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Preparation and characterization of nanocomposite textile filter for separation of the oil/water mixture using PA-PVA blends reinforced with carbon nanoparticles

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Abstract

The electrospinning technique used to prepare a nano fibre filter under limited conditions involves (20 kV applied voltage, 15 cm gap distance, 1 ml/h: flow rate, and 0.4 mm needle diameter). Several tests for prepared materials were conducted including [scanning electron microscopy (SEM-EDX), Fourier Transform Infrared (FTIR), contact angle, and particle size analysis]. As well as, the characterization of the water/oil mixture was tested before the filtration process and after it. According to the SEM data, smooth and homogeneous nanofibers with an approximate diameter of 350 nm and open porosity morphology were produced using the electrospinning approach. The liquid particles adhere to the filter fibers and work to shrink the holes in the filter, allowing the water to pass through the filter while recovering its characteristics, according to SEM results obtained after the filtration process. According to AFM data, the majority of pores have a diameter of around 100 nm, while the minority has a diameter of more than 100 nm but less than 2500 nm. The results of the catachrestic of water were saved after filtration of a sample by the prepared filter and it closed up the normal water properties by increasing the carbon nanoparticles ratios. The infrared data (FTIR) demonstrate that there is a physical interaction between the polymeric fibers and the added carbon particles since there is a drop in the intensity of infrared radiation after the carbon particles are added to the filter fibers. The hydrophilicity property is found to rise with increasing carbon nanoparticle ratio, and contact angle data demonstrate that this characteristic reduces with increasing carbon nanoparticle ratio, going from 72 o for pure nano filter to 44 o for filter with 0.3 percentage of carbon nanoparticles.

Keywords: Carbon nanoparticles, Electrospinning, Filtration, In-situ polymerization, Nano composites.

Introduction

Nano-composite materials are important in protecting the environment from pollution. The properties of other engineering substances are significantly enhanced by the reinforcement of nanoparticles[1-3]. Since there have been several oil spill tragedies as well as rapid industrialization during oil production or transportation, which has resulted in significant loss and long-term damaging consequences on the environment, oil/water separation has become a research hotspot recently. Many methods have been used to separate the emulsion of oil - water; as settling, heating, distillation, centrifuging, electrical treatment, chemical treatment and filtration [4], however, their efficiency in extracting oil from emulsified oil/water combinations is limited. Filtration is thought to be one of the most effective techniques for separating emulsions Reverse osmosis, nanofiltration, ultrafiltration, and microfiltration are the four types of filtration. One of the most popular techniques for filter separation is nanomembrane filtration. Consequently, researchers have concentrated on developing optimal oil spill absorbents with excellent selectivity and huge absorption capacity by adopting the proper surface structure and composition design [5]. Adding nanoparticles to nanocomposites improves their mechanical, thermal, optical, and antibacterial properties. Materials' hydrophobicity behaviour is classified into two types involve those that are hydrophilic and hydrophobic [5,6] In contrast to hydrophobic materials, which have self-cleaning surfaces and tend to reject water, hydrophilic materials tend to form hydrogen bonds with water and have a contact angle of less than 90 degrees [7-9].

The primary synthesis techniques for nanomaterial-based membranes are phase inversion, sintering, layer-by-layer, interfacial polymerization[10] and electrospinning [10-11]. Recent studies have shown that the electrospinning approach is a flexible and efficient method to create nanofibrous membranes, particularly polymer membranes with controllable compositions and structures [12-14]. Additionally, this method has become particularly powerful when combined with Nano materials that have remarkable features, such as high surface-to-volume ratio and multi-porous structures, as well as specific chemical, physical, and mechanical functions provided by easily and controllably incorporating other components [15-17]. Saikat et al 2018, created a superhydrophobic membrane by incorporating recycled carbon black (from Waste tires after treatment with extraction and high-temperature calcination) into a polyvinylidene fluoride (PVDF) solution. A steep increase of 18–20% was observed in the loss and storage moduli [18]. Yixuan et al. rganized a review study about electrospun nanofiber materials (ENMs), which are divided into two groups based on their function involving selective layers, and supportive substrates. They discussed the architectures and properties of conventional ENMs and mixed matrix ENMs for the function of the nanofiber layer as a selective layer. In-depth examples of parameter fabrication and adjustment techniques, including surface modification, inorganic and organic incorporation, polymer and co-solvent incorporation, are shown. It is essential to have a matching selective layer operating as a supporting layer for nanofiber layers. This study presents the numerous selective layers that were created on the nanofiber layer. Inorganic deposition, polymer coating, and interfacial polymerization are some of the fabrication techniques. Finally, the utilization of ENMs for water treatment is reviewed along with future prospects and the main industry challenges. The development of ENMs is anticipated to boost the prosperity and use of a number of industries, including water treatment, environmental protection, healthcare, and energy storage [19].

This study aims to prepare of nano textile filter using a branch poly amid (PA 66) / carbon nanocomposites solution via in-situ polymerization technique.

Materials and Methods

The samples were derived from the residue of the process of heating the extract products in a furnace of the treatment unit of the Furfural as in Fig. 1, which is assembled in the strainer refinery installed before the pump pushing the material mentioned for the evaporation unit. It was taken from Al-Daura refinery during the refining of crude oil (it is considered a by-product resulting from the incomplete burning of hydrocarbon compounds). The Furfural is used as a solvent to get rid of aromatized compounds with poor lubrication specifications of the extracted product, (an atypical oil product) that is heated at a temperature of no more than 240 °C. 1, 6 Hexa methylene diamine, Adipoyl chloride, Sodium hydroxide has been acquired from sigma Company. Cyclohexane was obtained from fluka AG, polyvinyl alcohol (PVA) was purchased from CDH (p) ltd. Formic acid from Alpha Chemika.

Figure 1. Raw materials of petroleum residue

Preparation of Carbon Nano-particles (CNPs)

Cyclohexane solvent was utilized to separate the furfural solvent from the raw material in the first step of the extraction procedure, which involved washing the raw material in a solvent-solvent mixture. After being washed, the raw materials are depicted in Fig. 2.

