




## Research Article

# Investigation of the retention of oils mixed with water by forming activated carbon added nanofibers

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## Abstract

Heavy metals, waste oils, garbage, petroleum-derived polymers, animal and medical wastes cause pollution in water resources. The resulting pollution poses a danger to aquatic creatures and harms these creatures. In this study, obtaining wetttable nanofibers for removing of oils mixed with water was investigated. It is aimed to increase the contact angle value by adding activated carbon produced from apricot kernels to the nanofibers produced from waste polystyrene. As a result, oil-water separation was carried out by increasing the hydrophobicity of the nanofiber and preserving its oil-holding property. In the experiments, 4 different concentrations of solutions were prepared using waste polystyrene and dimethylformamide (DMF)/(C<sub>3</sub>H<sub>7</sub>NO) and nanofibers were produced by electrospinning device. According to the SEM images, it was determined that the sample with the best fibre structure belonged to the 17% solution. On top of this, new nanofibers were produced by adding 5% and 10% by mass of activated carbon made from apricot kernels to the same solution. The contact angle values of the produced nanofibers were measured and it was observed that the hydrophobicity of the 10% activated carbon added nanofiber was the highest. In order to determine the water and oil absorption of the sample with the best structure and activated carbon added samples, it was tested by dropping 2 mL of water and 2 mL of oil on the experimental setup. As a result, it has been proven that this material, which has hydrophobic and oleophilic properties, can be used in oil/water separation processes, and the oil-derived waste polystyrene materials that cause water pollution are recycled.

Received  
03 August 2022

Accepted  
30 October 2022

## Keywords

Water pollution  
Nanofibre  
Oleophilic  
Hydrophobic  
Apricot kernel  
Activated carbon

## SULARA KARIŞAN YAĞLARIN AKTİF KARBON KATKILI NANOELYAF OLUŞTURULARAK TUTULABİLİRLİĞİNİN İNCELENMESİ

## Özet

Su kirliliğine, sulara karışan ağır metaller, atık yağlar, atılan çöpler, petrol türevli polimerler, hayvansal ve tıbbi atıklar vb. neden olmaktadır. Oluşan kirlilik suda yaşayan canlılar için tehlike oluşturmakta ve bu canlılara zarar vermektedir. Bu çalışmada suya karışan yağların sudan arındırılması için ıslanabilir nanoelyaf elde edilmesi amaçlanmıştır. Atık polistirenden üretilen nanoelyafın bünyesine, kayısı çekirdeğinden üretilmiş aktif karbon ilavesi yapılarak temas açısı değerinin artırılması hedeflenmiştir. Sonuç olarak nanoelyafın hidrofobikliğin artırılması ve yağı tutma özelliğinin korunması ile yağ su ayırma işlemi gerçekleştirilmiştir. Deneylerde, atık polistiren ve dimetilformamid (DMF)/(C<sub>3</sub>H<sub>7</sub>NO) kullanarak 4 farklı derişimde çözelti hazırlanmış ve elektrospinning cihazı ile nanoelyaf üretilmiştir. SEM görüntülerine göre lif yapısı en iyi olan numunenin %17'lik çözeltiye ait olduğu belirlenmiştir. Bunun üzerinde aynı çözeltiye kütlece %5 ve %10 oranlarında kayısı çekirdeğinden yapılmış aktif karbon eklenerek yeni nanoelyaf üretilmiştir. Üretilmiş olan nanoelyafın temas açısı değerleri ölçülmüş ve %10 aktif karbon katkılı nanoelyafın hidrofobikliğinin en fazla olduğu görülmüştür. Yapısı en iyi olan numuneyle aktif karbon katkılı numunelerin su ve yağ emme durumlarını belirlemek amacıyla oluşturulan deney düzeninde üzerlerine 2 ml su ve 2 ml yağ damlatılarak test edilmiştir. Sonuç olarak, hidrofobik ve oleofilik özelliğe sahip olan bu malzemenin yağ/su ayırma işlemlerinde kullanılabileceği ispatlanmış ve su kirliliğine neden olan petrol türevli atık polistiren malzemelerinin ise geri kullanıma kazandırılması gerçekleştirilmiştir.

## Anahtar Kelimeler

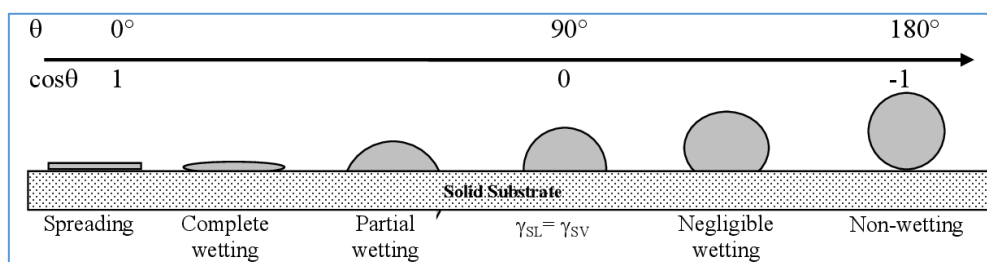
Su kirliliği  
Nanoelyaf  
Oleofilik  
Hidrofobik  
Kayısı çekirdeği  
Aktif karbon

## INTRODUCTION

Environmental pollution occurs with the formation of harmful effects on air, water and soil, which are the basic elements of nature. Types of environmental pollution are known as air pollution, water pollution, soil pollution, noise pollution and nuclear pollution [1]. The release of many harmful substances such as waste oils, petroleum and petroleum-derived substances, detergents, waste batteries to nature causes environmental pollution. The release of many harmful substances such as waste oils, petroleum and petroleum-derived substances, detergents, waste batteries to nature causes environmental pollution. Pouring waste vegetable oils into the sink clogs the sink and sewer pipes. One liter of vegetable waste oil pollutes approximately one million liters of drinking water. Cleaning up contaminated groundwater is very expensive and difficult. Groundwater is an important source of drinking water. In addition, vegetable waste oils mixed with waste water reach the sea and rivers and pose a danger to the lives of aquatic creatures [2]. Recently, due to oil spills caused by tanker accidents, oily factory wastes that arise with the increasing industry, and spilling of frying oil into sinks, waste oils mix into the waters and the pollution of water resources increases. This pollution reduces our clean water resources and poses a threat to aquatic creatures. Therefore, it is of great importance to clean the polluted water from oils. Separation of water and oil mixtures is seen as an important and ecological problem worldwide due to oil spills caused by frequent tanker accidents and oily factory wastes generated with increasing industry [3].

