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Characterization of polyethyleneterephthalate (PET) based proton exchange membranes prepared by UV-radiation-induced graft copolymerization of styrene

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ABSTRACT

Polymer electrolyte membranes (PEMs) were successfully prepared by simultaneous ultraviolet (UV) radiation-induced graft copolymerization of styrene (35 vol.% concentration) onto poly(ethyleneterephthalate) (PET) film, followed by sulfonation on the styrene monomer units in the grafting chain using 0.05 M chlorosulfonic acid ($ClSO_3H$). The radiation grafting and the sulfonation have been confirmed by titrimetric and gravimetric analyses as well as Fourier Transform Infrared (FTIR) spectroscopy. The maximum ion-exchange capacity (IEC) of the PEM was measured to be 0.04385 mmol g^{-1} at its highest level of grafting and sulfonation. They exhibited high thermal and mechanical properties as well as oxidative stability. They are highly stable in H_2SO_4 solutions and can be used in the acidic fuel cells. The membranes showed low water uptake as well as low proton conductivity than Nafion. In this study, the preparation of PEMs from commodity-type polymers is found to be very inexpensive and is a suitable candidate for applications in fuel cells.

1. Introduction

Radiation graft polymerization is one of the promising methods, which enables introduction of active monomer functional group at inner polymer chains in film for the modification of the chemical and physical properties of a wide range of polymer materials. Various kinds of grafting polymerization techniques – including ion-radiation induced, photo-induced (UV and chemical initiator), and plasma-induced grafting polymerizations – have been developed in the last few decades [1–3]. However the UV-radiation technique is more available and less expensive than other techniques [4].

Graft-type polymer electrolyte membrane (PEM) fuel cell is believed to be the best type of fuel cell that can be used as an alternative source of power, with promising potential to reduce the consumption of fossil fuel, as well as emission of CO_2. Graft-type PEM can be prepared by radiation-induced graft polymerization and subsequent chemical modifications of the grafting chains [5–7]. The method is advantageous for initiating grafting in many kinds of polymers owing to its extensive penetration in the polymer matrix and its rapid and uniform formation of radicals [8]. This technique allows introducing ion conducting (electrolytes) regions directly into polymer films, which have excellent thermal and mechanical properties. Up to now, perfluorinated membranes like Nafion, have been used extensively because of their characteristic higher durability under fuel cell operating conditions [9]. The higher cost, lower stability at higher temperature, and lower gas barrier property of the perfluorinated membranes necessitates the development of a higher performance PEM that will have high-energy efficiency, low cost, high durability, as well as excellent barrier properties against fuels (H_2) and oxygen.

Radiation-grafted sulfonic acid membranes have been regarded as the most promising alternative proton conducting materials for PEM fuel cells and direct methanol fuel cells [10], having wider applications in the field of electrolysis method of water desalination, electrolysis in fuel cell, etc., owing to their wider pH range working characteristics [6]. The radiation-induced graft copolymerization of various monomers such as acrylic acid, methacrylic acid, 4-vinylpyridine, acrylonitrile, vinyl acetate, *N*-vinyl-3-morpholinone, and 2-methyl-5-vinylpyridine onto polyethyleneterephthalate (PET) films has been investigated earlier on many occasions [8,11–13]. In this study, the PET-based PEM was prepared successfully by UV-radiation grafting of styrene onto PET films, and consequent selective sulfonation by chlorosul-

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fonic acid. The effects of different monomer concentrations and sulfonation on grafted membranes of PET have also been studied. The membrane has been characterized by different chemical and physical parameters, including water uptake, tensile strength, ion-exchange capacity, oxidative and acidic stability, proton conductivity, thermal durability and FTIR spectroscopy. These results are presented in the context of other perfluorinated membranes (e.g., Nafion) to evaluate its (PEM) potential use as proton exchange membrane in fuel cells.

2. Experimental

2.1. Materials

The base PET film was prepared from PET granules purchased from Thailand (MITSUIPET). The monomer, styrene (C_6H_5–C=CH_2) and the photoinitiator (Darocur$^{(R)}$ 1173) were purchased from Merck, Germany. Methanol (CH_3OH) was used as a solvent in grafting and for washing base film to remove impurities on its surface before use for grafting. Toluene ($C_6H_5CH_3$) was used to remove homopolymer after grafting. Chlorosulfonic acid ($ClSO_3H$) was used for sulfonation. All three (methanol, toluene, and chlorosulfonic acid) were provided by Merck, Germany. 1,2-Dichloroethane ($C_2H_4Cl_2$) purchased from BDH limited, Poole, England was used as a diluent. H_2O_2 and H_2SO_4 were also purchased from BDH limited, Poole, England. Sodium chloride (NaCl) and sodium hydroxide (NaOH) were used for titrimetric analysis and purchased from Wako pure chemical industries, Ltd., Japan.

