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Article

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Sulfonated electrospun polystyrene as cation exchange membranes for fuel cells

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Abstract

Fuel cells are one of important renewable energy sources represent scientific trends of contemporary researches that seek to find better alternatives to solve the negatives of organic fuels and biological damage to humans, environment and atmosphere. The most vital part in hydrogen fuel cells is the cation exchange membranes whose special and critical role. Cation exchange membranes have been prepared and characterized using expanded polystyrene (PS) from waste packaging. Electrospun polystyrene PS membranes were synthesized by electrospinning. The electrospun PS membranes were chemically modified by sulfonation reaction. Electrospun PS membranes were immersed in (20%) diluted sulfuric acid (H_2SO_4), as a sulfonating agent, for different periods of 1, 2, 3, and 4 h. Sulfonating reaction successfulness was demonstrated by FTIR investigations. SEM and EDS were used to detect the sulfonating and desulfonating reactions regarding the sulfonating time. Ion-exchange capacity (IEC) and the proton conductivity were reported and discussed with respect to different sulfonation time. IEC increased with increasing the sulfonation time. Obtained results showed that the membrane sulfonated for 2 h have the highest IEC, which was equal to 2.857 mmol/g. The proton conductivities of electrospun membranes increased with increasing sulfonation time. Maximum proton conductivity was (8.8×10^{-4}) S/cm resulted after 3 h sulfonation.

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Keywords: Electrospinning; Sulfonation; Expanded polystyrene; Ion exchange capacity; Proton conductivity

1. Introduction

Global trends are moving toward discovering and developing renewable energy resources as solar cells and fuel cells. To be competitive with fossil fuels types such as coal, natural gas and gasoline, renewable energy resources need to be in minimum cost and more reliability, etc [1].

Fuel cells (FCs) are electrochemical cells that cleanly transform the chemical energy of hydrogen (or another hydrocarbon fuel) to electrical energy effectively [2]. Fossil fuels are consuming readily and diminishing significantly to meet future energy demands. Minimum noises, less contamination as well as approximately zero byproducts

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are the most important benefits made fuel cells considered as a clean energy resource [3,4]. Electrical potential produced by a single fuel cell is about (0.6–0.7) V. Connection in series (Stacking) is the solution for this situation as voltage requirements were solved through it. Cell stacks are defined as repeating units, each unit is a fuel cell [5,6]. Many goals aspire to be achieved through stacking, like little loss in electrical interconnects and efficient cooling scheme [7]. Researches directed on diminishing difficulties of fuel cells, this is done through understanding the structural effects on performance [8].

In comparison with batteries, fuel cells are longer operation time, lighter in weight, higher response speed and do not require continuous recharging and replacement [9,10]. Cation or proton exchange membrane fuel cells (PEMFCs) is a common category of polymer electrolyte fuel cells (PEFCs), utilize a solid polymeric membrane between cathode and anode electrodes. This membrane is called cation or proton exchange membrane (PEM) which had characterized by unique properties enabled it to become the main part of PEMFCs. Proton exchange membrane allows the passage of positive ions while expel the negative ions [11]. PEMFCs are considered as preferable choice for transportation due to rapid start-up and rapid energy production even at low temperatures [12]. Efficient proton exchange membrane must have some important intrinsic characteristics enable them to work correctly in fuel cell environment [13,14].

Cation or proton exchange membranes had special physical and chemical properties like proton conductivity, zero electronic conductivity, acceptable mechanical strength, low fuel percolation and minimum water transport through diffusion [15,16]. Electrospinning technique was used to produce membranes suitable for proton exchange fuel cells as well as enhance membrane performance, ionic conductivity and power density [17,18]. Electrospinning produce fibers in both nano and micro scale [19,20]. Electrospinning principle is: applying high voltage on a polymeric solution forming a jet moves toward the collector and as the solvent evaporates, the fibers will be formed [21,22]. There are fundamental requirements of electrospun fibers such as controlled pore sizes, small diameters and specific surface area [23,24].

Electrospun fibers have wide spread application such as in solar cells, air filtration membranes, gas and chemical sensors, piezoelectric sensors, fuel cells, energy storage and ion exchange membranes for different applications [25].

1.1. Proton exchange membrane fuel cell (PEMFC)

PEMFCs make benefit of essential polymeric properties like light weight and chemical resistance to corrosive material and liquids [26]. Fig. 1 shows the schematic representation of a PEMFC.

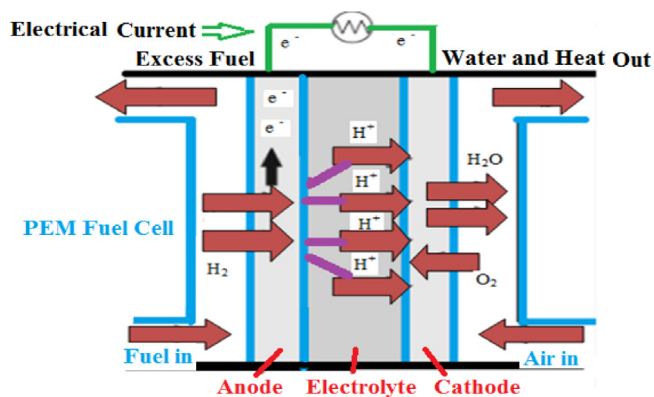


Fig. 1. The schematic diagram of a PEM fuel cell.