Separation of water and oil mixtures is seen as an important and ecological problem worldwide due to oil spills caused by frequent tanker accidents and oily factory wastes generated with increasing industry. Due to marine accidents, millions of tons of crude oil spill into the seas and cause the death of sea creatures. The organisms that are most affected by marine pollution are the microorganisms that maintain the material cycles in the ecosystem and the predators balance. Disruption of the cycles also prevents sea plants from performing photosynthesis [4]. The surface to be used in oil/water separation processes must be superhydrophobic and superoleophilic. By making different synthesis, carbon nanotube sponge, polymer film etc. materials are prepared and used in oil/water separation processes [3].

Hydrophobic means fearful of water, while hydrophilic means water-loving. Hydrophobicity is a measure of the airiness of a solid or mineral, usually evaluated by the contact angle ( $\theta$ ) [5]. The fact that the liquid does not spread on the solid surface and forms a contact angle means that the solid cannot be wetted by the liquid. In other words, it can be said that the magnitude of this angle varies according to the wettability of the solid. If the contact angle of a solid surface with a liquid has a value between  $90^\circ$  and  $150^\circ$ , these surfaces are called hydrophobic surfaces. In cases where the contact angle is less than  $90^\circ$ , these surfaces are called hydrophilic surfaces because the liquid wets the solid surface. In addition, surfaces where the liquid is spherical on the solid surface and the contact angle is greater than  $150^\circ$  are called superhydrophobic surfaces [6]. The contact angles between the solid surface and the liquid and the wettability of the solid are shown in Fig. 1 below.



**Figure 1.** The state of the contact angle between the solid surface and the liquid [7].

The surfaces that make an angle of  $90^{\circ}$ - $150^{\circ}$  with the oils dripped on them are called oleophobic, and surfaces that make an angle greater than  $150^{\circ}$  are called superoleophobic. Surfaces with an angle of  $10^{\circ}$ - $90^{\circ}$  are called oleophilic, and surfaces with an angle of less than  $10^{\circ}$  are called superoleophilic surfaces [8]. Activated carbon is the substance that has a high carbon content in the majority of its composition, and whose inner surface area and pore volume are increased by physical or chemical activation methods at high temperature [9]. Activated carbon is a good adsorbent in terms of its properties. Activated carbon, which has a wide application and usage area in many fields such as environment, medicine, cleaning of waste water, filtration, gas masks, decolorization, has become an indispensable material of the industry. Powder, granular and spherical activated carbon types are produced commercially. Powdered activated carbon is generally used in solution phase adsorption. Especially decolorization and medical solutions are the most common areas of use. Granular or pellet activated carbons are mostly used in water purification. Spherical activated carbon with high mechanical strength is mostly used in gas adsorption and gas purification areas [10]. It can be produced from materials such as nut shell, almond shell, apricot kernel, fruit shell, coconut shell, olive seed, tea pulp, sugar cane and coffee bean.

Nanotechnology is a new and rapidly developing field of science and technology that aims to bring new physical, chemical and biological properties to matter. Nano-sized materials are used in different studies in many fields because they show new and different functional properties. Materials science covers many fields such as electronics, optics, biology, medicine, chemistry, pharmaceuticals, cosmetics, textile, aerospace and aerospace industries and deals with synthesizing materials such as nanotubes, nanorobots, nano capsules and nanofibers [11].

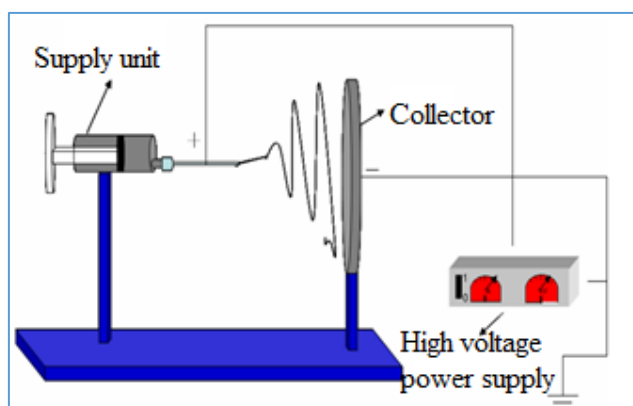
Nanofibers are fibers with a diameter of 100 nm or less. However, according to some sources, fibers with a size below 1 micrometer are also called nanofibers [12]. Nanofibers, a new generation material, have very few structural defects because they have small dimensions. So they are good in terms of functionality and mechanical properties. In addition, due to their small diameters, they have high surface-to-volume ratios or surface-to-mass ratios, therefore they have high specific surface areas [13]. Nanofibers are used in many fields such as tissue engineering, protective mask and clothing making, filtration, sensor making, drug release, catalyst support, wound closure, polymeric batteries and composite support [13,14]. If we consider historically, nanofibers are produced by drawing, mold synthesis, phase separation process, self-assembly and electrospinning (electro-spinning) etc. methods [12]. Today, electro-spinning method is the most used method among these methods. The parameters determining the structure and size of nanofibers are given in Table 1.

**Table 1.** The parameters that determine the structure and size of nanofibers [15].

<b>Parameters from the polymer solution</b>	<b>Process parameters</b>	<b>Environmental Parameters</b>
Molecular weight	Voltage	Temperature
Viscosity	Flow rate	Moisture
Surface tension	Solution temperature	Atmosphere type
Conductivity	Distance from needle to collector	Pressure
Dielectric constant		

Electro-spinning, a viable fiber spinning method, has its origins in the early 1930s. Artificial fibers were first produced by Formhals (1934) [16], using electric charges. He also dissolved cellulose acetate in acetone and produced artificial fiber with this substance.

