Research Article

Electrospinning of polystyrene polybutadiene copolymer for oil spill removal



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Abstract

Cleanup of spilled oil is a difficult process, and it depends on various factors, including the viscosity and the type of the oil spilled, the temperature and the type of water. Oil sorbent nanofibers were prepared from polystyrene polybutadiene copolymer (PSBR) by the electrospinning process using dimethylformamide (DMF), tetrahydrofuran (THF) or mixture of THF and DMF. The fibers surface morphology, crystallinity, hydrophobicity and the sorption capacity, reusability over potable water and seawater were studied. The electrospinning of PSBR in THF produced a structure having a combination of fibers, beads and cup-shaped structures, while the electrospun nanofibers from DMF have more uniform shape and structure. It was found that the average PSBR fibers diameters formed from 10 wt% in THF, DMF and mixture of THF and DMF with the ratio of 50:50 are 80, 1200 and 900 nm, respectively. The results revealed that the contact angle of PSBR fibrous sorbent increased by increasing the concentration of electrospun polymer solution and reached 153 degrees. Moreover, the sorption capacity for crude oil, gas oil and motor oil was 50, 62 and 125 g/g, respectively. The sorption capacity of PSBR nanofibers is higher than regular polystyrene (PS) nanofibers making it a highly promising material for oily water purification.

Keywords Polystyrene · Electrospinning · Nanofibers · Oil spill · Superhydrophobic

1 Introduction

Oil spill is one of the major sources of water contamination. Crude and fuel oils spill may occur due to the industrial accidents or oil transport accidents. Disastrous oil spills prompt the necessity to develop a friendly system and cost-effective technology to reduce the environmental impacts caused by such oil spills [1]. There are some common practical methods of the oil spill cleanup by using mechanical recovery, chemical treatment methods [2, 3] and sorbents [4, 5] which are considered as another class of materials that are able to soak up oil spills from water. The primary advantage of sorbents is the convenience removal of oil layer from contaminated area. However, it is known that oil-containing sorbents should be collected for oil recovery or for further eliminations. Fibrous sorbents should not be hydrophilic to avoid the sorption of water. The ideal fibrous sorbents have several characteristics including high oil selectivity, convenient application and transportation of fibers, cost-effective, nontoxic and harmfulness to the environment [6]. The sorbents used for oil sorption are classified as natural materials and organic synthetic fibers. The natural materials include milkweed, cotton and kapok which showed low hydrophobicity and a relatively high oil sorption capacity ranging 20–50 g/g [7].

Polymeric nanofibers have been fabricated by different techniques such as drawing [8], template synthesis [9], phase separation [10] and electrospinning [11]. The electrospinning technique is utilized for continuous fine fibers production (with inner diameters of 10 μ m down

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to 10 nm) by allowing a polymer solution or melt with an electrical driving force. The advantages of this technique are the low cost, relative easiness, the high versatility by controlling over the fiber diameter. Some phenomena such as the formation of beads and branched jet were reported to occur [12]. Zhu et al. [13] reported the use of electrospun polyvinyl chloride /polystyrene nanofibers as sorbent for oil removal, which revealed many advantages compared with commercial PP nonwoven fabric. The sorption capacities of PVC/PS sorbent for motor oil and diesel were 146 and 38 g/g, respectively. It was about 59 times that of a commercial PP sorbent. Alnaqbi et al. [14] investigated and studied sorbents of pure and recycled PS in the form of microfibers. These fibrous sorbents achieved a sorption capacity as high as 95 g/g of the crude oil spills.

In the current work, a low-cost, simple, versatile process for the preparation of superoleophilic and superhydrophobic by electrospinning process is carried out. The hydrophilicity and the oil sorption capacities of the prepared PSBR fibrous sorbents for gas oil, motor oil and crude oil for potable and seawater were evaluated. Additionally, the mechanism of oil sorption by fibers was discussed based on SEM analysis.