It is important to mention that electrospinning accomplished 1D material play a special role as catalyst and solid electrolyte membranes for PEM fuel cell [27,28].

At the anode side of typical PEMFC, oxidation reaction takes place as hydrogen divided to protons and electrons. Produced protons diffuse through membrane while the electrons reach the cathode through an external circuit, thus electric power will be resulted [29]. At the cathode, reduction reaction takes place as oxygen molecules will react with the passing protons. When electrons arrive through the external circuit, H₂O molecules will be produced [30,31].

1.2. Cation exchange membranes:

Ion-exchange membranes (IEM) are semi-permeable membranes transport certain dissolved ions, while preventing and blocking other ions [32]. IEM featured by high selectivity (ions with a charge identical to membrane will be excepted and unable to penetrate the membrane matrix) [33]. There are two major types of IEM: Cation exchange membranes (CEM) and anion exchange membranes (IEM). Cation exchange membranes consist of fixed anions, mobile cations (counter ions) and negative coions (the excluded ions from polymeric matrix) [34]. Fig. 2 shows the CEM structure. IEM properties will depend on the underlying materials they consist of as well as the fixed ions type and their concentrations. It's worth noting that in most cases, cation exchange membrane used in hydrogen fuel cells the used ions as constant charges are $(-SO_3^-)$ and $(-CO_2^-)$ [35].

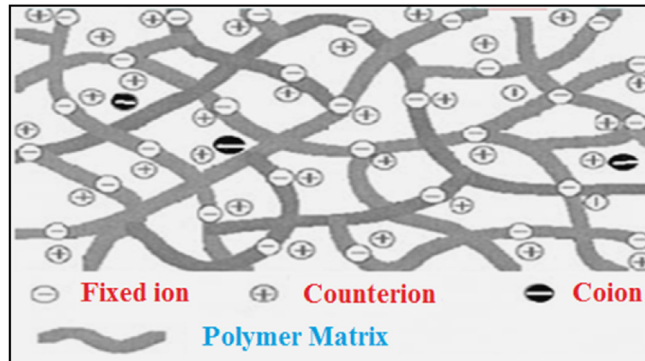


Fig. 2. Cation exchange membrane structure.

1.3. Sulfonation process

Sulfonation process is a chemical reaction used to add acidic group $(-SO_3H)$ to an organic molecule. Sulfonating agents are chemical compounds characterized by a special role in $(C-H)$ bonds replacement by (SO_3H) group through the chemical modification of membrane as the following equation:



Incorporating sulfonic groups into polymers matrix resulted in special properties like increased hydrophilic behavior, impart proton conductivity and unique applications like polymeric membranes for PEMFC [36]. There are two types of sulfonation: pre-sulfonation and post-sulfonation. Pre-sulfonation is defined as sulfonating reaction allowed to be completed before polymerization beginning. While post-sulfonation is defined as sulfonation reaction allowed to be completed after polymerization or by using finished polymer [37,38]. There are many compounds used as sulfonation agents like H_2SO_4 , SO_3 , HSO_3Cl , $CH_3CO_2SO_3H$ [39]. Some of the sulfonating agents are summarized in Fig. 3. In the present study, SPS membranes were produced by post sulfonation of electrospun

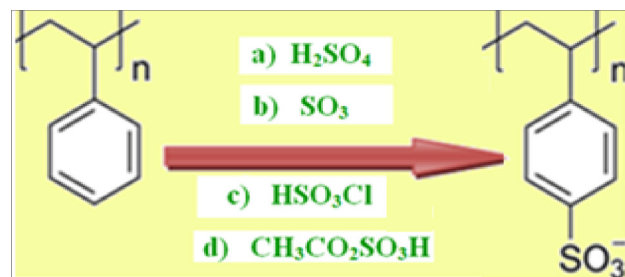


Fig. 3. Sulfonating agents used in sulfonation of polystyrene.

membranes by using 20% diluted sulfuric acid as sulfonating agent which is less affectivity and less aggressively to polymeric membranes comparing with concentrated sulfuric acid [40,41].

The aim of this work is preparation of cation exchange membrane by proving the possibility of PS membranes sulfonation by using dilute sulfuric acid instead of the concentrated acid, while the general thrust of research has often tended to sulfonation PS membranes using concentrated sulfuric acid to avoid the problem of lack in functional groups. The time required for desulfonation reaction (reaction opposite to sulfonation) was highlighted as critical parameter controls sulfonation, and by avoid reaching to this time, sulfonation reaction is controlled and desulfonation is prevented.