Although the method of producing artificial yarn using electric field has been tried for a long time, the electro-spinning method did not gain importance until the invention of Formhals [17]. The electro-spinning device consists of 3 basic parts, namely the high voltage power supply, the supply unit and a grounded collector (conductor plate, rotating cylinder etc.). A simple electrospinning setup is given in Fig. 2 [18].



**Figure 2.** A simple electrospinning mechanism [18].

In this method, a high voltage is applied between the capillary needle, which is a liquid solution, and the collector made of a metal with high conductivity, and an electric field is formed. When the voltage is at the threshold level, it overcomes the high surface tension on the liquid drop at the needle tip and takes a conical shape. This shape is called a “Taylor cone”. After a while, nanofibers begin to form on the collector [19]. With this technique, many polymer nanofibers have been produced from melt or solution until today [20,21]. The raw material of disposable cups, plates, yogurt cups and foam materials is polystyrene. In short, plastics are materials that have a polymeric structure and are produced from petroleum derivatives [22]. Plastics are used in many areas and cause environmental pollution if they are not recycled [23]. It was aimed to evaluate waste polystyrene foam as the main material of nanofibers, aiming to reuse plastics that have been used for many years and take many years to dissolve when released into the environment.

In this study, obtaining wettable nanofibers for oils mixed with water was investigated. It is aimed to increase the contact angle value by adding activated carbon produced from apricot kernels to the nanofibers produced from waste polystyrene. As a result, oil-water separation was carried out by increasing the hydrophobicity of the nanofiber and preserving its oil-holding property.

## MATERIAL AND METHOD

The waste foam materials (polystyrene) were used to produce nanofibers. First, hexane ( $C_6H_{14}$ ) and petroleum ether ( $C_7H_7BrMg$ ) were tried separately to dissolve the polystyrene foam. It has been observed that these solvents do not dissolve the polystyrene foam. Dichloromethane ( $CH_2Cl_2$ ) and ethyl acetate ( $C_4H_8O_2$ ) were used instead of these solvents. New area brand NE-300 model electrospinning device was made into nanofibers (Fig. 3).



**Figure 3.** Electrospinning device used in experiments.

Dimethylformamide (DMF) ( $C_3H_7NO$ ) was used as an alternative solvent because the yarn structure was thinner than desired. Nanofiber was formed and its structure was found to be in the desired state. Polystyrene/DMF solutions at 10%, 15%, 17% and 20% by mass were prepared and turned into nanofibers. The contact angles of the fibers and SEM images were examined at Selcuk University Advanced Technology Research Centre. SEM images of the obtained nanofibers were taken with Zeiss Evo LS 10 brand, Evo10.11.29 model scanning electron microscope and contact angle measurements were carried out with Dataphysics brand, Oca50Micro model contact angle device in order to determine the hydrophobicity of the samples. The result of the experiment, in which bubble formation, which is an undesirable situation, was observed the least, was obtained in electrospinning processes with 17% solution. Then, 5% and 10% activated carbon obtained from apricot kernels was added to this solution. The contact angles and SEM images of the activated carbon added samples were also examined. Finally, the samples with the best results were used to observe the water and oil absorption conditions in the experimental setup. The experimental setup used is given in Fig. 4.



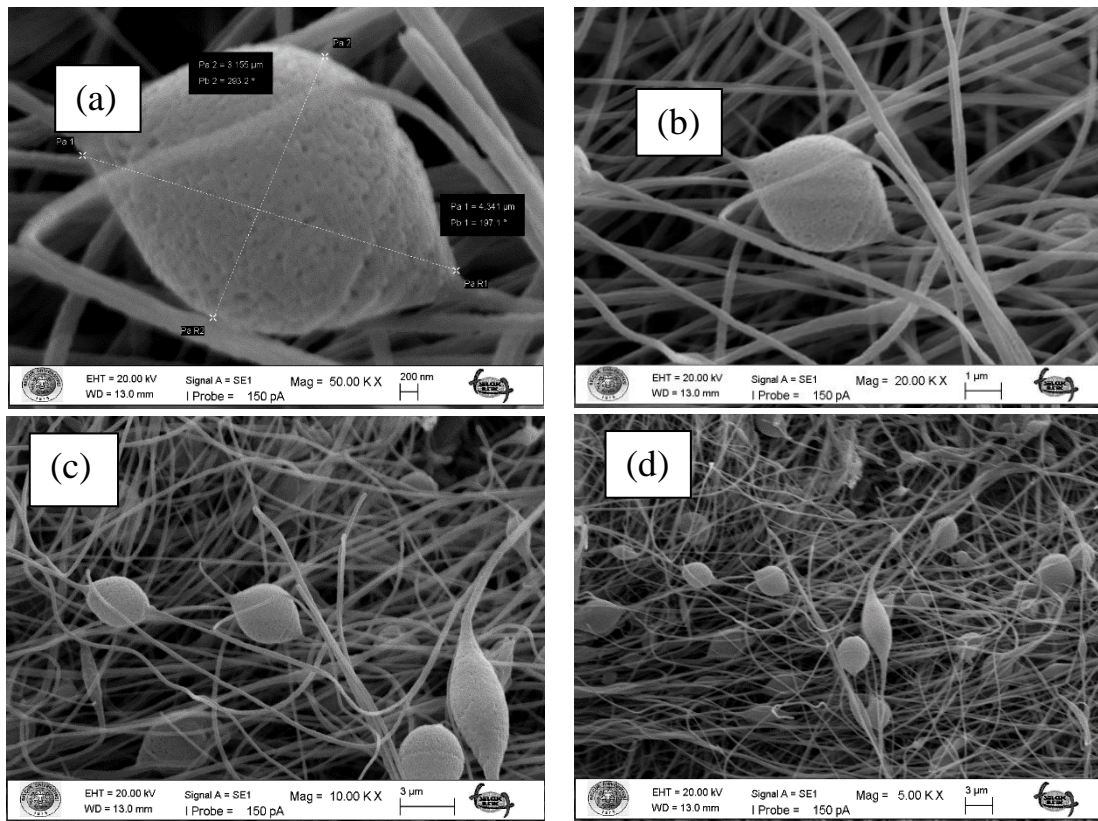
**Figure 4.** The experimental setup.