2.2. Preparation

PET granules of different masses were heated at different temperatures and cooled under different pressures for different occasions using the heat and the cold press machine (SHIMADZU Corporation, Japan), respectively. The homogeneous PET films were found for 5 g granules at 510 °F under 3500 psi pressure for 5 min in each case. The PET films of 0.20 mm thickness were cut into 1 cm × 5 cm rectangles and wiped with acetone to remove impurities on the film surface.

After drying, the washed films were immersed in 20 mL of methanol solution containing different concentrations (25, 30, 35, 40, and 45 vol%) of styrene monomer and 2% photo initiator for graft copolymerization under UV-radiation (SHIMADZU Corporation, Japan) for different times (1, 2, 3, 4, 5, and 6 h). After graft copolymerization, the grafted films were washed with a large amount of toluene to remove the homopolymers and residual monomer by the Soxhlet extraction process.

The degree of grafting was calculated using the following equation:

$$G\ \ (\%) = \frac{W_g - W_o}{W_o} \times 100 \tag{1}$$

where W_o and W_g are the film weights before and after graft copolymerization, respectively.

Finally, the grafted films were sulfonated by immersing in different concentrations (0.2, 0.1, 0.05, 0.03, and 0.01 M) of chlorosulfonic acid ($ClSO_3H$) in dichloromethane at different temperatures for different times, and washed using distilled water to remove unreacted chlorosulfonic acid ($ClSO_3H$) adhering on the films. The degree of sulfonation of dried sulfonated films was calculated by the following equation:

$$S\ \ (\%) = \frac{W_s - W_g}{W_g} \times 100 \tag{2}$$

where W_s and W_g are the weights of sulfonated films and grafted films, respectively.

2.3. Characterization of the membrane

The ion-exchange capacity (IEC) of the PEM was determined by titrimetric analysis. The dried membrane in the protonic form was immersed in a 0.1 M NaCl aqueous solution for 24 h at room temperature (30 °C). The H^+ ions, substituted by Na^+ ions, were released into the solution. The solution was titrated by a 0.05 M NaOH solution. The ion-exchange capacity (IEC) of the membrane was calculated using titration result with the following equation:

$$\mathrm{IEC} = \frac{C_{\mathrm{NaOH}} \times V_{\mathrm{NaOH}}}{W_{\mathrm{dry}}} \tag{3}$$

where C_{NaOH} (mol L^{-1}) and V_{NaOH} (mL) are the concentration and volume of NaOH solution required for neutralization of the residual solution, and W_{dry} is the weight of the dry sulfonated membrane in protonic form.

The water uptake of sulfonated membranes was calculated by the following equation:

$$\text{Water uptake}\ \ (\%) = 100 \times \frac{W_{\mathrm{wet}} - W_{\mathrm{dry}}}{W_{\mathrm{dry}}} \tag{4}$$

where W_{wet} is the weight of PEM after immersing the dry membrane in distilled water at different temperatures for different times and W_{dry} is the weight of the dried PEM.

The acid tolerance of sulfonated membranes was calculated by the following equation:

$$\text{Acid tolerance}\ \ (\%) = 100 \times \frac{W_{\mathrm{wet}} - W_{\mathrm{dry}}}{W_{\mathrm{dry}}} \tag{5}$$

where W_{wet} is the weight of PEM after immersing the dry membrane in H_2SO_4 at different concentration for different times and W_{dry} is the weight of the dried PEM.

The hydrogen peroxide (H_2O_2) tolerance of sulfonated membranes was calculated by the following equation:

$$H_2O_2\ \text{tolerance}\ \ (\%) = 100 \times \frac{W_{\mathrm{wet}} - W_{\mathrm{dry}}}{W_{\mathrm{dry}}} \tag{6}$$

where W_{wet} is the weight of PEM after immersing the dry membrane in H_2O_2 at different concentrations for different times and W_{dry} is the weight of the dried PEM.

The proton conductivity of the PEM was calculated from the following equation:

$$\text{Proton conductivity},\quad \sigma = \frac{l}{R} \times S \tag{7}$$

where R is the resistance (Ω) of the membranes, l is the thickness (cm) of the membranes and S stands for contact surface area (cm^2) of the electrode.

