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PRODUCTION OF PURE PVC, GRAPHENE AND CARBON NANOTUBE-DOPED PVC ELECTROSPUN NANOFIBER MEMBRANES: A HIGHLY EFFECTIVE METHOD FOR REMOVING ACID BLUE 74 DYE FROM WASTEWATER

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Abstract: Dyeing wastewater is produced by various industries, including textiles, cosmetics, tanning, and plastics. Annually, about 1.6 million tons of dyes are manufactured, with 10-15% released into the environment as wastewater. Organic dyes are particularly harmful pollutants, even at low concentrations. The adsorption technique has proven effective for removing these pollutants from water, significantly relying on the properties and efficiency of the adsorbent. Nanofibers are emerging as promising adsorbents for dye removal due to their unique properties and high efficiency. This research focuses on polymeric nanofibers to address global dye pollution, as they effectively eliminate various dyes and contaminants. However, their mechanical properties can be a limitation under harsh conditions. To enhance their strength and hydrophilicity, incorporating nanofillers like carbon nanotubes and graphene oxide has shown significant benefits. This study examines the impact of different nanofibers on the adsorption of Acid Blue 74 (AB74) dye. Pure, carbon nanotube-doped, and graphene-doped PVC nanofibers were produced via electrospinning. Their properties were analyzed using FTIR and SEM, while key parameters such as contact time, adsorbent dosage, dye concentration, and pH were assessed to understand the adsorption behavior. Additionally, dye adsorption isotherms and kinetics were investigated.

Keywords: Nanofibers, Wastewater treatment, Electrospinning, Dyes, Adsorption

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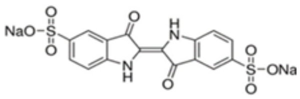
1. Introduction

Textile industries utilize large amounts of dyes to provide distinctive textures to fabric materials. Approximately 10% of the colors used in textile processes are discharged into wastewater. Wastewater from textile manufacturing facilities is recognized as one of the most polluting among various industries due to its significant content of hazardous waste. Treating contaminated water, such as non-industrial waste, is an effective method for recycling lost water (water treatment) (Mo et al., 2008). Various conventional technologies are employed to remove different pollutants (inorganic, organic, biological, and radiological) from aqueous solutions (Kaczyk et al., 2020). These technologies include ion exchange, chemical oxidation, reduction, precipitation, biological, electrodialysis, (Altıntaş et al., 2023) coagulation, electrocoagulation, flocculation, liquid-liquid extraction, evaporation (Fadlalla et al., 2020), phytoremediation, filtration/adsorption, reverse osmosis, and ultra-, nano-, or micro-filtration (Kurniawan et al., 2006; Ayodhya and Veerabhadram, 2018).

In the textile industry, Acid Blue 74 (AB74) is one of the most commonly used dyes for dyeing wool and silk (Ghanavati et al., 2021). This dye is highly toxic and carcinogenic. Exposure to AB74 can cause skin and eye irritation in humans (Li et al., 2012). Acid Blue 74, also known as Indigo Carmine or Indigotine, is a synthetic dye widely used in textiles, food, and the pharmaceutical industry. It is derived from indigo and is recognized for its vibrant color and stability. In environmental science, indigo carmine is often studied as a model pollutant due to its widespread use and potential impact on water quality. Its removal from wastewater is a significant focus in developing effective nano adsorbents and filtration techniques (Wang Q et al., 2019; Wang W et al., 2019; Wang et al., 2015). Below is an exploration of its properties, including its chemical structure, uses, and environmental impact in wastewater.

Acid Blue 74 (AB74) is a sulfonated derivative of indigo. It consists of a central indole structure with two aromatic rings linked by a double bond between two carbon atoms. The sulfonic acid group (-SO_3H) makes it water-soluble and allows it to dissociate in aqueous solutions (Attia et al.,, 2006). The presence of the sulfonate group enhances its solubility compared to the original indigo dye, making it suitable for various applications. Textiles are traditionally used in dyeing fabrics, especially in textile industries, for their vibrant blue color and in food and beverages (Rehorek et al., 2004). It is employed as a food colorant (E132) for various products, giving them an appealing blue hue. In medicinal, it is sometimes used in pharmaceutical formulations and as a color indicator in biological studies (Yagub et al., 2014). In cosmetics, found in personal care products for coloring purposes. By electrospinning technique, it is possible to produce PVC nanofiber membrane material with high specific surface area and high porosity, which is useful for adsorption and filtration applications (Wang et al., 2022).