## RESULT AND DISCUSSION

DMF was used as the solvent, which we observed in the preliminary experiments that it dissolves the waste polystyrene foam well. Solutions with 10%, 15%, 17% and 20% mass concentrations were prepared and electro-spinning was carried out. The SEM images of the



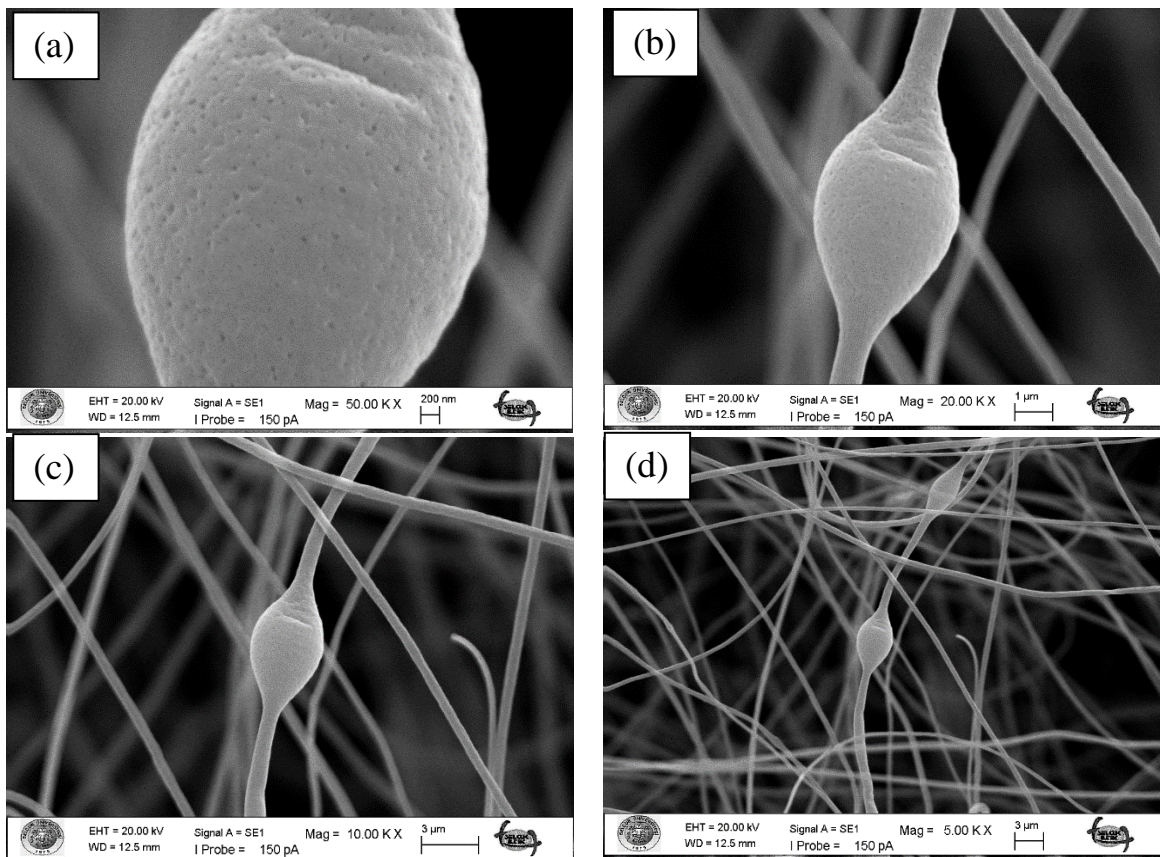
nanofibers obtained from the experiments are given below, respectively. SEM images of nanofibers obtained from 10% (by mass) prepared solution are given in Fig. 5.



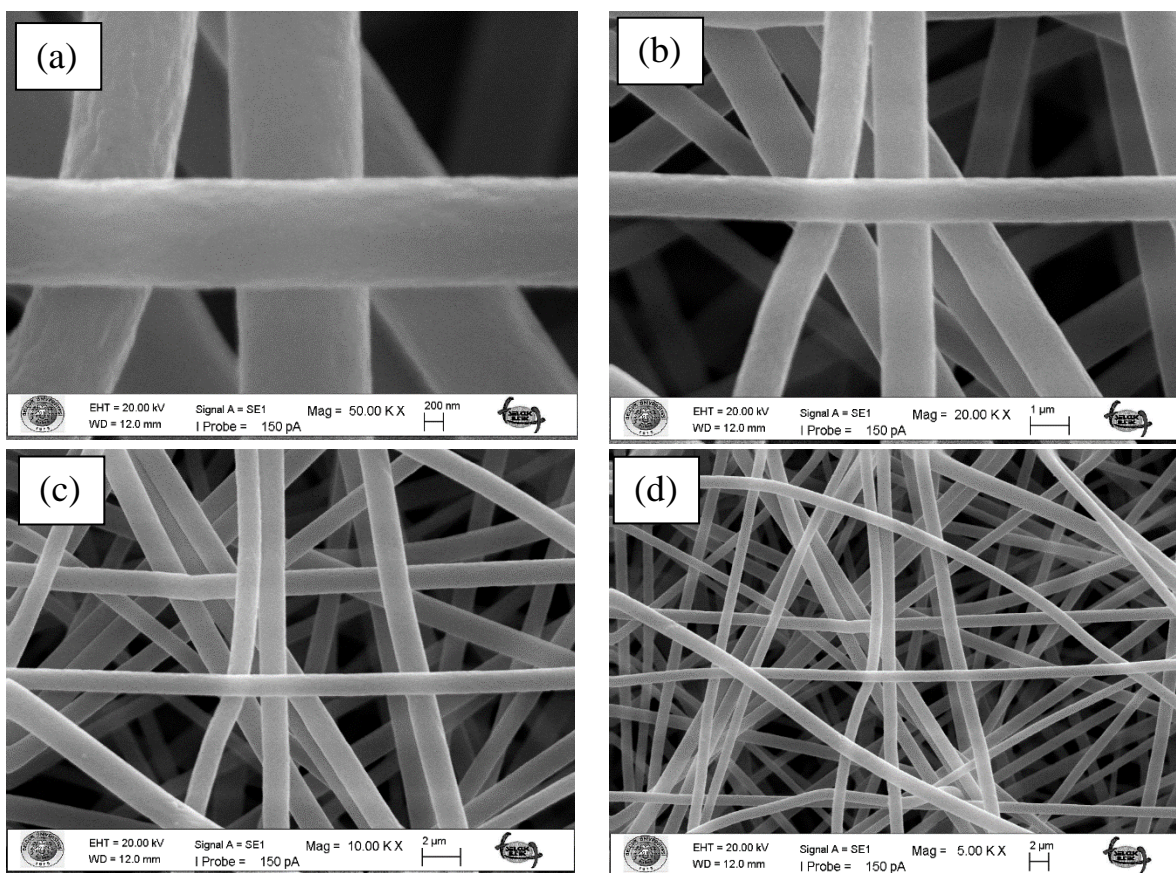
**Figure 5.** SEM image of spun nanofiber obtained from 10% solution a) 50.00 magnification, b) 20.00 magnification, c) 10.00 magnification, d) 5.00 magnification.