IR spectra of original, grafted and sulfonated PET films in the range of 400–4000 cm^{-1} were measured by Fourier Transform Infrared Spectrophotometer (FTIR) (SHIMADZU Corporation, Japan). The tensile strengths of sulfonated films were determined using Tensile Strength Testing Machine (INSTRON Corporation, UK). For measuring the resistance, the membrane of 1 cm^2 area was soaked in water at 80 °C for 24 h. The hydrated PEM was then platinized by Pt/C. The Pt/C acted as an electrode that was prepared as liquid ink form by thoroughly mixing together appropriate amounts of catalyst (a powder of Pt dispersed on carbon) and an alcoholic solution of the membrane material. The ink was painted to the both sides of the membrane and heated until the catalyst layer was dry. The dry membrane/electrode assembly was rehydrated by immersing in lightly boiling dilute acid solution followed by rinsing in distilled water. The prepared membrane/electrode assembly was inserted into the temperature and humidity chamber (ESPEC Corporation, Japan) and connected by Cu wire to impedance analyzer (SHIMADZU Corporation, Japan). The resistances of the membranes were read out from the Impedance Analyzer at a constant temperature (30 °C) and at a relative humidity of 80%.

3. Results and discussion

3.1. Preparation of the sulfonated film

Radiation-induced graft copolymerization of styrene onto PET films was carried out using simultaneous UV-radiation technique, and the grafted films were sulfonated by chlorosulfonic acid to prepare proton exchange membranes for the fuel cell. Fig. 1 shows the degree of grafting, *G* (%) of styrene monomer onto the PET film as a function of monomer concentration after 3 h under simultaneous UV-irradiation method. In this study, *G* (%) increases with increasing monomer concentration and reaches a maximum at 35 vol.%. The grafting copolymerization dominates over homopolymerization with increasing monomer concentration, but after 35 vol.% monomer concentration, it starts to decrease because of forming homopolymer rather than grafting. It was suggested that the homopolymerization effect increased the viscosity of the grafting solution hindering the movement of the monomers toward the substrates and consequently reduced the grafting yield [8,13].

Using 35% monomer styrene concentration, the grafting copolymerization was carried out with UV radiation at different radiation periods (Fig. 2). The degree of grafting increases with the increase of radiation time up to 4 h. With increasing radiation time, the number of radical sites formed in the grafting system also increases, i.e., more monomers diffuse to the PET film resulting in an increase in the degree of grafting. After 4 h radiation time, no appreciable increase in grafting was observed because of the saturation of the radical sites. So the equilibrium degree of grafting value (12.7%) was reached at 4 h.

The incorporation of sulfonic groups to the grafted PET film can enhance the solubility of the products, so that they could be cast into the membranes as PEMs. The sulfonation was performed by sulfonating agent, chlorosulfonic acid ($ClSO_3H$). The $-SO_3H$ is added to the aromatic ring by electrophilic substitution reaction [14]. Full sulfonation, i.e., one sulfonic acid group per aromatic group in the PET film, is expected for this type of reaction. But the extent of sulfonation can vary considerably with the conditions of the sulfonation reactions [14]. This study showed that the grafted PET film (grafting degrees of 8–13%) exhibited maximum sulfonated degrees of 10–12%. Previous studies on MA-grafted ETFE showed grafting degrees of less than 30% exhibited sulfonated degrees of 20–25%, which seems lower than those of the films with grafting degrees of more than 30% [15]. To determine the influence of the reaction conditions on the properties of the sulfonated membrane, the effect of the reaction time, temperature and chlorosulfonic acid concentration were investigated in this study.

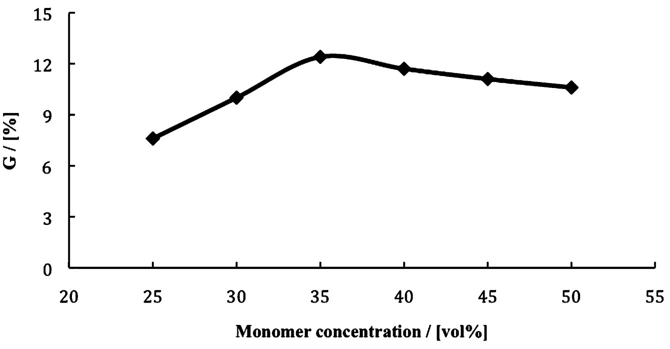

Fig. 1. Degrees of grafting of styrene onto the PET film as a function of monomer concentration.

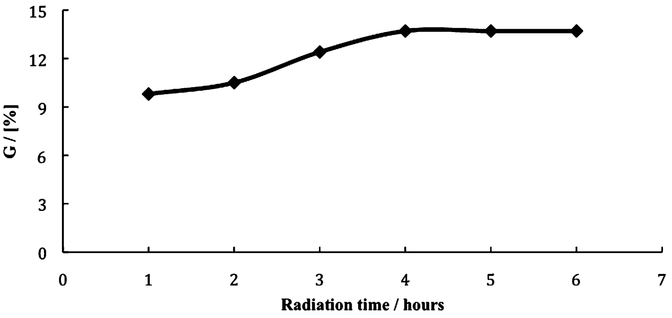

Fig. 2. Degree of grafting (*G* (%)) of styrene onto the PET film as a function of UV-radiation time.