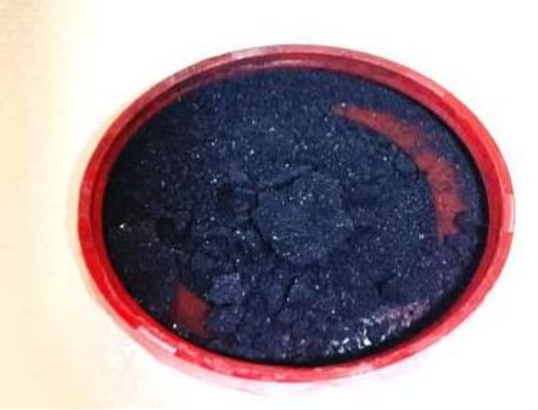


Figure 2. Shows the raw materials after washing by hexane solvent

The washing procedure was carried out three times with a considerable volume of hexane to completely remove the furfural compound and the related impurities and to obtain the pure raw material. The raw material was exposed to the elements for 36 hours before being dried in a drying oven set to 60 °C to ensure that the sample's whole hexane content had disappeared. The final step in the production of Nanocarbons entails electrically vibrating the sieves to separate the material and remove any large lumps that are not wanted. It uses a 45 micron sieve filtration level. The sample was cleaned with alcohol and then distilled water to remove impurities, and the raw material was completely purified by drying it for an hour at 80 °C. Then burning of the sample using thermal pellets coated with ready-made temperature-resistant plaster paste for one hour at 1050 º C in an inert and evacuated atmosphere as in Fig. 3. The test was repeated again to check that the percentages of carbon were high.

Figure 3. Shows the container of raw materials before covering & after covering

The burned sample was pulverized in a mortar to reduce the particle size after removing the larger particles and leaving only those with a diameter of 50 microns or less. Subsequently, to generate a sodium hydroxide solution with a concentration of 10 M, 20 g of sodium hydroxide was dissolved in 50 ml of distilled water along with 2 g of carbon. The mixture was then stirred until it was homogenous. The beaker was exposed to 900 watts of ultrasonic energy for three hours. The carbon atoms were then extracted from the solution using a centrifuge at a speed of 12,000 rpm, repeatedly rinsed with distilled water to eliminate the hydroxide, and finally separated to be ready for granular analysis.

Preparation of Poly amide 6, 6 – CNPs by In situ-polymerization Process

Using an in-situ polymerization process, pure PA 66 and composites of PA 66 reinforced with carbon nanoparticles were produced. The first solution consisted of 25 ml of distilled water and 1 g of 1,6 hexamethylene diamine. After that, 50 ml of sodium hydroxide NaOH (10M) was used to create the NC suspension, which was then ultrasonically dispersed for 15 minutes. 10 drops of the freshly created solution were added to 10 ml of the first solution in a 50 ml beaker. The second solution was prepared by combining 1 ml of a dipole chloride with 19 ml of cyclohexane and set aside and covered. A 50 ml beaker was tilted at a 45-degree angle, and 10 ml of the second solution was applied dropwise to the edge of the beaker. A polymer film formed at the interface of the two solutions as soon as they came together and attached to the beaker's outside. The string was gently pulled with a glass rod. Fig. 4 displays the prepared polyamide.

Figure 4. Prepared poly amide

Preparation of Nano-composite blend for electrospinning

Table 1 contains the several dosages utilized in the solution blending process to create the nano-composite blend.

Table 1. Contents of samples

samples	By in situ polymerization	With
A	1ml from dissolved PA 15%	9 ml from dissolved PVA
B	1ml from dissolved (PA +0.01 CNPs) 15%	
C	1ml from dissolved (PA +0.02 CNPs) 15%	
D	1ml from dissolved (PA +0.03 CNPs 15%	

All samples of poly amide 15% concentrated in formic acid were thoroughly stirred for 15 minutes at room temperature after dissolving. Each sample was then given 9 ml of PVA that had been dissolved in formic acid, the two were combined, and the mixture was subjected to a 30-minute ultrasonic treatment. 10 ml of the (A, B, C, and D) solutions were pumped using an electrospinning system for 10 hours, as in Fig. 5. A positive high voltage power source (20 kv) was attached to the metallic needle and the negative power supply was linked to an earthly metallic collector. The solution was then collected on aluminum foil.

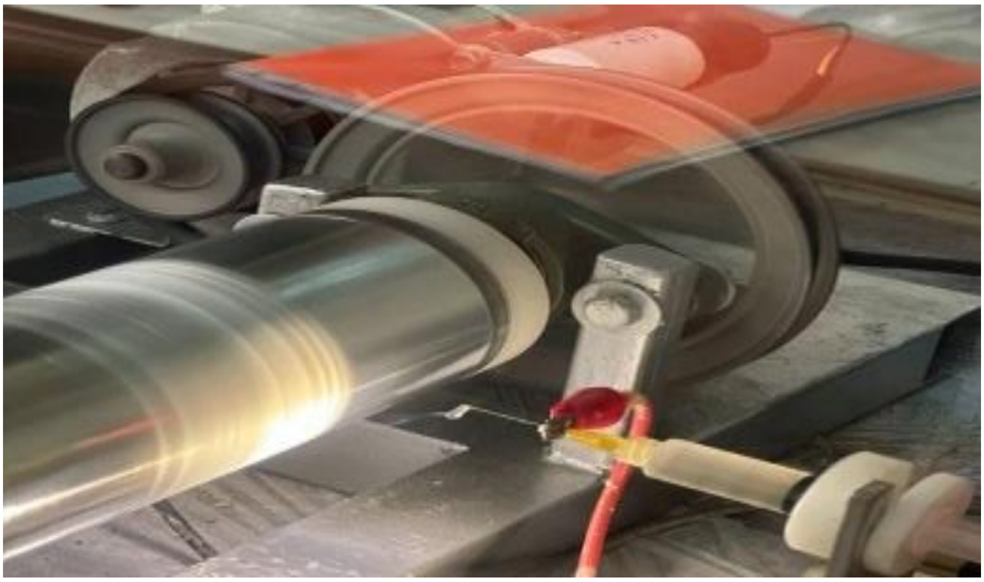


Figure 5. Electro spinning system

Oil/ water System Preparation and separation

40 g of distiller water and 1 g of fuel oil with the parameters listed in table 2 are combined in an ultrasonic mixer at 60 ° C for 15 min to create an emulsion. A constructed nano filter that was placed in a syringe pump was used for the separation system. The Nano filter then isolates the water due to its exceptional hydrophilicity and sequesters the oil through an absorption process [20].

