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Properties of Gypsum Boards Made with Cedrus Tree (Cedrus *libani*) Components. Part 2. Chemical and Technological Properties

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Araştırma Makalesi

Abstract − In this study, the variation of cedrus tree parts and gypsum during experimental panel manufacturing have been evaluated. The burning pattern on the surface of all test boards produced by adding cedrus's tree components (wood, bark, cone and needle) as reinforcement fillers to the gypsum structure did not reach the threshold limit of 150 mm that specified in the standard value as ISO 11925-2 standard but only limited spreading of char was observed. It was also found that cone looks like create better barrier against heat compare to needle, wood and bark in similar proportions with gypsum. In contrary, although bark could be absorbed and barrier to heat better than others but it may not support flammability that are not support to mass lost when subject to burning. Some chemical changes occurred in main constituents of lignocellulosic substances in water/gypsum mixture as evidence with FTIR spectrums. It has also realized that bark (SKa_6), cone (Sko_6), and needle (SI_6) in gypsum negative impact on thermal degradation that higher temperature for decomposition compare to wood-based board (Ska_1) at similar experimental manufacturing conditions. It has clearly seen that content of fillers dramatically effects thermal stability of gypsum based boards.

Keywords − *Gypsum board, FTIR, thermal stability, burning properties*

Sedir Ağacının (*Cedrus libani*) Farklı Kısımlarından Üretilmiş Alçı Levhaların Özellikleri. 2. Bölüm. Kimyasal ve Teknolojik Özellikler

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Öz-Bu çalışmada, sedir ağacının değişik kısımları ile alçı karışımından elde edilen deneysel levhalar üretilmiştir. Yanma deneylerinde, bütün sedir ağaç kısımların (odun, kozalak, kabuk, ibre) alçı yapısına ilave edilmesiyle üretilmiş deneysel panellerin yüzey yanma durumları, as ISO 11925-2 standardına göre 150 mm lik seviyeye ulaşmamış, sadece sınırlı bir yüzey yanma yayılımı gözlemlenmiştir. Ayrıca sedir kozalağı diğer ibre, odun ve kabuk karışımından üretilen alçı esaslı levhalara göre daha fazla engel oluşturduğu anlaşılmıştır. Bu duruma karşı olarak ise, kabuk daha fazla ısıyı absorpladığı ve daha iyi engel oluşturduğu gözlemlenmekle birlikte, yanma esnasında kütle kaybı üzerine olumlu etkisi gözlemlenememiştir. Lignoselülozik maddelerin (odun, kabuk, ibre, kozalak) su/alçı karışımı içerisinde bazı kimyasal değişimlere uğradığı FTIR analizleri ile gözlemlenmiştir. Benzer koşullarda üretilen kabuk (SKa_6), kozalak (Sko_6) ve ibre (SI_6) ilavesinin alçı esaslı levhaların termal bozulması üzerine, odun esaslı levhalar göre (Ska_1) negatif etki ettiği belirlenmiştir. Çalışmada alçı yapısına ilave edilen katkı maddelerinin alçı esaslı levhaların termal dayanım özellikleri üzerine etki ettiği belirlenmiştir.

Anahtar Kelimeler − *Alçı levha, FTIR, termal dayanım, yanma özelliği*

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1.Introduction

Due to population growth and expanding rural areas, there is a need to develop a cost-effective and sustainable materials to utilize for building assemblies. However, the gypsum has been utilized as construction and ornamental materials throughout human beings. It is chemically known as *calcium sulfate dihydrate* ($CaSO_4.2H_2O$) which are obtained through mining. The paper-faced gypsum boards, is a well-known low-cost material and frequently used to finish interior wall and ceiling surfaces, have been widely used since the 1950s. (Cam, 2019; Shiroma et al. 2016; Van Elten, 1996). They are often called *drywall, wallboard*, or *plasterboard* (Van Elten, 1996; Youngquist, 1999).

However, one of the main drawbacks of gypsum board as a building material is its heaviness and brittleness. Hence, it does not have strong impact resistance for many applications. This drawback could be overcome by combining with various type natural fibers (wood, seconder fibers, annual plants) in order to improve mechanical and technological performances (Cam, 2019; Sahin et al.,2019; Sahin and Demir 2019; Herhández et. al. 1999).

