

UNIVERSITI TEKNOLOGI MARA

**PREPARATION AND
CHARACTERIZATION
OF ELECTROSPUN SULFONATED
POLY (ETHER ETHER KETONE)
CROSSLINKED STRONTIUM
FOR PROTON EXCHANGE
MEMBRANE**

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Thesis submitted in fulfillment
of the requirements for the degree of
Master of Science
(Physics)

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AUTHOR'S DECLARATION

I declare that the work in this thesis was carried out in accordance with the regulations of Universiti Teknologi MARA. It is original and is the result of my own work unless otherwise indicated or acknowledged as referenced work. This thesis has not been submitted to any other academic institution or non-academic institution for any degree or qualification.

I, hereby, acknowledge that I have been supplied with the Academic Rules and Regulations for Post Graduate, Universiti Teknologi MARA, regulating the conduct of my study and research.

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ABSTRACT

Poly ether ether ketone (PEEK)s have been considered as a candidate material for membrane in PEMFC due to their good mechanical strength, high chemical, and thermal stability, and high proton conductivity. Strontium (Sr) cross-linked nanofibrous membranes based on sulfonated polyether ether ketone (SPEEK) were successfully prepared using the electrospinning technique. The degree of sulfonation of the SPEEK is 76 % was determined by $^1\text{H-NMR}$ using the area under the peak of the hydrogen shielding at the aromatic ring of the SPEEK. The value of the IEC is 1.92 meq/g and the FTIR showed O=S=O stretching vibrations of SPEEK at 1077 cm^{-1} and 1249 cm^{-1} indicate that SPEEK has successfully sulfonated. Metal-polymer physical cross-linking has been proposed as a way to improve the mechanical and chemical stabilities of the electrospun SPEEK membrane which we found in this finding can help to reduce water uptake compared with the non-crosslinked membrane. Physical cross-linking of Sr-SPEEK membranes were fabricated by the immersion process which was then electrospun into nanofibrous membranes. The optimum voltage, feed rate, and tip to collector distance for electrospinning set up were 12 kV, 0.30 mL/h, and 12 cm respectively. The morphologies of electrospun Sr-SPEEK membrane, fabricated under different concentrations of Sr were presented which show up to 6% concentration of Sr was able to be electrospun. The effect of crosslinking and electrospun membranes on water uptake, proton conductivity, and thermal behaviors was characterized. The expected outcome with the addition of a small amount of Sr has decreased the water uptake. On the electrical properties of the electrospun membrane, the conductivity for the sample with 6% Sr weight percentage has given 0.188 S/cm which is about 80% higher than the film cast Nafion-117 at 80°C as cited before. This particular result is a hallmark of the studies as it shows that a combination of nanostructured with crosslink is proven to work in favor of producing proton exchange membrane fuel cell (PEMFC) which performed much better than Nafion based membrane.

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