2 Materials and methods

2.1 Materials

Polystyrene Polybutadiene rubber (PSBR) pellets with Mwt = 275,000 g/mol and density = 1.04 g/cm³ and extruded film of thickness = 1 mm were obtained from Estyrenics, Egypt. Tetrahydrofuran (THF) (purity > 99.9%) was purchased from Sigma-Aldrich, Germany. Dimethylformamide (DMF) (purity > 99.8%) was purchased from Honeywell, Germany. Gas oil (density = 0.878 g/cm³, viscosity = 8.5 cSt at 40 °C) was supplied by Alexandria mineral oils company (AMOC), Egypt. Crude oil (density = 0.867 g/cm³, viscosity = 6.73 cSt at 40 °C) was supplied by Alexandria company for petroleum additives (ACPA), Egypt. Motor oil (density = 0.886 g/cm³, viscosity = 79.1 cSt at 40 °C) was purchased from Shell, Egypt.

2.2 Methods

2.2.1 Electrospinning of PSBR

PSBR solutions with different concentrations (10 wt%, 20 wt% and 30 wt%) were dissolved in THF, DMF and mixture of THF and DMF with a ratio of 50:50, these solutions were fed with a constant rate of 2 mL/h into a needle with a diameter of 0.5 mm by using a syringe pump (New Era Pump Systems, Inc. USA), the distance between the

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2.2.2 Characterization of PSBR nanofibers

FTIR spectrophotometer (Spectrum BX 11 spectrometer FTIR LX 18–5255 Perkin Elmer, USA) was used for characterizing the electrospun PSBR nanofibers. X-ray diffraction patterns for PSBR film and electrospun nanofibers were performed using (X-ray 7000 Shimadzu, Japan) at room temperature. The Bragg's angle (2θ) has the range from 2 to 35 degrees, to determine the degree of crystallinity of the samples. The X-ray source was Cu target generated at 40 kV and 30 mA with scan speed 12 deg/min. The surface morphology of the electrospun PSBR nanofibers was studied using the scanning electron microscope (SEM) (Joel JSM 5300, Japan). The samples of the electrospun nanofibers were cut under liquid nitrogen and placed on a brass plate. The nanofibers were then sputter-coated with a thin film of gold prior to the SEM investigation.

2.2.3 Contact angle measurements

The contact angle of the nanofibers surface was measured using a contact angle goniometer instrument of (Rame hart, Company, France). The sample coupons were with surface area of 5 cm \times 1.5 cm and were prepared by selecting, cutting pieces from different positions from the nanofibers sheet. The samples were placed on a glass slide plate and mounted with a tape. Then, one drop of distilled water (2µL) was placed on the surface of the sample by using a micro-syringe (Hamilton Company, Reno, NV). The contact angle was measured within a 10 s after the water drop was placed at 5 different spots.

2.2.4 Oil sorption capacity measurement

In a typical sorption experiment, 10 mL of the different oils (gas oil, motor oil and crude oil) was poured into a 250-mL beaker containing 90 mL of potable or seawater. A constant weight of the PSBR nanofibers sorbent (0.1 g) was then placed freely on the oil surface. After multiple time intervals 0.5, 5, 10, 15 till 30 min, the sorbent containing the adsorbed oil was collected, drained for 2 s. and placed on a watch glass for weighing as shown in Fig. 1. These experiments were performed at ambient temperature, and the weighing was performed by using an analytical balance with up to 0.01 mg (5 decimal places) readability. The



Fig. 1 The oil sorption capacity test procedures for crude oil, **a** weighing of the sorbent, **b** placing fiber in a beaker containing the oil and water, **c** the fiber after absorbing the oil through 30 min

oil sorption capacity (C) in g/g of dry sorbent was determined by Eq. (1) [14]:

$$C = \frac{M_{\rm t} - M_{\rm d}}{M_{\rm d}} \tag{1}$$

where M_t is the total mass of the sorbent after the oil drainage and M_d is the mass of the dry sorbent.

