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Research Article

Bibliometric analysis of disinfection by-product research trends in Türkiye

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ABSTRACT

The goal of this study is to reveal the time dynamics of studies systematically and comprehensively on drinking water treatment and disinfection, as well as the situation in the literature, by using the bibliometric analysis method to examine scientific publications in the field of "Disinfection By-Products" between 2001 and 2022. The data gathered from the investigated articles is shown using the visual mapping approach. In this regard, the research provides for an evaluation of the disinfection by-products literature. The study's database contained 115 scientific papers retrieved from Web of Science. Istanbul Technical University is the most productive university with 23 published articles on Disinfection By-products, followed by Suleyman Demirel University with 18 published articles. Trihalomethanes, haloacetic acids are the most studied types of carbonaceous disinfection by-products in published articles, and N-nitrosodimethylamine is one of the most widely published nitrogenous disinfection by-products. The precursors of disinfection by-products or the removal of disinfection by-products are the two main focuses of the purpose of all studies. Coagulation, advanced oxidation processes and membrane processes constitute the methods used in the control of disinfection by-products. Brominated, and nitrogenous DBPs have attracted much attention due to their high toxicity. Future studies on disinfection by-products should focus on water quality standards, precursor controls, toxicity, and health effects. The necessity of bibliometric analysis of disinfection by-products is a necessity to fill the existing knowledge gaps in global and regional studies.

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INTRODUCTION

In this study, either studies involving researchers located outside of Turkey or studies from universities that support disinfection by-products research in Turkey were used. However, the aim of this study is the bibliometric compilation of disinfection by-products studies completed in Turkey's water resources, regardless of where the researchers or research institutions are in the world. As a result of Web of Science search, disinfection by-products research was detected for the first time in our country in 2001. Three developmental stages were identified throughout the research. In the first part, disinfection by-products studies between 2001 and 2005 are preliminary studies of disinfection by-products (DBPs) in the country and generally include review studies. The studies in the second part are the studies that took place between the years 2005-2008

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and in this context, the studies that include monitoring studies of carbonaceous DBPs in our country. From 2008 to the present, nitrogenous DBPs and modeling studies have gained momentum in addition to C- DBPs.

The first study on DBPs in our country was carried out in 2001 [1]. Although this study was not carried out in Turkey, for the first time, there are researchers at Gebze Technology Institute in this study. In the study, the effect of water quality parameters was investigated in order to control the bromate formation after ozonation in biological activated carbon filter processes. Another of the first studies conducted in our country is a review article on the formation of DBPs in water and the effects of chlorine disinfection on health [2]. Another important article that made a difference in the early period is a review article investigating the disinfection of different wastewater sources with peracetic acid [3]. Although this article was not prepared about drinking water sources, it is important in terms of being inspiring.

The years in which DBPs studies were most striking were between 2004 and 2008. The first seasonal DBPs monitoring study in our country was carried out by spatio-temporal monitoring of total trihalomethane (THM) concentration at 22 different points in Ankara [4]. After this study, DBPs monitoring studies gained momentum. After the DBPs studies carried out in Ankara, DBPs studies were carried out in various reservoirs and water treatment plants in Istanbul [5–7]. In the same time period, the first DBPs monitoring study was carried out in İzmir [8]. The most comprehensive DBPs monitoring study carried out in our country was completed with formation potential tests in 29 different water sources from all regions of our country [9]. In 2005 Istanbul water resources, there are studies in which HAA analysis is carried out in addition to THM [10]. Studies on the removal of natural organic substances (NOM) that cause the formation of DBPs by advanced oxidation processes (AOPs) gained momentum in the early 2000s [11]. Studies on NOM removal have become popular in these years. In this period, NOM removal with adsorbents with different surface properties was also investigated [12]. In order to reduce the effect of dissolved organic substances (DOM), the effect of advanced oxidation and PAC was investigated in Istanbul water treatment plants [13]. Studies of different researchers on the precursors of DBPs are concentrated in this period [14]. In this study, researchers focused on the removal of DOM formations in low SUVA waters by adsorption of different resins and granular active carbon. Cancer risk study in Istanbul waters [15] and model studies established between water quality parameters and DBPs formation in Istanbul waters [16–18] which created awareness between 2004-2008 and guided other studies to be carried out. The effect of bromine as a water quality parameter on DBPs formation is also among the subjects studied in this period [19]. Different model studies have been developed for the rapid and practical evaluation of bromate formation in ozonated waters [20]. Another important study in this period is the toxicity studies from disinfection by-products [21].

When disinfection studies and DBPs knowledge increased, studies in many universities gained momentum since 2008. Until this period, monitoring studies carried out in surface waters only in Istanbul, Ankara and Izmir, as of this period, the monitoring of different city networks has started. Seasonal THM and haloacetonitrile (HAN) monitoring was carried out for the first time in İzmir city mains waters during this period [22]. It can be said that N- DBPs studies started with HAN monitoring. Another comprehensive study after the Istanbul, Ankara and Izmir studies is the study carried out in different processes of the Bursa drinking water treatment plant and measuring the concentrations of disinfection by-products [23]. There are studies in which THM monitoring was carried out in the water network for 15 months at 23 different points from Antalya well waters [24]. DBPs precursors were analyzed for 3 years in 6 different reservoirs in Konya [25]. In addition, studies were carried out in which THM and HAA analyzes were carried out at Altınapa Dam in Konya [26]. DBPs precursors and DBPs formation potential (FP) tests were carried out in Porsuk Stream, which supplies water to Eskisehir, and adsorption studies were carried out with different adsorbents for precursor removal [27]. In Aksaray water source, a precursor removal study was completed in order to reduce THM formation, similar to the study conducted in Eskisehir [28]. There are also studies to reduce THM formation in the water of Ulutan Lake in Zonguldak [29]. It carried its DBPs studies to a different point and carried out DBPs concentration measurement in swimming pools for the first time in Canakkale [30]. This study on swimming pools may have inspired the swimming pool studies completed in Bitlis [31] and Eskisehir [32]. The cities where DBPs studies are carried out in our country are shown in Figure 1.

In recent studies, DBPs studies have gained a different dimension and the formation of N-Nitrosodimethylamine (NDMA) from dissolved organic nitrogen (DON) forms from N- DBPs species has been investigated [33]. As of 2008, studies on N- DBPs types tend to increase. The effects of the use of chlorine and chloramine as an alternative disinfectant in drinking water treatment plants on the formation of THM and NDMA have also started to be included in research topics [34]. It can be said that halonitromethane (HNM) measurement was made for the first time in another study, which entered the literature in our country and had a researcher(s) from Turkey in the study team [35]. Similarly, more extensive studies have been conducted on the formation of C- DBPs and C- DBPs in waters affected by DBPs precursors in water resources [36]. Tests of the measurement methods of DBPs types with different techniques have begun to be carried out. Contrary to known methods, studies aiming to measure NDMA with LC-MS/MS technique are available in the literature [37]. NDMA formation mechanisms were also tried to be understood by

Figure 1. Cities in Turkey where seasonal DBPs monitoring is carried out.

using chlorine and chloramine as disinfectants in drinking water and wastewater samples [38]. A comprehensive investigation of NDMA formation was also carried out using different disinfectants in different water sources [39]. The studies in which NDMA formation potential tests were carried out in waters with and without wastewater interaction using chlorine dioxide and chlorine after pre-oxidation aimed to measure wastewater contamination in water resources [40]. The effects on the water resources affected by natural disasters in the USA and therefore on DBPs types (two C- DBPs and two N- DBPs) were investigated [41]. One of the most comprehensive studies completed in 2022, 4 THM, 9 HAA, 6 HAN and 9 HNM species in water distribution systems using different water sources such as Isparta, Antalya-Konyaaltı, Antalya-Kumluca and the formation potential tests of 28 DBPs species in Egirdir Lake and Karaagaç Natural Water Source (Kumluca) have been tested for one year [42].

In this study, bibliometric analysis of DBPs studies between 2001-2022 was carried out. In this context, studies have evolved from laboratory-scale studies to field-scale studies and finally to model studies. As a result of the developing technology and toxicity studies, while C- DBPs studies were on the agenda in the first years, N- DBPs studies were added to C- DBPs studies in the following years and their number is increasing. DBPs precursors removal studies, DBPs laboratory studies, DBPs water network studies and DBPs modeling studies maintain their importance and up to date. The main purpose of this study is to determine the deficiencies and future demands of DBPs studies.

METHODOLOGY

The study's goal was to conduct a bibliometric analysis of works published between 2001 and 2022 that had the terms "disinfection by-products" in the abstract, keywords, and title sections. The bibliometric study was shown using the VOSviewer (1.6.19) package software. The bibliometric analysis approach entails using statistical tools to determine qualitative and quantitative changes in a certain scientific study topic, to profile past publications on the subject, and to highlight patterns within a discipline. The method's objective is to evaluate scientific papers and deliver the results to scientists or other stakeholders, which makes it beneficial. The subjects investigated using this approach, the authors working on these subjects, and their distribution by country or publishing type are statistically evaluated, allowing the overall state of a given field to be presented in accordance with the data acquired.

VOSviewer (1.6.19) was used in this study to visualize the bibliometric analysis approach. VOSviewer is a software application that allows you to create and see maps based on network data. The goal of visualization is to make it easier to interpret vast amounts of complicated data by showing significant characteristics of the data. Furthermore, the data gathered with VOSviewer is displayed, giving researchers with more accurate information as well as visual metadata.

The bibliometric analysis procedure is divided into three major stages: search criteria determination, database selection, and data analysis [43]. The first stage involves determining the search criteria. At this point, the researchers were identified by scanning the database for disinfection by products topics. The gathered data were then classified based on the subject's significance, the features of the scientific papers, and the publication time. The second stage is to calculate the number of articles by picking scientific papers based on the search parameters in the databases. The data analysis and visualization step are the third and final stage. The stages of the bibliometric analysis process is shown in Table 1.

Table 1. Stages of the bibliometric analysis process

Stages of the process	Selection criteria	Result
Search criteria	Research and analysis of terms used by researchers in databases on disinfection by-products Characteristics of selected scientific publications and determination of publication period	Title, summary, and keywords ("Documents type: Article"), ("Countries/Region: Turkey"), (Web of Science Categories: Environmental Sciences or Engineering Environmental or Water Resources) or ("Web of Science Index: Science Citation Index Expanded-SCI Expanded")
Selection of database	Determination of the number of scientific publications in the Web of Science database	Web of Science – 115 Science Citation Index Expanded
Data analysis	Visualization of bibliometric analysis	VOSviewer

PERFORMANCE OF PUBLICATIONS

Publication Output

According to the data obtained from the Web of Science database for the research on the disinfection by-product; Between 2001 and 2022, "All Fields" disinfection by-products were searched. As a result of these searches, a total of 115 scientific publications were reached. All these publications are SCI-Expanded studies.

The distribution of these scientific publications by years between the period of 2001-2022 is given in Figure 2. As seen in Figure 2, it has been observed that scientific publications about disinfection by-products have increased from time to time and have found a field of study. The year in which the most scientific publications were produced was 2007 with 12 articles, while the year in which the least publications were produced was 2001 with 1 article. As a result, it can be said that the issue of disinfection by-product as a working area has gained importance in recent years.

Subject categories, journals, and institution

The geographical examination of the researchers who contributed to the literature on disinfection by-products with scientific publications is presented in Figure 3. There are 112 institutions that have contributed to the literature, but the top 10 institutions are included in the graph. Istanbul Technical University and Suleyman Demirel University are at the top in terms of the number of scientific publications and these institutions are among the institutions that have an important place in disinfection by-products. In the research of disinfection by-products on Web of Science, 19 different subject categories were determined. The articles in the

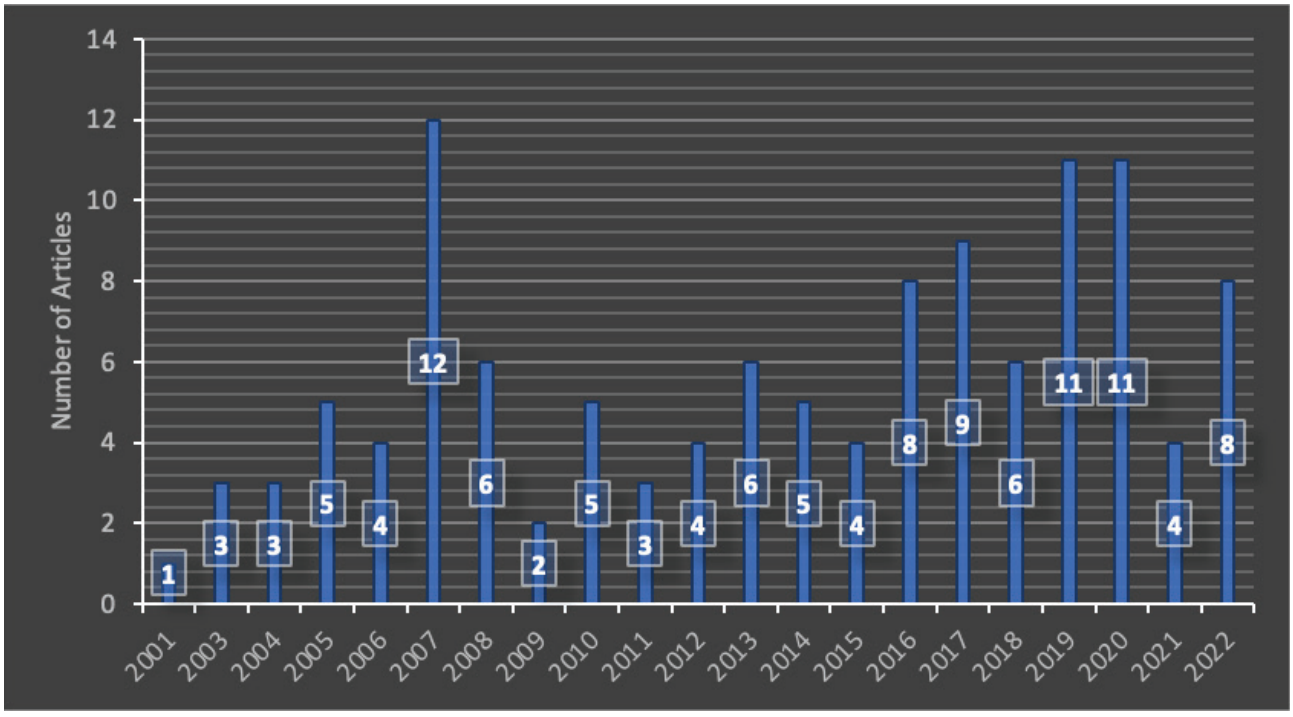

Figure 2. Distribution of scientific publications on disinfection by-products for the period 2001–2022 by year.

Figure 3. Distribution of scientific publications on disinfection by-products by institutions between 2001-2022.

Environmental Sciences category constitute 67% of the published articles. The second most common topic category is Engineering Environmental with 41 published articles, and the next topic category is Water Resources with 39 published articles. According to statistics, 115 scientific articles have been published in 42 different SCI-Expanded journal types.

Cluster sizes indicate the excess use of keywords, cluster colors mean that keywords are used together. The lines between the clusters show that the keywords are in relation to each other. As seen in Figure 4, the concepts frequently used with disinfection by-products were determined as trihalomethanes, ozonation, drinking water, humic acid, chloramination, toxicity, disinfection.

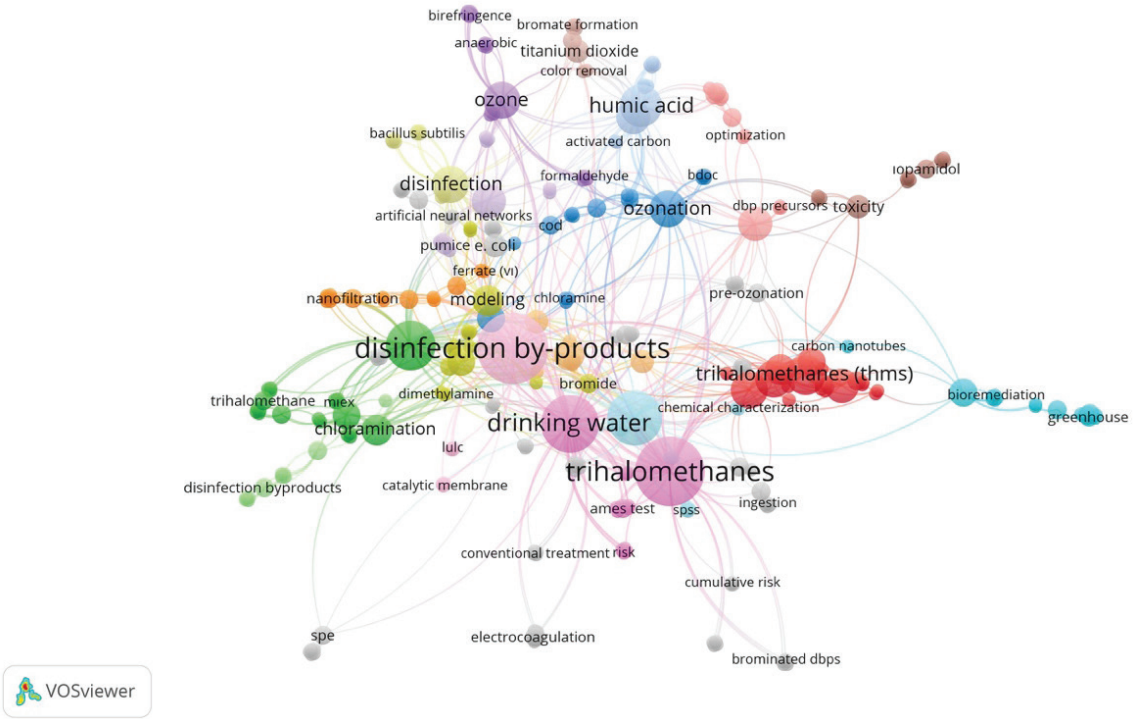

Figure 4. Bibliometric network analysis of keywords used in scientific publications on disinfection by-products for the period 2001-2022.

The bibliometric analysis conducted between 2001 and 2022 shows that Mehmet Kitis is the most productive author. Mehmet Kitis carried out his first study in the field of DBPs in 2004. The mentioned author is also the most cited author as a result of the bibliometric study with this article. Mehmet Kitis carried out his studies on DBPs in 2004 (2 articles), 2006, 2007 (5 articles), 2009, 2010 (3 articles), 2018. The scope of his work focuses on review articles and natural organic matter removal in the first years, and in the following years he focuses on the negative effects of DBPs species on human health. In addition, the related author's studies on multiple linear regression model studies between DBPs species and water quality parameters and on precursor removal using membrane processes are included. Thanks to the articles written by the mentioned author according to the needs of the period, he has made him one of the most effective researchers. The status of the authors according to the number of published articles is shown in Figure 5, and the ranking of the most cited articles is shown in Table 2.

ATTENTION-RAISING ISSUES

Although membrane processes were first used in water and wastewater treatment applications in the 1960s, a significant increase in the number of articles is observed with the beginning of the 2000s [44]. Membrane processes and water treatment DBPs have also added a different dimension to their studies. Removal of THM and HAA precursors by using nanofiltration (NF) membrane technology has started to be used in our country since 2008 [45]. Ultrafiltration (UF) membrane technology, as well as NF membrane technology, continued to be used in the subsequent years to remove the precursors of DBPs [46]. The removal of NOM, which are DBPs precursors, has been studied in UF ceramic membranes from membrane technologies [47].

With the speciation of disinfection by-products, the presence of much more cytotoxic and genotoxic DBPs species compared to THM and HAA has been detected [48]. In our country, there are studies investigating the genotoxic and carcinogenic effects of DBPs species [49]. In order to determine the health effects of DBPs through ingestion, dermal contact, and inhalation, a remarkable study was conducted [50].

Organic substances, inorganic substances, algal organic substances, and disinfectants such as chlorine, chloramine, chlorine dioxide and ozone must be present in the aquatic environment for the emergence of disinfection by-products [51] . Therefore, in DBPs control, either precursors should be reduced, or disinfectant doses should be reduced [52]. Along with the precursors of DBPs, studies for the simultaneous removal of endocrine disrupting chemicals and pharmaceutical personal care products have started [53]. In this context, it has been investigated that more than one pollutant group can be removed at the same time. Removal of algal organic matter (AOMs) that occurs as a result of eutrophication, a common problem today, has also been studied [54]. THM concentrations can be estimated in the developed multiple linear regression (MLR) models [55]. In this study, single-walled carbon nanotube and multi-walled carbon nanotube were added to the coagulation process as an innovative approach.

Figure 5. Number of articles published by the most productive authors.

Table 2. Top 10 most cited articles

Author(s)	Article Name	Journal	Citations	Year
Kitis, M	Disinfection of wastewater with peracetic acid: a review	Environment International	487	2004
Ates, N; Kitis, M and Yetis, U	Formation of chlorination by-products. in waters with low SUVA-correlations with SUVA and differential UV spectroscopy	Water Research	179	2007
Pehlivanoglu-Mantas, E and Sedlak, DL	Measurement of dissolved organic nitrogen forms in wastewater effluents: Concentrations, size distribution and NDMA formation potential	Water Research	147	2008
Selbes, M; Kim, D; Ates, N; Karanfil, T	The roles of tertiary amine structure, background organic matter and chloramine species on NDMA formation	Water Research	107	2013
Yildiz, YS; Koparal, AS and Keskinler, B	Effect of initial pH and supporting electrolyte on the treatment of water containing high concentration of humic substances by electrocoagulation	Chemical Engineering Journal	100	2008
Uyak, V and Toroz, I	Investigation of bromide ion effects on disinfection by-products formation and speciation in an Istanbul water supply	Journal Of Hazardous Materials	87	2007
Uyak, V; Yavuz, S; Toroz, I; Ozaydin, S; Genceli, EA	Disinfection by-products precursors removal by enhanced coagulation and PAC adsorption	Desalination	85	2007
Uyak, V; Koyuncu, I; Oktem, I; Cakmakci, M; Toroz, I	Removal of trihalomethanes from drinking water by nanofiltration membranes	Journal Of Hazardous Materials	84	2008
Kitis, M; Kaplan, SS; Karakaya, E; Yigit, NO; Civelekoglu, G	Adsorption of natural organic matter from waters by iron coated pumice	Chemosphere	81	2007
Kirisits, MJ; Snoeyink, VL; Inan, H; Chee-Sanford, JC; Raskin, L; Brown, JC	Water quality factors affecting bromate reduction in biologically active carbon filters	Water Research	78	2001

DBPs studies are promising for future research. The trend of DBPs studies in the world shows a continuous increase over the years. The main reason for this is that although only an upper limit for THMs has been set by legislation in Turkey, different DBPs groups have also been included in the legislation in developed countries. In addition, while articles on C-DBPs are generally published in our country, the number of articles published on nitrogenous, brominated, and iodinated DBPs is higher in developed countries. Nitrogenous, brominated, iodinated DBPs are much more cytotoxic and genotoxic for human health. Therefore, tendencies towards these types DBPs should be increased, supported, and investigated. After these research are completed, the gap between the knowledge of DBPs of developed countries and the knowledge of DBPs of Turkey will begin to close.

CONCLUSIONS

10491 studies were found by searching the disinfection by-products in the Web of Science database. After selecting the document type "Article", country/region "Turkey", the web of science category "Environmental Sciences, Environmental Engineering, Water Resources, Chemical Engineering", and the web of science index "SCI Expanded", the number of research articles decreased to 115. The 115 articles obtained were limited to 19 different subject categories and 42 different journals. Most of the articles belong to Environmental Sciences subject category. The journal of Water Research is recognized as the most productive journal with 12 published articles. 106 institutional articles were published in 42 different journals. These articles include studies carried out either by native or foreign researchers in institutions in Turkey or by local researchers working in institutions located outside the country. Among all institutions, Istanbul Technical University is the institution with the highest number of articles published, and Suleyman Demirel University is the second institution with the highest number of articles published. Mehmet Kitis from Suleyman Demirel University can be considered as the most influential person on disinfection by-products, as he is the most cited author. The most used ones according to the keyword analysis are trihalomethanes, ozonation,

drinking water, humic acid, chloramination, toxicity, disinfection. Trihalomethanes were the most analyzed disinfection by-product type between 2001-2022 in Turkey. Control of the precursors of disinfection by-products is the main objective of many articles. The coagulation process, membrane processes, and advanced oxidation processes are the main treatment methods used to control disinfection by-products. In addition, the formation of disinfection by-products in water distribution networks and their toxic effects on health should also be examined. Toxic disinfection by-products should be included in water quality regulations for drinking water safety.

CONFLICT OF INTEREST

The authors declare no potential conflicts of interest regarding the research, authorship and/or publication of this article.

DATA AVAILABILITY

The data used to support the findings of this study are included within the article.

AUTHOR'S CONTRIBUTIONS

All author is contributed equally to bring out this article.

ETHICS

There are no ethical issues with the publication of this manuscript.

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Research Article

Acoustic safety assessment of palm oil mill workers

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ABSTRACT

Mechanization of work processes in agro-based industries through the employment of improved tools and/or equipment powered by either fuel or diesel engines reduce tedium and drudgery on the workers as well as improve the overall productivity and production. However, it also leads to health threat to the workers through environmental noise pollution. This study assessed the occupational noise exposure of workers in three different palm oil mills. The noise level was measured using a noise metering device (model 1352) and noise descriptors (L10, L50, L90, Lav, LAeq, Total Sound Pressure Level (Tspl), DOSE (%) and TWA). The occupational health effects of noise exposure were deduced using a semi-structured questionnaire. The noise exposure was assessed during the palm nut digestion, and nut and pulp separation job operations as the noise emanates from the diesel powered engine, palm nut digester and nut and pulp separator. The average noise levels obtained from the palm oil mills during palm nut digestion, 99.36 (SD = 4.16) dBA and pulp separation, 98.18 (SD = 3.07) dBA, exceeded the recommended 85 dBA for an 8-hour exposure period permissible noise exposure limit standard by the NIOSH. The independent t-tests for noise level between off-working and working periods disclosed $p < 0.05$ – significant noise exposure. The self-reported effect of the noise exposure level on the workers was interference with communication - 100%, tinnitus - 70%, ringing sensation - 76.75%, and noise stress - 73.30% of the workers. This research will inform the operators of the need for appropriate personal protective equipment usage and equip Government regulatory agencies to make appropriate regulations in order to protect oil mill workers.

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INTRODUCTION

The oil palm tree is one of the economic crops in Nigeria whose by-products has crucial role in food production, raw materials provision for industries, income generation, employment set up, foreign exchange earnings, and national economic development [1]. Among the oil palm tree products are palm oil. Dada et al. [1] documented that before the 1970s, palm oil alone was one of the major sources of foreign exchange earning to Nigeria. Its significance was observed between the year 1950 and 1960 as the palm oil produced in Nigeria made an average of 34% of total world palm oil export in the world. The physical strenuous and repetitive activities involved in the palm oil production process affected the interest in its production. With the mechanization of the work process in most agro-based industries, palm oil production industry inclusive [2], the overall productivity and production process, as well as the reduction in tedium and drudgery from the work processes, has been positively affected. This transformative component of development in itself has significantly and necessarily improved physical stress issues in the work environments [3].

Some of the industrial machines acquired and used in most work processes are adapted with internal combustion engines while some due to the electric power supply challenge in the nation, are alternatively powered by fuel or diesel engines which inescapably generate noise in the work environment as such poses health threat to the workers [4-7]. Tekin et al.'s [8] studies observed that even though exposure to excessive noise pollution in the work environment affects the workers' health, the study concluded that there was no statistically significant difference in the attention level of the workers before and after

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machine operations. Ibhadode *et al.* [9] stated that encouragement of industrialization in the face energy insufficiency or crisis prevalent in most third world nations, Nigeria inclusive, effectuates environmental noise pollution through diverse privately owned and operated sources of power. The noise intensity being emitted by industrial machinery during job operations cannot be overemphasized as it is classified among the occupational hazards that significantly affect the health of workers in most work environments [10, 11].

The harmful effect of exposure to the high intensity of noise on the workers' health may be immediate, long-term or both. This covers a range of psychological, physiological, and perhaps pathological responses [12]. Occupational diseases caused by exposure to noise are stated to be preventable. To address the issue of noise in the working environment, the background information on the work factors should be assessed. The steps in an effective noise exposure prevention program consist of the identification of sources of noise, the intensity, effects and the proposed appropriate precautionary and/or control measures. Oil palm mill has come to stay considering that palm oil is an important product in world trade [13] and is playing an important role in Nigerian economy [14]. Previous scientific studies on noise level measurements and evaluations found in the works of literature in different occupational dispensations included evaluation and analysis of the intensity of noise from various generators in the commercial areas [15], distraction effect of excessive noise pollution from mining machines during mining operations [8], assessment of noise emission from vibrator-block factories and the impact on human health [16], and occupational noise exposure in an amassed sawmill site was evaluated and analyzed by Azodo et al. [17]. However, no publicly available published literature was found that assessed the occupational exposure of workers in the palm oil mill.

Chong et al. [18] opined that recognition and prevention of occupational noise risks are crucial for the improvement of occupational health risks perception and regulation in the work environment. It is therefore, necessary to assess the set of conditions, limits and forces which surround and have direct influence on the effective and efficient operation of mechanized palm oil production process. This study assessed the occupational noise exposure of palm oil mill workers using noise metering device and noise descriptors, as well as determine the subjective effects of the noise exposure of mechanized palm oil production process. This research provides the needed information for oil mill operators and Government regulatory agents to act on in order to improve the occupational health of oil mill workers.

MATERIALS AND METHOD

The physical occupational noise exposure measurement from mechanized palm oil process was conducted in three palm oil mills located in Ogidi, Idemili North Local Government Area of Anambra State, Nigeria (Figure 1). These palm oil mills were purposefully selected as study sites for this study. The palm oil mills identified by the names of the operators who also were the owners of the mills were identified with English alphabets A, B and C for this study. The geographical coordinates of the palm oil mills obtained with a handheld Garmin Global Positioning System (GPS) 72H (Garmin Ltd. Kansas, United States) are presented in Table 1.

The palm oil operators were contacted at their work environment and intimated about the study; the purpose, their detailed involvement in the study, as well as their permission. The consent

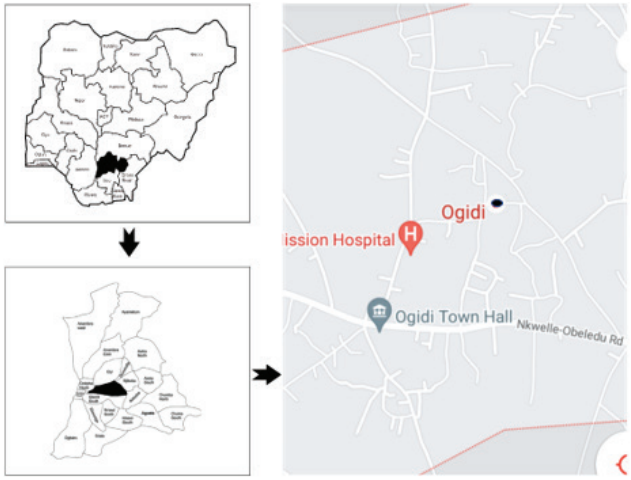

Figure 1. Map, showing the study site location.

Table 1. Geographical coordinates of the palm oil mills assessed

Palm oil mills	Geographical coordinates	
	Longitude	Latitude
A	N 6°8'42"	E 6°54'26"
B	N 6°9'20"	E 6°54'37"
C	N 6°8'47"	E 6°54'8"

was obtained verbally from the three palm oil mill owners. A pre-survey of the study site locations (palm oil mills) revealed that palm nut digestion, and nut and pulp separation were the mechanized palm oil production process common in the three palm oil mills. These machines were powered by diesel engines in the three palm oil mills. Other features found similar in three palm oil mills is the production floor layout of the palm oil mills. The factory sections showing the palm nut digester, nut and pulp separator and the diesel-powered engine used in the palm oil job operations is presented in Figure 2. The plant layout for the palm oil mills showing the points from which the measurement was obtained is presented in Figure 3 below. Table 2 explained the

Figure 2. The palm oil mills showing palm nut digester, nut and pulp separator and the diesel-powered engine.

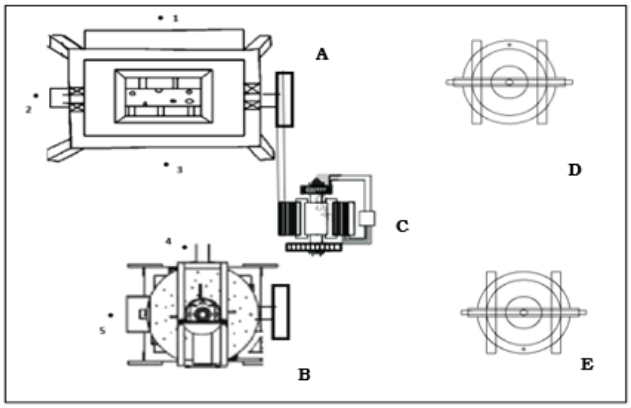

Figure 3. The schematic plan of the palm oil mills showing the measurement points (A= Pulp separator, B = Palm fruit digester, C = Diesel engine, D = Palm oil press E = Palm oil press).

measurements points noted in Figure 3 from which the reading where obtain in the palm oil mills.

Verbal conversation with the operators also revealed that the production process rate is affected by the number of customers per day which is higher in the dry seasons with an average of 600 – 700 kg of palm nuts handling per day. This guided the study period as it was conducted in February and March 2019.

The noise exposure level in the work environments was measured at the ears of the workers in the different duty positions of the workers during the palm nut digestion, and nut and pulp separation job operations. The physical measurement of the noise levels was done using Benetech Sound Level Meter (SLM) type GM1352 internally calibrated by Benetech (Shenzhen Jumaoyuan Science and Technology Co., Ltd., China). The response frequency of the SLM is 31.5 Hz – 8 kHz at an accuracy of ±1.5 dB. The resolution setting of the SLM is 0.1 dB. Its measurement range is SLM is 30 – 130 dB. The noise exposure level at the palm oil mills was measured and recorded before and during each of the work activity at an exposure time interval of 60 seconds using the Benetech sound level meter and a digital professional handheld liquid crystal display (LCD) stopwatch (Shenzhen, China) during the job operations.

Five sets of measurements were taken for each of the mechanized palm oil production operations, making a total of 10 readings for each of the palm mills and 30 readings for the three palm oil mills assessed. The measured sound level at the palm oil mills was computed and expressed as A-weighted equivalent sound pressure level (LAeq) using equation (1). The value obtained represented the total sound energy being produced throughout the assessment.

$$L_{Aeq} = 10log_{10}\left[\frac{1}{N}\sum_{I=1}^{N}\left(anti\ log\frac{L_{Ai}}{10}\right)\right] \quad (1)$$

Where

LAeq = A-weighted equivalent sound pressure level

LAi = A-weighted sound pressure level in dB

N = total number of measurements

The independent samples t-test analyses were used to establish if there was a significant statistical difference between (a) off- and working periods at the palm oil mills and (b) working periods at the palm oil mills and maximum permissible noise level exposure.

The workers' occupational noise exposure analysis was carried out on noise exposure limit recommended by National Institute for Occupational Safety and Health (NIOSH) for A-weighted decibel of 8-hr time-weighted average (85 dBA as an 8-hr TWA). The relation between exposure levels and duration was determined using equation (2)

$$T_{SPL} = \frac{480}{2^{(L-85)/3}} \quad (2)$$

Where

T_{SPL} = Exposure duration of the sound pressure level

L = exposure level and

3 = the exchange rate

The daily noise dose of occupational noise exposure was determined by the periods of different noise levels, the daily dose (D) by each of the participants by the relation

$$D = \sum\frac{C_n}{T_{SPL}} \quad (3)$$

Where

C_n = total time of exposure at a specific noise level, and

T_{spl} = exposure duration for which noise at this level becomes hazardous

The value obtained for the daily dose can be converted into an 8-hr TWA using the relation

$$TWA = 10.0 \times (log\frac{D}{100}) + 85 \quad (4)$$

The obtained Time Weighted Average (TWA) noise levels for each of the workers were subjected to acoustic safety and health risks analysis using equation (5). The output was classified as safe and unsafe noise exposure levels using the revised recommended standard criteria for occupational noise exposure by National Institute for Occupational Safety and Health presented in Table 3 as a guide.

$$TWA = \sum\frac{(wf_i \cdot t_i)}{\sum t_i} \in [0,1] \quad (5)$$

$$\begin{bmatrix} TWA_{unsafe} \text{ with } wf_i = \begin{bmatrix} 1\ if\ I_{sound\ level} > I_{85\ dBA} \\ 0\ if\ I_{sound\ level} \leq I_{85\ dBA} \end{bmatrix} \\ TWA_{safe} \text{ with } wf_i = \begin{bmatrix} 1\ if\ I_{sound\ level} < I_{85\ dBA} \\ 0\ if\ I_{sound\ level} \geq I_{85\ dBA} \end{bmatrix} \end{bmatrix}$$

Table 2. The noise level measurement points and the processing activity in the palm oil mills.

Noise level measurement points	The concerned worker	Job operation involved
1	Oil mill worker	Collection of the palm fruit pulp
2	Oil mill worker	Collection of palm kernel
3	Machine operator	Feeding in the digested palm fruit in the palm fruit separator and monitoring the process
4	Machine operator	Feeding in the palm fruit in the palm nut digester and monitoring the process
5	Oil mill worker	Collection of the digested palm fruit

Table 3. Acoustic risks and safety analysis interpretation of the noise intensity level from lawnmowers on groundskeepers for 85 dBA as an 8-hr TWA

TWA noise levels	85 dBA as an 8-hr TWA exposure level interpretation
≤85dBA	safe noise exposure levels
≥85dBA	unsafe noise exposure levels

Where

TWA = Time weighted average noise levels

$wf_{i=}$ A-weighting factor variable which depends on $I_{sound\ level}$ values

t_i = Time in hours

The self-reported effect of noise exposures a result of the job operations in the palm oil mills was assessed using a questionnaire. The questionnaire was developed after a review of previous studies on the effect of noise on humans. The questionnaire covers the physical and physiological effect of noise on the participants. The questionnaire developed for this study was subjected to scrutiny for validity and reliability by two professionals in acoustic and human safety engineering. The questionnaire also underwent a pre-test for clarity and the precise presentation of questions. The questionnaire underwent a series of edits and modifications as suggested by the professionals regarding the objective of the study, who later gave their approval for the production of the final copy of the questionnaire used for the study. The proposed participants in this survey who were workers in the palm oil mills were contacted face-to-face for participation in the study by the researchers, informed of the study, its purpose, and procedure, and verbally assured of the confidentiality of their responses to the questionnaire. Responses were only obtained from contacts who agreed to participate in the study. The participation was voluntary, and as such, no incentive was offered. The analysis of the obtained data from the survey was carried out using Statistical Package for Social Science (SPSS 20.0) software and Microsoft Excel spreadsheet version 2001.

RESULTS AND DISCUSSIONS

The sources of the noise were mainly the diesel-powered engine (the power drive source), palm nut digester and nut and pulp separator. The palm nut digester and the nut and pulp separator machines were driven by diesel powered engine in the three palm oil mills assessed. The detailed noise level measured in the oil mill includes off work hours and working hours (Apendix 1) during the working hours two categories of measurements were made including noise level from palm nut digestion and noise level from pulp separation process (Apendix 2).

Table 4 shows the descriptive statistics of the noise level in three different palm oil mills during the job operation the palm nut digestion, and nut and pulp separation job operations.

The healthful and safe environment need of industry covers the business objectives as well as that of the individual employee. Deficiencies, inadequacies, and negation of the human factor's aspect for the effectiveness and efficiency of the workplace affect the whole work process. This study which focused on the acoustic safety of palm oil mill workers using a digital noise metering device and noise descriptors considered the intensity of noise for exposure duration as noise has the characteristics of negating the workers' health life in the work environment. The measured sound levels from the palm oil mills and the computed noise descriptors from the data obtained are presented in Table 5.

The variation of the measured noise level intensity between the off-working and the working hours analyzed using independent-sample t-test showed that the occupational noise levels during working process were statistically significantly higher (mean = 98.80 ± 3.75) when compared to off-working periods (mean = 59.60 ± 6.41) with $p < 0.001$ (Table 6). The obtained noise intensity level during the job operation (with mean value of 98.8 from 1800 measurements) was higher than the recommended permissible noise exposure limit standard by NIOSH (85). This implies that noise levels during the job operation superseded the recommended as permissible noise exposure limit standard.

The A-weighted equivalent sound pressure level (LAeq) computed so as to obtain a single constant noise level value that represented an equivalent total sound energy the palm oil mills workers were exposed to during their job operation over the assessment period showed a range of 94 - 109, 94.4 - 106.4, 96 - 108 dBA for

Table 4. Descriptive statistics of the measured noise level during the off hours and working hours

Descriptive statistics	A			B			C		
	OH	PD	PS	OH	PD	PS	OH	PD	PS
Max	78.50	108.5	112.5	81.20	107.3	114.6	75.9	107.7	111.6
Min	50.00	93.00	91.8	51.70	93.00	91.8	48.40	94.70	94.70
Mean	59.82	97.88	99.35	61.56	97.87	99.33	57.61	98.79	99.41
SD	6.33	3.04	4.52	6.39	3.26	4.91	5.90	2.81	2.75

Note: OH= Off working hours, PD = Palm nut digestion, PS = Pulp separation

Table 5. Descriptive statistics of the measured noise level during the off hours and working hours

Descriptive statistics	A		B		C	
	OH	WH	OH	WH	OH	WH
Max	78.50	114.60	81.20	111.60	75.90	107.70
Min	50.00	91.80	51.70	93.00	48.40	94.70
Mean	59.82	99.34	61.56	97.88	57.61	99.09
SD	6.33	4.71	6.39	3.18	5.90	2.77

Note: OH= Off working hours, WH= Working hours

Table 6. Independent sample t-test for noise level between off-working and working periods

Descriptive statistics					t-test for Equality of Means		
Measurement periods	**N**	**Mean**	**SD**	**SEM**	**T**	**Df**	***P*-value**
Off-working	1800	59.67	6.41	0.15	-223.42	3598	0.00
Working hour	1800	98.80	3.75	0.09			

palm oil mill A, B and C respectively. The daily dose (D) of the noise exposure for each of the palm oil mills as a result of the job operations showed that a maximum daily dose of 208.3% for palm oil A, 130.8% for palm oil B and 174.5% for palm oil C while the minimum daily dose was 52.9% for palm oil A, 52.7% for palm oil B and 53.0% for palm oil C. To ascertain the workers' safety, the value obtained for the daily dose was converted to an 8-hr TWA. The workers' occupational noise exposure analysis carried out on noise exposure limit recommended by National Institute for Occupational Safety and Health (NIOSH) for A-weighted decibel of 8-hr time-weighted average (85 dBA as an 8-hr TWA) gave a range of 90.3 - 105.8, 90.3 - 98.1 and 90.3 - 102.5 dBA (Table 7). This showed that the occupational noise exposure on the workers in the three-palm oil exceeded the noise exposure limit recommended by National Institute for Occupational Safety and Health (NIOSH) for A-weighted decibel of 8-hr time-weighted average (85 dBA as an 8-hr TWA) which was not healthy for the workers. L_{10}, the noise descriptor, represented the mean of the lower limit of the fluctuating noise value obtained in this study, and it was recorded for the palm oil mill A. The upper limit of the noise level (L_{90}) to which the workers were exposed when compared to the three palm oil mills, A, B, and C, showed that the value obtained for palm oil mill A was the highest. Other analyses of the noise descriptors, including L_{50}, L_{av}, T_{spl}, DOSE (%), and TWA, were present in Table 7. The result of the noise descriptor analysis presented in the table showed that workers in palm oil mill A were exposed to the highest noise level values, with average values recorded as L_{Aeq} (101 dBA), L50 (98.6 dBA), L_{max} (105.5 dBA), and T_{spl} (27 dBA).and a noise dose percentage of 80.3. This study is proof that the frequent increase in the sophistication of machinery, which replaces manual labor in our industries relentlessly, adds to the degree of noise pollution in every corner of the world's environment.

Work situation characteristics which comprise of what happens to the individual in his work environment as a human is one of the greatest assets visible in any organization essential for achieving the set tangent. The identification of the effects of noise intensity which is imperative in understanding the relationship between the workers and the work environment as the judicious commitment to the whole system of safe work process and environment for the reduction of accidents at work and rates of occupational diseases makes the whole system economically active populations [19]. The acoustic safety analysis for the noise exposure done with a one-tailed criterion (see equation 5) following the recommended exposure time guideline for continuous time-weighted average noise exposure split into two bins (unsafe and safe noise intensity level) for A-weighted decibel of 8-hr time-weighted average (85 dBA as an 8-hr TWA). The time-weighted average which indicates the safety and the occurrence frequency of noise exposure to sound pressure showed absolute unsafe noise exposure level above the NIOSH limit (Table 8).