For lignocellulosics, there is a general agreement that the inhibitory effect on mineral adhesive hydration is due to their kind and content of extractives. However, the influences of the extractives may not be just a reason of the absolute extractive content of a given material. It has already proposed that calcium sulfate dihydrate (gypsum) crystals are relatively long and have a hexagonal form while dimension of the crystals are altered under interactions of extractives (Simatupang and Geimer 1990).

Singh and Garg (1994) investigated natural and glass-fibre-reinforced boards based on gypsum plaster. They have discussed the ***β****-hemihydrate* plaster with high strength water-resistant gypsum binder made from phosphogypsum and the plasterboards/composites (sisal, coir and glass fibres). Hernández-Olivares and his friends (1999) proposed that cork and plaster are mutually compatible and cork–gypsum composite suggested for use in some building applications. Li and his group (2003) treating cotton stalk fiber surface with styrene acrylic emulsion to improve interfacial state of cotton stalk fiber/gypsum composite. Carvalho and his group (2008) produce a composite material made of gypsum reinforced with cellulose fibers from discarded Kraft cement bag. It has suggested that natural fibers absorb large amounts of water, causing the water/gypsum ratio of the paste to increase. However, this material is a technically better substitute for the brittle gypsum board, and it stands out particularly for its characteristics of high impact strength and high modulus of rupture. Sahin and his friends (2019) proposed that the addition of rice straw to the wood/gypsum mixture affects to extending hardening time while improve heat resistance (insulation) properties of panels at some level. However, post-consumer waste paper, old corrugated container (OCC) and secondary fiber addition (cellulosic additives) to gypsum in panel structure impact on the heat transmittance, were found to be lowered with addition of that lignocellulosic matters (Sahin and Demir, 2019).

It has already well predicted that the production of gypsum-biomass based products is very complex phenomenon. However, the approaches of quality development of lignocellulosic need to be carried out, along with a systematic method to measure the impact of reinforcement elements (cedrus tree components). Hence, a systematic investigation has been carried out with selected types of cedrus tree components (wood, bark, cone, needle) substrates to determine the effects on gypsum based composite manufacturing approaches and the chosen methods. The first part of this study, *"Properties of Gypsum-Based Boards Prepared with Cedrus Tree (Cedrus libani) Components. Part 1. Physical and Mechanical Properties"* have already been published in the Journal of Bartin Faculty of Forestry (Sahin and Cam, 2022). In the second part of this study, we seek to provide clear effects of the chemical and technological properties of gypsum based composite materials.

2. Material and Method

The reinforcement filler materials used in gypsum are cedrus tree *(Cedrus libani)* and its components of wood, bark, cones, and needles used in the study were collected from a local forest office in Isparta, Turkiye. All these raw materials were turned into particles through a hammer mill and screened. Particles remaining on the 2-3 mm were used in gypsum-based panel production. The particles were then dried at atmospheric conditions until air dried (12-15%) moisture content. Commercially available gypsum that carried TS EN 13279-1 B4/20/2 standard form as provided and without further processing was used. Six different type and a total of 62 (31x2) experimental gypsum-based boards were made. The detailed information on lignocellulosic matter, gypsum properties and experimental panel preperaing procedures could be found elsewehere (Cam, 2019; Sahin and Cam, 2022).

For determining ignitability of boards, a single flame combustion tests were carried out. Special type of flame combustion test system was built and conducted according to TS EN 13501-1 (Çam, 2019). For flame spreading, the system was conducted according to TS EN-ISO 11925-2 standard. The size of 90x250 mm pieces prepared from experimental boards and placed on the test apparatus at vertical position. Single-source small flame simulation on the edge of the bottom of samples applied at 45^{o} slope and distance of 20 mm from the middle of samples was initiated. The total test duration was 60 s. At the end of test, a visual observation of the sample was made and results recorded as positive or negative whether the specimen ignition occurs or not and the flame spreads in the vertical direction more than 150 mm above.

