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Assessment of the Properties of Proton Exchange Membrane in Formulated Polystyrene/ Polyisopreneblend for Fuel Cell Application

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Abstract

Researchers in recent times in the field of Fuel Cell Technology are passionately seeking alternative polymerelectrolyte membranes (PEMs), being the central component of fuel cell because of the very high cost and some disadvantages associated with the current commercial membrane, Nafionionomer. This has prompted this study by synthesizing PEM from formulated polystyrene/polyisoprene via sulphonation process at initial acid concentration (0.001 mol/L) to obtain membranes of different Ion Exchange Capacities (IECs) and Degrees of Sulphonation (DS). IECs obtained were 0.879, 1.314, 2.07 and 2.63 mmol/g for formulated PS/PI of 20/80, 30/70, 40/60 and 50/50 %, respectively with corresponding DS of 1.6, 2.38, 3.62 and 4.76 %, respectively. But DS increased with increase in the initial acid concentration as 0.0010, 0.0013, 0.0016, 0.0019 and 0.0023 mol/L achieved DS of 2.37, 14.74, 32.89,38.56 and 41.97, respectively. The membrane achieved proton conductivity ranging between 10^{-3} to 10^{-2} S/cm, which competed well with the commercial state-of-the-art membrane. The water uptake and retention ability as well as the swelling ratio properties were found to be superior to the polyisoprene membrane of previous study; while the polyisoprene membrane of lower DS (28.84 %) absorbed 68.87 % water by weight within 6 days, the newly synthesized membrane of ≥ 2.5 -fold higher DS (41.97 %) absorbed 66.78 % by weight of water in 10 days. This shows a better swelling resistance that can stand the test of time under PEM fuel cell operation.

Keywords: Fuel cell, PEM, Sulphonation, IEC, DS, Sulphur content, Water uptake, Polystyrene, Polyisoprene.

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1.0 INTRODUCTION

The drive for efficient but affordable **I** proton exchange membranes that can serve as alternatives to Nafion®ionomer for fuel cell application has continued to engage researchers in the field of fuel cell technology. Fuel cell. especially, proton exchange membrane fuel cell is recognized as a very viable, efficient, portable and green energy source due to its high power density, lightweight and compact, rapid response to varying load, quick start up and low operating temperature (Idibie, 2017a). However, realizing its full commercialization has been

impeded because of the very high cost of the membrane, and it is the central component of the fuel cell because it is sandwiched between the anode and cathode electrodes. Besides, it conducts the protons produced at the anode, as well as acts as a barrier between the fuel and oxidant to prevent fuel oxidation and over potential (Idibie, 2017b). Apart from the non-affordability near of the perflorinatedionomerNafion due to monopoly, it dehydrates at high temperature (>80°C) with accompanied loss of conductivity (Quanet al., 2005; Cheddie and Munroe, 2005; Shibasaki et al., 2005). Although, the membrane exhibits high proton conductivity

 $(\sigma \ge 10^{-2} \text{ S cm}^{-1})$ and durability under fuel cell operation, but the aforementioned disadvantages have necessitated the drive for alternatives. Meanwhile effort in the past has directed in synthesizing been proton exchange membrane for fuel cell using polyisoprene. Although the properties of the synthesized polvisoprene elastomer demonstrated competitive proton conductivity with the state-of-the-art Nafion that could qualify it for PEM fuel cell application, but it showed the tendency of absorbing excess water at high degree of sulphonation that produced the high proton conductivity $(10^{-3} - 10^{-2} \text{ Scm}^{-1})$ with attendant high swelling. This is a situation that could lead to dissolution and breakdown of the membrane under use. Therefore. polyisoprene with a good prospect for PEM fuel cell application should be reinforced to overcome this challenge. On this basis, this study aims to explore polymer blending effects on polyisoprene choice for fuel cell application by blending it with polystyrene and then assess the characteristic effects, especially the water absorption and its proton conductivity for fuel cell application. The selection of polystyrene is harped on its properties such as stiffness, hardness. thermal stability (Ashby, 2013), light-weight and non-toxicity. Apart from these qualities that it possesses, it is an inexpensive plastic material (Satterthwaite, 2017). Therefore, we presumed that the blend of polystyrene and polyisoprene will offer the advantages of plastic-rubber domain, particularly in its water absorption resistance and, thus durable under fuel cell application.