When the SEM images were examined, it was observed that the structure of the nanofibers was not homogeneous in Fig. 5. In addition, it was determined that the nanofiber formation was defective and that they had beaded structures. SEM images of nanofibers obtained from 15% (by mass) prepared solution are given in Fig. 6. In Fig. 6, it is seen that the structure of nanofibers is not homogeneous and there are bead-like structures as in Fig. 5. In Fig. 7, it can be seen that perfect nanofibers are formed. When SEM images are examined, it is seen that the nanofiber structures are homogeneous and their diameters are very close to each other.



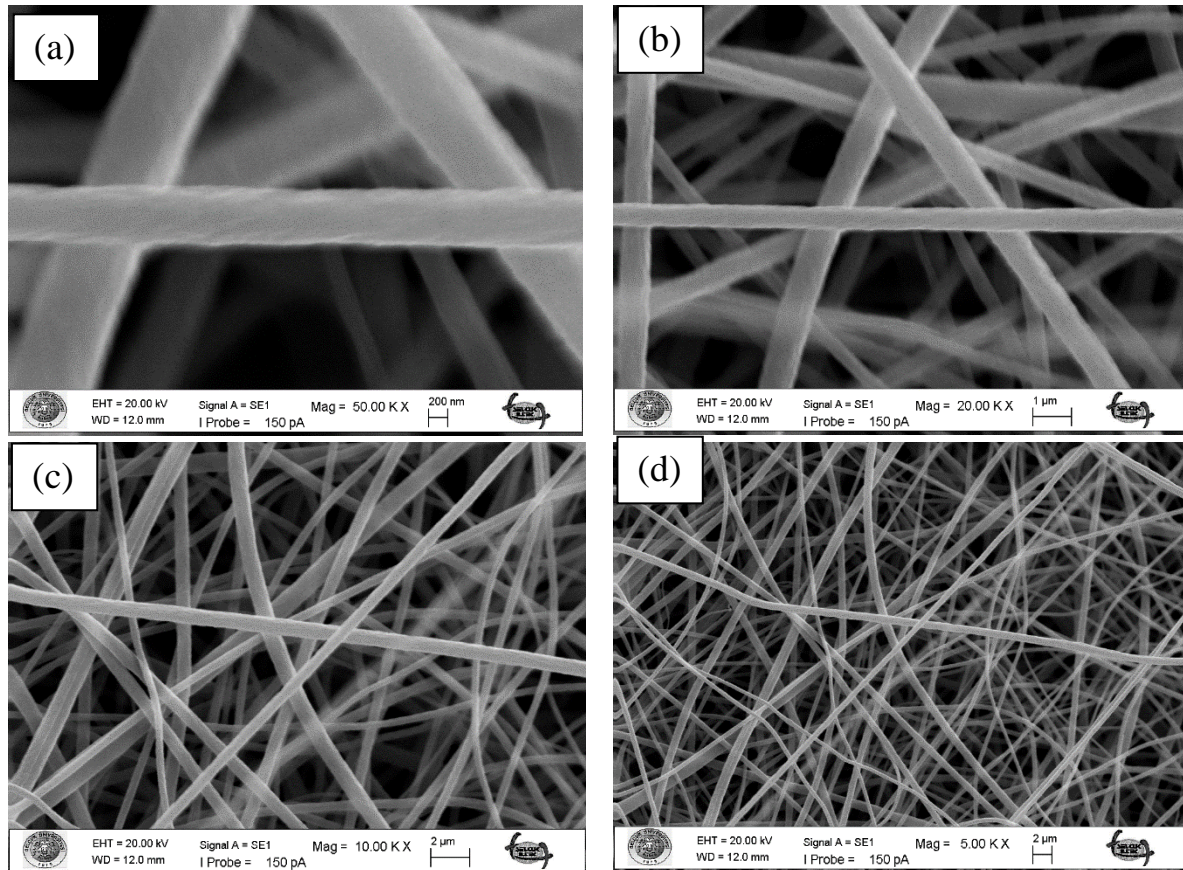


**Figure 6.** SEM image of spun nanofiber obtained from 15% solution a) 50.00 magnification, b) 20.00 magnification, c) 10.00 magnification, d) 5.00 magnification.



**Figure 7.** SEM image of spun nanofiber obtained from 17% solution a) 50.00 magnification, b) 20.00 magnification, c) 10.00 magnification, d) 5.00 magnification.

