



UNIVERSITI PUTRA MALAYSIA

**RADIATION AND TEMPERATURE EFFECTS ON ELECTRICAL
PROPERTIES OF POLY (VINYL ALCOHOL) - SODIUM SALICYLATE
POLYMER ELECTROLYTES**

NOORHANIM BINTI AHAD

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**RADIATION AND TEMPERATURE EFFECTS ON
ELECTRICAL PROPERTIES OF POLY (VINYL
ALCOHOL) - SODIUM SALICYLATE POLYMER
ELECTROLYTES**

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NOORHANIM BINTI AHAD

**MASTER OF SCIENCE
UNIVERSITI PUTRA MALAYSIA**

2011

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PROPERTIES OF POLY (VINYL ALCOHOL) - SODIUM SALICYLATE
POLYMER ELECTROLYTES**

By

NOORHANIM BINTI AHAD

**Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia,
in Fulfilment of the Requirements for the Degree of Master of Science**

October 2011

UPM

DEDICATION



I dedicate this thesis to my family especially my beloved father and mother and also to all my friends.

Abstract of the thesis presented to the Senate of Universiti Putra Malaysia in
fulfilment of the requirement for the Master of Science

**RADIATION AND TEMPERATURE EFFECTS ON ELECTRICAL
PROPERTIES OF POLY (VINYL ALCOHOL) - SODIUM SALICYLATE
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NOORHANIM BINTI AHAD

October 2011

Chairman : Professor Elias Bin Saion, PhD

Faculty : Science

The solid composite polymer electrolytes (SCPEs) are materials that have attracted great attention for their application in the development of solid-state ionic devices. The physical and chemical properties of SCPEs can be modified by treatment with ionizing radiation. One major problem relating to these solid electrolytes is that they have tendency to crystallize at room temperature resulting in a reduction of the ionic conductivity. In this study, radiation processing technique was chosen to increase the ionic conductivity at room temperature. The SCPEs consists of poly (vinyl alcohol) (PVA) and sodium salicylate (SS) as ionic blend were prepared by solvent-casting technique at different SS concentrations (0, 10, 20, 30, 40, 50 wt %) and the film samples were irradiated with gamma radiation at different doses (0, 10, 20, 40 and 50 kGy) at room conditions. The irradiated and unirradiated film samples of different compositions were each placed between two parallel-plate metal electrode and the electrical conductivity and dielectric properties were measured using an impedance analyzer at frequencies ranging from 20 Hz to 1 MHz. The unirradiated samples were also treated thermally and the conductivity and dielectric properties were

measured at different temperatures of 303 to 353 K. The X-Ray diffraction (XRD) analyses were performed to characterize the change of molecular structure of the SCPEs with radiation dose and compositions of the ionic blend. For the unirradiated samples, the complexation and interaction in SCPEs were measured by using Fourier Transform Infrared (FTIR) Spectroscopy and the thermal stability of the SCPEs was investigated by thermogravimetric analysis using a Perkin Elmer TGA Instrument.

The results show that the conductivity and dielectric properties of PVA-SS SCPEs depend on composition of the ionic blends, temperature and radiation dose. The conductivity and dielectric value increase with increasing SS composition due to increase of the number of charge carriers, mainly ions introduced in the blends. The sample of 50 wt. % SS irradiated with dose of 50 kGy exhibits the highest conductivity at room temperature is $\sim 10^{-7}$ to 10^{-6} S/cm. The dc conductivity follows the equation $\sigma_{dc} = \sigma_0 \exp(D/D_0)$ which is characterized by the dose sensitivity, D_0 . It was found that the lowest dose sensitivity is the characteristic of PVA-SS polymer electrolyte of the highest conductivity (50 wt.%, $D_0 = 0.017$). The dc conductivity is associated with sodium and hydrogen ions and free ions that mainly induced from γ -ray interaction. The temperature effects on the conductivity of SCPEs follow Arrhenius equation, $\sigma_{dc} = \sigma_0 \exp\left(\frac{E_A}{kT}\right)$ which characterized by the activation energy, E_A and for 50 wt. % SS sample, the highest conductivity is at 353 K ($\sim 10^{-7}$ to 10^{-6} S/cm). The highest activation energy is the characteristic of pure PVA polymer ($E_A = 0.729$ eV). The effect of temperature on conductivity is due to increase of ionic mobility of charge carriers by a factor proportional to kT . From the XRD analysis, the results show that the degree of crystallinity of the PVA decreases with the

