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Crude Oil Desulfurization by Polystyrene/Graphene Nanocomposite Membranes

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Abstract

The main aim of this paper is to evaluate the effect of graphene nanoplatelets (GNPs) on the desulfurization of the polystyrene nanofibers. Hence, different loadings of GNP, 0.5, 1, 1.5, and 2 wt.% GNP was incorporated into the polystyrene nanofibers using electrospinning technique. Several characteristics of the electrospun membranes were investigated. The obtained field emission scanning electron microscopy (FE-SEM) images confirmed the formation of uniform fibers and proper distribution of the particles in the electrospun structures. In addition, thinner fibers with smaller pore sizes were formed by addition of the GNPs into the nanofibrous mats. Moreover, the hydrophobic behavior reduced by increment of GNP content in the polystyrene nanofibers. Fourier transform infrared spectra (FTIR) confirmed the interaction of the polymer chains with the loaded filler particles. Furthermore, introduction of the GNP nanofillers in the decrease of the crude oil sulfur content from 2.8% to 2.6% *via* addition of 0.5 wt.% of GNPs into the as-spun fibers. Further reduction of the sulfur content (to 1.8%) was also obtained by embedding 2 wt.% of GNPs. Overall, the obtained data manifested that the electrospinning composite membranes are good candidate material for removing sulfur from crude oil.

Keywords

composite membranes, electrospinning technology, graphene nanoplatelets, adsorption, desulfurization

This article was originally published with an error. This version has been corrected/amended in the Corrigendum. Please see the Corrigendum (https://doi.org/10.3311/PPch.40074)!

1 Introduction

Since the first usage of polymer nanocomposites, they have been gaining attention as the most important category of composite materials due to superior material characteristics [1]. Polymer-based nanocomposites have displayed significant enhancement in various aspects of material properties [2–6]. Introduction of nanomaterials into the polymer composites leads to reduction of the final material weight, as well as facilitation of the preparation process in comparison with addition of the micro-sized ones (e.g., carbon or glass fibers) [7]. The aforementioned valuable characteristics of the polymer nanocomposites have caused progress of their applications in numerous fields [8]. In recent years, different types of nano-additives were incorporated into the polymer-based materials [9]. Among them, graphene-based structures have appropriate mechanical, electrical and thermal characteristics due to their great aspect ratio [10–14]. Graphene is one of the most usage allotrope of carbon. This nanomaterial arranges in two dimensions which formed a lattice structure as a monolayer with sp^2-hybridized [15, 16]. Since the discovery of this wonder nanostructure in 2004, studies on it and its derivatives have drastically enhanced [17, 18]. Regarding to the mentioned desirable features, they have been widely used as nano filler in nanocomposite materials such as solar cells [19], sensors [20], hydrogen storage [21], and capacitors [22].

Polymer nanocomposites is recently employed to fabricate membranes used to achieve various purposes. One of the applications of polymer-based membrane is desulfurization. Desulfurization is a method communally applied for reduction of sulfur content in crude oil and overcome its adverse action. Sulfur is known as an impurity in crude

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oil which has resulted in air pollution in several countries, and its components are unwanted in fuels because its undesirable effects that may cause many issues in various refining stages. In desulfurization process, an absorbent material leads to sulfur removal through physical or chemical reactions [23]. It has been widely shown that materials with more active surfaces, higher specific surface area and proper pore sizes could act as more ideal adsorbents [24]. Desulfurization techniques by adsorption are more adequate since it is providing a high removal efficiency with little operating requirements, several lines of evidence suggest that activated carbon [25], zeolites [26], sludge [23], and bentonite [27] could be applied as versatile adsorbent materials. During desulfurization procedure, adsorption complexes could be formed through π–π interacting between sulfur components and applied adsorbent material.

Compared with van der Waals interactions, the π–π stacking reveals stronger binding. However, such interactions could be easily broken resulting from temperature increment or pressure decrement leading to regeneration of the adsorbent material [26]. Several researchers claimed modification and enhancement of the sulfur removal through incorporation of a cation into the adsorbent materials [28]. Alhamed and Bamufleh [29] represented a model for desulfurizing of diesel by utilizing carbon-based adsorbent material modified with zinc chloride cations. In another attempt, Shah et al. [30] confirmed superior desulfurizing of diesel by embedment of tin cation into the activated carbon adsorbent compared with that of the untreated activated carbon.

Electrospinning has been introduced as a promising technology for fabrication of continuous fibers in a wide tunable diameter range from nano to micro scales [31]. Application of electrospun fibers in various fields has been evaluated mainly due to high aspect ratio of the nano-sized fibers [32]. Tissue engineering [33, 34], exchanger proton membranes [35], filtration [36], electrochemical devices [37, 38], and solid phase microextraction (SPME) applications [39, 40] are of the well-known application fields of the electrospun fibers.