The detrimental effect of a high level of noise on human health has both immediate and long-term effects on the workers. The range of self-reported effect of the noise exposure level on the 30 workers from the three palm oil treadmills who participated in this study were interference with communication, tinnitus, ringing sensation, and noise stress (Figure 1). This study depicts that occupational noise affects human health and well-being.

Table 7. Descriptive statistics of the computed noise descriptors

Descriptive statistics	L10	L50	L90	Lmax	LAeq	Tspl	DOSE (%)	TWA
Palm oil mill A								
Max	102.9	109.2	112.6	114.6	109	64.7	208.3	105.8
Min	92.2	92.2	92.2	92.2	94	3.4	52.9	90.3
Mean	96.4	98.6	103	105.5	101	27	80.3	93.0
Palm oil mill B								
Max	101.8	105.3	108.8	111.6	106.4	55.1	130.8	98.1
Min	93.3	94	94.9	95.2	94.4	4.5	52.7	90.3
Mean	96	97.5	100.3	101.9	98.6	27.5	60.7	91.1
Palm oil mill C								
Max	101	108.3	111	112.6	108	38.1	174.5	102.5
Min	95	95.6	96.5	96.7	96	3.8	53.0	90.3
Mean	97.4	98.9	101.4	102.8	99.8	20.0	57.0	90.7

Table 8. The A-weighted decibel of 8-hr time-weighted average safety analysis

Palm oil mills	Noise exposure levels (dBA)	Operation Characteristics	Frequency(percentage)
A	93.0	Unsafe	60(100%)
B	91.1	Unsafe	60(100%)
C	90.7	Unsafe	60(100%)

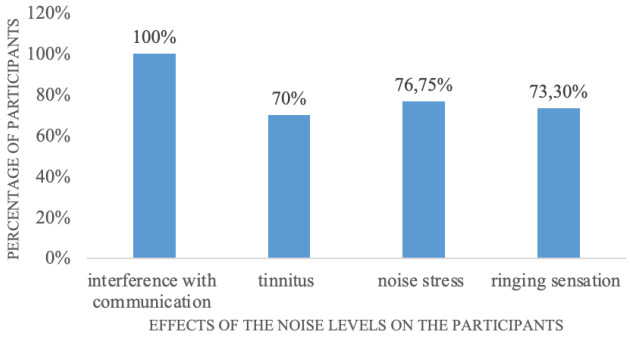

Figure 1. Effects of the noise levels on the palm oil mill workers.

CONCLUSION

The identification of noise intensity and its effects on the occupational health of the palm oil mill workers were measured and analyzed using the noise descriptors (L10, L50, L90, Lav, LAeq, Total Sound Pressure Level (Tspl), DOSE (%), and TWA). The values obtained were high when compared to the noise exposure limit recommended by the National Institute for Occupational Safety and Health. Further analysis of the data showed an unsafe noise exposure level above the recommended noise exposure limit in the palm oil mills. The self-reported effect of noise exposure as a result of the job operations in the palm oil mills was assessed using a questionnaire that showed that the level of noise exposure in the palm oil mills translated into interference with communication, tinnitus, ringing sensations, and noise stress effects on workers. Considering the crucial role palm oil plays in human food availability, foreign exchange earnings, and national economic development, workers' health must be protected from the ill effects of noise. This study, therefore, recommended the use of personal protective devices such as ear mufflers to reduce the intensity of noise exposure for the workers.

CONFLICT OF INTEREST

The authors declare no potential conflicts of interest regarding the research, authorship and/or publication of this article.

DATA AVAILABILITY

The data used to support the findings of this study are included within the article.

AUTHOR'S CONTRIBUTIONS

All authors are contributed equally to bring out this article.

ETHICS

There are no ethical issues with the publication of this manuscript.

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Research Article

Evaluating compost for hydrogen and methane rich gas production via supercritical water gasification

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ABSTRACT

The compost produced from organic wastes (MSW, city market's wastes and wood dust) was selected to be processed via supercritical water gasification (SCWG) in order to produce gas product consisting of hydrogen and methane mainly. The effects of parameters such as temperature, reaction time and KOH as an additive were determined and around 55 vol.% of H_2 and CH_4 in the gas product was found after 30 min reaction time together with KOH, at 500 °C. The red mud catalysts did not improve the gasification yields even though they increased the calorific value of the product gas.

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INTRODUCTION

Utilizing organic wastes as a resource for clean energy and chemical feedstock production has been gaining much attention recently since the global municipal solid waste (MSW) production was recorded as 2.01 billion tonnes per year according to a report published in 2018 by Worldbank. Unfortunately, around 33% of this waste is not managed properly and creating risks to the environment [1]. Currently the main disposal method of wastes is landfilling, as 37% of waste produced is landfilled globally. 33% of total waste is managed by open dumping which is commonly seen in economically low developed countries. The share of recovery through recycling and composting remains at 33% and 11% of the waste is incinerated for energy production [1]. Turkey as a developing country produced 32.2 million tons of MSW and 67.2% of it was landfilled to controlled sites, and 20.2% was delivered to open dumping sites in 2018. While the recovered waste amount had a share of 11.9%; 0.38% was composted showing that Turkey needs new routes to deal with the MSW to maintain an environmentally and economically more sustainable waste management [2].

Composting is a biological process to convert organic wastes such as sewage sludge, municipal solid waste etc., into soil amendments. By definition, it is homogenization of mixed organic wastes by means of thermophilic process at a temperature range of 45 to 65 °C [3,4]. The final products after the process have nutrients for the soil, humic acid and metals together with high moisture content around 40 to 60 wt.% [5]. There are reports in the literature criticizing the usage of compost as soil amendment since it has pathogens and heavy metals which are hazardous for the humans. Although vermicomposting, which is the stabilizing the

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organic wastes or compost by the digestion of earthworms and micro-organisms at 35°C reduce somewhat human pathogens, regulations by the governments limit the usage of compost and vermicompost due to their hazard to human health [6]. Therefore, if new evaluation methods were suggested, compost as a cheap product gathered from the MSW could be a valuable feedstock and a new route for waste management.

As a thermo-chemical conversion method, supercritical water gasification process could be applied to compost to produce H_2 and CH_4 rich gas fuel. The high-water content of the compost, therefore could participate in the production of gas products since water undergoes tremendous changes when the conditions reach critical point (374°C, 22.1 MPa). This will prevent a costly pre-drying process which will be required for a pyrolysis or conventional gasification of compost to produce gas fuel.

In this study, the compost produced from organic wastes (MSW, city market's wastes and wood dust) was selected to be processed via supercritical water gasification (SCWG) in order to produce gas product consisting of hydrogen and methane mainly. Its being waste driven material and its cost around 0.4 €/kg make compost a favourable feed for energy fuel production since composting could be regarded as a homogenization process for the feed. In addition, SCWG process eliminates the costly drying process therefore the high moisture content becomes an advantage. The effects of parameters such as temperature, reaction time, particle size and solid/water ratio were investigated. Red mud as another waste material was activated and used as catalyst together with KOH to determine their effects on the product yields.

EXPERIMENTAL

Samples

Compost sample was purchased from a local company, from Izmir, Turkey. The compost originally contained around 65 wt.% of water, therefore it was air dried, sieved and separated to 3 different mesh sizes for determining the effect of particle size. Red mud was supplied by Alumina Plant in Seydisehir, Turkey, as a sludge and it was filtered and dried at 110 °C. Original red mud contained Fe_2O_3 (37.7%), Al_2O_3 (17.3%), SiO_2 (17.1%), Na_2O (7.1%), CaO (4.5%), and TiO_2 (4.8%). Activated red mud was prepared by placing 600 mL of red mud slurry (~30 % solid content) into a 1 L beaker and heating up to 95 °C while mixing. The pH of the slurry adjusted to be 4-6 by adding 35-37 % of HCl (d=1.18 g/cm^3) dropwise and finally at 95 °C it was held for one hour. NH_3 (25 %, d=0.91 g/cm^3) was added into mixture until obtaining a pH of 8. The mixture was heated further for 1 hour at 95 °C and then filtered and washed. Finally, the filter cake was dried at 110 °C overnight, calcined at 500 °C for 2 hours. KOH was purchased from Sigma-Aldrich.

Experimental Set-up

The supercritical water gasification experiments were performed in a batch autoclave reactor system where the details were given in elsewhere [7]. 1.11 g of compost along with 10 ml of deionized water were fed to the reactor, after the particle size and intended water content of the feed was adjusted. The reactor then purged with nitrogen, sealed and heated with a heating rate of 12 °C/min to the designated temperature. When the set temperature reached, the temperature kept constant for the specified reaction time. After completion of the gasification, the reactor was cooled to room temperature by means of a fan. The gas product was collected with a gas-tight syringe and its volume was measured. The reaction effluent was filtered, washed with deionized water and the remaining char was dried and its weight were determined. A sample from the final gas product was injected to HP 7890 A gas chromatography device which is equipped with TCD, FID, TCD detectors connected in series, after each experiment to determine the gas composition. The gross calorific value of gas products was calculated according to the equation (1)

$$HHV = \sum_{i=1}^{n} X_i HHV_i \quad (1)$$

Where i,.., n=each combustible gas in the product mixture and Xi denotes for mass fraction of each gas in the product mixture. HHVi is the higher heating value of each gas [MJ/kg] (values taken from [8]).

RESULTS

Supercritical water gasification of compost was performed and the resulting gas composed of hydrogen, methane and carbon dioxide mainly. Small amounts of carbon monoxide and hydrocarbon gases (ethane, ethylene, propane and butane) which were referred as C_2-C_4 gases, were also produced during SCWG of compost. The effects of temperature, reaction time, particle size, solid content of feed and catalyst were investigated to determine the optimum reaction conditions in order to produce gas fuel with high calorific value.

FTIR spectrum of compost given in Figure 1 reveal that functional groups originated from carboxylic acids (humic substances) exist in its structure. While broad band observed at 3278 cm^{-1} could be attributed to H bonded OH groups, peak around 2920 cm^{-1} could represent C-H stretch of aliphatic structures within the compost. The peak between 1620 and 1660 cm^{-1} may indicate the C=O vibration of bonded conjugated ketones, carboxylic acids and esters, and the C=C vibration of aromatic components. Finally, broad peak at 1032 cm^{-1} belonging to polysaccharides, can also present Si–O stretch of clay minerals and Si– O–Si vibration of silica since clay minerals are also characterized by a huge band around 1030 cm^{-1} [9,10].

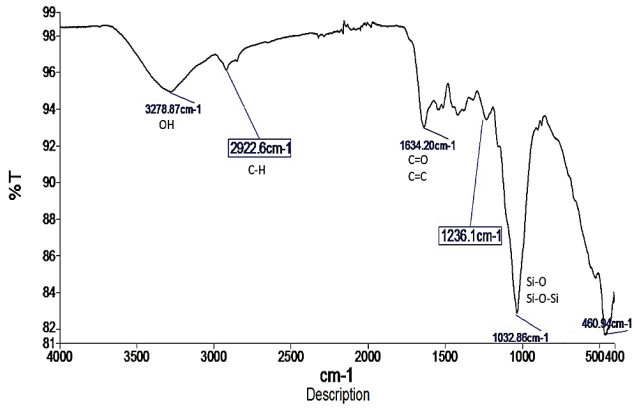
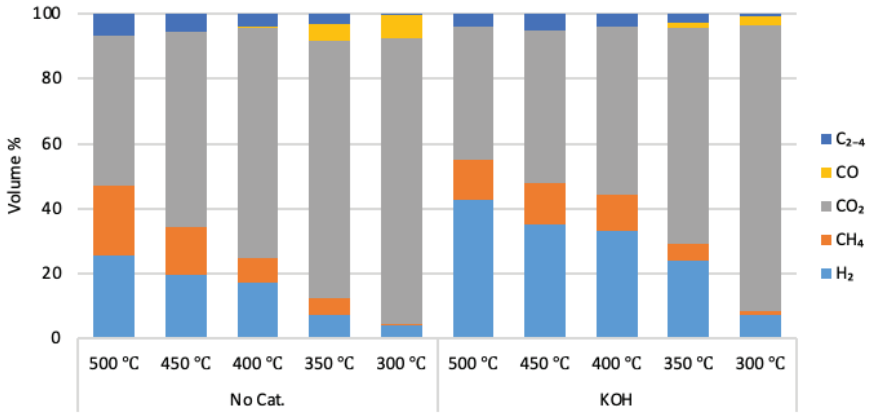


Figure 1. FTIR spectrum of raw compost.

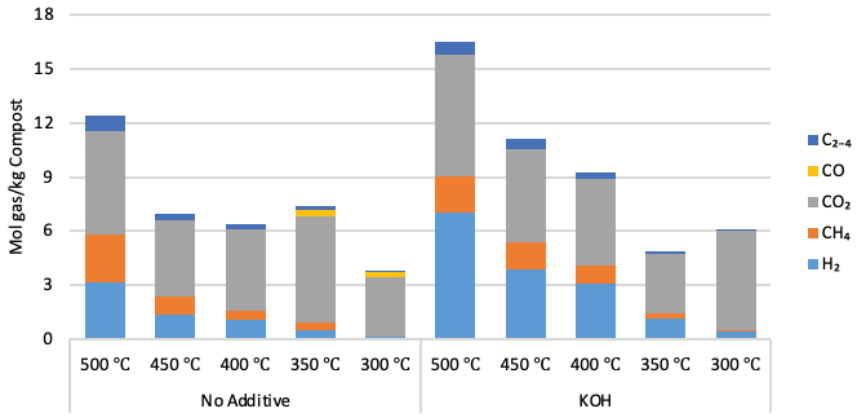
The ash content of compost was determined by oxidizing at 700 °C for 2 hours in a furnace. It was found to be 27.5, 15.4 and 31.1 wt.% for particle sizes of x<1.18, 1.18<x<2.35 and 2.35<x<3.45 mm, respectively.

The Effects of Temperature

Temperature was the most influential parameter during SCWG process, as the gas composition and product yield greatly changed with the increasing temperature as shown in Figure 2a and 2b. At 300 °C and 350 °C, 89 vol.% and 79 vol.% of the product gas consisted of CO_2, respectively. In hydrothermal medium, at low temperatures initially decarboxylation reactions of intermediates resulting from hydrolysis of organics occur, which is the main source of CO_2 [11]. H_2 and CH_4 yields increased to 3.2 mol/kg and 2.6 mol/kg compost, respectively, as the temperature reached 500 °C. Methanation of CO_2 and CO, and water-gas shift reactions become dominant when the supercritical conditions of water achieved [12]. Although CO in the product gas was found at lower temperatures, with the aforementioned reactions' increasing selectivity at higher temperatures, CO was not detected at temperatures above 450 °C. KOH additions further increased H_2 yields as it was 7 mol/kg compost at 500 °C, since alkalis promote water-gas shift reaction. In addition, above critical point, free-radical reaction mechanism governs the decomposition of biomass so more H_2 production is observed [13].



(a)



(b)

Figure 2. (a) Product gas composition and (b) yield during SCWG of compost at various temperatures.

The Effects of Reaction Time, Particle Size and Water Content of the Feed

The effect of time on the gas composition and yield was investigated at 0, 30, 60 and 90 minutes of reaction times at 500 °C, and the results are given in Figure 3a and 3b.

The amount of total produced gas during SCWG of compost ascended with the increasing reaction time. At 0-minute reaction time, 7.6 moles of gas was obtained per kg of compost processed while it increased almost thrice the amount and reached 21.5 moles at 90 minute reaction time. The gas composition was improved with the increasing reaction time as well, since CO_2 was dropped to 38.1 vol.% yielding 61.9 vol.% combustible gases at the end of 90 minutes. The CH_4 yield increased gradually until the 60 minute of reaction time while it boosted to 6.7 from 2.3 moles when the time increased to 90 minutes. H_2 too, increased steadily with the increasing reaction time, which could be the results of methanation of CO_2 (1) and water-gas shift reaction (2);

$$CO_2 + 4H_2 \rightarrow CH_4 + 2H_2O \quad (1)$$

$$CO + H_2O \rightarrow CO_2 + H_2 \quad (2)$$

The effect of particle size was also investigated at 500 °C and the results represented in Figure 4a and 4b.

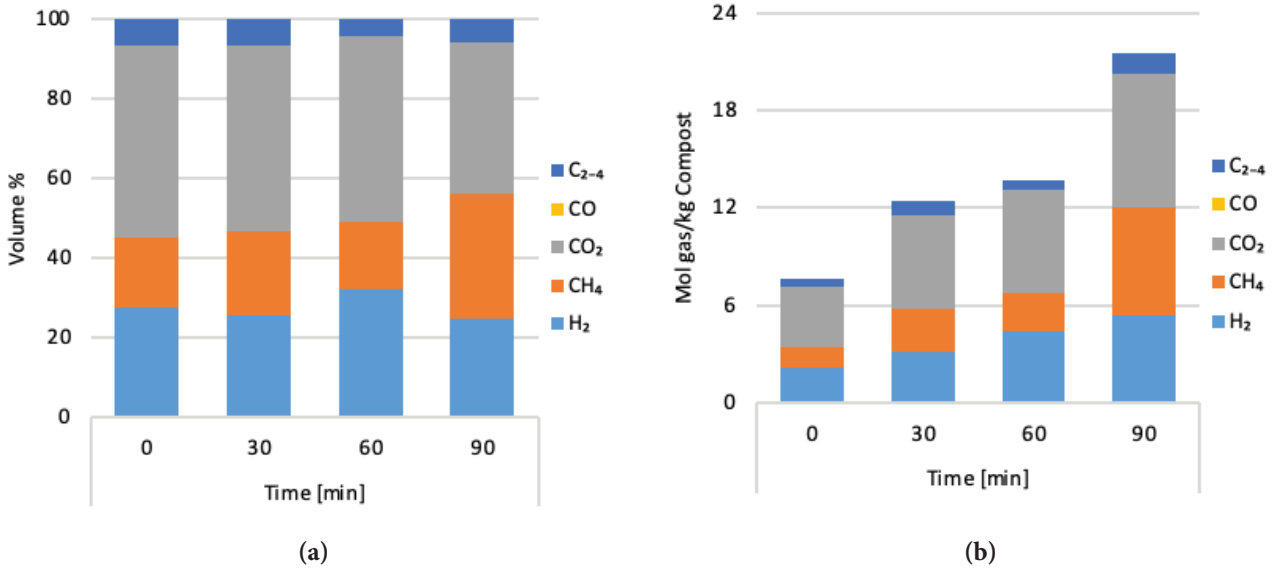
The results revealed that mid-sized particles experienced slightly higher product yields and gas composition. The ash content of mid-sized particles was determined to be the lowest, therefore it contained more volatiles compared to other particle sizes. As a result, total gas production was slightly increased. Nevertheless, this could be an indication of the homogeneous structure of compost consisting of various kinds of wastes and biomass, even though in general the studies revealed that the changing the particle size affects less during SCWG process compared to other parameters [14].

The effect of water content of compost was studied at three different concentrations of 10, 20 and 30 wt.% dry compost and Figure 5a and 5b reveals the results. The sum of H_2 and CH_4 in total in the product gas was around 42 vol.% in all concentrations while the share of H_2 increased with the increasing concentration. This might be due to water-gas reaction, since CO composition was 0 vol.% at 10wt.% while it was increased to 6.8 vol.% at higher concentrations. Water-gas shift reaction is an equilibrium reaction, therefore the increase in CO composition was balanced with the less water content of the feed, as a result the increase in H_2 composition was limited. In addition, less moles of gas per kg of feed was produced, indicating that the higher feed concentration decreased the gasification efficiency. This might be a result of "cage effect", creating a limitation in the reaction rates caused by the water molecules [15].

The Effects of Catalysts

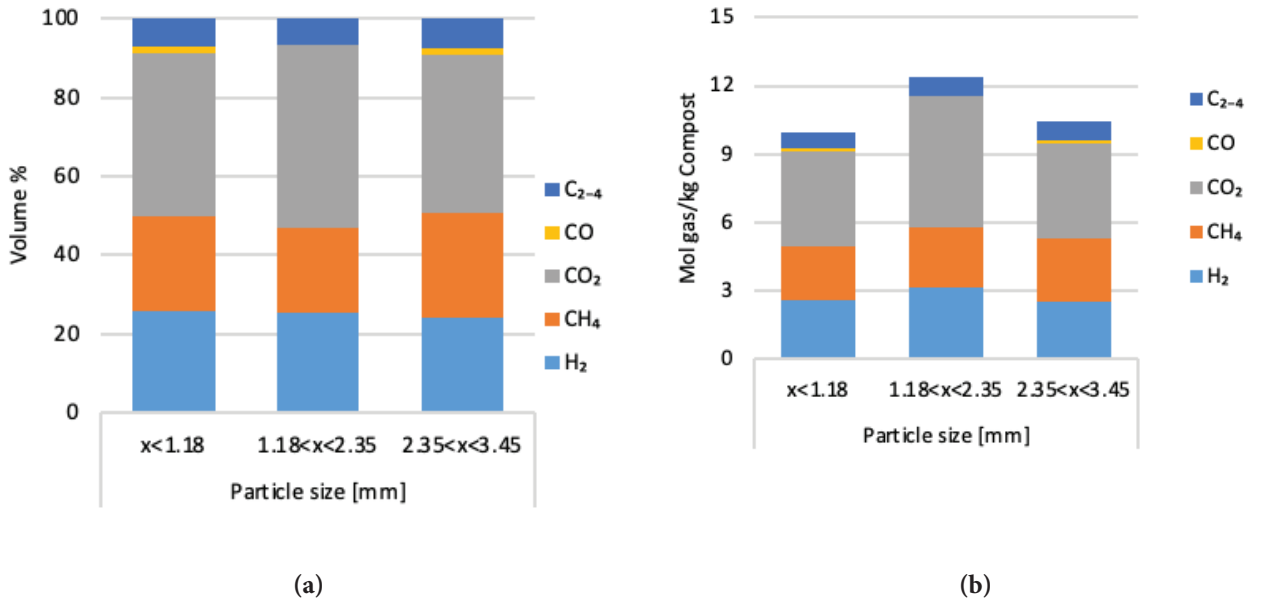
The catalytic effects of original red mud (ORM), activated red mud (ARM) and KOH were investigated and the results are shown in Figure 6a and 6b. The red mud catalysts yielded more C_2-C_4 and CO, whereas CO was not detected in the presence of KOH. The red mud contains metal oxides which are affective in oxidation of biomass in hydrothermal medium [16]. CO would be a partial oxidation product of compost gasification, while the red mud catalysts slightly reduced H_2 composition and increased CO_2 composition in the product gas.

The addition of KOH promoted the water-gas shift reaction, hence H_2 increased from 25.5 vol.% to 42.6 vol.%.



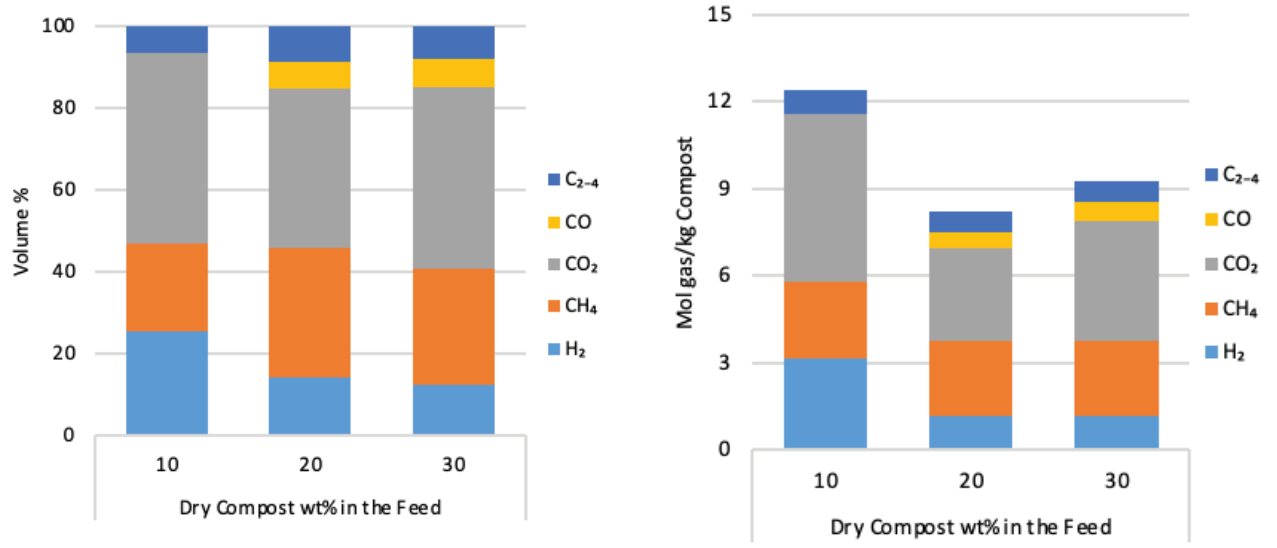
(a) **(b)**

Figure 3. (a) Product gas composition and (b) yield during SCWG of compost at various reaction times at 500 °C.



(a) (b)

Figure 4. (a) Product gas composition and (b) yield during SCWG of compost at various particle sizes at 500 °C.

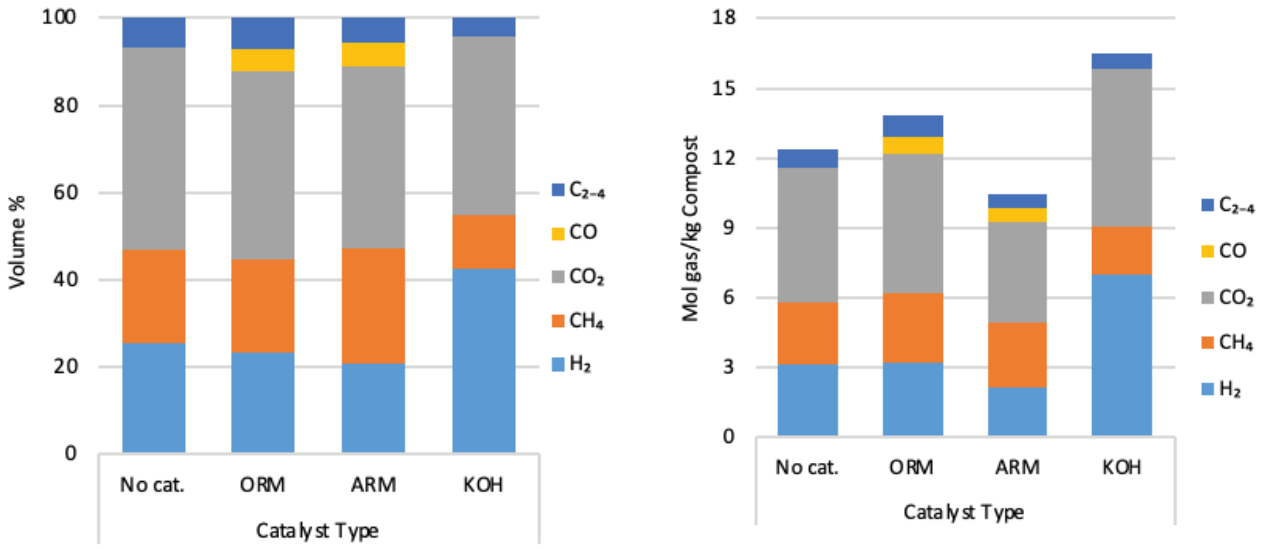


(a) (b)

Figure 5. (a) Product gas composition and (b) yield during SCWG of compost at various dry solid concentrations at 500 °C.

The yields of gases were affected via catalyst presence, whereas the highest total moles of gas per kg of compost was achieved as 16.5 mol/kg in the presence of KOH. ORM addition did not increase remarkably while ARM addition decreased the total gas produced. The slight or negative effects of red mud catalysts could be attributed to compost structure, since naturally compost may contain some heavy metals [17].

On the other hand, the calorific value of the product gas was the highest when SCWG of compost performed with 10 wt.% dry compost at 500 °C for 30 minutes. However, as mentioned before, when the solid concentration of feed increased, gasification yield decreased due to cage effect. In the end, a product gas with 20.1 MJ/Nm^3 calorific value obtained without any catalysts, at 90 minutes of reaction time. Although KOH increased H_2 yield, since volumetric energy density of hydrogen is low, the calorific value of the



(a) (b)

Figure 6. (a) Product gas composition and (b) yield during SCWG of compost with different catalysts at 500 °C.

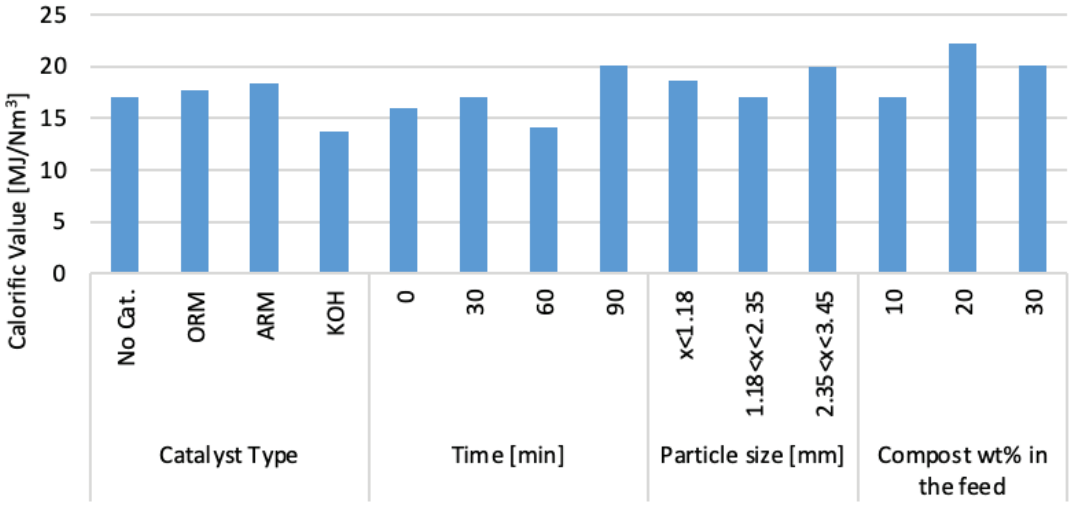


Figure 7. Gross calorific values of product gas obtained during SCWG of compost at different conditions at 500 °C.

product gas was not improved. If the aim is producing a specific gas component, then a catalyst with a high selectivity should be chosen.

CONCLUSION

Supercritical water gasification of compost was evaluated to determine the optimum conditions. High H_2 yield was achieved in the presence of KOH. In addition, around 55 vol.% of H_2 and CH_4 in the gas product was found after 30 min reaction time together with KOH, at 500°C. Although the red mud catalysts increased the calorific value of the product gas obtained, the total gas yield did not improve. The product gas with the highest calorific value was gathered as 20.1 MJ/Nm^3 when the reaction time increased to 90 minutes. While the change in particle size did not affect the gasification yields, increasing the amount of dry solid in the feed dramatically decreased the efficiencies.

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CONFLICT OF INTEREST

The authors declare no potential conflicts of interest regarding the research, authorship and/or publication of this article.

DATA AVAILABILITY

The data used to support the findings of this study are included within the article.

AUTHOR'S CONTRIBUTIONS

All authors are contributed equally to bring out this article.

ETHICS

There are no ethical issues with the publication of this manuscript.

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Research Article

The effect of bags law on environmental behavior and habits-Mersin example

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ABSTRACT

The use of plastic bags (PB) has increased continuously over time because of their multipurpose property. Due to its environmental effects, Türkiye has approved a new law to limit the use of plastic bags. According to the approved Turkish plastic bags law (PBL), plastic bags of 15 microns or more are prohibited from being given to the customer free of charge. The new law's effects were investigated by applying a face-to-face survey consisting of 13 questions to 1537 people in four districts close to Mersin city center. In addition to that, field surveys were managed to the common markets in the target area. From the target sample, 159 males and 128 females had not bought the PB after the law. Whereas 184 males and 178 females had bought PB for 10 times or more. The monthly income has a weak correlation with the plastic bags purchasing times. Families with 1-3 capita, 4-6 capita, and larger families who never bought plastic bags have moderate negative correlations. Families with 4-6 capita and larger families have a significant strong positive correlation at the ($P<0.01$). It was found that people aged 50 years and over who participated in the survey were less aware of the negative impact of bag use on the environment than younger ones. Garbage plastic bag consumption has not increased as expected. The managed field surveys proved that. The results from the surveys showed a decrease in PB consumption with a ratio between (60-80%)..

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INTRODUCTION

Plastic Bags (PB) was designed for the first time by StenGustafThulin. It was patented and commercialized in 1965 [1]. It aimed to facilitate the transportation products from market to the destination. The single-use average time was 12 min [2]. Shopping bags were used over the world because of their thin-film structure, waterproof, lightweight, and cheap (free in markets). Many expressions are describing plastic bags Types. Some of these are listed in Table 1 [3].

From the first day, the demand for plastic bags has increased due to their practicality. The expansion in the large shopping center also increased the demand by providing free-of-charge plastic bags to ensure customer satisfaction. Approximately 0.5-1.0 bags are consumed in a year in the world. This means that 1.4-2.7 billion bags are used in a day and more than one million bags in 1 minute

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Table 1. Plastic bags type's definitions [3]

Shopping plastic bags (SPB)	It obtained from seller at the market point to carry products. It formed partially or completely from polymers.
HDPE bag	High-density polyethylene (HDPE) bags, and in general it has a thickness less than 35 microns. It is often referred to as supermarket style bags, single bags, disposable plastic bags, and lightweight plastic shopping bags. Generally used in supermarkets or grocery stores.
Boutique bag	It made from Low-density polyethylene (LDPE) bags. It normally has printed trademarks and usually supplied from clothes and electrical markets.
Green bags	Reusable "green" bags. They are durable bags made of polypropylene and designed for multiple uses.

[4]. PB consumption is higher in countries with high income levels. However, in low-income countries, the negative effects of insufficient and excessive use of laws are greater [5]. It has been determined that the annual per capita consumption of plastic bags is 1370 in Hong Kong, 286 in the USA, 263 in Israel, 252 in Taiwan, 235 in Japan and 223 in China [6]. In addition, it has been reported that before 2019, the amount of plastic bag production in Türkiye was 35 billion pieces per year and that one person used an average of 440 plastic bags per year [7]. As a result, a tremendous quantity of naturally non-recycled plastic bags was thrown. When the contribution of non-recycled plastic bags on environment pollution became significant, many governments had been forced to arrange legally the uses of plastic bags [8].

The arrangement procedure had taken three shapes; the first one related to imposing taxes on the disposal of plastic bags; the second is related to charging the plastic bags; the last one related to collecting or exchanging the plastic bags for fees. Many governments applied the first vision [9, 10]. In the beginning, the applied tax reduced the using of plastic bags [11, 12]. Despite the high taxes, the decreasing in using plastic bags did not continue, since the people returned to their habits [13, 14]. Prohibitions and exchanging the plastic bags for fees have been more effective in reducing bag use. In 1994 Denmark introduced a tax law on plastic bags disposal [15]. Countries followed two main policies in reducing the use of plastic bags. While developed countries chose to tax, developing countries preferred to prohibit. The benefit of taxation policy is revenue generation, public awareness and growth of recycling. The prohibition policy aims at a cleaner environment, tourism attraction, environmentally friendly shopping and reducing the ecological footprint [16]. In 1998 India followed Denmark in enacting legal laws [17]. The emerged effects of plastic on the environment led many countries to introduce legal regulation [15].

The first conference related to environmental issues was held in Bern in 1913. The European environmental advisory board was organized in 1965 at the United Nations building [18, 19]. The first international conference on environmental rights was held in Stockholm in 1972 by the United Nations [20].

In Türkiye, the first environmental regulations were included in Municipalities Law No.1580 [21] and Public Health law No.1593 [22] in 1930. These laws include regulations on the waste collection, storage, and protection of the environment and human health. Article 56 of the 1982 Constitution states that people have the right to live in a healthy and sustainable environment, and the state and citizens have to develop and protect the environment [23]. Environmental Law No. 2872 was approved in 1983. In 1991, the control of the effects of the solid waste on the environment began with the Regulation on Solid Waste Control [24]. Until these days, many legislation were regulated, developed, and implemented with consistency with European Union Legislation. After the scientific demonstration of the negative effects of plastics on the environment, many countries such as the Republic of Türkiye had adopted regulations that limit the use of plastic bags.

Arı and Yılmaz (2017) examined the attitudes of 321 people living in Eskişehir, a metropolis in Türkiye, towards the use of plastic and cloth bags. The study conducted during the period when nylon bags were free showed that environmentally conscious consumers tended to use cloth bags instead of plastic bags [25]. The research conducted with 434 people in Gümüşhane, a small province in eastern Türkiye, revealed that after the regulation, consumers reduced the use of plastic bags, but they also had doubts about their sustainability [26]. After the implementation of the bag law in 2019, a questionnaire was applied to 363 university personnel in Isparta. All the findings have shown that the plastic bag pricing in effect contributes to the goals and targets of reducing bag consumption to a certain extent [27]. This research was carried out after the bag law was enacted. In addition, face-to-face questionnaire was applied to 1537 people. In addition, Mersin is a coastal city and the inhabitants of Mersin show a more cosmopolitan structure. All these details add originality to this work.

To reduce the use of plastic bags and plastic packaging, the approved plastic bags law (PBL) (7153) on 10 December 2018 prohibits the free charge using of plastic bags with a double thickness of more than 15 microns. The PBL aimed to reduce the annual use of 400 plastic bags per person up to 90 units per person until 31.12.2019, and 40 units per person until 31.12.2025 [28].

MATERIAL AND METHOD

Mersin province is located at the center of the southern coast of Türkiye at the Mediterranean Sea. In 2018, the total population in Mersin reached 1814468 capita [29]. They are distributed over 13 districts. Mersin extends over an area of 15.853 km^2 [30]. Figure 1 shows the district of Mersin.

The districts were selected according to their economic status, social structure, and the number of shopping centers in the region. Erdemli district is further away from the city center than other districts. People living in this region are mostly engaged in agriculture. The number of people coming from outside migration is low. They have a more traditional lıfe way. Mezitli is a region where Mersin province has developed greatly in recent years. In Mezitli, the agricultural area has decreased by 80% in the last decade, and these have been replaced by large multi-story sites. There are many shopping centers in this area. Yenişehir district is located close to the center. In this area, there are many shopping centers as well as residences. The economic situation of the people living in this region is high. The Mediterranean district is located in the old city center of Mersin. The economic and social situation of the people living in this region is very complicated. In general, old warehouses are located in this region. Also, this region is the most region affected by migration (internal and external migration).

By applying Equation 1, the sample size for each district was obtained. In total, 1537 questionnaires were prepared and distributed over the target area. The survey lasted 10 days, the results were arranged using Excel program. Table 2 shows the population of each district and the sample sizes.

Table 2. The population and the sample size of each district

District	Population (Mersin, 2018)	Sample size
Erdemli	140331	383
Mezitli	194019	384
Yenişehir	258694	385
Mediterranean	264618	385
Total		1537

Effect of Plastic Bags Law on the People Habits Exploration

The effect of the PBL on the people habits was explored in the target area by field surveys for the period from 20/4/2019-01/05/2019 face to face by students

Figure 1. Mersin province (Turkish State Meteorological Service, 2019).

Table 3. The prepared questionnaire

Question	Answer			
Gender	**Male**		**Female**	
Age	18-25	25-35	35-50	>50
Education	Reading/Writing	Primary sch.	High Sch.	University
Monthly income	<1000	1100-2000	2100-4000	>4000
Family size	1-3	4-6	7-10	>10
Frequency of shopping	< weekly	weekly	2 time / week	Every day
Do you know PBL?	Yes		No	
Do you use Market bags as garbage bags?	Yes		No	
Did you buy a market bag after PBL?	No	Yes (3-5)	Yes (6-10)	Yes (>10)
Are you purchasing garbage bags?	Yes		No	
How many daily garbage bag do you waste?	1-2	3-4	4-5	>5
How much do you buy garbage bags monthly? (Pack of 20)	1-2	3-4	4-5	>5
Do you think the market bags (plastic) are harmful to the environment	Yes		No	

from Environmental Engineering department - Mersin University.

The data related to people's habits were recognized and designed as a questionnaire. It had been distributed over sample sizes that achieve a 95% confidence level with 5 confidence interval. Sample sizes were determined using equation 1 [31].

$$\text{Sample size} = \frac{Z^2 \times P \times (1-P)}{C^2} \quad (1)$$

Where, Z, P, and C are the Z-score, Percentage of the population picking a choice, and Confidence interval, respectively.

The questionnaire had 13 questions. Each question has been prepared to be clear and understandable. The results from questionnaires were analyzed for each district. Table 3 shows the questions.

SPSS software (SPSS v20.0) was used in the statistical analysis. The Pearson correlation test was used to study the relation between the different factors.

Effect of Plastic Bags Law on Shopping Center (Market)

The effect of the law on the shopping center was explored by a separate survey. The most famous and popular shopping centers (market) were selected to be the target. The shopping centers were visited separately by the surveyors. The data from shopping were used in two approaches; the first is to examine the effect of LPB on the bag consuming from market view. The second is to support the questionnaire results.

RESULT AND DISCUSSION

Effect of the Consumer Gender on the Plastic Bags Consumption

It had been thought that the application of the law is affected by consumer gender. It had assumed that males purchased plastic bags more than females.

In Erdemli district, 55% of the sample was male, and the percentage of females was 45%. In Mezitli and Akdeniz males recognized 51.5% and 62% of the sample respectively. In Yenişehir the percentage of males reached 45% of the sample. Figure 2 shows the gender size within the sample in each district.

From the target sample, 159 males and 128 females had not bought the PB after the law. Whereas 184 males and 178 females had bought PB 10 times or more. The largest female and male part had bought PB 3-5 times. The number of males and females who had bought PB 6-10 times

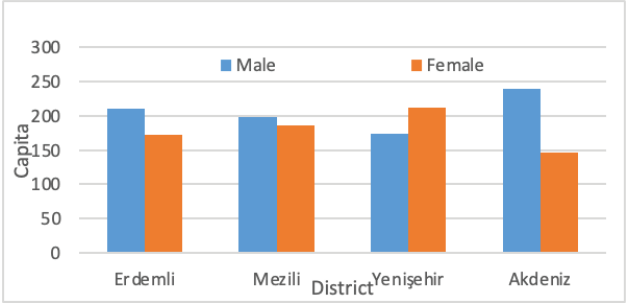

Figure 2. Gender size within the sample.

was 169, 161 respectively. Figure 3 shows the PB purchase times according to gender along the target area.

Effects of the Monthly Income and Family Size on the Plastic Bags Consumption

The monthly income and the family size effects on the PB purchase were studied over the four districts. Over the random sample, there were 211 questionnaires with income less than 1000 Tl, and 384 for the income level between 1000-2100. The most statement about income range was 2200-4000 with 531 answers. There were 338 questionnaires for income larger than 4000 TL. Table 4 shows the families' sizes corresponding to their income levels.

The families with sizes of 1-3 comprised 45% of the total families who participate in the survey in Akdeniz. While in Erdemli, Mezitli, and Yenişehir it is about 40%. In Mezitli, and Yenişehir 50% of the families were 4-6 per capita. The families with a size of 7-10 formed percent of 7-12%. The big families (more than 10) constituted 7.8% in Erdemli. However, it composed just 1.5 % of the other districts. Figure 4 shows the family sizes in the random samples.

The study assumed that families with higher incomes would buy PB more than others. While the limited income families would save more than others. To test this assumption, the Pearson correlation test was performed and the results are shown in Table 5.