2. Experimental part

The experimental work of the research was done in the same manner described in our previous research [42], it will be explained briefly. (25)% expanded Polystyrene solution was firstly prepared by dissolving expanded polystyrene EPS from waste packaging in (DMF) as a solvent. Heating and stirring the solutions to (100) °C for (20–25) minutes is important to obtain homogeneous solution. The collected electrospun membranes were cut to small pieces (2 × 2) cm, then submerged into 20% dilute sulfuric acid) for different periods (1, 2, 3, and 4) h. After sulfonation, the excess acid was removed from sulfonated electrospun membranes by using deionized water.

3. Electrospun membranes characterization

The chemical bonds of the electrospun membranes were characterized using Fourier Transform Infrared spectrophotometer. Which reveals and detect sulfonic acid (–SO₃H) peaks. The morphologies were cleared using scanning electron microscope (SEM). Presence and quantity of sulfur were demonstrated by energy dispersive spectroscopy (EDX).

3.1. Ion exchange capacity (IEC)

Ion exchange capacity IEC is a relative concentration measurement for acidic groups within polymer electrolyte membranes. IEC of membranes is determined by salt splitting titration method at room temperature [43].

The membranes in the acidic forms (H⁺) are converted to the sodium forms. The membranes were cut into small pieces firstly were dipped in 25 ml of 2M NaCl solution through time equal to 12 h m temperatures. Then, the membranes were removed and the remaining liquid was titrated using phenolphthalein used as indicator against a 0.01 M NaOH solution IEC of the SPS was calculated from the following equation [44]:

$$IEC = \frac{\text{Consumed of NaOH} (\text{Molarity of NaOH})}{\text{Dried membrane weight}} \text{ mmol/g} \quad (2)$$

3.2. The proton conductivity

The proton conductivity (σ) was investigated at wet state by an electrochemical impedance spectroscopy (EIS) technique at room temperature using LCR Meter device. Small pieces (2 × 2) cm of sulfonated electrospun membranes were immersed. in deionized water for 12 h [45], and then the excess water was removed. The membranes were mounted on Al foil mask using silver paste to contact the membrane by very high conductivity wire [46]. The coated membranes were measured at potential equal to (1V) using an alternating current at different frequencies range from 20 Hz to 2 MHz. The proton conductivity was obtained from the following equation:

$$\sigma = \frac{L}{R * A} \quad (3)$$

where

σ : The proton conductivity (S/cm)

L : distance between two electrodes (cm)

A : effective area (cm²)

w : electrode width (cm)

R : electrical resistance (Ω)

4. Discussion and results:

4.1. Fourier transforms infrared spectra:

Sulfonation process of PS electrospun nanofiber membranes confirmed clearly by FTIR. FTIR spectra of PS and SPS are depicted in Fig. 4. Fig. 4(a) represents pure PS membrane while Fig. 4(b, c, and d) represents SPS electrospun membranes sulfonating for 1, 3, and 4 h. The principle chemical bonds that presents in pure and sulfonated PS electrospun membranes are: the peak at $(3025) \text{ cm}^{-1}$ is related to the aromatic stretching ($=\text{C}-\text{H}$), while sharp peak at 2921 cm^{-1} is referred to asymmetric stretching ($-\text{CH}_2-$), and the peaks at 1492 cm^{-1} and 1451 cm^{-1} are corresponding to aromatic ($\text{C}=\text{C}$) stretching. The appearance of these peaks matches with Seung H. H. et al. [47].

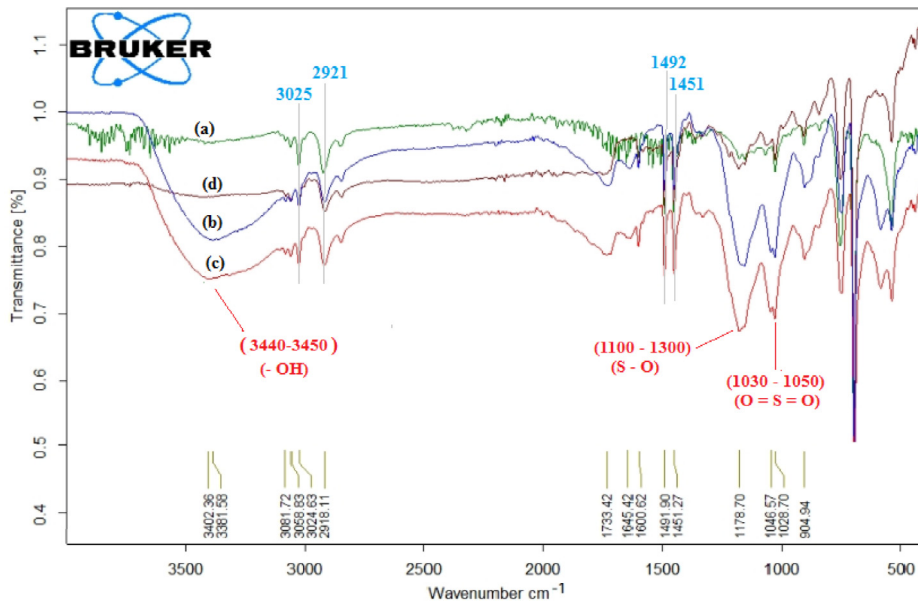


Fig. 4. FTIR spectra (a): pure PS (b): 1 h sulfonation (c): 3 h sulfonation (d): 4 h sulfonation.