2.0 MATERIALS AND METHODS

2.1 Materials

Analytical grade (> 98 %) chemicals were used in this study and where obtained from (Charlec Laboratories, Nigeria). These include: 1, 2, dichloroethane, chlorosulphonic toluene, acid, ethanol. commercial polyisopreneelastomer and polystyrene (Charlec Laboratories, Nigeria and Karbochem RSA). Four neck round bottom flask fabricated reactor and multimeter.

2.2 Methods

i. Blend formulation and sulphonation of polystyrene/polyisoprene

Each portion of polyisoprene and polystyrene were separately dissolved in toluene and I, 2, dichloroethane. respectively. where polystyrene/polyisoprene (PS/PI 20 g) ranging from 20/80, 30/70, 40/60 and 50/50 % which were formulated, were sulphonated using chlorosulphonic acid in a four-neck round bottom flask fabricated reactor according to the method described by Idibie and co-worker (Idibie, 2017a; Idibie et al., 2009). The sulphnation reaction was carried out using different initial concentrations of the acid (0.0010, 0.0013, 0.0016, 0.0019 and 0.0023 mol/L) that were cooled in an iced bath in a gradual addition to the solution mixture of PS/PI that was undergoing vigorous stirring in the four-neck round bottom flask reactor, under argon atmosphere, and at room temperature. The reaction was allowed to run at varying time between 1 to 20hrs initially and then shorten to 18 hrs of equilibrium time. At each set time, the reactions were terminated by the addition of ethanol, and the precipitated sulphonated were polystyrene/polyisoprene (SPS-PI) recovered and washed using deionised water until 6 -7 pH of wash was attained. Each batch of the sulphonated copolymer was later oven dried (80°C) between 1–2 hrs.

ii. Ion exchange capacity and degree of sulphonation determination

Ion Exchange Capacity (IEC) and Degree of Sulphonation (DS) of the Sulphonatedpoly (styrene-isoprene) were determined through elemental analysis of the percentage of sulphur content (Sc) present in the sulphonated PS/PI. Once the Sc was determined, the IEC was then calculated with Equation (1) (Bebin, 2005):

Where: Sc = sulphur content in percentage weight rate, MWs = molecular weight of sulphur, while 1000 is the multiplication factor to obtain IEC value in mmol/g. Thereafter, the respective values of IEC obtained from Equation 1 were used to arrive at the varying degrees of sulphonation of sulphonatedPS-PI using Equation 2 (Paturzo *et al.*, 2005):

$$DS = \frac{MW_{SPS-PI} \times IEC}{MW_{PS-PI} \times MW_{SO_3H}} \dots \dots \dots \dots (2)$$

Where: MWSPS-PI= the molecular weight of the sulphonated polystyrene-polyisoprene, IEC = ion exchange capacity, and MWPS-PI = the Molecular weight of unsulphonated polystyrene-polyisoprene and MW_{SO_3H} = molecular weight of SO₃H.

iii. Casting of membrane into thin film

SPS-PI was cast into a thin membrane according to the method described by Idibie, 2018a. 10 g of sulphonated PS/PI was dissolved in 250 ml mixture of toluene/ dichloroethane at elevated temperature, and which form a casting solution. The solution was then cast onto a clean polymer paper support with the use of a laboratory doctor blade casting machine that was set to different thicknesses with the aid of feeler gauges. The casting was carried out by drawing the casting head of the blade along the length of the substrate, and allowed to cure for 3 days by exposing it to air, and followed by oven drying at temperature below 80°C for a period of another 2 hrs, and later vacuum dried for another 2 hrs to take away any residual solvent.