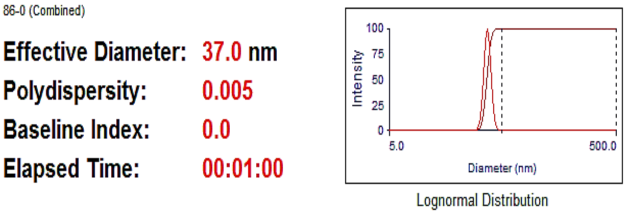
Results and Discussion

Particle size analysis

Analyses of particle sizes are shown in Fig. 6. The produced carbon nanoparticles' respective particle sizes in the samples are roughly 37 nm. It has been established that using a sonication probe, Nano size can be successfully reached in less time. Additionally, the polydispersity factor is close to zero at roughly 0.005, indicating good stability and anti-agglomeration properties.

Table 2. Properties of oil/water

Test name	Result	Standard test method
Density @ 15.0 c (g/cm3)	0.9765	ASTM D4052
Viscosity @ 50 c (mm2/s)	274	– 22
Total sulfur (w%/w)	4.6	ASTM D 445
Carbon residue(w%/w)	9.2	ASTM D 4294
Water and sediment (v%/v)	< 0.025	ASTM D 524 ASTM D 1796



86-0 (Combined)

Effective Diameter: 37.0 nm
Polydispersity: 0.005
Baseline Index: 0.0
Elapsed Time: 00:01:00

Figure 6. Effective diameter of prepared Nano carbon

FTIR analysis of prepared filter

Fig. 7 (a-d). Shows the FTIR analysis of PA, PA + CNPs, PVA+PA nanofibers, and PVA+PA+ CNPs.

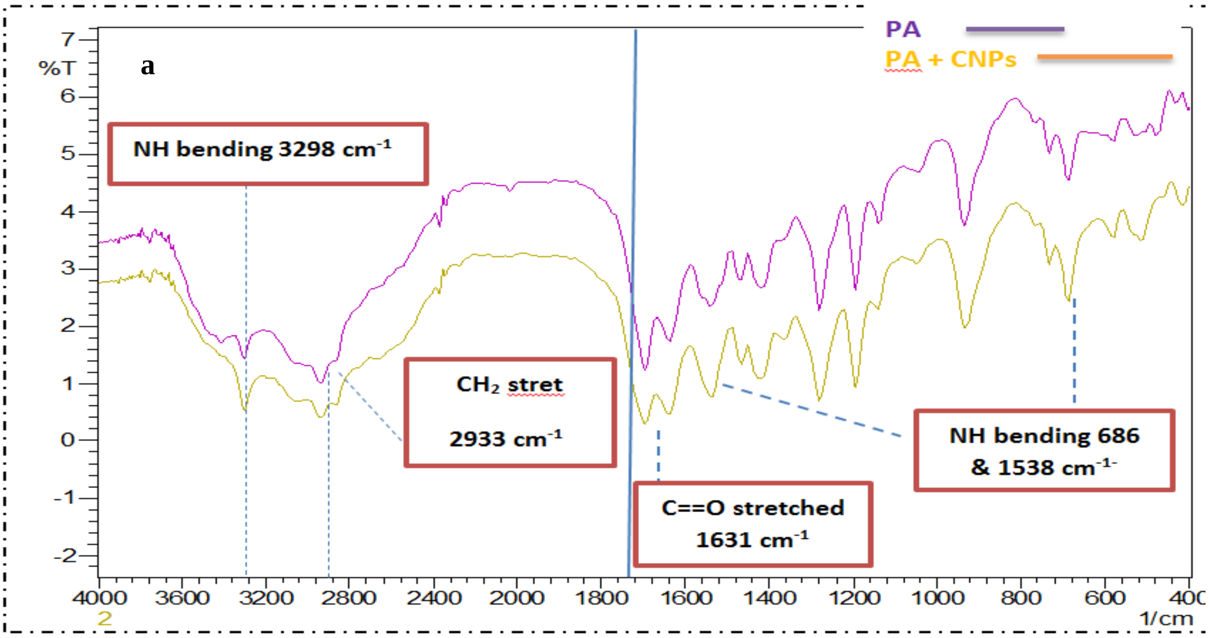


Figure 7 a. FTIR analysis of PA and PA+CNPs

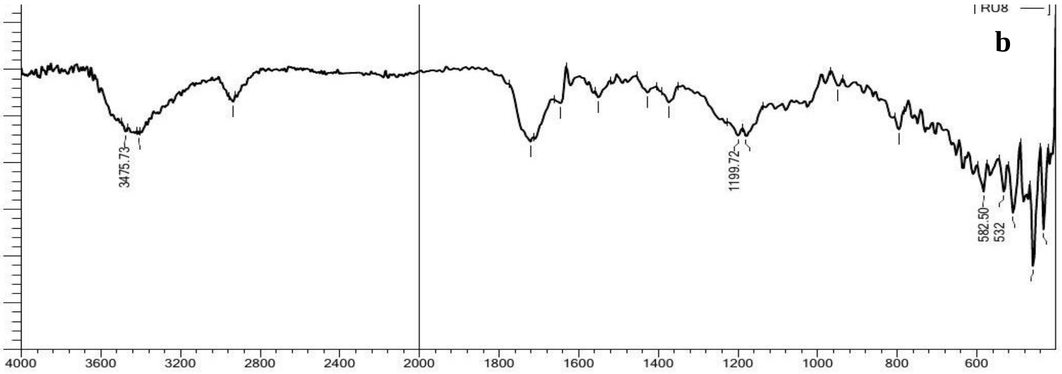


Figure 7b. FTIR analysis of PVA+PA blend nanofibers (matched with Darwish et al.[21])