In this study, polystyrene membranes consisting of electrospun nanofibers incorporated with different amount of graphene nanoplatelet (GNP) fillers have been investigated. The membranes were fabricated through a standard electrospinning technology. These membranes were characterized under different tests to explore their properties. Then, the potential of the as-prepared membranes was employed as adsorbent materials for reduction of sulfur content in crude oil.

2 Experiment

2.1 Materials

Polystyrene with an average molecular weight M_w = 250,000 g mol^{-1} was provided from Sigma Aldrich (USA). N,N-dimethylformamide (DMF) was purchased from Central Drug House Ltd. (India). GNPs with an average lateral size of 15 nm and a thickness of 6–8 nm were provided from SkySpring Nanomaterials, Inc. (Canada).

2.2 Membrane preparation

In this study, electrospinning technique was employed to fabricate the membranes. To fabricate the membrane, the following steps were performed. First, polystyrene polymer was added to DMF solvent with a constant concentration of 20 wt.%. The mixture was prepared by stirring vigorously for 2 h at lab temperature to obtain a homogenous polymeric solution. Then, 0.5, 1.0, 1.5, and 2 wt.% of GNP was added to the obtained solutions. The GNPs were dispersed in the as-prepared polymeric solutions through magnetic stirring for 30 min. Subsequently, the suspension was sonicated (Model 300VT Ultrasonic Homogenizer, USA) for 3 min. An electrospinning device (Bio-electrospinning/electrospray system ESB-200, South Korea) was applied to fabricate the electrospun fibers from the prepared solutions [41]. The electrospinning solutions were placed in 10 mL syringe. A stainless-steel needle (22 gauge) was conjugated with the syringe. The polymeric solutions were fed by the syringe pump to the electrospinning electrical field with a constant feed-rate of 1 mL h^{-1}. The collector was placed at a distance of about 15 cm from the syringe needle. The electrical field (about 20 kV) between the syringe needle and collector was generated by a high power supply. The electrospinning process was continued for 2 h for fabricating the membranes, as illustrated in Fig. 1 [42].

2.3 Characterization

Field emission scanning electron microscopy (FE-SEM) device (MIRA 3-XMU) was utilized to examine the fiber morphology, porosity, size distribution, and the fibers surface roughness of the electrospun polystyrene nanofibers membranes, by taking images at higher magnification. Histograms were obtained by using image analyzer [43] and Auto CAD software [44] to illustrate the average fiber diameter and average porosity. SEM-EDX device (MIRA 3-XMU) was employed for estimating chemical composition of the electrospun polystyrene membranes. FT-IR device (BRUKER, TENSOR-27) to identify

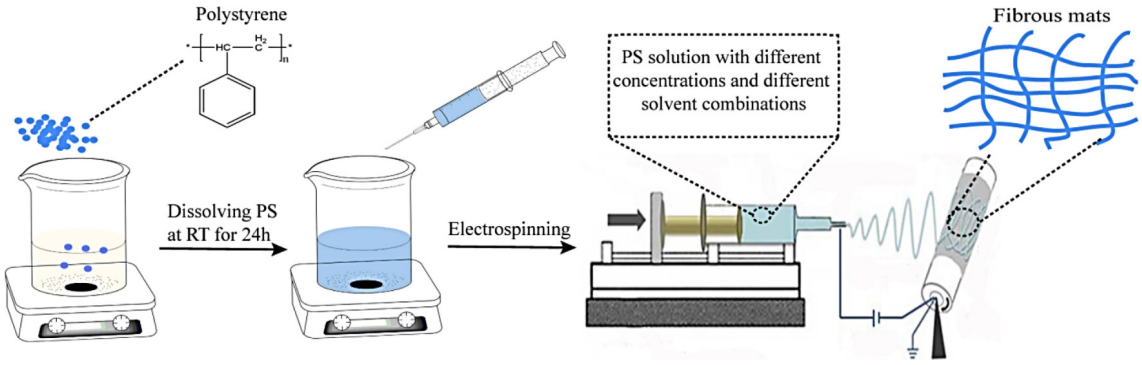

Fig. 1 Polystyrene membrane manufacturing by electrospinning following Ref. [42]

the chemical bonds within the GSP filled polystyrene membranes. The wettability of the membranes was measured through estimation of contact angle between water/crude oil and the as-prepared membranes using a contact angle instrument (CAM 110, Germany). During this examination, water and crude oil were automatically dispensed from a syringe onto the membrane surface and the device analyzed the taken image for the contact angle in about 3 sec. At the end, to evaluate the sulfur content in crude oil before and after passing through the prepared electrospun composites, a sulfur analyzer device (XRF-EDX 3000, Sindie OTG) was used.