iv. Measurement of proton conductivity

Measurement of the proton conductivity of the synthesized membrane was carried out using alternating current impedance over a frequency range of 1-106Hz in 1M H₂SO₄ as an electrolyte (Idibie*et al.*, 2018). This involves using membranes of different degrees of sulphonation ranging from 2.37 – 41.97% and with varying thicknesses (120, 150, 220, and 270 μ m) where the intersection value of the high frequency impedance curve was taken as the membrane resistance, and

the proton conductivity was thus calculated according to equation 3:

$$\sigma = \frac{T}{RS}$$
.....(3)

Where: σ = proton conductivity (S/cm), T (cm) = membrane thickness and S (cm²) = surface area of the membrane and R = resistance obtained from the impedance plane.

v. Water absorption and desorption capacities

The water absorption capacity of the synthesized membrane was studied by immersing a known weight (0.17 g) and different dimensions of membrane (120, 150, 220, and 270 μ m) in a beaker containing distilled water for a few days until they were saturated and removed from the water, and the water uptake was determined using a digital weighing balance. The weight difference between the wet and dry membrane was obtained via Equation 4: Water absorption (%)

$$=\frac{W_{wet}-W_{dry}}{dry} \times 100..... (4)$$

Where: W_{wet} and W_{dry} = weights (g) of the wet membranes and dry membranes, respectively.

Also, the water desorption capacity of the membrane was also determined by immersing (0.17g)of the synthesized membrane in distilled water for 8 hrs and then removed and exposed to air and at different temperatures of 24, 35, 45, 65 and 80°C, thereafter the weight of the membrane was measured at intervals of one hour, and the percentage of water desorbed was determined using Equation 5.

Water desorption (%)

$$= \frac{W_{wet} - W_{dry(t)}}{W_{wet} - W_{dry}} \times 100 \dots (5)$$

Where: W_{wet} = weight of wet membranes and W_{dry} = weight of the dry membranes at time

(t) in grams, respectively. While the membrane swelling ratio was determined according to equation 6:

$$\frac{T_{wet} - T_{dry}}{T_{dry}} \times 100.....(6)$$

Where: T_{wet} = thickness of wet membranes and T_{dry} = thickness of dry membranes.

3.0 RESULTS AND DISCUSSION

3.1 Sulphur content (Sc), ion exchange capacity (IEC) and degree of sulphonation (DS)

The sulphur content, ion exchange capacity degree of sulphonation of and the sulphonated membranes are a function of the acid used in the suphonation process. Result as presented in Table 1 showed that the formulated polymerblend, was a good candidate of sulphonation, and the amount of Sc increased with time of sulphonation and the ratio of PS/PI. While the lowest time of 1 hr achieved Sc of 0.0120 %, 20hrs of reaction achieved Sc of 0.0280 % using 20/80 % of PS/PI, whereas 50/50 % PS/PI at 1 hr and 20 hrs of sulphonation achieved Scof 0.0680 and 0.0841 %, respectively. This shows that the more the resident time, the more the acid got attached to the polymer matrix. Also, the more the polymer blend, the more the sites of This trend was also observed on the IEC and DS of the sulphonatedpolymer blend. Figures 1 and 2 are the IEC and DS of the SPS-PI as a function of time. The figures showed that at 1 hr of sulphonation, IECs obtained were 0.375, 1.437 and 2.125 mmol/g 0.875, for formulated PS/PI of 20/80, 30/70, 40/60 and 50/50, respectively, and at 20hrsthey were 0.879, 1.314, 2.07 and 2.63 mmol/g, respectively. In the same vein, DS obtained at 1hr were 0.68, 1.57, 2.61 and 3.85 %, respectively, and at 20 hrs DS achieved were 1.6, 2.38, 3.62 and 4.76 %, respectively. The concentration of acid in the membrane is known to be related to the amount of ionic groups in the membrane, hence it is referred to as the measure of the degree of sulphonation(Idibie et al., 2018b), while the ion exchange capacity (IEC) is noted as an indicator of the proton conduction sites (Sagetha, 2005).