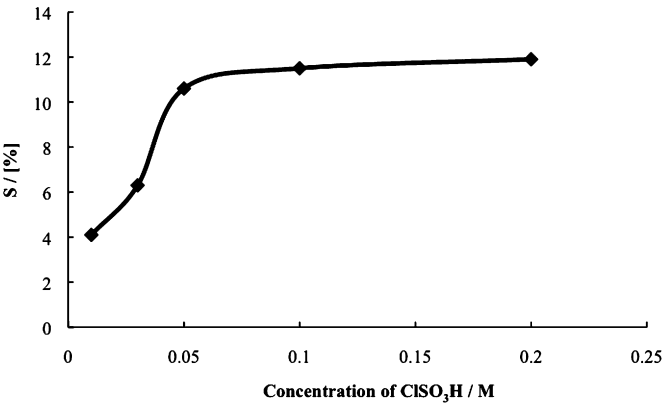

Fig. 3. Degree of sulfonation (*S* (%)) versus concentration of $ClSO_3H$ at room temperature (30 °C) for 2 h.

Fig. 3 shows that the degree of sulfonation, *S* (%) increases with increasing chlorosulfonic acid concentration. For fuel cell applications, it is important that the graft-type PET has a homogeneous distribution of sulfonic acid in the perpendicular direction to the film surface so as to exhibit high proton conductivity between two electrodes in a fuel cell device [15]. More homogenous distribution of sulfonic acid to the film surface gives more rigid structure having higher tensile strength. We examined the tensile strengths of the sulfonated films (Table 1) prepared from different concentrations of chlorosulfonic acid and selected the optimum concentration of $ClSO_3H$ to be 0.05 M. The sulfonation reaction at higher $ClSO_3H$ concentration, is accompanied with decreasing tensile strength of the film. This may be due to side reactions, resulting in the degradation of the sulfonated chains that make the film more fragile [15].

The degree of sulfonation, *S* (%) also increases with increasing reaction time and temperature due to saturation of grafted PET films by the $-SO_3H$ groups (Fig. 4). An elevated temperature drastically increased the sulfonation reaction speed; and in a quite short time, the maximum degree of sulfonation could be obtained [14]. In the current study, maximum *S* (%) was found with increasing temperature for a shorter reaction time.

Table 1
Tensile strength and degree of sulfonation of sulfonated films prepared from different concentrations of chlorosulfonic acid ($ClSO_3H$).

Concentration of $ClSO_3H$ (M)	Degree of sulfonation, *S* (%)	Tensile strength (MPa)
0.2	12.3	8
0.1	11.2	12
0.05	10.6	29
0.03	3.1	31
0.01	2.9	36

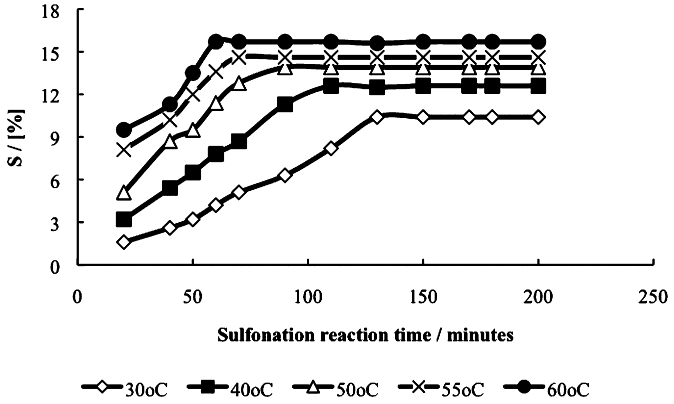

Fig. 4. Degree of sulfonation (*S* (%)) of grafted film by 0.05 M $ClSO_3H$ as a function of reaction time at different temperatures.

3.2. Characterization of the sulfonated films

Titrimetric and gravimetric analyses are more advantageous methods for evaluating techniques for grafting polymer films because they easily determine the quantity of the sulfonic acid group [15]. The structural characterization of the sulfonated membranes by titrimetric analysis was confirmed by FTIR spectroscopy.

Fig. 5 shows the relationship between ion-exchange capacity (IEC) and the degree of sulfonation of the grafted membranes at room temperature. The results show that IEC increases with increasing *S* (%) due to increased $-SO_3H$ groups with a higher degree of grafting. The amount of SO_3H groups is responsible for the ion exchange and the water uptake in the sulfonated membranes. With increasing the degree of grafting, more styrene is introduced in the PTE film, and accordingly the equivalent sulfonic acid group is incorporated. Due to the repulsive forces between the condensed sulfonic acid groups at a higher degree of grafting, the water uptake tends to reach a saturated level slowing the increase of IEC. In this study, at 15.7% degree of sulfonation, the IEC reaches a maximum of 0.04385 mmol g^{-1}. IEC of the current membrane is quite low because of the lower degree of grafting and sulfonation of the PET film under UV-radiation treatment.