3 Results and discussion

3.1 Morphology of the electrospun fibers

The surface morphology of the electrospun polystyrene fibers is shown in Fig. 2. As observed, the as-spun fibers exhibited a bead-on-string structure. This may be linked with the low polymer concentration. So, the incomplete entanglement of the polymer chains led to breaking of the solution components, and therefore formation of droplets (not fibers) during the electrospinning procedure [45].

The fiber diameter distribution and the pore size histogram of the polystyrene fibers are displayed in Fig. 3. As it is apparent, the electrospun membrane contains fibers with the average diameter of 1.68 ± 0.02 μm and mean pore size of 1.28 ± 0.02 μm. The as-spun fibers were selected for fabrication of composites due to several advantages such as high surface area, thin fibers, high porosity, and simple spinning process.

Fig. 4 illustrates the morphology of the electrsopun polystyrene fibers with incorporated GNP filler. As observed, the bead-shaped deformations are significantly reduced. This could be attributed to the improvement of the solution conductivity which has resulted in the formation of more uniform fibers. In other words, increment of the elongation

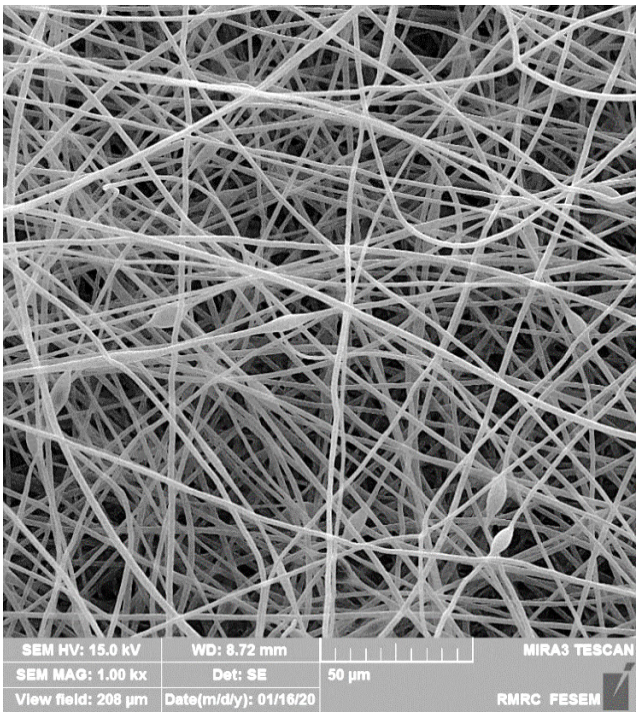

Fig. 2 FE-SEM image of the electrospun polystyrene membrane

force and subsequently the elongation rate resulted finer fibers without any beads in their structures [46, 47]. These results are well displayed in Figs. 5 and 6. As observed, by increasing the GNP content, both average fiber diameter and average pore size were degraded remarkably. For example, by incorporating 2 wt.% of GNPs into polystyrene fibers, the average diameter of the fiber is reduced from 1.7 to 0.89 ± 0.02 μm. The obtained results in the current research are in good agreement with the study carried out by Li et al. [48].

According to [48], the morphology of the neat polystyrene fibers differs from that of the polystyrene/GNP ones. Adding GNP filler to the as-spun membrane, the fabricated structure changes from a smooth structure to a rough membrane due to the proper dispersion of the GPN additive in the