A Shimadzu (IR Prestige-21 series) FTIR spectrophotometer was used to evaluate the chemical groups present in the boards was made from selected samples. All FTIR measurements were performed under liquid nitrogen blanket. Data were collected in the 100-4000 cm^{-1} wavenumber region with 3 scans for each sample. Thermogravimetric Analysis (TGA) is carried out using a Perkin Elmer SII instrument in order to measure changes in properties of boards as a function of increasing temperature (with constant heating rate). Thermogravimetric Analyzer, supported by a PC and software for control and data handling. Approximately 20 mg sample is introduced into a quartz sample's pan and heated to a preset temperature profile using nitrogen as purge gas at a scanning rate of 10 ^{o}C/min. The data were collected in the 0-1000 ^{0}C.

3. Results and Discussions

A single flame combustion tests were carried out to determine burning propagation characteristic of the experimental panels (Figure 1). It was found that the burning pattern on the surface of all test boards produced by adding cedrus's tree components (wood, bark, cone and needle) as reinforcement elements to the gypsum structure did not reach the threshold limit of 150 mm that specified in the standard value as ISO 11925-2 standard (Fig.1 A). However, the reaction to fire with experimental boards also evaluated and it looks like only limited spreading of burn was observed (Fig.1 B). These results could be expected for gypsum-based boards that it is usually rated as A that a *non-flammable class* of material. Moreover, it is important to note that all reinforcement fillers used for experimental board in this study is known to be flammable that may result some level flamme behavior as realized in this study. But the test boards looks like still *a non-flammable class* (A) while flame did not reach the threshold limit of 150 mm (Fig. 1 A). In addition, these materials do not carry out any flame, even if the source of fire is removed after 5 min (Fig. 1 B).

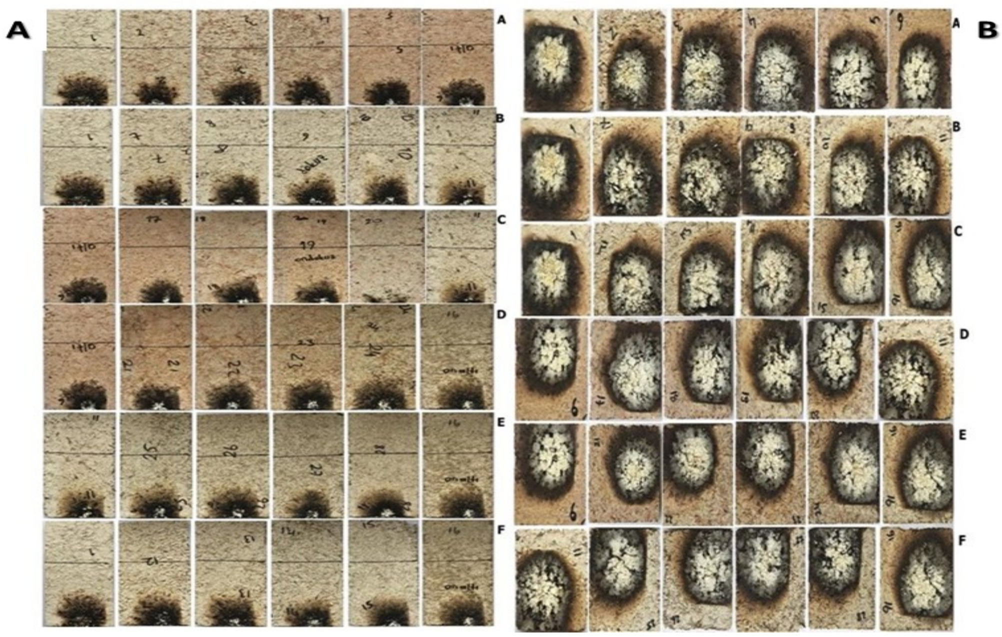

Figure 1. Flammability (A) and surface burning properties (B) of samples (A: SKa type boards; B: SKo type boards; C: SI type boards; D: KaKo type boards; E: KaI type boards; F: KoI type boards)

The heat transmission experiments are valuable in predicting insulance properties. In this sense, the heat-transmittance experiments were conducted at 5.0 min of duration and comparative properties of six different types gypsum-based boards are presented in Figure 2.

