

**SYNTHESIS AND CHARACTERIZATION OF PVA -  
METAL COMPLEX COMPOSITES FOR  
ELECTROCHEMICAL DOUBLE LAYER CAPACITOR  
(EDLC) DEVICES**

**BY**

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## ABSTRACT

In the current work, the preparation of the polymer electrolyte (PE) systems consists of PVA polymer matrix, ammonium thiocyanate ( $\text{NH}_4\text{SCN}$ ) as ionic source, Cu(II)-, Ce(III)-, and Cd(II)-complex as metal-complexes and glycerol as plasticizer are performed by solution cast technique. The polymer electrolytes are used for application in electrochemical double-layer capacitor (EDLC) device. Due to poor optical, electrical, and electrochemical properties of PVA, it is integrated with the metal-complex and glycerol to increase amorphous phase, electrical conductivity, and decrease optical band gap for the electrolyte to be used for fabricating EDLC device with very high performance. X-ray diffraction (XRD) has shown that the highest conducting plasticized electrolyte with Cu(II)-complexes possess the lowest degree of crystallinity. The possible interactions within the PE elements are verified using Fourier transform infrared (FTIR) spectroscopy. The FESEM images reveal that the surface morphology of the samples showed a uniform smooth surface at high glycerol concentration. This is in good agreement with the XRD and FTIR results. The highest conducting plasticized electrolyte without metal-complexes is found to be  $1.82 \times 10^{-5} \text{ S cm}^{-1}$  while the conductivity increased up to  $2.25 \times 10^{-3} \text{ S cm}^{-1}$  by the addition of Cu(II)-complex into the PE. The conductivity is found to be affected by the ionic mobility ( $\mu$ ), diffusion coefficient ( $D$ ), and number density ( $n$ ) of ions. From UV-Vis spectroscopy study, the optical parameters (absorption edge, refractive index ( $N$ ), dielectric constant ( $\epsilon_r$ ), dielectric loss ( $\epsilon_i$ ), and optical band gap energy ( $E_g$ )) of pure PVA and composite films are measured. Examination of the  $\epsilon_i$  optical parameter is carried out to measure the  $E_g$ , while types of electronic transition in the films are determined based on the Tauc's method. From transference number measurement (TNM), ions are considered as the dominant charge carrier and the transference number for ions and electrons for the highest conducting electrolyte (PGNC-4) are 0.971 and 0.029, respectively. PGNC-4 is electrochemically stable up to 2.15 V. Galvanostatic charge-discharge (GCD) measurement of the EDLC has been supported with cyclic voltammetry (CV) analysis. CV curves are determined by inserting the plasticized electrolytes between two activated carbon (AC) electrodes, and it shows a nearly rectangular shape at low scan rates. The specific capacitance and energy density of the EDLC for the highest conducting plasticized electrolyte with Cu(II)-complexes (PGNC-4) are nearly constant within 1000 cycles at a current density of  $0.5 \text{ mA/cm}^2$  with average of 155.322 F/g and 17.473 Wh/Kg, respectively. The energy density of the EDLC in the current work is in the range of battery energy density. The EDLC performance was found to be stable over 1000 cycles for the PGNC-4 system. The low value of equivalent series resistance shows that the EDLC has good electrolyte-electrode contact. The EDLC for the PGNC-4 system exhibited the initial high power density of  $4.960 \times 10^3 \text{ W/Kg}$ .

## خلاصة البحث

في الدراسة الحالية، تحضير انظمة الكتروليت البوليمري (PE) يتكون من مصفوفة بوليمر (PVA)، ثيوسيانات الامونيوم ( $\text{NH}_4\text{SCN}$ ) كمصدر الأيون و معقد  $\text{Cu(II)}$ ,  $\text{Ce(III)}$ ,  $\text{Cd(II)}$  كالمعدن المعدنية و الجلسرين كملدن او المصق و ذلك عن طريق تقنية الصب المحلول. يستخدم البوليمرات الاكترولية في التطبيق و فبركه متمسعه الكتروكيميائية ثنائية الطبقة (EDLC). بسبب ضعف الخواص البصرية و الكهربائية و الكهروكيميائية لـ PVA تم دمجها و تكملتها مع المعقد المعدن و الكريسول لزياده الطور الامورفي اللابلورى و كذلك التوصيل الكهربائي و تقليل افجوه الطاقه البصرية للالكتروليت لكي تستخدم فبركه EDLC ذات كفاءة العاليه.