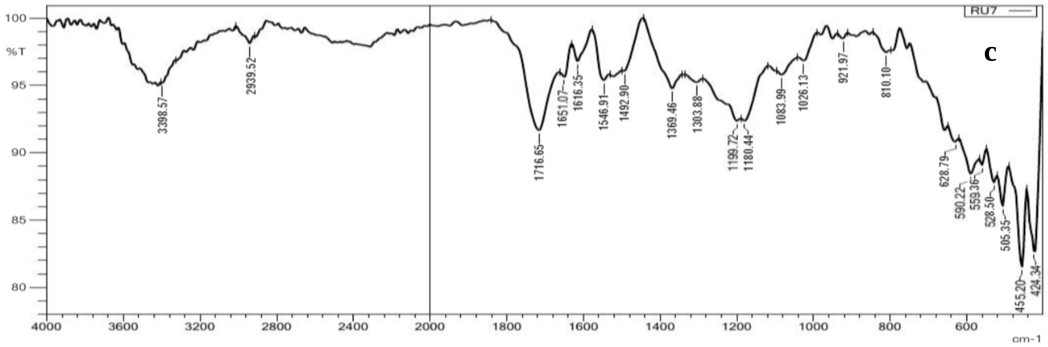


Figure 7c. FTIR analysis of PVA+PA + CNPs 0.02 wt% blend nanofibers

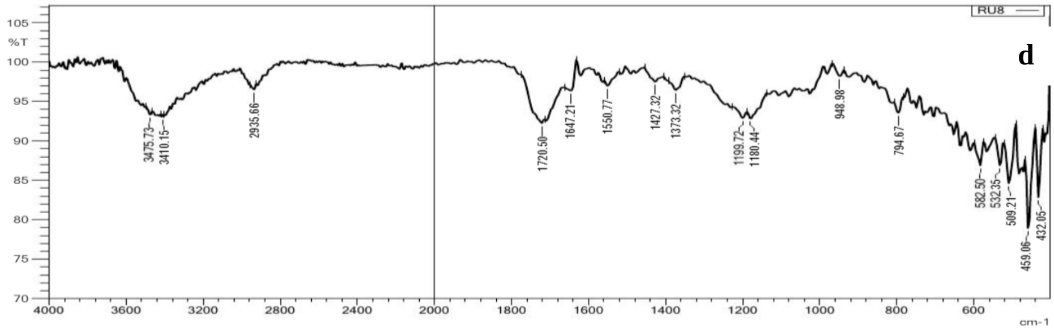


Figure 7d. FTIR analysis of PVA+PA + CNPs 0.03 wt% blend nanofibers
Figure 7. FTIR of Nanofibers Samples

The typical PA66 vibration frequency peaks are found at 3298cm^{-1} (NH bending vibration), 2,933 cm^{-1} (CH_2 stretching), 1631 cm^{-1} (C = O stretching, amide I), 1536 cm^{-1} (NH bending vibration), and 686 cm^{-1} (NH bending vibration) [21]. The stretching peaks of the methylene groups (2850-3000 cm^{-1}) and N-H groups in amide (1550 cm1), which correspond to those of PA66, were visible in the FTIR spectra of the PA66-CNPs. Particularly, the stretching peak of PA66-CNPs at 1631 cm^{-1} in the FT-IR spectrum showed considerable strength [20--21]. We see that carbon particles behave as darkening zones that restrict the transit of infrared rays and enhance their reflection, resulting in a decrease in the intensity of infrared transmittance. we notice the main peaks of PA, and PVA was presented It is interesting to note that the absorption peaks corresponding to the hydrogen-bonded N—H (3000–3650 cm^{-1}) and C== O stretching (1450–1800 cm^{-1}) motions of PA and OH stretching (3000–3700 cm^{-1}) of all PVA shifted to higher wavenumber as in Fig. 7b-d. The shifting of the hydrogen-bonded, C== O vibration to lower frequencies can be attributed to the gradually strengthened interaction between the carbonyl and hydroxyl groups in each PA & PVA series Based on these results, it is reasonable to suggest that the presence of PVA in PA can interfere with the hydrogen-bonded carbonyl groups originally present in PA, and lead to new interactions between the carbonyl and hydroxyl groups as the PVA content of each blend series increases. On the other hand added of the carbon nanoparticles leads to shifting of absorption peaks at some positions [22].

Water Characteristic (Before, After) Separation:

Table 3 shows Water Characteristic before and after the separation the oil:

Table 3. Properties of water before & after the filtration process

Property	Oil/water mix	After separation				Distill water
		A	B	C	D	
Density g/cm^3	1.05	0.99	0.99	0.97	0.97	0.9
Viscosity Cp	0.98	0.97	0.96	0.96	0.98	6.9
Ph	6,54	6.6	6.73	6.7	3.3	5
Turbidity	27.1	13.8	8.4	6	4.5	4
Conductivity µSim/cm	12.25	19.27	8.63	5	5	0.96

Fig. 8. Shows the sample after the separation process.

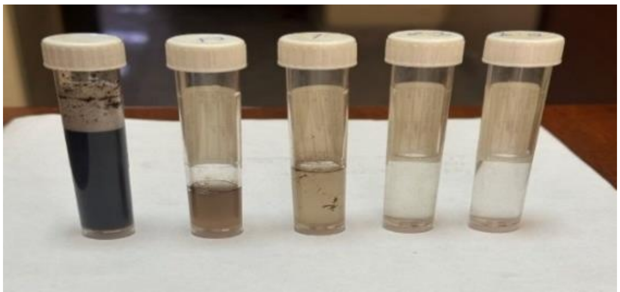
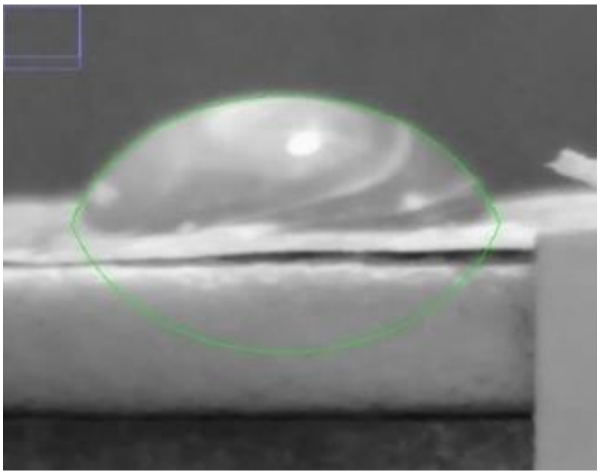


Figure 8. Water samples before, after separation process

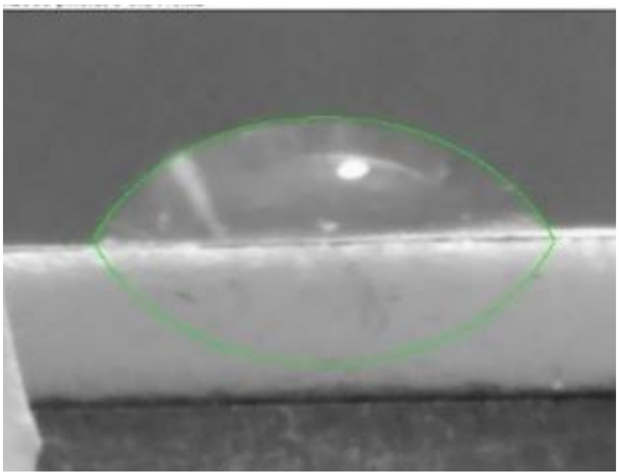
We observe that the oil and water are successfully separated using a nanofilter that has been created using an electrospinning technique, and the qualities of pure water are restored to their original state.