3 Results and discussion

3.1 Structural property and crystallinity of PSBR nanofibers

As shown in Fig. 2a, polystyrene bands are identified at their corresponding wavenumbers listed in the literature. The IR band of PSBR nanofibers at 3060–2850 cm⁻¹ corresponds to the stretching mode of aromatic CH, 1944–1803 cm⁻¹ corresponds to the CH asymmetric stretching mode and 1475–1360 cm⁻¹ is assigned to the stretching mode of aromatic C=C double bond. The sharp peak at 966 cm⁻¹ is attributed to the isomerized cis-polybutadiene band of hydrogen out of plane vibrations of cis-CH=CH groups [15].

Figure 2b, c shows the diffraction patterns of PSBR film and nanofibers. There are two broad peaks centered at 2θ of 10° and 18° for PSBR film and at 10° and 20° for PSBR nanofibers. The small shift in the second broad peaks has been attributed to the rapid vaporization of the solvent during the electrospinning process which did not allow sufficient time for ordering of the molecules,



Fig. 2 a The FTIR spectrum of the PSBR nanofibers and XRD diffraction patterns of: b PSBR film, c PSBR nanofibers

leading to the formation of small defective crystals [16]. The X-ray diffraction patterns are consistent with the mixture of amorphous polystyrene and polybutadiene.

3.2 Morphological study of PSBR nanofibers

Figure 3 displays the SEM images of PSBR fibers formed from a solution of 10 wt% of PSBR dissolved in different solvents. The PSBR image shown in Fig. 3a presents cupshaped and porous polygonal structures with few numbers of fibers. The formation of cup-shaped structures is attributed to the charged polymer solution jet initiating from the Taylor cone whipping after a certain distance, which is around 0.5 cm, breaks up into separate droplets. Each droplet looks like a circle, and the phase separation into solvent-rich region and polymer-rich region is observed at this stage. This results in the formation of a hole and a complete evaporation of solvent resulting in cup shape formation [17, 18]. On the other hand, porous polygonal structures formation is due to hindered solvent evaporation and a rapid evaporation of the volatile solvent, THF, which lower the temperature and condensation of water vapor. Consequently, the copolymer is separated to different phases, and the pores on the electrospun fibers are formed in solvent-rich regions.[19, 20]. It is noted that there are a number of beads formed during the electrospinning process with a low concentration of PSBR [21]. The electrospinning of PSBR in THF produces a structure having a combination of fibers, beads and cup-shaped structures. While in DMF, the electrospun nanofibers have more uniform shape and structures. This is attributed to high dielectric constant of DMF (36.7 at 20 °C), while THF has a relatively low dielectric constant (7.6 at 20 °C) [22]. The presence of beads reduces the surface area to volume ratio, which is crucial property of nanofibers. Beads structures are considered as a defect in the nanofibers and affect the quality of the formed nanofibers and can be attributed to the elasticity of the solution. PSBR solutions having low extensional viscosity or low relaxation time tend to form beads. This mechanism arises from Rayleigh instability which driven by the surface tension suppressed by viscoelastic behavior of the jet. As the viscoelastic force suppresses the instability, smooth fibers are formed. Consequently, the formation of beads on fibers is caused by the surface tension of the PSBR solution which is decreased by increasing the PSBR concentration [23]. The charge density on the electrospinning jet considers the stretching force to elongate the solution. Improper charge density results in insufficient elongation viscosity to overcome Raleigh instability which leads to beads formation [24]. It is noted that the average PSBR fibers sizes formed from THF, DMF and mixture of THF and DMF with the ratio of 50:50 are 80, 1200 and 900 nm, respectively.

By increasing the concentration of the PSBR solution to 20 wt% in DMF, the polymer solution viscosity increases resulting in syringe tip blockage, and therefore, no fibers were obtained. On the other hand, the 20 wt% PSBR solution within THF forms nanofibers, beads and few numbers



Fig. 3 SEM micrographs at two magnifications of PSBR fibrous sorbents obtained by electrospinning of 10 wt% solution of PSBR in different solvents: **a** pure THF, **b** pure DMF and **c** mixture of THF and DMF with a ratio of 50:50

SN Applied Sciences A Springer Nature journal of cup-shaped structures as shown in Fig. 4a. Smooth and uniform fibers with beads are formed from the 20 wt% of PSBR dissolved in the mixture of THF and DMF with ratio of 50:50 as presented in Fig. 4b. It is found that the fibers diameter is increased significantly with an average fiber size of 700 nm for pure THF solvent and 1200 nm for mixture of THF and DMF with the ratio of 50:50.