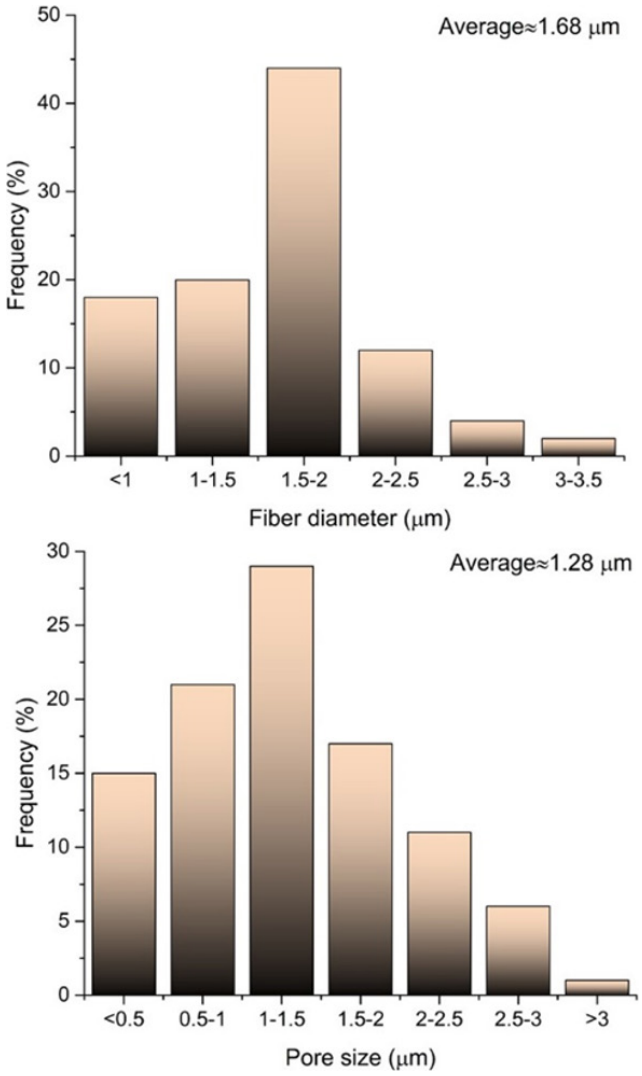

Fig. 3 Fiber diameter and pore size distribution histograms of the electrospun polystyrene

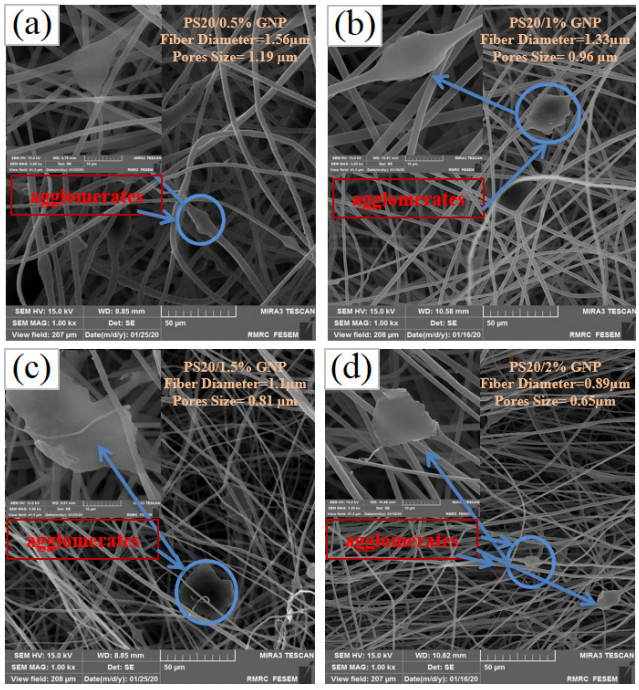

Fig. 4 FE-SEM images of the polystyrene membranes loaded by various concentrations of GNP filler (a) 0.5, (b) 1, (c) 1.5, and (d) 2 wt.%

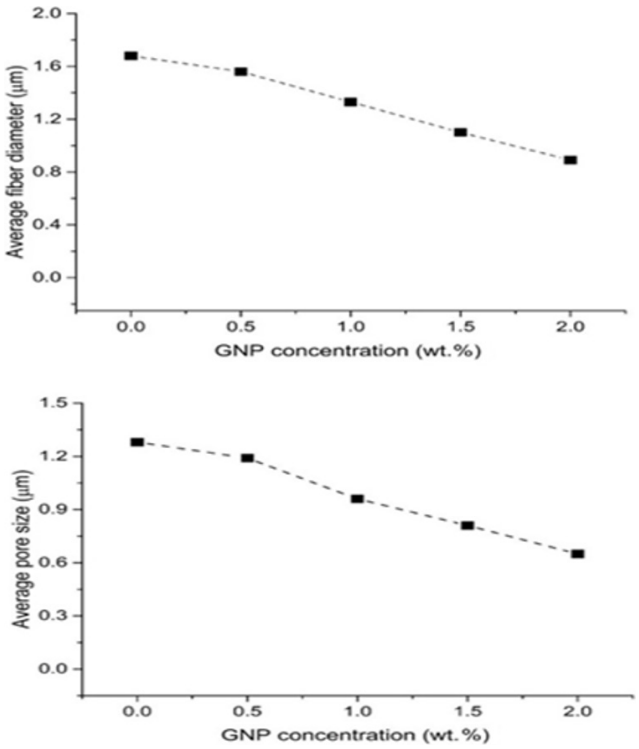

Fig. 5 The mean fiber diameter and pore size for the as-spun fibers with various loadings of GNP filler

polystyrene matrix [48]. It is well observed that by increasing the GNP content, the roughness of the polystyrene fiber surface is significantly increased (refer to Fig. 7).