increase of SS composition. As the compositions increase to 30, 40 and 50 wt.% SS, the XRD patterns become crystalline because several peaks have been emerged but the intensity of these peak turns decreases when the dose increases. From the results, we have demonstrated that the radiation can be used to increase the conductivity of SCPEs for the development of solid-state ionic devices.



Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Master Sains

KESAN SINARAN DAN SUHU KE ATAS SIFAT ELEKTRIK POLI (VINIL ALKOHOL) – NATRIUM SALISILAT ELEKTROLIT POLIMER

Oleh

NOORHANIM BINTI AHAD

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Komposit pepejal elektrolit polimer (SCPEs) merupakan bahan menarik yang diberi keutamaan kerana mempunyai aplikasi dalam memajukan peranti ionik keadaan pepejal. Ciri-ciri fizik dan kimia SCPEs boleh diubah suai dengan menggunakan rawatan sinaran mengion. Satu masalah besar yang berkaitan dengan SCPEs ini adalah SCPEs cenderung untuk menghablur pada suhu bilik menyebabkan penurunan kekonduksian ion. Dalam kajian ini, teknik pemprosesan menggunakan sinaran telah dipilih untuk meningkatkan kekonduksian ion pada suhu bilik. SCPEs mengandungi poli (vinil alkohol) (PVA) dan natrium salisilat (SS) sebagai pencampur ion telah disediakan dengan menggunakan kaedah acuan-pelarut pada komposisi SS yang berlainan (0, 10, 20, 30, 40, 50 wt %), dan filem-filem ini disinarkan dengan sinar gama pada dos yang berlainan (0, 10, 20, 40 and 50 kGy) pada suhu bilik. Sampel filem yang telah dan belum dirawat dengan sinaran diletakkan di antara dua elektrod logam plat selari dan ciri-ciri kekonduksian elektrik dan dielektrik diukur dengan menggunakan kaedah analisis impedans pada frekuensi daripada 20 Hz kepada 1

MHz. Sampel yang tidak dirawat dengan sinaran juga dipanaskan pada suhu 303 K hingga 353 K dan sifat kekonduksian dan dielektrik diukur. Analisis XRD juga dilakukan untuk percirian perubahan struktur SCPEs terhadap dos dan komposisi campur ion. Untuk sampel yang tidak dirawat dengan sinaran, pengkompleksan dan tindak balas di dalam SCPEs diukur dengan menggunakan spektroskopi FTIR dan kestabilan haba SCPEs disiasat dengan menggunakan analisis termogravimetri dari sebuah alat Perkin Elmer TGA.

Hasil kajian menunjukkan bahawa sifat kekonduksian elektrik dan dielektrik bagi PVA-SS SCPEs bergantung kepada komposisi campur ion, suhu dan dos sinaran. Nilai kekonduksian dan dielektrik meningkat dengan peningkatan komposisi SS disebabkan oleh penambahan bilangan pembawa cas, terutamanya ion yang terhasil di dalam campuran. Sampel 50 wt.% SS yang disinarkan dengan dos 50 kGy mempamerkan kekonduksian ion tertinggi pada suhu bilik adalah $\sim 10^{-7}$ to 10^{-6} S/cm. Kekonduksian dc mematuhi persamaan $\sigma_{dc} = \sigma_0 \exp(D/D_0)$ yang dicirikan oleh kepekaan dos, D_0 . Didapati bahawa kepekaan dos terendah dicirikan pada PVA-SS elektrolit polimer yang mempunyai kekonduksian tertinggi (50 wt.%, $D_0=0.017$). Kekonduksian dc dikandungi oleh ion natrium dan hidrogen dan ion bebas yang terutamanya terdorong daripada tindak balas sinar gama. Kesan suhu ke atas kekonduksian SCPEs mematuhi persamaan Arrhenius, $\sigma_{dc} = \sigma_0 \exp\left(\frac{E_A}{kT}\right)$ yang dicirikan oleh tenaga pengaktifan, E_A dan untuk sampel 50 wt.% SS, kekonduksian tertinggi adalah pada 353 K ($\sim 10^{-7}$ to 10^{-6} S/cm). Tenaga pengaktifan tertinggi dicirikan pada polimer PVA ($E_A = 0.729$ eV). Kesan suhu ke atas kekonduksian adalah disebabkan oleh peningkatan kelincahan pembawa cas dengan faktor berkadar langsung kepada kT . Daripada analisa XRD, hasil kajian menunjukkan bahawa