New bonds were appeared in FTIR spectra, represent successful sulfonation process. Similar results have registered for sulfonated PS in Imene et al. [48]. Sulfonation process, occurs on phenyl rings, was inferred through new vibration characteristic of sulfonic acid groups. The new chemical bonds at $(3440-3450) \text{ cm}^{-1}$ for the stretching ($-\text{OH}$) in the ($-\text{SO}_3\text{H}$) group, the broad band $(1100-1300) \text{ cm}^{-1}$ (exactly at 1178 cm^{-1}) is referred to ($\text{S}-\text{O}$) and at $(1030-1050) \text{ cm}^{-1}$ for a symmetric stretching of the ($\text{O}=\text{S}=\text{O}$). The appearance of these new peaks indicates successful sulfonation process occurred during specific periods till 4 h.

The disappearing of ($-\text{SO}_3\text{H}$) peak from the spectra of samples treated for 4 h Fig. 4(d) could be used to follow the sulfonating reaction progress with time. After long time exposure to sulfuric acid a hydrolysis of ($-\text{SO}_3\text{H}$) group taken place, this reverse reaction is called desulfonation. Woei Jye Lau et al. verified the presence of competing desulfonation that could be controlled by concentration of sulfonating agent, time and temperature [49].

4.2. Scanning Electron Microscope (SEM):

The variation in morphology between pristine and sulfonated PS electrospun membranes was observed by scanning electron microscope SEM. Fig. 5 indicates SEM image of pure PS, the average fiber diameter was $(1.8-2) \mu\text{m}$. PS electrospun membranes sulfonated for different period (1, 3, and 4 h) are shown in Figs. 6, 7, and 8) respectively.

The average fiber diameters of PS membranes sulfonated for (1, 3, and 4) hr was $(2.5-3) \mu\text{m}$, $(3-3.5) \mu\text{m}$, and $(2-2.5) \mu\text{m}$. As it evidences from SEM images, sulfonation was accompanied with increasing in average fiber diameters, this could be observed clearly from Figs. 6 and 7. The explanation of average fiber diameter enhancement

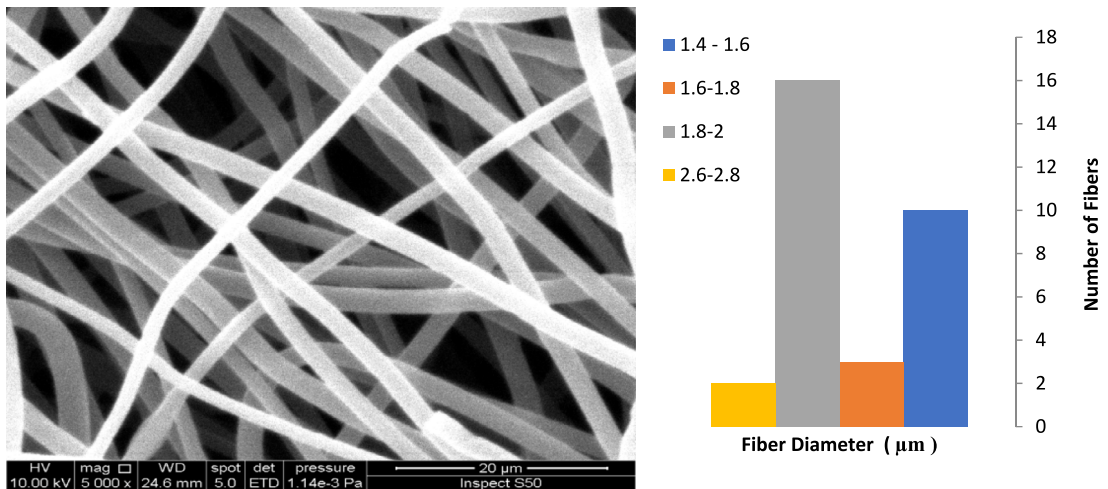


Fig. 5. SEM image and average fiber diameter for pure PS membrane.