3.2 Energy dispersive X-ray spectroscopy

The result of the energy dispersive analysis (EDS) of the electrospun polystyrene fibers is demonstrated in Fig. 8.

According to the obtained spectrum, low energy peaks appeared in the range of 0.1 to 0.6 keV. The aforementioned peaks are attributed to carbon and oxygen, respectively. In addition, the observable peaks in the range of 1.5 to 2.2 keV correspond to the elemental gold applied for coating the membranes.

Fig. 9 (a) and (b) display the EDS spectra of the electrospun polystyrene membranes with different loadings of GNP filler. Increasing the GNPs concentration from 0.5 to 2 wt.% in the electrospun fibers, the intensity of carbon was enhanced from 2500 to 5000 and this result confirm that carbon is the main component in the membrane, also oxygen was present confirming existence of the GNP filler in the as-spun fibers and the reason of existing was from environmental conditions. Gold present in EDS spectra is

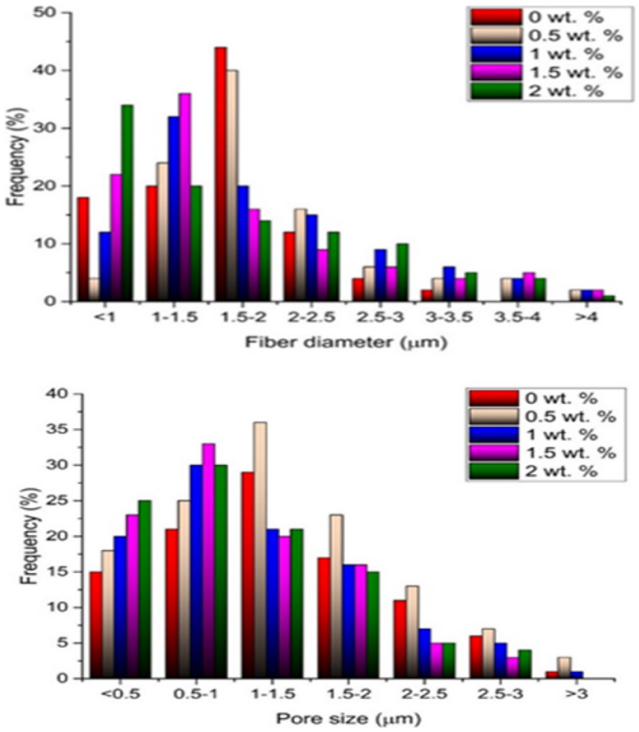

Fig. 6 Fiber diameter and pore size distribution histograms for the as-spun fibers incorporated with various loadings of GNP filler

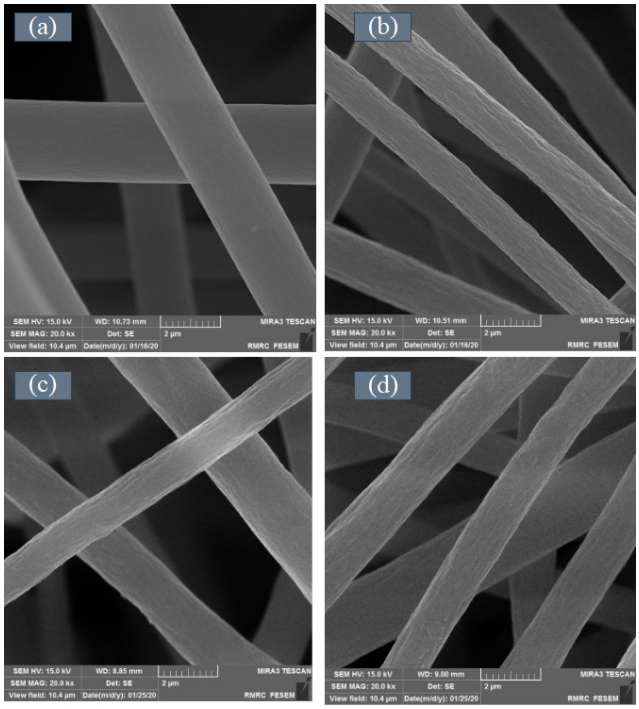

Fig. 7 Effect of GNP content of (a) 0.5, (b) 1, (c) 1.5, and (d) 2 wt.% on the roughness of the polystyrene fibers taken by FE-SEM

related to coating the electrospun membranes with a conductive layer to produce high resolution FE-SEM images.