Table 1. Characteristic properties of Acid Blue 74 (AB74) dyes

Properties of Dye	Acid Blue 74 (AB74)
Common name	Indigo Carmine
Color index number	73015
Chemical name(IUPAC)	Disodyum [2(2′)E]-3,3′-diokso-1,1′ ,3,3′ -tetrahidro [2,2′-biindoliliden]-5,5′-disülfonat
Molecular weight	466.35 g mol^{-1}
Chemical formula $C_{16}H_8N_2Na_2O_8S_2$	
Suitablefor detection wavelength	650 nm

The interaction of AB74 dye with nano adsorbents is governed by multiple adsorption mechanisms, primarily involving physisorption and chemisorption (Ahmed and Lalia, 2015). Key factors like pH, temperature, initial dye concentration, contact time, and the specific properties of the nano adsorbents play crucial roles in determining the extent and efficiency of adsorption. For effective wastewater treatment solutions, it's essential to optimize these parameters and tailor nano adsorbent materials to effectively capture and remove contaminants like AB74 dye. Adding graphene or carbon nanotubes can significantly enhance the adsorption properties of PVC. These materials possess high surface areas and can provide additional adsorption sites. They may also improve mechanical properties and electrical conductivity. In this study the AB74 dye adsorption of the synthesized nanofibers was described exploring the influence of several parameters, including contact time, adsorbent dosage, dye concentration, and pH of solution.

2. Materials and Methods

2.1. Materials

The PVC used in the study was supplied by Sigma-Aldrich; its viscosity is 0.80 dL / g, density is 1.4 g / mL (at 25 °C), and molecular weight is Mw = ~80,000. The supplied PVC was dissolved in a mixture of THF and DMF prepared in a weight ratio of (1:1) to 15% by weight. Indigoid dye AB74 (Mw = 466 g mol^{-1}) was obtained from Sigma–Aldrich as a commercially available dye (85%) and used without further purification (Briesemeister et al., 2024).

2.2.Methods

2.2.1. Nanofiber production

The polymer solution was placed into 10 mL syringes in the pump unit. Nanofibers were produced at 27 kV voltage and 0.6 mL/h feed rate. During production, the ambient temperature was 25 °C on average, and the relative humidity was between 50%-60%. The distance between the syringe tip and the drum was 24 cm (Xue et al., 2019). Under the given conditions, the nanofiber sheets obtained were kept at ambient temperature for 3 days so that the solvent that might remain on them could evaporate. The thickness of the nanofiber sheets obtained varied between 0.004-0.005 mm. Three different types of PVC nanofiber sheets were produced. Three different products were obtained pure PVC nanofiber sheets, 2% carbon nanotube-doped PVC nanofiber sheets, and 2% graphene-doped PVC nanofiber sheets. For the PVC solution to be as homogeneous as possible, the solution was first placed in the magnetic stirrer for about half an hour until the solid particles dissolved and then kept in an ultrasonic bath for 1 hour (Ozkan et al., 2019). For our study, the electrospinning production method was preferred among nanofiber production methods. The electrospinning device used in production is given in Figure 1.

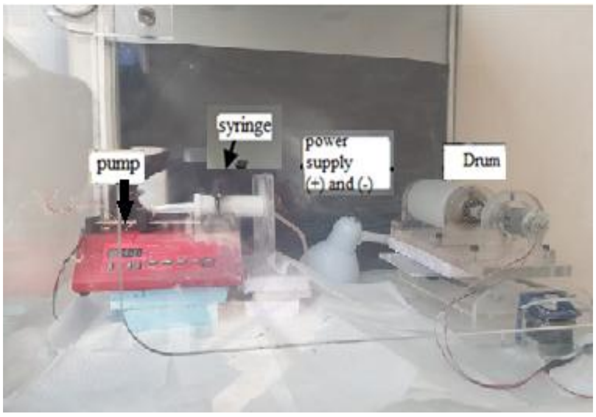


Figure 1. Electrospinning device used in nanofiber production.