H_2O_2 can be produced at a very low degree in hydrogen fuel cell, so the oxidative stability of PEMs is especially important for fuel cell applications. The oxidative stability of the membranes was investigated by different concentrations of H_2O_2 at 30 °C for 8 days. The membranes showed no visible change (maximum 1.5% weight loss) even after treatment with 0.5 M H_2O_2 for 8 days, and still remained transparent, tough and flexible confirming their excellent oxidative stability. The membranes could be used in acidic fuel cell, if they are stable in the acidic solution. So, the tolerance of sulfonated film in different concentrations of H_2SO_4 solution has also been investigated in this study. There is no visible change in weight of sulfonated film (only 5% weight loss) after treating with 0.1–1 M H_2SO_4 for 8 days (Fig. 6b) suggesting that the sulfonated film has good stability in the acidic media of the hydrogen fuel cell.

In Fig. 7, the water uptake of sulfonated film is presented as a function of soaking time at different temperatures. The water uptake of PEMs has a profound effect on their conductivity and mechanical properties [16]. Since water molecules attach to the acid functionality and facilitate proton transport, an appropriate amount of water in PEMs is necessary for proton transport according to the vehicle mechanism [17]. However, too much water

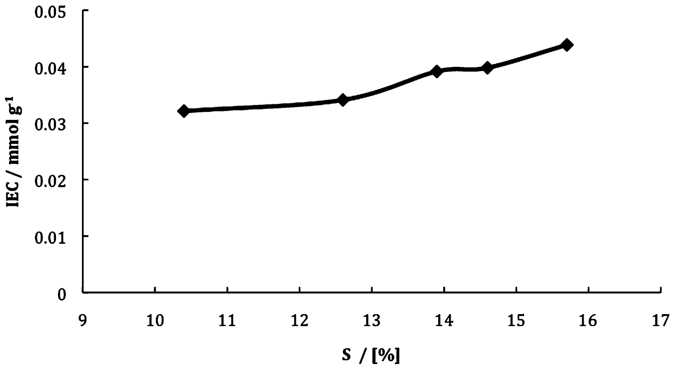

Fig. 5. Ion-exchange capacity (IEC) as a function of degree of sulfonation (*S* (%)).

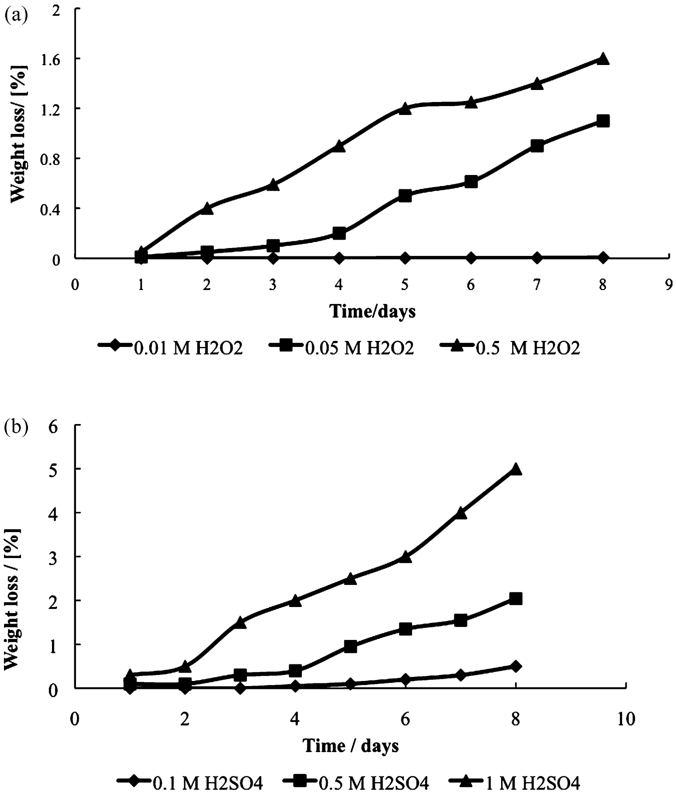

Fig. 6. Tolerance of sulfonated film in different concentrations of (a) H_2O_2 and (b) H_2SO_4 solution.