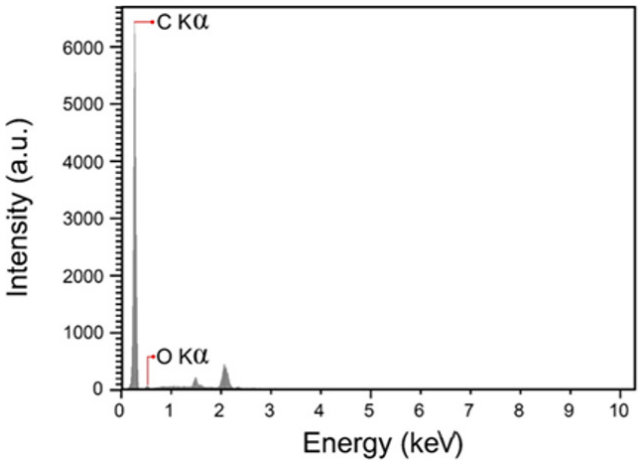

Fig. 8 EDS spectra of the electrospun polystyrene membrane

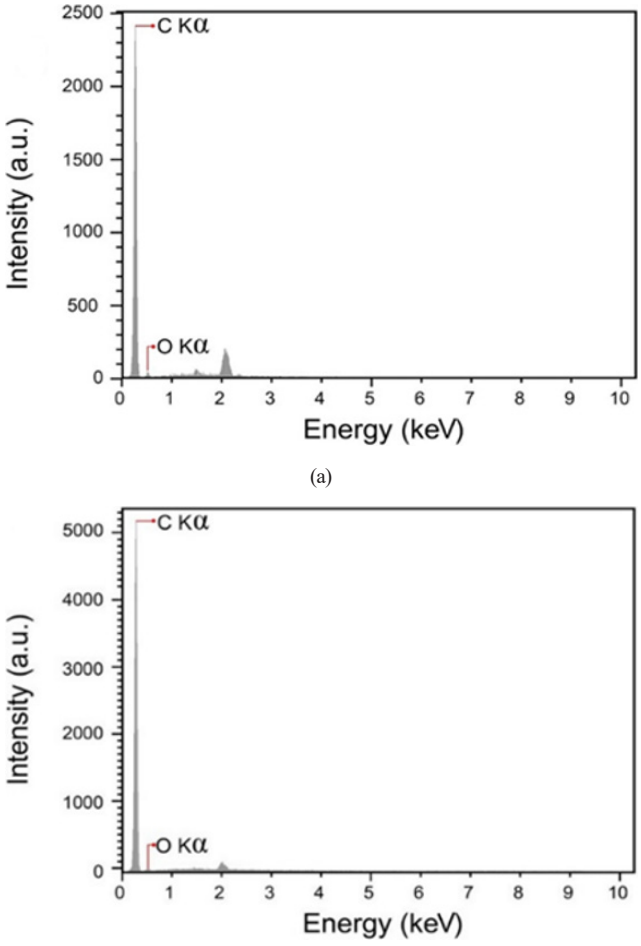

Fig. 9 EDS spectra of the polystyrene membranes incorporated with different GNP concentrations (a) 0.5 and (b) 2 wt.%

3.3 Fourier transform infrared spectroscopy

FTIR spectra of the polystyrene nanofibrous mat is displayed in Fig. 10. According to the obtained spectra,