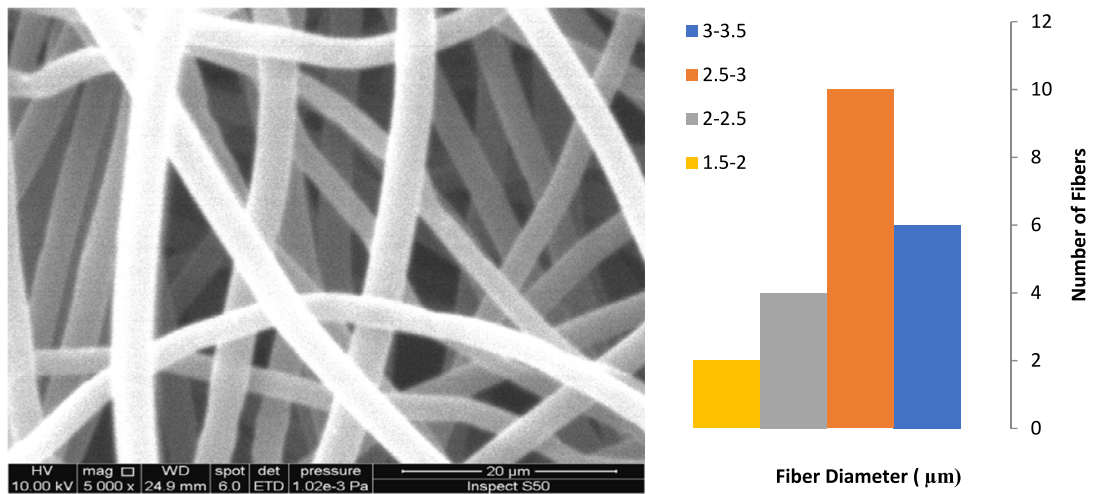


Fig. 6. SEM image and average fiber diameter for PS membrane sulfonated for 1 h.

is due to the increase in acidic groups or sulfonic groups which cause excessive swelling with water. In other words, increasing the sulfonation reaction time will cause hydrophilic behavior enhancement of polymeric fibers. Consequently, polymeric fibers retain more amount of water and average fiber diameter will be increased. The average fiber diameter decreased in membrane sulfonated for 4 hr as in Fig. 8. This may be referred to desulfonation process which has a critical effect reverse to sulfonation.

Desulfonation will cause lack of acidic group and thus, lack in hydrophilic behavior. Fig. 9 indicates the relationship between the average fiber diameter and sulfonation time. It is important to mention that the fibers maintain their fibrous structure during exposure to dilute sulfuric acid solution without dissolving or dissociation and without any additional surfactant treatment, that refer to its less harsh effect on polymer.

4.3. Energy Dispersive Spectroscopy (EDX)

Energy Dispersive Spectroscopy (EDX) is a powerful characterization allows elemental analysis of the sample surface. EDX microanalysis spectrum provides qualitative as well as quantitative information [50]. Fig. 10 represents EDX spectra and element mapping of pure PS, while Figs. 11, 12, and 13 represent EDX and element mapping of

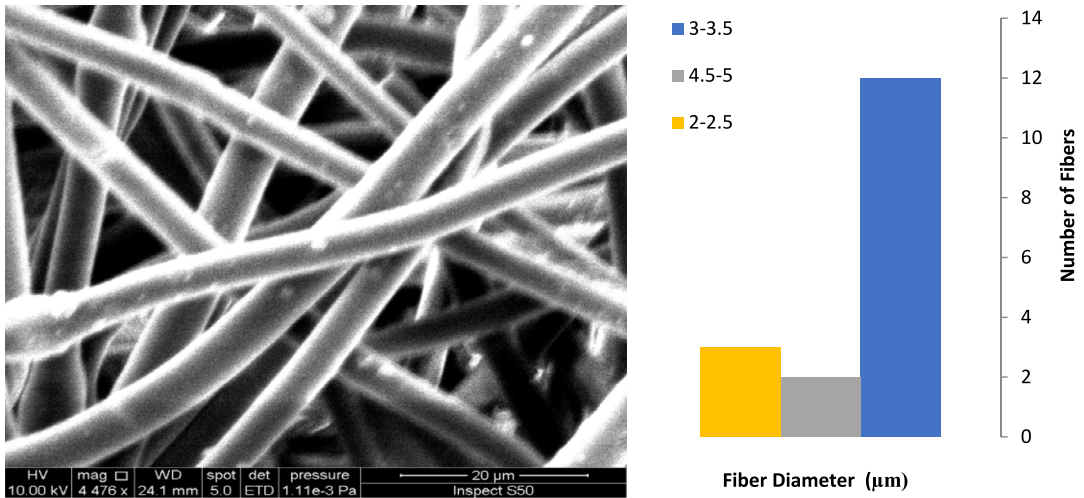


Fig. 7. SEM image and average fiber diameter for PS membrane sulfonated for 3 h.