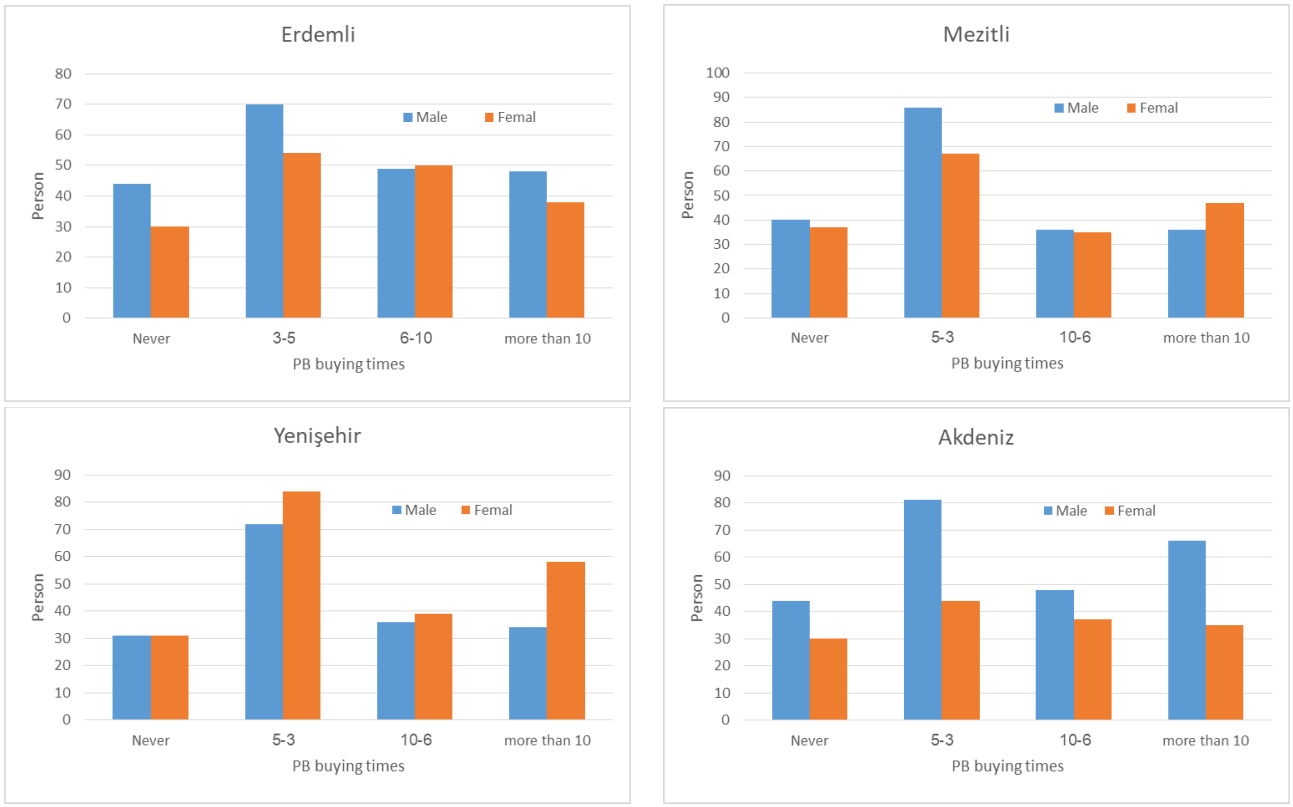

Figure 3. The PB purchase times according to gender along the target area.

Table 4. Family sizes corresponding to income level

		IncomeLevel				Total
		<1000	1000-2000	2100-4000	>4000	
Family Size	**1-3**	112	168	194	149	623
	4-6	137	160	259	135	691
	7-10	26	41	57	30	154
	>10	9	15	21	24	69
Total		284	384	531	338	1537

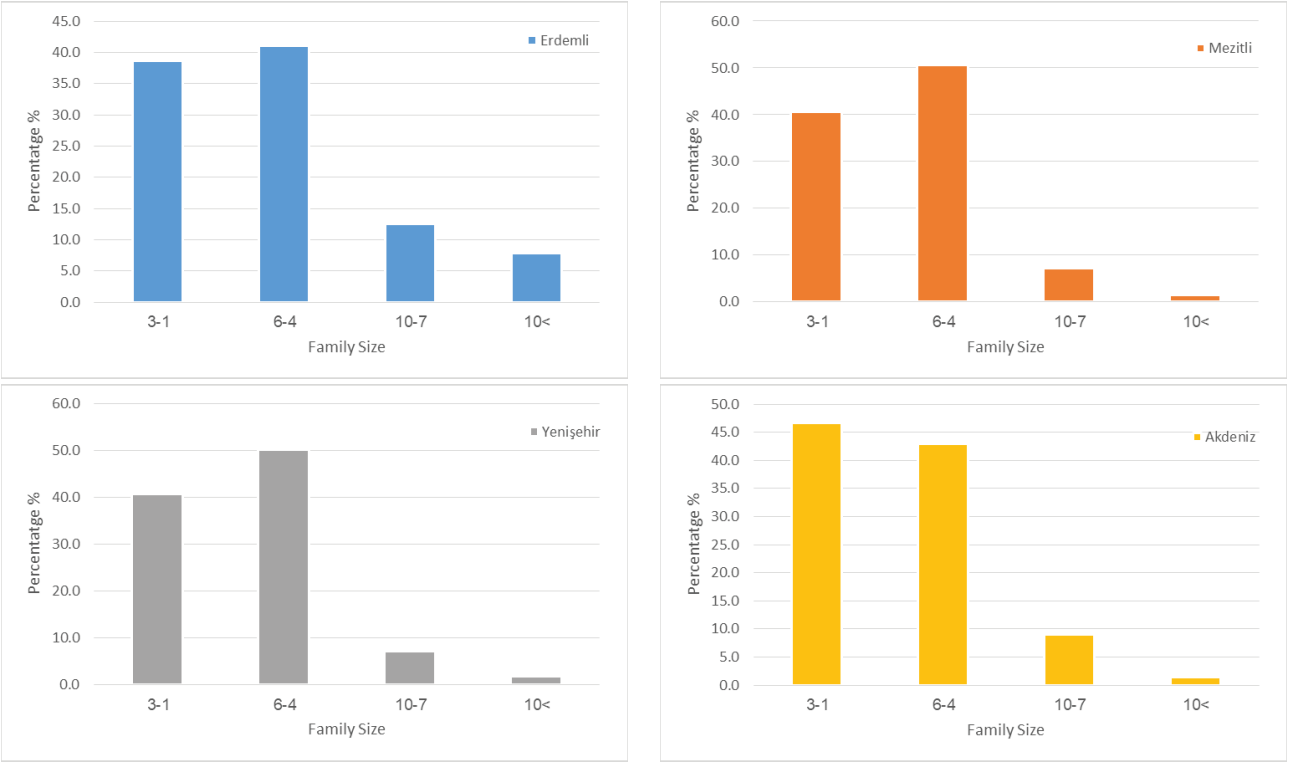

Figure 4. Families' sizes in the random sample over the four districts.

Table 5. Pearson correlation test for income level and PB purchase

Income level for each district	PB purchase
Erdemli	0.016*
Mezitli	-0.035*
Yenişehir	0.076*
Mediterranean	0.046*

PB: Plastic bags, *$p < 0.05$; **$p < 0.01$

Table 6. Pearson correlation test for family size and PB purchase

	Never	3-5	6-10	>10
Family (1-3)	-0.532	0.090	0.607	0.902*
Family (4-6)	-0.871	0.955*	0.726	0.957*
Family (7-10)	0.771	0.685	0.779	0.009
Family (>10)	-0.541	0.981*	1.000**	0.967*

PB: Plastic bags, **: Correlation is significant at the 0.01 level (2-tailed), *: Correlation is significant at the 0.05 level (2-tailed).

Correlation results between the income level and the PB purchase show a very weak correlation ($p < 0.05$) over the study area. The results reject the above assumption. These results could be explained by the price of plastic bags. The plastic bag can be bought for 0.25 TL, whereas the green bag price is 2 TL.

For the family size, it was thought that the larger family will tend to use green bags and will not buy plastic bags. While families with less capita will continue using plastic bags. The family size and the plastic bags buying were correlated by the Pearson correlation test and the results are shown in Table 6.

Pearson correlation test show a strong relationship between family size and the PB purchase. Families with 1-3 capita, 4-6 capita, and larger families who never bought plastic bags have a moderate negative correlation. While families with 4-6 capita and larger families have a significant strong positive correlation at the ($p < 0.01$). There are significant correlations at ($p < 0.05$) for the families with 4-6 capita and larger families with plastic bags purchase rates larger than 10 times. These results clearly indicate that there is a relation between family size and PB purchase.

Effects of the Age and Education Level on the Plastic Bags Consumption

The age and education level had been expected to affect the consumption of PB. To study the effect of age, four different categories were set as shown in Table 2. The average samples ages in Erdemli, Mezitli, Yenişehir, and Akdeniz were 38.13, 35.6, 35.8, and 34.9 years, respectively.

Table 7. Distribution of education levels according to age

		Education				Total
		Reading-writing	Primary	High School	University	
Age	**18-25**	22	40	71	269	402
	25-35	37	57	141	168	403
	35-50	62	92	162	169	485
	>50	52	66	58	71	247
Total		173	255	432	677	1537

The education level was categorized into; reading-writing, primary school, high school, and university. According to Table 7, there were 677 questionnaires with an education level of the university. And just 173 people can write and read level.

Pearson correlation test was performed to study the relationship between the age and the PB buying times. The results from the test are shown in Table 8. The results in general shows a weak correlation between age and PB purchasing times. In Erdemli district, the correlation was positive with a confidence of 95%. The other districts show a negative correlation between the age and the times of PB purchasing ($p < 0.01$ for Mezitli and Akdeniz, $p < 0.05$ for Yenişehir). This means that age has a relative relationship with PB purchasing in those districts.

The PB purchasing times are assumed to be affected by the education level. In order to study this side in Mersin, the data were analyzed by Pearson correlation test. The results shown in Table 9 show a positive weak correlation between the education level and the Purchasing times of PB over the four districts. In general, the environmental awareness of educated people is expected to be higher. Increased environmental awareness through education is an expected result, but the survey results do not confirm this hypothesis.

Effects of the Plastic Bags Law on the Garbage Bags

People used plastic bags (market bags) to carry baggage to their houses. After that, they usually used them as garbage bags. After the adoption of the relevant law and decreasing the use/purchase the market bags, it was expected to increase in purchasing of garbage bags. The results obtained from the Pearson correlation test show a weak positive correlation between garbage purchasing and plastic bags Table 10.

Effects of the Plastic Bags Law on the Shopping Centres (Markets)

The field survey results at the most popular market in the four districts show a decrease in PB consumption with

Table 8. Pearson correlation test for age and PB purchase

		Age	PB Purchase
Erdemli	**Age**	1	
	PB Purchase	0.03*	1
Mezitli	**Age**	1	
	PB Purchase	-0.184**	1
Yenişehir	**Age**	1	
	PB Purchase	-0.066*	1
Mediterranean	**Age**	1	
	PB Purchase	-0.193**	1

PB: Plastic bags, *$p < 0.05$; **$p < 0.01$

Table 9. Pearson correlation test for the education level and PB purchase

		Education	PB Purchase
Erdemli	**Education**	1	
	PB Purchase	0.01	1
Mezitli	**Education**	1	
	PB Purchase	0.223**	1
Yenişehir	**Education**	1	
	PB Purchase	0.057	1
Mediterranean	**Education**	1	
	PB Purchase	0.214**	1

PB: Plastic bags, *$p < 0.05$; **$p < 0.01$

Table 10. Pearson correlation test for the garbage bags purchasing and the PB purchase

		GB Purchase	PB Purchase
Erdemli	**GB Purchase**	1	
	PB Purchase	0.045	1
Mezitli	**GB Purchase**	1	
	PB Purchase	0.036	1
Yenişehir	**GB Purchase**	1	
	PB Purchase	0.022	1
Mediterranean	**GB Purchase**	1	
	PB Purchase	0.141**	1

GB: Garbage bags PB: Plastic bags *$p < 0.05$; **$p < 0.01$

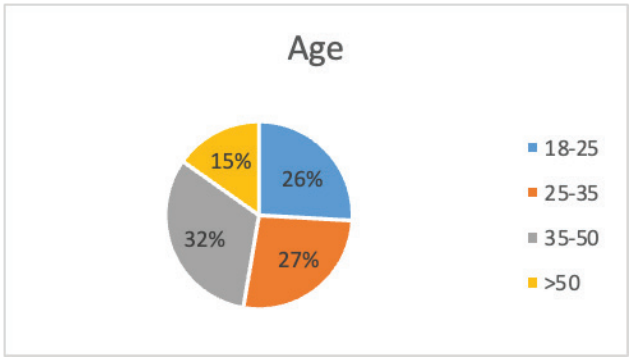

Figure 5. Age contribution to the awareness of the plastic hazards.

a ratio between (60-80%). Additionally, it does not appear to be any indication of increased purchase of garbage bags. These results support the statistical results above. Another note that should be taken into consideration when assessing the law, is the increasing in the consumption of 15-micron bags which is expected from the law.

Effects of the Awareness on the Plastic Bags Consumption

The level of awareness of the hazards of plastic bags was determined. The awareness level was examined according to age and education level. Over the four districts, 84.3% of the questionnaires agreed that plastic bags harm the environment. The persons aged 35-50 years comprised the max percentage with 32.1%, whereas the lowest percentage was for the persons with ages above 50 years. Figure 5 shows the contribution of age to the awareness of plastic bags hazards. It was found that people aged 50 years and over who participated in the survey were less aware of the negative impact of bag use on the environment than younger ones.

The education level affects the awareness of plastic bags hazards. The results in Figure 6 reflect that human with high education is aware of the hazards of plastic bags. This result is consistent with those obtained by Mogomotsi et al. [8]. The survey showed that the environmental awareness gained through education has not reflected to correct behavior [32]. It was found that most of the respondents did not change the habit of using the bag while saying that the bag pollutes the environment.

CONCLUSION

The study is an attempt to evaluate the effects of the newly approved Turkish plastic bags law (PBL) on environmental behavior and people's habits in four districts of Mersin. The study was based mainly on the results of the questionnaire prepared for this purpose, the questionnaire was consisting of 13 questions and was distributed to 1537 people. Gender, age, family size, income level, and education level correlated to the rate of plastic bags purchased by the Pearson correlation test. Gender and family size were founded to affect the plastic bags purchase. Whereas the remaining factors had a very weak correlation coefficient. The garbage plastic bags consumptions correlated weakly to plastic bags consumptions ($r = 0.141$ with $p<0.01$). The managed field surveys proved this result. The results from the surveys in the four districts showed a decrease in PB consumption with a ratio between (60-80%). The same question was asked in the face-to-face survey conducted to 821 consumers in Istanbul, Türkiye's most populous city. The result obtained is similar to this study [33]. Another note that should be taken into consideration when assessing the law, is the increase in the consumption of 15-micron bags which is expected from the law. At the end of the study, it was determined that there was no increase in the sale of garbage bags offered for sale in the markets, whereas the use of bags lower than 15 microns given free of charge increased by 100-110%. The survey showed that the environmental awareness gained through education has not reflected to correct behavior. It was found that most of the respondents did not change the habit of using the bag while saying that the bag pollutes the environment. As a result of this study, it found that the Laws and prohibitions can reduce the use of plastic bags. However, it would be most effective if the laws were conducted in parallel with the conducting of an education program about the bad effects of plastic bags. According to a 2018 study in the American Economic Journal: Economic Policy, the five-cent tax on disposable bags reduced the use of disposable bags by 40 percent [34]. In our study, it was found that the targets have not been reached yet. However, it would be more effective if a training program on the law and the harms of plastic bags is carried out in parallel. According to a 2019 review of available studies, fees and taxes are expected to decrease in usage by 66% in Denmark, by more than 90% in Ireland, and by 74% to 90% in South Africa, Belgium, Hong Kong, Washington D.C., Santa Barbara. has caused. In the United Kingdom and Portugal and Botswana and China, it decreased by approximately 50% [34]. The survey research was conducted in 2019, when the tax and wage law was put into effect. The survey results show that the plastic grocery bag tax and fee in 2019 is not well understood by the people T.C. Ministry of Environment, Urbanization and Climate Change reported that with the pricing initiated in 2019, the use of plastic bags in Türkiye decreased by 75 percent, thus preventing the generation of 354 000 tons of plastic waste. When evaluated together with the survey results, the tax application has reduced the use of bags. After the law, there was an increase in the consumption of thin bags placed for weighing products in the greengrocer sections of the markets. This shows that people do not give up on their habits of using bags, instead they turn to free bags. Uniform surveys should be established and administered periodically to identify changes in people's plastic bag use habits. In this way, awareness will increase. The effect of education and information on behavior change will be determined. Markets and shopping centers that adopt the

concept of zero waste are becoming widespread in the world. Encouraging practices should be implemented for consumers to prefer these places.

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CONFLICT OF INTEREST

The authors declare no potential conflicts of interest regarding the research, authorship and/or publication of this article.

DATA AVAILABILITY

The data used to support the findings of this study are included within the article.

AUTHOR'S CONTRIBUTIONS

All authors are contributed equally to bring out this article.

ETHICS

There are no ethical issues with the publication of this manuscript.

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Research Article

Biodegradation of high cellulose-lignin content agricultural wastes in bioreactors

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ABSTRACT

The bioreactor landfill is a solid waste disposal method that provides rapid degradation of solid waste and acquisition of methane. Bioreactors in which leachate circulation is carried out are generally operated anaerobically. The biodegradation of wastes with high lignin and cellulose content is very difficult. Especially under anaerobic conditions (moreover, if there is a lack of moisture), such wastes almost never decompose. In this study, the degradation of waste sunflower stalks that are difficult to biodegrade and have a high lignin-cellulose content and the production of methane gas in semi-aerobic bioreactors have been investigated. Sunflower stalks were loaded into the bioreactors in different proportions and mixed with the organic fraction of municipal solid waste (OFSWM). The bioreactors have been operated under different operating conditions. The contents of cellulose, hemicellulose, lignin, and initial and final organic matter in the wastes loaded into the bioreactors were examined. Parameters such as pH, COD, BOD5, TKN, NH4-N in leachate were analysed and the amounts of total and methane gas were measured. Initially, all bioreactors have been operated anaerobically. In the decomposition of the sunflower stalk, while 43% of the organic matter removal was achieved in the anaerobic bioreactor, 60% of the organic matter removal was realized in the semi-aerobic/anaerobic bioreactor. The other agricultural wastes were then subjected to decomposition under semi-aerobic/anaerobic operating conditions. As a result of the study, it can be said that semi-aerobic pretreatment accelerates the decomposition of agricultural waste with a high lignin and cellulose content, decreases the COD values of leachate, and increases the amount of methane.

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INTRODUCTION

Agricultural waste, known as biomass, has significant potential to produce sustainable energy from renewable fuels [1]. Lignocellulosic materials, such as agricultural waste, are widely accepted as the most suitable raw materials for the production of biomethane due to their relatively low costs, high availability, and lack of direct competition with the production of food and feed [2]. Lignocellulosic biomass is composed of cellulose, hemicellulose, lignin, oil, starch and proteins [3].

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Sunflower stalks, which are lignocellulosic biomass, produce 78 to 182 million tons globally and are typically disposed of as garbage or burned in fields, resulting environmental pollution [4]. Sometimes, sunflower remains (stems, leaves, heads) are left in the fields after the seed harvest [5]. In contrast, the high cellulose and hemicellulose content of the sunflower stalk makes it a potential raw material for the production of biomethane [6]. However, in the anaerobic decomposition of sunflower stalk, which contains high-crystalline cellulose, a lignin of a natural structure and hemicellulose, hydrolysis is rate-limiting and adversely affects methane production [7].

Pruning waste contains organic compounds such as lignin, which prevents and slows the aerobic degradation of cellulose, which is resistant to biodegradation, or hemicellulose, which is slow/moderately degradable [8].

Hazelnut husks are green plant structures that surround and protect the hazelnut during the growth process. At harvest time, this plant structure is collected with the hazelnut, separated from the hazelnut in the factory, and converted to waste. Turkey, which hosts 73% of the world's hazelnut production, is the world's largest hazelnut producer (400.000–450.000 tons/year). Each year after the hazelnut harvest, the hazelnut waste is burnt or left in the field [9]. Anaerobic decomposition of organic substances containing lignocellulosic matter, such as hazelnut husks, takes a long time and produces inefficient biogas [10].

In recent years, it has been demonstrated that bioreactor landfill technology supports the decomposition and stabilization of biodegradable organic waste by adding water, recirculating leachate, or injecting air [11].

Bioreactor systems are landfill systems that use advanced controlled microbial processes to convert and balance organic waste compounds that can be easily or partially decomposed in as little as 5-8 years [12]. The bioreactor areas may operate under various operating conditions. 4 types of bioreactor systems have been developed in line with the studies carried out so far. These are; anaerobic, aerobic, facultative, and hybrid bioreactors [13].

In this study, the effect of adding an organic fraction of solid waste to the biodegradation of sunflower stalk was examined. The other objective is mixing these wastes at different rates within themselves by applying different operating conditions in bioreactors and observing methane production.

MATERIALS AND METHOD

Laboratory-Scale Simulated Bioreactor

Two stainless steel cylindrical bioreactors with a height of 30 cm, a diameter of 10 cm and a reactor volume of 2.4 L were used in experiments (Figure 1). The anaerobic reactor has a part that allows air to be supplied with the help of a diffuser from the lower part to provide aerobic conditions. It was operated under mesophilic conditions (33-35°C). According to the meteorological data in the region, enough water was added the reactor in rainy days.

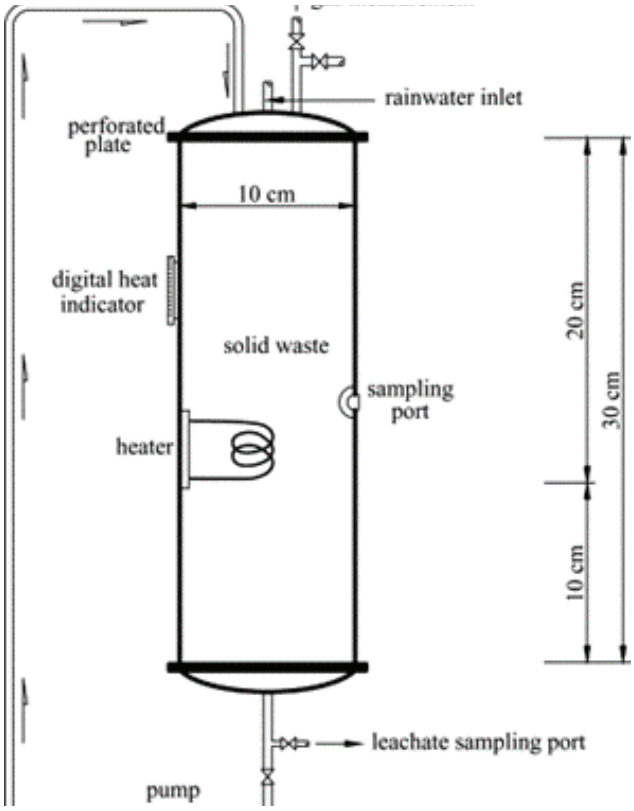


Figure 1. Laboratory-Scale Simulated Bioreactor.

Anaerobic and Semi-aerobic/ Anaerobic Bioreactor Operating Conditions

Leachate recirculation in bioreactors under anaerobic and semi-aerobic/anaerobic operating conditions was achieved with peristaltic pumps. Leachate recirculation between 10% and 15% (300 ml) of reactor volume has been shown to be appropriate in several studies in terms of methane yield [14]. The peristaltic pumps used in leachate recirculation were set to operate for 15 minutes every 8 hours with a timer. For the measurement of total gas and methane gas, specially manufactured scaled glass materials were installed in each reactor.

In bioreactors that have been operated under semiaerobic / anaerobic conditions, reactors are aerated for 1 hour in the form of 5 minutes of aeration and 3 minutes of rest with the help of a diffuser 5 days a week to ensure that aerobic conditions have been realised. After the aerobic operation process was completed, the system have been operated anaerobically for methane production.

Loading of Agricultural Solid Waste and Organic Fraction of Municipal Solid Waste Mixtures into Bioreactors

The organic fraction of municipal solid waste (OFMSW) used in bioreactors was obtained from kitchen waste in the central cafeteria of Pamukkale University, and the sunflower stalk (SS) was obtained from the sunflower pruning waste from a sunflower field in the Tavas district of

Table 1. Amount of lignin, cellulose, and hemicellulose (%) of the sunflower stalk

	Hemicellulose (%)	Lignin (%)	Cellulose (%)
Sunflower Stalk	43.67±0,8	17.23±0,2	24.46±0,3

Table 2. Water content and organic matter analysis in waste mixtures used in bioreactors

	Water Content (%)	Organic Matter (%)
RA1	71±0,3	82±3,3
RA2	76±5,2	89±4,4
RSA1	26±2,3	88±1,2
RSA2	12±0,4	85±0,3

Denizli province. The first anaerobic bioreactor (RA1) was filled with 20% OFMSW + 80% SS, and the second reactor (RA2) was filled with 50% OFMSW + 50% SS, and waste was loaded into both reactors and the reactors have been operated for 90 days.

For semi-aerobic / anaerobic treatment, the mixture waste rates were determined as 20% OFMSW + 80% SS (RSA1) and the second reactor was 100% SS (RSA2). Reactors have been operated for 90 days. Sewage sludge supplied from Denizli Wastewater Treatment Plant Anaerobic Sludge Tank was used as inoculum for bioreactors. Table 1 shows the amount of lignin, cellulose, hemicellulose (%) of sunflower stalk used in reactors. Table 2 shows the results of the water content and organic matter analysis of the prepared waste mixtures before loading into the reactors.

Analytical Methods

The total COD was measured colorimetrically by the closed reflux method in bioreactor leachate samples [15]. Leachate samples' pH were measured using the model pH meter HANNA HI 221. Volatile fatty acid concentration was measured by the titrimetric method determined by Anderson and Yang [16]. BOD_5 was measured using the WTW Oxi Top IS system. Ammonium nitrogen and Total Kjeldahl Nitrogen analyses were performed with standard method. Total gas generated in the bioreactors was measured by passing through a solution containing 10% NaCl and 2% H_2SO_4 using the liquid displacement method [17]. The methane gas generated in the bioreactors was measured by passing through a solution containing 3% NaOH using the liquid displacement method [18]. The analyses of water content and organic matter in solid waste were carried out before and after loading in bioreactors according to the standard method [15]. The contents of hemicellulose, lignin, and cellulose were determined using the processes specified by [19].

RESULTS AND DISCUSSION

Sunflower Stalk Biodegradation in Anaerobic Bioreactors

COD, pH, VFA Variations in Leachate Produced from Anaerobic Bioreactors

Figure 2 (a) shows change in COD concentrations. In leachate samples from anaerobic bioreactors, the initial COD concentrations were, respectively; 22026 mg/l in RA1 and 14250 mg/l in RA2. The COD removal yields were calculated as 90% in RA1 and 97% in RA2. Dumlu [20] reported that the digestion of agricultural waste consisting of roots, stems, leaves, and fruits resulting from the production of tomatoes, peppers, cucumbers, eggplants, and zucchini was achieved as a result of the 60.49% COD removal yield of 100 days of anaerobic digestion. The cellulose content of the agricultural waste used in that study was 26.40% and the lignin content 12.28%. It can be said that the sunflower stalk used in our study has a lower cellulose content than in this study, and higher COD removal yields are achieved as a result of mixing the sunflower stalk with OFMSW.

Acid accumulation occurred in bioreactors in the first stages of anaerobic treatment and a decrease in pH values. This situation was observed in Figure 2 (b). In order to provide alkalinity to the environment and to increase the pH values, 0.6% $NaHCO_3$ solution was added until the desired pH values were obtained at regular intervals from the 21st to the 51st day of the enterprise. pH values were measured between 4.96-7.43 and 4.98-7.48, respectively, in RA1 and Rector2.

It has been reported that VFA occur during the acidogenesis stage in anaerobic breakdown and often cause a decrease in pH, while low pH affects the activity of methanogens and thus affects a rapid decrease in methane production [21]. The VFA concentrations measured on day 38 were 6335 mg/l and 3290 mg/l in RA1 and RA2, respectively (Figure 2 (c)). VFA concentrations decreased in a similar way to COD concentrations [22]. During the operation of the reactors, RA2 had higher pH values than RA1. As a result, lower VFA concentrations were measured in RA2. VFA is inversely proportional to pH, meaning that the higher the occurrence of VFA, the lower its pH [23].

Cumulative Gas Generated in Anaerobic Bioreactors

During the decomposition of wastes under anaerobic conditions, organic matter is converted into biogas through microbial activities [24]. The cumulative total gas was measured as 12.42 L in RA1 and 18.88 L in RA2 over a 90 days operating period respectively (Figure 3). The high ratio of sunflower stalks, which had a high content of cellulose and

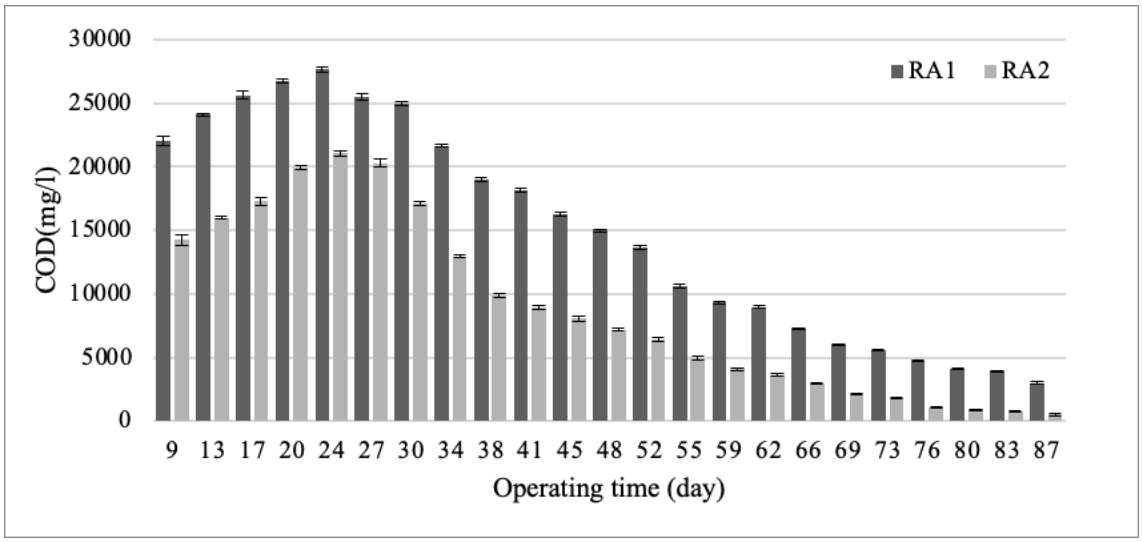
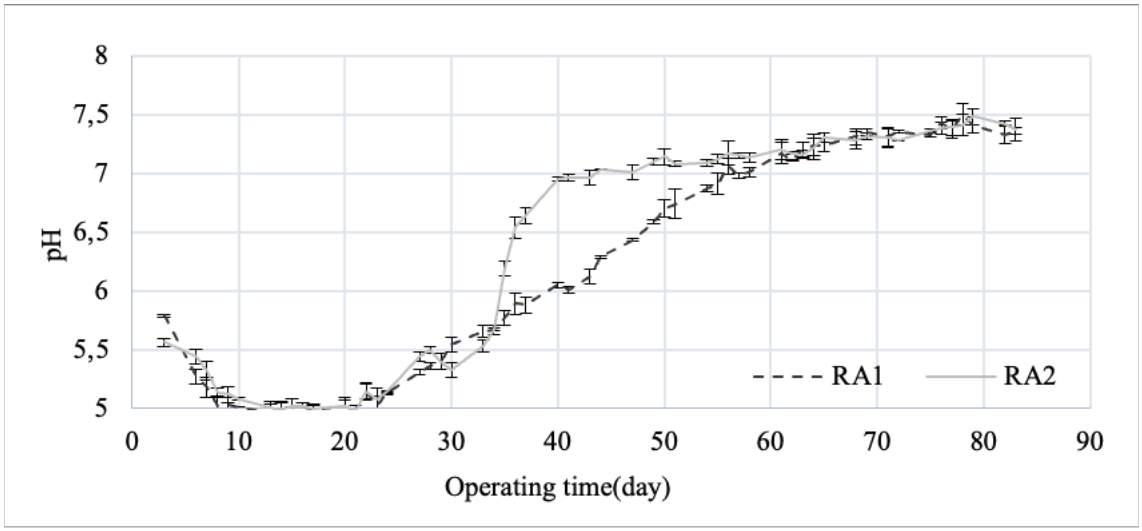
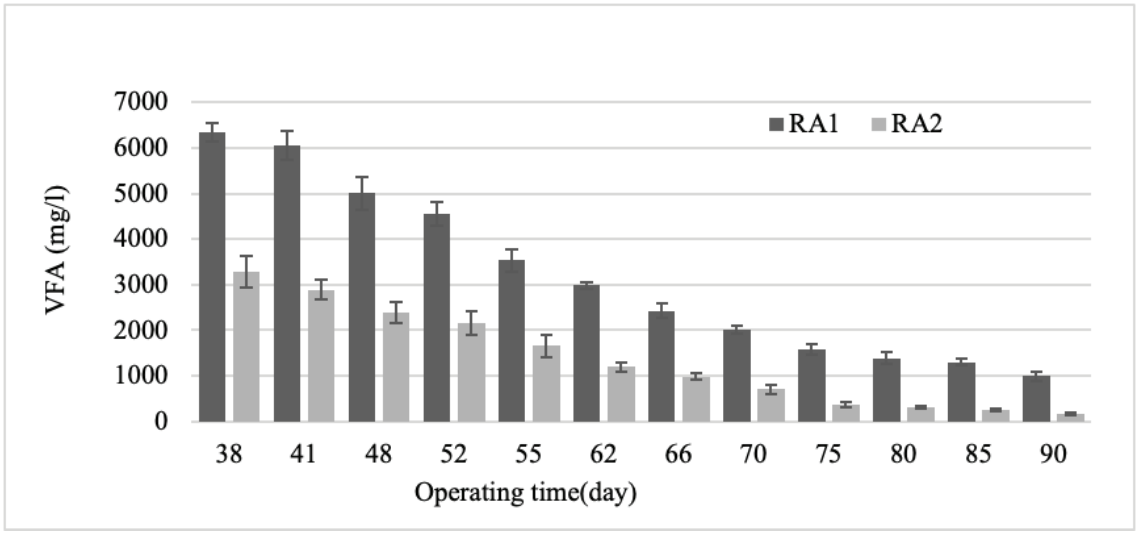


Figure 2. (a) Change in COD concentrations.



(b) pH change of bioreactor leachate.



(c) Change in VFA concentrations.

lignin in RA1 was caused to reducing of the total amount of gas. Especially in anaerobic conditions, the difficult decomposition of wastes with a high lignin and cellulose content is the most important factor in this process. Since the decomposition was slow, the total gas formation was also slow.

Cumulative Methane Gas Production in Anaerobic Bioreactors

The methane gas measured at the beginning of period is thought to be produced by hydrogen-consuming methane bacteria. The cumulative quantities of methane calculated during the 90-day operating period were measured as 3.90 L (Figure 4 (a)) in RA1 and 8.13 L (Figure 4 (b)) in RA2, respectively. The percentage of methane in the reactors is 34% in RA1 and 44% in RA2 during the operating period. In the study, in which different MBT (mechanically biologically treated) wastes containing cellulose were used, approximately half of our reactors were used, it was observed that the methane content varied between 58% and 62% [25]. In another study, Zhurka et al. [5] found a methane potential

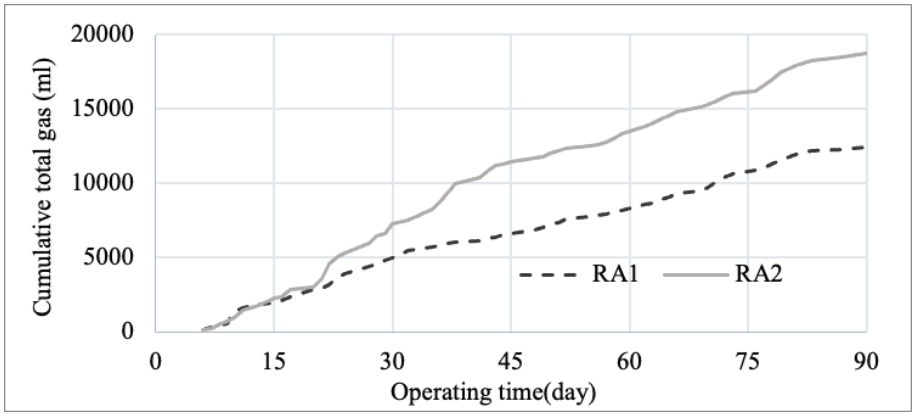


Figure 3. Cumulative total gas generated in bioreactors.

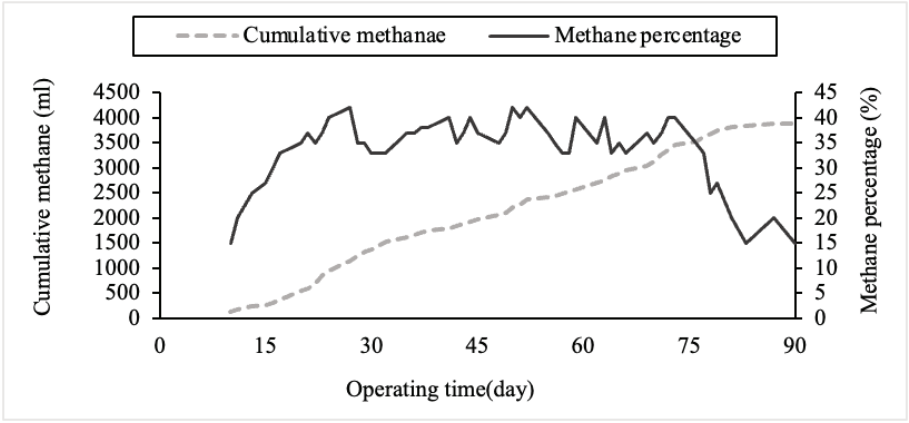
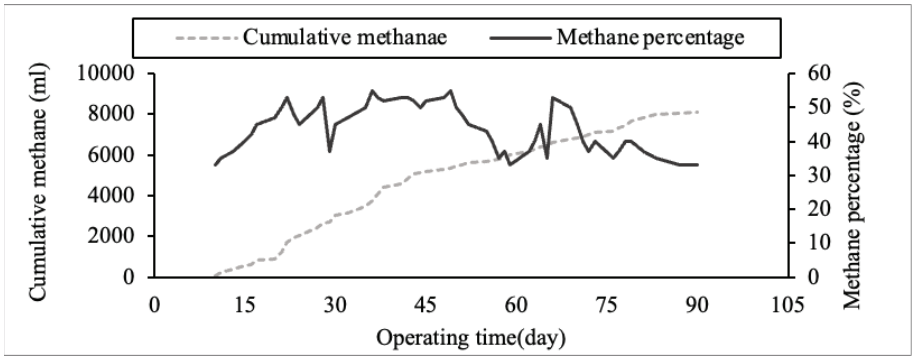


Figure 4. (a) Cumulative methane gas and percentage of methane formed in RA1.



(b) Cumulative methane gas and percentage of methane formed in RA2.

of 132 mL CH4 g^{-1} raw vs. without pretreatment and 144 mL CH4 g^{-1} raw vs. from sunflower stalks using identical alkaline pretreatment.

The highest percentage of methane was 42% in RA1 and 55% in RA2. Lignocellulosic materials are limited by their slow degradation and therefore low methane yield [26]. In parallel with the fact that the amount of sunflower stalk (with a 24.46% cellulose content) is higher in RA1 than in RA2, the amount of methane formed is less. The excess food waste in RA2 has a higher methane potential than the lignocellulosic biomass [27]. In addition, an increase in methane gas amounts was observed in parallel with COD removal yields.

Variations in BOD_5 Concentrations in Leachate Produced from Anaerobic Bioreactors

The BOD_5 measurements at different 5 days during the operation period of the bioreactors are shown in Table 3. The BOD_5/COD ratio is indicative of stabilisation in reactors, and the BOD_5/COD ratio is initially 0.16 in RA1 and 0.18 in RA2. The BOD_5/COD ratio between 0.02 and 0.13 means a low biodegradability and indicates an excess amount of organic compounds that are difficult to decompose [28]. On the 34th, 45th and 59th days of the operation, an increase in the BOD_5 /COD ratio was observed as the organic materials in the reactors were broken down and passed into the leachate. On day 73, as a result of the consumption of organic substances by methane bacteria, a decrease in the BOD_5 / COD rates was observed.

As a result of the loading of sunflower stalk wastes with high content of lignin into the reactors, low BOD_5/COD ratios are observed. When BOD_5 removal yields are calculated, it is 76% in RA1 and 92% in RA2.

Variation of NH_4-N and TKN in Leachate Produced from Anaerobic Bioreactors

When proteins break down as a result of the breakdown of organic matter, ammonium ions are released. These ammonium ions are potent inhibitors of methanogenic bacteria [29]. During the operation of the bioreactors, NH4-N (Figure 5(a)) and TKN (Figure 5(b)) concentrations were measured on 3 separate days. Low concentrations of NH4-N and TKN were measured on day due to the use of anaerobic microorganisms in the system for their N and amino acid needs, as organic materials decompose and pass into leachate. On day 75, an increase in NH4-N and TKN concentrations was observed again. This increase in concentrations indicates that the degradation of the sunflower stalk with a high lignin and cellulose content is still ongoing. Furthermore, continuous leachate recirculation in all reactors is also effective in this increase.

Table 3. Change in the BOD5 concentrations of bioreactor leachate

	RA1					RA2				
	20th day	34th day	45th day	59th day	73rd day	20th day	34th day	45th day	59th day	73rd day
BOD_5 (mg/l)	4275±469	5200±742	5376±223	3354±134	1224±202	4978±323	4538±142	2978±169	1345±183	357±26
COD (mg/l)	26714±142	21669±164	16291±55	9315±90	5565±70	19912±159	12965±127	8050±164	4075±117	1785±36
BOD_5 / COD	0.16	0.24	0.33	0.36	0.22	0.25	0.35	0.37	0.33	0.2

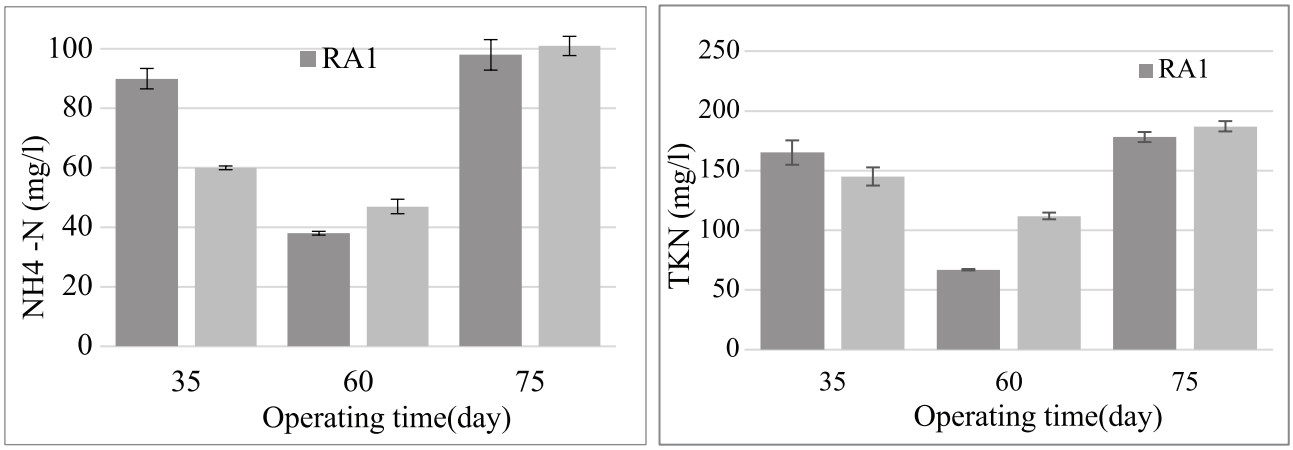


Figure 5. (a) Variation of NH4-N concentration (b) Variation of TKN concentration

Sunflower Stalk Biodegradation in Semi-aerobic/ Anaerobic Bioreactors

COD, pH, Variations in Leachate Produced from Semi-aerobic/Anaerobic Bioreactors

In leachate samples of semi-aerobic/anaerobic bioreactors, initial COD concentrations were 10181 mg/l and 8777 mg/l in RSA1 and RSA2, respectively (Figure 6 (a)). Toptas and Yay [30], in the intermittent air, increased COD concentrations in the first 38 days and the highest value was measured at 29800 mg/l. There has been a rapid decline since day 38. The COD concentration measured at the end of operation in the intermittent reactor operated for 245 days is 396 mg/l. It is seen that the oxygen supplied to the system from here has a faster decomposition effect. Similar to this study, the COD concentration in RSA1 was 651 mg/l and the COD concentration in RSA2 was 1326 mg/l as a result of the 90 days operating period. The COD removal efficiency is 97% in RSA1 and 94% in RSA2.

The optimum pH range for methane bacteria in mesophilic systems is between 6.5-8.0. When the pH increases below 6.0 and above 8.3, the process is severely restricted [31]. Figure 6 (b) shows an increase in pH values is observed from the 10th day. This increase in pH values shows that the hydrolysis and acidification phases occur faster with the aeration effect in the reactors. Cossu et al. [32] showed that pre-aeration had a beneficial effect on the increase in pH in the subsequent anaerobic phase. The highest pH values in the reactors were measured on day 42, which were 7.77 in RSA1 and 8.47 in RSA2. During the 90 days operating period, the pH values in the reactors were observed to be between 5.57 and 7.77 in RSA1 and 5.64 to 8.47 in RSA2, respectively.