تبين من الحيوذ الاشعة السينية (XRD) ان الكتروليت المعدن ذات التوصيلية العاليه مع معقدات  $\text{Cu(II)}$  تمتلك أقل درجة من التبلور. تم استخدام التحليل الطيفي لتمويل الفوريرى للاشعه تحت الحمراء (FTIR) و ذلك للتحقق من التفاعلات الممكنة للعناصر داخل الالكتروليت البوليمري. من خلال دراسة اشكال (FESEM) تبين له عند تراكيزه العاليه لكليسرين تتميز سطوح الاغشية المستحضرة بأفها وساء و منظم و متوافقه مع نتائج FTIR, XRD. تم الحصول على توصيلة الكتروليت الوصل الملمدين بدون معقدات المعدنيه بمقدار  $1.82 \times 10^{-5} \text{ Scm}^{-1}$  و تزداد هذه القيمة الى  $2.5 \times 10^{-3} \text{ Scm}^{-1}$  عند اضافة معقد  $\text{Cu(II)}$  الى اكرتوليت البوليمري (PE). و من خلال الدراسة، تبين ان التوصيلية الكهربائيه تتأثر بحركية الايونيه ( $\mu$ ) و معامل الانتشار (D) و تركيز الايونات (n). من دراسة الطيف الاشعة المرئية و فوق البنفسجية (UV-Vis)، تم قياس الباراميترات (حافه الامتصاص، معامل الانكسار (N) و ثابت عزل الكهربيات ( $\epsilon_r$ ) و فقد عزل الكهربائي ( $\epsilon_i$ ) و كذلك فجوة الطاقه البصرية ( $E_g$ ) للاغشيه البوليمريه PVA و الحركيه. من خلال فحص الباراميتر الضوئي  $\epsilon_i$  تم قياس فجوه طاقه البصريه ( $E_g$ )، بينما حددت الانتقالات الالكترونية بأنواعها للافلام تحت الدراسه اعتمادا على طريقة (Tauc) تاوك. نتائج قياسي الرقم التحويلى TNM، تؤكد على أن غالبية حاملات الشحنات هي الايونات، و نتائج TNM للايونات و الاكترونات للالكتروليتات ذات توصليه عاليه لـ PGNC-4 تكون 0.029 و 0.971 على التوالي. من خلال الدراسة، تبين أن PGNC-4 مستقر كهربائيا حتى 2.15 فولت، و تم دعم قياسات تفريغ الشحن الكلفاني (GCD) لـ EDLC من خلال تحاليل فولتميري الدورى (CV)، حيث تم تحديد منحنيات (CV) و ذلك من ادراج الكتروليتات الملمدنه (plasticized) تحت الدراسه بين قطبي كاربون المنشط (AC) حيث خمرت شكلا تقريبا للمستطيل عند معدلات المسح المنخفض. و تظهر من خلال النتائج ان السعة النوعية و كثافه الطاقه للالكتروليتات الملمدنيه مع معقدات PGNC-4 Cu (II) يمتلك الموصلية عاليه ثابتة تقريبا خلال 1000 دورة عند كثافه التيار بمعدل  $0.5 \text{ mA/cm}^2$   $17.474 \text{ Wh/kg}$  على التولى في الدراسه الحاليه، الكثافه الطاقيه EDLC عند نطاق كثافه الطاقه البطارية، و اداء EDLC مستقر على مدى 1000 دورة لنظام PGNC-4، و قيمة الواطيه للمقاومه المكانية المتواليه دلالة على توصيلية جيدة بين الالكتروليتات و القطب. اظهر EDLC لنظام PGNC-4 كثافة لقدرة العاليه الأولية لـ  $4.960 \times 10^3 \text{ W/kg}$ .

## **APPROVAL PAGE**

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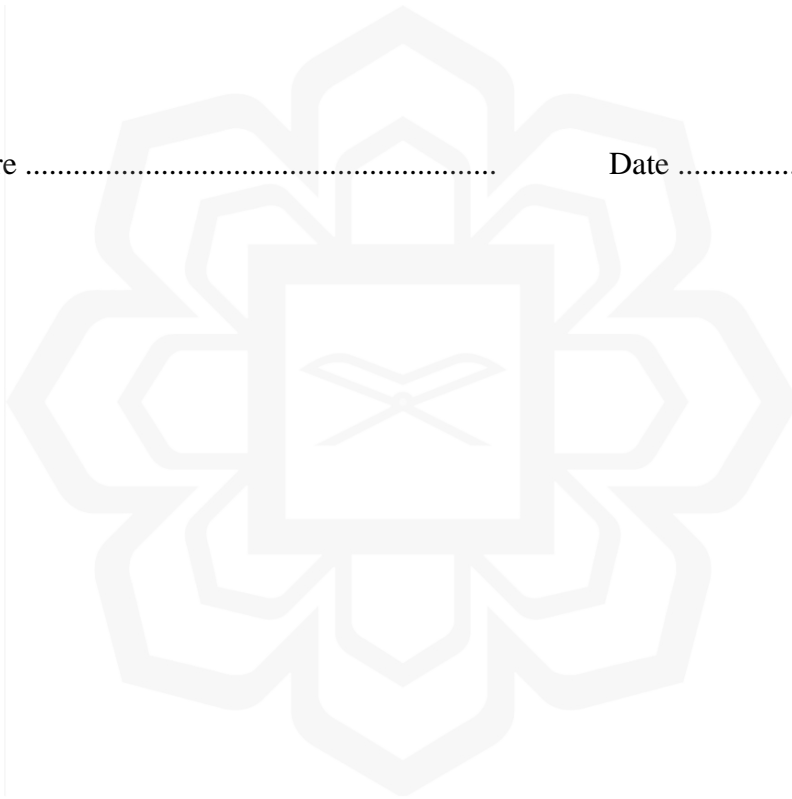
## DECLARATION