The samples presented various temperatures that the boards made with only cedrus wood/gypsum proportions show (SKa_1) 119.5 ºC heat in board surface. For Type 1 boards the highest heat value of 135.7 ºC was found with SKa_2 board followed by 132 ºC with SKa_4 and 130.5 ºC with SKa_6 boards, respectively. For Type 2 boards, only SKo_4 sample show 129.9 ºC heat value and all other samples show lowering heat transmission as cone particles increase in mixture. This is important considering cone looks like create better barrier against heat compare to bark in similar conditions. Like Type 2 boards, similar trend was also found for Type 3 boards that SI_3 show the highest heat transmission value of 141.7 ºC while other samples show smooth trend and marginally changing values. For samples prepared from cedrus bark/cone with gypsum (Type 4), increasing cone content show increasing level of heat transmission up to $KaKo_3$ sample then lowering with increasing cone in mixture. The highest heat transmission value of 143.8 ºC was measured with $KaKo_3$ sample while the lowest value of 117 ºC was found with $KaKo_6$ that only made with cone (without any other lignocelluolosic additives). The boards made with bark/needle proportion (Type 5) show interesting trend that increasing needle in proportions usually show lowering heat trasmission values. The lowest heat value of 119.2 ºC was found with KaI_6 boards that only made with bark (no needle additives). An opposite trend was observed with cedru's cone/needle proportions that increasing needle in mixture usually effects increasing heat transmission properties in all mixtures and conditions. The lowest heat transmission value of 117 ºC was found with SKo_6 board that only made with cone (without needle) while the highest heat transmission value of 127.7 ºC was found with KoI_5 boards that highest amount of needle (400 gr) while the lowest proportions of cone (100 gr) in mixture.

However, the results clearly indicate that the cone particles from cedrus tree have positive impact on thermal insulation properties that increasing its ratio lowering effects on thermal transmittance properties some level. Similarly, needle show better barrier against heat transmittance properties than bark and marginally better than wood. It could be ordered as properties of cedru's tree components (when used 100% in gypsum) for heat transmittance properties in experimental boards in that order of; cone (117 ºC) < needle (119.2 ºC) < wood (119.5 ºC) < bark (130.5 ºC).

