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Improvised centrifugal spinning for the production of polystyrene microfibers from waste expanded polystyrene foam and its potential application for oil adsorption



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Abstract

A straightforward approach to recycle waste expanded polystyrene (EPS) foam to produce polystyrene (PS) microfibers using the improvised centrifugal spinning technique is demonstrated in this work. A typical benchtop centrifuge was improvised and used as a centrifugal spinning device. The obtained PS microfibers were characterized for their potential application for oil adsorption. Fourier transform infrared spectroscopy results revealed similarity on the transmission bands of EPS foam and PS microfibers suggesting the preservation of the EPS foam's chemical composition after the centrifugal spinning process. Scanning electron microscopy displayed well-defined fibers with an average diameter of 3.14 \pm 0.59 μ m. At the same time, energy dispersive X-ray spectroscopy revealed the presence of carbon and oxygen as the primary components of the fibers. Contact angle (θ_{CA}) measurements showed the more enhanced hydrophobicity of the PS microfiber (θ_{CA} = 100.2 \pm 1.3°) compared to the untreated EPS foam (θ_{CA} = 92.9 \pm 3.5°). The PS microfiber also displayed better oleophilicity compared to EPS foam. Finally, the fabricated PS microfibers demonstrated promising potential for oil removal in water with a calculated sorption capacity value of about 15.5 g/g even at a very short contact time. The fabricated PS fiber from the waste EPS foam may provide valuable insights into the valorization of polymeric waste materials for environmental and other related applications.

Keywords: Expanded polystyrene foam, Polystyrene microfiber, Centrifugal spinning, Oil adsorption

Introduction

Expanded polystyrene (EPS) foam is a ubiquitous industrial and commercial product often used for disposable cutlery, insulating, and cushioning material in packaging and construction [1]. EPS is primarily composed of aromatic hydrocarbon polymer of styrene that is being produced in millions of tons per year due to its high market demand



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[2]. The increasing utilization of EPS and other polystyrene (PS)-based products has become a serious environmental concern due to its non-biodegradability and limited reusability. Approximately 32.7 million tons of EPS were manufactured annually as of 2012 [3], and an estimate of 25–30% space of landfills worldwide is occupied by these used polystyrene products [4]. Reports also claimed that PS-based wastes are infesting the seas and other water bodies [2–4]. This poses an increasing interest in recycling EPS into value-added products such as fiber material [5, 6].

Fiber technology is a longstanding field that utilizes fiber-structured materials for various industries such as textile and clothing, medical and hygiene, and filtration. In particular, microfibers are classes of very fine fibers possessing a diameter of not more than 10 μ m [7]. Due to their large surface area and extra-fine structure, microfibers possess more outstanding properties than their ordinary fiber counterparts [8]. Microfibers can be generated from natural sources such as cellulose, cotton, silk, flax, and animal wool. However, the fabrication of microfibers from natural sources requires extra steps for isolation and purification due to the presence of other biological components available in these raw materials. In this regard, microfiber fabrication using synthetic polymeric materials such as polystyrene has become a convenient option due to its purity and availability [7, 8].

Synthesizing microfibers from polystyrene has been widely demonstrated using various fabrication techniques. For instance, the production of PS fiber and microfiber scaffolds were produced through solution blow spinning, melt blowing, electrospinning, and centrifugal spinning technique [9-11]. Among the mentioned fabrication techniques, centrifugal spinning is considered one of the most recent and promising strategies due to its fast and high-yielding fiber production with minimal concern on the solution precursor's electric or thermal property [12]. Moreover, this method does not require a set-up with sensitive control on pressure and temperature. Unlike electrospinning, which is the most commonly employed technique for nano/microfiber fabrication, centrifugal spinning can synthesize microfibers without using a high voltage power source [13]. Due to its simple mechanism and instrumentation, centrifugal spinning could also be modified and incorporated into other fabrication techniques such as electrospinning centrifugal spinning and melt-blown centrifugal spinning techniques to improve the quality of the fibers to be produced. In fact, a number of works have demonstrated the successful fabrication of various nano/microfibers using a centrifugal spinning technique, including porous carbon microfibers [14], antimony tin/carbon composite microfibers [15], polypropylene [16], and many more [17]. The centrifugal spinning technique has also been used to produce PS fibers for various applications [11, 18, 19]. Although the centrifugal spinning technique emerged as a promising tool in fiber fabrication, its utilization is still limited due to its unavailability, especially in small laboratories. Nonetheless, the simplicity of its mechanism and instrumentation also opens an opportunity for researchers to modify, improvise, and develop the technology regarding the available resources at hand.