Contact Angle Analysis

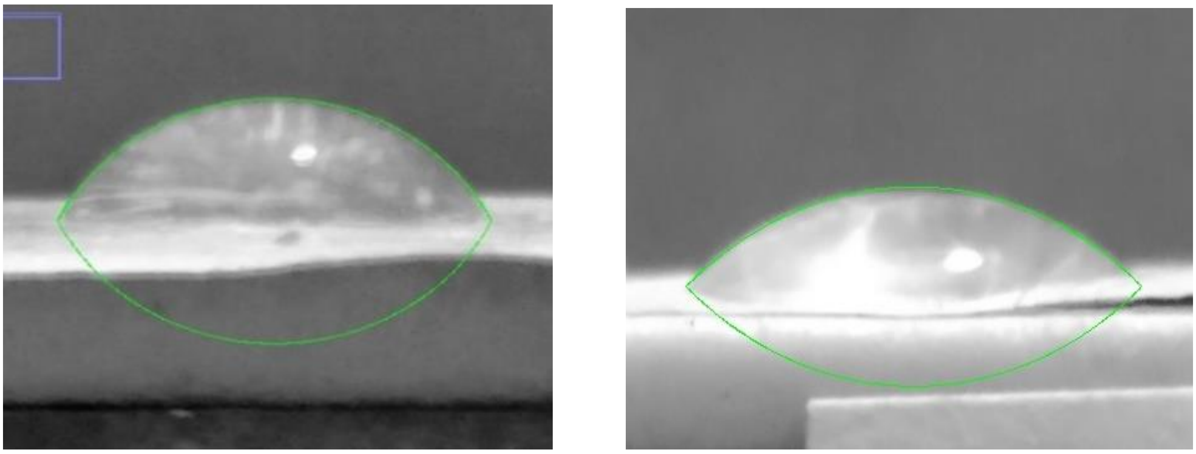
Fig. 9(a-d) shows the contact angle of sample A Nano filter (PA+PVA) is (72.12 ° C) and gradually decreases until it reaches 40.46 with sample D that consists of (PA+PVA+ 0.03 wt CNPs) nanofibers, this leads to high hydrophilic characterization with increasing percentage of Nano carbon, and this Nano filter is able to separate the oil from water with high efficiency[23].



9a. C.A of PVA+PA 72.123 °



9b. C.A of PVA+PA +0.01 CNPswt%: 66.543 °



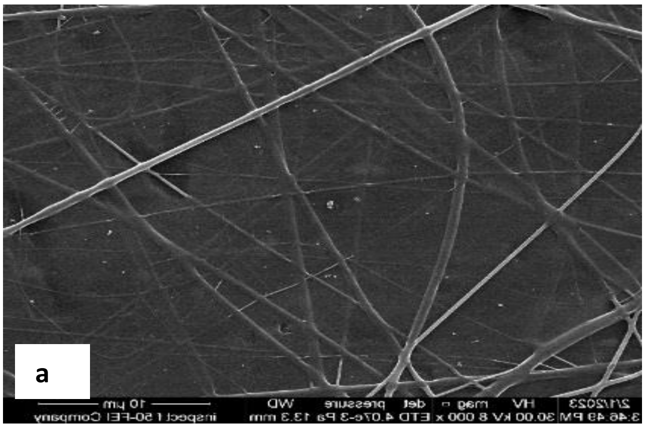
9c. C.A of PVA+PA+0.02 CNPs wt%:63.54 º

9d. C.A of PVA+PA+0.03 CNPs wt%:40.46 º

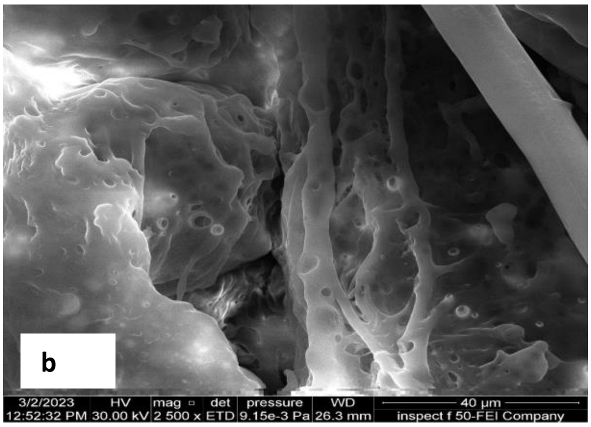
Figure 9(a-d). Contact Angle of nanofibers a. PVA+PA b. PVA+PA +0.01 CNPswt% c. PVA+PA +0.02 CNPs wt% d. PVA+PA +0.03 CNPs wt%

Morphology of Samples by SEM Images

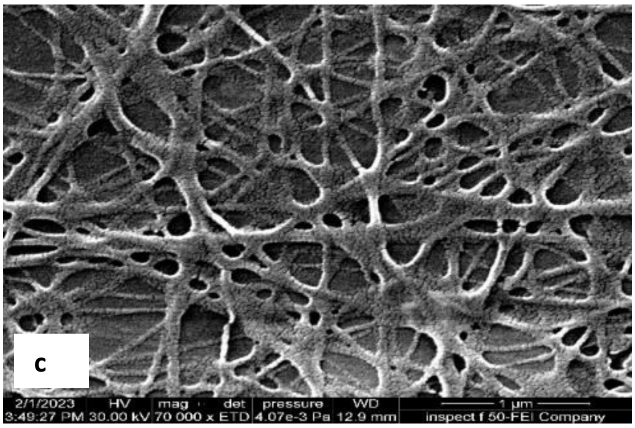
Different combination formations of the nanofibrous of polymers and NCP with various ratios were shown in the SEM pictures Fig. 10 (a-h), which revealed their various microstructures.