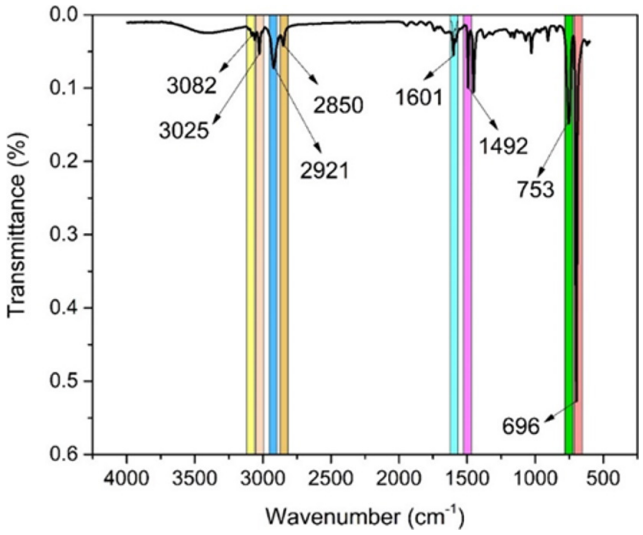

Fig. 10 FTIR spectra of the electrospun polystyrene membrane

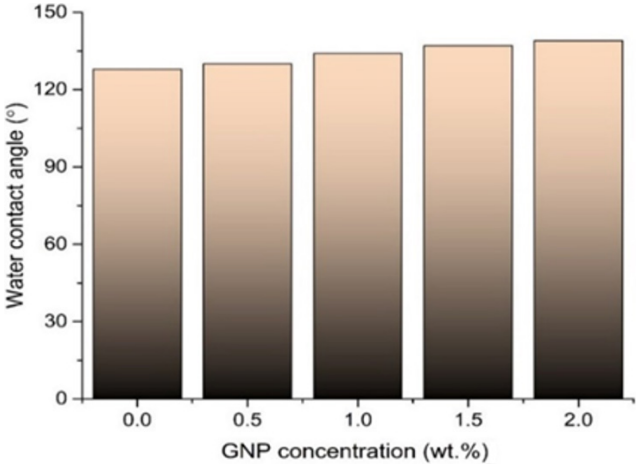

Fig. 12 The effect of GNP concentrations on the measured contact angle property

aromatic C–H bond stretching vibration was appeared in the wave numbers of 3082 cm^{-1} and 3025 cm^{-1}. In addition, aromatic C=C bond stretching vibration is observed at 1601 cm^{-1}, 1492 cm^{-1}, and 1451 cm^{-1}, which indicates the existence of the benzene rings. Moreover, the transmittance peaks at the wave numbers of 753 cm^{-1} and 696 cm^{-1} correspond to C–H out-of-plane bending vibration, confirming that there is only one substituent in the benzene ring. Furthermore, the peaks at the wave numbers of 2921 cm^{-1} and 2850 cm^{-1} are linked to the methylene group which is tied well with the observations of Fang et al. [49].

Fig. 11 shows the FTIR spectra of the electrospun polystyrene fibers incorporated with various amounts of GNP filler. In general, the peak intensities were increased by loading more filler particles into the electrospun fibers, specifically in the cases of C–H out-of-plane bending and aromatic C=C stretching vibrations. In addition, C–H out-of-plane bending vibration was shifted from 753 cm^{-1} to 750 cm^{-1} by incorporation of 2 wt.% GNP filler. Based on Chayad et al. [50] this could be referred to locating the carbon atoms in structure of the polystyrene fibers.

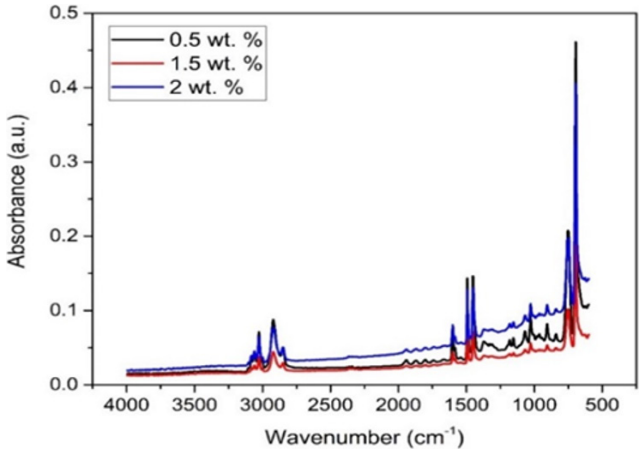

Fig. 11 FTIR spectra of the electrospun polystyrene fibers loaded by various concentrations of GNP

3.4 Contact angle measurement

The effect of GNP introduction into the electrospun fibers on the contact angle is been presented in Fig. 12. Apparently, the contact angle was increased from 128° to 139° by addition of 2 wt.% of GNP to the nanofibrous mats. The observed trend of the contact angle feature could be attributed to the effect of GNPs on the morphology of the fabricated fibers. In fact, in the polystyrene/GNP, the surface roughness of the fibers enhanced significantly. Subsequently, a lower surface energy was resulted in the electrospun membranes. Similar behaviors were obtained by the other researchers [51–53]. Notably, polystyrene/GNP membranes showed hydrophobic behavior. However, they represented high affinity for the crude oil. Therefore, a value of zero was obtained for contact angle in the case of using crude oil droplet instead of water.

3.5 Sulfur analyzer

The influence of the GNP concentration on the sulfur removal from the crude oil is illustrated in Fig. 13. Based upon the obtained results, the sulfur content declined from 2.8% to 2.55% *via* addition of 0.5 wt.% GNP into the as-spun fibers. Further reduction of the sulfur content (1.8%) was also approached by embedding 2 wt.% of GNP into the electrospun mat. Hence, desulfurization efficiency was enhanced from 4.3 to 35.7% by incorporation of more GNP nanofillers into the electrospun fibers. This