3.2 Variation of initial acid concentration on degree of sulphonation

The effect of varying the initial acid concentrations was also studied. The result as presented in Figure 3 showed that DS increased with increase in the initial acid concentration, as 0.0010, 0.0013, 0.0016, 0.0019 and 0.0023 mol/L achieved DS of 2.37, 14.74, 32.89, 38.56 and 41.97, respectively.

| Time, hr | Sulphur content (&) | | | | |
|----------|---------------------|-------------|-------------|-------------|--|
| | 20/80 PS/PI | 30/70 PS/PI | 40/60 PS/PI | 50/50 PS/PI | |
| 0 | 0 | 0 | 0 | 0 | |
| 1 | 0.0120 | 0.0280 | 0.0460 | 0.0680 | |
| 3 | 0.0160 | 0.0300 | 0.0480 | 0.0700 | |
| 6 | 0.0200 | 0.0320 | 0.0540 | 0.0740 | |
| 9 | 0.0220 | 0.0340 | 0.0560 | 0.0760 | |
| 12 | 0.0240 | 0.0360 | 0.0600 | 0.0800 | |
| 15 | 0.0260 | 0.0400 | 0.0620 | 0.0820 | |
| 18 | 0.0280 | 0.0420 | 0.0640 | 0.0840 | |
| 20 | 0.0284 | 0.0423 | 0.0642 | 0.0841 | |

 Table 1: Sulphur content of sulphonated PS/PI at varying ratios

attachment of the acid to the matrix of the polymer.



Figure 1: IEC of the SPS-PI as a function of time at varying PS/PI ratios.



Figure 2: DS of the SPS-PI as a function of time at varying PS/PI ratios.



Figure 3: Variation of initial acid concentration on DS.

3.3 Proton conductivity

The proton conductivity of the synthesized membrane was found very promising, ranging between 10^{-3} to 10^{-2} S/cm. Result presented in Figure 4 showed that conductivity increased with increase in DS

but inversely to the thickness of the membrane.



Figure 4: Proton conductivity versus degree of sulphonation.

This pattern is in agreement with previous works (Idibie, 2017a; Idibie*et al.*, 2009; Idibie*et al.*, 2018a; Abdukareem*et al.*, 2010). For the lowest and highest degrees of sulphonation 2.37 and 41.97 %, the proton conductivities for membranes of 0.120, 0.150, 0.220 and 0.270 mm were 7.1×10^{-3} and 2.5×10^{-2} , 5.9×10^{-3} and 2.0×10^{-2} , 3.9×10^{-3} and 9.9×10^{-3} and 3.2×10^{-3} and 9.3×10^{-3} S/cm², respectively.

3.4 Hydrodynamic properties

water absorption and desorption The capacities of the synthesized membrane as investigated as shown in both Tables 2 and 3 revealed that as the degree of sulphonation increased, water absorption per gramme of dry membrane increased. This pattern was also in agreement with previous works (Idibie, 2017a; Idibieet al., 2009; Idibieet al., 2018a; Abdukareemet al., 2010), and this phenomenon is harped on the fact that high degree of sulphonation leads to high ion exchange capacity. Ranging from the lowest to highest DS(2.37, 14.74, 32.89, 38.56 and 41.97%), the lowest and highest water uptakes were 4.89 and 21.64 %,10.57 and 22.33 %, 24.37 and 41.29 %, 29.45 and 49.01 % and 38.98 and 66.78 %, respectively, indicating a moderate water uptake ability of the synthesized membrane of all the DS except that of the highest DS (41.97) that was above 50 %.