Figure 5 shows the SEM micrographs of PSBR fibers obtained from 30 wt% PSBR solutions dissolved in THF and mixture of THF and DMF with the ratio of 50:50. At 30 wt%, the formation of smooth and uniform nanofibers without any beads due to the increase in viscosity of the PSBR solution is noticed. Using DMF as solvent for the 30 wt% solution leads to syringe tip blockage, and the average fiber size is of 2800 nm for pure THF solvent, and 3000 nm for mixture of THF and DMF with the ratio of 50:50. This rising in the fiber diameter is attributed to the increase in the viscosity of the PSBR polymer resulting from the high molecular weight and due to presence of the polybutadiene rubber [23].

3.3 Hydrophobicity of PSBR nanofibers

PSBR nanofibers show an increase in contact angle by increasing the concentration from 10 to 30 wt%, and the highest contact angle achieved for the PSBR nanofibers is 153° by using 20 wt% dissolved in a mixture of THF

and DMF with the ratio of 50:50 as shown in Fig. 6a. This significant increase in the values of the contact angle is attributed to the increase in surface roughness and the formation of grooves on the fiber surface resulting from the polybutadiene rubber, that raise the total surface area and subsequently an increase in the surface roughness as displayed in Fig. 3b. It is also noted that droplet attachment force increases relatively with increasing fibers spacing in contact with the droplets [25]. Figure 6b shows a water droplet of about 10 μ L allowed to contact the surface of the PSBR nanofiber sample, indicating that the PSBR nanofibrous sorbent is superhydrophobic. In contrast, when 10 μ L gas oil droplets are dropped on its surface, the gas oil droplets immediately spread and are adsorbed on the nanofibers sorbent.

3.4 Oil sorption of PSBR nanofibers

Figure 7 shows the maximum sorption capacities of the gas oil onto the fibrous sorbents of the three different concentrations of PSBR prepared from different concentrations in THF, DMF mixture of THF/DMF with the ratio of 50:50, respectively, as a solvent after 30 min. It is observed that the highest sorption capacity achieved by the fibers prepared from the polymer solution of 20 wt% PSBR in the mixture of THF/DMF with the ratio of 50:50 is 62 g/g. The high of oil sorption capacity of PSBR

Fig. 4 SEM micrographs at two magnifications of PSBR fibrous sorbents obtained by electrospinning of 20 wt% solutions of PSBR in different solvents: **a** pure THF and **b** mixture of THF and DMF with the ratio of 50:50



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Fig. 5 SEM micrographs at two magnifications of PSBR fibrous sorbents obtained by electrospinning of 30 wt% solutions of PSBR in different solvents: **a** pure THF and **b** mixture of THF and DMF with the ratio of 50:50



Fig. 6 a The water droplet image for the electrospun PSBR nanofibers with contact angle of 153°. b Wettability of water versus oil of 20 wt% solutions of PSBR nanofibers sample prepared in a mixture of THF and DMF with the ratio of 50:50



nanofibers is due to the high surface roughness and the large surface area resulting from the grooves formation on the fiber surface as well as fiber pores as observed in Fig. 4a, b inset [26]. The main oil sorption mechanisms by these porous fibrous sorbents are the adsorption and capillary action [27]. The different parameters, such as the fiber characteristics (diameter, surface roughness, surface area, density, etc.), the oil properties (density, viscosity), the pore size and distribution in the sorbent, as well as the interactions between sorbent and oil affect the sorption mechanism and the oil sorption capacity [28].