darjah penghabluran bagi PVA menurun dengan peningkatan komposisi SS. Apabila komposisi SS bertambah kepada 30, 40 dan 50 wt.%, bentuk XRD bertukar menjadi hablur kerana kemunculan beberapa puncak tetapi keamatan puncak tersebut menurun apabila dos meningkat. Daripada hasil kajian, kami telah menunjukkan bahawa pemprosesan kaedah sinaran boleh digunakan untuk meningkatkan kekonduksian elektrik SCPEs untuk pembangunan peranti ionik keadaan pepejal.



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I certify that an Examination Committee has met on 28 October 2011 to conduct the final examination of Noorhanim Binti Ahad on her Master of Science thesis entitled “Radiation and Temperature Effects on Electrical Properties of Poly (Vinyl Alcohol)-Sodium Salicylate Polymer Electrolytes” in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The Committee recommends that the students be awarded the relevant degree. Members of the Examination Committee are as follows:

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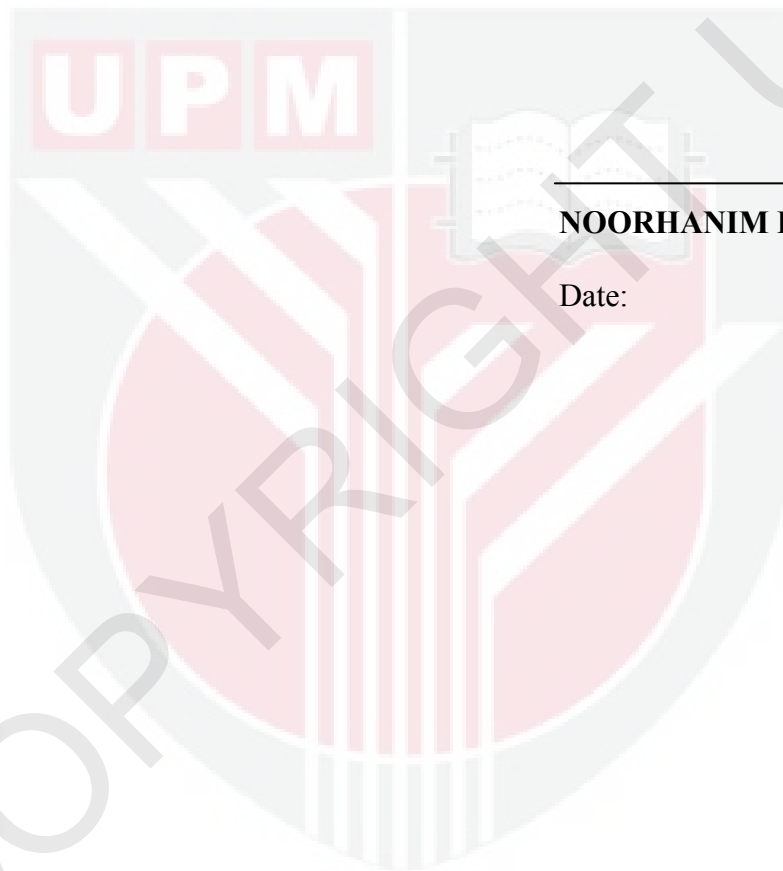
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DECLARATION

I declare that the thesis is my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously, and is not concurrently, submitted for any other degree at Universiti Putra Malaysia or at any other institution.



NOORHANIM BINTI AHAD

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