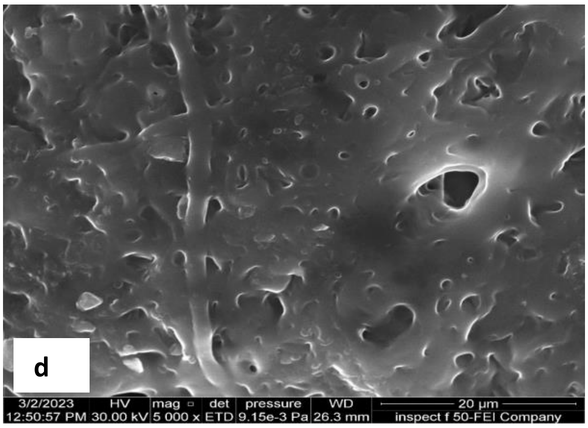
SEM of PA+PVA filter before fliration



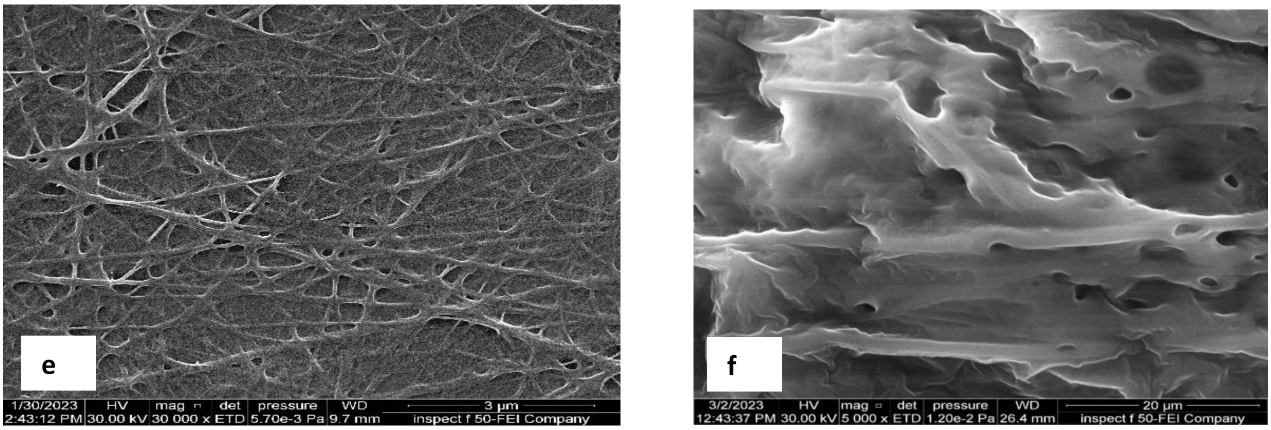
SEM of PA+PVA filter after fliration



SEM of PA+PVA+0.1wt% CNP filter before flirtation

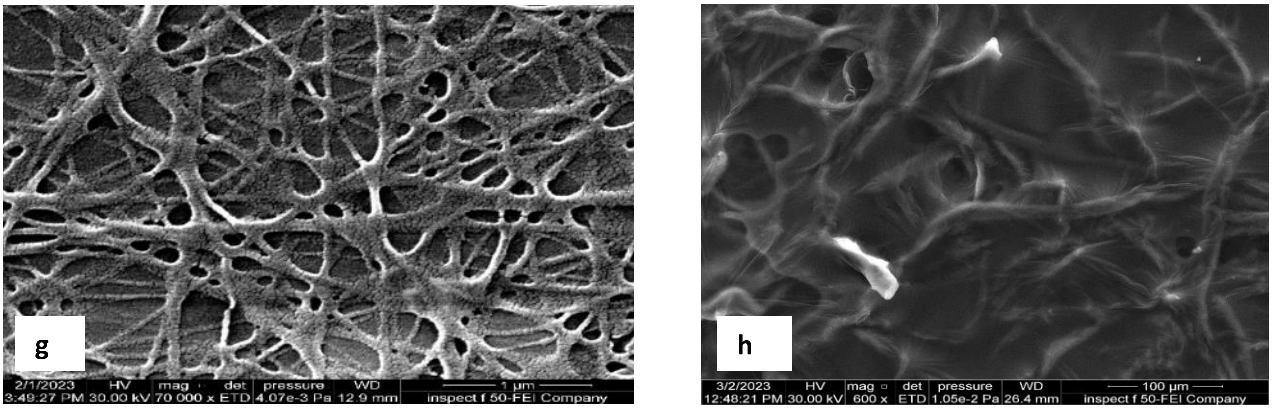


SEM of PA+PVA+0.1 wt% CNP filter after flirtation



SEM of PA+PVA + 0.2 wt% CNP filter before filtration

SEM of PA+PVA + 0.2 wt% CNP filter after flirtation



SEM of PA+PVA + 0.3 wt% CNP filter before flirtation

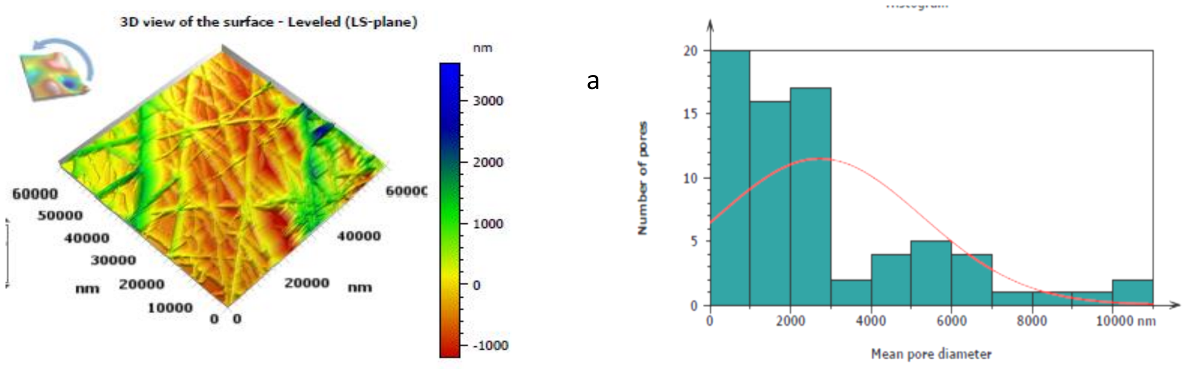
SEM of PA+PVA + 0.3 wt% CNP filter after flirtation