Cumulative Gas Generated in Semi-aerobic/ Anaerobic Bioreactors

The cumulative gas quantities during the 90 days operating period were measured as 26.64 L in RSA1 and 39.6 L in RSA2 (Figure 7). Due to intermittent air supplied in the first 41 days of operation of the reactors, pH values were measured higher than those of the anaerobic system. When the amount of gas generated in reactors is compared, the total amount of gas in RSA2 is considerably higher than in RSA1. Although there are 100% sunflower stalks in RSA2, the amount of gas formed in parallel with the pH values is higher in RSA2 because the pH values are higher than in RSA1.

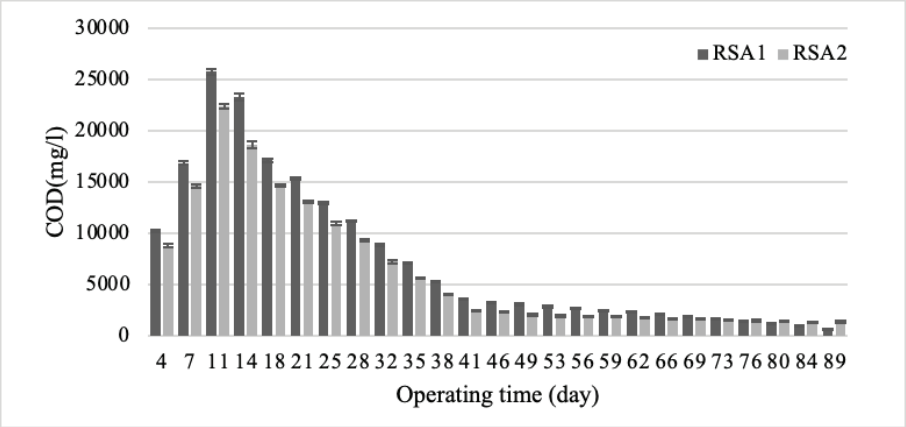
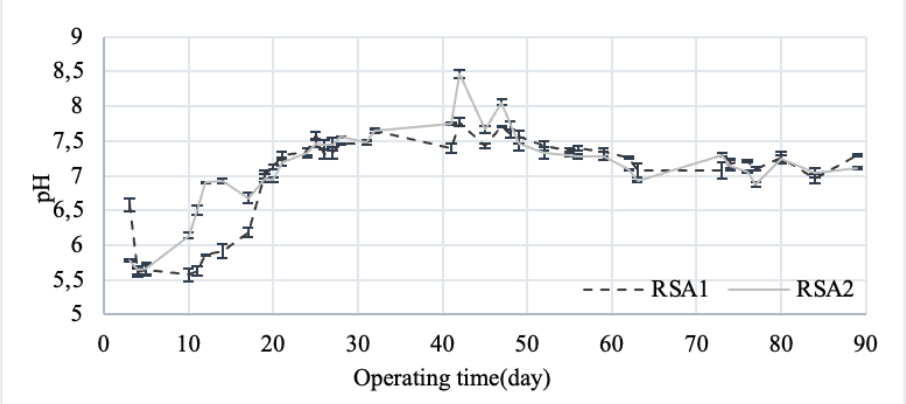


Figure 6. (a) Change in COD concentrations.



(b) Changes in pH of the bioreactor leachate.

Cumulative Methane Gas Production in Semi-aerobic / Anaerobic Bioreactors

Traditionally, exposure to oxygen or air is avoided in anaerobic digestion systems to minimise its negative impact on anaerobic microorganisms. However, it has recently been found that methanogenesis also occurs outside of anaerobic environments such as oxygenated freshwater and soil [33]. Nguyen and Khanal [34] reported that exposure to small amounts of oxygen / air (micro-aeration) benefits anaerobic digestion by increasing hydrolysis, improving methane yield, stabilizing the process, and removing hydrogen sulphide, among others. It has been observed that a high rate of methane gas is formed in reactors compared to the anaerobic system due to the air supplied to the reactors (Figure 8 (a) and (b)). When the aeration process in the reactor was completed and the anaerobic operation was

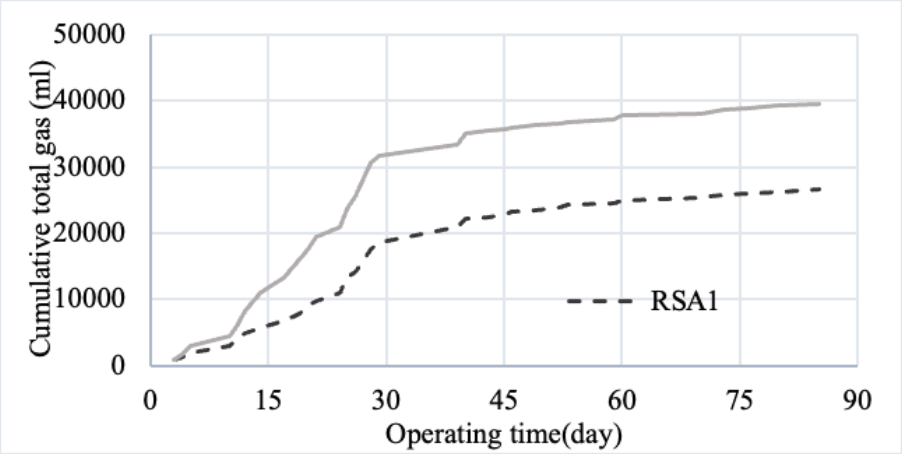


Figure 7. Cumulative total amount of gas generated in bioreactors.

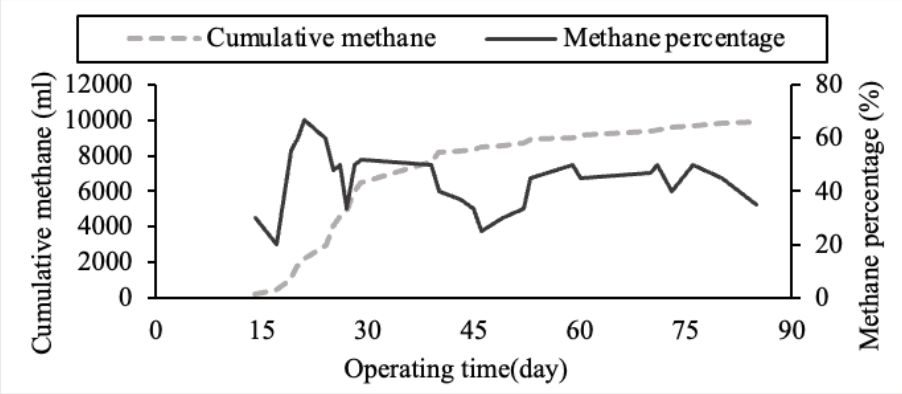
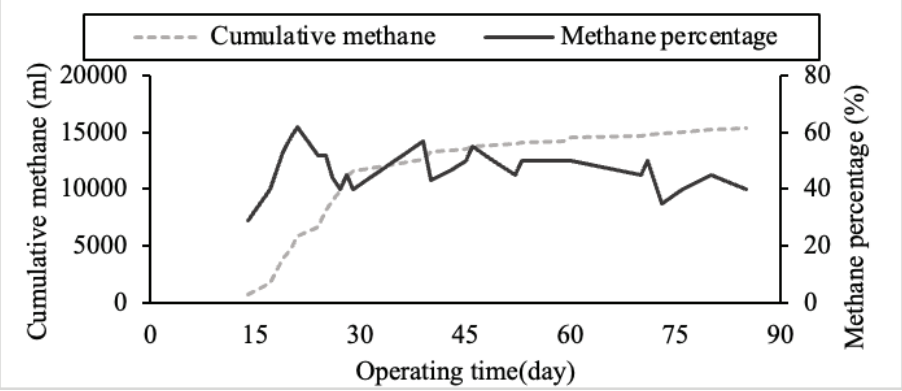


Figure 8. (a) Cumulative methane gas and percentage of methane formed in RSA1.



(b) Cumulative methane gas and percentage of methane formed in RSA2.

continued, less methane gas was formed than the aerobic part. In the aerobic part, methane bacteria consumed the organic substances in the environment and when the anaerobic part was passed, less methane gas was formed than the aerobic part due to the fact that there was less organic matter in the environment. Cumulative quantities of methane in semi-aerobic / anaerobic bioreactors were 9.93 L in RSA1 and 15.36 L in RSA2, respectively. The percentage of methane in the reactors was 44% of the total gas in RSA1 and 47% in RSA2, respectively, during the operating period. The percentage of methane was highest on day 21 at 66.6% in RSA1 and 62% in RSA2.

Variations in BOD_5 Concentrations in Leachate Produced from Semi-aerobic / Anaerobic Bioreactors

According to Table 4, it was observed that organic substances digest and pass through leachate through the air distributed to the reactors intermittently before the anaerobic system. On day 38, the BOD_5 / COD ratios increased to 0.4 in RSA1 and 0.35 in RSA2, which is an indication that organic substances are broken down and passed into leachate. BOD_5 concentrations on day 80 were 154 mg/l and 262 mg/l in RSA1 and RSA2, respectively. The BOD_5 / COD ratios are 0.13 in RSA1 and 0.19 in RSA2.

Variation of NH_4-N and TKN in Leachate Produced from Semi-aerobic/Anaerobic Bioreactors

Since the reactors were intermittently aerated during the first 41 days of operation, a decrease in TKN and NH4-N was observed (Figure 9 (a) and (b)). An increase in NH4-N concentrations was observed when the system was operated anaerobically after intermittent aeration. Nag et. al. [35] evaluated the aerobic-anaerobic storage method using intermittent aeration. The mass of ammonium nitrogen decreased sharply from its high values as a result of the stripping of air and the removal of free ammonia. The observed decrease in NH_4^+ concentrations is in line with findings from previous studies showing that hybrid conditions created by cyclic/intermittent aeration are suitable for reducing total nitrogen, especially ammonium concentrations.

Comparison of Anaerobic and Semi-aerobic/Anaerobic Bioreactors

Although RA1, one of the anaerobic bioreactors, and RSA1, one of the semi-aerobic / anaerobic bioreactors, had the same mixture ratios of wastes, in RA1, the hydrolysis phase, which is one of the advantages of the aerobic process, took place faster and in a shorter time compared to the anaerobic system, so the organic substances contained in the wastes passed into the leachate in a shorter time and higher removal efficiencies were obtained. In RSA2, although 100% sunflower stalks were used, COD decreased at faster rates in short periods in both reactors. This is because the aerobic process is faster than the anaerobic process. The COD concentrations measured at the end of a long operating period indicate that wastes with high lignin and cellulose content are better decomposed under

Table 4. Change in the BOD5 concentrations of bioreactor leachate

	RSA1				RSA2			
	21st day	38th day	56th day	80th day	21st day	38th day	56th day	80th day
BOD_5 (mg/l)	5058±613	2119±257	394±84	154±12	3923±209	1394±111	462±42	262±15
COD (mg/l)	15328 ±108	5299±42	2626±95	1186±19	13078±113	3983±55	1848±51	1377±60
BOD_5 / COD	0.33	0.4	0.15	0.13	0.3	0.35	0.25	0.19

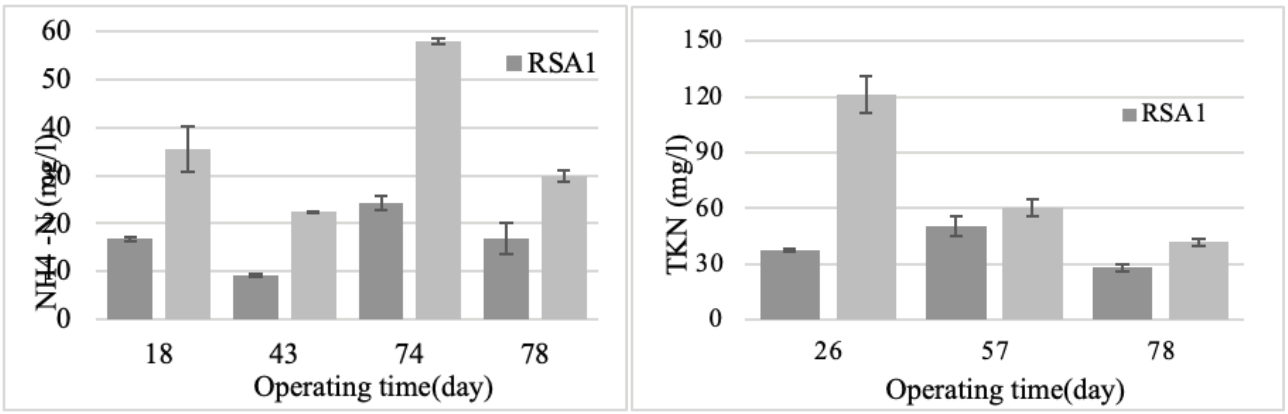


Figure 9. (a) Variation of NH_4-N concentration (b) Variation of TKN concentration

semi-aerobic / anaerobic operating conditions than anaerobic process.

The pH values in semi-aerobic / anaerobic operating conditions are higher than the pH values in anaerobic operating conditions. The reason for this is that the hydrolysis and acidification phases of the intermittent air supplied to the reactors take place at shorter times and the reactors pass to the methanization phase faster.

The total amount of gas measured in semi-aerobic / anaerobic operating conditions is higher than anaerobic bioreactor. The decomposition of sunflower stalks with a high lignin and cellulose content in semi-aerobic / anaerobic conditions occurs faster than under anaerobic operating conditions, and the pH values are higher due to the intermittent air, which causes more gas to be formed.

In comparison to anaerobic operation, the amount of methane gas formed in semi-aerobic / anaerobic operation conditions was higher than in the anaerobic system. This is since the hydrolysis and acidification phase is completed in shorter periods of time and then the transition to the methanation phase is faster due to the aeration of semi-aerobic / anaerobic bioreactors with intermittent air for 41 days. Ebrahimian et. al found that methane production from untreated stem and capitulum was very low compared to other crude samples and suggested that a pretreatment is required for anaerobic degradation of lignocellulosic biomass [36]. As in this study, aerobic pretreatment confirms the increase in the methane gas amount.

At the end of the experiment, it was observed that the removal of ammonium nitrogen in aerobic-anaerobic reactors was slightly higher than in the aerobic reactor. Studies in the literature show that a semi-aerobic bioreactor landfill can not only accelerate the landfill stabilization process and reduce the concentration of organic matter in the leachate, but also reduce the concentration of ammonia in the leachate [37, 38].

Due to intermittent aeration made due to the loading of sunflower stalk waste with a high lignin content of lignin into the reactors, higher BOD_5 / COD rates are observed according to anaerobic operating conditions with the breakdown of organic materials and their passage into leachate. When the BOD_5 removal yields are calculated, it is 97% in RSA1 and 93% in RSA2.

CONCLUSION

According to the analysis results, 90% and 97% COD removal, 84% and 94% VFA removal, 76% and 92% BOD_5 removal and 43% and 52% organic matter removal were achieved in RA1 and RA2, respectively. The cumulatively calculated total gas amounts were 12.42 L and 18.88 L in RA1 and RA2, respectively. Cumulative methane amounts were 3.895 L in RA1, 8.13 L in RA2 and methane percentage was measured as 34% in RA1 and 44% in RA1. It was observed that the decomposition rate was slower in the reactor with more sunflower stalks than in the reactor with more domestic waste due to the high lignin and cellulose content. In the next operating condition, semi-aerobic / anaerobic operating conditions, the reactor with more sunflower stalks was selected and its degradation was observed under semi-aerobic / anaerobic conditions.

Under semi-aerobic / anaerobic operating conditions, sunflower stalks were mixed with organic fraction of municipal solid waste at a ratio of 20% OFMSW + 80% SS in RSA1 and 100% sunflower stalks in RSA2 and their decomposition and methane production in bioreactors were observed for 90 days. The reactors were intermittent aerated for 41 days and anaerobically operated for 49 days for methane production. Under semi-aerobic operation conditions, RSA1 and RSA2 achieved 86% and 89% COD removal, respectively. After 90 days of operation, RSA1 and RSA2, respectively; 97% 94% COD removal, 97% 93% BOD removal and 60% and 58% organic matter removal were achieved. The cumulatively calculated total gas amounts of RSA1 and RSA2 were 26.64 L and 39.6 L, respectively. Cumulative methane amounts in RSA1 and RSA2; 9,929 L and 15,351 L methane gas was measured and the percentage of methane in the reactors was 44% and 47% of the total gas.

Due to the high lignin and cellulose content of agricultural waste, the decomposition process takes place in long stages with anaerobic treatment methods. Therefore, it has been determined that the decomposition of agricultural wastes with a pre-aerobic treatment to accelerate the decomposition process and then their anaerobic operation for methane production contribute to the decomposition process of the wastes.

ACKNOWLEDGMENT

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CONFLICT OF INTEREST

The authors declare no potential conflicts of interest regarding the research, authorship and/or publication of this article.

DATA AVAILABILITY

The data used to support the findings of this study are included within the article.

AUTHOR'S CONTRIBUTIONS

All authors are contributed equally to bring out this article.

ETHICS

There are no ethical issues with the publication of this manuscript.

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Environmental Research & Technology

Research Article

Investigation of the usability of the electrocoagulation method in malachite green removal from water solution

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ABSTRACT

In this study, the removal of Malachite Green dye in synthetically prepared aqueous solution by electrocoagulation process was investigated. In the study, initial dye concentration, electrolyte amount, mixing speed, current density, electrolysis time, pH value, and distance between electrodes parameters that affect the removal efficiency of the electrocoagulation method were investigated. As a result of the study, optimum parameters were found as initial dye concentration of 200 mg/L, electrolyte amount of 150 mg/L, stirring speed of 100 rpm, current density of 8 mA/cm^2, pH 4.5 value, the distance between electrodes 1 cm, and electrolysis time 20 min. 93.6% color removal efficiency and 37.5% COD removal were obtained under optimum conditions.

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INTRODUCTION

Negative changes in the chemical, physical, bacteriological, radioactive and ecological characteristics of the water source are defined as water pollution. Water pollution occurs as a result of the discharge of substances or energy wastes that will directly or indirectly cause preventive deterioration in biological resources, human health, fisheries, water quality and the use of water for other purposes [1].

The textile industry is one of the longest and most complex industries in the manufacturing industry. The textile industry consumes a large amount of water in its production processes. The wastewater generated as a result of the textile industry is in the toxic class. These wastewaters, which can be in different colors according to the raw material used, spoil the natural appearance of the water environment and reduce the light transmittance. The decrease in light transmittance and dissolved oxygen causes damage to aquatic organisms and prevents the use of water resources [2].

The substances used to color the materials are called colorants. These substances are mainly divided into two as dyestuff and pigment. Dyestuffs can be dissolved in the dyeing process. On the other hand, do not dissolve in the substance to which they are transported.

Malachite green (MG) is one of the basic dyes and is widely used in the dye and textile industry. MG's mixing with water and its high concentration in water cause serious problems for all living organisms, including humans, due to its toxic, carcinogenic, and mutagenic factors. In particular, it shows biological toxicities to humans and living things by consuming fish produced in Malachite green dyed or

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polluted water and by causing irritating the gastrointestinal tract. Thus, the removal of these organic dyes will directly benefit the environment and living things [3].

Many different methods are used in the treatment of dyestuffs. The toxic properties of dyestuffs and the high content of organic substances resistant to biodegradation in textile wastewater limit the applicability of biological methods [4]. In the treatment with the chemical coagulation/flocculation technique, this method cannot be preferred as a treatment alternative because the sludge formation is higher compared to other methods and because the dyestuffs are dissolved in the wastewater in the enterprises where the reactive dyeing technique is used [5]. Chemical oxidation methods are widely used in the treatment of textile wastewater and their color removal efficiency is high [5]. However, the potential for the formation of substances known to be toxic as a result of oxidation reactions is high [6].

Towards the end of the 20th century, a process called electrocoagulation was developed. This process consists of anode material, cathode material, a conductive liquid power supply components in a reactor [7].

This study, it is aimed to investigate an alternative method for the treatment of wastewater containing dyestuffs. In this context, the removal of Malachite Green dyestuff by electrocoagulation method was investigated and optimum values were determined for initial dyestuff concentration, mixing speed, pH optimization, electrolyte concentration, electrolysis time, current density, and distance between electrodes.

MATERIALS AND METHODS

Materials

Malachite green dyestuff, whose properties are given in Table 1, was obtained from Sigma Aldrich. In the study, AA TECH ADC-3303D brand power supply, Edmünd Bühler GmbH brand mixer and aluminum electrodes with 50 x 70 x 2 mm dimensions and 30 cm^2 active surface area were used. Hack DR-3900 brand spectrophotometer was used to find the dyestuff removal.

Methods

In the studies, color removal from the aqueous solution prepared in the laboratory with the dyestuff was investigated by the electrocoagulation process. The prepared 2500 mg/L stock solution was stored under cold and dark conditions and diluted with distilled water to bring it to the desired concentration.

Table 1. Properties of Malachite Green Dye

Molecule Formula	$C_{23}H_{25}ClN_2$
Molecular Weight (g/mol)	364.9
Wavelength (λ)	617 nm

The dyestuff prepared at the desired concentrations in the experiments was added to the 600 ml beakers against the risk of overflow by using 250 ml volume and put into the system. Then, the variables of dyestuff concentration, electrolyte amount, current density, pH, mixing speed, the distance between electrodes, and electrolysis time were investigated to find the optimum conditions.

Two electrodes, 1 anode and 1 cathode, were used in the monopolar connected state inside the reactor. The distance between the electrodes was adjusted to 1 cm until the optimum value was found.

In the experiments, 0.1 M HCl and 0.1 M NaOH chemicals were used to adjust the pH value. The electrodes were kept in the cleaning solution for 10 minutes before the experiments and after being washed with distilled water, they were dried and made ready for weighing and the next experiment.

During the experiment, 5 ml samples were taken from the system and the samples were subjected to centrifugation at 6000 rpm for 5 minutes and analyzed in a spectrophotometer at 617 nm.

Equations Used in Calculations

In Table 2, the equations of the calculations made as a result of this study and the explanations of the parameters applied when using these equations are given.

RESULTS AND DISCUSSION

Optimization Parameters

Initial Dye Concentration

In the electrocoagulation process working with aluminum electrodes, solutions with concentrations of 50, 100, 150, 200 and 250 mg/L were prepared and their removal efficiencies were investigated in order to determine the optimum concentration in the aqueous solution containing the dyestuff. Other parameters in the process; The amount of electrolyte was 100 mg/L, stirring speed was 100 rpm, current density was 6 mA/cm^2, the distance between the electrodes was 1 cm, the electrolysis time was 30 minutes and the original pH was used. As the concentration value increased, the removal efficiency decreased from 99.88% to 76.88%, and it was observed that the required time for removal was prolonged. At the beginning of the study, it was observed that the removal efficiency of wastewater with high concentrations was lower compared to the studies with low dye concentrations. When Figure 1 is examined, since electricity consumption is taken into account in terms of treatment cost, it is determined as 200 mg/L with less energy. Figure 2 shows the effect of initial concentration on color removal efficiency and energy consumption.

Electrolyte Amount

The pollutant removal efficiency and operating cost are related to the conductivity of the solution. The conductivity

Table 2. Equations Used in Calculations

Calculated Parameter	Equation	Parameters to use
Current Density	$J = \frac{I}{2 \times S}$	• J : Current density (mA/cm^2) • I : Current intensity (mA) • S : Electrode area
Dyestuff Removal	$Dyestuff\ removal\ (\%) = \frac{(C0 - Ct)}{C0} X100$	• C_0 : Initial dyestuff concentration • C_T : Dye concentration at time T
Mole Amount of Substance to Precipitate	$m = \frac{I\,x\,t}{n\,x\,F}$	• I : Current Intensity (Amps) • t : Time (sec) • n : Ion charge (+3 will be taken for aluminum) • F : Faraday constant (96485 ^{0}C mol^{-1})
Theoretical Amount of Dissolution at the Anode Electrode	$M_{ATe} = \mathrm{m} \times (M_W)$	• M_{ATE}: Theoretical Amount of Dissolution at the Anode Electrode • M_W : Molecular weight
Current Efficiency	$E = \frac{M_A}{M_{ATe}}$	• E : Current Efficiency • M_A : The amount of dissolution in the anode electrode material in the experiment (g)
Total amount of dissolved aluminum (M_T)	$M_T = M_A + M_K$	• M_K : The amount of dissolution in the cathode electrode in practice (g)
Energy Consumption Value	$W = \frac{V \times I \times t}{v}$	• W : Energy Consumption Value (kW.hour/m^3) • V : Potential Difference Occurring in the System • I : Applied Current Intensity • T : time (hour) • v : Total Solution Volume in the Reactor
Energy Consumption	$Ec = V \times I \times t$	• Energy Consumption (Ec) • V: potential difference (volts) • I: Current flowing through the circuit (ampere) • t: Process time (hours)
Chemical Oxygen Demand	$COD\ (mg\ L) = \frac{(A-B)\,xNx8000}{Sample\ (ml)}$	• A: Iron Ammonium Sulphate consumption of the blind (mL) • B: Iron ammonium sulfate consumption of the sample (mL) • N: Normality of Iron Ammonium Sulphate Solution (N)

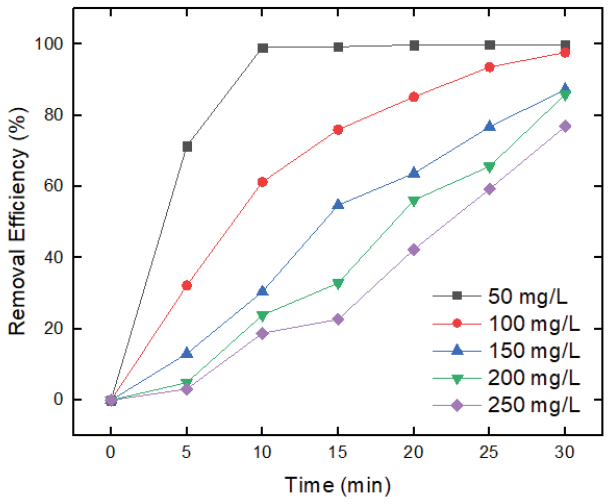

Figure 1. Results of color removal efficiency versus time with Al electrode at different initial concentrations (C_{salt}=100 mg/L, 100 rpm, J=6 mA/cm^2, original pH, Electrodes Distance=1 cm, t=30 min).

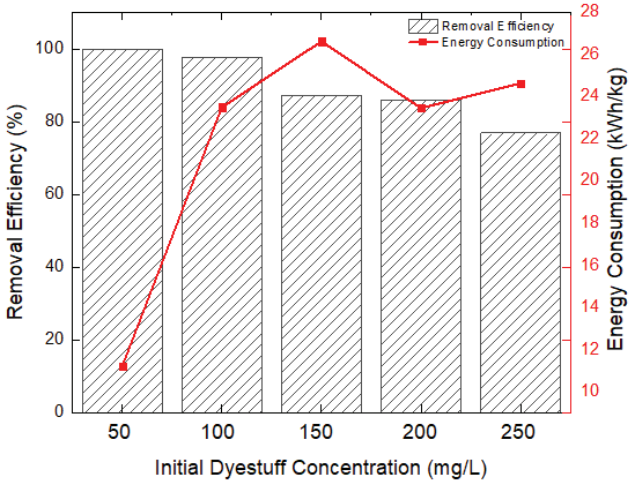

Figure 2. Effect of initial concentration on color removal efficiency and energy consumption (C_{salt}=100 mg/L, 100 rpm, J=6 mA/cm^2, original pH, Electrodes Distance=1 cm, t=30 min).

of the solution is adjusted by adding salts such as sodium chloride and sodium sulfate to the wastewater with low conductivity [8]. After the initial dyestuff concentration was determined as 200 mg/L, the amount of electrolyte was determined. In order to determine the optimum amount of electrolyte, NaCl concentrations of 50, 100, 150, 200 and 250 mg/L were added to the solution and their removal efficiencies were investigated. It was observed that as the amount of NaCl increased, the removal efficiencies increased from 69.5% to 93.5%. As the amount of added NaCl increased, the solution conductivity also increased, and accordingly, the voltage value decreased. As the voltage value decreased, the required energy amount also decreased. It was observed that the energy consumption value for kilogram pollutant amount decreased from 35.9 to 3.69 kWh/kg as the amount of electrode increased. When the kilogram pollutant amount and removal graph were examined, it was seen that the optimum NaCl amount was 150 mg/L and the removal efficiency was 88.05%. In the study, the amount of electrolyte was determined as 0.15 g/L. Higher removal was achieved with less amount of NaCl. Figure 3 shows the effect of electrolyte concentration on color removal efficiency and energy consumption.

Mixing Speed

Stirring prevents the formation of a concentration gradient from the electrocoagulation cell and increases the velocity of the ions in the cell. As the mixing speed increases, the pollutant removal efficiency increases. After the amount of electrolyte was determined, the mixing speed was determined. In order to determine the optimum mixing speed, the mixer 100, 150, 200, 250 and 300 rpm values were set on the magnetic stirrer. It was observed that the removal efficiency decreased as the mixing speed increased. Increasing

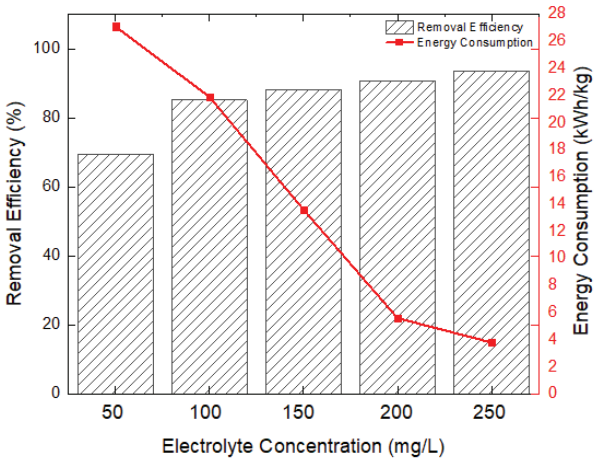

Figure 3. Effect of electrolyte concentration on color removal efficiency and energy consumption (C_0=200 mg/L, 100 rpm, J=6 mA/cm^2, original pH, Electrodes Distance =1 cm, t=30 min).

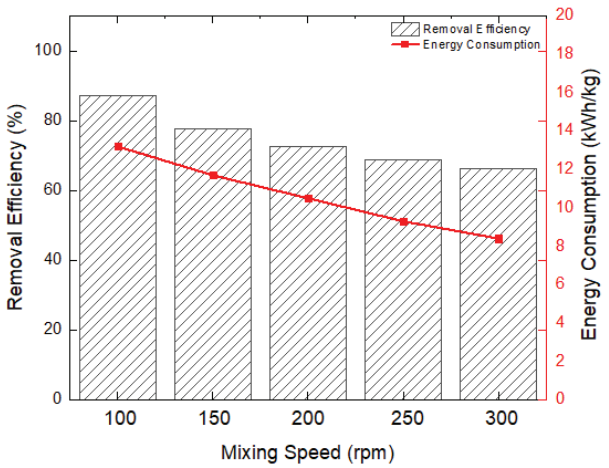

Figure 4. The effect of change in mixing speed on color removal efficiency and energy consumption (C_0=200 mg/L, C_{salt}=150 mg/L, J=6 mA/cm^2, original pH, Electrodes Distance =1 cm, t=30 min).

the mixing speed requires extra energy. In Figure 4, the effect of the change in mixing speed on the color removal efficiency and energy consumption is given. When Figure 4 is examined, it is seen that the removal efficiency decreases when the mixing speed is increased. The optimum mixing speed was determined as 100 rpm.

Current Density

Current density affects electrocoagulation efficiency, coagulation rate, bubble generation rate and size. The anodic dissolution rate increases with the increase in current density. In this way, the number of metal hydroxide clumps increases, increasing the pollutant removal efficiency [8]. It is one of the most important parameters that directly affect the removal efficiency in the electrocoagulation process. After the mixing speed was determined, the current density was determined. 2, 4, 6, 8 and 10 mA/cm^2 are set on a digital power supply to determine the optimum current density. It was observed that the removal efficiency increased as the current density increased. As the current density increases, the amount of energy required also increases. As seen in Figure 5, it has been observed that the difference between the applied current density and the removal efficiency does not increase much as it approaches the highest value. Since the removal values between 10 mA/cm^2 and 8 mA/cm^2 are close to each other, 8 mA/cm^2 was determined as the optimum current density.

pH Effect

pH is an important factor as it directly affects the reactions taking place in the electrocoagulation process. It is important both because of the formation of hydroxyl radicals at the cathode and the presence of metal hydroxides at the anode. After the current density was determined, the pH value was determined. In order to find the optimum pH

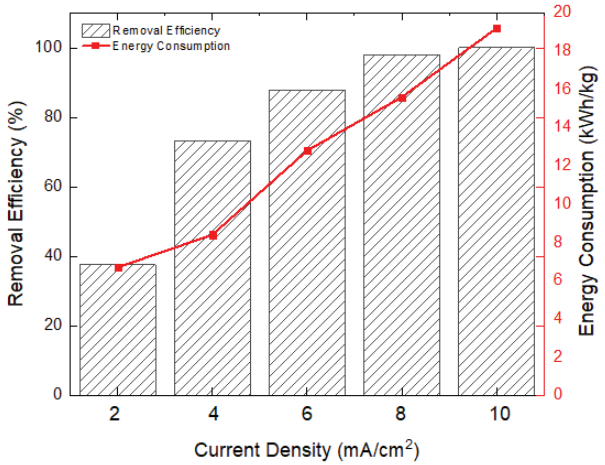

Figure 5. The effect of current density on color removal and energy consumption(C_0=200 mg/L, C_{salt}=150 mg/L, 100 rpm, original pH, Electrodes Distance =1 cm, t=30 min).

value, solutions of 2.5 -4.5 -6.5-8.5 were prepared. It was observed that the difference between pH value and removal efficiency did not increase much as it approached the highest value. At the end of 20 minutes, when the pH value was 4.5 and 6.5, the removal efficiency was found to be 98.28% and 96.95%. The optimum pH value was determined as 4.5. It has been observed that pH changes do not directly affect the processing time. The effect of pH change on color removal and energy consumption is given in Figure 6.

Distance Between Electrodes

Here, the effect of the distance between the electrodes on the removal efficiency was investigated. In order to find the most suitable distance between the electrodes, the effect on the removal efficiency was examined by opening 1-1.5-2 cm between the electrodes. It was observed that the removal efficiency decreased when the distance between the electrodes was increased. It was observed that the energy consumption increased when the distance between the electrodes was increased. When looking at the time-dependent removal graph, it was seen that the highest yield was obtained in the 1 cm range. In Figure 7, the effect of the distance between the electrodes on color removal and the energy consumption is given.

Electrolysis Time

If the electrolysis time changes, the amount of ions and hydroxide flocs produced change. In order to determine the optimum electrolysis time, the results of the previous

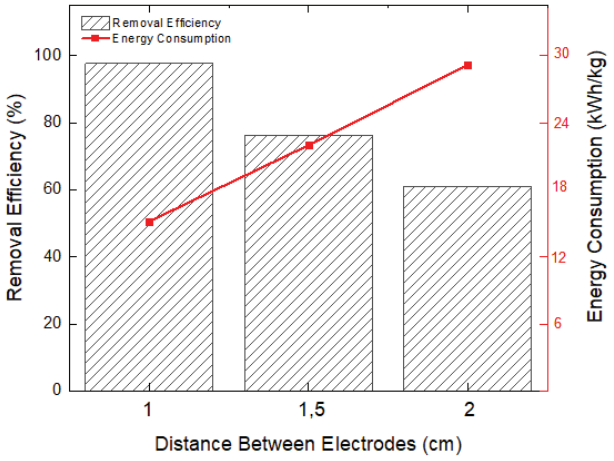

Figure 7. The effect of the distance difference between the electrodes on color removal and energy consumption (C_0=200 mg/L, C_{salt}=150 mg/L, 100 rpm, J=8 mA/cm^2, original pH, t=30 min).

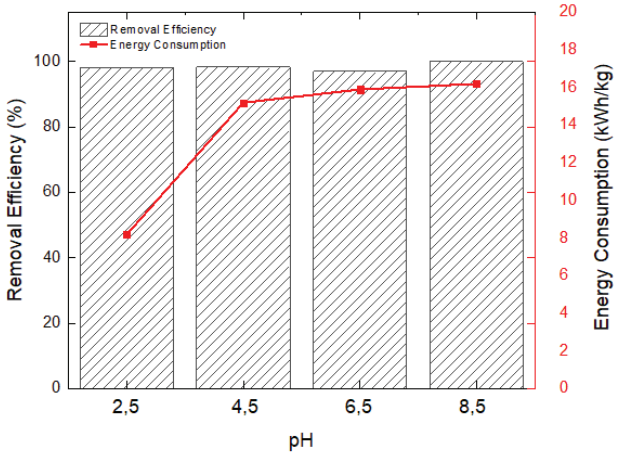

Figure 6. The effect of pH change on color removal and energy consumption (C_0=200 mg/L, C_{salt}=150 mg/L, 100 rpm, J=8 mA/cm^2, Electrodes Distance =1 cm, t=30 min).

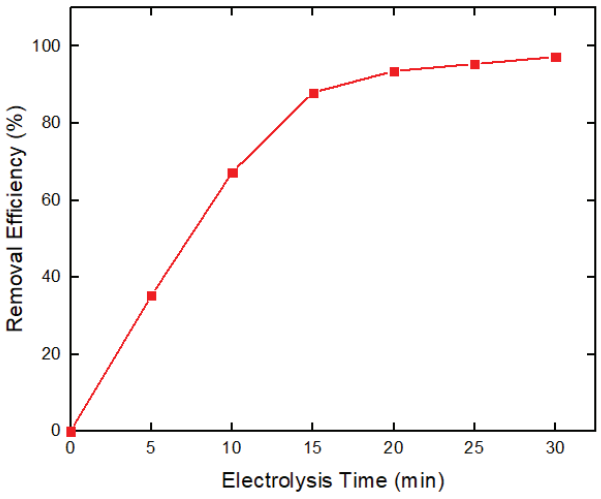

Figure 8. The effect of electrolysis time on color removal (C_0=200 mg/L, C_{salt}=150 mg/L, 100 rpm, J=8 mA/cm^2, original pH, Electrodes Distance =1 cm).

Table 3. Calculation results according to optimum values

Parameter	Results
Current Density (J)	8 mA/cm^2
Current Efficiency (E)	0,80
Total Dissolved Aluminum Amount (M_T)	0,0431 g
Energy Consumption (W)	10,58 kW.saat/m^3
Electricity Cost	5,22 TL/m^3
NaCI Cost	0,594 TL/m^3
Aluminum Cost	5,405 TL/m^3
Chemical Cost	0,594 TL/m^3

experiments were evaluated. When evaluated together with other optimum parameters in the process, the optimum time was found to be 20 minutes. It was observed that the removal efficiency increased as the electrolysis time increased (Figure 8). It has been observed that the removal is reduced if the electrolysis time is extended more than necessary.

Table 4. Analysis results at optimum conditions

	Initial	Final
Temperature ^{0}C	21	29
pH	4,40	6,17
Conductivity (µS)/cm	1728	614
COD (mg/L)	2561	1604
Color (Pt-Co)	3500	260

Calculations Based on Optimum Values

In Table 3, the results of the calculations made according to the optimum values of the study and in Table 4, the results of the analysis made under the optimum conditions are given.

COMPARE OTHER STUDIES

Table 5 shows what some other researchers have done before; optimum conditions of dye solution removal from aqueous solutions by electrocoagulation are given.

Tablo 5. Compare with other studies

DyeStuff	Parameters	Electrode	Dye Stuff Removal	References
Remazol Brillant Blue	Dyestuff Concentration: 100 mg/L, Current Density:1 A/m^2, Electrolysis time: 20 min	Al	%98.1	[9]
Remazol Brillant Blue	Dyestuff Concentration: 50-200 mg/L, Current Density: 0,5-5 A/m^2	Al	%99.6-98.2 %96.8-99.4	[10]
Procion Yellow H-EXL	Dyestuff Concentration: 500 mg/L, Elekcrolide concentration: 6,5 mg/L, Current Density: 7 mA/cm^2	Fe	%91.5-98.4	[8]
Metilene Blue	Dyestuff Concentration: 100 mg/L, Current Power: 20 A	Al	%94	[11]
Reaktif Yellow 160	Current Density: 100 A/m^2 Electrolysis time: 10 min Dyestuff Concentration: 100 mg/L,	Al	%96.4	[5]
Direct Red 23	Current Density: 0,1 mA/cm^2 Mixing Speed:150 rpm	Al	%98	[12]
Basic Blue	Current Density: 333 A/m^2 pH 8	Al	%97	[13]
Malachite Green	Dyestuff Concentration: 150 mg/L, Electrolysis time: 20 min pH 8	Steel	%99,5	[14]
Malachite Green	Dyestuff Concentration: 100 mg/L, Current Density: 76,5 A/m^2 pH 8 Electrolysis time: 30 min	Al-Fe	%99.9	[15]
Malachite Green	Dyestuff Concentration: 200 mg/L Current Density: 8 mA/cm^2 Electrolysis time: 20 min	Al	%94.5	This Study

CONCLUSIONS

In this study, the effects of electrocoagulation process operating parameters on color removal were investigated. Optimum conditions in the process; The initial dyestuff concentration was 200 mg/L, the electrolyte amount was 150 mg/L, the stirring speed was 100 rpm, the current density was 8 mA/cm^2, the pH was 4.5, the distance between the electrodes was 1 cm, and the electrolysis time was 20 minutes. During the studies, it was observed that color removal increased over time. It was observed that the color removal efficiency decreased when the initial dyestuff concentration was increased. It was observed that the amount of dissolution from the anode increased when the current density was increased. It was observed that the required power amount decreased when the amount of electrolyte added to increase the conductivity was increased. As a result of increasing the current too much, the water temperature increased after the paint was removed.

In this study, 93.6% removal efficiency and 37.5% COD removal efficiency were achieved after 20 minutes in the removal of malachite green dyestuff in an aqueous solution by electrocoagulation method under optimum conditions. Color measurement was made from the Pt-Co unit under optimum conditions and 92.5% removal efficiency was observed.

The necessary consumption calculated in the examination of the treatability of synthetic wastewater prepared with malachite green dyestuff was calculated according to the unit energy price of January 2021, and the energy cost to treat 1 m^3 of wastewater was found to be 11.22 TL/m^3. The electrocoagulation process has succeeded in providing a high rate of color removal in wastewater containing dyestuffs, but it has been found to be insufficient in COD removal. It should be used together with other treatment processes in wastewater with high COD value.

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CONFLICT OF INTEREST

The authors declare no potential conflicts of interest regarding the research, authorship and/or publication of this article.

DATA AVAILABILITY

The data used to support the findings of this study are included within the article.

AUTHOR'S CONTRIBUTIONS

All authors are contributed equally to bring out this article.

ETHICS

There are no ethical issues with the publication of this manuscript.

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Research Article

Comparison of waste lithium-ion batteries recycling methods by different decision making techniques

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ABSTRACT

Today, to reduce fossil fuel consumption and to prevent gas emissions that are increasing day by day, vehicles working with electrical energy have started to be produced and developed. The environmental impact of the batteries of electric vehicles, which are increasing in number, is an undeniable fact and is predicted to be a major problem. In this study, three different alternative recycling processes were selected for waste lithium-ion batteries (LIBs), namely pyrometallurgical process, hydrometallurgical process and direct recycling. These processes were compared in terms of their technical, economic, environmental and social aspects using a Multi-Criteria Decision Making (MCDM) approach. From this point of view, in this study, entropy method which is an objective method was used to weight the criteria and Analytic Network Process (ANP) and TOPSIS methods were used to prioritise the alternatives in order to determine the best process for the recycling of waste LIBs. The alternatives were determined as being pyrometallurgical process, hydrometallurgical process and direct recycling, and these alternatives were evaluated in terms of environmental, economic, technical, and social dimensions. Afterwards, sensitivity analysis was performed. The ranking results showed that direct recycling is the best alternative (with values of 0.68 and 0.8101 for ANP and TOPSIS, respectively). In addition, sensitivity analysis was applied for the robustness of the results. As a result of the sensitivity analysis, direct recycling was found to be the best alternative.