2.2.2. Characterization of adsorbents

The FTIR spectra of untreated pure PVC nanofibers membranes, PVC/CNT nanofiber membranes, and PVC/G nanofiber membranes were analyzed using a PerkinElmer 1600 Fourier Transform Infrared (FTIR) spectrometer, covering a range from 4000 to 400 cm^{-1}. The surface morphology and topography of the electrospun nanofibers were examined with a Jeol JSM-6335F scanning electron microscope (Wang et al., 2015;Wang et al., 2016). Infrared (IR) spectra were also collected during the study. To determine the thermal properties of the produced nanofibers, DSC analyses were carried out in the presence of nitrogen gas (N_2) in the Perkin Elmer DSC-4000 model Differential Scanning Caliper (DSC) device at a heating rate of 20 °C min^{-1} and the range of 30-400 °C (Ozkan, 2019)

2.2.3. Adsorption tests

This study obtained three different adsorbents by electrospinning with PVC on different nanofiber membranes. The adsorption performance of all three adsorbents in the aqueous solution and the samples was tested. As the adsorbate, AB74, a basic anionic dye, was used to represent the total dyes in the adsorption studies. To evaluate the adsorptive removal capacity of PVC nanofiber membranes for the AB74 dye, 0.030 g of the adsorbents were weighed, mixed with 25 mL of AB74 solutions between 50 and 500 mg/L, and treated separately until equilibrium was reached for ten hours. Each flask was shaken in a Nuve ST 402 shaking water bath at a constant speed of 150 rpm. Then, adsorbent particles and adsorbate molecules were separated with the help of a vacuum filtration device. The concentrations of AB74 remaining in the filtrate were determined at 650 nm in a UV-VIS spectrophotometer in the mg/L concentration unit. The results were then converted to mg/g by Formula (1) and % by Formula (2).

$$Q_e\ (mg/g) = \frac{C_o - C_e}{m} \quad (1)$$

$$Q_e\ (\%) = \frac{C_o - C_e}{C_o} \times 100 \quad (2)$$

Where Qe (mg/g) and Qe (%) mean the amount of AB74 molecules adsorbed on the adsorbent at equilibrium, Co and Ce represent the initial and equilibrium AB74 concentrations (mg/L), respectively. m is the adsorbent mass (g).

The experiments were conducted at three different temperatures: 5 °C, 15 °C, 25 °C, 35 °C, and 50 °C, to assess the effect of temperature. Additionally, the pH of the dye solutions was varied between 2 and 12 to investigate the impact of pH, with the shaking duration set at 10 hours. After filtering the dye solutions, absorbance values were measured at 650 nm. The measured absorbance data were then converted to concentration data using a calibration graph specific to the dye.

3. Results and Discussion

3.1. Results of Characterization Analysis of Adsorbents

3.1.1. SEM analysis

SEM analysis is a crucial tool for characterizing the surface morphology and physical properties of adsorbent materials. It helps determine the particle shape, porosity, and size distribution of the adsorbent. Additionally, nanofibers with high porosity exhibit superior dye removal capabilities due to their increased surface area and greater availability of adsorption sites.

In the study, SEM images of PVC, carbon nanotube-doped PVC, and graphene-doped PVC nanofibers obtained by electrospinning were obtained with the ZEISS GeminiSEM brand SEM device, which is shown in Figure 3. (Zakaria, 2021).

Figure 2. Nanofiber membranes obtained from PVC polymer, a) Pure PVC Nanofiber membranes, b) 2% carbon nanotube-doped PVC nanofiber sheets, c) 2% graphene-doped PVC nanofiber sheets.