Figure 10. (a-h). SEM images of Nanofibers samples

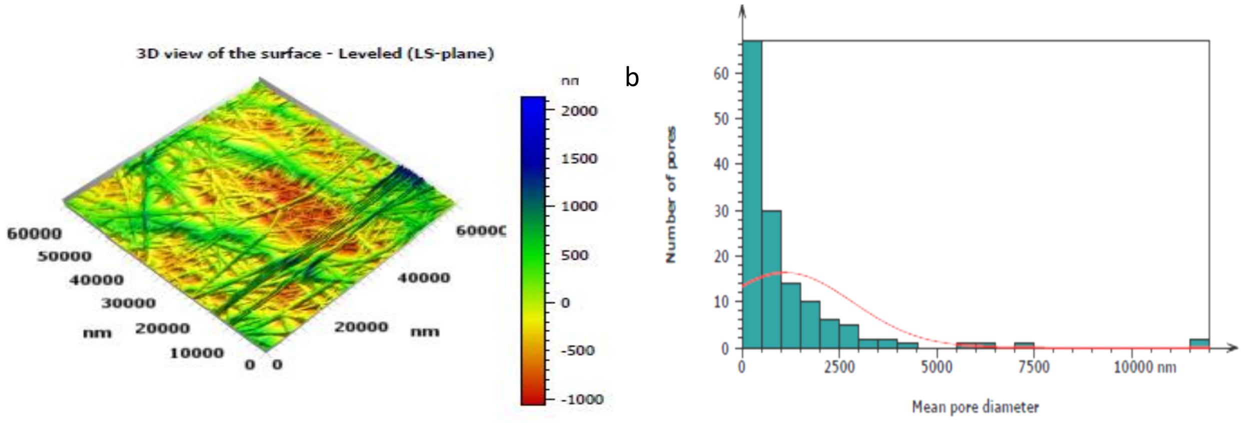
We found that the generated nanofibers have a porous structure; this is because the nanofibers were created by an electrospinning performed using immiscible blends. Due to the stability of the electrospinning method, low beads nanofibers were also generated. However, the oil molecules were properly bonded by the nanofiber filters, allowing water to pass through it for a container of water.

AFM and Porosity

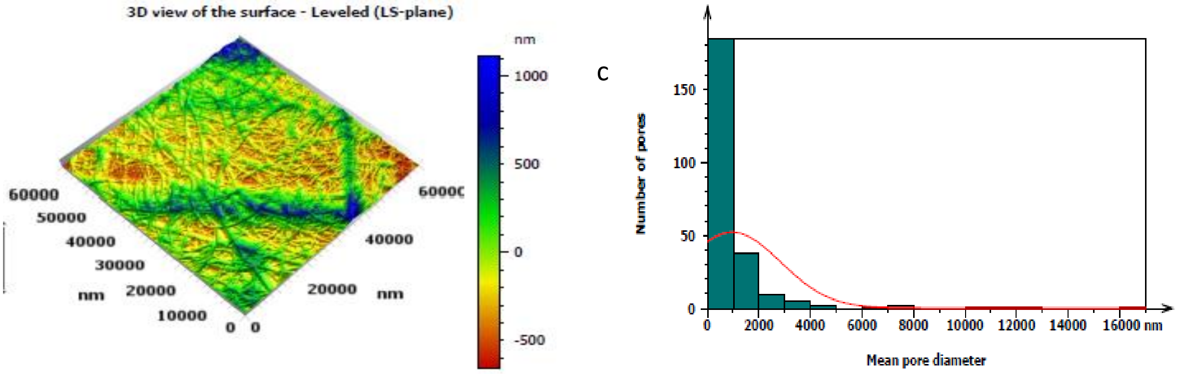
Fig. 11(a-d) shows the AFM and porosity distribution of filter nanofibers.



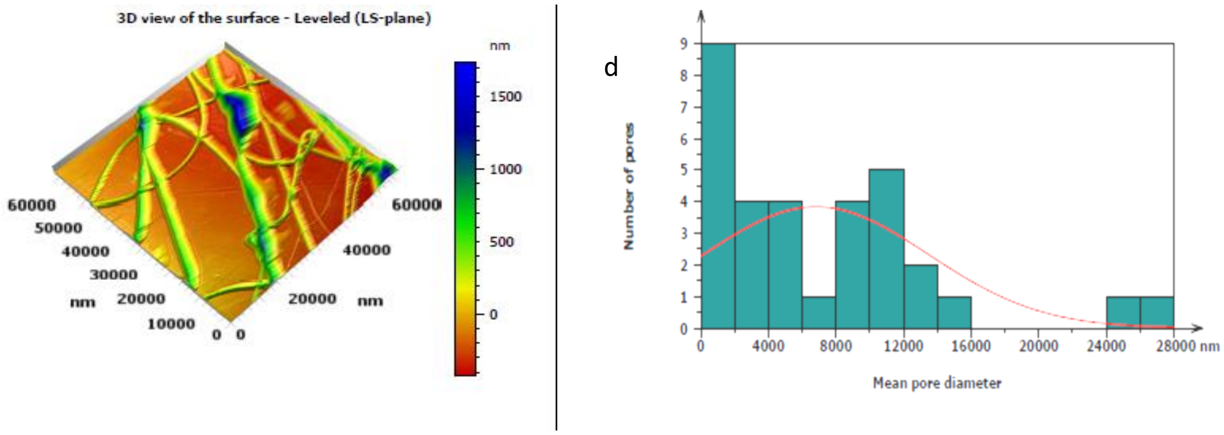
11a .AFM image and porosity histogram of Pure PVA + PA nanofibers