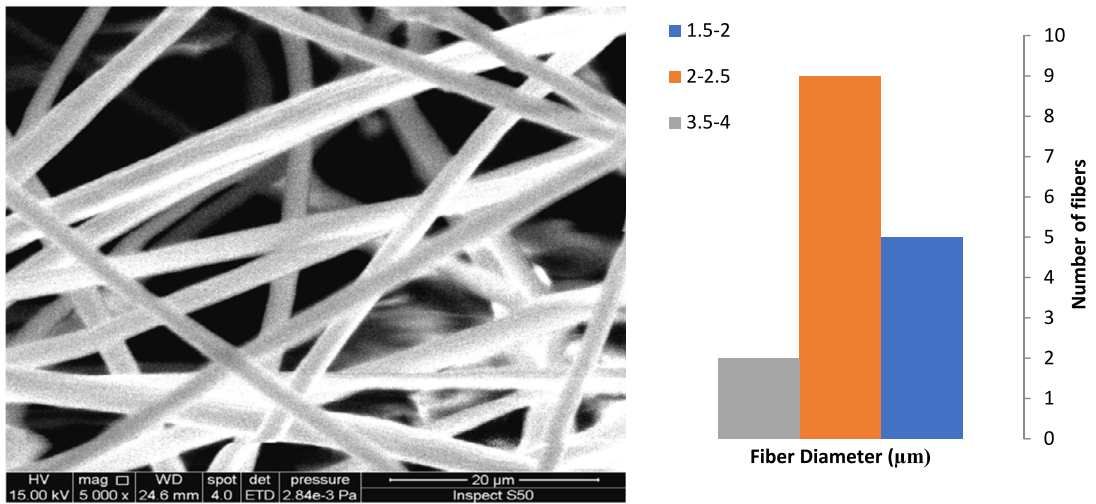


Fig. 8. SEM image and average fiber diameter for PS membrane sulfonated for 4 h.

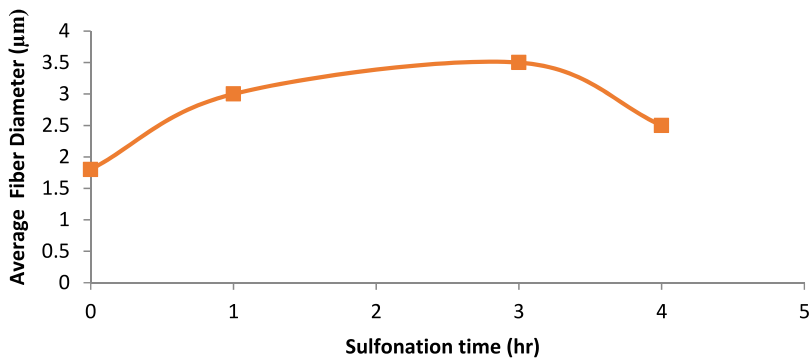


Fig. 9. The relationship between average fiber diameter and time of sulfonation.

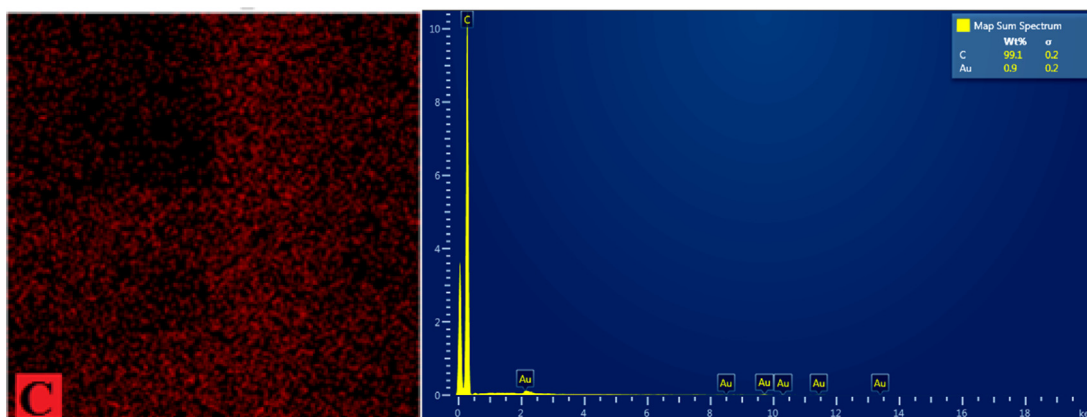


Fig. 10. EDX spectra for pure PS membrane.