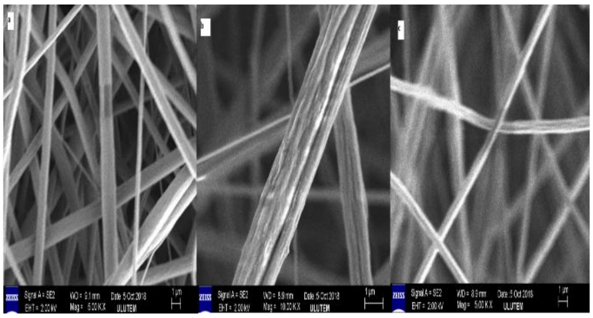


Figure 3. SEM Analysis a) PVC b) PVC-CNT c) PVC-G nanofibers.

The random distribution of nanofibers of all examined membranes is seen in the SEM image in Figure 3. The SEM images also show that all of the nanofibers produced are obtained in a droplet-free and homogeneous distribution (Ray et al., 2016).

3.1.2. FTIR analysis

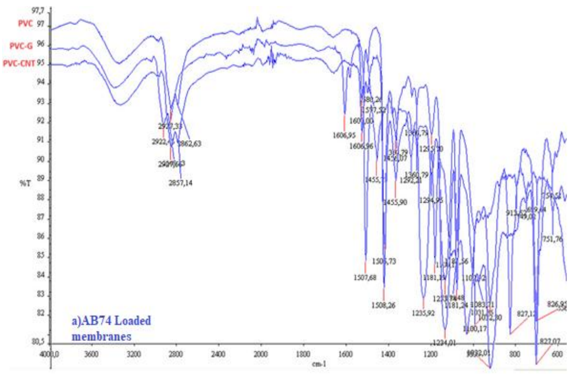


Figure 4. a) IR spectra for AB74 loaded nanofiber membranes.

The spectra of AB74 and AB74 loaded PVC, PVC/CNT, and PVC/G in Figure 4 are similar. Since AB74 covers the surface, the spectrum of AB74-loaded adsorbents is similar to the original AB74 spectrum. The adsorption capacity of adsorbents for AB74 is high. The shift in permeability can explain the formation of similar spectra. After AB74 dye adsorption, first of all, it is seen that the relative permeability (T) percentages increase compared to the original adsorbents.

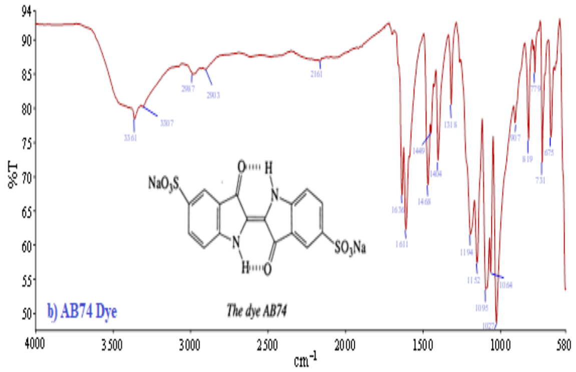


Figure 4. b) Spectrum of original AB74 dye.

The increase in permeability is a natural result resulting from the occupation of functional groups by dye molecules. The spectra taken after adsorption show that PVC nanofiber membrane has extra peaks compared to other adsorbents, and some peaks have shifts in wavelength values. The most significant change occurs after adsorption with PVC/G nanofiber membrane. When the IR spectra of the AB 74 dye given in Figure 4 are examined, typical AB74 peaks appear between 1517–1623 cm^{-1} due to the surface coating feature. This situation is also seen in the spectra of many other adsorbents taken after AB74 adsorption. In the IR spectra taken after dye adsorption for membranes, extra peaks consisting of hydroxyl groups are seen around 2850 cm^{-1} compared to the pure PVC. However, since the groups characterizing the adsorbents are generally the peaks that appear toward the right side of the spectrum, this situation does not constitute a problem (Zakaria, 2021).

3.1.3. TGA/DSC analysis

Thermal analysis measurements were performed with TGA Shimadzu, a TGA-50 device (thermogravimetric analysis) from room temperature to 400 °C (at a heating rate of 10 °C/min), and with DSC Shimadzu, a DSC-50 device (differential scanning calorimeter) from room temperature to 200 °C (at a heating temperature of 20 °C/min) under a dynamic nitrogen atmosphere (10 mL/min low flow rate).

