ภาคผนวก ค

ผลงานที่ได้ไปนำเสนอในงานประชุมวิชาการ

ผลงานที่ได้ไปนำเสนอในงานประชุมวิชาการ มี 1 ผลงาน ดังนี้
ชื่อเรื่อง "Low toxicity Simplified Synthetic Route for Polysulfone-based Solid-State Alkaline Electrolytes Membranes" ใน 38th Congress on Science and Technology of Thailand, October 17 – 19, 2012, Chiang Mai, Thailand.

Low toxicity Simplified Synthetic Route for Polysulfone-based Solid-State Alkaline Electrolytes Membranes

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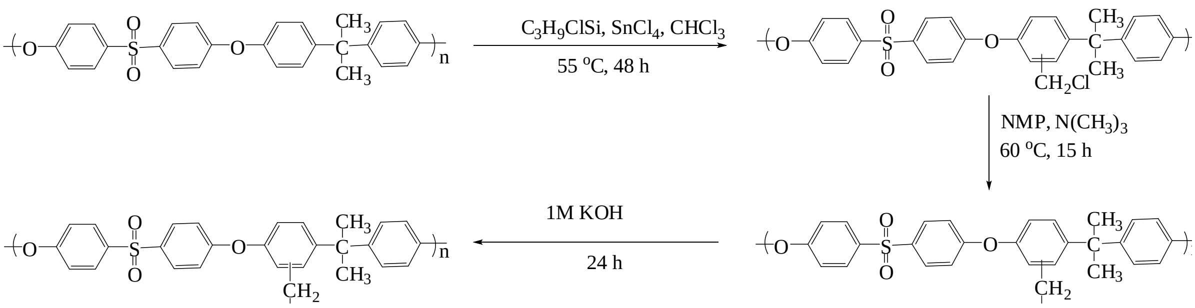
Abstract: Recently the alkaline system for fuel cell enhance their presence because of possibility of no-precious-metal catalyst and low over potential at cathode reaction. Alkaline anion exchange membranes (AEM) of high ionic conductivities are made from polysulfone (PS) by adding a chloromethyl pendant group to the PS, follow by reacting the chloromethyl group with amine to form quarternary ammonium pendant groups which act as the counter ion for hydroxide anion. To avoid the use of such hazardous materials, in our work we used paraformaldehyde, chlorotrimethylsilane, N-methylpyrrolidone and ethanol as a reagent for providing conversion. This is a simplified synthetic route to quaternized polysulfone (QAPS) using reagents of low carcinogenic toxicity. QAPS was prepared by chloromethylation followed by quaternization using trimethylamine as an amination agent. Finally, alkalinization was carried out to transform the PS from the Cl^- form to the OH^- form using 1M KOH solution. ^1H NMR (300 MHz, $CDCl_3$, DMSO-d_6, 25 °C, TMS) and FT-IR spectroscopies were used to confirm the AEM structure at each synthetic step. Water uptake (WU%) and swelling degree (SD%) were estimated to determine the percentage of water uptake by the membrane, and the ion-exchange capacity (IEC) was determined by the back titration method. The swelling behavior of polysulfone-based solid-state alkaline electrolyte membrane was closely related to the degree of water uptake (25 WU%, 7.5 SD%) and the ion-exchange capacity was 1.05 mmol g^{-1}, which is sufficient for electrolyte membranes used in alkaline fuel cells



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Keywords: Simplified synthetic route, polysulfone, solid-state alkaline, electrolytes membrane

Low toxicity simplified synthetic route for polysulfone-based solid-state alkaline electrolytes membranes

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Introduction

Recently the alkaline system for fuel cell enhance their presence because of possibility of no-precious-metal catalyst and low over potential at cathode reaction. Alkaline anion exchange membranes (AEM) of high ionic conductivities are made from polysulfone (PS) by adding a chloromethyl pendant group to the PS, follow by reacting the chloromethyl group with amine to form quarternary ammonium pendant groups which act as the counter ion for hydroxide anion. To avoid the use of such hazardous materials, in our work we used paraformaldehyde, chlorotrimethylsilane, N-methylpyrrolidone and ethanol as a reagent for providing conversion. The objective of this research is a simplified synthetic route to quaternized polysulfone (QAPS) using reagents of low carcinogenic toxicity.