This work demonstrated the fabrication of PS microfiber from waste EPS foam via a centrifugal spinning technique using an improvised centrifugal spinning set-up. A simple tabletop centrifuge was modified as the spinning apparatus. To the best of our knowledge, this is the first time that an ordinary centrifuge was used as a centrifugal spinning apparatus. Interestingly, this improvised method demonstrated high yield and

fast production of microfibers from waste EPS foam as the primary polymeric material. The morphological and chemical profiles of the produced microfiber were also presented. The potential application of the produced PS microfiber as an oil adsorbent material in the water-oil system was also investigated. This improvised process offers new opportunities for the fast and easy production of various microfibers using a readily available and simple device. This also presents a promising step in recycling different polymeric waste products into new functional materials for various applications.

Methods/experimental

Materials collection

Waste EPS foam from laboratory equipment packaging was cleaned thoroughly by washing with running tap water and distilled water. The washed EPS were dried and cut into pieces. Tetrahydrofuran (THF, 95% Scharlau) was used as received without further purification and used as the solvent in this study. A functional benchtop centrifuge (HC-16A, Hanshin Medical Co. Ltd) was used as the improvised centrifugal spinning apparatus. A commercially available mineral multi-grade motor oil (Shell Helix HX3) was used as the test oil in the oil adsorption experiment. All aqueous mixtures required in this study were prepared using deionized water.

Preparation of PS solution and centrifugal spinning of PS microfibers

A polystyrene solution (20% w/v) was prepared by dissolving a weighed amount of EPS in 100 mL of THF. The solution was stirred and homogenized by rapid stirring for at least 12 h at room temperature. The centrifugal spinning of PS microfibers was done using an improvised ordinary centrifuge machine set-up powered by a DC motor (220 V) with a variable rotational speed. In this study, the rotational speed of the motor was fixed at 4000 rpm. Cylindrical plastic vials with holes (0.014 mm diameter) were used as a sample reservoir and were mounted on the centrifuge's rotating pole. The holes-to-fiber collector distance was fixed at around 15 cm throughout the experiment. All the spinning operations were conducted at room temperature.

Characterization of the fabricated PS microfibers

The composition of EPS foam and PS microfibers was determined using an FTIR-ATR spectrophotometer (Perkin-Elmer Spectrum 1000). Surface morphological and elemental characterizations were examined using the SEM-EDX technique (Horiba, X-3CT). Meanwhile, contact angle (θ_{CA}) measurements were obtained *via* the sessile drop method using a tensiometer (Biolin Scientific, ThetaLite).

Application of PS microfibers for oil adsorption in water

The potential application of the fabricated PS microfiber as an oil adsorbent material was investigated by following the oil sorption protocol reported in previous studies [20-22]. In brief, the PS microfibers (approximately 0.12 g) were submerged in a water/oil (2:1 v/v) mixture with a total volume of 250 mL. For comparison, the experiment was repeated for unmodified EPS foam. The adsorption of both PS microfiber and EPS foam in water alone was also investigated. The sorption capacity was calculated using the equation,

sorption capacity =
$$\frac{M_f - M_i}{M_i}$$

where M_f and M_i are the mass of the adsorbent after and before the immersion, respectively. The immersion duration was varied from 5 to 30 seconds.

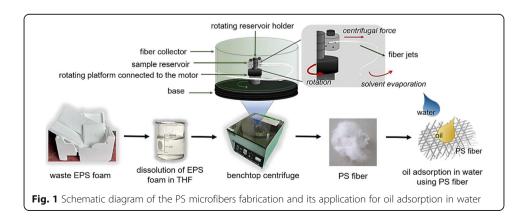
Results and discussion

Characterization of PS microfibers

The schematic illustration of the experimental workflow in this study is presented in Fig. 1. In a standard centrifugal spinning process, a fluid or a polymer solution is placed in a reservoir connected to a rotating motor. This reservoir has multiple holes on its sides that serve as the outlet of the fluid. Once the reservoir rotates and acquires enough speed, the surface tension of the polymer solution is unbalanced by the strong centrifugal force directed outward the reservoir. As a result, a formation of fiber jets around the rotating reservoir was observed. The produced fiber jets were collected by the fiber collectors around the rotating holder. Interestingly, this process can be executed using a typical benchtop centrifuge with minor modifications, as demonstrated in this study.