| Time (days) | DS 2.37% | DS 14.74% | DS 32.89% | DS 38.56% | DS 41.97% |
|-------------|----------|-----------|-----------|-----------|-----------|
| 0 | 0 | 0 | 0 | 0 | 0 |
| 1 | 4.89 | 10.57 | 24.37 | 29.45 | 38.98 |
| 2 | 8.17 | 14.36 | 27.89 | 33.09 | 45.32 |
| 3 | 11.38 | 17.61 | 30.45 | 37.23 | 49.73 |
| 4 | 14.04 | 19.97 | 33.16 | 40.56 | 53.29 |
| 5 | 16.79 | 21.67 | 35.09 | 42.23 | 57.45 |
| 6 | 1823 | 23.76 | 37.36 | 44.89 | 60.09 |
| 7 | 19.98 | 22.10 | 39.26 | 46.08 | 63.91 |
| 8 | 20.37 | 21.90 | 40.17 | 47.48 | 65.23 |
| 9 | 21.64 | 22.32 | 41.28 | 49.08 | 66.78 |
| 10 | 21.64 | 22.33 | 41.29 | 49.01 | 66.78 |

Table 2: Water uptake capacity at varying degree of sulphonation at constant membrane thickness (220 µm)

Table 3: Water uptake capacity at varying membrane thickness at constant degree of sulphonation (38.56 %)

| Time (days) | 120 µm | 150 µm | 220 µm | 270 µm |
|-------------|--------|--------|--------|--------|
| 0 | 0 | 0 | 0 | 0 |
| 1 | 32.67 | 30.51 | 29.45 | 20.43 |
| 2 | 36.01 | 34.57 | 33.09 | 24.24 |
| 3 | 40.10 | 38.71 | 37.23 | 37.37 |
| 4 | 43.91 | 41.10 | 40.56 | 30.56 |
| 5 | 46.85 | 43.65 | 42.23 | 33.21 |
| 6 | 49.32 | 45.18 | 44.89 | 36.56 |
| 7 | 50.87 | 47.23 | 46.08 | 38.21 |
| 8 | 51.78 | 49.30 | 47.48 | 40.32 |
| 9 | 52.91 | 51.23 | 49.08 | 42.94 |
| 10 | 52.91 | 51.23 | 49.01 | 42.97 |

This superior property is а over monopolyisoprene that was conducted in previous work (Idibie, 2017a) where membrane of lower DS (28.84 %) absorbed 68.87 % by weight within 6 days, whereas the newly synthesized blend ofpoly(styreneisoprene) of 41.97 % absorbed 66.78 % by weight of water in 10 days. This is an indication that the synthesized membrane is tougher and of better moderate water absorption ability, which can stand the test of time under PEM fuel cell operation without dissolving or breaking down. Again, all the membranes investigated showed initial rapid water absorption but decreased with time until saturation was accomplished on the 10th day. This shows that the membrane can hold up good amount of water for proper proton mobility and conductivity even at the lowest DS (Idibie, 2017a; Idibie, 2018b). The result of the effect of membrane thickness on water absorption as shown in Table 3, revealed that water absorption increased as the thickness of the membrane decreased, hence membrane of thicknesses 120, 150, 220 and 270 μ m at constant degree of sulphonation (38.56 %) absolved 54.92, 51.23, 49.01 and 42.97 %, respectively.

The result on the investigation of the swelling ratio of the membranes being the percentage change in membrane thickness per gramme of the dry membrane, as shown in Figure 5 revealed that the swelling ratio is

inversely proportional to the membrane thickness, as smaller the thickness of the membrane the higher the swelling ratio. The swelling ratios obtained were 14, 9, 5, and 2 % for membranes of 120, 150, 220 and 270 μm, respectively. Relating the swelling ratio to previous work, the swelling ratio of monopolyisoprene was higher > 20 % (membrane thickness 120 μm) (Idibie, 2017a) compared to the copolymer that was < 15 %(membrane thickness 120 µm).