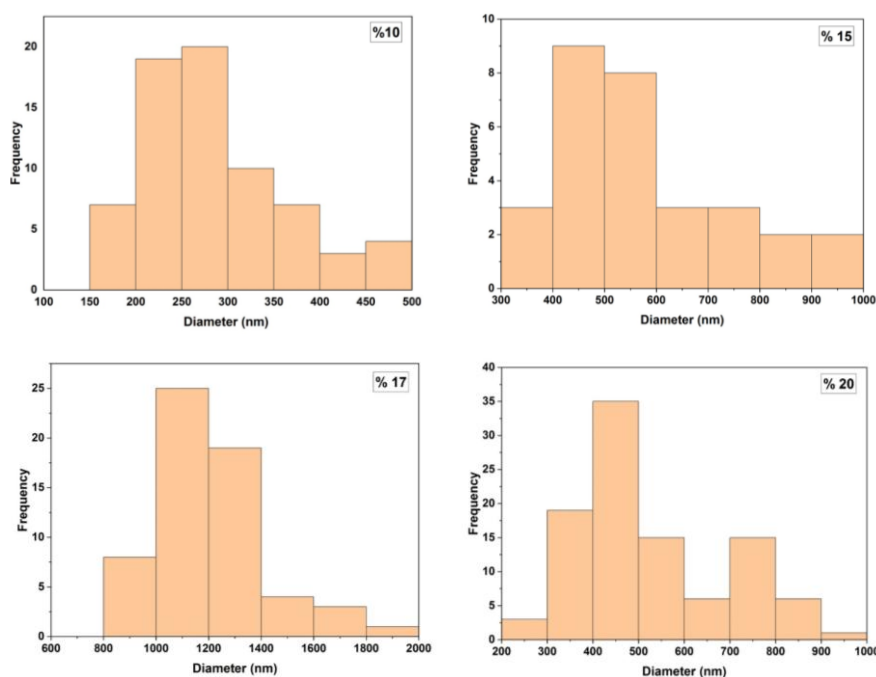




**Figure 8.** SEM image of spun nanofiber obtained from 20% solution a) 50.00 magnification, b) 20.00 magnification, c) 10.00 magnification, d) 5.00 magnification.

As shown in Fig. 8, flawless nanofibers were obtained. Although there are no beaded structures, it is seen that the fiber diameters are not homogeneously formed. As a result, it was seen that the nanofiber obtained with 17% DMF/polystyrene solution has a very perfect structure. Histogram plots of the diameters of spun nanofibers obtained from DMF/polystyrene solutions of different mass were obtained (Fig. 9).





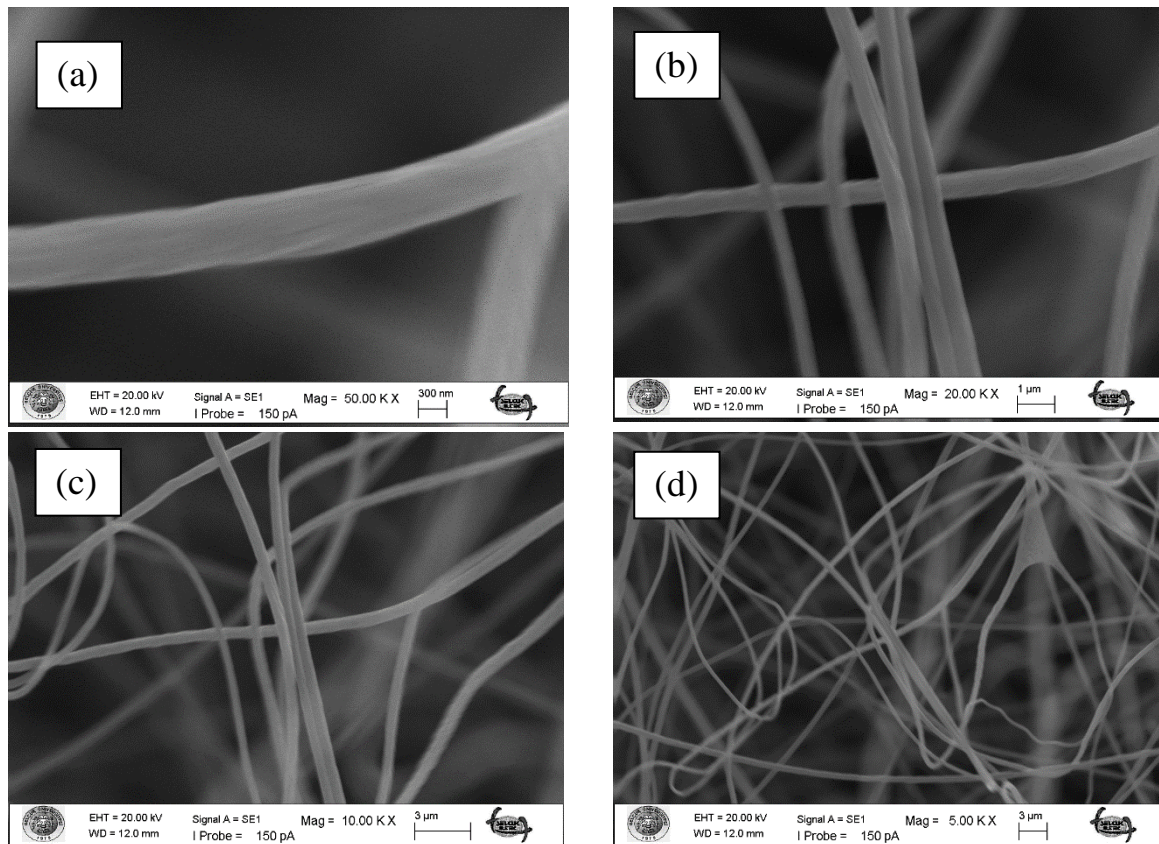
**Figure 9.** Frequency dependent diameter histograms of spun nanofibers obtained from solutions different in mass.

When Fig. 9 is examined, it has been determined that the fiber diameters obtained from the 10% concentration solution are around 150 nm minimum and 475 nm maximum. According to the fiber diameter distribution diagram, it is seen that most of the fibers have diameters ranging from 250-300 nm and irregular bead-like structures are formed after 300 nm. It has been determined that the fiber diameters obtained from the 15% concentration solution are around 300 nm minimum and 950 nm maximum. According to the fiber diameter distribution diagram, it is seen that most of the fibers have diameters ranging from 540-600 nm and irregular bead-like structures are formed after 600 nm. The fiber diameters obtained from the 12% concentration solution were found to be around 1200 nm, while the fiber diameters obtained from the 20% concentration solution were found to vary between 300-900 nm. According to these results, it is clearly seen that the nanofiber prepared with 17% DMF/polystyrene solution has the most perfect structure. The contact angle value was increased by adding activated carbon to the nanofibers produced from waste polystyrene. As a result, oil-water separation was achieved by increasing the hydrophobicity of the nanofibers and maintaining the oil-holding property. For this reason, experiments were carried out by adding 5% and 10% by mass of activated carbon obtained from apricot kernels into a 17% solution. The photograph of powdered activated carbon obtained from apricot kernels used in the experiments is given in Fig. 10. SEM images of the obtained nanofibers are shown in Fig. 11 and Fig. 12, respectively.