Figure 5 shows DSC graphs of pure, carbon nanotube-doped, and graphene-doped PVC nanofiber membranes. The DSC curves of the samples are given in graphs and interpreted comparatively. Accordingly, they showed exothermic behavior. The glass transition temperature is high for PVC nanofibers.

As seen in Table 2, the highest glass transition temperature belongs to the CNT-doped PVC-nanofiber membrane, while the lowest belongs to the pure PVC-nanofiber membrane. The peak length is the longest for the pure PVC-nanofiber membrane, which shows that it has the lowest exothermic reaction rate among all materials.

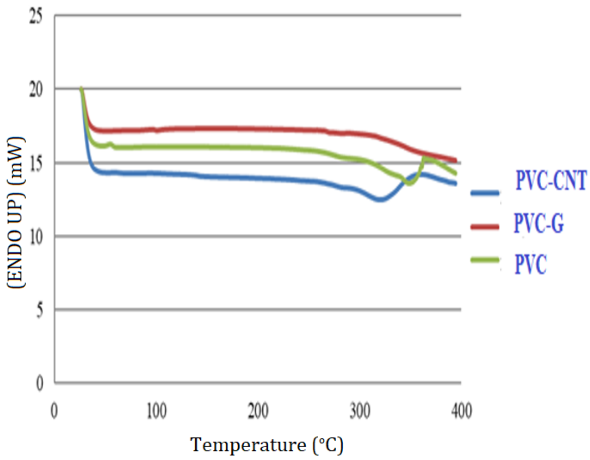


Figure 5. DSC graphs of pure, carbon nanotube-doped, and graphene-doped PVC nanofiber membranes.

Table 2. DSC test results of the produced nanofiber membranes

	PVC-CNT	PVC-G	Pure PVC
T_g	95 °C	94 °C	51 °C
T_{on}	288 °C	267 °C	321 °C
T_{max}	322 °C	270 °C	348 °C
ΔH	-165.8641 J/g	-2.0228 J/g	-174.9227 J/g
P_H	1.4048 mW	0.0677 mW	1.7277 mW
T_{off}	348 °C	258 °C	362 °C

3.2.Results of Adsorption Of AB 74 Dyes on Nanofiber Membranes

3.2.1. Effect of pH on adsorption of AB 74 dye on PVC nanofibers

250 mg/L batch dye solution with pH adjusted between 2-12 was prepared and treated with 3.0 g/L adsorbent for 10 hours. After filtration, the remaining dye concentrations in the solution without being adsorbed were determined by UV-VİS spectrophotometer. According to Figure 6, it has been determined that there is a possibility of working in a wide pH range, especially for AB74 adsorption. Although it was determined that acidic conditions were suitable for AB 74, pH five would be more suitable. As seen in Figure 6, the highest adsorption capacity (qt) was obtained at pH =5 and 43.57 with a CNT-doped PVC nanofiber membrane. However, the decrease in adsorption capacity with increasing pH value to 8 is related to the decrease in dye adsorption due to the competitive effect of OH- and anionic dye to be adsorbed on the solid surface. The wide adaptation of the surface and pore structure of the copolymer particles to HR molecules can be considered the main factor in removing dyes (Ahmed et al., 2017).

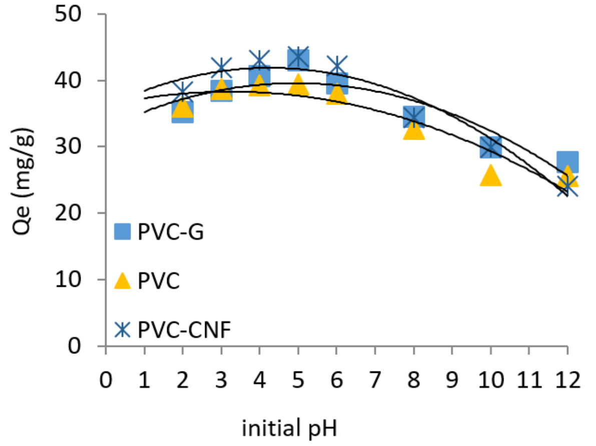


Figure 6. Effect of initial solution pH on adsorption of AB74 (Initial dye concentration: 250 mg/L, ads. dose: 3.0 g/L, agitation time: 10 h).