By visual inspection, the waste EPS was clearly transformed into fibrous material after centrifugal spinning using the improvised apparatus shown in Fig. 2. It was observed that there was an irregular formation of PS film on the fiber collector when EPS solution was lesser than 20%. On the other hand, no fiber formation was observed for the spun solution at a high PS concentration (much greater than 20%). The percent yield of microfiber production using 20% (w/v) of the polymer solution was calculated to be around 91%, while the spinning process lasted for approximately 2 min for every 15 mL of polymer solution placed in the reservoir.

The EPS foam and PS microfibers' compositional properties were determined and compared as shown in Fig. 3. It can be observed that there is neither an appearance nor disappearance of any transmission bands after EPS was converted into PS microfibers. This implies that there are no changes in the chemical composition of the EPS even after it was dissolved in THF, centrifugally spun, and dried. The transmission band at 3031 cm⁻¹ indicates the aromatic C-H stretching vibration, while the band at 2919 cm⁻¹ indicates the vibration of the CH₂ group [23]. Also, the transmission bands at 1600 cm⁻¹, 1488 cm⁻¹, and 1453 cm⁻¹ were attributed to the aromatic C=C



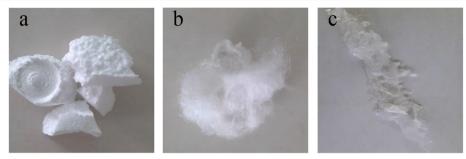
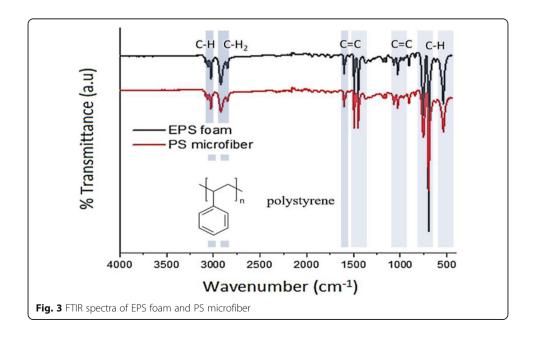


Fig. 2 Actual photograph of the a EPS foam, b PS microfibers, and c PS film formed from low PS concentration

stretching vibration of the benzene ring as expected in the PS structure [24]. Strong vibrational modes at lower wavenumber, 754 cm⁻¹, and 706 cm⁻¹, show the out-of-plane bending vibration of C-H typical for polystyrene [25].

The morphology of the fabricated PS microfibers was examined using SEM, as presented in Fig. 4. Successful formation of long and intertwining microfibers was observed with an average diameter of $3.14 \pm 0.59\,\mu m$ and fiber length expanding up to few millimeters. Higher magnification revealed its well-defined and round fibrous structure with the absence of bead formation. This suggests the successful transformation of waste EPS foam to fine PS microfibers using the improvised spinning apparatus. However, upon closer inspection, the surface of the microfibers was found to be irregularly rough. EDX analysis revealed the presence of carbon as the most abundant elemental component of the microfibers. It is known that PS also consists of hydrogen; however, it is naturally not observable through EDX analysis. A signal of oxygen appeared in the EDX spectrum, which may be due to the residual oxygen from the THF used as the solvent in preparing the PS blend prior to the spinning process. This result generally implies that the polystyrene's chemistry was preserved even after the spinning process was done. The microfibers' chemical composition that is exclusively composed of



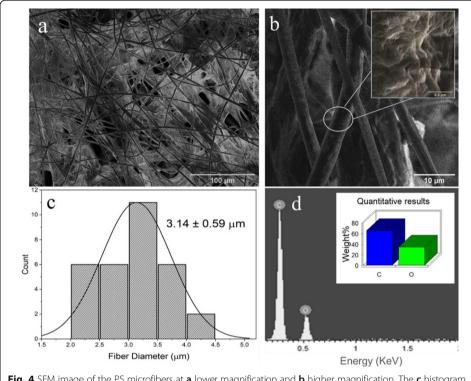
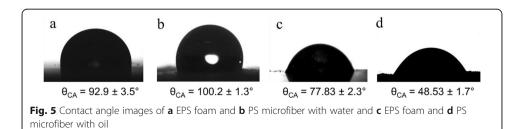


Fig. 4 SEM image of the PS microfibers at $\bf a$ lower magnification and $\bf b$ higher magnification. The $\bf c$ histogram of the microfiber diameter was also shown together with the $\bf d$ EDX result

carbon and oxygen based on the EDX result may suggest that the microfibers are chemically benign and could be employed for biomedical and environmental applications.