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INTRODUCTION

The greenhouse gas emissions caused by the increasing fossil fuel consumption with the industrialization is the most important reason for the global climate change, which has been the problem of the whole world in the last century. Today, 20% of the CO_2 emissions in the European Union countries originate from road transport and a significant amount of fossil fuels are used in road transport [1]. Internal combustion engine vehicles are the cause of a non-negligible rate of fossil fuel consumption [2]. At the Paris Climate Summit held in 2016 with the participation of many countries, the importance of using hybrid and fully electric vehicles (EVs) in reducing global warming was emphasized [3]. In addition, countries committed to

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zero-emission transport at COP27. France and Spain, one of the largest vehicle manufacturers in Europe, have signed the Zero Emissions Vehicle Declaration, aiming for 100% zero emissions in light vehicle and pickup truck sales by 2035 at the latest in leading markets and by 2040 in others [4]. EVs are seen as having zero CO_2 emissions, but to ensure this is true, energy must come from non-fossil fuel sources such as nuclear and alternative energy [5]. EVs, which are increasing in number day by day, are seen as the technology of the future in the world and their use is encouraged in most countries. On the other hand, in parallel with the production and use of these electric vehicles, the production and use of electric vehicle batteries is also increasing. As a result of the studies, it is estimated that by 2030, 140 million EVs will be on the roads worldwide, and with this, 11 million tons of Li-ion battery waste will be generated. Annual waste streams of these batteries are expected to reach 340,000 metric tons by 2040 [6]. Therefore, while aiming to minimize the damage to the environment, these batteries bring with them another problem that causes environmental pollution [7].

Lithium-ion batteries (LIBs), developed in the late 20th century, have led to technological advances in the energy storage and portable electronics and electric vehicle sectors. Compared to other batteries, LIBs stand out thanks to their features such as almost zero memory effect, low self-discharge rate and large power storage capacity with a very high energy density/weight ratio [8]. LIBs generally consist of cathode, anode, separator, electrolyte and casing with sealing function. Lithium ions move along the electrolyte from the negative electrode anode to the positive electrode cathode, during discharge. During charging, the transport of lithium ions is reversed and the ions move from the cathode to the anode. Electrons leave the electrode active material from the current collector, which is a metal with high conductivity, to reach the external circuit. The separator, which allows the electrons to leave the cell and remains between the anode and the cathode, prevents the electrodes from short-circuiting, and also allows the exchange of lithium ions [9]. The schematic representation of the LIB is given in Figure 1.

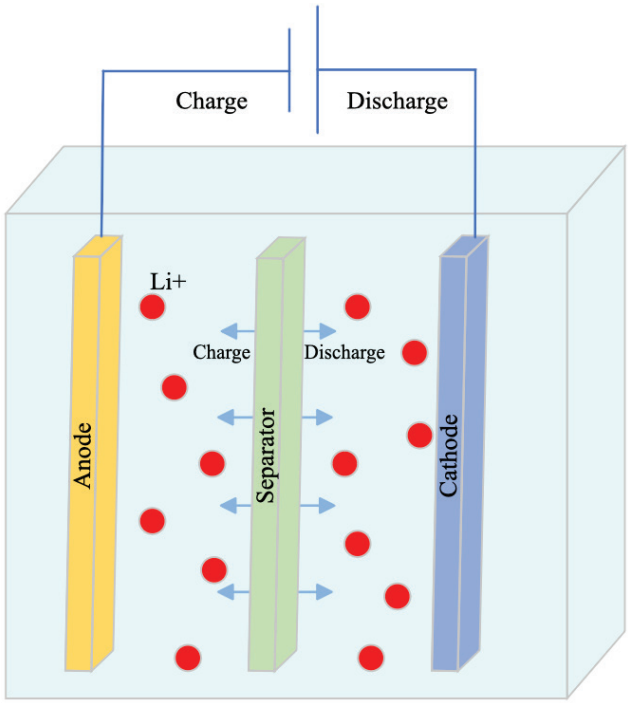

Figure 1. Schematic representation of a LIB [Adapted from 10].

LIBs generally contain transition metal oxides or phosphates, aluminum, copper, graphite, organic electrolytes containing harmful lithium salts, and other chemicals. Therefore, their reuse and recycling processes are very important. In addition, metals such as lithium, cobalt, nickel, copper and aluminum contained in LIBs are very valuable so these waste batteries must be collected and treated appropriately to prevent the disposal of potentially hazardous materials such as cobalt, nickel, manganese, cadmium, lead, etc. [10–12]. In the early 2010s, about 30% of lithium was used for the production of ceramics and glass. In the following years, batteries are thought to cause close to 60% of lithium consumption due to the use of lithium in small electronic devices such as smartphones and laptops and in larger systems such as electric vehicles and energy storage systems [13]. The increase in raw material demands from the EV market is projected to create short-term bottlenecks in lithium and battery-grade nickel supply and long-term excessive copper demand [14]. Lithium is limited as it is not a renewable resource. The supply of lithium has increased with the production of electric vehicles and, accordingly, the production and use of lithium-containing batteries. Only a small amount of used lithium is recycled, and it is thought that lithium shortages may occur if no solution is found to increase lithium recycling [15]. Looking at the production of lithium from raw materials, two hundred and fifty tons of ore spodumene (lithium aluminum silicate) or seven hundred and fifty tons of mineral-rich brine are required to produce just one ton of lithium. Processing raw materials of this scale can also have significant environmental impacts [16,17].

For these reasons above, recovery of metals is of great importance. In 2021, there were two hundred thousand metric tons of EV batteries suitable for recycling. It is predicted that this amount will reach seven million metric tons by 2035 [18]. The recycling of LIBs and the recovery of rare metals are also important for the transition to a circular economy. In the production, use and recycling of LIBs and their materials, circular economy principles are of great importance. Materials containing strategic rare earth elements such as lithium, cobalt and nickel are commonly used in lithium-ion batteries. Efficient use of these materials in a circular economy reduces the resources used and helps to sustain natural resources [19].

At this point, the use of Electrochemical Impedance Spectroscopy (EIS) technique is recommended before recycling process. This provides useful information to optimise the reuse or conversion of batteries for recycling and to minimise the environmental impact of waste batteries. EIS measurements can be used to obtain data on cell performance, to characterise the electrode material and the condition of the cell and to detect damage to the spent LIB. This increases LIBs' contribution to the circular economy and reduces the use of natural resources such as rare metals [20–22]. Thus, these precious metals can be recovered and reintroduced into production processes by recycling end-of-life LIBs [19]. The most commonly used methods for recycling LIBs are pyrometallurgical process, hydrometallurgical process and direct recycling [23–25]. In order to choose the best one among these three methods, it is necessary to consider different aspects such as environmental, economic, technical and social concepts. Taking these concepts into account, MCDM is a widely used and convenient tool for comparing methods.

According to our best knowledge, in the literature, two studies on the comparison of the processes used in the recycling of waste LIBs with Multi-Criteria Decision Making (MCDM) methods were found. The first of these were studied by Sangwan and Jindal (2012) and in this research werev developed an integrated MCDM model to compare different alternatives (disassembly, mechanical conditioning, pyrometallurgy, hydrometallurgy combination (A1), disassembly, mechanical conditioning, hydrometallurgy combination (A2), disassembly, pyrometallurgy, hydrometallurgy combination (A3), pyrometallurgy, hydrometallurgy combination (A4) and pyrometallurgy (A5)) used in the recycling of LIBs. A fuzzy Analytic Hierarchy Process (AHP) was used in the first step to calculate the weights for the different criteria. In the second step, the ranking of the different recycling processes has been calculated by means of fuzzy Technique for Order of Preference by Similarity to Ideal Solutions (TOPSIS). It was found that a combination of pyrometallurgy and hydrometallurgy was the best recycling process for LIBs [26]. In another study, Chakraborty and Saha (2022) compared 9 alternatives created by combining different recycling processes. For the comparison, fermatean fuzzy environment, entropy measure, and aggregation operators based MCDM models were developed and solved. As a result, it was seen that the Alternative 8 (blending of mechanical shredding, electrolyte extraction, electrode dissolution and cobalt electrochemical reduction) gave the best result and the Alternative 3 (blending of dismantling, acid leaching, chemical precipitation and solvent extraction) was the last in the ranking [27].

In the light of the literature studies, it is thought that there is a lack of literature on MCDM studies for the recycling of waste LIBs and more studies should be carried out with different methods. From this point of view, in this study, entropy method which is an objective method was used to weight the criteria and Analytic Network Process (ANP) and TOPSIS methods were used to prioritise the alternatives in order to determine the best process for the recycling of waste LIBs. The alternatives were determined as being pyrometallurgical process, hydrometallurgical process and direct recycling, and these alternatives were evaluated in terms of environmental, economic, technical, and social dimensions. Afterwards, sensitivity analysis was performed.

The rest of the paper is organised as follows: general information about MCDM is given in the materials and methods section and the two selected methods (ANP and TOPSIS) are described. In the same section, the selected recycling alternatives and criteria were examined in detail, and the decision matrix is constructed. Information about sensitivity analysis used to determine the stability of the results is given. The results obtained from two different MCDM methods and sensitivity analyses are presented in the results and discussion section. Finally, the conclusion summarizes the findings and indicates the future research direction.

MATERIALS AND METHODS

MCDM tools are generally used to reach the optimum decision when faced with multiple alternatives with conflicting and unmeasurable decision criteria. This method is widely used in decision making processes in science and engineering. MCDM is based on the decision-maker making a choice between at least two criteria. MCDM has many methods and when a decision maker wants to solve a problem, the first thing to do is to determine the method. Afterwards, it is necessary to create the criteria of the problem and determine the alternatives of the problem. The decision maker evaluates the alternatives based on the criteria and makes the right decision according to the best result among the alternatives [28].

Due to their different mathematical approaches, ANP and TOPSIS were preferred as MCDM methods in this study. Whereas TOPSIS is an objective method for measuring Euclidean distances, ANP is a subjective method based on pairwise comparison. This involved seeing how these different mathematical approaches might influence outcomes. In addition, ANP and TOPSIS are very active areas of MCDM research, and there are several studies that combine these two methods and compare their results [29–31].

The flowchart of the study is given in Figure 2. Firstly, the alternatives and the criteria were determined and decision matrix was performed. The criteria were weighted with the Entropy Method, and then the alternatives were evaluated with ANP and TOPSIS methods.

Determination of Alternatives

In this study, considering the literature studies, the three most commonly used methods, pyrometallurgical process,

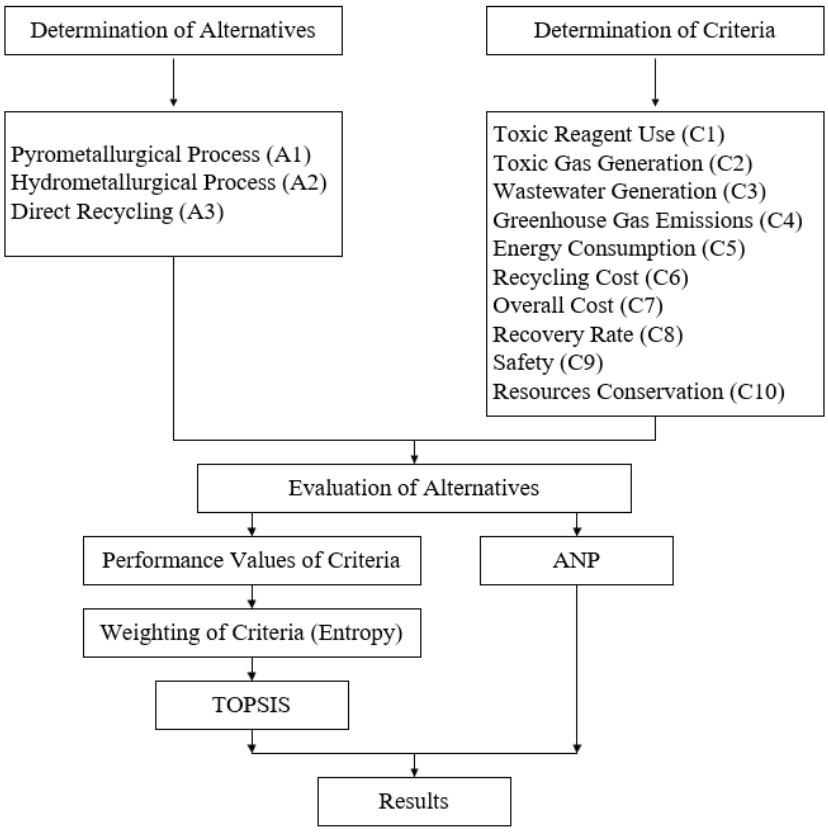

Figure 2. Flow chart.

hydrometallurgical process and direct recycling were selected as alternatives [23–25].

Pyrometallurgical Process (Alternative 1, A1)

The pyrometallurgical process is a high temperature melting process. In this process, LIBs are first incinerated in a foundry where compounds are decomposed and organic materials such as plastics and separators are incinerated. New alloys are then produced through carbon reduction. In the later stages, usually hydrometallurgical, the metal alloys are separated to recover the pure materials. In this process, only expensive metals such as cobalt, nickel and copper can be recovered with high efficiency, while the anode, electrolyte and plastics are oxidized and provide energy for the process. Lithium, aluminum, silicon, calcium and some iron are obtained in the slag phase. Slags can be processed by hydrometallurgical process to obtain pure metals or metal salts. Aluminum, on the other hand, acts as a reducer in the furnace, reducing the need for fuel [32,33]. The reason lithium cannot be recovered by pyrometallurgical processes is that organic materials such as paper, plastic and battery electrolyte burn and lithium remains in the slag. The metals contained in this slag have the potential to leak into the environment after going to storage [34].

Some of the thermal processes used during the pyrometallurgical process are; pyrolysis, melting, distillation and refining [35]. *Pyrolysis* is the decomposition of organic material by an intensive application of heat in the absence of oxygen. This process can be used to neutralize batteries and eliminate electrolytes as well as organic materials such as plastic and paper. In vacuum pyrolysis, the heating process is carried out in a vacuum in order to lower the boiling temperature and prevent secondary chemical reactions from occurring. *Melting* uses heat and chemical reduction to obtain metal, leaving slag and gases behind. *Distillation* can be used to thermally separate metals. Metals are evaporated at different temperatures and then condensed. *Distillation* can also be carried out using a vacuum. Since the decrease in pressure also reduces the evaporation temperature, there is no need for very high temperatures. In addition, thermal processes can be used to refine metals to high purity, eliminating unwanted materials [34].

During the pyrometallurgical process, a large amount of energy is consumed due to the operating temperature (~1500 °C) [36].

Figure 3 shows the flow chart of the pyrometallurgical recycling process.

The advantages of pyrometallurgical processes are as follows:

- It consists of simple operations.
- There is no need for operations such as separation and size reduction.

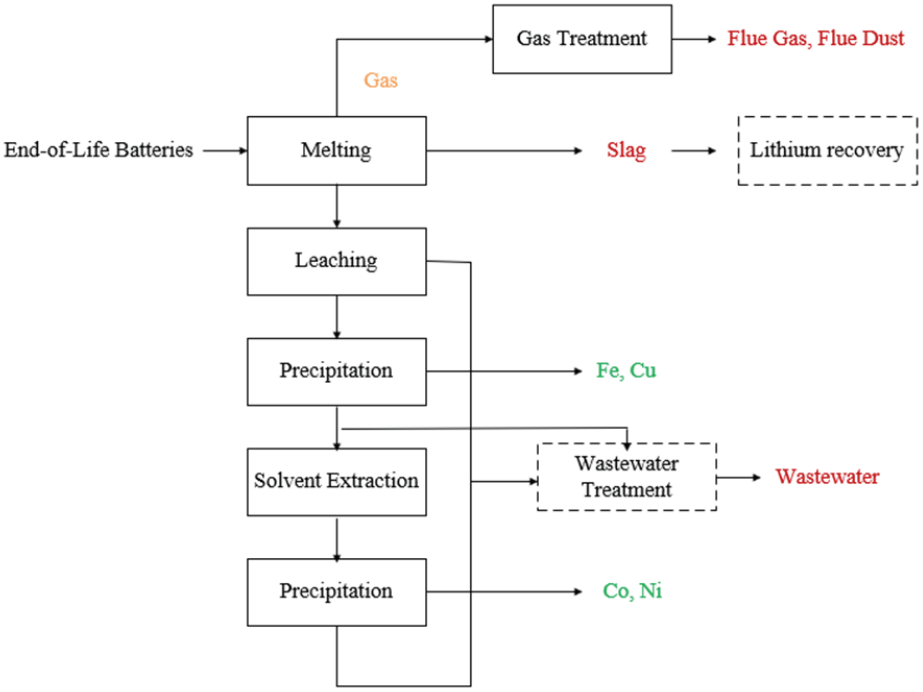

Figure 3. Flow chart of the pyrometallurgical recycling process [37].

(Green color represents products, orange color represents by-products and red color represents wastes and the dashed areas represent optional processes).

- Outputs that can be used in the production of new cathode materials are formed as a result of the process.
- The disadvantages of pyrometallurgical processes are as follows:
- CO_2 is produced during the melting process and a high amount of energy is consumed.
- The alloy requires extensive processing and is therefore costly.
- Most materials such as plastic, graphite and aluminum cannot be recovered [32].

After this process, harmful emissions may occur, including carbon dioxide, carbon monoxide, sulfur dioxide, volatile organic compounds and dust from scrap metals [34].

Hydrometallurgical Process (Alternative 2, A2)

Aqueous chemistry is used in the hydrometallurgical process. This process is carried out by leaching in acids or bases and then by concentrating and purifying [32]. This process is performed to recover LIBs after a pre-treatment [38]. Mechanical processes applied to batteries, such as shredding and dismantling, are part of the hydrometallurgical process. After these processes applied to the batteries, an acid solution is used to separate the elements. Even if the liquid solution can be used almost directly for the production of Ni-Co sulfates, elements such as lithium or copper may be lost. Shredding and disassembly can also cause material loss, and safety problems may arise depending on the amount of charge present in the battery [39]. For LIBs, ions in solution are separated by processes such as ion exchange, solvent extraction, chemical precipitation, electrolysis and precipitated as different compounds [32].

Figure 4 shows the flow chart of the hydrometallurgical recycling process.

The advantages of hydrometallurgical processes are:

- High purity materials may occur.
- Many of the LIB components are recoverable.
- The process is carried out at low temperature.
- Compared to the pyrometallurgical process, lower CO_2 emissions occur.
- The disadvantages of hydrometallurgical processes are:
- Cost increases as separation is required in this process.
- Since elements such as cobalt, nickel, manganese, iron, copper and aluminum in solution have similar properties, it is difficult to separate them and requires high cost.
- The cost is increasing to treat the resulting wastewater [32].

In hydrometallurgy, cobalt, lithium, manganese, nickel and, if present, graphite can be recovered [40]. Hydrometallurgical and pyrometallurgical processes can also be used together. With the pyrometallurgical process, the safety problems that may arise from the different chemical compositions, structures and charge states of the batteries are eliminated. In the hydrometallurgical process, the

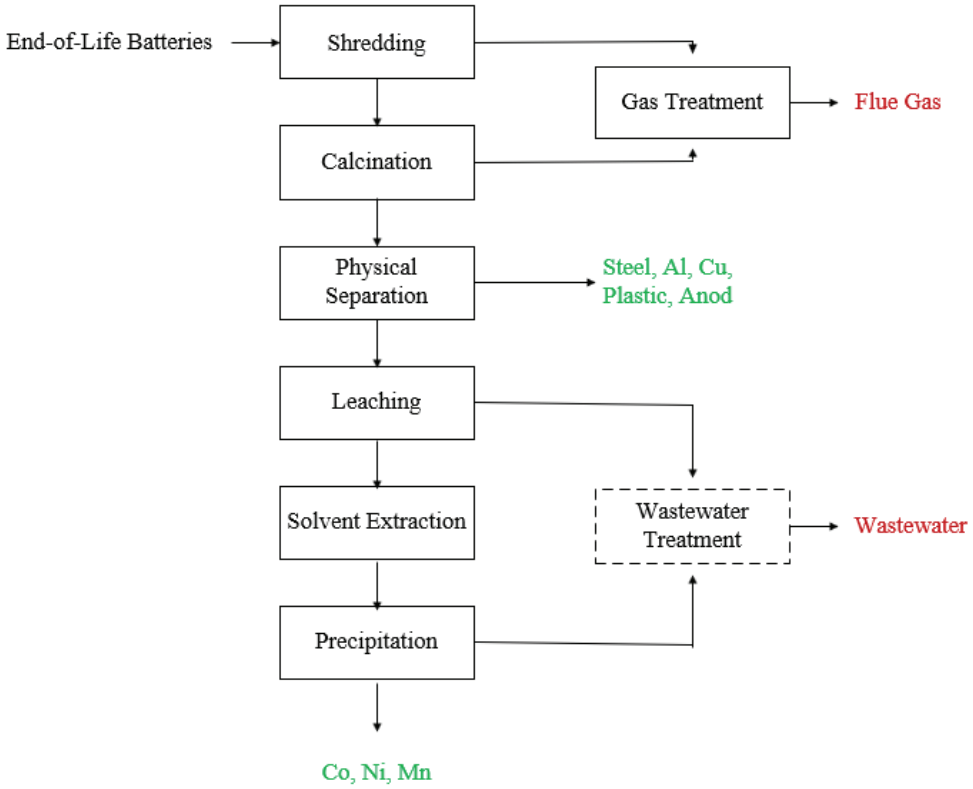

Figure 4. Flow chart of the hydrometallurgical recycling process [37].

(Green color represents products, red color represents wastes and the dashed areas represent optional processes).

separation and processing of the materials recovered in the slag is carried out with the help of different types of acids and chemicals [39].

Direct Recycling (Alternative 3, A3)

Direct recycling aims to recover the cathode material without any chemical change in the structure of the recovered material and to produce new batteries by renewing them. It is a physical recovery method in which processes such as separation by using gravity and magnetic separation are in question [9].

Significant energy inputs are needed for extensive processing of material recovered in pyrometallurgical and hydrometallurgical processes [41]. With the direct recycling method, in principle, the wastes produced during recycling are minimized and the cathode materials are recovered as reusable cathode mixtures instead of individual metals [42].

The direct cathode recycling process, similar to hydrometallurgical processes, begins with an evacuation and disassembly step, in which the external cell equipment can be individually removed and recycled. Direct cathode recycling involves removing the electrolyte using liquid or supercritical CO_2, then reducing the size of the recovered components and separating the cathode materials [41].

In this process, first of all, end-of-life LIBs are discharged and disassembled until they reach the cell level. They are then treated with supercritical CO_2, which can extract the electrolyte. After reducing the temperature and pressure, the CO_2 is removed from the electrolyte and this electrolyte can be reused for the manufacture of new batteries. Cells that do not contain electrolytes are separated and broken down. The cell components are then separated using physical methods, and the cathode materials can be brought together and reused in new batteries. Direct cathode recycling, which saves and regenerates powder cathode material for use in subsequent batteries, draws attention due to its low energy consumption and high recovery rate [43].

Figure 5 shows the flow chart of the direct recycling process.

High temperature, strong acid leaching and extensive gas purification are required for pyrometallurgical and hydrometallurgical recycling processes. This results in high costs, high energy consumption, water pollution and recycled materials with low resale value. Since cathode materials account for 30-40% of the total cost, a cost-effective and environmentally friendly direct recycling process providing reusable cathode materials can reduce energy consumption and cost of battery materials [44], [45]. Considering

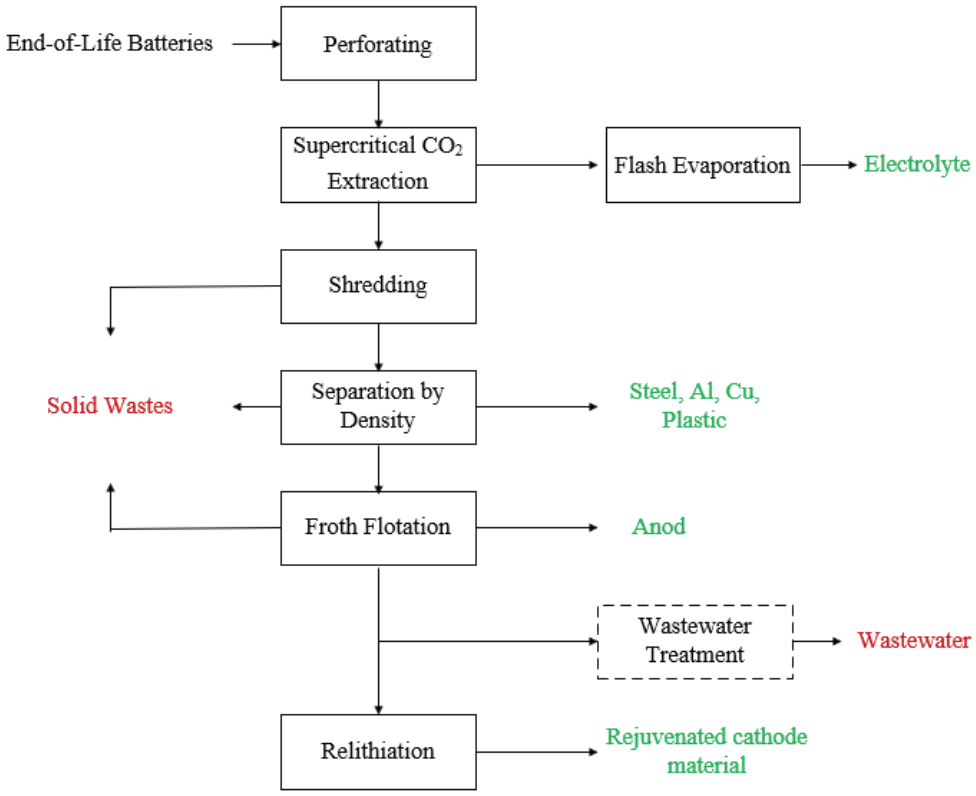

Figure 5. Flow chart of the direct recycling process [37].

(Green color represents products, red color represents wastes and the dashed areas represent optional processes).

all recycling processes, no recycling process can recover all materials and a small amount of waste has to be sent to landfill [34].

A comparison of the methods is given in Table 1.

Determination of Criteria

As a result of the literature survey, ten criteria have been defined in environmental, economic, technical, and social dimensions. These criteria and their explanations are given in Table 2.

Performance Values of Criteria

Considering the following explanations for the criteria, the decision matrix was created and then the mentioned methods were applied.

(C1) Toxic reagent use: In this criterion, the scoring was determined as 2 for pyrometallurgy, 8 for hydrometallurgy, and 4 for the direct recycling method. Although the hydrometallurgical process promises high efficiency for recycling, strong, dangerous and environmentally harmful acids are used in this process. In this method, preference of organic acids instead of dangerous inorganic acids is one of the important focus points [46]. In the direct recycling method, toxic solvents and acids are rarely used. In direct recycling, most methods that focus on re-functionalizing the active material are based on high-temperature treatments [9].

(C2) Toxic gas generation: Scoring made in this criterion was determined as 8 for pyrometallurgy, 4 for hydrometallurgy, and 2 for direct recycling [34].

(C3) Wastewater generation: In this criterion, the scoring was determined as 3 for pyrometallurgy, 8 for hydrometallurgy, and 2 for the direct recycling method. Wastes resulting from hydrometallurgical processes; the leaching stage is water and chemicals from co-precipitation and washing. In order to reduce or eliminate the generated wastewater and related costs, research on wastewater treatment, water reuse or reducing the amount of water in the process is still ongoing [32]. In the hydrometallurgical process, difficulties in recovery of low pH leaching and leaching of metals such as Al, Cu and Fe, formation of harmful by-products such as Cl_2, SO_x and NO_x and the resulting wastewater treatment are considered as important problems [6]. In the last stage of this process, wastewater is formed in the solvent extraction method used for separation [46].

Table 1. A comparison of the methods [9,37,38]

	Pyrometallurgical Process	Hydrometallurgical Process	Direct Recycling
Type of the Method	**Chemical**	**Chemical**	**Physically**
Recovered Materials	Copper compounds	Copper	Copper
	Iron compounds	Steel	Steel
	Co^{2+} in output	Aluminum	Aluminum
	Ni^{2+} in output	Graphite	Graphite
	Lithium compounds	Plastics	Plastics
	Aggregate (from slag)	Lithium carbonate	LCO
		Co^{2+} in output	NMC (111)
		Ni^{2+} in output	NMC (622)
		Mn^{2+} in output	NMC (811)
		Electrolyte solvents	NCA
		Electrolyte salts	LMO
			LFP
			Electrolyte solvents
			Electrolyte salts
Advantages	High recycling rates	High recycling rates	Environmentally friendly
	Solvent free	High purity product formation	High specificity
	Simple operation	A wide variety of metals are recovered	Low energy consumption
		Low energy consumption	High recovery rate
		Less waste gas	Reduction in recovery costs
Disadvantages	High temperatures are needed	Complex process	Not specific
	May need other operations to effectively recover materials	Usage of toxic reagents	Does not allow simultaneous processing of different cathode materials
	Li and Mn are not recovered	Costly operation	
	More toxic gas generation and toxic gas treatment costs	Excess wastewater generation	High operational and equipment requirements are needed
	High energy consumption	Long processes	

LCO: Lithium cobalt oxide, NMC: Lithium nickel manganese cobalt oxide, NCA: Lithium nickel cobalt aluminum oxide, LMO: Lithium manganese oxide, LFP: Lithium iron phosphate

Table 2. Used criteria

Criteria No	Criteria Group	Criteria Name	Unit	Preference
C1	Environmental	Toxic Reagent Use	1-9	Decreasing
C2		Toxic Gas Generation	1-9	Decreasing
C3		Wastewater Generation	1-9	Decreasing
C4		Greenhouse Gas Emissions	kg/kg	Decreasing
C5		Energy Consumption	MJ/kg	Decreasing
C6	Economic	Recycling Cost	$/kg	Decreasing
C7		Overall Cost	$/kg	Decreasing
C8	Technical	Recovery Rate	1-9	Increasing
C9		Safety	1-9	Increasing
C10	Social	Resources Conservation	1-9	Increasing

(C4) Greenhouse gas emissions: Greenhouse gases originate from the smelting process in the pyrometallurgy process. In the hydrometallurgical process, the upstream production of chemicals contributes significantly to total greenhouse gas emissions. Based on 1 kg of end-of-life LIB, 2.21 kg of greenhouse gas is emitted for the pyrometallurgy process and 2.27 kg for the hydrometallurgy process. For the direct recycling method, this value is only 0.5 kg, and since this value is significantly lower than other methods, the direct recycling method has the potential to reduce emissions and be economically competitive [46]. According to a study, the direct recycling method has the potential to reduce greenhouse gas emissions from cathode material recovery by 81-98% and SOx emissions by 72%-100% [9].

(C5) Energy consumption: For end-of-life LIBs (NMC), the energy consumption per kg for the direct recycling method is about 4.2 MJ, which is only 25% and 13.8% of

the pyrometallurgical and hydrometallurgical processes, respectively. Lighter processing conditions are a big factor in the direct recycling method [46]. According to a study, energy consumption per kg of $LiCoO_2$ is 108 MJ for pyrometallurgy, 89 MJ for hydrometallurgy and 91 MJ for direct recycling. The energy consumption for $LiCoO_2$ production per kg without any recycling process is approximately 151 MJ [25]. The average values were calculated as 108 MJ and 16.8 MJ for the pyrometallurgy process, 30.4 MJ and 89 MJ for the hydrometallurgy process, and 4.2 MJ and 91 MJ for the direct recycling method. The energy consumption values for pyrometallurgy, hydrometallurgy and direct recycling were determined as 62.4 MJ, 59.7 MJ, 47.6 MJ, respectively.

(C6) Recycling cost: Recycling costs are set at \$2.9, \$2.2 and \$1.6 per kg for pyrometallurgy, hydrometallurgy and direct recycling, respectively. The difference in recycling costs between the three methods is small because the costs include not only energy and material input, but also operating labor, maintenance and repair, and laboratory costs [46]. The final step of the hydrometallurgical process involves the separation and purification of the separated and filtered metallic components of the LIBs. Common processes such as solvent extraction, chemical precipitation and electrochemical deposition are used for separation. The solvent extraction process is widely used because of its ion selectivity advantages and the high extraction efficiency (>95%) offered by the many available extractants. The disadvantage of this process is the high upfront cost of extractors and the waste treatment cost, given the large volume processes [47].

(C7) Overall cost: Overall costs are \$0.5, \$0.3 and \$0.4 for pyrometallurgy, hydrometallurgy and direct recycling, respectively. Overall costs include administrative costs, distribution and selling costs, and R&D costs, and there is no significant difference between these three recycling methods. Although the costs of the three recycling methods are close to each other, the direct recycling method generates the highest profit compared to the hydrometallurgical and pyrometallurgical process because new LIBs can be produced directly with this method [46].

(C8) Recovery rate: Scoring was made based on the rate of material that pyrometallurgy, hydrometallurgy and direct recycling methods could recover, and this scoring was determined as 3 for pyrometallurgy, 5 for hydrometallurgy, and 8 for direct recycling. Dai et al. [37] showed material recovery efficiency for different recycling methods in their study. Looking at the values given in their study for plastics and electrolyte solvents, a material recovery efficiency of 50% is assumed as there may be less incentives for recycling compared to higher values of cobalt and nickel or metals with generally more stable demands. Besides, due to the lack of data on the direct recycling method, it is assumed that this method has the same material recovery efficiency (excluding the cathode materials) as the other two recycling methods. The recovery efficiency for cathode materials is assumed to be 90%, given the difficulties associated with separating the cathode material from the rest of the battery components.

(C9) Safety: In this criterion, the scoring was determined as 4 for pyrometallurgy, 2 for hydrometallurgy, and 7 for the direct recycling method. In the pyrometallurgical process, there is little safety risk in this process, as the cells and modules are all exposed to extreme temperatures with a reductant for metal reclamation [48]. In the pyrometallurgical process, there are different heating stages of the furnace during combustion. Thanks to the slow heating of waste batteries, the risk of explosion is reduced. In the hydrometallurgical process, there may be material loss due to shredding and dismantling, and safety problems may arise depending on the amount of charge present in the battery. Explosions may occur if batteries are not discharged before they rupture [39].

(C10) Resources conservation: In this criterion, the rate of recycled material and the amount of energy consumed were taken as a basis and scoring was made accordingly. This scoring was determined as 3 for pyrometallurgy, 5 for hydrometallurgy, and 8 for direct recycling.

In the application of the entropy and TOPSIS methods, the decision matrix was first created (Table 3).

Weighting of Criteria (Entropy Method)

The Entropy Method, also known as Shannon's Entropy method, is one of the objective weighting methods that is based on completely unbiased data and can overcome the shortcomings of subjective weighting methods. The Entropy Method, which is versatile and efficient, eliminates human-induced problems and gives results that are more in line with the facts [49]. One of the reasons why this method is suitable for use in MCDM problems is that it allows calculating the importance weights of the criteria without resorting to personal judgments and considerations [50]. Since the entropy weight indicates the degree of useful information, it can be concluded that the criterion with the larger entropy weight is more important in terms of decision making/evaluation [51].

Table 3. Decision matrix

	C1	C2	C3	C4	C5	C6	C7	C8	C9	C10
A1	2	8	3	2.21	62.4	2.9	0.5	3	4	3
A2	8	4	8	2.27	59.7	2.2	0.3	5	2	5
A3	4	2	2	0.5	47.6	1.6	0.4	8	7	8

The Entropy Method consists of the following steps:

Step 1: A decision matrix (Equation 1) is made, where the rows consist of alternatives and the columns consist of criteria. This situation is as follows for the decision matrix K:

$$K = \begin{bmatrix} b_{11} & \cdots & b_{1n} \\ \vdots & \ddots & \vdots \\ b_{m1} & \cdots & b_{mn} \end{bmatrix} \text{m x n} \quad (1)$$

Step 2: In order to get rid of the differentiation in units, the decision matrix is normalized and the Equation 2 is used to normalize the criteria:

$$S_{ij} = \frac{b_{ij}}{\sum_{j=1}^{m} b_{ij}} \text{ j = 1, 2 ,…, m; i = 1, 2 ,…, n} \quad (2)$$

Step 3: Entropy values (ed_j) are calculated according to Equation 3:

$$ed_j = -k \sum_{j=1}^{m} S_{ij} \ln S_{ij} \quad (3)$$

k = entropy constant = $(lnm)^{-1}$

$0 \leq ed_j \leq 1$ and $s_{ij} lns_{ij} = 0$ if $s_{ij} = 0$

However, ed_j values increase as the information transmitted by the j_{th} criterion decreases.

Step 4: The degree of differentiation (Equation 4) is calculated for each criterion:

$$fd_j = 1 - ed_j \quad (4)$$

Step 5: The weight value (Equation 5), which is the degree of importance, is found for the i_{th} criterion:

$$ad_j = \frac{fd_j}{\sum_{i=1}^{n} fd_i} \text{ j=1, 2, …, m} \quad (5)$$

Looking at the above equation, it can be stated that criteria with large entropy weights are more important [52].

Evaluating of Alternatives (Analytical Network Process (ANP))

ANP is one of the widely used multi-criteria decision-making methods to solve various real-world problems due to its ability to consider the interrelated and complex relationships between decision elements and its ability to simultaneously apply qualitative and quantitative attributes. ANP provides a network of relationships between criteria that leads to more reliable results. Calculates complex relationships between decision elements by replacing a hierarchical structure with a network structure [53]. A decision problem analyzed with ANP is usually examined through a hierarchy or network of controls for benefits, costs, opportunities and risks. ANP uses the same basic comparison scale (1-9) (Table 4). This comparison scale enables the decision maker to intuitively combine experience and knowledge and show how many times an item dominates another item according to the criteria [54].

Step 1: In this step, the problem is defined and a decision model is created. The purpose, criteria, sub-criteria and alternatives related to the problem are clearly stated.

Step 2: The relationships between the criteria and sub-criteria of the problem are determined.

Step 3: Priority vectors are calculated from pairwise comparisons between criteria.

Step 4: Consistency analyzes of the comparison matrices are performed. To determine whether the comparisons are consistent, the Consistency Ratio (CR) must be calculated for each matrix after the comparison matrices have been created. The CR is obtained by dividing the Consistency Index by the Randomized Consistency Index. If the CR value is less than 0.10, it can be said that the pairwise comparisons are consistent. If values are greater than 0.10, there is inconsistency in the comparison. In this case, the comparisons should be repeated.

Step 5: The supermatrix is created. A new matrix is created by multiplying all the values in the unweighted supermatrix and the weights of the set. This matrix can be expressed as a weighted super matrix. All columns of the matrix are the same, and each gives the relative priorities of the elements in each set, with the priorities of the elements

Table 4. Comparison scales used in ANP [55]

Importance Level	Definition	Explanation
1	Equally Important	It contributes equally
3	Moderately Important	One option is slightly preferable than the other
5	Strongly Important	One option is strongly favored over the other
7	Very Strongly Important	One option is strongly favored over the other, and its superiority is clearly evident
9	Extremely Important	Evidence in favoring one option over another has the highest possible degree of validation
2, 4, 6, 8	For compromise between the above values	They are used when there is no good word to define for compromise. They represent the average values that can be given.

normalized to one. The supermatrix is taken to a power large enough to equalize the priorities at some point. The resulting matrix is called the limit supermatrix.

Step 6: The best alternative is chosen. With the resulting limit supermatrix, the importance weights for each criterion are determined. The best alternative in the problem of choice is the one that is the most important alternative and has the highest importance in the decision process.

The Super Decision program was used and "benefit-opportunity-risk-cost" analysis and "benefit-cost-risk (BCR)" analysis was performed to evaluate the recycling methods of LIBs. In order to achieve this, benefit and opportunity clusters were combined [56].

The following formulation is used in the program:

Formula: bB + oO + c (1/C) + r (1/R)

Here, firstly, evaluations were made within each cluster and the weights of each cluster were used as r = 1/2, c = 1/3, b = 1/6 and o = 0 to reach the result.

In Figure 6, BCR model for ANP are shown.

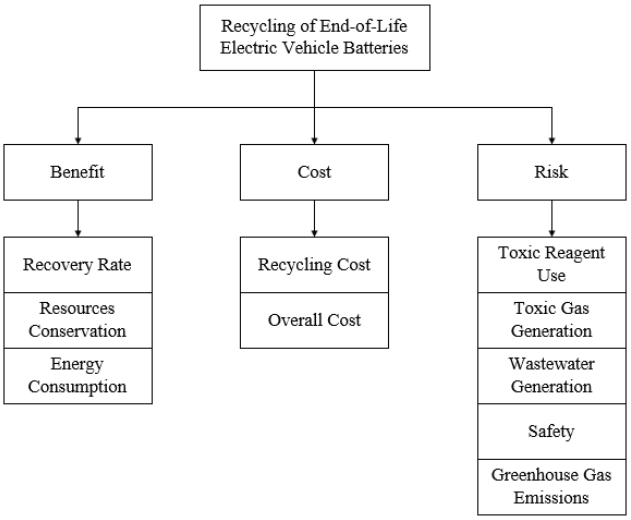

Figure 6. Benefit-cost-risk cluster criteria.

Evaluating of Alternatives (TOPSIS)

TOPSIS, which was first developed by Hwang and Yoon (1981), was developed based on the idea that the chosen alternative should have the shortest distance from the positive ideal solution and the farthest distance from the negative ideal solution to solve the multi-criteria decision-making problem. However, the best alternative should not only have the shortest distance to the positive ideal solution, but also the longest distance to the negative ideal solution. In short, the positive ideal solution consists of all the best achievable values of the criteria, while the negative ideal solution consists of all the worst achievable values of the criteria [57].

The steps used to implement TOPSIS are as follows:

Step 1: A normalized decision matrix (Equation 6) of useful and non-useful criteria is created.

$$r_{ij} = \frac{xij}{\sqrt{\sum_{j=1}^{J} xij^2}}, \; j = 1, 2, 3, \ldots, J; \; i = 1, 2, 3, \ldots, n \qquad (6)$$

x_{ij} = original score of the decision matrix

r_{ij} = normalized score of the decision matrix

Step 2: A weighted normalized decision matrix is created by multiplying the w_i weights of the evaluation criteria with the normalized decision matrix r_{ij} (Equation 7).

$$v_{ij} = w_{ij} * r_{ij}, \; j = 1, 2, 3, \ldots, J, \; i = 1, 2, 3, \ldots, n \qquad (7)$$

Step 3: The positive ideal solution (Equation 8) and the negative ideal solution (Equation 9) are determined.

$$A^+ = \{v^*_1, v^*_2, \ldots, v^*_n\} \text{ Maximum values} \qquad (8)$$

$v^+_i = \{\max(v_{ij}) \text{ if; } j \in J; \min(v_{ij}) \text{ if; } j \in J^-\}$

$A^- = \{v^-_1, v^-_2, \ldots, v^-_n\}$ Minimum values

$$v^- = \{\min(v_{ij}) \text{ if; } j \in J; \max(v_{ij}) \text{ if; } j \in J^-\} \qquad (9)$$

Step 4: The separation measures of each alternative from the positive ideal solution (Equation 10) and the negative ideal solution (Equation 11) are calculated.

$$s_i^+ = \sqrt{\sum_{j=1}^{n} (v_{ij} - v_j^*)}, \; j=1, 2, \ldots, J \qquad (10)$$

$$s_i^- = \sqrt{\sum_{j=1}^{n} (v_{ij} - v_j^-)}, \; i=1, 2, \ldots\ldots, J \qquad (11)$$

Step 5: The closeness coefficient (Equation 12) is calculated according to the ideal solution of each alternative.

$$CC_i = \frac{d_i^-}{d_i^* + d_i^-}, \; i=1, 2, \ldots, J \qquad (12)$$

Step 6: Alternatives are ranked from the most valuable to the least valuable according to the decreasing values of the closeness coefficient. The alternative with the highest closeness coefficient (CC_i) is selected [58].

Microsoft Office Excel program was used to evaluate with TOPSIS method. The entropy values (w_j) of the criteria weighted by the Entropy Method were used in the implementation of TOPSIS.

Sensitivity Analysis

Sensitivity analysis examines the effect of changing the coefficient values determined in a MCDM on the optimal solution of the problem. The decision maker can make

better decisions if they can determine how critical each criterion is, in other words, how sensitive the current ranking of alternatives is to changes in the weights of the criteria [59,60].

In this study, a sensitivity analysis was conducted to determine the stability of the results. It is done to examine whether changing the weight of a criterion causes a change in the priority order of alternatives. Sensitivity analysis was performed with a decision matrix created with modified importance level values (Table 5).

RESULTS AND DISCUSSION

The sum of the weights of the criteria determined for all three alternatives was taken and the calculation was made by dividing each criterion by this weight sum, and thus a normalized decision matrix was obtained. Then, the entropy values of the criteria were found and the degree of differentiation of the information (d_j) was calculated. Finally, the entropy criterion weights (w_j) were calculated (Table 6).

According to the weighting processes made by using the Entropy Method, it has been seen that the most important evaluation criterion is wastewater generation. Greenhouse gas emissions (C4), toxic reagent use (C1) and toxic gas generation (C2), safety (C9), resource conservation (C10) and recovery rate (C8), recycling cost (C6), overall cost (C7) and energy consumption (C5) followed the wastewater generation criteria, respectively.