I hereby declare that this thesis is the result of my own investigations, except where otherwise stated. I also declare that it has not been previously or concurrently submitted as a whole for any other degrees at IIUM or other institutions.

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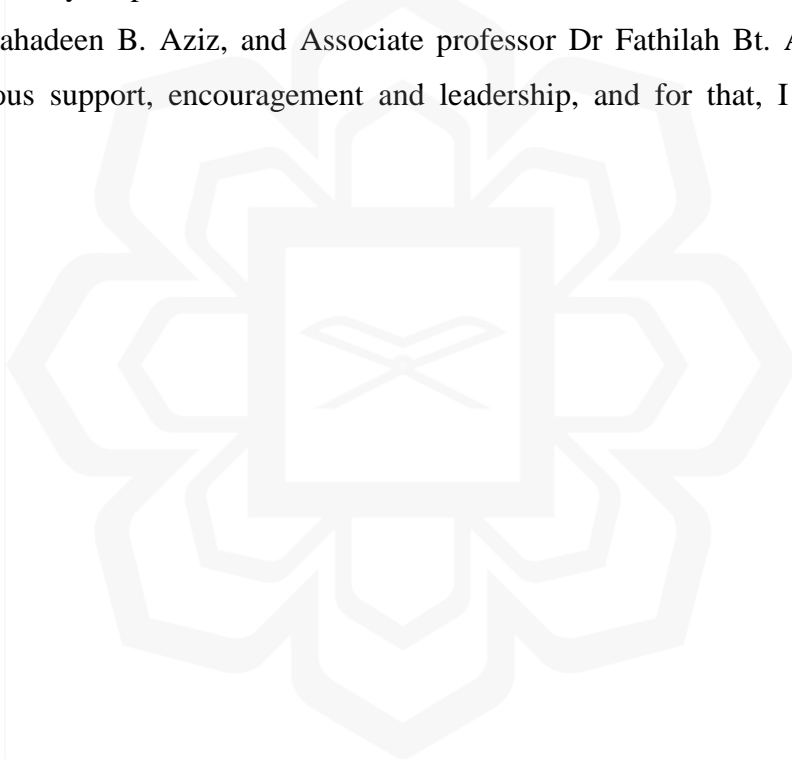
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## LIST OF ABBREVIATIONS

Al <sub>2</sub> O <sub>3</sub>	Aluminium oxide
Al <sub>2</sub> SiO <sub>5</sub>	Aluminium silicate
BmImBr	1-butyl-3-methylimidazolium bromide
BmImCl	1-butyl-3-methylimidazolium chloride
CB	Conduction band
CH <sub>3</sub> COOK	Potassium acetate
NH <sub>4</sub> I	Ammonium iodide
NH <sub>4</sub> NO <sub>3</sub>	Ammonium nitrate
CPE	Constant phase element
CV	Cyclic voltammetry
EDLC	Electrochemical double-layer capacitor
FESEM	Field emission scanning electron microscopy
ESR	Equivalent series resistance
FTIR	Fourier transform infrared spectroscopy
CuI	copper iodide
CuS	Copper monosulfide
DBG	Direct band gap
DMFC	Direct-methanol fuel cell
DOP	Diocetyl phthalate
H <sub>2</sub> SO <sub>4</sub>	Sulfuric acid
H <sub>3</sub> PO <sub>4</sub>	Orthophosphoric acid
KOH	Potassium hydroxide
LED	Light-emitting diode
H <sup>+</sup>	Hydrogen ion
Li <sup>+</sup>	Lithium ion
LiClO <sub>4</sub>	Lithium perchlorate
LSV	Linear sweep voltammetry
MC	Methylcellulose
NH <sup>4+</sup>	Ammonium ion
NH <sub>4</sub> CH <sub>3</sub> CO <sub>2</sub>	Ammonium acetate