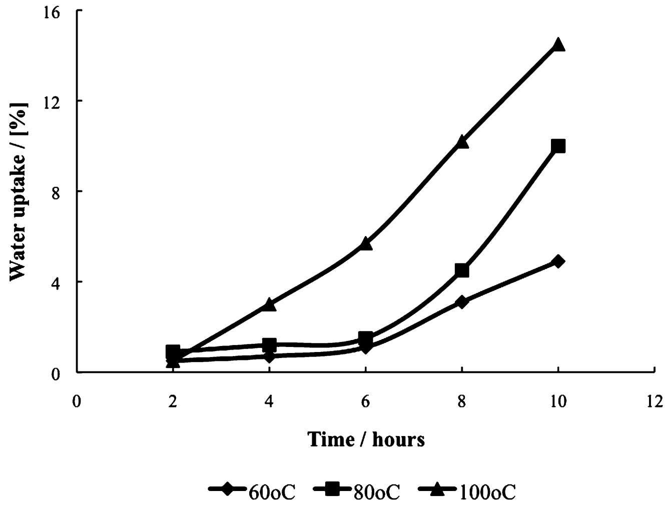

Fig. 7. Water uptake of sulfonated film with respect to time at different temperatures.

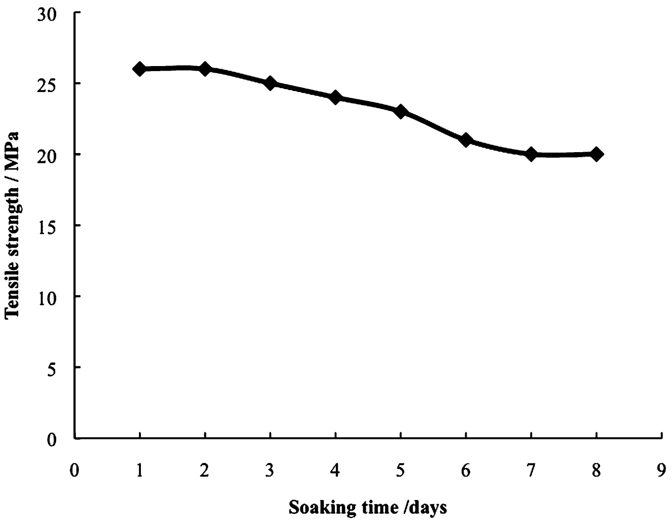

Fig. 8. Tensile strength versus soaking time of sulfonated film in H_2O at 80 °C for 5 days.

uptake results in membrane swelling, mechanical fragility, and low dimension stability [17,18]. In this study, there was small change in weight of sulfonated film (low water uptake) at 60 °C. A small increase of absorbed water content was observed after 6 h at 80 °C but water uptake was increased at 100 °C at the initial soaking time (2 h). After 10 h soaking time, the maximal water uptake (14.5%) was observed at 100 °C. With increasing temperature, the increase in the content of the sulfonated styrene side chain grafts leads to a decrease in the PET domain in the membrane, resulting in the decrease of the hydrophobicity of the membrane. So, the radiation-grafted membrane can be assumed to have larger hydrophilic part, with more dispersed water channels at higher temperature that might facilitate water transport together with the protons. The water uptake of the film (this study) is low but may be sufficient for facilitating proton transport in the fuel cell. Also low swelling made the membrane possess excellent dimensional stability and mechanical strength at the hydrated state.

Fig. 8 shows tensile strengths as a function of soaking time of PEMs in aqueous media at 80 °C for 8 days. Here tensile strength slightly decreases with increasing soaking time in water and after 6 days soaking time, the tensile strength remains constant. So, the treated membranes still retained excellent mechanical properties (e.g., flexibility, toughness) without any practical change in appearance.

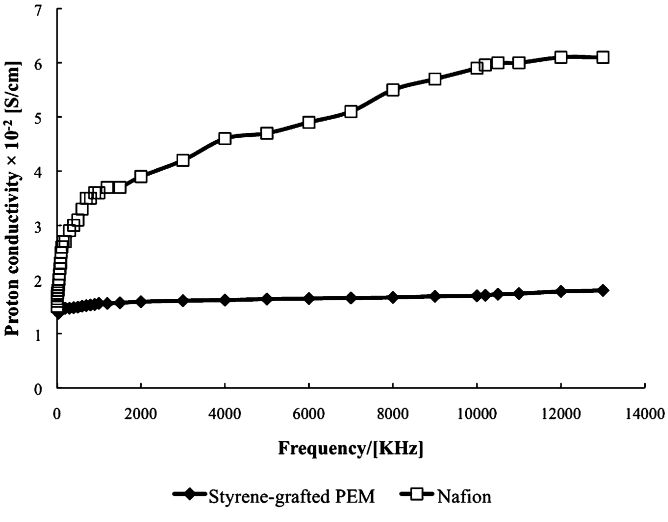

Fig. 9. Proton conductivity of the sulfonated PET film and Nafion at different frequencies.