The criteria weights obtained by using the Entropy Method were transferred to the BCR model by normalizing for each cluster. Synthesis command in the main menu is used in ANP, and general results are obtained by performing synthesis in the highest-level network. The results obtained from ANP are shown in Table 7.

It is seen that the most useful criterion for the benefit cluster is the "Direct Recycling" Method, which has a very high difference compared to the other two alternatives. Looking at the cost cluster, "Pyrometallurgical Process" is the costlier alternative. In the results of the risk cluster, "Hydrometallurgical Process" is determined as the most risky alternative. According to the ANP synthesis results, the best alternative was "Direct Recycling (A3)".

Table 8 shows the positive ideal and negative ideal separation measures, the relative closeness to the ideal solution, and the ranking results for TOPSIS. According to Table 8, the "Direct Recycling (A3)" alternative is in the first place in order of priority. It is followed by "Pyrometallurgical Process (A1)" and "Hydrometallurgical Process (A2)", respectively.

As mentioned before a sensitivity analysis study was conducted to determine the stability of the results. Sensitivity analyses were performed for six linguistic criteria (1-9 scaling). These criteria are toxic reagent use (C1), toxic gas generation (C2), wastewater generation (C3), recovery rate (C8) safety (C9), and resource conservation (C10). The weight values used for the sensitivity analysis are given in Table 9. From Tables 10 and 11 it can be observed that direct recycling is the most favourable alternative for

Table 5. Decision matrix for sensitivity analysis

	C1	C2	C3	C4	C5	C6	C7	C8	C9	C10
A1	**3**	**6**	**4**	2.21	62.4	2.9	0.5	**2**	**6**	**2**
A2	**5**	**5**	**6**	2.27	59.7	2.2	0.3	**4**	**3**	**6**
A3	**7**	**3**	**3**	0.5	47.6	1.6	0.4	**7**	**8**	**7**

Changed values are shown in bold characters

Table 6. Entropy criterion weights (w_j) values

	C1	C2	C3	C4	C5	C6	C7	C8	C9	C10
wj	0.1539	0.1539	0.1868	0.1606	0.0072	0.0307	0.0227	0.0804	0.1234	0.0804

Table 7. ANP results

	Benefit	Cost	Risk	Overall	Ranking
A1	0.15	0.43	0.34	0.22	2
A2	0.35	0.30	0.40	0.10	3
A3	0.5	0.27	0.26	0.68	1

Table 8. TOPSIS results

	Si+	Si-	Ci+	Ranking
A1	0.1483	0.1511	0.5047	**2**
A2	0.2039	0.0790	0.2792	**3**
A3	0.0503	0.2147	0.8101	**1**

(Si^+ is the distance from the ideal solution, Si^- is the distance from the negative ideal solution and Ci^+ is the relative closeness to the ideal solution.)

Table 9. Entropy criterion weights values for sensitivity analysis

C1	C2	C3	C4	C5	C6	C7	C8	C9	C10
0.0869	0.0596	0.0644	0.2363	0.0105	0.0451	0.0333	0.1816	0.1112	0.1707

Table 10. Sensitivity results of benefit, cost and risk clusters

	Benefit	Cost	Risk	Sensitivity Results	Sensitivity Ranking
A1	0.11	0.43	0.36	0.26	2
A2	0.25	0.31	0.53	0.32	3
A3	0.64	0.26	0.11	0.42	1

Table 11. Sensitivity results for TOPSIS

	Si+	Si-	Ci+	Ranking
A1	0,1854	0,0601	0,2448	**3**
A2	0,1593	0,1129	0,4148	**2**
A3	0,1084	0,1820	0,6267	**1**

the recycling of waste LIBs, even with the change of the weights of the criteria. As a result of the sensitivity analysis, a change in the overall ranking can be observed with the pyrometallurgical process ranked second and the hydrometallurgical process ranked third.

The results obtained for sensitivity analysis from ANP are shown in Table 10. The same rankings were obtained in the sensitivity analysis results as in the ANP results. "Direct Recycling" Method is the most useful criterion for the benefit cluster, "Pyrometallurgical Process" is the costlier alternative and "Hydrometallurgical Process" is determined as the most risky alternative.

When the general results are examined, it is seen that "Direct Recycling" is the best alternative. According to the sensitivity analysis results for TOPSIS, "Direct Recycling (A3)" is again the best alternative for recycling of LIBs (Table 11).

CONCLUSIONS

Most of the research studies on end-of-life LIBs focus on recycling methods that reduce environmental pollution and try to prevent natural resource consumption, thus contributing to environmental sustainability and circular economy. In this study, three different recycling methods were compared via MCDM. Firstly, the criteria were weighted with the Entropy Method, and then alternatives were evaluated with two different methods, ANP and TOPSIS. According to the weighting processes made by using the Entropy Method, it has been seen that the most important evaluation criteria are wastewater generation and greenhouse gas emissions, respectively. These criteria are followed by the use of toxic reagents with the same weight and the formation of toxic gas. According to the ANP overall results, it was seen that the best alternative was Direct Recycling. Direct Recycling Method is followed by Pyrometallurgical Process and Hydrometallurgical Process, respectively. In TOPSIS, positive ideal and negative ideal separation measures, relative closeness to the ideal solution and ranking results were obtained. In order of priority, the Direct Recycling alternative is in the first place followed by Pyrometallurgical Process and Hydrometallurgical Process, respectively. This is because the Direct Recycling alternative has more advantages in environmental management

for end-of-life LIBs than other alternatives. As a result of the sensitivity analysis, Direct Recycling was found to be the best alternative for the recycling of LIBs in both ANP and TOPSIS methods. Direct recycling, designed to recover cathode material with morphological integrity. By reducing the number of processing steps required to re-synthesize cathode materials, this process has a comparatively low environmental impact. A main drawback of direct recycling is that the process is dependent on the input of specific cathode types for the recovery of high value materials. So, it is highly dependent on an efficient classification of battery types based on easy to understand labelling according to cell chemistry (Recycling of Lithium-Ion Batteries—Current State of the Art, Circular Economy, and Next Generation Recycling). For future studies, it is recommended that studies with criteria different from those used in this paper can be conducted in the future using different MCDM methods, such as Elimination and Choice Translating Reality (ELECTRE) and Preference Ranking Organisation Method for Enrichment Evaluations (PROMETHEE), among others. In addition, it is possible to develop the approach here with other methods such as Life Cycle Assessment and Life Cycle Cost Analysis. In this way, more accurate and more detailed information can be obtained to compensate for the limitations of this study.

CONFLICT OF INTEREST

The authors declare no potential conflicts of interest regarding the research, authorship and/or publication of this article.

DATA AVAILABILITY

The data used to support the findings of this study are included within the article.

AUTHOR'S CONTRIBUTIONS

All authors are contributed equally to bring out this article.

ETHICS

There are no ethical issues with the publication of this manuscript.

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Research Article

Mixing effect on bio-methanation, settleability and dewaterability in the anaerobic digestion of sewage sludge fractions

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ABSTRACT

Biomethanation and dewaterability characteristics of primary sludge (PS), secondary sludge (SS) and mixed sludge (MS) fractions were assessed after anaerobic stabilization under parallel batch and continuous mixing conditions at 35°C in order to investigate the performance of the separate digestion system. Similar methane conversion/yield values were obtained in PS, SS and MS digestion with continuous mixing's positive effect only in PS digestion. Continuous mixing resulted in 50% increase in the methane yield (600(+/-100) mL/g VS_{add}.d. SS digestion produced a comparable methane yield at 650(+/-100) mL/g VS_{add}.d showing no effect due to mixing pattern. Settling and dewaterability characteristics of the stabilized PS were superior to stabilized SS samples. A reverse relationship was obtained between settling and dewaterability characteristics where intermittent mixing enhanced settling ability while continuous mixing resulted in higher dewaterability of the stabilized sludges. Polyelectrolyte (PE) addition showed a negative effect on the settleability of the sludges. Low degree mixing resulted in 50% sludge volume reduction and an SVI of 64 mL/g VS compared to 25% volume reduction and 82 mL/g SVI in the continuous mixing mode for the stabilized PS. A similar trend for the stabilized SS but weaker values with 25% volume reduction and an SVI 182 mL/g in the intermittent mixing mode compared to 15% volume reduction and 200 mL/g VS SVI indicated a much lower settleability in the continuous mixing mode and compared to stabilized PS.

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INTRODUCTION

Sewage sludge is produced as primary sludge (PS) and secondary sludge (SS) fractions in high volumes and mixed to be thickened (as mixed sludge (MS)) and then digested in completely mixed stirred anaerobic reactors applying high retention time as the most common strategy in the municipal wastewater treatment plants (WWTPs) despite they possess different biodegradability and dewaterability characteristics [1]. The biogas produced as a result of partial reduction in the volatile solid (VS) content is used in the electricity generation and contribute to the recovery of the operational costs. Many upgrading approaches deal

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with the minimization of the sludge volume, augmentation of the biological stabilization and dewaterability degree [2].

Dewaterability of sewage sludge affects the daily chemical dosage and produces a major cost in the sludge line of the municipal WWTPs. Dewaterability of sludges depends on many factors as particle size distribution, bound water, protein, fat and carbohydrate content whereas for SS, produced as the waste biological sludge, floc-structure and extra polymeric substances (EPS) play a major role in the water release in the decanters [3].

In biological treatment systems, it is important to mix the reactor to ensure efficient contact of the substrate with biomass. The mixing process is an important parameter that affects the efficiency and biochemical reaction times of the reactor. There are many advantages for anaerobic digesters in the mixing process; the homogeneity of organic/volatile matter and biomass, prevention of dead spaces, efficient contact of microorganisms with the substrate. In particular the rate of anaerobic hydrolysis increases, the methane gas produced by methanogens strips out more easily, lower retention time is needed compared to non-stirred reactors. Efficiency of the biogas production can be increased by 50% due to dilution+homogenization effect on the inhibitory products enhancing microbial reactions [4]. Contrarily, batch/intermittent mixing can improve the syntrophic relationship between methanogens and acetogens in the biomass and improve degradation in the case of fat-oil-grease (FOG) matter through lessening the degree of inhibition by long chain fatty acids [5]. As a result, methanogenic activity can proceed at a higher level in the batch than continuous mixture. Dewaterability of the final stabilized sludge is another important parameter determining the chemical cost spent in the decanters towards the final disposal of the sludge enabling reduction in the sludge volume and increase to a desired solid content. Both digestability and dewaterability of the sewage sludge fractions were seldom investigated comparatively for PS, SS and MS to optimize the sludge line in the municipal WWTPs.

In this study raw PS and SS and MS samples were subjected to anaerobic digestion and the final stabilized sludge fractions were comparatively evaluated based on dewaterability (chemical costs), conductivity (potential for land use) and volatile solid content (carbon content) as well as digestion performance in terms of methane yield. The outcome is aimed to indicate the mixing pattern's impact and different biodegradability and final biosolid quality that affect directly the operational costs and the advantage and/or disadvantage of separate sewage sludge digestion system.

METHODOLOGY

The experimental study was conducted in semi-continuous lab-scale reactors with 2000 mL total volume in parallel as continuous mixing (CM) and intermittent mixing (IM) modes for PS, SS and MS digestion at 35°C (Figure 1).

Figure 1. Lab-scale anaerobic digesters (a) IM and (b) CM modes.

PS samples were collected at the outlet of the primary settlers in the Konya municipal WWTP. SS was collected from the sludge return line in the activated sludge unit. Both PS and SS were thickened to increase the solid contents. The mixed sludge (MS) was prepared using 54:46% (v:v) of PS and SS samples, respectively. Inoculum sludge was grown in a previous study of sewage sludge digestion. The total solid (TS) and volatile solid (VS) concentrations ranged at 16380-23350 mg TS/L and 10690-15080 mg VS/L (PS) and 6430-7050 mg TS/L and 5250-5710 mg VS/L (SS). Organic loading rates (OLR) were applied as 0.53, 0.83 (PS), 0.29, 0.42 (SS) and 0.57 (MS) g VS/L.d for the digesters.

Hydraulic retention time (HRT) was kept at 20 d for all the reactors. Digesters were fed one time daily. Anaerobic digestion performance was monitored daily whereas effluent sludge settling and dewaterability were determined after the steady state was reached. Continuous mixing was provided in the PS-K, SS-K and MS-K digesters placed in the water bath by magnetic stirrers at 150 rpm placed under the water bath whereas parallel digesters, PS-A, SS-A and MS-A, kept in the incubator were subjected to intermittent mixing during the day time (Figure 1a, b). Anaerobic digestion performance was monitored by methane production via liquid displacement method and stabilized biosolid quality was determined as volatile solids (VS-2540 E), conductivity (WTW İnolob Cond 7110), Imhoff settleability and sludge volume index as mL/g VS (SVI-2710 D) and

dewaterability as capillary suction time (CST) (2710 G) and time-to-filter (TTF)(2710 H) according to standard methods [6]. 304M Venture Innovations, Inc was used for CST determination. A vacuum filtration unit was used for TTF determination. Both measurements were obtained as seconds. 200 mL of sludge was used for Imhoff settling and SVI calculation. Methane yield as specific methane production was calculated as dividing the daily methane production by the VS amount of raw sludge fed in gram. A cationic polyelectrolyte (cationic acrylic acid derivative polyacrylamide) (PE) was used as aid for settling and added at 0.5, 1, 1.5, 2 and 2.5 g/200 mL sludge dosages making a concentration range as 2.5, 5, 7.5, 10 and 12.5 g/L of sludge.

RESULTS

The anaerobic digestion of PS produced the lowest methane yield at 400(+/-200) mL/g VS_{add}.d in the intermittent mixing (IM) mode (Figure 2a). Continuous mixing (CM) resulted in 50% increased methane yield (600(+/-100) mL/g VS_{add}.d). Higher degree of mixing was more efficient indicating that hydrolysis was the rate limiting stage and was enhanced for the slowly-degradable matter in the PS. SS digestion performance was low in daily methane production but produced a comparable methane yield level at 650(+/-100) mL/g VS_{add}.d due to its low VS feeding and showed no effect due to the mixing pattern indicating that biodegradability is limited and the hydrolysis stage can not be enhanced by adjusting the mixing degree (Figure 2b). Low VS concentration is considered a disadvantage for SS as low OLRs are applicable that limit the methane produced [7]. No effect of mixing degree was observed on methane yield in MS digestion at similar levels (650 (+/-50) mL/g VS_{add}.d) evidencing the dominant effect of SS in the mix (Figure 2c).

Higher methane yield values were obtained compared to previous studies of PS and SS digestion [8-10]. Winter and Pierce obtained a steady but low biogas yield at 250(+/-20) mL/g VS_{add}.d in SS digestion with a 65-70% methane content whereas higher but fluctuating value between 200-650 mL/g VS_{add}.d in the PS digestion with a 65% methane content [8]. The OLRs were higher as 1.48 and 1.67 kg VS/m^3.d for PS and SS digestion, respectively. The higher performance was correlated to lower OLR applied in this study. Methane yield decreases in cases of over exceeding on the hydrolysis rate by the OLR applied. Braguglia et al. obtained a methane yield of 170 mL/g VS_{add}.d in the SS digestion and applied pre-treatment methods to increase the yield [9]. Pinto et al. investigated an optimum proportion of PS and SS in the MS at an OLR of 1.62 kg VS/m^3.d and obtained a maximum methane yield of 372 mL/g VS_{add}.d [10]. The findings indicated that the content of the PS was the major enhancing factor on the methane yield of anaerobic sludge digestion and a variable for all municipal wastewaters.

Volatile solid (VS) concentration of the stabilized PS dropped and stabilized at 8000(+/-1000) mg/L in the end

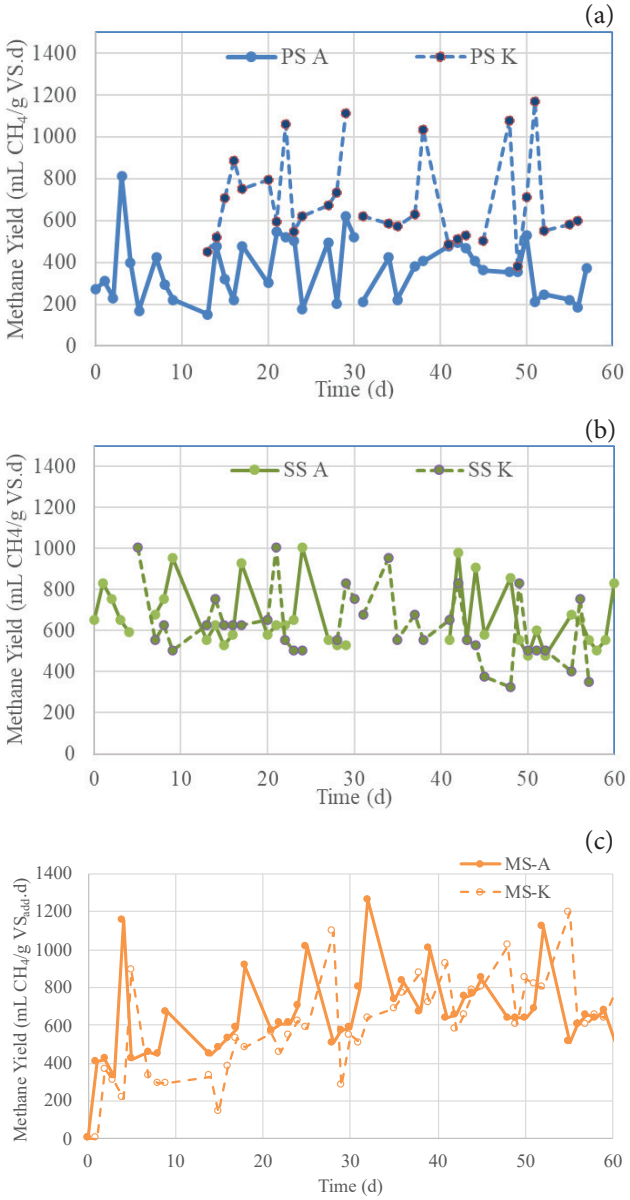

Figure 2. Methane yield values for (a) PS, (b) SS and (c) MS digesters.

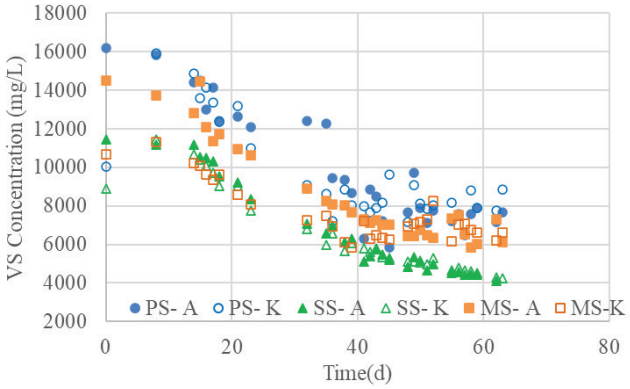

Figure 3. VS Concentration of the stabilized sludge types.

of the first 30 d-period showing no effect of mixing mode (Figure 3). Higher biomass content supported the high biodegradability of raw PS in comparison to SS digestion where VS concentration continuously dropped down to a lowest level of 4200 mg/L which supported low biodegradability and/or nutrient availability. Resistance of the SS' content to the reducing hydrolytic environment of anaerobic digestion originates from its content as bacterial cells and microbial products and has been reported in many studies [8,11]. Stabilized MS had a slightly lower VS content compared to stabilized PS. No effect of mixing degree was observed on the VS concentration for all sludge types. As a digestion performance VS removal was obtained at 39-56% (PS-A), 33-52% (PS-K), 20-27% (SS-A), 18-25% (SS-K), 27-29% (MS-A) and 23-28% (MS-K) indicated a slightly higher removal degree in the IM mode. In concordance with the methane yield data it can be concluded that biomass inhibition or loss (mostly methanogens) may have taken place in PS digestion in the low degree mixing resulting lower VS concentration (higher VS removal).

As the final biosolid quality, the conductivity of the stabilized PS showed the highest level (5.2-5.2 mS/cm) compared to stabilized SS at 4.2 and 4.4 mS/cm and MS at 4.7 and 5.0 mS/cm in the CM and IM conditions, respectively (Figure 4). The low level for SS indicated a suitability for limited agricultural use. Mixing degree affected the ion concentration of all the stabilized sludges meaning that higher microbial synthesis was promoted in the CM mode compared to low degree mixing.

Mixing degree of the stabilized sludges (in the absence of PE) was significantly effective on the settling property as low degree mixing resulted in lower Imhoff settling volume with 50% sludge volume reduction and an SVI of 64 mL/g VS compared to 25% volume reduction and 82 mL/g SVI in the CM mode for the stabilized PS (Figure 5a). A similar trend for the stabilized SS but weaker values with 25% volume reduction and an SVI 182 mL/g compared to 15% volume reduction and 200 mL/g VS indicated much lower settleability character in the CM mode and compared to stabilized PS (Figure 5b). Stabilized MS exhibited good

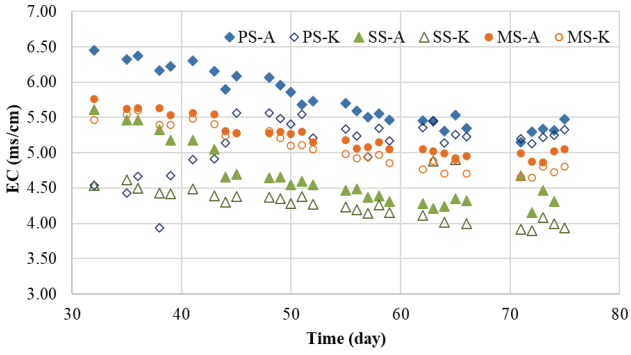

Figure 4. Electrical Conductivity (EC) of the stabilized sludges.

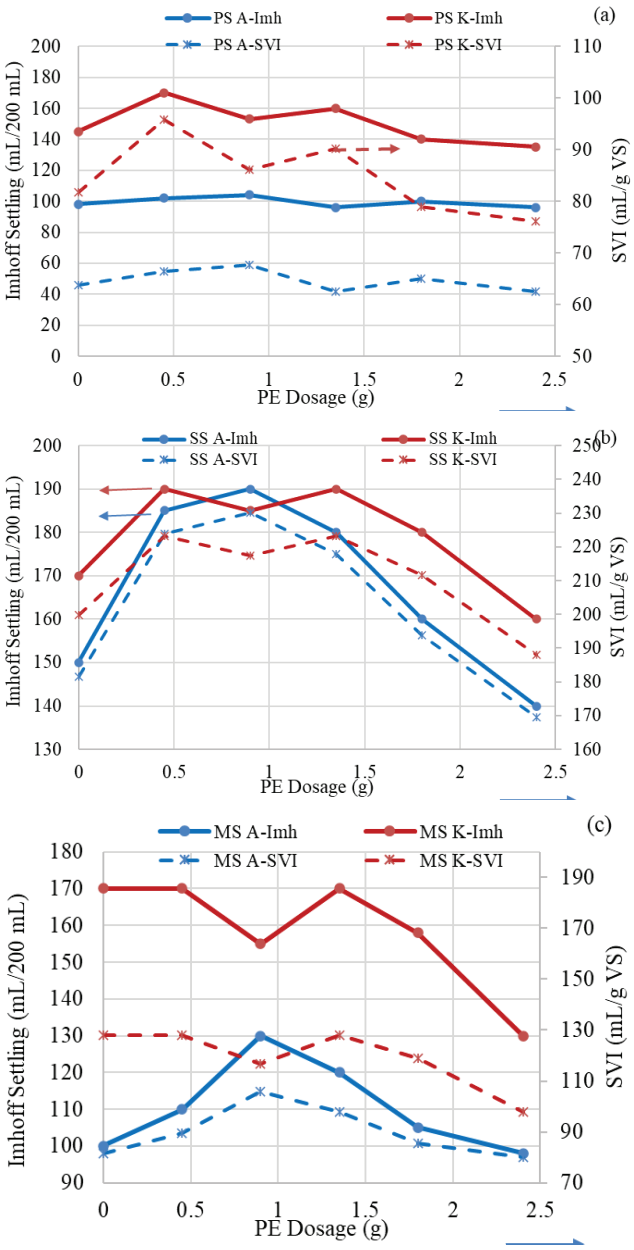

Figure 5. Imhoff settling and SVI values for stabilized (a) PS, (b) SS and (c)MS biosolids.

settleability and SVI approaching stabilized PS characteristics in the IM mode whereas bad settleability and SVI approached stabilized SS characteristics in the CM mode (Figure 5c). PE dosing exhibited no effect on the settleability and SVI for stabilized PS. A worsening effect was noticeable at all dosages with the exception of the highest dosage for stabilized SS regardless of the mixing degree. The worsening pattern was observed with the stabilized MS in the IM mode (no effect with the maximum dosage) whereas the maximum dosage enhanced settleability and SVI by 20% and 26%, respectively.

The mixing degree exhibited an opposite pattern in the dewaterability of the stabilized PS that occurred at different levels as CST:77-84 s and TTF: 560-618 s in the IM mode and much higher water release abilities with CST: 15-16 s and TTF: 195-205 s in the CM mode. Similarly for stabilized SS samples, low mixing degree produced lower dewaterability with higher CST (93-100 s) and TTF (1685-1702 s) values than CM mode with CST:71-74 s and TTF: 890-1160 s values. CM promoted dewaterability for stabilized SS and PS. Similarly, stabilized MS produced much lower CST (19 s) and TTF (281-238 s) values in the CM than CST: 74-80 s and TTF: 760-887 s in the IM mode. The results implied that compacted flocs were formed with higher settleability but poorer water release ability in the low mixing operation. High water release was an indicator of the high free water content and loose flocs that occurred in the CM operation which was shown with poorer Imhoff settling results.

Dewaterability at a higher degree for stabilized PS was shown previously in similar comparative studies with stabilized SS at higher OLRs [12,13]. Lower settling and dewaterability characteristics of the SS were correlated to a higher concentration of colloidal solids inducing a higher zeta-potantial and high content as EPS matter that holds bound water [14-17]. Reverse effect of mixing degree on the settling and dewatering abilities indicated that free water content was higher in the case of IM and was separated off the flocs more efficiently by simple settling whereas lower dewaterability was caused by the high number of bacterial cells and lower amount of products [18,19]. On the other hand, CM worsened settling but enhanced water release by the lower number of cells and higher number of cell products due to harder environmental conditions where bacteria increased the EPS production. Similarly, the negative effect of PE addition was explained as the increase of bounding energy of the sludge that already contained polymeric substances and bound water resulting in floc expansion [19].

CONCLUSIONS

A parallel relation was obtained between mixing density and methanogenic activity+dewaterability with a negative impact on the settleability of the stabilized sludges in PS, SS and MS digestion. Settling and dewaterability characteristics of the stabilized PS were superior and required no PE addition compared to stabilized SS and MS samples. A reverse relationship was obtained between settling characteristics and dewaterability for all sludge types indicating denser floc formation with high settling but poor water release abilities in a low degree of mixing. No effect of solid content was calculated on the CST and TTF results in order to indicate the highest settling and water release abilities of stabilized PS despite the highest VS content requiring no need for PE addition which also did not enhance settleability at a significant level up to 12.5 g/L sludge for stabilized SS and MS.

The outcome pointed out to the benefits of the separate/parallel digestion of PS and SS when compared with MS digestion where:

- a substantial chemical and energy cost can be eliminated through mechanical settling allowing 50% volume reduction and thickening of the stabilized PS,
- a substantial savings in the chemical cost using PE in the decanters after the PS digestion,
- a substantial saving in the chemical cost with a partial intrusion of the PS into SS flow to ameliorate SS's settling ability and dewaterability significantly.

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CONFLICT OF INTEREST

The authors declare no potential conflicts of interest regarding the research, authorship and/or publication of this article.

DATA AVAILABILITY

The data used to support the findings of this study are included within the article.

AUTHOR'S CONTRIBUTIONS

All authors are contributed equally to bring out this article.

ETHICS

There are no ethical issues with the publication of this manuscript.

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Case Report

Temporal evaluation of the Beni Haroun dam's (Algeria) raw water quality, through a literature review

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ABSTRACT

Several authors have carried out physic-chemical analyses of the raw water from the Beni Haroun dam since it was exploited, and others continue to do so. The aim of this work is to present a temporal synthesis (2003-2018) of the variation in their quality. It depends on several parameters, such as climate, socio-economic development and population growth around its tributaries. The parameters studied are : Temperature, pH, EC, TDS, and in (mg/l) Ca^{2+}, Mg^{2+}, Na^{+}, K^{+}, Cl^{-}, SO_4^{2-}, HCO_3^{-}, NO_3^{-}, NO_2^{-}, NH_4^{+} and PO_4^{3-}, as well as organic parameters such as: COD, BOD_5 , dissolved O_2 and certain heavy metals (µg/l): Pb, Cd, Zn, Fe, Cr, Cu and Mn. The results of this collection of publications show that the quality of the raw water from the Beni Haroun dam varies over time, depending on the season and mainly on the water supply resulting from rainfall, which leads to dilution of the various chemical elements. The air temperature modifies the water temperature, which affects the organic composition. The conclusion shared by all the authors is that raw water must undergo prior treatment before being used for drinking or irrigation.

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INTRODUCTION

In Algeria, the growing need for fresh water is outstripping available resources, as a direct consequence of socio-economic and demographic development. The mobilization of surface water in dams therefore remains one of the possible solutions for managers, depending on the prevailing hydroclimatic conditions [1]. However, these surface waters are vulnerable to various pollutions and are often of poor quality. They may contain significant amounts of natural organic matter such as humic substances but also organic compounds from various pollutant discharges or intensive agricultural practices [2]. The importance of the studies of the quality of the water of the dams, lies in the fact of their use in the food of the populations, the animals and in irrigation [1].

The Beni Haroun dam, located on the Wadi El Kebir watershed (WS), is a very important structure in the region of Mila, and in the entire eastern region of Algeria, by the volume of water it is able to store (about one billion m^3), and also by its location which allows it to ensure the need for Drinking Water Supply (DWS) and Irrigation Water (IW) of six neighboring wilayas [3, 4].

Several studies, in the different frameworks (doctorate, magister, master, and license), were interested in the waters dams physic-chemical quality evaluation in Algeria; others

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took particularly as objective to follow and analyze the raw waters quality and the organic parameters of the Beni Haroun dam ; and some used the remote sensing and GIS methods to map this quality [4]. To achieve their objectives, these works have either collected data from the ANRH (National Agency for Hydrous Resources) or the ANBT (National Agency for dams and Transfer), or carried out the sampling and chemical analyses themselves.

MATERIALS AND METHODS

DESCRIPTION OF THE STUDY FRAMEWORK

The Beni Haroun dam is located in the Wilaya of Mila, on the Wadi El Kebir, in the Kebir Rhumel watershed (East-Algeria) (Figure 1), it belongs to the sub-humid domain, with an annual precipitation of about 654.80 mm and an interannual mean temperature of 16.43°C, for the series 2003-2013 [5]. Wadi Rhumel feeds it from the West and Wadi Endja from the East.

The catchment area of Kebir Rhumel, with an area of 6595 km^2.

It is naturally bordered by the Constantinois Ouest and Constantinois Centre catchment areas (WS n° 03) to the north, the Hauts Plateaux du Constantinois catchment area (WS n° 07) to the south, the Soummam catchment area (WS n° 15) to the west and the Seybouse Wadi catchment area (WS n° 14) to the east [3, 4, 6].

The Beni Haroun dam is of the rectilinear weight type, in BCR (Roller compacted concrete), with a length of 710 m at the crest, levelled at the 216.3 m coast, a height of 118 m above the foundation. The lake of the reservoir hugs the captured part of Wadi El Kebir and the two valleys of Wadi Rhumel and Wadi Endja, on a surface of 39.29 Km^2, that is to say nearly 4,000 ha. The rainfall-reservoir balance makes it possible to determine a net destocking by evaporation, equivalent to an average annual tranche of 350mm. The reservoir allows to store 963 Hm^3 of water, that is to say a

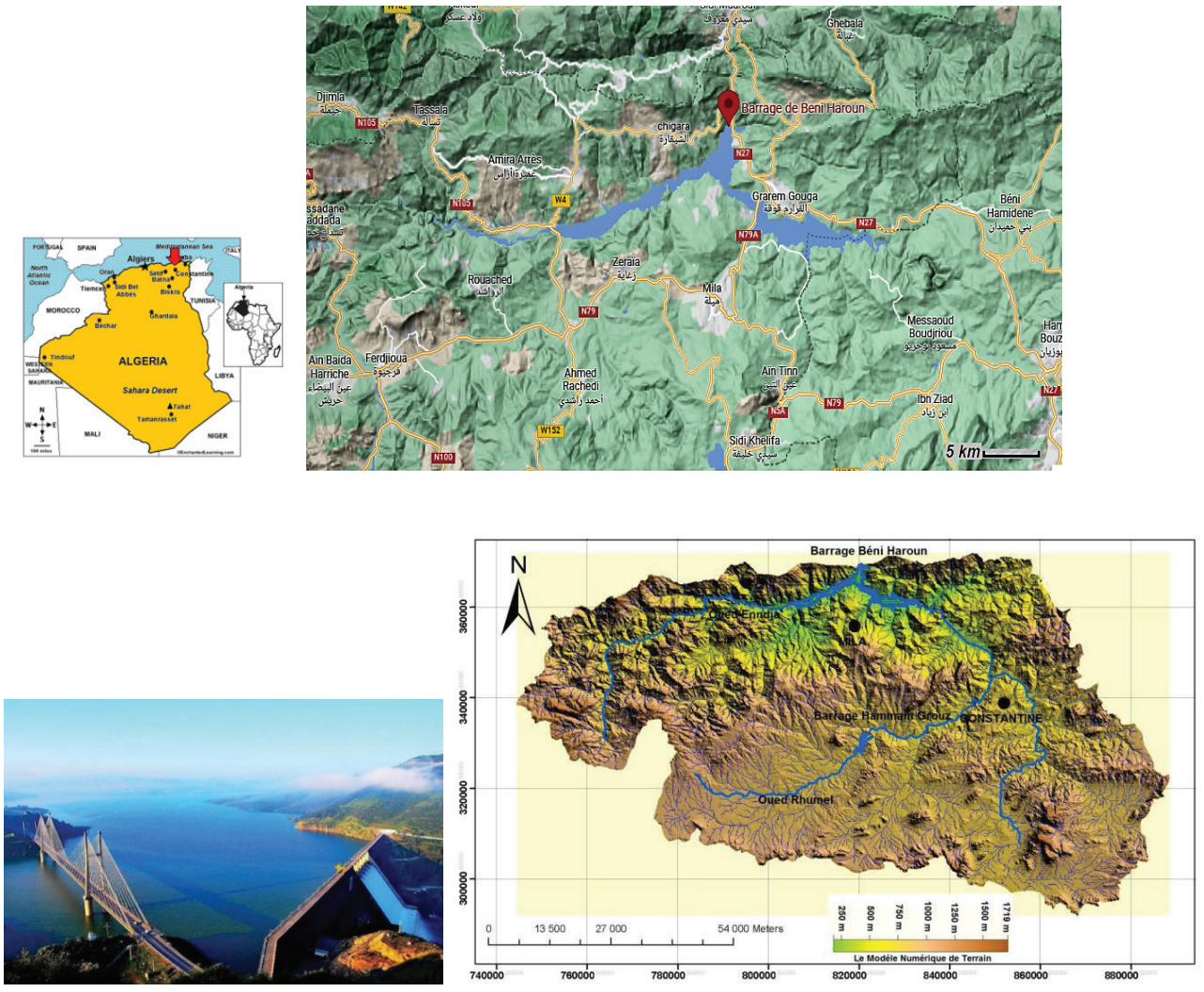

Figure 1. Geographical location (Google map 2023), and view of the Beni Haroun Dam (reservoir).

useful volume of 732 Hm^3, it allows to regulate an annual contribution of 435 Hm^3, with a reserve of 1 billion m^3 of water reached on February 12th, 2012; it is put in exploitation in 2003. It provides a drinking water supply to 4 million inhabitants, spread over six Wilayas, which are: Mila, Jijel, Constantine, Oum El Bouaghi, Batna, Khenchela, and four irrigated areas with a total area of about 40,000 ha. It is manage by the ANBT [1, 3, 4, 6, 7].

METHODS AND DATA USED

To carry out this study of the quality of the raw water of the Beni Haroun dam, through time, we have collected studies of doctorate, master, magister and licenses, which have treated the subject, over a period of 16 years, from 2003 to 2018. Each one has its own objective, there are those who study the evolution of the temporal variation of the quality of the raw water of the dam [8-22]. There are those who make the assessment in order to search for indicators of pollution in the water, in the fauna and flora that form the ecosystems populating the site [6, 23] by the determination of heavy metals and the analysis of sediments of the dam [24-27] and there are those who apply statistical methods [15, 22] or even mapping and GIS methods [4] to show the spatial variability of certain physical elements. There are also studies of the quality of Wadi El Kebir before the realization of the dam of Beni Haroun from 1983 to 2003 (Figure 2) [6, 16].

The physic-chemical elements followed in this work are: Temperature, pH, electrical conductivity (EC), dry residue (DR), suspended matter (SM), Ca^{2+}, Mg^{2+}, Na^+, K^+, Cl^-, HCO_3, SO_4^{2-}, NO_3^-, NO_2^-, NH_4^+ and PO_4^{3-} in (mg/l), as well as organic parameters such as: COD, BOD_5, dissolved O_2 and some heavy metals (µg/l): Pb, Cd, Zn, Fe, Cr, Cu and Mn.

RESULTS AND DISCUSSIONS

The monitoring of dam water quality is very important, in order to protect against anthropic pollution, which can reach the ecosystems present in these waters, that is why the National Agency of Hydraulic Resources (ANRH) has installed a network of control and physico-chemical measurements of waters along all the wadis feeding the dams on the whole Algerian territory; as well as the establishment of a scale of classification going from good to very bad, based on the values of the various physico-chemical parameters present in this water (Table 1). On the Kebir Rhumel watershed, there are four control stations; we have the station 100315 at Hammam Grouz Dam on Wadi El Athmania. The three stations: 100403 at Ain Smara, 100624 at El Menia and station 100601 at Grarem Gouga upstream of the Beni Haroun Dam, are on the Rhumel Wadi.

The ABH (River Basin Agency) and the ANRH (National Water Resources Agency) established in 2009 a classification grid for surface water quality (Table 1) [2, 6], it is based on the values of different chemical elements present in these

Table 1. Water quality grid for dams adopted by the ABH and ANRH in 2009 [2].

	Unit	Class 1 Good	Class 2 Average	Class 3 Mediocre	Class 4 Very bad
pH		6.5 - 8.5	6.5-8.5	8.5-9	>9 and <65
(Dry Residue) DR	mg	300-1000	1000- 1200	1200-1600	>1600
TDS	mg/l	0-30	30-75	75-100	>100
Ca^{2+}	mg/l	40-100	100-200	200-300	>300
Mg^{2+}	mg/l	<30	30-100	100 -150	>150
Na^+	mg/l	10-100	100-200	200-500	>500
Cl^-	mg/l	10- 150	150-300	300-500	>500
SO_4^{2-}	mg/l	50-200	200-300	300-400	>400
Dissolved oxygen (DO)	%	90-100	50-90	30-50	< 30
BOD_5	mg/l O_2	<5	5-10	10-15	>15
COD	mg/l O_2	<20	20 -40	40-50	>50
OM (organic matter)	mg/l	<5	5-10	10-15	>15
PO_4^{3-}	mg/l	≤ 0.01	0.01 -0.1	0.1-3	>3
NH_4^+	mg/l	≤ 0.01	0.01 -0.1	0.1-3	>3
NO_2^-	mg/l	≤ 0.01	0.01 -0.1	0.1-3	>3
NO_3^-	mg/l	≤ 10	10-20	20-40	>40
Fe^{2+}	mg/l	0-0.5	0.5-1	1-2	>2
Mn^{2+}	mg/l	0-0.1	0.1 -0.3	0.3-1	>1

Table 2. Comparison with the descriptive statistics of the raw water of Beni Haroun Dam (2003-2018)

	Mean	Min	Max	Class
pH	7,78	7,27	8,45	**1-2**
TDS	96	25	27,3	**1**
Ca^{2+}	113,69	65,61	195,74	**1-2**
Mg^{2+}	68,99	44,18	145,61	**2-3**
Na^{+}	93,56	30,61	123,41	**1-2**
Cl^{-}	177,63	30,88	334,57	**1-2-3**
SO_4^{2-}	232,86	162,50	347,33	**1-2-3**
BOD_5	6,36	3,00	9,70	**1-2**
COD	64,34	44,50	97,65	**3**
PO_4^{3-}	1,04	0,05	2,64	**2-3**
NH_4^{+}	1,69	0,13	4,72	**3-4**
NO_2^{-}	1,38	0,17	2,64	**3**
NO_3^{-}	11,35	0,55	24,37	**1-2-3**
Mn^{2+}	0,17	0,14	0,22	**2**

waters. We compared the results of statistics (Min, Max and Average) of raw water of Beni Haroun dam, for the period of 2003-2018, with those of the grid. We notice that they present a good to average quality for the following elements: pH, TDS, Ca^{2+}, Mn and Na^{+}, mediocre for Cl^{-}, SO_4^{2-}, PO_4^{3-}, NO_3^{-} and NO_2^{-}, poor for NO_2^{-}, COD and very poor for NH_4, which exceeds 3 mg/l . In general, it can be considered to be of average quality.

The establishment of graphs, on a logarithmic scale, of raw water from the site of the Beni Haroun dam on Wadi El Kebir, before its construction, over the period 1984 - 2002 (Figure 2.a and Table 3), where we have collected the descriptive statistics (Min, Max and Avrg), allows us to note that these waters have a poor quality to very poor, while on Figure 2.b, all the elements show some stability through time, except for a few particularities:

- NH_4^{-}, shows a decreasing peak from 1986 (2.25 mg/l) to 1987 (0.1 mg/l), followed by an increase in 1988 (1.55 mg/l).
- A decrease in TDS, from 1.47 mg/l (1984) to 0.0245 (1990).
- NO_2^{-}, shows a double increasing slope from 0.16 mg/l (1996) to 2.15 mg/l (2000), and from 0.3mg/l (1998) to 1.87 mg/l (2002).
- The rest of the elements have kept a certain temporal stability in their concentrations.

Figure 3 shows the descriptive statistics of the chemical elements of the raw water of Beni Haroun Dam between 2003 and 2018, or we notice that:

- NO_3^{-}, NO_2^{-}, NH_4^{+}, PO_4^{3-}, BOD_5 and O_2 have low values compared to the other elements.
- Cl^{-}, SO_4^{2-} and HCO_3^{-} have high concentration values.
- Ca^{2+}, Mg and Na have relatively average values.

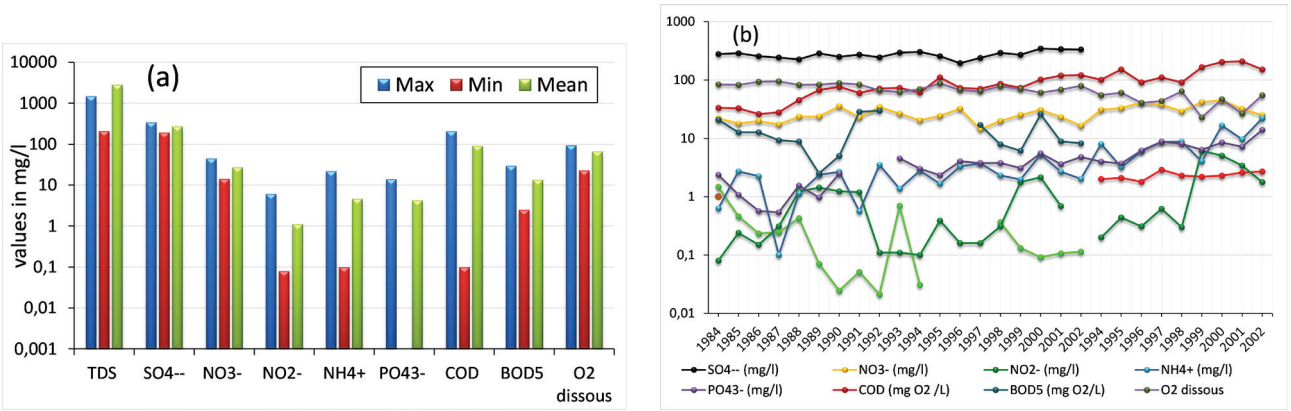

Figure 2. a and b, Water quality of Wadi el Kebir before the construction of the Beni Haroun Dam, before 2003.