Contact angle measurements

Contact angle measurement of the PS microfibers was conducted to investigate their wettability with water and oil samples (Fig. 5). For comparison, the contact angle measurement on EPS foam was also performed. The PS microfibers exhibited a water contact angle of the value of $100.2 \pm 1.3^{\circ}$, validating its hydrophobic property. This value is comparable to the water contact angle observed in PS microfibers produced using other methods [26]. Interestingly, this value is about 7% larger than EPS foam's water contact angle ($\theta_{CA} = 92.9 \pm 3.5^{\circ}$). The round morphological feature of the microfibers may resist the water molecule penetration, thereby trapping air between the intertwining PS fibers. This further resulted in forming a stable heterogeneous solid-liquid and liquid-



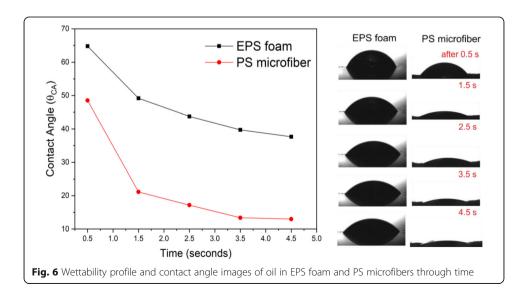
gas interface under the water droplet that may improve PS microfibers' hydrophobicity compared to untreated EPS foam [27]. Meanwhile, a contact angle of 48.5 \pm 1.7° was observed for the PS microfibers with mineral machine oil as the test. This is notably smaller than EPS foam's oil contact angle value (θ_{CA} = 77.8 \pm 2.3°), signifying the PS microfibers' improved oleophilic behavior.

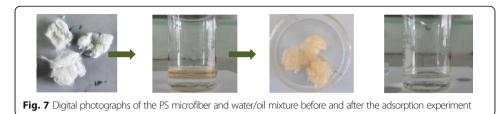
Oil adsorption experiment

To further investigate the oil adsorption of the samples, the θ_{CA} profile was examined through time, as reflected in Fig. 6. As can be seen, there was a dramatic decrease in the θ_{CA} from 48.5° to 21.1° observed as the contact time of oil onto the PS microfiber progressed from 0.5 to 1.5 s. This decrease (up to 56%) indicates the fast adsorption of oil in the fiber matrix. The θ_{CA} value consistently decreased to 12.9° after 4.5 s. Beyond this period, the tensiometer could no longer measure the contact angle, suggesting the complete adsorption of oil on the PS microfiber sample. Meanwhile, EPS foam also demonstrated a decrease in the θ_{CA} from 64.7° to 49.2° at 0.5 to 1.5 seconds. A gradual decrease to 37.6° was observed after 4.5 s. However, the oil θ_{CA} of the EPS foam remained at around 33.2° even after 5 seconds indicating its limited oil adsorption capacity.

The application of the fabricated PS microfibers as oil absorbent material in a water-oil system was investigated, as shown in Fig. 7. The actual photograph of the PS microfibers and the water-oil set-up before and after the oil adsorption experiment is presented. The white color of the PS microfiber prior to the adsorption eventually turned yellowish after submerging in the oil/water system, indicating oil adsorption on the microfibers. The removal of oil in the water was also highly observable (Fig. 7), wherein the water became clear after the application of the microfiber as an adsorbent material.

The sorption capacity of the PS microfibers in the water-oil system at different immersion times was also examined, as shown in Fig. 8. For comparison, the sorption capacity of the samples in water alone was also demonstrated. As expected, both samples exhibited a very low sorption capacity with water due to the polystyrene's





hydrophobicity. On the other hand, the fabricated PS microfibers showed higher oil sorption capacity than the untreated EPS foam. It is believed that the fibrous microstructure of the spun PS may afford high surface energy that increases the capillary action and adsorption of oil into the PS microfibers. These qualities are not observed in the EPS foam, which has a smooth surface and compact structure. It was also observed that the sorption capacity increases as the immersion time was extended up to 15 s until saturation was attained. The oil sorption capacity of the fabricated microfibers reached an equilibrium with a value of 15.5 g/g. This fast saturation might be due to the large surface area afforded by the PS microfibers. The spaces and voids formed between the intertwining microfibers might have served as sites for oil adsorption. The obtained sorption capacity of the fabricated PS microfiber as obtained in this study is comparable or even better than the sorption capacity obtained from plant-derived materials like palm fruit bunch, corn and soybean fibers, cocoa pods, and other microfiber-based oil adsorbents materials such as polypropylene and superhydrophobic

Conclusions

carbon nanotubes as summarized in Table 1.