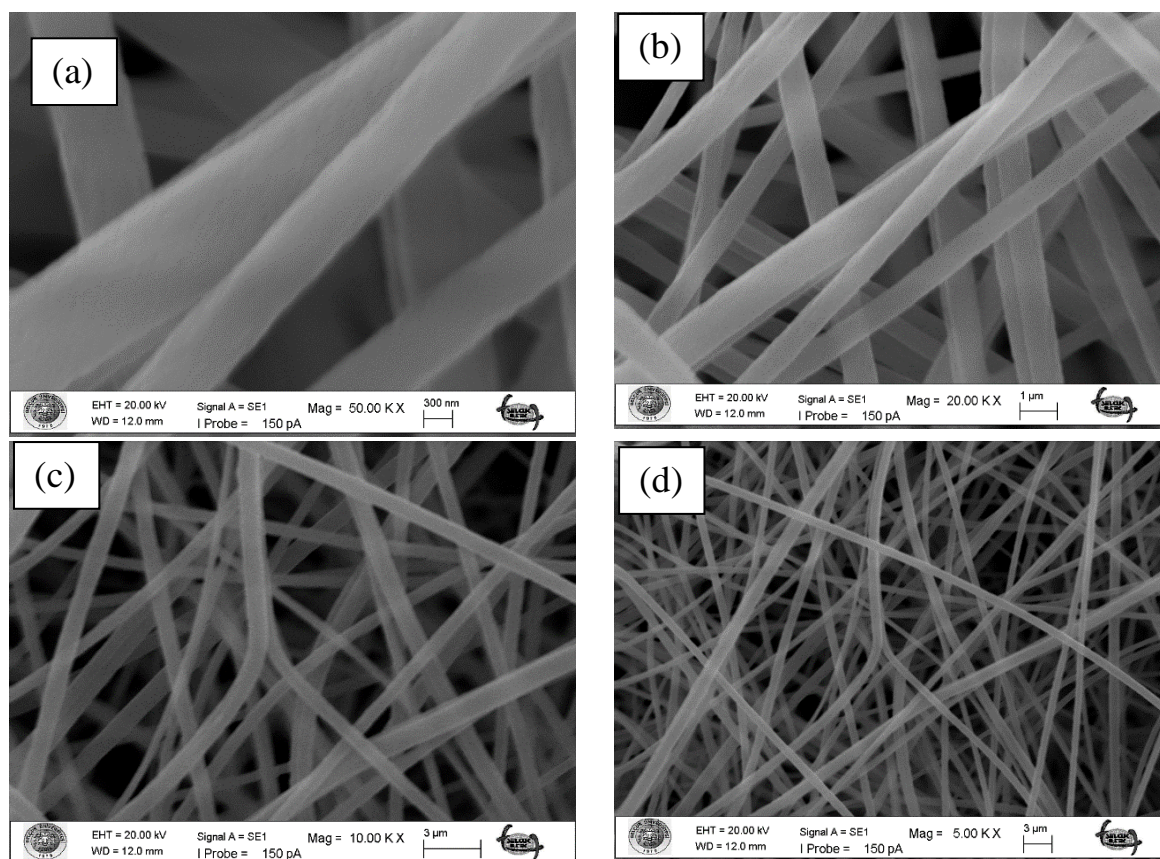


**Figure 10.** Photograph of powdered activated carbon from apricot kernels.

As seen in Fig. 11 and Fig. 12, it is seen that there were perfect structures in both nanofiber formations. However, considering the nanofiber diameters and homogeneous distribution, it was determined that the best results were obtained with the addition of 10% activated carbon. The properties and content of the obtained nanofibers, photographs of the obtained nanofibers and contact angle values are given in Table 2.


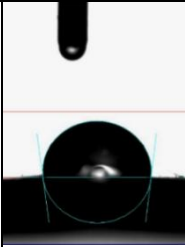

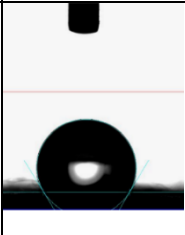


**Figure 11.** SEM image of 5% activated carbon doped spun nanofiber a) 50.00 magnification, b) 20.00 magnification, c) 10.00 magnification, d) 5.00 magnification.


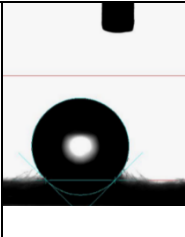

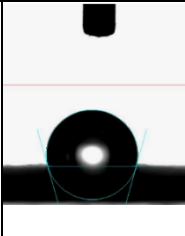
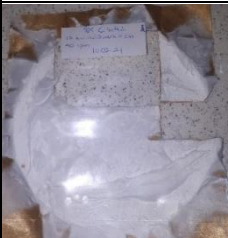
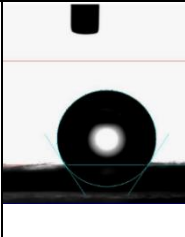

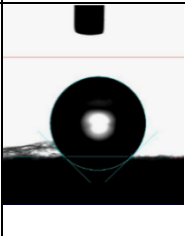


**Figure 12.** SEM image of 10% activated carbon doped spun nanofiber a) 50.00 magnification, b) 20.00 magnification, c) 10.00 magnification, d) 5.00 magnification.

**Table 2.** Properties and content of obtained nanofibers, photos of nanofibers, contact angle values and contact angle images (The magnetic force that allows the fiber to reach the collector (17 KV), sample delivery rate per hour (inversely proportional to fluidity) (0.02-0.03 mL/h), The distance between the needle and the collector (11 cm) and the number of rotation per minute of the totalizer (50 rpm).