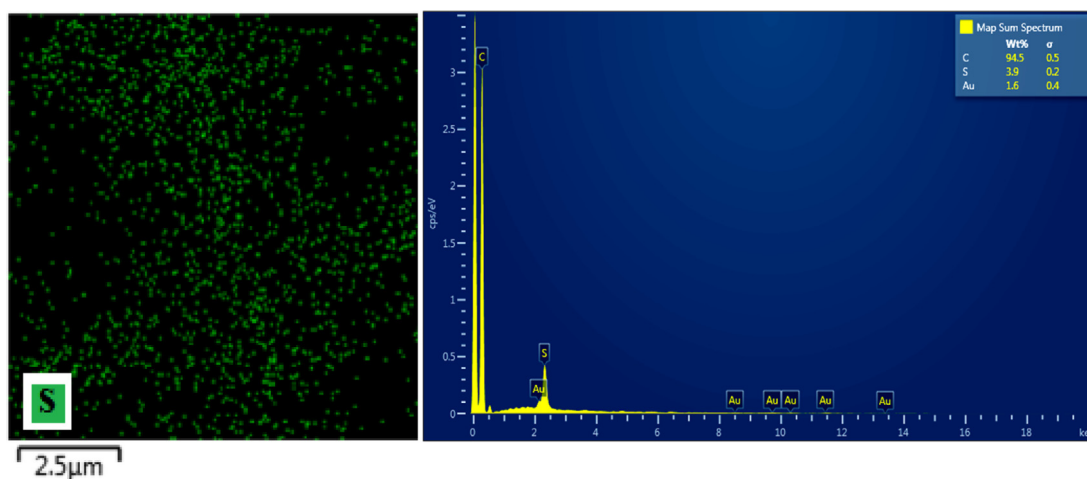


Fig. 11. EDX spectra for PS membrane sulfonated for 1 h.

PS electrospun fibers sulfonated for (1, 3, and 4) h, red and green color spots represent carbon and sulfur elements respectively.

EDX element analysis spectrums associated with the elements mapping for each case of sulfonating shows clearly the difference in sulfur concentrations in the fiber structure, these results had matched with Won G. J. Et al. [51].

As it is clear from Fig. 10, there is no sulfur in pure PS membrane and carbon is the only element. In Figs. 11 and 13, the presence of sulfur enhanced with increasing sulfonation reaction time. EDX results accomplished with FTIR and SEM morphology results because clear reduction in sulfur was found, and that indicates the beginning of desulfonation phenomena after 4 h exposure to diluted sulfuric acid.

4.4. Ion Exchange Capacity (IEC):

IEC values of SPS were increased with sulfonation reaction time as reveals in Fig. 14. The maximum IEC value appeared after 2hr sulfonation (2.857 mmol/g), then decreased with sulfonation times increasing. This may be attributing to the formation of diffusion barrier as the concentration of SO₃H group density increases with increasing the sulfonation time. The diffusion barrier hampers the motion of [H⁺] through the membrane which leads to reduce IEC. Mohamed M. E attributed the reduction in IEC to the hydrophobic diffusion barrier formation during the reaction [52]. IEC values of electrospun membranes listed in Table 1.

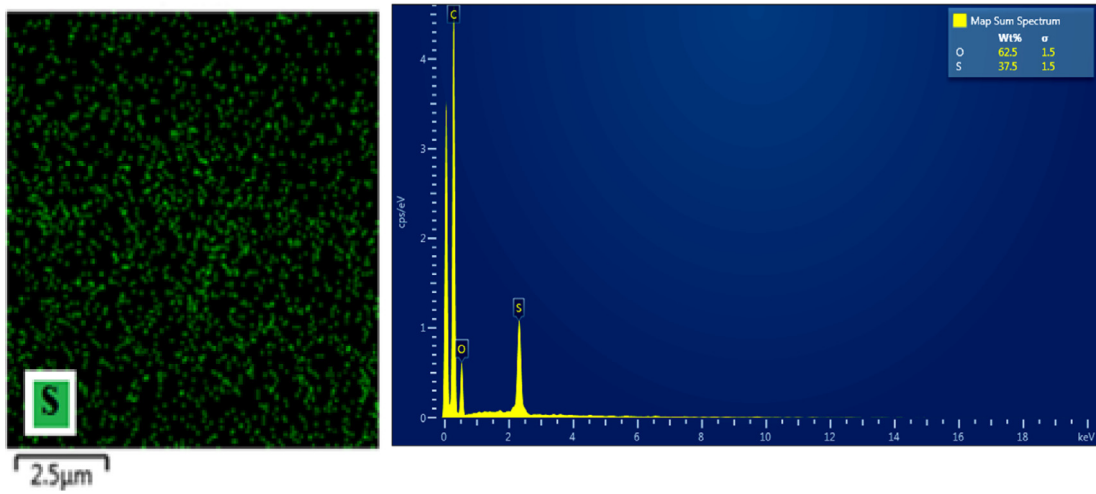


Fig. 12. EDX spectra for PS membrane sulfonated for 3 h.