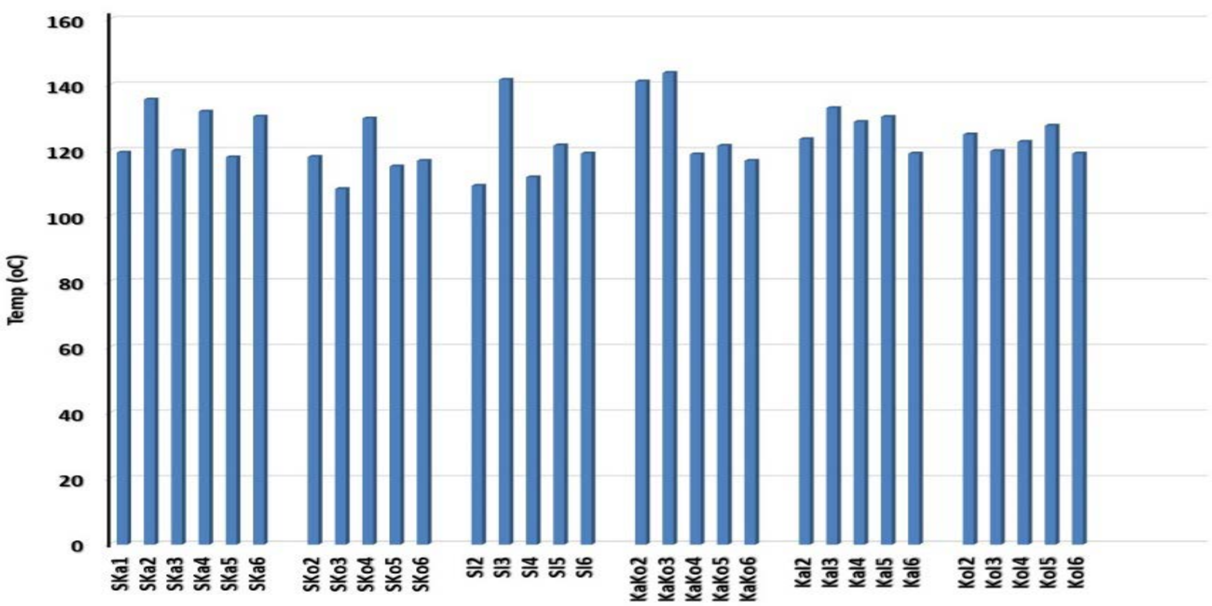

Figure 2. The heat trasmission values of boards

The weight loss of boards after mass burning test is shown in Figure 3. The lowest mass lost of 8.47% was calculated with SKa_2 while the highest mass lost of 24.59% was found with SI_3 board. It is very difficult to correlate all reinforce fillers effect on weight lost properties after 5.0 min. burning tests. However, some interesting results was found. It was observed that increasing cone and needle in wood proportions (Type 2 and Type 3 boards) usually effects increasing weight loss. Similar trend were also observed for Type 4 and 5 boards that increasing cone in bark and needle in bark proportions were also showed increasing weight lost. In contrast, for cone/needle based gypsum boards (Type 6), increasing needle content in cone proportions lowering effects on weight lost of boards. This is a clear evidence for bark and needle that they not support char spreading properties of experimental boards manufactured from various proportions of cedru's tree components.

It could be ordered as mass lost properties of cedru's tree components (when used 100% in gypsum) for burning properties in experimental boards in that order of; bark (11.01%) <needle (13.06%) <wood (13.36%) <cone (18.73%). It should be noted that although cedrus's bark effect lowering mass lost properties followed by needle, wood and cone, there is inverse correlation was observed for heat transmittance properties of boards (Fig.2). With having these informations, it could be hypotheses that heat transmittance and mas lost are independently occur during burning process, and they may not correlate to each other. It could be explained that bark could be absorbed and transfer to heat better than others in gypsum board structure. But it may not support flammability that are not support to mass lost when subject to burning.