3.2.2. Effect of temperature on adsorption of AB 74 dyes and adsorption ısotherms

B250 mg/L dyestuff solutions were treated with 3.0 g/L adsorbents at room temperature for 10 hours. The concentrations of dyestuff remaining without adsorbing in the solution were determined by UV-VİS spectrophotometer. That is, the adsorption of dyestuffs on the adsorbents occurs spontaneously and voluntarily (Figure 7).

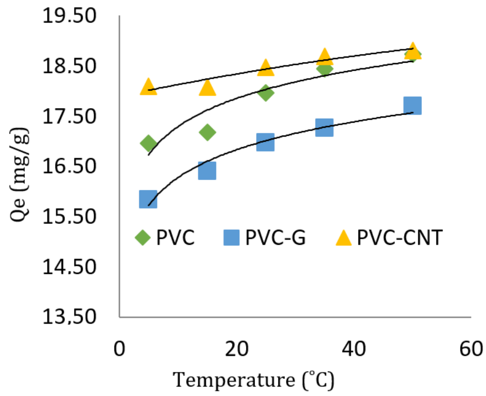


Figure 7. Effect of temperature (Initial dye conc.: 250 mg/L, pH: 5.0, ads. dose: 3,00 g/L).

The adsorption equilibrium isotherm is important because it gives a clear clue about the distribution pattern of dye molecules between the liquid and solid phases when the adsorption process reaches an equilibrium state. The isotherm is vital for the design of adsorption systems, and its shape can explain the homogeneity and heterogeneity of the adsorbent surface. The equilibrium data of dyes were analyzed by fitting them to the Langmuir equations to find the appropriate model to describe the adsorption mechanism. Langmuir isotherm equation 3 is;

$$\frac{C_e}{q_e} = \frac{C_e}{q_{max}} + \frac{1}{bq_{max}} \quad (3)$$

Table 3. Adsorption constants obtained according to the Langmuir model in the adsorption of AB 74 onto PVC nanofibers

	Qmaks(mg/g)	*b*(L/mg)	slope	R^2
PVC	39.42	0.0107	0.0136	0.999
PVC-G	40.26	0.0095	0.0112	0.996
PVC-CNT	43.57	0.0109	0.0121	0.997

3.2.3. Effect of contact time on adsorption of dyes

The time dependence of the adsorption process is called adsorption kinetics. The equilibrium time for AB 74 dye adsorption on PVC nanofibers was determined in the 0–12 h range. For this purpose, a series of dye solutions containing 500 mg/L and pHs between 4–6 were treated with 3.0 g/L adsorbents at different intervals. After the mixtures taken at different time intervals were filtered, the AB 74 dye remaining in the filtrate was determined by UV-GB at 630 nm wavelength (Bozbeyoğlu and Gündoğdu, 2023). Dye adsorption (%) graphs were drawn against shaking time (min) from the results.

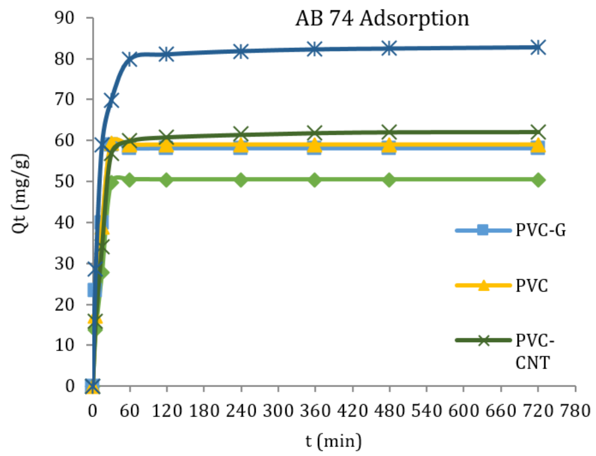


Figure 8. Changes in dye adsorption over time.