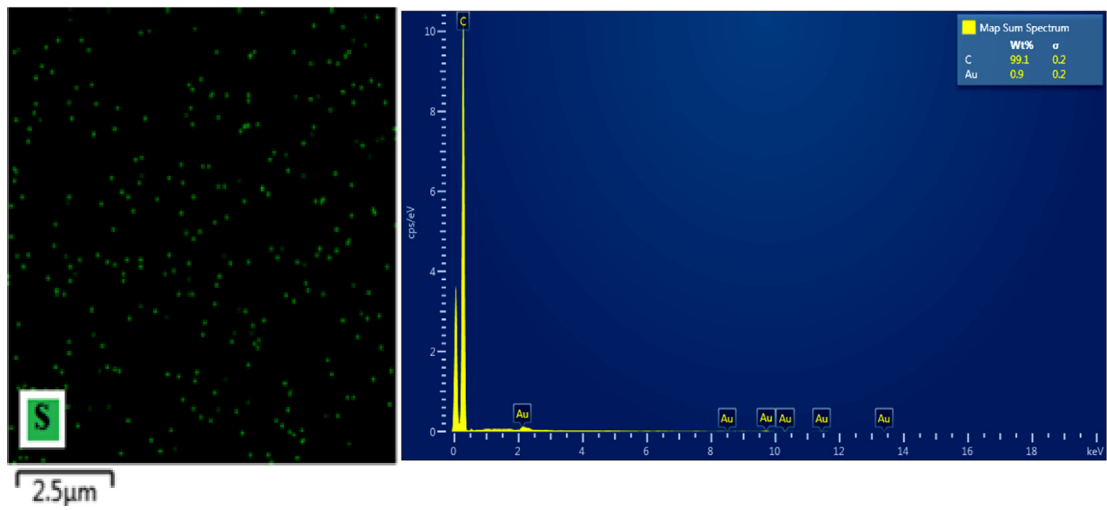


Fig. 13. EDX spectra for PS membrane sulfonated for 4 h.

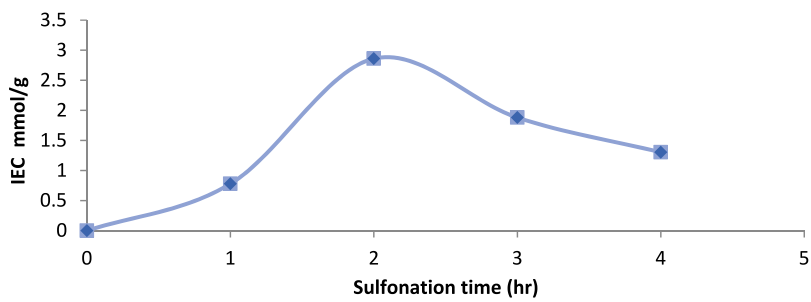


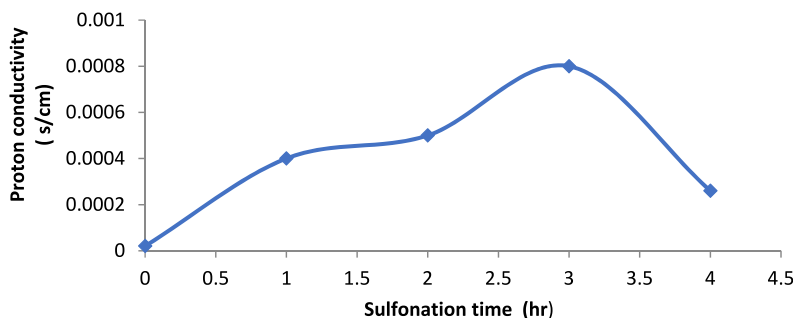
Fig. 14. Increasing IEC with sulfonation time.

Table 1. IEC and proton conductivity for electrospun membranes.

The polystyrene membrane	IEC (mmol/g)	Proton conductivity (S/cm)
Pure PS membrane	0	2.04×10^{-5}
PS membrane sulfonated for 1 h	0.78	4.1×10^{-4}
PS membrane sulfonated for 2 h	2.857	5.032×10^{-4}
PS membrane sulfonated for 3 h	1.88	8.81×10^{-4}
PS membrane sulfonated for 4 h	1.304	2.61×10^{-4}

4.5. Proton conductivity:

It was improved by, Maryam et al. that the introduction of sulfonic, acid groups to the hydrocarbon-based polymers would lead the polymeric chains to gain, proton conductivity [53]. As our FTIR and EDX results confirm the increasing in sulfonic acid group and sulfur concentration in PS fibers with sulfonating time till 3 h. It will become easy to attribute the increasing in proton conductivity of PS membranes, as shown in Fig. 15, to the sulfonic acid group increment. Losing sulfonic group by desulfonation phenomena is responsible in reducing the proton conductivity of PS membrane sulfonated for 4 h. Proton conductivity and IEC values of electrospun membranes are listed in Table 1.

**Fig. 15.** Proton conductivity of electrospun S/cm.

5. Conclusion

Cation exchange membranes were prepared effectively through sulfonation of electrospun fibers by with 20% diluted sulfuric acid. Different sulfonation periods were used. Acidic groups or sulfonic groups were confirmed through FTIR analysis. Sulfonation is very critical reaction, successful sulfonation reaction reached after (1 and 3) h treatment. Desulfonation reaction phenomena detected after sulfonation time equal to 4 h exposure to the diluted acid. So controlling sulfonation time could be used to avoid desulfonation. Sulfonation and desulfonation reactions were demonstrated by FTIR, SEM and EDX. The membranes maintain their fibrous structure without dissolving under the effect of acidic environment. IEC and proton conductivity increased with increasing time of sulfonation. Maximum IEC and proton conductivity was (2.857 mmol/g) and (8.8×10^{-4} S/cm) respectively.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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