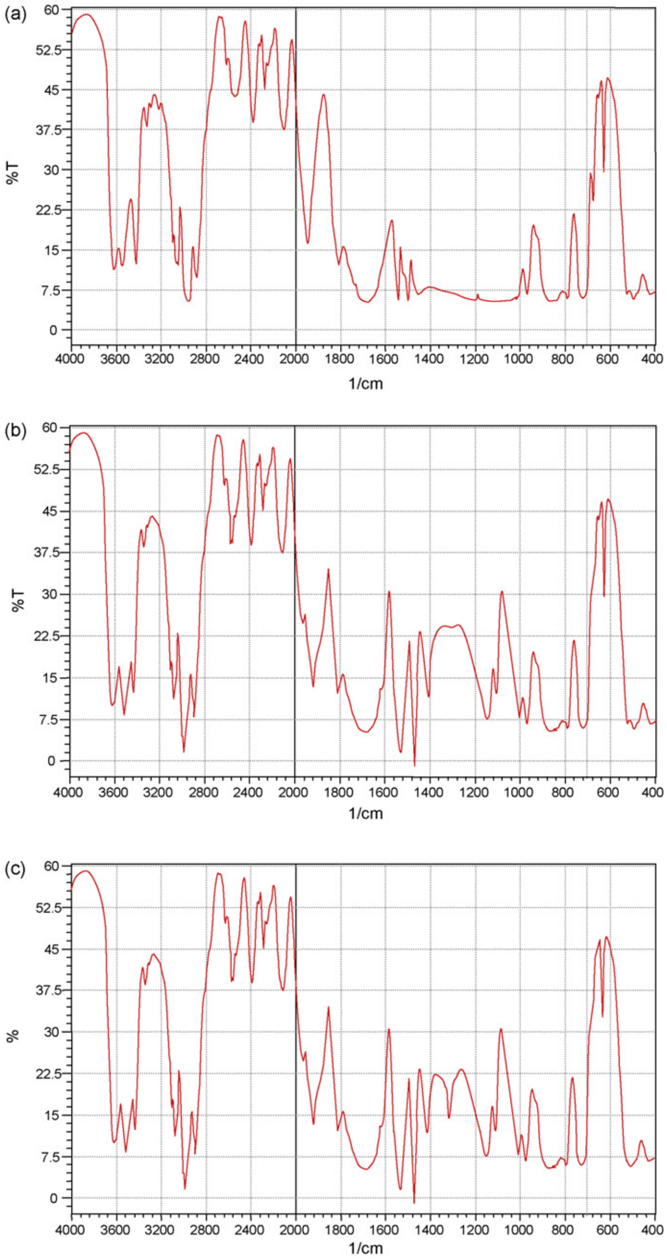

Fig. 10. IR spectrum of original (a), grafted (b) and sulfonated PET film (c).

The variation of resistance of sulfonated PET films with different frequency ranging from 1 to 13,000 kHz was observed at constant temperature (30 °C) and a relative humidity (RH) of 80%. The measurement was not taken at hydrous condition (RH of 100%) so, the membranes may lose water slowly, with a resulting loss of conductivity and flexibility. The resistance of Nafion was also measured with the same instrument under same condition for comparison. The resistance of the PET film decreases with increasing frequency up to 1500 kHz then remain almost constant with increasing frequency up to 13,000 kHz. The maximum value of resistance at minimum frequency for sulfonated PET film was observed to be 0.455 kΩ. As the proton conductivity affects the performance of a fuel cell directly through ohmic losses, in order to obtain a high power density, the area of resistance of the membrane should be low. The observed proton conductivity of the membranes (0.015 S cm^{-1}) is four times lower than that of Nafion (0.06 S cm^{-1}) (Fig. 9). The proton conductivity is directly dependent on the degree of sulfonation [19], so the styrene grafted PEM with low degree of sulfonation has lower proton conductivity than Nafion. According to previous studies on some of the grafted, post-sulfonated and direct polycondensated membranes [20–22], the proton conductivity is around a magnitude order of 10^{-3} to 10^{-4} S cm^{-1} under the hydrous conditions. In that respect, the electrical properties of sulfonated PET film will be a significant improvement in its capacity as an electronically insulator membrane required in fuel cells.

Fig. 10 represents FTIR spectra of an original, grafted and sulfonated PET film. PET shows several peaks in the ranges of 1450–1600 cm^{-1} for C=C (aromatic ring); 3600 cm^{-1} for –OH; 1650–1750 cm^{-1} for C=O (ester); 3000–3100 cm^{-1} for C–H (aromatic ring) by which PET could be confirmed. After grafting of styrene monomer on the PET film, peak intensity is increased at 1450–1600 cm^{-1}, which proves the monomer was grafted. Sulfonation at the grafted PET film was confirmed by showing two peaks at 1146 cm^{-1} (asymmetrical stretch) and 1056 cm^{-1} (symmetrical stretch) for sulfonic acid group.