Figure 5: swelling ratio versus membrane thickness.

On the other hand, the result obtained on investigating the effect of temperature on water desorption of the membrane as presented in Table 4, showed that the synthesized membrane lost water as temperature increased, especially at the highest temperature (80oC). This implies that if the membrane is to be used at a very high temperature ($\geq 80^{\circ}$ C) for over a day, proper

attention to maintain its humidification is therefore needed to prevent dehydration and thus loss of proton mobility and conductivity. Comparatively, previous work (Idibie, 2017) showed that the monopolyisoprene dehydrated faster by losing water higher (87 %) in one day but the newly synthesized PS/PI blend experienced reduced water desorption (78 %) in same one day, with the advantage of maintaining ionic mobility and

Table 4: Water losing capacity at varying temperature and at constant membrane thickness (220 μm)

| $\begin{array}{cccccccccccccccccccccccccccccccccccc$ | Time (days) | 24ºC | 35ºC | 55ºC | 65°C | 80ºC |
|--|-------------|-------|-------|-------|-------|-------|
| 285.7396.3698.2399.21100391.3298.9998.97100100495.2499.41100100100597.9699.49100100100698.0499.49100100100799.2199.49100100100899.21100100100100 | 1 | 78.67 | 93.79 | 97.96 | 98.69 | 99.89 |
| 391.3298.9998.97100100495.2499.41100100100597.9699.49100100100698.0499.49100100100799.2199.49100100100899.21100100100 | 2 | 85.73 | 96.36 | 98.23 | 99.21 | 100 |
| 495.2499.41100100100597.9699.49100100100698.0499.49100100100799.2199.49100100100899.21100100100100 | 3 | 91.32 | 98.99 | 98.97 | 100 | 100 |
| 597.9699.49100100100698.0499.49100100100799.2199.49100100100899.21100100100100 | 4 | 95.24 | 99.41 | 100 | 100 | 100 |
| 698.0499.49100100100799.2199.49100100100899.21100100100100 | 5 | 97.96 | 99.49 | 100 | 100 | 100 |
| 799.2199.49100100100899.21100100100100 | 6 | 98.04 | 99.49 | 100 | 100 | 100 |
| 8 99.21 100 100 100 100 | 7 | 99.21 | 99.49 | 100 | 100 | 100 |
| | 8 | 99.21 | 100 | 100 | 100 | 100 |

proton conductivity at prolonged time.

4.0 CONCLUSION

Synthesis of proton exchange membrane from well formulated polystyrene/ а polyisoprene blend showed promising and superior properties over the monpolymer, polyisoprene (PI). Although both the mono and polymer blend were able to achieve the desired proton conductivity (10⁻² S/cm), however, the water retention ability as well as the swelling ratio properties of the PS/PI were superior to the PI. While the PI dehydrated faster by losing water higher (87 %) in one day whereas the PS/PI experienced reduced water desorption (78%) in same one day, with the advantage of maintaining proton conductivity at prolonged time. Previous work on the swelling ratio of PI membrane was found higher > 20 % (120 µm) compared to the PS/PI membrane that was < 15 % of the same membrane thickness (120 µm). Finally, while PI membrane of

lower DS (28.84 %) absorbed 68.87 % water by weight within 6 days, the newly synthesized polymer blend of \geq 2.5-fold higher DS (41.97 %) absorbed 66.78 % by weight of water in 10 days. This shows a better water absorption resistance as compared to the PI membrane.

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7.0 CONFLICT OF INTEREST

There was no conflict of interest.

8.0 AUTHORS' CONTRIBUTIONS

All the authors contributed from the bench work to the write up. They all made financial commitment. However, the corresponding author's contribution was more both in concept, execution and funding of the project.

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