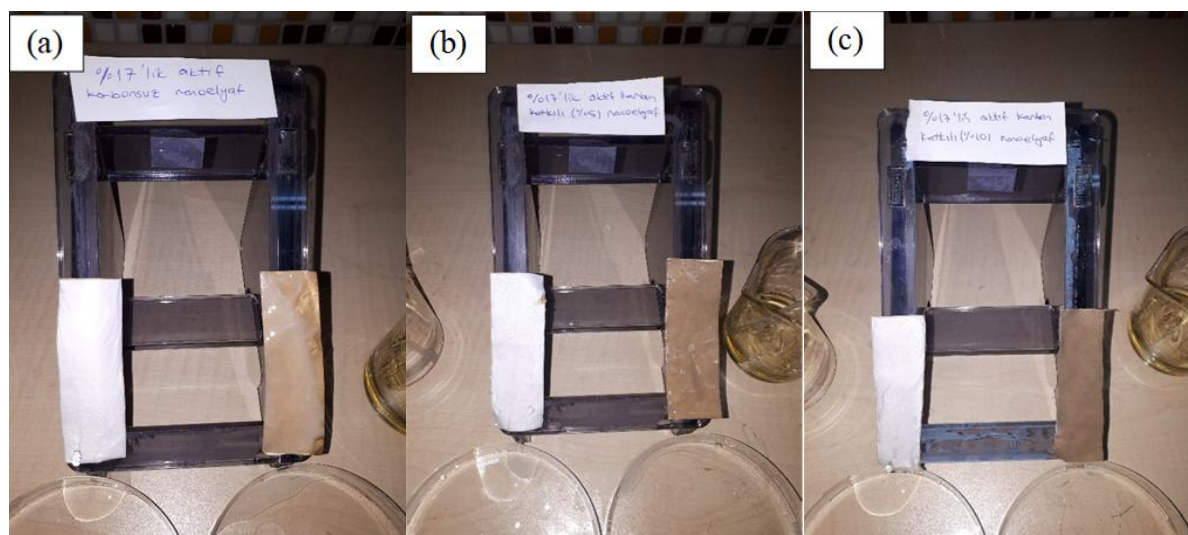
Nanofiber Content and Properties	Nanofibers	Contact Angle Values ( $\theta$ )	Contact Angle Images
10% by mass (1 g polystyrene, 9 g DMF)		97.5	
10% by mass (1.5 g polystyrene, 8.5 g DMF)		114.8	



17% by mass (1.7 g polystyrene, 8.3 g DMF)		121.4	
20% by mass (2 g polystyrene, 8 g DMF)		105.5	
17% by mass, 5% activated carbon added (1.7 g of polystyrene, 8.215 g of DMF and 0.085 g of activated carbon)		123.8	
17% by mass, 10% active carbon added (1.7 g of polystyrene, 8.13 g of DMF and 0.17 g of activated carbon)		136	

It was determined that the contact angles ( $\theta$ ) of the pure nanofibers prepared with DMF/polystyrene solution varied between  $97.5^\circ$ - $121.4^\circ$ . It was observed that the contact angle ( $\theta$ ) values increased and reached  $123.8^\circ$ - $136^\circ$  values in activated carbon added fibers made from apricot kernels. It was determined that the sample with 10% activated carbon added had the best contact angle value. By looking at the fiber structures and contact angle values in the priority SEM images, it was appropriate to test the sample made from 17% solution and the activated carbon added samples prepared in two different ratios in the experimental setup. For this, 6 identical nanofibers of  $2 \times 6$  cm dimensions were used. In order to observe the oil/water absorption of these 3 different samples, 2 mL of water was added to the first of each sample and 2 mL of sunflower oil to the other with a pipette. Experimental setups are given in Fig. 13. According to the results of the experiment, it was observed that the oil was retained, not the water, by the nanofibers. In a study by Zhuo et al., (2008) [24], they pointed out that

polyurethane was dissolved in DMF and the nanofiber material obtained from this solution was produced perfectly. In addition, in another study, it was stated that the development of functional materials is of great importance in order to solve the problems of oily wastewater and water pollution. It has been stated that solutions based on filtration and adsorption methods can be produced by using various materials and materials with surface superhydrophobic properties [25].



**Figure 13.** Experimental setups a) 17% activated carbon-free nanofibers, b) 17% activated carbon-added (5%) nanofibers, c) 17% activated carbon-added (10%) nanofibers.

## CONCLUSION

This study was carried out in order to find a solution to the pollution caused by the mixing of oils that cause water pollution into the water. In the light of the information gathered, it was determined that this problem could be solved by producing special hydrophobic materials. As a result, many polymer types that can be used in nanofiber production and reused by recovering from wastes have been determined by this method. It was concluded that these polymers can be dissolved in different solvents and different solutions can be prepared. For this, a solution of 4 different concentrations was prepared using polystyrene and DMF and turned into nanofibers with an electrospinning device.

According to the SEM images taken, it was determined that the sample with the least bead formation and the best fiber structure was nanofiber made from 17% solution. Then, in order to evaluate the wastes, activated carbon, which is a good adsorbent and produced from apricot kernels, was added to the 17% solution. It has been determined that the nanofibers with 10% activated carbon doped have a perfect structure. It was observed that the hydrophobic property of the nanofiber surface increased with the increase of the measured contact angle value. In line with the experiments, it has been proven that the oils that mix with the water can be retained thanks to the nanofibers we have created, as it does not retain water and absorbs oil.

Alternatively, it can be suggested that the amount of polymer, activated carbon and solvent in the solution can be changed and their effects on nanofibers can be observed. It may also be possible to change the type of waste material from which the activated carbon is made. The thickness and dimensions of the nanofiber can be adjusted according to the desired situation so that it can be used in advanced systems, that is, to increase its durability. In different studies, it can be examined whether it absorbs different types of waste oil such as hazelnut, corn, motor oil and other substances.

## ACKNOWLEDGMENT

This study, with the application number of TÜBİTAK-1689B012135210, qualified to participate in the final stage of the "Polar Research Projects" (TÜBİTAK-Kutup Araştırma Projeleri) competition for 2021 high school students.

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