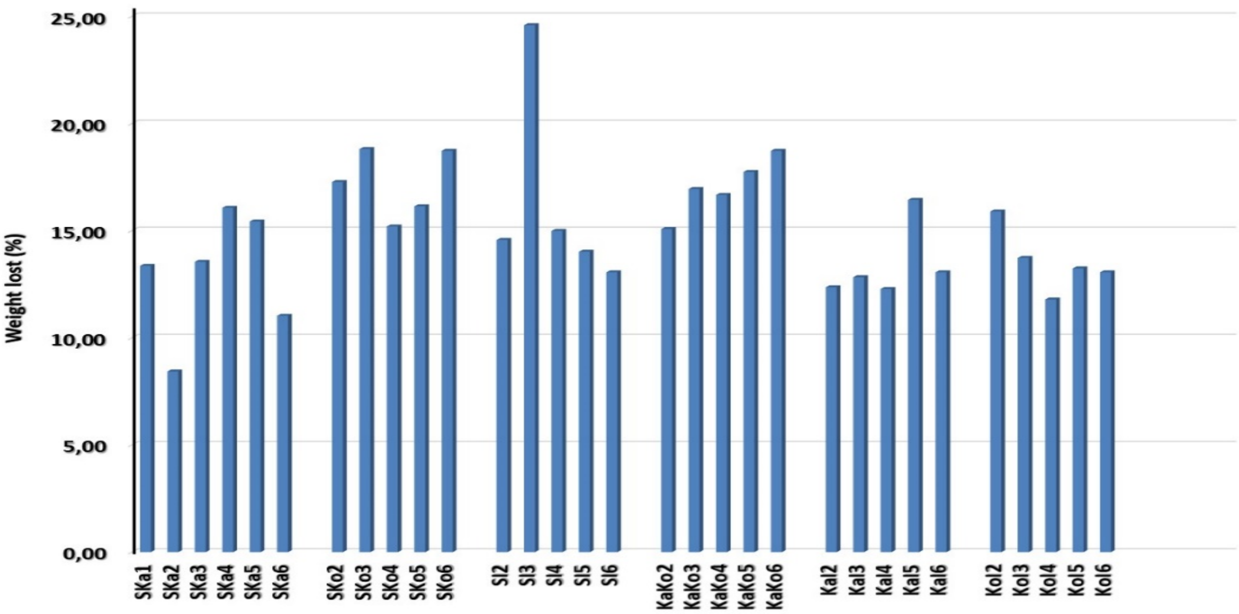

Figure 3. Weight lost properties of boards after 5 min burning tests

The comparative FTIR spectra of selected experimental boards are shown in Figure 4 (a-g). The FTIR spectra were obtained in range of 500–3600 cm^{-1}. However, the characteristic spectrum of lignocellulosics and gypsum concentrate in the range of 800–3400 cm^{-1} and the major peaks in this range had been identified. As seen in Figure 4, all spectra exhibit multi-modal absorption in the 600-1000 cm^{-1} region due to plenty of –OH groups in lignocellulosics (cellulose, hemicellulose and lignin). However, the out-of-plane C-H vibration was assigned at 750 cm^{-1}. The band at 900-1200 cm^{-1} is attributed to C-C out of the plane stretching, C-C-O stretching at 1080 cm^{-1}; C-O-C symmetric stretching 1150 cm^{-1} C=O stretching in 1745 cm^{-1} band, -CH_3 asymmetric stretching in 1450 and 1360 cm^{-1} band, symmetric stretching peak in 2947 cm^{-1} band identified (Chieng et al., 2013). It has proposed that characteristic -CH stretch bands from methylene and methyl can be seen in the range of 2800-3000 cm^{-1}. O-H stretching, which can come from cellulose groups in the range of 3200-3600 cm^{-1} or from water on the fiber surface, can be seen (Dink et al., 2016; Iorio et al., 2018).

Al Dabbas and his friends (2014) conducted research to determine FTIR spectra of pure gypsum. They proposed that gypsum has a strong band around 1425 cm^{-1}, characteristics of the C–O stretching mode of carbonate together with a narrow band around 875 cm^{-1} of the bending mode. They also suggested that the stretching vibrations of the H_2O molecules in the gypsum occur at 3540 and 3400 cm^{-1} while a strong band around 1128 cm^{-1} which splits into two components at around 1141 and 1116 cm^{-1} are assigned to the stretching and bending modes of sulfate. In this respect, gypsum ($CaSO_4.2H_2O$) and lignocellulosic substrates has usually show similar functional groups at similar band. Moreover, the less intense vibrations at 1450-1700 cm^{-1} and C-C and C-O-C peak areas (Fig. 4 c, d, g) indicates the generation of new surface chemistry which is related to gypsum induced/dried chain modification.

When we look at the FTIR spectra of samples, C-H stresses in the 2800-3400 cm^{-1} band are noticeable in variations using gypsum. The reason for this may be the gypsum being grafted to lignocellulosic substartes. It has already well-established that drying affects lowering distance between microfibrils and cross-links in cellulose structure (Sahin and Arslan 2008). This modification can be effects level of H-bonds in cellulose. The result found with FTIR evaluation support this information. On the basis of FTIR measurements it was concluded that some modification had taken place during solvent diffusion and solvent induced chain modification occurred in main constituents of lignocellulosic substances (wood, bark, cone, and needle).

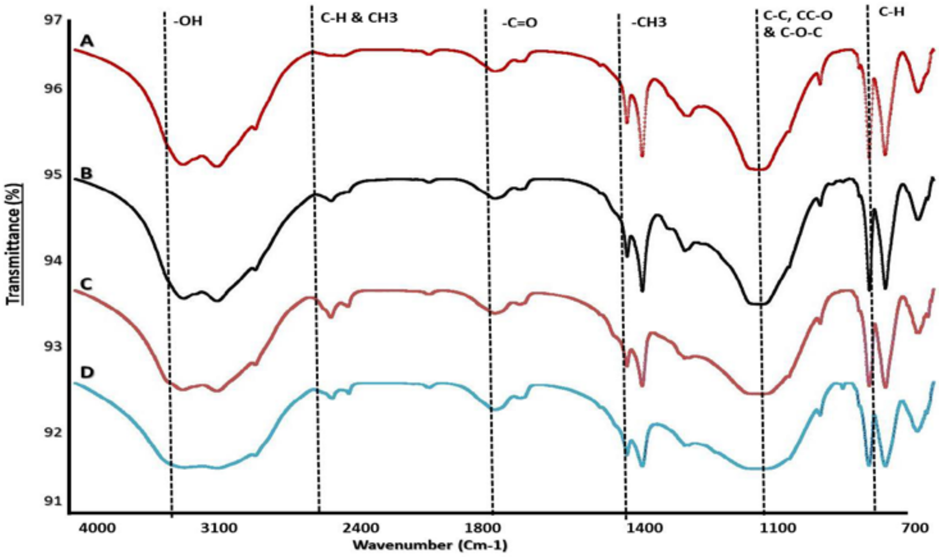

Figure 4. FTIR spectra of samples (A: SKa_1; B: SKa_6; C: SKO_6; D: SI_6)