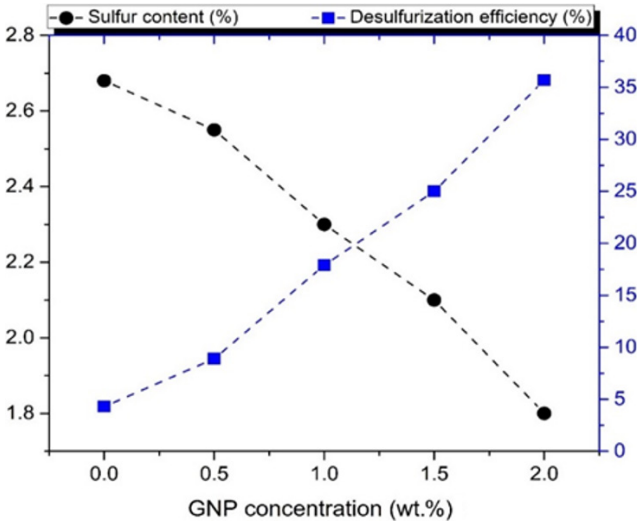

Fig. 13 Sulfur content and desulfurization efficiency of various electrospun membranes

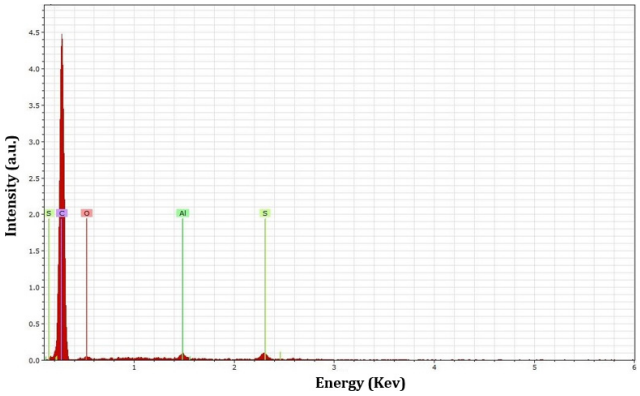

Fig. 14 EDS analysis of the polystyrene fibers after passing through crude oil

could be due to the sp^2 configuration of the GNP additives which results in $\pi-\pi$ interactions and therefore improves the desulfurization efficiency [54–57].

Fig. 14 shows the EDS analysis of the electrospun polystyrene membrane after passing through the crude oil. This test confirms which types of contamination the electrospun polystyrene membrane can capture. As observable, the as-examined membrane contained elemental sulfur and aluminum.

Fig. 15 demonstrates the EDS analysis of the electrospun mat when incorporated with 2 wt.% GNP after passing through the crude oil. As displayed, the as-examined membrane capturing ability enhanced with incorporated with

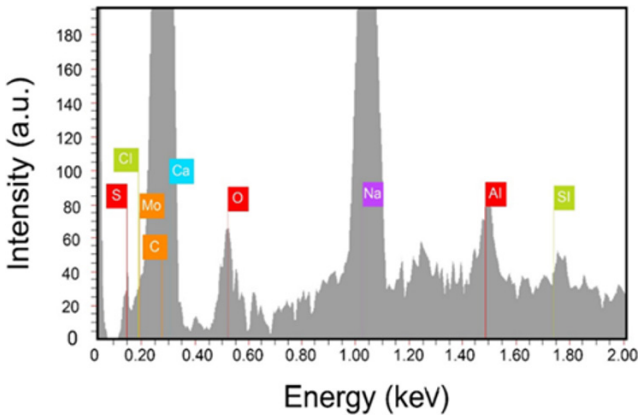

Fig. 15 EDS analysis of the polystyrene fibers loaded by 2 wt.% GNP filler after passing through crude oil

graphene and can capture several elements (sulfur, sodium, calcium, chloride, molbidium, aluminum and silicon) compared to the electrospun membranes without additives.

4 Conclusion

In summary, electrospun polystyrene fibers incorporated with various ratios of the GNP were successfully fabricated through a standard electrospinning technique. Various features of the electrospun membranes were evaluated.

The results showed the formation of fine and uniform composite fibers along with homogenous distribution of the loaded GNP particles in the fiber structures. In addition, finer fibers, smaller pore size, and rougher surfaces were created by increasing the GNP amount in the electrospun fibers. Moreover, introduction of 2 wt.% GNP filler into the electrospun fibers led to increase the hydrophobicity from 128° to 139°. Furthermore, the sulfur content decreases from 2.8% to 2.6% in the presence of 0.5 wt.% of GNPs. Further reduction of the sulfur content was obtained by incorporating 2 wt.% of GNP into the electrospun polystyrene fibers, resulting in enhancing desulfurization efficiency from 4.3 to 35.7%. At the end, these fabricated membranes work well as filters, reducing the percentage of sulfur in crude oil in places where the sulfur content is very high.

Acknowledgement

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