The centrifugal spinning technique was employed to recycle waste EPS foam into PS microfibers. EPS foam's conversion to fiber material was done using an improvised tabletop centrifuga as centrifugal spinning apparatus. Morphological analysis from the SEM result revealed the formation of round and intertwined fine fibers with an average diameter of about $3.14\,\mu m$. The chemical composition was consistent even after the spinning process, as indicated in the FTIR spectra of the EPS foam and PS microfibers. Moreover, the EDX result revealed carbon as the primary elemental component of the PS microfibers. The hydrophobicity and oleophilicity of both EPS foam and PS

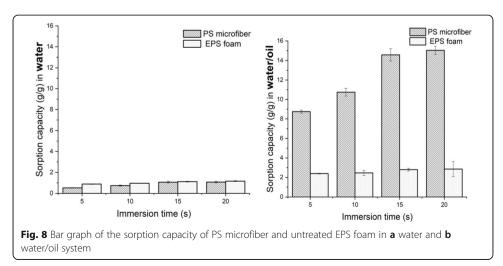


Table 1 Reported sorption capacity of recycled biomass and other carbon-based sorption materials

Adsorbent	Sorption capacity (g/g)	Maximum contact time	References
Empty palm fruit bunch	1.1239–2.8	1–72 h	[28]
Acetylated cocoa pods fiber	8	15 min	[29]
Pyrolyzed barley straw fiber	7.6–9.2	30 min	[30]
Mercerized cattail, corn, and soybean fiber	8	10 min	[31]
Human hair	7.47	60 min	[32]
Nonwoven polypropylene microfibers	14	5 min	[33]
Graphene oxide-coated microfibers	6.8	n/a	[34]
Polycarbonate/carbon nanotubes	12.62	20 s	[35]
Gamma-irradiated and electrospun Polystyrene nanocomposites	16	n/a	[36]
Centrifugally spun (improvised) PS microfibers	15.5	20 s	This work

microfibers were examined by contact angle measurements. Interestingly, PS microfibers manifested a higher water contact angle value compared to EPS foam indicating the enhanced hydrophobicity of the centrifugally spun PS microfibers. On the other hand, the PS microfibers exhibited a more oleophilic behavior than EPS foam, suggesting promising oil adsorption application potential. Finally, an oil adsorption experiment in an oil/water system revealed the outstanding performance of the fabricated PS microfibers for oil adsorption with a sorption capacity of approximately 15.5 g/g even at a very minimal immersion time. The demonstrated work offers new insight in fabricating value-added products from waste materials using readily accessible, environment-friendly, and economical processes for environmental and other related applications.

Abbreviations

EPS: Expanded polystyrene; PS: Polystyrene; θ_{CA} : Contact angle; THF: Tetrahydrofuran; FTIR-ATR: Fourier transform infrared spectroscopy with attenuated total reflectance; SEM-EDX: Scanning electron microscopy and energy-dispersive X-ray spectroscopy

Supplementary Information

The online version contains supplementary material available at https://doi.org/10.1186/s44147-021-00030-y.

Additional file 1.

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Authors' contributions

All the authors have read and approved the manuscript. MLMB conducted the fabrication of microfibers and oil adsorption experiments and wrote the manuscript. JNP conducted the contact angle measurements and analysis and helped revised the manuscript. JPLO assisted in the fabrication and characterization of PS microfibers. SDA provided the equipment and validated the contact angle analysis. ACA collated the characterization data and edited the manuscript. AB conducted the SEM-EDX characterization; FSL and RYC supervised the laboratory work, checked the manuscript, and examined the characterization results.

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Availability of data and materials

The data used and/or analyzed during the conduct of this study are available from the corresponding author on reasonable request.

Declarations

Ethics approval and consent to participate

Not applicable

Consent for publication

The authors hereby declare that they participated in the study and in the development of the manuscript. The authors further declare that they have read the final version and give their consent for the article to be published in this journal.

Competing interests

The authors declare that they have no competing interests.

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