One way to understand the thermal breakdown properties in the interaction between constituents of gypsum-based boards and heat, which could be the basis determination of Thermo Gramatic Analyssis (TGA) under controlled heating. Figure 5 show the comparative thermographs of selected samples. In recent studies, it has already well predicted that the TGA migrographs of cellulose-based materials including gypsum based composite materials typically show four different heating regions and better to evaluate these materials within that regions (Cam, 2019; Demir, 2019; Sahin et al., 2019; Kaya and Sahin 2016). In this regard, the evaluation of TGA spectra have been made within these suggestions.

According to Figure 5, the boards started to degrade at 160-180 °C and began to decompose at 370-390 °C. However, the mass loss of 80-100 °C in samples is thought to be due to water loss and started to degrade due to hemicellulose at 180 °C. However, all samples began to considerably decompose at a temperature above 400 °C. It could be suggested that all organic compounds (cellulose, lignin, hemicellulose) completely break down up to 700 ^{o}C. Moreover, it can be realized that bark (B: SKa_6), cone (C: Sko_6), and needle (D: SI_6) in gypsum looks like negative impact on thermal degradation that higher temperature for decomposition compare to wood based board (A: Ska_1) at similar experimental manufacturing conditions.

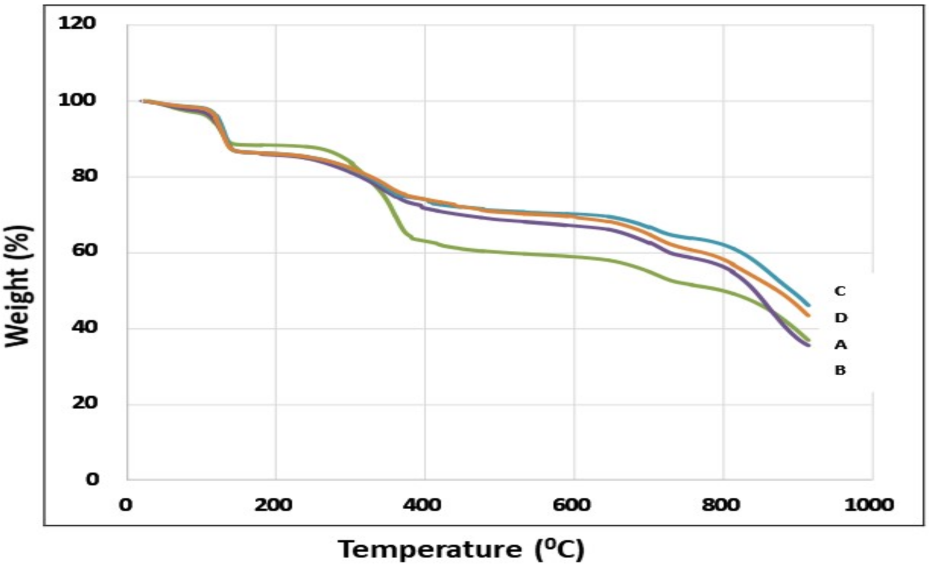

Figure 5. TGA spectra of samples (A: Ska_1; B: SKa_6; C: Sko_6; D: SI_6)