Fig. 1. Preparation of QAPS with OH^- anions via chloromethylated intermediates

Methodology

Polysulfone (PS) was chloromethylated using chlorotrimethylsilane as a chloromethylation reagent, resulting in the formation of Chloromethylated Polysulfone (CMPS). CMPS was converted to a quaternized form using trimethylamine and precipitated into ethanol. The powder was dissolved in N-methylpyrrolidone, followed by aminated with a 25 wt% trimethylamine. The resulting solution was cast onto a flat glass plate and dried in an oven. The membrane was immersed in KOH solution for 24 h to replace the Cl^- anion in the polymer with OH^-. ¹H NMR (300 MHz, $CDCl_3$, DMSO-d_6, 25 °C, TMS) and FT-IR spectroscopies were used to confirm the AEM structure at each synthetic step. Water uptake (WU%) and swelling degree (SD%) were estimated to determine the percentage of water uptake by the membrane, and the ion-exchange capacity (IEC) was determined by the back titration method.

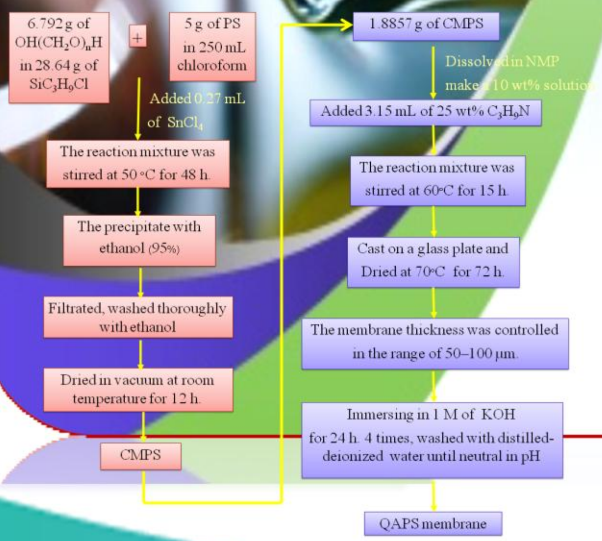

Fig. 2. Chart showing the preparation of QAPS.

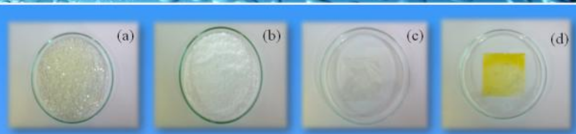

Fig. 3. The synthetic products of the (a) PS, (b) CMPS, (c) QAPS-Cl and (d) QAPS-OH.

Result and Discussion

¹H NMR spectra corresponding to the final product, QAPS-OH. In addition to the well-resolved peaks corresponding to H atoms on the QAPS chain, as shown in Fig. 4a and 4b, a split peak can be observed at δ = 4.6 ppm (d, j = 27 Hz, 2H, CH_2) and a new peak emerges at δ = 5.9 ppm (br, w, 1H, OH^-), indicating that the Cl^- anion has been successfully substituted by OH^- anion, as shown in Fig. 4b.

The IR absorption band of PS clearly indicates the presence of the sulfonic group (O=S=O) in the polymer back bond at 1149.5 and 1238.2 cm⁻¹. The IR absorption bands at 1485.1 and 1585.4 cm⁻¹ are characteristic of the phenyl group. A new characteristic peak with high intensity was observed at 1683.7 cm⁻¹, corresponding to the aminated aromatic salts, as shown in Fig. 5.

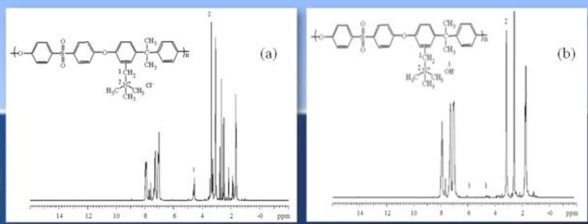

Fig. 4. ¹H NMR spectra of (a) QAPS-Cl and (b) QAPS-OH in DMSO-d6

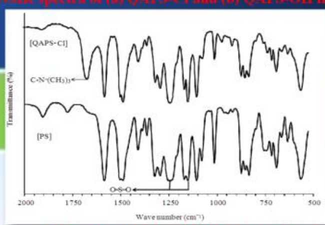

Fig. 5. FT-IR spectra of PS and QAPS-Cl

Conclusion

A simplified synthetic route to quaternized PS using reagents of low toxicity. Result, the swelling behavior of polysulfone-based solid-state alkaline electrolyte membrane was closely related to the degree of water uptake (25 WU%, 7.5 SD%) and the ion-exchange capacity was 1.05 mmol g⁻¹, which is sufficient for electrolyte membranes used in alkaline fuel cells.

Acknowledgement

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References

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