3.5 Effect of oil viscosity on sorption capacity of PSBR nanofibers

Nanofibers prepared from a solution of 20 wt% PSBR in a mixture of THF/DMF with the ratio of 50:50 are used to evaluate the effect of the oil viscosity on the sorption capacity. Gas oil, crude oil and motor oil with different viscosities of 8.5, 6.73 and 79.1 cSt at 40 °C, respectively, are tested in this experiment. All experiments are conducted at constant volume of the oil. Figure 8 shows the sorption capacities of PSBR nanofibers as a function of the sorbent weight after 30 min of sorption. It is found that the sorption capacities

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Sea Water

Potable Water



0

Gas Oil

Fig. 8 The sorption capacity of 20 wt% of PSBR nanofibers prepared in mixture of THF and DMF with the ratio of 50:50 nanofibers for gas oil, crude oil and motor oil (Error bars represent standard deviation of mean, n = 3)

Crude Oil

Types of Oil

Motor Oil

Gas Oil

of PSBR nanofibers are 62, 50 and 125 g/g for gas oil, crude oil and motor oil, respectively. The adsorption capacity is increased with the increase in the oil viscosity due to the improved adherence of the oil onto the fibers surface. A high oil viscosity may result two opposite effects. These enhance the sorption capacity by improving the adherence of the oil onto the fiber surface, and decreasing the sorption by suppressing the oil penetration into the pores of the sorbent [29]. Although the rate of oil penetration into a capillary is inversely proportional to the oil viscosity, porous structure on nanofiber surfaces has the advantage of heavy oil adhesion. This indicated that a minimum amount of each of the sorbents is essentially used for collecting the oil from water.

3.6 Effect of water type on the sorption capacity of PSBR nanofibers

Figure 9 illustrates the results of the sorption capacity of nanofibers sorbents prepared from 20 wt% PSBR in

Fig. 9 The oil sorption capacity of 20 wt% PSBR in a mixture of THF/ DMF with the ratio of 50:50 in potable water versus seawater for gas oil, crude oil and motor oil (Error bars represent standard deviation of mean, n = 3)

Motor Oil

Crude Oil

Type of oil

a mixture of THF/DMF with the ratio of 50:50 in potable water versus seawater for gas oil, crude oil and motor oil. A slight decrease in the nanofibers sorption capacity of the oil spilled in seawater by almost 5 g/g for gas oil, 6 g/g for crude oil and 9 g/g for motor oil. This reduction is attributed to the increase in the total dissolved solids and salts concentration in the seawater which enhances in the seawater surface tension [30] and consequently an increase of the cohesive energy between the oil and the seawater [31]. It is concluded that the interfacial surface energy between oil and seawater is larger than that in oil and potable water.

3.7 Reusability of fibers sorbents

Approximately 80-90% of the absorbed oil is recovered from the PSBR sorbents fibers. This ratio is inversely proportional to the sorption capacity of fibers, resulting from the strong interactions between oil and fiber surface. The sorption efficiency decreases with the repetitive sorption and desorption, and in the second sorption, PSBR fibers absorbed approximately 70–80% of the oil sorbed in the first attempt. These conclude that PSBR sorbents can be recycled multiple times for oil cleanup by utilizing a suitable mechanical extraction mechanism.

4 Conclusion

Superhydrophobic–superoleophilic PSBR nanofibers were successfully prepared by the electrospinning technique using different concentrations of PSBR solutions. It was found that increasing the solution concentration resulted in increasing the fibers diameter and decreasing the cup-shaped beads formation. The sorption capacities of the prepared nanofibers were 62 g/g for gas oil, 50 g/g for crude oil and 125 g/g for motor oil. The formation of grooves on the nanofibers surface increased the surface roughness and the total surface area leading to an increase in oil adsorption on the nanofibers surface. In addition, increasing the oil viscosity increased the oil sorption capacity. The sea and salty water declined the oil sorption capacity of the nanofibers.

Compliance with ethical standards

Conflict of interest On behalf of all authors, the corresponding author states that there is no conflict of interest.

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