After considering all of its characteristics, the newly prepared PEM gives high utility with lower cost, higher mechanical and oxidative stability, better acid resistivity, and moderate proton conductivity in long term operations. So, the prepared PEMs can contribute to a very significant improvement in nonflourinated membrane science, to function in fuel cells. In addition, PEMs for fuel cell have attracted much interest as a convenient and viable alternative source of power, with promising potential to reduce the excess consumption of fossil fuel and discharge of CO_2. Besides, there is no sound pollution during the operation of fuel cell. The fossil fuels emit a large amount of CO_2, a major green house gas that is largely responsible for global warming. In the future, fossil fuels crisis may also appear. In saving the earth from pollution, the eco-friendly fuel cell technology will be a better alternative to fossil fuels.

4. Conclusion

Polymer electrolyte membranes (PEMs) for fuel cells, also termed as proton exchange membranes, have been successfully prepared by UV-radiation grafting of styrene onto PET films and consequent selective sulfonation by chlorosulfonic acid. The PEMs displayed excellent thermal, mechanical and electrical properties as well as chemical stability in acidic and hydrogen peroxide media. The maximum ion-exchange capacity (IEC) and the proton conductivity of the PEM were measured to be 0.04385 mmol g^{-1} and 0.015 S cm^{-1}, respectively. The proton conductivity of the membrane is lower than that of the Nafion membrane (i.e., expecting lower fuel cell performance), but they are a lot cheaper compared to Nafion or other equivalent membranes.

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References

[1] P.A. Dworjanyn, J.L. Garnett, J. Polym. Chem. Polym. Lett. Ed. 26 (1988) 135–138.
[2] J.R. Hollahan, Plasma Chemistry Industrial Application, Wiley, New York, 1974.
[3] C.A. Wilkie, C. Deacon, Eur. Polym. J. 32 (1996) 451–455.
[4] S. Hasegawa, Y. Suzuki, Y. Maekawa, Radiat. Phys. Chem. 77 (2008) 617–621.
[5] T.R. Dargaville, G.A. George, D.J.T. Hill, A.K. Whittaker, Prog. Polym. Sci. 28 (2003) 1355–1376.
[6] M.M. Nasef, E. Hegazy, Prog. Polym. Sci. 29 (2004) 499–561.
[7] J. Chen, M. Asano, T. Yamaki, M. Yoshida, J. Membr. Sci. 256 (2005) 38–45.
[8] M.M. Nasef, J. Appl. Polym. Sci. 77 (2000) 1003–1012.
[9] M. Doyle, G. Rajendran, Handbook of Fuel Cells, vol. 3, John Wiley & Sons, 2003, pp. 351–395.
[10] P.R.S. Reddy, G. Agathian, A. Kumar, Radiat. Phys. Chem. 73 (2005) 169–174.
[11] M.H. Rao, K.N. Rao, Radiat. Phys. Chem. 26 (1985) 669–672.
[12] M.P. Carreon, R. Aliev, R. Ocampo, G. Burillo, Polym. Bull. (Berlin) 44 (2000) 331–335.
[13] M. Kattan, E. El-Nesr, J. Appl. Polym. Sci. 102 (2006) 198–203.
[14] K. Kang, P.H. Kang, Y.C. Nho, J. Appl. Polym. Sci. 99 (2006) 1415–1428.
[15] S. Takahashi, H. Okonogi, T. Hagiwara, Y. Maekawa, J. Membr. Sci. 324 (2008) 173–180.
[16] X.H. Ma, C.J. Zhang, G.Y. Xiao, D.Y. Yan, J. Power Sources 188 (2008) 57–63.
[17] K.D. Kreuer, J. Membr. Sci. 185 (2001) 29–39.
[18] X.H. Ma, L.P. Shen, C.J. Zhang, G.Y. Xiao, D.Y. Yan, G.M. Sun, J. Membr. Sci. 310 (2008) 303–311.
[19] X. Glipa, M.E. Haddad, D.J. Jones, J. Roziere, Solid State Ionics 97 (1997) 323–331.
[20] P. Staiti, F. Lufrano, A.S. Arico, E. Passalacqua, V. Antonucci, J. Membr. Sci. 188 (2001) 71–78.
[21] J.M. Bae, I. Honma, M. Murata, T. Yamamoto, M. Rikukawa, N. Ogata, Solid State Ionics 147 (2002) 189–194.
[22] J. Jouanneau, R. Mercier, L. Gonon, G. Gebel, Macromolecules 40 (2007) 983–990.