The TGA micrographs has quantitatively evaluated and results are comparatively shown in Figure 6. *In heating zone (Tb; 78-84 °C);* the water and some volatile compounds assumed to remove, cedrus's wood/bark proportions not influenced in this region that only 2.1% and 1.99% mass loss found for SKa_1 and SKa_6 samples, respectively. But boards made with cone and needle have dramatically effects on mass lost that 15.74% was found for SKo_6 and 14.38% for SI_6 samples, respectively. *In cell wall degradation zone (Tm1; 124-129 °C);* similar results observed as Tb zone that boards made from only cone-gypsum (SKo_6) and needle-gypsum (SI_6) instead bark (SKa_6) shows considerable higher mass lost values. The highest mass loss of 21.32% found for SKO_6 sample, followed by board with 21.32% mass lost found for SI_6 sample of 30.06%. *In cell wall organic constituents completely break down zone (Tm2; 358-438 °C);* More less similar trend was observed as Tb and Tm1 zone that boards made from only cone (SKO_6) and needle (SI_6) show higher mass lost then board made from wood/bark and only bark with gypsum proportions (SKa_1 and SKa_6). In this respect, the mass lost of samples were found to be 29.57% for SKa_1; 27.28% for SKa_6; 27.18% for SKo_6 and 29.11% for SI_6 samples, respectively. The degradation in this temperature range is thought to belong to the lignin in the structure of the additives. In all samples, the rate of mass loss decreased around 365 °C, and after this temperature, composites with cone and needle supplements gave neck in the temperature range of 365-500 °C. The reason for this is thought to be the lignin that is different behavior under heat than hemicellulose and cellulose. Because it is a mixture of 3 types of benzene-propane units with high molecular weight and cross-links. The thermal stability of lignin is therefore very high and difficult to separate (Fengel and Wegener, 1984; Sjostrom 1981). *In non-organics degraded zone (Ts; 537-622 °C);* The highest mass lost (40.45%) was calculated for SKa_1 sample, followed by SI_6 (34.06%), SKo_6 (29.77%) and SKa_6 (33.08%), respectively. It is clear that the inorganic gypsum has started to degrade above 400 °C. As can be seen from these values, the cedru's cone and needle based gypsum boards show high level of degradation at all temperatuve levels whereas cedru's wood and bark based gypsum boards show very low decomposition level initially and then it increase as temperatures increases. This is also a clear evidence that the content of fillers dramatically effects thermal stability of gypsum based boards.

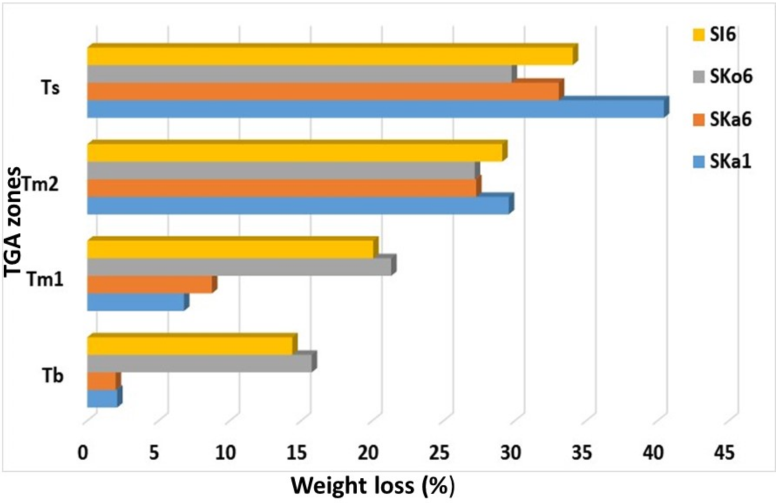

Figure 6. Thermal decomposition of experimental boards (0-622 ^{o}C)

4. Conclusions

The gypsum-based boards have some drawbacks that heavy and brittle material in many applications. In this respect, numerous studies have conducted for determining the suitable configurations on the end use applications. But many properties such as; strength, physical, and insulation are primarily influenced by the density and the binder (gypsum)/non-gypsum ratio in network structure. However, the chemical and physical properties could be a direct function of the interface bonding in gypsum that are greatly affected by the type, dimensions, geometry and arrangement of the reinforcement (filler) elements. Although the properties of gypsum-based panels could be influenced by the processing variables and reinforcement elements, the low value wood components may create some opportunities to match product performance to end-use requirements. It could be suggested to prepare sustainable panel products from gypsum-lignocellulosic mixtures which helps to utilize renewable sources for non-strength required purposes (i.e. thermal insulation).

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