Table 3. Descriptive statistics of raw water from Beni Haroun Dam (1984-2002)

	Turbidity NTU	EC (mS/cm)	TDS (mg/l)	SO_4^{2-} (mg/l)	NO_3- (mg/l)	NO_2- (mg/l)	NH_4+ (mg/l)
Max	92,00	2,90	1475	346,10	45,00	6,09	22,00
Min	4,39	1,80	212	194,29	14,33	0,08	0,10
Mean	20,26	2,32	2844	274,44	27,39	1,11	4,64

	PO_4^{3-} (mg/l)	COD (mg O_2 /L)	BOD_5 (mg O_2 /L)	O_2 dissolved (mg O_2 /L)
Max	14,00	209,00	29,83	95,29
Min	0,001	0,10	0,001	22,89
Mean	4,25	91,05	12,75	67,24

- Pb and Fe stand out, with maximum values exceeding 150 mg/l.
- Mn is always low with a maximum of 0.22mg/l.
- Cd, Zn, Cr and Cu, their maximums do not exceed 44 mg/l.

Figure 4a shows the evolution over time, from 2003 to 2018, of the quality of raw water of Beni Haroun dam, heavy metals, or we notice that:

- Almost all the elements have a certain stability of concentration, with the exception of SO_4^{2-} and Cl^-.
- Fe decreases, sharply, between 2003 and 2004, and continues its descent until 2017, then it increases in 2018 (97 µg/l).
- The element Pb increases in 2011 (186.72 µg/l), decreases in 2014 (83.82 µg/l) and returns to a stability around 100 µg/l, from 2015 to 2018.

In **Figure 4b**, we observe the temporal evaluation of major and minor elements, where we notice that:

- Almost all the elements show some stability over time, except for the SO_4^{2-}.
- SO_4^{2-} begins a descent from 2004 (347.33 mg/l) to 2018 (226.26 mg/l).
- In 2013, all elements show a remarkable decrease, except for HCO_3 which increases with a value of about 65 mg/l.
- The element Cl^- increases from 30.88 mg/l (2013) to 334.57mg/l (2016) and declines, relatively, in 2017 (198.50 mg/l).

A ***PCA (Principal Component Analysis)*** is establish for the variables (chemical elements) and the elements (years of realization of the analyses), in order to see the different existing correlations, or we observe:

- The formation of three groups on the two representations in Figure 5 a and b.
- K^+ and HCO_3 form the first group, for the variable and 2013, for the elements, indicating the increase in their concentration in that year.
- Organic nitrogen compounds (pollution indicators) and elements of anthropic origin, which increased in 2003, mainly form the second group.

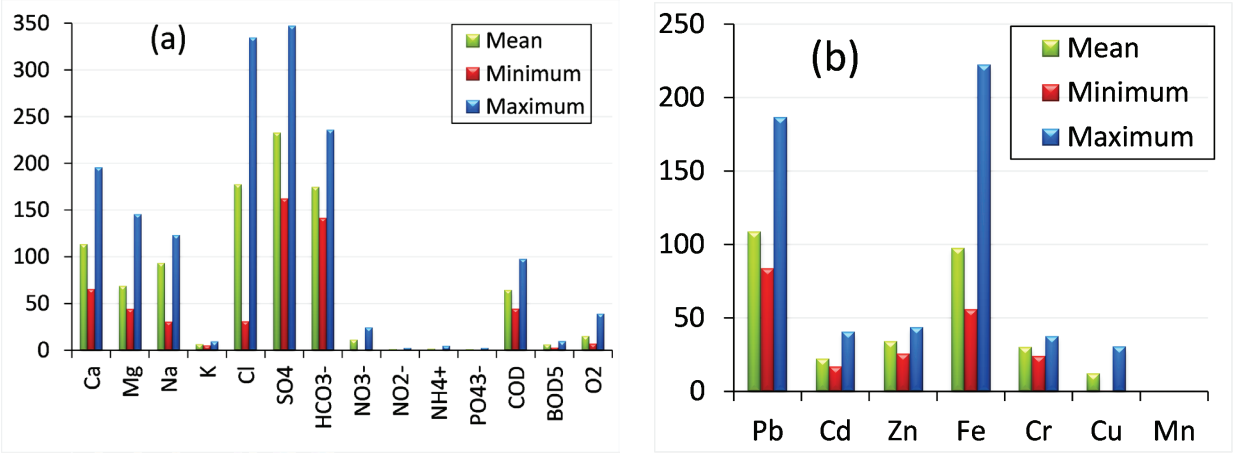

Figure 3. Descriptive statistics of chemical elements in the raw water of Beni Haroun Dam (1984-2002).

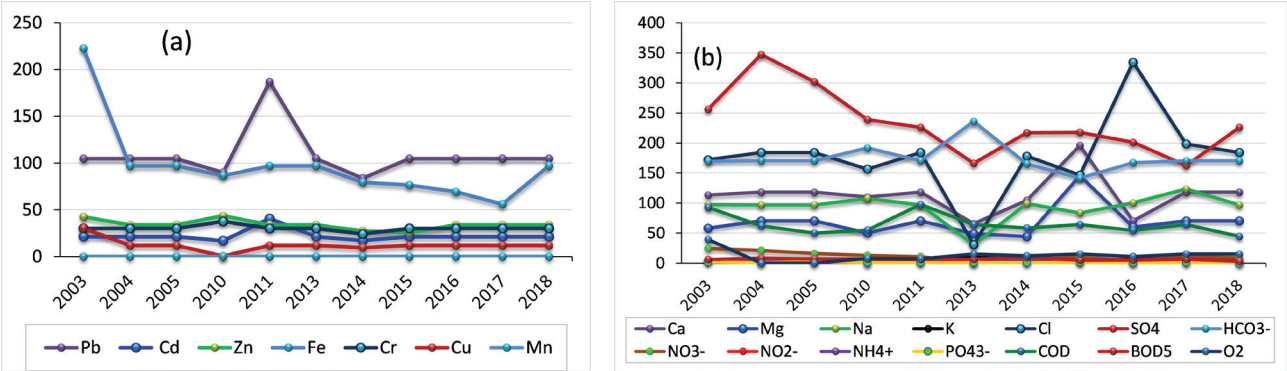

Figure 4. Water quality of Beni Haroun Dam, average, min and max from 2003 to 2018.

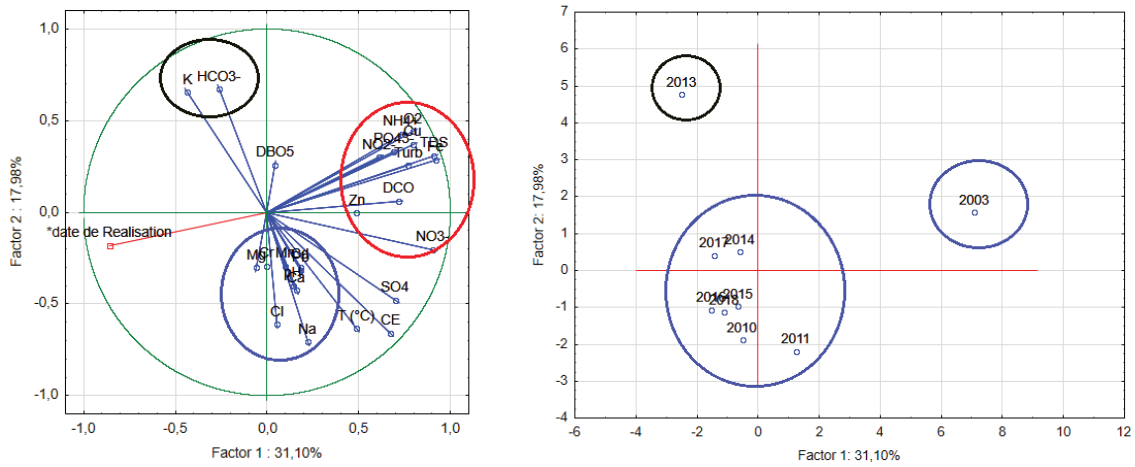

Figure 5. PCA plot (a: variables, b: elements) of raw water from Beni Haroun Dam (2003-2018).

- The third group is composed of elements of geological origin, and which keep their concentrations relatively stable during the rest of the years, represented in the center of the graph of the elements.
- The variable 'date of realization' does not influence the quality nor the quantity of chemical elements.

DISCUSSIONS

For this section, we have collected the main interpretations issued, according to the goals pursued, which are listed and attributed to their authors; we start with the work done before the construction of the dam: Kerdoud (2006) and Maarouf (2012). The others carried out after the impoundment of the dam, in 2003, are present in chronological order of their year of completion.

The analysis and monitoring of the pollution parameters of the Kebir Rhumel basin for a period of twenty years (1984 - 2005), allows us to advance that some analysis parameters are related to the natural structure of water such as pH, conductivity, turbidity, chloride ions, sulfate ions, etc.. In addition, others concerning undesirable substances such as the content of nitrates (NO_3^-), phosphates (PO_4^{3-}), which are the main factors responsible for the proliferation of algae and the acceleration of the eutrophication phenomenon. The pollution indicator parameters such as COD, BOD_5 , dissolved O_2 and saturated O_2, present worrying concentrations especially in the summer season **(realization years of the study: 1984-2005)** [16].

The watercourses that flow into the future site of Beni Haroun are characterize by poor water quality. A generalized contamination of the watershed by organic matter, nitrogenous matter and phosphorus matter is the consequence of a strong urban concentration. The high values of these parameters have a tendency to increase from upstream to downstream as the number of urban groups increases. The waters having undergone treatment, which remains low and leave some values exceeding the standards of discharge required **(realization years of the study: 1994-2003)** [6].

The temporal evolution of the content of the physico-chemical elements according to the volume of water in

the dam shows that the majority of the elements evolve in an inverse way to the volume of the dam; this can be explain by a phenomenon of dilution and concentration.

The pollution elements, notably COD, BOD_5 and the COD/BOD_5 ratio show that the raw water of the dam presents a high proportion of non-degradable organic matter linked to anthropic action. These waters cannot be used for drinking water without prior treatment, and they are generally of good chemical quality (**years of study: 2003-2013, annual monitoring)** [28].

Wadi El Kebir and its tributaries (Wadi Boumerzoug, Smendou, Ktone, Enndja, Kébir, Rafraf, Metlili ...) are subject to very important demographic, industrial and agricultural pressures; this fact obliges us to control the level of a possible contamination of waters and sediments.

The analyzed parameters, revealed cases of disturbances and alterations that were translated by a pH with alkaline tendency (not exceeding 8.5) and a generally high EC, reflecting an excessive mineralization, an increase in the rate of dissolved oxygen, that reflects an advanced process of self-purification, important rates of dry residue and major mineral elements, whose contents of cations (Ca^{2+}, Mg^{2+}, Na^+ and K^+) and anions (Cl^-, SO_4^{2-}), show a significant salinity **(realization years of the study: 2010, monthly monitoring)** [26].

The quality of the raw water from the Beni Haroun dam shows that it has a high hardness and mineralization with low levels of phosphates, nitrate and organic matter. Chlorides and hardness, present stable contents in time. The evolution of the different parameters during the study period shows the effects of biological incorporation, evaporation and sedimentation. This quality is good in winter and depends on seasons, it is. While the excessive hardness remains characteristic of the waters of the dam **(realization years of the study: 2010, monthly values)** [10].

The results obtained show that the waters of the dam have a high hardness and mineralization with low levels of phosphates, nitrate and organic matter. Chlorides and hardness, present stable contents in time and space. The quality of the raw water of the dam depends on the season. They are of good quality in winter **(the study years: 2010, monthly monitoring**) [29].

Secondary metabolites of pesticides in sediments, which is certainly due in part to the particular climatic conditions that preceded the sampling stage. These conditions are very favorable to the degradation of pesticides either by the biotic or abiotic mode. The organic load estimated by measuring the BOD_5 and COD shows that the concentrations of COD are high and suggest the presence of organic macro pollution upstream of the dam **(realization years of the study: 2011)** [27].

The water and sediments have a rather degraded quality with a certain homogeneity in their compositions. It is certain that the dam is influenced by discharges from urban sites in the Wilayas of Constantine and Mila **(year of the study: 2011)** [24].

For irrigation, the raw water from the Beni Haroun dam is of good quality and presents no risk to crops. The Cl^-, Na^+ and K^+ ions are mainly of external origin and can come from the saliferous formations of the Triassic or marl and clay formations. Na^+ and K^+ are derive from the alteration of silicate minerals. On the other hand, the element Cl^- can have a meteoric origin, i.e. from the leaching of the lithological formations by the rainfall **(realization years of the study: 2012-2013)** [13].

The results of the physico-chemical analyses from 2005 to 2014 show that a large percentage of the contents related to agricultural, industrial and domestic pollution is notable, which indicates that the raw water of the dam's reservoir cannot be used for human consumption without prior treatment. Synthesis of the physico-chemical parameters. The year 2005 allowed classifying this water between average and bad. While in the year 2014, it was of good to average quality. The surface water is practically not drinkable without prior treatment, due to the presence of various substances of natural or anthropic origin **(years of study: 2005-2014)** [2].

Measurements carried out in situ, show that the raw water of the dam has an alkaline tendency, with an average temperature around 18 °C and electrical conductivity (EC >1000 µS/cm). According to the assessment grid of the general water quality in 2014 is qualified as good quality (class 2) **(realization years of the study: 2014**) [17].

The main objective of the study is to evaluate the level of organic pollution in the raw water of Beni Haroun dam, and to compare their evolution over time during 6 months. A generally high EC, but not exceeding the standards, reflecting an excessive mineralization; an increase in the dissolved oxygen rate that reflects an advanced process of self-purification; nitrogenous matter contents (ammoniums, nitrite and nitrates) reflecting a good quality water probably due to the fact that the wastewater of the important agglomerations are treated in the treatment plants **(realization years of the study: 2013-2014)** [30].

Nutrients, whose content in nitrogen compounds (nitrate, nitrite and ammonium) exceed the standards, so according to the standards of ANRH, the waters of Wadi Rhumel are of poor quality. On the other hand, the water of Wadi Endja is of good quality **(year of the study: 2014)** [31].

Currently, the use of water from the Beni Haroun dam for irrigation purposes is practiced without any evaluation of the quality of this water by the farmers. A periodic control is recommend. The results of samples taken between September and March have shown an alarming increase in concentrations parameters. We also note, an additional contribution of chemical substances by runoff and in the rainy period **(realization years of the study: 2014-2015**) [12].

The quality control of the raw water and sediments of the Beni Haroun dam have allowed the identification of a pollution by PAHs (Polycyclic Aromatic Hydrocarbons)

and pesticides. It was also found that fish are quite significantly contaminated by persistent organic pollutants. The organic load estimated by measuring the BOD_5 and COD, shows that the concentrations of COD are high, which suggest the presence of macro organic pollution by non-biodegradable organic pollutants, and an accumulation of organic matter upstream of the dam **(realization years of the study: 2015)** [23].

It appeared that the quality was globally average, characteristic of a surface water with its variability and the importance of certain physicochemical parameters. The values indicate that the waters studied have a pH close to neutrality, a high mineralization and an acceptable hardness. For the elements considered undesirable or toxic (copper, iron, manganese and bromine) present low levels. Although the mineral and organic pollutions do not seem excessive, it is obvious that the water intended for consumption requires various physicochemical treatments in order to provide a water in conformity with the standards of potability **(realization years of the study: 2014-2015**) [18].

Water temperature variations depend on seasonal variations. The most alkaline values (pH) are attribute to the summer season. This alkalinity favors the productivity of phytoplankton. TSS contents in the raw water of the Beni Haroun dam are very variable both in time and in space; they depend on the nature of the land crossed, the season, the rainfall and the discharges. Apart from the periods of flooding, the TSS content does not present any anomaly. The highest values of Nitrate are attribute to the spring and summer seasons of the two years (2015-2016) with the exception of peaks appearing during the rainy season. The enrichment of water in nitrates during the rainy periods suggests that these ions come from the leaching of soils by runoff, inputs of waste of plant and animal origin rich in nitrogenous organic compounds and inputs of urban origin. The Beni-Haroun dam, give it a normal quality meeting the quality of fresh surface water intended for the production of drinking water. The nitrites also characterize the environments with active eutrophication. These non-negligible values are essentially the consequence of domestic discharges, fecal matter from grazing herds and phosphate fertilizers used in agriculture on the land surrounding the dam. The presence of sulfates is primarily relate to the geological nature of the bedrock and terrain in the area. The highest levels are recorded during the summer season, which may be the consequence of the phenomenon of evaporation that will tend to concentrate the salts **(years of study: 2015-2016**) [14].

The monitoring of the evolution of the physic -chemical quality of the raw water of Beni Haroun Dam, over a period of seven months (from September 2015 until March 2016), sampled monthly have shown that it is characterized by: a temperature below 25 °C which is seasonal, influenced by air temperature. A weakly alkaline pH between 7.4 to 7.9; an average of 1204.29 µs/cm for the electrical conductivity which is quite high, translating an excessive mineralization, important rates of dry residue and TSS revealing an organic pollution marked by strong organic and mineral loads. The evolution of the dissolved oxygen rate reflects an advanced self-purification process and generally well oxygenate waters. A BOD_5, a high COD, ammonium contents, nitrogen contents of nitrates, nitrites translating a water of good quality. Without forgetting a possible continuous arrival of some pollutants in the dam **(realization years of the study: 2015-2016)** [9].

The quality of the raw water of the Beni Haroun dam reveals relatively low concentrations for most chemical elements, except for chlorides, which have quite high levels, which are relate to the strong evaporation in the dam lake. A variation of major elements is observe from one tributary to another, this variation is due to the geological nature of the land crossed by the water, given the geological diversity of the basin. The analyses show that the most important pollutant load (organic) comes from the wadis Mila and Rhumel; this is due to the wastewater discharges of the agglomerations of Mila and Constantine. The study of the characteristic ratios shows a predominance of chlorides compared to sodium, which explains a second origin of chlorides, probably anthropic. Calcium comes from the evaporitic formation. A carbonate origin for calcium, and evaporitic for sodium, as well as sulfates. We also note the good quality of the water of the dam during the period of high water, without forgetting the existence of organic pollutants at the level of two tributaries feeding the dam **(realization years of the study: 2016**) [19].

The interpretation of the physic-chemical analyses show that: The pH of the waters is close to neutrality with a slight alkaline character; the conductivity and hardness are higher; the organic load is high in all the waters tested. However, the proportion of humic substances remains appreciable, indicating the natural origin of these organic materials; The Beni Haroun dam is considered to be the most polluted dam among the eleven dams studied, since it is characterized by poor physic-chemical quality both organic and mineral **(realization years of the study: 2017**) [21].

The raw water from the Beni Haroun dam that feeds the plant was of average quality overall, with some parameters having high levels. While some parameters seemed to be largely corrected by the treatment in the plant, other parameters such as organic matter, ammonia or bromides seemed less well influenced by the treatment of water in the plant **(realization years of the study: 2017)** [20].

The quality of the Beni Haroun dam varies between good, average and polluted for most of the measured parameters **(years of the study: 2018)** [11].

Nutrient levels are high in the raw water of the Beni Haroun dam. Nitrites and ammonium evolve in parallel with each other and the volume of water in the dam in the months of December, January and March, and inversely during the other months **(years of the study: 2017-2018**) [22].

CONCLUSIONS

After consulting the various works of evaluation and control of the quality of raw water of Beni Haroun dam, carried out by different authors over different periods (2003-2018). We note that the studies do not take into account all the parameters, and that there are always a few missing elements. We have gathered a set of conclusions, where we can see that each of the authors has issued a conclusion specific to the objective and the period of realization of his study, from which we can extract some deductions that converge in the same direction:

- Water quality is a function of the seasons, and is influence by climatic parameters such as air temperature (which modifies water temperature) and precipitation (water inputs help dilute concentrations).
- Some chemical elements (cations Ca^{2+}, Mg^{2+}, Na^{+} and K^{+}, and anions Cl^{-} , $SO4^{2-}$) are of geological origin.
- Others (Pb, Cd, Zn, Fe, Cr, Cu, Mn) are of anthropogenic origin.
- The raw water from the dam cannot be use directly for consumption and irrigation without undergoing adequate prior treatment.
- The quality of these waters is likely to be degrade by the proliferation of urban sites in the path of the tributaries feeding the dam.
- Organic nitrogen compounds vary with seasonal water inputs.
- Finally, they all formulated the perspective of establishing and maintaining a system of continuous monitoring and periodic control of the water quality of the dam.

CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

DATA AVAILABILITY

The data used to support the findings of this study are included within the article.

AUTHOR'S CONTRIBUTIONS

All authors are contributed equally to bring out this article.

ETHICS

There are no ethical issues with the publication of this manuscript.

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Review Article

Waste classification and separation practices from Türkiye and selected countries of the world

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ABSTRACT

Waste is an integral part of our lives. It is a fundamental by-product of human activities. Waste is divided into several groups, including medical, hazardous, municipal, biodegradable, industrial, and inert waste. Both the waste producer and its owners have many obligations imposed upon them by the appropriate standards and laws in force in each country. Waste classification and segregation have many benefits, including less environmental pollution; improved living conditions for plants, animals, and humans, and the ability to obtain better raw materials for recycling through segregation. In this study, waste classification and separation systems are assessed. Waste classification method that considers the source, basic composition, and physical, chemical, and biological properties of the waste. Manual, mechanical, and optical systems were used to separate the waste. Nowadays, smart waste classification and segregation systems are being developed for automation. They separate mixed waste effectively. It is a very modern and efficient method that requires less work to function properly than basic waste classification and segregation methods. It is also a faster, more professional method that avoids incorrect sorting of waste. It can be called a future-oriented way of waste disposal and should gradually be introduced into our civilization. Examples of this technology include smart bins, automatic bottle vending machines, and automatic segregation/sorting. Solving the waste classification and separation problem is one of the issues that need special attention in the coming years. The constant development of technology related to this topic is a staple of the circular economy.

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INTRODUCTION

It is natural that by performing many activities, man creates superfluous substances and objects that he wants to get rid of. The concept of waste is very broad because of its diversity. Among other things, we distinguish municipal waste, hazardous waste, biodegradable waste, and industrial waste. Each type of waste needs a suitable treatment system. The problem of waste and how to manage

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it has been known to mankind for a long time. However, in ancient times, it received much less attention than in modern times. It is inseparable from human development and the acquired ability to transform and produce goods from readily available raw materials. In the past, humans have created waste that could be completely absorbed by the environment. This was due to limited raw material processing skills. In other words, man did not transform the acquired materials, using them only to satisfy his basic needs. Thus, it did not disturb the natural circulation of matter in nature. Unfortunately, with the development of the human species and technology, this trend has changed. The man was able to produce much more processed products that satisfied not only elementary but also secondary needs. The amount of waste produced increased every year, but the most rapid increase occurred in the 16th century when the industrial revolution began. They assumed a series of economic, social, and technological changes that were initiated in England and Scotland. Until then, human work was mostly manual labour, resulting in limited production of basic materials. The revolution was characterized by the mechanization of production, in which manual labour was replaced by machines. This greatly accelerated the production of goods. People began to produce new materials, from which more diverse waste began to be produced. During this period, vacant landfills began to appear (due to excess waste). Over the past few years, urban migration has been on the rise, further exacerbating this problem. This was related to a better standard of living in urban agglomerations compared to rural areas. There were many more well-paid jobs in the cities. Centres were established in cities to meet the needs of people at that time, which intensified the migration of people using them. All these elements were the reason for the increase in the production of by-products that were difficult to manage [1].

Nowadays, we observe a constant upward trend in waste production, whose effective storage and management are becoming frequent problems. Increasing environmental problems and a global character have led countries to take serious steps both locally and globally. Also, the gradual decrease and rapid consumption of natural resources have made the concept of sustainability popular, and it has made the concept of environmental development widespread, considering that the balance between development and the environment should be maintained. For this reason, the concept of effective waste management began to be developed. Its aim is to reduce the negative impact of this waste on the environment. A significant part of the discussed waste management concerns solid municipal waste, which is generated because of the activities of many areas, including industry, households, and trade. Proper waste management practices vary by country and region (rural and urban). Differentiation also implies individual handling of waste from the industrial or residential sectors. In this study, the evaluation of solid waste has been handled with a systematic approach, and the concept of solid waste management has been developed by accepting that it is an issue that needs to be managed. It has become essential to use the developing technology for the collection, transportation, and disposal of solid wastes. One of the first steps required for the technology to be integrated into the system is correct classification, followed by separation and various disposal processes [2]. Observing the efforts of different countries in the activities carried out within the framework of waste management, one gets the impression that the performance of these processes is not uniform. The key issue is intervening in education on proper waste sorting/segregation as well as further treatment measures [3]. The biggest responsibility in this matter lies with the people. Solid waste management, which can vary according to the level of development and socio-political characteristics of societies, will no longer be an issue with measures taken at the individual level and conscious steps taken jointly. In this study, waste management practices in Türkiye and selected countries around the world are discussed.

TRADITIONAL WASTE CLASSIFICATION AND SEPARATION APPROACHES

Waste classification and separation are two of the most important issues in waste management. It is also a fundamental part of sustainability. Both activities are closely related and enrich each other. Classification enables proper waste sorting, and, as a result, proper waste segregation, collection, and disposal. Proper waste management is possible only through the correct classification and segregation of waste. The way these treatments are carried out depends on the country of occurrence and its characteristics. These are often deeply different systems that have been built on a common elemental pattern. To better understand the concepts of classification and segregation, in the following section, these basic schemes will be shown and presented.

Classification of Waste

Waste segregation is a particularly important part of waste management. This system facilitates other processes related to waste treatment. Wastes with similar or identical characteristics could be listed using reliable classification. The potential for waste treatment and recovery is very favorable. Therefore, the selection of waste classification criteria is necessary. The classification of waste is based on its origin, physical and chemical properties, toxicity, physical state, degree of danger to the environment and people, adequacy, and treatment [4]. Different properties must be considered for each waste classification criterion. For example, when classifying waste by toxicity, special attention should be paid to the content of the most hazardous component, which determines the carcinogenic potential of the waste, the degree of hazard, and water, soil, and atmosphere pollution or flammability [5]. Waste classification rules are set forth in the relevant legal acts in force in unions of countries, for example, in the European Union (EU) or

only in a certain country. Legal acts enacted individually by a country can vary from country to country.

In Türkiye, waste management is one of the main issues addressed by the Ministry of Environment, Urbanisation, and Climate Change (MoEUCC). Against this backdrop, announced the "National Action Plan on Waste Management to 2023" in 2016, which assesses the situation of waste management in the country, analyzes the waste management mechanism, and sets the country's waste management targets in the short term. Waste management has become an ever-evolving field and has become the raw material market for Türkiye. According to the report "Management of Municipal Waste in Turkey" published by the MoEUCC in 2016, landfills, and recycling facilities have increased from 15 to 82 and from 46 to 1226, respectively, compared to 2003 and 2016 [6]. Domestic, construction, and industrial waste are at the top of the waste bracket in Türkiye. Hazardous waste is incinerated at different facilities. İZAYDAŞ, Petkim, Tüpraş, Erdemir, and cement plants have the necessary permits and licenses to meet their energy needs in this way. In Türkiye, waste is sorted by code, like in many other countries. In Poland, there are 20 waste groups and waste classifications. The Waste Transport Table, formerly known as the National Waste Transport Form and completed online through the Integrated Environmental Information System, can dispose of its waste with the condition that the Ministry be notified of the waste codes of the carrier and the disposer. For example, the code for paper and cardboard packaging is 15 01 01 [7].

In Poland, the classification of waste is carried out in accordance with the applicable waste catalogue. Waste is divided into groups, subgroups, and types, and it is also specified which waste is hazardous. For this reason, each waste has its own six-digit code, in which the first two digits indicate its group -the source of waste- the next two subgroups -the component or process from which the waste is generated- and the last two digits -the type- the chemical composition of the waste. Hazardous waste is additionally marked with an asterisk "*" There are twenty waste groups and many subgroups and types. For example, the leaves have the code 200201 because they can be included in the group of municipal waste together with the selectively collected fractions (20), the subgroup of garden and park waste-02, and the type of biodegradable waste-01 [8]. Another example can be found in Australia, where the classification of waste is slightly different. In Australia, the national waste classification system is based on two foundations: waste streams and waste depots. The most frequently mentioned source sectors in this country are municipal solid waste (MSW), commercial and industrial (C&I) waste and construction and demolition (C&D) waste. However, detailed rules for the classification of trash are set by the governments of each of Australia's jurisdictions; in some jurisdictions, waste from various materials is classified strictly. For example, "fly ash" waste in Victoria, Queensland, and Western Australia is considered hazardous waste, while in South Australia it is not [9, 10]. In Japan, the classification of waste begins with its division into household waste and business activity waste. Household waste includes waste that can be incinerated or recycled at the end of its life cycle as well as waste that must be managed individually (non-combustible, toxic, or oversized). The Japanese government pays great attention to recycling, including in this group such items as paper, clothing, plastic bottles, cans, and bottles. This is a commonly used classification in developed countries. The difference is that Japan has created a separate category for recycled waste, which is white food trays. In the case of the second branch (business wastes), the process consists of the correct classification of waste generated in industrial activities. There are cases where part of the by-product can be classified and treated as household waste, but most of the waste is treated as industrial waste [11].

Separation of Waste

Waste separation is another basic and important element in the waste management system. Separation is the activity of placing specific types of waste in appropriate containers. After proper classification, waste is separated according to the material from which it was created. The waste segregation process most often takes place in the places where waste is generated, that is, in households or production companies [12].

The waste segregation system is not yet fully implemented in Türkiye. Many local authorities still collect mixed waste. However, with the zero-waste approach, improvements are made in the selective collection of waste, especially in public institutions and agencies, supermarkets, and various social facilities. All public institutions and agencies have been trained and recycling bins have been installed. The Zero Waste Project, which started being implemented in 2017, generated 16.5 million tons of paper and cardboard, 4.1 million tons of plastic, 1.7 million tons of glass, 0.4 million tons of metal, and 1.5 million tons of organic and other recyclable waste, for a total of approximately 24.2 million tons. Recyclable waste is brought into the economy. Furthermore, this project increased the recovery rate from 13% in 2017 to 22.4% by August 2021. It is hoped that by the end of 2023, this rate will have risen to 35%. Another practice considered part of waste reduction in Türkiye is the pricing of plastic bags, which aims to reduce plastic waste. With the decision taken in 2019, the use of plastic bags has decreased by 75%, and the formation of 354000 tons of plastic waste originating from plastic bags has been prevented. Because of the decision taken, the import of plastic raw materials required to produce plastic bags was reduced; approximately 2.44 billion Turkish Lira (TL) was saved, and 14640 tons of greenhouse gas emissions were prevented [13]. Many universities are still establishing waste collection centres today. As one of Türkiye's most important universities, Middle East Technical University (METU) which is an international research university with

around 27000 students and one of Türkiye's largest campuses, encompassing 4500 hectares, is highly appreciated for its solid waste management system. Bahçelioğlu's study includes the evaluation of the effectiveness of existing solid waste management strategies by various methods (survey, etc.) starting from waste generation. According to the study's findings, the daily average solid waste production factor on METU campus is 0.40 kg/day/person, with the total amount of waste produced ranging between 5.8 and 10.3 tons/day/weekly. It has also been stated that the total recyclable waste collected is 13% of the total waste [14]. Recycling facilities and/or businesses acquire waste-derived raw materials through a variety of partnerships with regional governments. The city has integrated waste separation boxes, waste oil collecting boxes, waste accumulators, and battery stations so that they can be used as raw materials in specific locations. According to the system created by Kepez Municipality in Antalya, a waste oil collection vehicle picks up used motor oil from specific locations on specific days and times. Once more, several information technologies (IT) firms (including Turkcell, a digital operator in Türkiye) coordinate programs for the segregated collection and recycling of electronic waste. Several foundations (including the Turkish Educational Volunteers Foundation (TEGV) and Turkcell Partnership) have stated that they accept electronic waste in terms of separate collection and evaluation and that they contribute to the circular economy. They also stated that the money they will make from this will be used to fund scholarships for students [15]. Reimbursement systems, which reward consumers for loading coupons or cards from waste bottles, are becoming more common thanks to the collaboration of environmental and software firms. It has been determined that training is necessary for the systems to be implemented successfully. As a result, new environmental-related courses were introduced to the curriculum. The separate collection of waste, particularly zero waste, has been the subject of short videos, public service announcements (PSAs), and numerous advertisements [16]. A sustainable waste management system and circular economy practices require that recyclable waste be collected separately at the source and that the recycling process be carried out in a planned manner. Separate collection of waste at the source, public participation in the zero-waste process, and raising awareness is essential for the success of this process.

The form of waste segregation at the source of its generation is also known as "selective waste collection" and forms the first element of a well-organized waste management system. Selective waste collection consists of collecting it individually from each property. For example, the selective collection of municipal waste (Figure 1) mainly involves the collection of recyclable materials such as metal, glass, plastic, or paper. This method of collecting waste has many advantages. These include, first, a smaller amount of waste sent to landfills; a collection of waste distinguished by the subsequent technology of their treatment; or the possibility

Figure 1. Selective collection of municipal waste [19].

of obtaining clean secondary raw materials. It is also worth noting that the reprocessing of secondary raw materials is much more beneficial than production using primary raw materials; it has a positive effect on the environment by reducing pollution and decreasing energy expenditure. Various types of (dedicated) containers, litter bins, and bags are used for selective collection of waste at the source. Another way of sorting waste is secondary sorting, which takes place in disposal plants. Both methods of waste segregation are used in Poland [17, 18].

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The segregation technology prepares waste for reuse and contributes to the possibility of recovering individual raw materials with the indicated degree of purity, which is why it is an extremely important process. Many countries around the world pay special attention to this element of waste management. In this case, it is worth looking at the inhabitants of Germany. Germany is a country that is an innovator in waste segregation in Europe due to its

high consumption and high living standard. When waste became a big problem there, interventions began. The principle of the three Rs (reduce, reuse and recycle) has started. The focus was primarily on the segregation of communal trash and its proper management. Initial sorting of household waste has become the most important. Special containers for waste batteries or electronic equipment were also implemented and installed in easily accessible places (e.g., supermarkets). The problem of bottles has also been solved; appropriate vending machines for accepting bottles have been designed and installed (Figure 2). Therefore, the user has the option of returning the previously paid deposit in the coupon. The coupon could be used for shopping, donated to charity, or exchanged for money. Also, each raw material intended for recycling is marked with an appropriate label to make the proper classification easier. This is an important point because, for example, if it were necessary to distinguish packaging made of polypropylene, polyethene, polystyrene, or polyvinyl chloride, there would be a problem because they are quite similar. The focus was also on the educational issue, and from an early age, efforts were made to inculcate the idea of proper trash segregation. Each of these steps helped reduce the massive amount of garbage. The basis for solving this problem was the improvement of waste segregation systems [20]. In Japan, the segregation of waste is analogous to the generally accepted pattern, i.e., the person or entity producing the waste is obliged to separate it properly and take it to the designated place. In the city of Utsunomiya (Japan), each resident has a list of the days when their garbage is collected from the common waste collection station. It is the resident`s duty to carefully select the generated waste and collect it in the household, awaiting the day when they can hand it over for further disposal. The Japanese pay a lot of attention to ensuring that the collected waste is properly prepared for the next processing stage. Residents must clean their garbage, shred it, and dry it to facilitate their further journey. Utsunomiya city government documents provide a detailed description of waste handling. We can, among other things, find information about household appliances that must be recycled. These include air conditioners, TVs, freezers, washing machines, etc. When getting rid of one of the above-mentioned devices, it is the owner's responsibility to pay the recycling fee and the pick-up service charge. A similar method of handling occurs with oversized waste, where the owner of the waste pays a fee for the excess kilograms of waste generated [11].

Figure 2. Vending machines for accepting bottles [21].

SMART WASTE CLASSIFICATION AND SEPARATION APPROACHES

Due to the economic development models adopted, the phenomenon of more people migrating to cities, and the different codes of conduct in different countries, waste management is a major problem to be solved. The most common waste treatment method is the manual method, which requires the use of workers. People who sort and store garbage can get many diseases due to the harmful substances contained in it. In addition, employees face many accidents that can seriously affect their health. These are automated systems that perform the sorting and segregation of mixed waste and, unlike previous methods, are more efficient and take less time to perform these operations. Technological development has enabled the creation of intelligent waste classification and segregation systems. These systems are more advantageous than existing methods and systems due to the following features:

- Make the sorting and separation process faster and more professional,
- Be healtier in terms of human health,
- Eliminate occupational health and safety problems associated with waste segregation,
- High discriminant performance,
- Recover lost time in sorting mixed waste,
- Preventing errors in waste sorting,
- Cheaper and with less environmental risk.

A smart sorting and separation system can be used to automatically sort waste, reduce human intervention, and prevent infection and contamination. It has a lower risk of error than traditional waste sorting and grading systems. It's a much faster system that works by limiting or eliminating human activity.

There are some smart waste applications and practices in Türkiye. In Manisa Akhisar Municipality, the amount of waste could be monitored from the centre with the smart container system. Thanks to the related system, the route can be redetermined, and unnecessary fuel consumption is prevented. In addition, regular monitoring of the container occupancy rate ensures the elimination of odour and visual pollution elements that may occur. It is stated that 4500 waste containers are included in the system within the scope of the smart waste collection system [22]. Muğla Bodrum Municipality has made some improvements to its waste management plan within the scope of the zero-waste

project. Container and vehicle tracking systems and the Clean City Tracking System have started to be implemented using Radio Frequency Identification Technology (RFID). Development activities in related systems are still ongoing [23]. The underground garbage container application was implemented by İstanbul Başakşehir Municipality. Within the scope of the zero-waste project, the smart garbage collection and sorting mechanisms, which have been implemented because of the efforts to make waste management more efficient and at the same time reduce the carbon footprint, are used by the Başakşehir Municipality. Thanks to the smart garbage collection system prepared using the RFID system, just like in Akhisar and Bodrum municipalities, preventing unnecessary fuel consumption and protecting the environment and public health are among the main objectives [24]. Antalya Muratpaşa Municipality uses LED screens to display the occupancy rate of garbage containers to users as part of smart waste management. In addition, the system obtains the electrical energy it needs from solar panels within the scope of renewable energy [25]. Nevşehir Municipality implemented a smart container system, mobile tracking system, route arrangements, and vehicle tracking system applications based on data in 2016. In cooperation with the Evreka firm and the municipality, it is stated that monitoring and follow-ups are carried out in 350 active containers, and because of the application, a decrease of 24% is experienced in the distances covered by the vehicles. In addition, it is reported that 15% of fuel savings were recorded [26].

A well-known and increasingly popular example is the previously mentioned reverse vending machines (RVM). These are automatic machines where people can recycle empty bottles, cans, and beverage containers. In return, the machine returns a receipt with which the owner can go to the point to collect the previously paid cash deposit, which is added to each recyclable bottle. RVM is a multifunctional device. When returning an empty bottle, the machine starts scanning the item, recognizing its size and material, and checking whether the bottle is empty. The bottles that will be approved by the machine are properly classified and segregated. Recyclable items are rapidly mechanically converted to provide size reduction, thereby increasing machine capacity. The converted items are sorted into appropriate containers that will be delivered to recycling companies. The system is widely used around the world, e.g., in Denmark. It is a country that is a leader in the effectiveness of the implemented solution. Over 92% of all purchased bottles are recycled using, e.g., RVM, which enables each person to contribute to improving the environment [27].

Another example is the Danish company “DON’T WASTE IT”, which introduced an innovative improvement to garbage collection during large events. Their product is a universal lid for a trash can that is properly marked and has specially measured openings for the collection of appropriate garbage. “Waste Top” fits commonly used containers with a capacity of 240 L. This eliminates the need to purchase dedicated containers, allowing you to make the necessary changes based on the amount of waste in each bin. Proper sorting of waste at the source increases the probability of avoiding errors in further parts of waste management and affects the construction of a closed circuit [28].

The other example comes from Norway and concerns an automated industrial waste sorting plant made by Bjorstaddalen in cooperation with Zen Robotics. The facility is in the municipality of Skien. A robotic station using artificial intelligence could carry out an independent waste sorting process with a capacity of 150000 tons per year [29].

Sweden is also actively implementing innovative measures that have a positive impact on more efficient waste management. In Alingsås Municipality, the concept of smart waste bins was presented, which were programmed to communicate and rely solely on solar energy. Thrown garbage is automatically compressed by the device. Smart bins communicate the current level of garbage filling. This facilitates the work of the municipality, adapting the way of emptying the bins to their real needs. By analyzing the degree of basket filling, it is possible to determine which locations the number of containers should be increased, and which should be reduced [30].

Smart waste classification and separation is a new concept with high potential for further development in the future. Nevertheless, it will not completely solve the problem of waste segregation and classification if the society is not sufficiently educated on the proper handling of waste.

CONCLUSION

Inadequate infrastructure, increased costs, the use of landfills because of local and regional agreements, and the implementation of joint decisions between districts and municipalities regardless of waste characterization are the issues that need to be improved in waste management. It is possible to increase waste recovery rates with the development and improvement of waste separation technologies, process selection based on waste characterization, and necessary infrastructure investments. In this context, any improvement that will reduce costs will be the key to local and regional development within the scope of waste management. Implementation of the "polluter pays principle" in industrial solid waste management and providing incentives and payment facilities within the reward system to companies that make clean production or contribute to recycling will be beneficial to waste management, especially for Organized Industrial Zones.

Waste management practices are various due to their diversity. Among other things, there is a distinction between municipal waste, biodegradable waste, and industrial waste. The problem of waste and how to manage it has been known to mankind for a long time. However, nowadays this problem has become significantly worse due to a steady increase in waste production and improper waste management. For this reason, the concept of effective waste management began to develop. It is primarily concerned

with the waste classification and segregation. This makes it possible to effectively pursue the introduction of a circular economy, which should replace its traditional model. Waste classification and separation offer many benefits, including reducing environmental pollution and improving the living conditions of plants, animals, and people, as well as obtaining better raw materials for recycling. Waste classification methods consider the source, basic composition, and physical, chemical, and biological properties of the waste. Manual, mechanical, and optical systems are used to separate waste. Smart waste classification and segregation systems are currently developed. These systems are automated. They separate and classify mixed waste efficiently. They are very modern and effective methods that require less work to function properly than traditional waste sorting and segregation methods. They are also faster and more professional methods of preventing incorrect waste segregation. This is what could be called a forward-looking way to dispose of waste and should gradually be introduced into our civilization. It is a way to effectively solve global waste problems facing humanity related to the state of the environment and thus maintain an appropriate level of economic growth.

DATA AVABILITY STATEMENT

The authors confirm that the data that supports the findings of this study are available within the article. Raw data that support the finding of this study are available from the corresponding author, upon reasonable request.

AUTHOR'S CONTRIBUTION

Conceptualization, N.D.-S.; investigation, K.U, N.D.-S., P.M.S, A.S.; methodology, K.U, P.M.S, A.S.; writing-original draft, K.U, N.D.-S., P.M.S, A.S.. All authors have read and agreed to the published version of the manuscript.

CONFLICT OF INTEREST

The authors declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

ETHICS

There are no ethical issues with the publication of this manuscript.

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Review Article

Green design or multiple re-useable product packaging as regards solid waste in selected areas in Awka Metropolis, Nigeria

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ABSTRACT

This study enumerates how product packaging amount to solid wastes among other Awka MSW constituents with the view to unfold green design or multiple re-useable product packaging as achievable means of minimizing solid waste in Awka Metropolis, Nigeria. Perhaps, there are locations with well managed or minimized packaging waste; accordingly, this study includes the review of some literatures on Awka MSW so as to note the extent of studies that have been published and made available in trendy media. Thus, the study is descriptive, literature-based and naturalistic observation -based in terms of estimated counting during the visitation to the selected areas. The findings reveal that studies on solid waste characterization, health, perception, behaviour of the populace in relation to solid waste and sustainability are ubiquitous. Still, the aspect of product packaging waste in the Awka metropolis has not been extensively studied; thus, present study fills this gap by aiming at unfolding green design or multiple re-useable product packaging as achievable means of minimizing solid waste in Awka Metropolis, Nigeria. However, this study in concord with prior studies shows that the product package among other solid waste occurs in a similar way in residential locations except other locations such as markets, schools, hospitals, municipal and industrial areas which have more of the litters of non-green packaging materials. Example is the plastic package with the largest quantity of generated solid waste within selected location in relation to other solid waste. Thus, the policy approach under which producers are given significant responsibility for waste prevention/diversion of post-consumer products should be ongoing through Private - Public Sector Partnerships (PPP) harmonized with EPR. This can be done by enhancing the concern of everybody (manufactures, consumers, organizations and other entrepreneurs including waste pickers/scavengers). Situation where by producers persuade the consumers through gainful or attractive means to return used product packages can be encouraged. Thus, the present study unfolds green design or multiple reuses of product packages as a means of attaining zero waste in Awka metropolis.