11b. AFM image and porosity histogram of PVA + PA+0.01 wt% nanofibers



11.c AFM image and porosity histogram of PVA + PA+0.02 wt% nanofiber



11.d AFM image and porosity histogram of PVA + PA+0.03 wt% nanofibers

Figure 11. AFM image and porosity histogram of nanofibers samples

Fig. 11. shows that the prepared pure PVA + PA nanofibers have porous structures with a porous diameter between a few nanometers and 2000 nm (2 µm) and a small number of porous with a diameter bigger than 2000 nm, while the diameter of the porous was decreased with adding (0.01 wt%) the carbon nanoparticles to a smaller value, about a small number of nanometers to less than 500 nm, a , Additionally, there exist a modest number of porous structure between 1000 nm and 2500 nm and between 500 nm and 1000 nm. When the amount of carbon nanoparticles is increased to 0.02 weight percent, the diameter of the pores decreases between a few nanometers to less than 1000 nm and a small

number to fewer than 2000 nm. The decrease in porous diameter that occurs when the percentage of carbon nanoparticles is increased is caused by the reduction in nanofiber diameter that results from raising solution conductivity and lowering viscosity [21-22]. Carbon nanoparticles act as lubricants, lubricate the solution layer, and reduce the viscosity of the mixture. Due to the agglomeration of carbon nanoparticles and a decrease in the conductivity of the solution and an increase in viscosity, which causes an increase in the fiber diameters, adding 0.03 weight percent of CNPs causes an increase in the porous diameter between some nanometers to 2000 nm and a number of porous diameter between 2000 - 6000 nm, 8000 nm, and 16000 nm [24-26].

Conclusion

According to the results from this study, nanofiber filters that were prepared by the electrospinning technique are effective in separating binary systems with different densities, like oil and water.

A blend of polyamide and polyvinyl alcohol armed with varying concentrations of carbon nanoparticles was used to create porous compositions of filters with various diameters. I concluded that the diameter of the porous decreased with an increase in the percentage of carbon particles to 0.02 % by weight, as well as, it increased with an increase in the number of carbon particles up to 0.03 % by weight. Water recovered its standard properties after the filtration process, the density dropped from 1.05 to 0.9 g/cm^3, and the mixture's viscosity slightly dropped but stayed around 0.9 cP. By using reinforcement ratios of carbon nanoparticles of 0, 0.01, 0.02, and 0.03 weight percentages, the turbidity is dramatically reduced from 27.1 to 13.8, 8, 4, 6, and 4.5 NTU, and is extremely near to the turbidity of pure water with a strengthening proportion of 0.03 weight percentages. Additionally, after filtration with filters made with carbon particle reinforcement rates of 0.02% and 0.03% by weight, the conductivity drastically drops from 12.25 to 5 µS/cm.

Acknowledgment

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Authors' Declaration

- Conflicts of Interest: None.
- We hereby confirm that all the Figures and Tables in the manuscript are ours. Furthermore, any Figures and images, that are not ours, have been included with the necessary permission for re-publication, which is attached to the manuscript.
- No animal studies are present in the manuscript.
- No human studies are present in the manuscript.
- Ethical Clearance: The project was approved by the local ethical committee at University of Babylon and University of Technology.

Authors' Contribution Statement

The contribution of each researcher was complementary to the contribution of the other researcher to complete this research in the final form.

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تحضير وتوصيف فلتر نسيج نانوي لفصل خليط الزيت / الماء باستخدام خلائط البولي امايد/ بولي فينيل الكحول المسلحة بجسيمات الكربون النانوية

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الخلاصة

تهدف هذه الدراسة إلى تحضير مرشح نسيجي نانوي باستخدام البولي امايد المتفرع 66 المحضر بطريقة بلمرة التكثيف البسيطة والذي يتم تقويته فيما بعد بدقائق الكاربون المحضرة من المخلفات النفطية . تم استخدام بلمرة الموقع لتحضير المتراكبات النانوية للبولي امايد 66 عن طريق بلمرة الموقع , تم تحضير خليط من البولي امايد المسلح بدقائق الكاربون النانوية مع محلول البولي فينيل الكحول , تم التدعيم بنسب مختلفة من الكاربون النانوي تضمنت 0.01 و 0.02و 0.03 نسبة وزنية مئوية . تم استخدام تقنية الغزل الكهربائي لتحضير فلتر الألياف النانوية في ظل ظروف محددة تتضمن (20 كيلو فولت كجهد مطبق ، ومسافة الضخ 15 سم ، 1 مل / ساعة كمعدل انسياب ، وقطر الابرة 0.4 مم). تم إجراء العديد من الاختبارات للمواد المحضرة بما في ذلك وفحص المجهر الإلكتروني الماسح ، وتحويل فورييه للأشعة تحت الحمراء ، زاوية الترطيب ، وتحليل حجم الجسيمات]. الماء / الزيت قبل عملية الترشيح وبعدها. وفقًا لبيانات ، تم إنتاج ألياف نانوية ناعمة ومتجانسة بقطر تقريبي 350 نانومتر وتم ملاحظة تشكيل تراكيب مسامية مفتوحة باستخدام تقنية الغزل الكهربائي . تم ملاحظة من صور المجهر الالكتروني الماسح ان الجزيئات السائلة تلتصق بألياف المرشح وتعمل على تقليص الفتحات الموجودة في الفلتر ، مما يسمح للماء بالمرور عبر الفلتر مع استعادة خصائصه ، وفقًا للنتائج التي تم الحصول عليها بعد عملية الترشيح. وفقًا لبيانات تم الحصول عليها يبلغ قطر غالبية المسام حوالي 100 نانومتر ، تقل المسامات الناشئة بين ليف واخر بزيادة نسبة الكاربون النانوي بسبب نقصان قطر الليف ، وتزداد اقطار الالياف في اكبر نسبة من الكاربون النانوي وتزداد معها قطر المسامات . تم ملاحظة ان الماء يستعيد خواصه الطبيعية بشكل كبير جدا بعد اجراء عملية الترشيح بالفلاتر المحضرة. توضح بيانات الأشعة تحت الحمراء أن هناك تفاعلًا فيزيائي بين الألياف البوليمرية وجزيئات الكربون المضافة نظرًا لوجود انخفاض في كثافة الأشعة تحت الحمراء بعد إضافة جزيئات الكربون إلى ألياف المرشح . اثبتت نتائج زاوية الترطيب ان للفلاتر خاصية المحبة للماء وتزداد مع زيادة نسبة الكربون النانوية حيث تقل زاوية الترطيب مع زيادة نسبة الكربون النانوية ، من 72 درجة مئوية لمرشح النانو النقي إلى 44 درجة مئوية للمرشح مع 0.3 نسبة مئوية من دقائق الكربون النانوية.

الكلمات المفتاحية: الغزل الكهربائي، بلمرة الموقع، دقائق الكاربون النانوية، فلترة، متراكبات نانوية.