Figure 8 shows a sharp rise in the dye concentration during the 60-minute contact time due to the adsorption of anionic dye onto the negatively charged PVC nanofiber membrane material via electrostatic attraction forces. After 120 min, no significant change in the dye concentration was observed for the membrane material AB 74 system.

3.2.4. Desorption and reuse experiments

Acetone, ethanol, NaCl, and HCl were used as eluents to investigate the reusability of PVC nanofiber membrane material. Before the desorption experiment, AB 74 dye (25 mL, 3.5×10-5 mol.L^{-1}) was loaded onto 0.0030 g of membrane material for 10 h. Then, AB74-loaded PVC nanofiber membrane materials were separately transferred to Erlenmeyer flasks containing 25 mL of ethyl alcohol, 0,1 M HCl, 0,1 M NaCl, and acetone and shaken at 25 °C for 10 h. Desorbed AB 74 concentrations were measured using the spectrophotometric method, and the dye desorption efficiency of the spent membrane material was calculated. The desorption studies, shown in Figure 9, showed that the desorption performance of acetone and ethanol used in a 1:1 ratio was better than that of NaCl and HCl (Gundogdu, 2022).

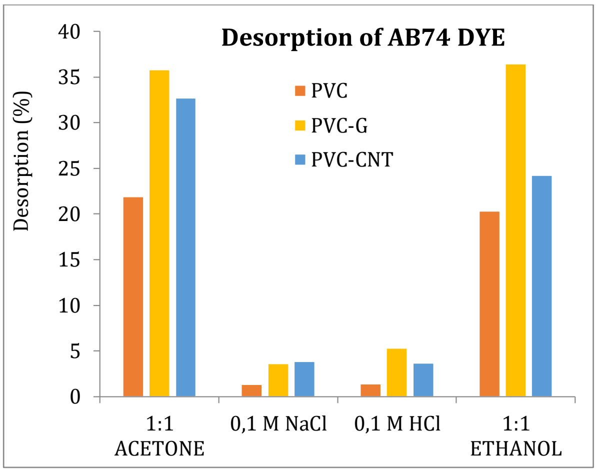


Figure 9. Desorption studies.

4. Conclusion

AB 74 dye is a widely used synthetic dye with important applications across industries, but its environmental implications require careful management. Effective waste treatment solutions to minimize the discharge of indigo carmine into water systems are critical to protecting aquatic ecosystems and ensuring water quality.

- In the studies, suitable conditions for factors such as pH, temperature, and time's effect on AB 74 dye solutions were determined using a model solution, and dye removal from wastewater was carried out.
- It was determined that the optimum pH range for AB 74 dyes was around five and that the shaking time increased the adsorption capacity to 60 minutes, reaching equilibrium after 60 minutes.
- It was determined that the adsorption capacity of pure PVC nanofiber membrane was lower than that of graphene-doped PVC nanofiber membrane, and the adsorbent with the best adsorption capacity belonged to the carbon nanotube-doped PVC nanofiber membrane.
- When the equilibrium data of AB 74 dye were examined, the Langmuir isotherm model was determined to be the most suitable model with a high R^2 value for describing the adsorption mechanism.

Author Contributions

The percentages of the authors' contributions are presented below. All authors reviewed and approved the final version of the manuscript.

	V.Ö.	P.B
C	50	50
D	50	50
S	50	50
DCP	50	50
DAI	50	50
L	50	50
W	50	50
CR	50	50
SR	50	50
PM	50	50
FA	50	50

C=Concept, D= design, S= supervision, DCP= data collection and/or processing, DAI= data analysis and/or interpretation, L= literature search, W= writing, CR= critical review, SR= submission and revision, PM= project management, FA= funding acquisition.

Conflict of Interest

The authors declared that there is no conflict of interest.

Ethical Consideration

Ethics committee approval was not required for this study because of there was no study on animals or humans.

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