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INTRODUCTION

Green design can be synonymous to eco-friendly design or sustainable design. A sustainable design should reduce negative impacts on the environment, health or well-being of the people. To be eco-friendly is to be less threatening or harmful to the environment and the living organism, most especially human being. Similarly, green design involves the aspect of safe environment by focusing on waste prevention and organic environment void of toxicity. The length of product life, safety, waste management and to mention a few are important aspect of green design. This seems to be difficult to achieve in product packaging design of certain product as the manufacturers do make use of such non-green substance that requires some hazardous chemicals. Examples are components of solid wastes from electronic devices, automobiles, and to mention a few products. Thus, present study enumerates how product packaging amount to solid wastes among other Awka Municipal Solid Waste (MSW) constituents with the view to unfold green design or multiple re-useable product packaging as achievable means of minimizing solid waste in Awka Metropolis, Nigeria.

In quest of green design, the 10R principles should be the goal of contemporary designers and other stakeholders. The 10R includes the following; viz: Reduce, Reuse, Recycle, Renew, Refill, Repair, Re-manufacture, Replace, Refine and Remove [1]. It is the notion of present study that green design should harmonize human being, nature/environment and the products being used in a symbiotic association. For purpose of this symbiotic association, reducing the solid waste in the environment will benefit everyone because waste will be minimized. Reuse as a factor in green design will be economical in the sense that the manufacturers will not need to produce more of the product package because the available ones can be reused. This is also beneficial to everyone as the number of waste will be minimized. Thus, this approach can be more environmental friendly and enhance waste prevention as well as save money. 'Reuse' is somehow similar to refill in the sense that product content can be replace into the product package after the initial use. This aspect of reuse in green design is not new; but more innovation and technological development or certain logistics depleted this practice. It can be observed that this depleted practice as a result of certain logistics is a kind of backward attitude described by these phrases - 'grow first', clean up later' [2]. The 'grow first' notion will continue to create more waste if green designs are not put into use. In such situation, as the manufacturers are producing more non-green design then more profits are made (they are attached to benefit oriented aspects in order to have a better market of such product [3] but at the expense of more waste generation, environmental waste, exposure of people to hazardous toxic materials. It is also worthy of note that in a country like Nigeria, 'clean up later' is difficult to achieve. However, the government agencies are trying but the effort is not enough. Now, the pursuit of Sustainable Development Goal (SDG) is very germane to our wellbeing and stakeholders should be mindful of it. Stakeholders should try the possibility of reuse, refill, and recycle to mention a few in order to reduce the negative impact of non-green design on the environment [4]. Enterprises can also use it as a means of competitive advantage [5]. Thus, it can become means for strong driving force in the market towards sustainable consumption pattern [2]. This is because many stakeholders such as the manufactures will start to reduce waste disposal problems at the consumption and manufacturing level. Present study is of the notion that there should be reversal to the practice of 'reuse' in product packaging design especially in order to minimize solid waste. Thus, present study enumerates how product packaging amount to solid wastes among other Awka MSW constituents with the view to unfold green design or multiple re-useable product packaging as achievable means of minimizing solid waste in Awka Metropolis, Nigeria.

Design For Environment (DFE)

This is an innovative design approach where by environmental factors are considered very crucial in the design and manufacturing process of a product or service. DFE attempts to minimize the adverse influence of design on the environment. DFE is mutual to product life cycle in the sense that the raw materials, manufacturing and package design, distribution of the product, utilization or functionality of the product and end of life of such products are all considered. Thus, DFE is about better design, product performance as well as well-being of the ecosystem in the aspect of safety, health, and to mention a few. With this notion, present study acknowledges that green design is not new and several enterprises or stakeholders in the world have been making effort to operate according to DFE. However, this study recommends it not as a new development but a re-consideration of green design or multiple reuse product packaging as regards solid waste in selected areas in Awka metropolis. This justifies the notion of present study for exploring the existence of solid waste in Awka metropolis selected areas with the view to see how product packaging amount to solid wastes among other constituents of Municipal Solid Waste (MSW). Thus, this study generally appraises packaging as regards solid waste in selected areas in Awka metropolis with the view of recommending green design or multiple reuse-able product packaging.

PRODUCT PACKAGING

Packaging is the means of protecting products (such as food, electronics, manufactured good items, etc.) from damage. The aforementioned is the primary function of product packaging. It makes the delivery of products to be in good condition for the purpose of successful trade. This aspect of product packaging facilitates transportation, handling, storage and preservation. By putting DFE into consideration, lifecycle of packaging from production,

distribution and retailing, until the disposal of that package should be put into consideration [3]. Thus, the choice of raw materials for the packaging should be according to the 10R principles (it should be packaging materials that 'Reduce' environmental waste and other forms of hazards; stakeholders should be able to 'Reuse' the packaging materials in a beneficiary way; to 'Recycle' the materials should not be impossible, stakeholders should be able to 'Renew' the packaging material; to 'Refill' the product content should be reasonable and acceptable to the consumer; 'Repair', 'Re-manufacture', 'Replace', 'Refine' should be possible. In a nut shell, it should not give room for waste.

Having think of waste prevention, it may be sarcastic but reasonable to believe that aesthetic appearance of the product package should prevent waste in the environment. To certain consumers, product packaging may be very attractive or attention grabbing because of its graphic design. It is then surprising to find these attractive product package in the waste bin. Is it not an artwork? Because of the aesthetics of product packaging, people who love beautiful things should not trash them. Pedestrians who buy biscuit along the road trash the packaging immediately. The pedestrians also see metallic container (.e.g. can), paper (.e.g. carton), plastic and to mention a few all round littering the surrounding, by the roadside and even inside the waste bin [6]. Someone's mindset is that these product packages are graphically appealing and should not end as waste. Thus, present study enumerates how product packaging amount to solid wastes among other Awka Municipal Solid Waste (MSW) constituents with the view to unfold green design or multiple re-useable product packaging as achievable means of minimizing solid waste in Awka Metropolis, Nigeria.

Traditional and Modern Product Packaging

Traditional packaging such as plant leaves, bamboo baskets, animal skins or clay pots are typical early packages.

These are made from natural or organic materials such as banana leaf as seen in Figure 1 for food packaging. If we compare this traditional packaging with modern product packaging, it will be discovered that the transition is all about innovation and technological development.

Figure 1. Banana leaves as example of traditional packaging.

Both modern and traditional packaging still function as protection, preservation, containment and ease of distribution or transportation. But the aspect of marketing, communication, protection, preservation, containment and ease of distribution or transportation will be different due to the technological advancement.

It is naturalistic to pronounce that the traditional packaging will contribute to environmental pollution (.i.e. in aspect of temporary unpleasant odour, quicker decay or degradation which will eventual lead to green manure) but its pollution is symbiotic to the ecosystem unlike certain modern packaging such as plastic. The traditional packaging (.i.e. plant leaves and others) are biodegradable. Metallic packaging like tin/can will rust after some period of times; according to the Mumbai government it will degenerate between 100 to 500 years. Likewise, other modern product packaging materials like plastic also takes a very long time to degenerate. Then, green design which is an innovation for modern packaging can be seen to be modeled in accordance to the behaviour of traditional packaging.

In Figure 2, majority of the conspicuous solid waste are the discarded modern packaging materials. Plastic related package and metallic substance can be seen while others can be considered to be degraded by undergoing rapid decomposition. Paper which is one of the modern product packaging is considered environmentally friendly .i.e. biodegradable. It is one of the modern product packaging material considered to be according to green design. In Table 1, paper takes 10 to 30 days to decompose compare to other packaging materials which takes above 500 years to degenerate.

Figure 2. Various solid wastes which consists of discarded modern product packaging like metallic can and plastic bottles to mention a few.

Thus, present study enumerates how product packaging amount to solid wastes among other Awka Municipal Solid Waste (MSW) constituents with the view to unfold green design or multiple re-useable product packaging as achievable means of minimizing solid waste in Awka Metropolis, Nigeria

SOLID WASTE

Waste can be something which the owner no longer want at a given time and specific position because to him or her such things have no current or perceived market value

[7].If wastes are refuse, garbage or rubbish things that are derived from places of human and animal habitation, then they are as a result of the activities of nature .i.e. human being; however, waste that are biodegradables are more beneficial to nature. Hence, can be considered to be symbiotic [8].

Table l. Decomposition Rates of Solid Wastes [9]

Solid Waste	Approximate time of degeneration
Vegetable and fruit peels, leftover foodstuff, etc	A week or two
Paper	10 -30 days
Cotton cloth	2-5 days
Wood	10-15 years
Woolen items	1 year
Tin, aluminum, and other metal items such as cans	100 -500 years
Plastic bags	One million years
Glass bottles	Undetermined

The biodegradable ones are the decomposable refuse while the non-decomposable are not degradable. Thus, solid waste either non-biodegradable or biodegradable can be considered as unwanted substance either solid, semi-solid, liquid or non-gaseous products which are discarded as a result of human or animal activity. They consists of complex mixture of different substances present in such garbage, or food waste, paper, glass, cars and other household wastes in our surroundings which may be beneficial or disadvantageous to the ecosystem.

Threats to the environment and human health as result of the heaps of garbage (solid wastes) that pile up in neighbourhoods and ineffective management [10] is one of the aspects of the disadvantages in recent times in most Nigerian urban cities. Thus, present study enumerates how product packaging amount to solid wastes among other Awka Municipal Solid Waste (MSW) constituents with the view to unfold green design or multiple re-useable product packaging as achievable means of minimizing solid waste in Awka Metropolis, Nigeria

The Table 1 is a publication by the Mumbai government showing different rates of decomposition of Solid Waste. From Table 1, certain product packaging product such as plastic is estimated to take up to one million years to decompose. Tin, aluminum and other metals approximately takes between 100 to 500 years to decompose while glass bottles are undetermined. So, if ignored can wreck the environmental suitability and existence of any well-meaning people. Such deterioration in environmental quality affects adversely the health and longevity of human beings and other living organisms.

AIM AND OBJECTIVES

Present study enumerates how product packaging amount to solid wastes among other Awka Municipal Solid Waste (MSW) constituents with the view to unfold green design or multiple re-useable product packaging as achievable means of minimizing solid waste in Awka Metropolis, Nigeria. The specific objective is to investigate whether product packaging amount to solid waste in the similar manner across the selected location within Awka metropolis. Probably, there are locations where there are well managed and minimized packaging waste. Accordingly, the study includes the review of some literatures on Awka

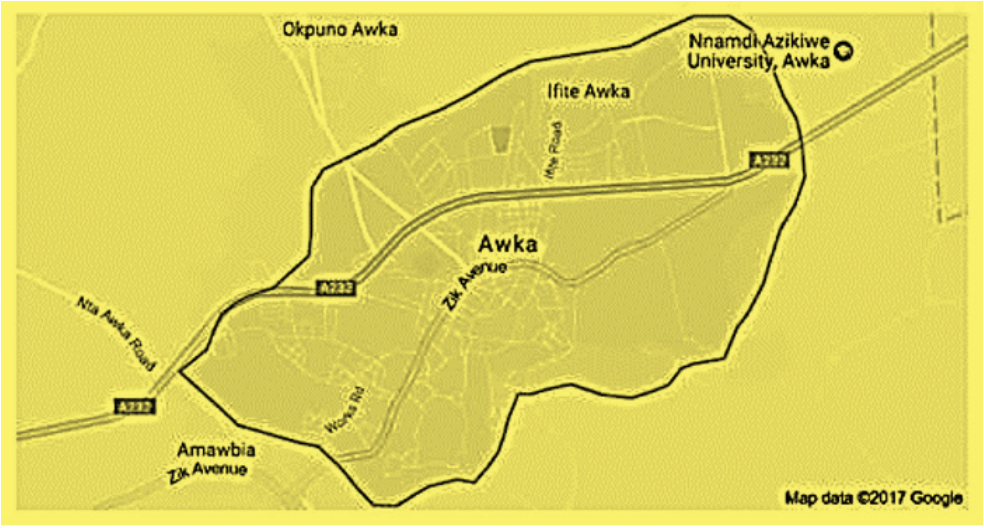

Figure 3. Closest places to Nnamdi Azikiwe University (NAU).

MSW so as to note the extent of studies that have been published and made available in trendy media.

SCOPE OF THE STUDY

This study focuses on selected Awka MSW as regards product packaging, and review of selected literatures on Awka MSW.

Study Area

The study area includes selected area in Awka, Anambra State in Nigeria. Figure 3 is the map showing the closest places to the Nnamdi Azikiwe University (NAU). Proximity to the NAU, is one of the criteria for choosing selected Awka MSW as regards product packaging, and review of selected literatures on Awka MSW.

METHOD

This study is descriptive and it is based on the review of selected prior literatures on Awka MSW as regards product packaging. The researchers searched for the statements '*Product packaging as integral components of solid waste in Awka Metropolis*' on the Google Chrome (GC), Semantic Scholar (SC), ResearchGate (RG) and Google Scholar (GS) on the February 4, 2023. Figure 4 shows the pie chart depicting the extent of related information obtained from the aforementioned sources.

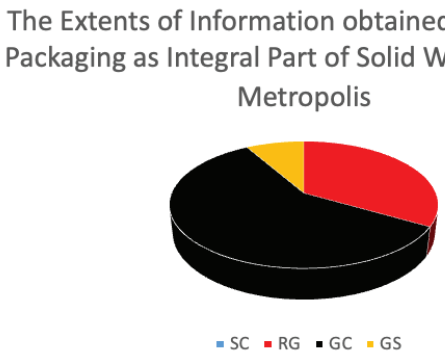

Figure 4. Pie chart of the extent of related information obtained from the GC, SC, RG and GS.

GC has the largest articles related to the scope of this study (see Figure 4); it provides the link to the journals of the articles or the other sources of the articles. Some of the PDF scholarly articles are downloaded directly from the GC without loading the exact source of the articles. Examples are the three (3) articles from different journals downloaded directly from GC; namely, International Journal of Environment and Pollution Research (IJEPR); Journal of Environment and Earth Science (JEES); NG- Journal of social Development (NG- JSD). By using set theory notation,

$$\{\{GS \cap GC\} \cup \{RG \cap GC\}\}' = \{\text{IJEPR, JEES, NG- JSD}\} \quad \text{(i)}$$

$$n\{\{GS \cap GC\} \cup \{RG \cap GC\}\}' = \{3\} \quad \text{(ii)}$$

The set notation in equation (i) and (ii) shows that there are three (3) articles not yet made available in GS and RG but are only in GC. Thus, it is intelligent to consult more than one internet sources in order to get suitable prior researches for this present study. The Venn diagram in Figure 4 shows the relationship that exist among the internet sources browsed for the purpose of this study. SC is disjointed from the rest because there are no related articles obtained from it.

In Figure 5, there is no intersection between GS and RG; it is surprising that available related articles are not simultaneously existing in both internet sources (.i.e. GS and RG). GC only behave as a link between GS and RG; thus, this proves that GC is a cross-platform web browser while other internet sources are media for academic scholars.

$$n\{GS \cap GC\} = 1 \quad \textit{(iii)}$$

Equation (iii) shows the intersection of GS and GC; only one article is simultaneously existing in both internet sources. This is a research published by MDPI Recycling (see equation iv). Equation (v) and (vi) show the intersection of RG and GC consisting of Journal of Applied Science and Environmental Management (JASEM), and Journal of Environmental Management and Safety (JEMS).

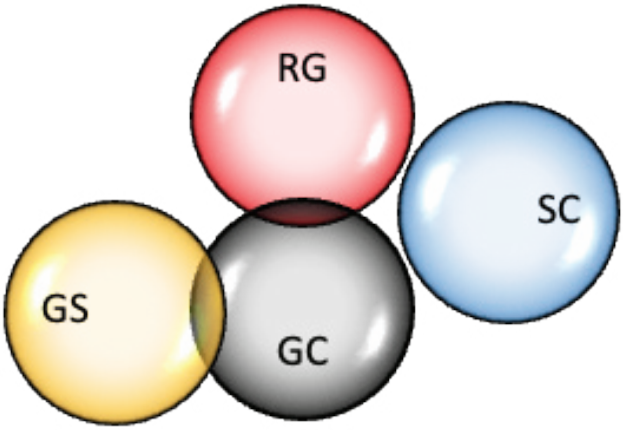

Figure 5. Venn diagram for the relationship between the GC, SC, RG and GS in terms of the selected journals.

$$\{GS \cap GC\} = (MDPI\ Recycling\} \quad (iv)$$

$$n\ \{RG \cap GC\} = 3 \quad (v)$$

$$\{RG \cap GC\} = \{JASEM, JEMS, JEMS\}\} \quad (vi)$$

In equation (vii), there are no related article existing simultaneously in GC, GS and RG. The total numbers of articles are 8 as seen in equation (viii) which is the union of the sets.

$$n\ \{RG \cap GC \cap GS\} = \emptyset \quad (vii)$$

$$n\ \{GS\ U\ GC\ U\ RG\ U\ SC\} = 8 \quad (viii)$$

$$\{GS\ U\ GC\ U\ RG\ U\ GC\} = (MDPI\ Recycling, IJEPR, JEES, NG\text{-}JSD, JASEM, JEMS, JEMS, JEH) \quad (ix)$$

The result of the internet search shows eight research papers as seen in Table 2; thus present study reviews only these eight papers. Naturalistic observation also is involved during the visitation to selected areas in order to observe how product packaging forms integral components of solid waste in Awka metropolis (see Figure 6 for diagrammatic representation of the procedure for data collection).

RESULTS AND DISCUSSION

Figure 7 depicts the extent to which solid wastes in selected areas within Awka metropolis have been studied. Based on the findings from prior studies, solid waste in residential areas as well as dump sites are often examined by prior researches (see Figure 7). That is, there are more studies on the location of solid wastes within the residential areas as well as dump sites within Awka metropolis. Not much researches have been carried out on solid waste in locations like water bodies, road, abattoirs, abandoned building, market/commercial centres, and school offices to mention a few.

Table 2. Eight (8) Research Topics from Seven (7) Journals reviewed

S/N	Journal	Topic	Authors	Internet Sources
1	MDPI Recycling	A Statistical Regression Method for Characterization of Household Solid Waste: A Case Study of Awka Municipality in Nigeria	Ezeudu, Ozoegwu & Madu	GC and GS
2	IJEPR	Evaluation of People's Perception on Plastic Waste Management, A Study of Nnamdi Azikiwe University in Awka, Anambra State	Onwuka and Ajator	GC
3	JEES	Evaluation of Domestic Solid Waste Disposal in Two Selected Housing Estates in Awka, Anambra State (Case Study of Udoka and Real Estates)	Ozoemene, Obienusi, & Ezenwaji,	GC
4	NG- JSD	Effect of Solid Waste Management on Sustainability of Clean Environment that Promotes Healthy Living in Nigeria: A Study Of (ASWAMA) Awka, Anambra State of Nigeria	Obi, Orga, and Ogadimma	GC
5	JASEM	Survey of Waste Disposal Methods in Awka Metropolis	Bill, Chidi Christopher and Ewuzie	RG and GC
6	JEMS	Developing an Effective Urban Waste Management System for Sustainable Environment: A Case of Awka Capital Territory, Anambra State.	Okeke and Anukwonke	RG and GC
7	JEMS	Behavioural and Household Characteristics Influencing Solid Waste Generation in Awka, Anambra State, Nigeria	Egbu, Umunakwe, and Ogbonna	RG and GC
8	JEH	Solid Waste Management in Awka Metropolis and Public Awareness : Sensitizing the Populace Through the Use of Social Cartoons	Onwuekwe and Okoye	RG

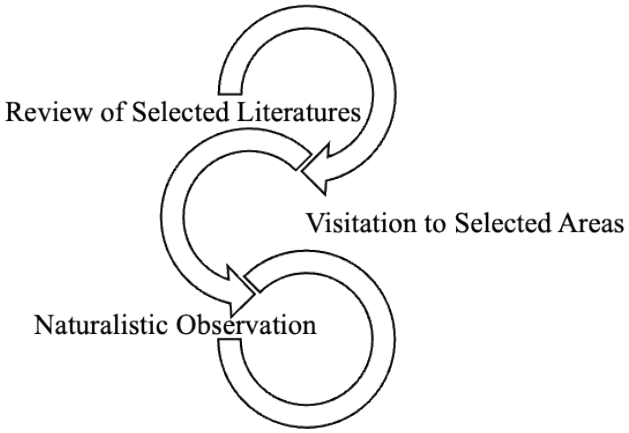

Figure 6. Procedure for data collection.

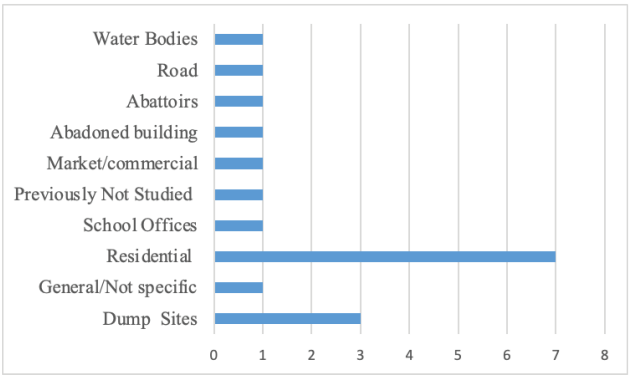

Figure 7. Locations of Solid Waste within Awka Metropolis based on prior studies.

Table 3 shows the selected areas within the Awka metropolis as studied by prior studies (.i.e. the eight (8) research papers reviewed in this current study). Studies on previously not studied location are scarce (see Figure 7). Also, studies that are generalized (.i.e. that are not specific to particular location) within Awka metropolis are few (see Figure 7). Having, based the rationale of current study on DFE, to re-examine the existence of solid waste in Awka metropolis selected areas with the view to see how product packaging amount to solid wastes among other constituents of Municipal Solid Waste (MSW) is considered significant to the scope of current study. Thus, present study will build on this existing body of knowledge in order to explore the existence of solid waste in Awka metropolis selected areas with the view to seeing how product packaging amount to solid wastes among other constituents of Municipal Solid Waste (MSW).

It can be assumed that the authors (.i.e.Ezeudu, Ozoegwu and Madu, [11]) probably studied locations where there are less of industrialized packaged products (see Table 3 and 4).

This is because majority of the solid waste are organic which is believed to be more related to green environment. In another more positive way, it can be assumed that the few plastic which are found in the environment can also be minimized further by adhering to the 10R principles (.i.e. Reduce, Reuse, Recycle, Renew, Refill, Repair, Re-manufacture, Replace, Refine and Remove). Onwuka and Ajator [12] also affirms that majority of the people never conceive the idea of re-using the solid waste (see Table 4); thus, they dispose them anyhow or incessantly. Eventually, more solid waste are generated because the 10 R principles are not followed. This can be the reason for the composition of the dump sites as studied by Ozoemene et al. [13] which are more of plastics and other non-biodegradable. This also attest to the fact that the solid waste within the selected areas in Awka metropolis can be further minimized if green design or multiple reuse is inculcated.

The study according to Egbu et al. [17], shows that the wealthy people generates more solid waste because they purchase more products that are industrially processed and packaged in different form of product packages such as glass bottles, can, and paper to mention a few. As a study carried out on residential location, the study shows that the putrescible form of the solid waste are the highest of the solid waste. The putrescible are more of food related

Table 3. Selected Awka Area as studied by the Authors

S/N	Selected Awka Area	Authors
1	Previously Not-Studied Location	[11]
2	Three (3) Hostels in Nnamdi Azikiwe University	[12]
3	Two (2) Selected Housing Estate	[13]
4	Surroundings of the Dump Sites in Awka	[14]
5	Some Dump Sites and School/Office premises in Awka metropolis	[15]
6	Waste Disposal Points around Residential, Commercial Settlements/Market Areas and Abattoirs; Natural Features and Water Bodies' .e.g. rivers; Abandoned Roads and Buildings etc used as Dump Sites in Awka.	[16]
7	Residential neighbourhoods of Awka.	[17]
8	No specific location within Awka metropolis but it concerns the inhabitants or populace	[18]

Table 4. The focus and findings of prior studies

The Focus of the Eight (8) Prior Studies	Findings of the Eight (8) Prior Studies
Zero-intercept first-order polynomial regression used for household solid waste characterization [11]	The results from the proposed method proved more accurate when compared with traditional averaging techniques (.i.e. organic (73.2%), plastic (8.0%), recyclable (20.3%) and to mention a few).[11]
Perception and awareness of people about plastic waste and the process of pyrolysis [12]	Lack of awareness on the process of pyrolysis, thus they handle waste anyhow [12]
Composition of these solid waste at the dump sites; as well as performance & whether MSW has effects on the health of the inhabitants [13]	Composition of dump sites includes non-biodegradable such as plastics, polythene bags, and scrap metals. Also, respondents are aware that solid waste can affect health of the inhabitants [13].
Challenge & extent of Anambra State Waste Management Authority (ASWAMA) operation of enhancing clean environment [14],	Operations of ASWAMA in solid waste management have not been effective in ensuring a sustainable and clean environment in Awka, Anambra State [14]
Waste disposal method and perception of the respondents [15]	People have particular preferred disposal method (e.g. burying and burning ,etc) without consideration for health and risky consequences [15]
Sustainable plan for the effective management of solid waste [16]	ISWM, IIF and PPP undertaken in a consultative manner remain as the plan that can guide the technological, social, financial options for the Awka metropolis to move towards becoming a zero waste city [16]
Explanation of the nature of generated household solid waste by using planned behaviour and rational economic behaviour theory [17].	Putrescible forms the highest component of solid waste and some of the ways by which solid waste in the aspect of clothing and books have been reduced is by passing them over to the extended family for reuse. The more the income of the people the more, the amount of generated solid waste [17]
Using cartoon illustrations for information dissemination to sensitize inhabitants of Awka metropolis on the consequences of indiscriminate refuse disposal [18]	The study demonstrates the possibility of using Cartoon illustration towards attaining green environment [18].

substances such as waste from vegetables, and fruits to mention a few. Less non-biodegradable .i.e. non-putrescible are also present compared to the putrescible. Having assumed that majority of the people never conceive the idea of re-using the solid waste [12], then it can be believed that solid waste can be minimized if green design or multiple reuse are practiced.

This suggestion is not the duty of the populace (i.e. consumers) but the policy makers, government, health organization, designers and the manufacturer. These stakeholders can make the 10 R principles (.i.e. Reduce, Reuse, Recycle, Renew, Refill, Repair, Re-manufacture, Replace, Refine and Remove) to be part and parcel of the social environment. Thus, to reduce waste disposal problems at the consumption and manufacturing level will be more realistic.

Onwuekwe and Okoye [18] just like radio or TV broadcast also used cartoon illustrations to sensitize the inhabitants of the Awka metropolis on the consequences of indiscriminate refuse disposal (see Table 4). These cartoon illustrations should be used as mural paintings on the wall of public places. Also, the cartoons can be included in the newspapers, magazine and other print media prominent within Awka metropolis. From observation, the numbers of explanations that might be provided by cartoon illustrations, mural paintings, publicity design, TV or radio broadcast are not enough to bring about zero waste environment in Awka metropolis. Moreover, Obi et al., [14] findings show that the operations of ASWAMA in solid waste management have not been effective in ensuring a sustainable and clean environment in Awka, Anambra State (see Table 4). Although, the government are trying to apply some measures in the aspect of providing incinerators and disposal of waste within the Awka metropolis.

Okeke and Anukwonke, [16] also opined that Integrated Solid Waste Management (ISWM), Integration of the Informal Sector (IIF) and Private - Public Sector Partnerships (PPP) undertaken in a consultative manner serve as the master plan that can guide the technological, social, financial options for the Awka metropolis to move towards becoming a zero waste city (see Table 4).

PPP consists of the government and private sector cooperation to be responsible for solid waste collection and treatment. ISWM focuses on designing a new waste management system, or rationalize an already existing one while the IIF consists of other sectors of solid waste management

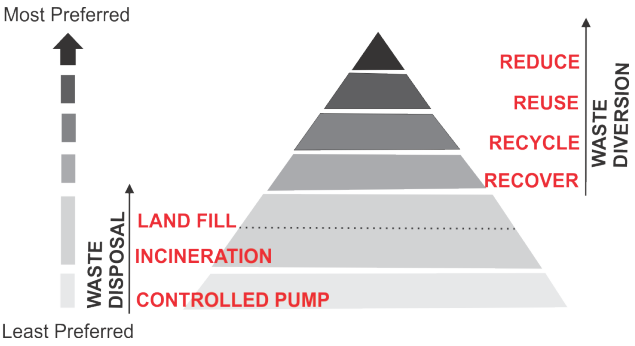

Figure 8. Level of preference for Integrated Waste Management by the World Bank.

that are not in anywhere operating with the government or recognized organization [16].

It seems that the government and other stakeholders focus on the least preferred waste management such as land fill and incineration to mention a few (see Figure 8).

The most preferred waste management by the World Bank such as Reduce, Reuse, Recycle and Recover to mention a few waste diversion are not yet well focused on by the government and other stakeholders. Expecting consumers to be engage in this aspect of waste diversion is not realistic because they do not have the capability or potential to do such. Majority of the consumers are not aware of pyrolysis, thus they handle waste anyhow [12]. From the study of Bill et al., [15], the populace have preferred method (.e.g. burning and burying) of refuse disposal which may not be favourable to health and well-being (see Table 4). Thus, less responsibility should be expected from the populace in order to attain green environment as many of them are not obliged or obligated to take appropriate step. Let the producers of these products (such as packaged food, beverages and to mention a few) as well as other stakeholders oblige, persuade and obligate the people through impressive and effective means. The policy makers, government, health organization, designers and the manufacturer have more capability and responsibility to reduce waste disposal problems at the consumption and manufacturing level. This is because they have more power to positively influence the possibility of reuse, refill, and recycle to mention a few in order to reduce the negative impact of those solid wastes generated as result of the consumption of their products which consumers buy. It can be envisioned that the involvement of these stakeholders .i.e. the enterprises can be as well used as a means of competitive advantage. Thus, it can become means for strong driving force to move towards becoming a zero waste environment.

Classification of the Focus of the Eight Reviewed Studies as Regards Present Study

Solid waste characterization: Solid waste characterization involves the collection and sorting of solid waste so as to ascertain the types of materials. Present study used naturalistic observation by visiting selected areas within Awka metropolis so as to see how product packaging amount to solid wastes among other constituents of Municipal Solid Waste (MSW). Estimates of the amount product package seen are done by estimating in percentage and not by weighing. Present study fails to use Zero-intercept first-order polynomial regression for household solid waste characterization as done in prior studies.

Perception and Behaviour: This aspect deals with the people living in the environment where these solid waste are generated. It is the activities .i.e. the behaviour of these people that definitely lead to the generation of these solid waste. They are consumers; thus, they will definitely exhibit consumption behaviour. The level of welfare of the citizens and the high number of working people can reflect the behaviour in terms of kinds of waste that could be generated by their activities. In the aspect of product package waste, a study reveals the amount of organic wastes (kitchen wastes, park and green wastes) and packaging waste (paper, cardboard, bulky cardboard, plastics, glass, metals and bulky metals) through the activities of some citizens [19]. This kind of behaviour forms the main parameter for environmental pollution [20]. For instance, more than one thousand billion liters of packaged beverages are consumed worldwide [20]. If 1,292 billion liters of packaged beverages are recorded to have been consumed worldwide in 2020, then the waste generated by the consumption of beverage products has increased in recent years [20]. Thus, present study used naturalistic observation to see how product packaging amount to solid wastes among other constituents of Municipal Solid Waste (MSW) in Awka metropolis.

Health: Present study is not focusing on the health aspect of solid waste in Awka metropolis. However, it is expected that when solid wastes are illegally disposed along road sides, sewage canals and reserved open spaces they contribute to floods and breeding of rodent vectors that eventually spread diseases such as intestinal schistosomiasis and soil transmitted helminths [21]. Prior studies have shown that people are careless about consideration for health and risky consequences [15] in Awka metropolis in terms of handling solid waste.

Disposal Method: Present study is not focusing on the disposal method of solid waste in Awka metropolis as other prior studies have done. Prior studies have shown that people have particular preferred disposal method (e.g. burying and burning, etc) in Awka metropolis.

Sustainability: It covers preferences that aim that creating long-term value and competitive advantage by considering eco-friendly factors. This relates to the aspect of green design or multiple reuse which this study aims at recommending.

PRODUCT PACKAGE WASTE AMONG OTHER SOLID WASTES IN SELECTED AWKA METROPOLIS

Selected locations include residential, market, hospital, school, municipal, and industrial location. Wastes from food, pure water bags, cloths, polythene, tins, paper, plastics, empty cartons, glass, sticks, leather; enamel plate, ceramics, metals, leaves, grass, rubber, and wood to mention a few are observed to be littering the residential locations. In the market places, wastes from food, plastics, polythene, pure water, empty cartons, paper, wood; cloths, leather, tins, ash, rubber, leaves, grass, metals, ceramics, fabrics, bricks, glass, and bottles to mention a few also litters the area. Few broken glass, glass bottles, plastics, paper, polythene, pure water, and few food waste are among the solid waste around school premises. In the hospital, laboratory wastes, surgery wastes, pathological wastes, maternity wastes, syringes, expired drugs and chemicals are among the wastes observed. However, these are properly disposed in the hospitals. Those waste found littering certain part of the hospital premises include plastic bottles and other types of plastics inside the gutter. Discarded large motor parts, large appliances, furniture, refrigerators, tyres, metal scraps, dirt, silt from gutters, leaves, content of refuse, cloths, plastics, paper, pure water satchels are found as waste in the municipal locations. In the industrial locations, Solid wastes resulting from industrial processes and manufacturing operations, wood, plastic and metal scraps, hazardous wastes, radioactive materials, conduit pipes, wire insulation scraps, nails, blocks, cement bags, and to mention a few are present.

Figure 9 shows the estimates of the observation in terms of frequency depicted by the use of bar chart. Also, Friedman test is conducted to determine whether product packaging form an integral components of solid waste in the same way in the selected location within Awka metropolis. Every foreign manufacturer as well as local producers have to enter into agreement with the government concerning the waste prevention in as much as their products are been consumed in such region. Paper package as shown in Figure 9 is one of the ways by which package contribute to solid waste. It is present in all the selected areas with a mean rank of 4.36 by using Friedman's analysis by rank. Compare to other solid waste which are not among product packages, there are lesser waste caused by paper package. There are little paper packages in the municipal and industrial locations. This is assumed to be because they decomposes easily (10-30 days).

However, there are more of paper packages in the residential and school areas. In the overall, safe environment by focusing on waste prevention and organic environment void of toxicity have been manageable in the aspect of paper packages in Awka metropolis. Green design is very well in place in paper package while multiple reuse is not obvious among the users of paper package.

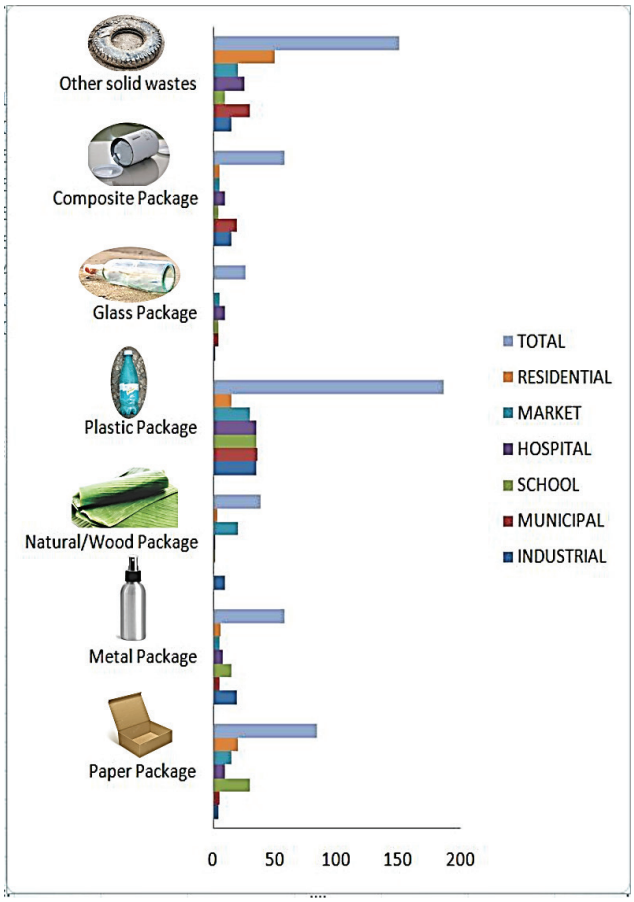

Figure 9. Level of Package Waste in Relation to Other Solid Wastes.

It is surprising that metal packages (mean rank =3.71) do not cause more solid waste. This is assumed to be as a result of the level of recycling that might be happening to the metal packages. The influence of the scavenger which can be classified among the PPP have reduced the quantity of metal package in Akwa metropolis. Compare to other solid waste which are not among product packages, there are lesser waste caused by metal package because a lot of the 'would-be' wastes are picked up by the scavengers. In Figure 9, there are little metal packages in the market, municipal and residential locations because of the influence of the scavengers who are everywhere sourcing for metal packages. Thus, the recycling of metal packages have not allowed more solid waste to be generated in Awka metropolis.

Compare to other solid waste which are not among product packages, there are lesser waste caused by natural/wood package. This is because the use of wood for packaging is declining. Wooden crate is no more popularly used compare to how it was previously used several years ago (see Figure 10). The natural ones such as baskets made from raffia, cane and palm trees still found littering the market areas.

Figure 10. Wooden crate.

With a mean rank of 2.21, natural /wood packages compare to other solid wastes have not generated waste beyond management except the baskets in the markets and wood dusts/ blanks in industrial locations. There are little or no natural/wood packages in the school, municipal and residential locations. In the overall, safe environment by focusing on waste prevention and organic environment void of toxicity have been manageable in the aspect of natural/ wood packages in Awka metropolis.

It is also surprising that glass packages (mean rank =2.0) do not cause more solid waste. Even though, a lot of products like beer, wine, and mayonnaise to mention a few are contained in glass package there are lesser waste of this kind of package. This is assumed to be as a result of the level of multiple reuse and recycling that might be happening to the glass packages. The influence of the scavenger which can be classified among the PPP have reduced the quantity of glass package in Akwa metropolis. Also, some itinerate workers go from house to house buying glass packages. Compare to other solid waste which are not among product packages, there are lesser waste caused by glass package because a lot of the 'would-be' wastes are picked up by the scavengers. In Figure 8, there are little or no glass packages among waste around residential locations because of the influence of the multiple reuse and scavengers who are everywhere sourcing for glass packages. Restricted location such as school and hospital where these itinerate workers cannot freely enter still consist of glass package refuse waste (see Figure 8). Thus, the multiple re-use and recycling of glass packages have not allowed more solid waste to be generated in Awka metropolis.

Compare to other solid wastes which are not among product packages, there are lesser waste caused by composite package (mean rank = 3.43). Example of other solid waste (mean rank = 5.57) not among the product packages is tyre (see Figure 9). Only plastic packages (mean rank = 6.71) in relation to other solid waste in Awka metropolis have the largest quantity of generated solid waste. This is assumed to be due to the fact that many products are contained in plastic materials. Ranging from soft drink, fruit drink, and milk drink to other edible or non-edible products [22]. This popularity can cause the increasing number of plastic package among other solid wastes in Awka metropolis. Thus, there is need for green design or multiple reuse of plastic packages in order to attain safe environment devoid of waste and toxicity in a long term perspective.

The result of that analysis indicates that the null hypothesis should be rejected at a significant level of 0.05, χ^2 (6) = 27.174, p = 0.000 (see Table 5). Thus, the way product packaging (.i.e. paper, metal, plastic, glass, natural /wood and composite package) form an integral components of solid waste in Awka metroplis are not similar within the selected location within Awka metropolis.

Also a T-test is conducted to determine whether product packaging as integral of other solid waste in previous study on residential location occurs in similar way as this present study. The result of the T-test score indicates 0.3002 at p-value of 0.05 (t=0.3002; df = 8; p = 0.05, two-tailed) which is less than p-value. Thus, the null hypothesis is accepted (.i.e. the product package among other solid waste occurs in similar way in the residential locations).

RECOMMENDATION

This study recommends green design or multiple re-use of product package as way of attaining zero waste environment in Awka metropolis. The 10R principles should be the goal of the stakeholders. Packaging can be subjected to taxes, re-use and re-cycling requirement. The policy that affect product design should also affect the disposal means of the product. Companies producing these products can be held responsible for the disposal of the product by a particular fee and take up the responsibility of recycling every waste as result of their products' consumption. Similar policy such as Deposit Refund System (DRS) can also be adopted. DRS is a recycling system in which consumers pay a small deposit value for bever- age containers, which can be refunded upon return of the used container to a collection point [20]. This is to avoid costly disposal of waste. EPR is a policy approach under which producers are given a significant responsibility of treatment or disposal of the post-consumer products. The

Table 5. The rejection of null hypothesis at a significant level of 0.05

Null Hypothesis	Test	Sig.	Decision
Product packaging form an integral components of solid waste in the same way in the selected location within Awka metropolis	Friedman Test	000	Reject the null hypothesis

government should spread their tentacles through Private - Public Sector Partnerships (PPP) to mannufactures of various products in order to establish this policy. This can be more effective in a developing country if PPP and EPR are harmonized. This will ensure government, non-government organization, and individuals participation in attaining Zero waste. This harmonization can be implemented by ensuring effective monitoring and compliance. EPR should not be responsible for only the financial burden but reduce packaging waste through necessary collaboration, incentives and encouragement with various stakeholders in the distribution chain of their products as well as other contractors or the general public (consumers). This can be achieved by harmonizing PPP and EPR. The entrepreneurial aspect of the private sector and quest for opportunity to invest by contractor can create more dynamism, finance and better managerial efficiency when EPR and PPP are harmonized. The public sector which includes the government and the populace will create social responsibility, involvement, and better compliance to the implementation of zero waste through the harmonization of EPR and PPP. This will relief the burden of the authority's waste management budget, due to higher effectiveness and access to external finance from both EPR and PPP. As a result of such harmonization, the waste pickers or scavengers will not be cut off from the system because there will be avenue for them to operate under PPP. Even when EPR systems are implemented by manufacturing companies setting up their own collection systems with the provision of incentives to households will be avenue for such as category of entrepreneur to be involved. This is because the avenue will upgrade the practice of the waste picker to be dignified and admirable as result of the link they will have with the producers of such products/brand. Thus, the harmonization of these policies can help reach a sustainable and inclusive solution**.** The government can ascertain this inclusive and sustainable solution through mandatory and government led, comprehensive financial responsibility , clear communication and training on EPR systems, and engagement of waste pickers.

CONCLUSION

Studies on solid waste characterization, health, perception, behaviour of the populace in relation to solid waste and sustainability are ubiquitous but the aspect of product packaging waste in Awka metropolis have not been extensively studied. Based on these prior studies, the residential locations have been more widely studied than other locations within Awka metropolis. The finding of present in concord with prior studies shows that the product package among other solid waste occurs in similar way in the residential locations. However, the situation of product packaging waste to other solid waste is not similar in other locations (i.e. market, schools, hospitals, municipal and industrial areas). This study recommends DFE not as a new development but a re-consideration of green design or multiple reuse product packaging as regards solid waste in selected areas in Awka metropolis. Green design should harmonize human being, nature/environment and the products being used in a symbiotic association. For purpose of this symbiotic association, reducing the solid waste in the environment will benefit everyone because waste will be minimized. This is also beneficial to everyone as enterprises can also use it as a means of advantage to help the acceptability of brand as it can become means for strong driving force in the market towards sustainable consumption pattern. This is because many stakeholders such as the manufactures will start to reduce waste disposal problems at the consumption and manufacturing level. Thus, policy approach under which producers are given a significant responsibility of waste prevention/diversion (.i.e. persuading the consumers through gainful or attractive means to return used product packages to the producers) of the post-consumer products should be ongoing through Private - Public Sector Partnerships (PPP). Companies producing these products can be held responsible for the disposal of the product by a particular fee and take up the responsibility of recycling every waste as result of their products' consumption. Improved or similar policy such as Deposit Refund System (DRS) can also be adopted . Situation where by producers persuade the consumers through gainful or attractive means to return used product packages can be encouraged. This should not stop the job opportunity of the waste picker/scavenger. Green design or multiple re-useable product packaging should be integrated to production or manufacturing cycle. The government should spread their tentacles to EPR harmonized with Private - Public Sector Partnerships (PPP) in order enhance the concern of everybody (manufactures, consumers, organizations and other entrepreneurs). Every foreign manufacturer, importer as well as local producers have to enter into agreement with the government concerning the waste prevention in as much as their products are been consumed in such region. Thus, present study unfolds green design or multiple reuse of product package as a means of attaining zero waste in Awka metropolis.

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CONFLICT OF INTEREST

The authors declare no potential conflicts of interest regarding the research, authorship and/or publication of this article.

DATA AVAILABILITY

The data used to support the findings of this study are included within the article.

AUTHOR'S CONTRIBUTIONS

All authors contributed equally to bring out this article.

ETHICS

There are no ethical issues with the publication of this manuscript.

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