SYNTHESIS AND CHARACTERIZATION OF LASER-IRRADIATED POLYANILINE/RICE HUSK (SILICA) NANOCOMPOSITES FOR ELECTRICAL CONDUCTIVITY APPLICATIONS

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DEDICATION

To my loving and caring mother Qismah Shihab, my father Oudah Mezan, my beloved wife Janan Obaid, and my sons Mohammad Salim, Zahraa Salim and Moqtadah Salim.

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ABSTRACT

Polymer nanocomposites contain inorganic fillers that have metallic particles dispersed in polymer matrices. These are very appealing for optical and electrical conductivity applications. Polyaniline/ silica (rice husk ash CCl (SiO_2)), nanocomposites (PANI/RHACCl (SiO_2)) nanocomposites can be manipulated through different methods of fabrication for desired purposes. An innovative method for nanocomposite materials fabrication has been developed using sol-gel that has been applied to polyaniline/RHACCl (Silica) nanocomposites. Laser irradiation is also used as a way to manipulate the structure of the nanocomposite to investigate the effect of enhancing new optical and electrical properties. The laser irradiation power of value 13.17 w/cm^2 leads to the formation of graphite oxide and carbon percentage increment, which is based on laser irradiation time and distribution percentage of RHACCl nanoparticles. The sodium silicate was obtained using hydrochloric acid (HCl) wash to produce silica. Polyaniline was synthesized by chemical oxidative polymerization, then mixed with RHACCl (SiO_2) in the presence of toluene and trimethylamine to form. The samples were analyzed by the structural, chemical, morphology, optical-electrical conductivity, and thermal properties behaviors. The amorphous nature of silica was confirmed using XRD analysis. The sample size was obtained from (6-9 nm) by FESEM morphology studies. EDX atomic weight was measured at 57.10% of carbon, 26. 78% of oxygen, 3.49% of silicon, 10.09% of nitrogen, 0.68% sulfur, and 1.87% of chlorine. UV-Vis absorption wavelength was determined at 382 nm. The best energy band gap of PANI was 1.13 eV and the best laser irradiation time was (40) min. Direct current (DC) electrical conductivity was calculated for the best ratio conductivity values were found to be at PANI / RHACCl (SiO_2) NCs after laser irradiation (1: 0.5 = $1.10x10^{-1}$ S cm^{-1}, 2: 0.5 = $1.36x10^{-1}$ S cm^{-1} and 3: 0.5 = $2.08x10^{-1}$ S cm^{-1}) in the time 40 min.

ABSTRAK

Polimer Nanokomposit mengandungi bahan pengisi tidak organik yang mempunyai zarah logam terserak di dalam matriks polimer. Ianya usaha yang sangat menarik terutama dalam aplikasi kekonduksian optik dan elektrik. Polianilin/silika (sekam padi terproses CCl (SiO_2)) nanokomposit (PANI/RHACCl (SiO_2) nanokomposit) boleh dimanipulasi melalui kaedah fabrikasi yang berbeza untuk tujuan yang diingini. Kaedah inovatif fabrikasi bahan nanokomposit melalui teknik sol-gel telah digunakan dalam kajian aplikasi PANI/RHACCl (SiO_2) nanokomposit. Penyinaran laser juga digunakan sebagai kaedah memanipulasi struktur nanokomposit dalam menyiasat kesan peningkatkan sifat optik dan elektrik baharu. Kuasa penyinaran laser dengan nilai 13.17 w/cm^2 membawa kepada pembentukan grafit oksida dan kenaikan peratusan karbon, yang berdasarkan masa penyinaran laser dan peratusan pengedaran nanopartikel RHACCl. Natrium silikat diperoleh menggunakan pencucian asid hidroklorik (HCl) untuk menghasilkan silika. Polianilin telah disintesis oleh pempolimeran oksidatif kimia yang mengandungi kumpulan berfungsi aktif dalam molekul, kemudian dicampur dengan RHACCl (SiO_2) dengan kehadiran toluena dan trimetilamin untuk membentuk. Sampel oleh struktur, kimia, morfologi, kekonduksian optik-elektrik, dan kelakuan sifat terma. Sifat amorf silika telah disahkan menggunakan analisis XRD. Saiz sampel diperoleh daripada (6-9 nm) oleh kajian morfologi FESEM. Berat atom EDX diukur pada 57.10% karbon, 26. 78% oksigen, 3.49% silikon, 10.09% nitrogen, 0.68% sulfur, dan 1.87% klorin. UV-Vis ditentukan pada 382 nm. jurang jalur tenaga terbaik PANI ialah 1.13 eV dan masa penyinaran laser terbaik ialah (40) min. Kekonduksian elektrik arus terus (DC) dikira pada nilai kekonduksian nisbah terbaik adalah pada penyinaran laser dengan nisbah (PANI / RHACCl (SiO2) NCs (1: 0.5 = 1.10x10^{-1} S cm^{-1}, 2: 0.5 = 1.36x10 $^{-1}$ S cm^{-1} dan 3: 0.5 = 2.08x10^{-1} S cm^{-1}) adalah pada masa sinaran 40 min.

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LIST OF SYMBOLS AND ABBREVIATIONS

%	-	Percentage
π	-	Pi
Ω	-	Ohms
=	-	Equal to
≤	-	Less than equal to
°	-	Degree
°C	-	Degree celsius
3D	-	3 dimensional
Å	-	Angstrom
β	-	Beta
θ	-	Angle
σ	-	Conductivity
A	-	Area
(a-C)	-	amorphous carbon
at%	-	Atomic percentage
C	-	Capacitance of dielectric constant
cm	-	The capacitance
CPE	-	Constant phase element
Cu	-	Copper
D	-	Diameter
DC	-	Direct current
EDX	-	Energy Dispersive X-ray
FESEM	-	Field emission scanning electron microscope
FTIR	-	Fourier transforms infrared
f	-	Frequency
FWHM	-	Full width at half maximum
g	-	Graem
H	-	Hour

Hz	-	Hertz
IS	-	Impedance spectroscopy
IT	-	Information Technology
k	-	Rate constant (min^{-1})
K	-	Kelvin
Kg	-	Kilogram
L	-	Liter
M	-	Mass of tested sample (g)
m	-	Meter
mg	-	Milgram
MHz	-	Megahertz
mL	-	Mililiter
NCs	-	Nanocomposite
NPs	-	Nanoparticles
°C /min	-	Degree Celsius per minute
PANI	-	Polyaniline
PVA	-	Polyvinyl Alcohol
RH	-	Rice Husk
RHA	-	Rice Husk Ash
RHACCl	-	Rice Husk Ash(Silica)
RT	-	Room temperature
s	-	Second
S	-	Siemens
s.g	-	Space group
SC	-	Scandium
Si	-	Silicon
SiO_2	-	Silica
T	-	Thickness
XRD	-	X-Ray diffraction

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CHAPTER 1

INTRODUCTION

1.1 Background of the study

A nanomaterial is the cornerstones of nanoscience, nanotechnology, and nanoscale that have contributed to a vast and multidisciplinary research area that is increasing dramatically worldwide in the last few years (Liu *et al.*, 2017). The future for advanced nanomaterials is faced by four fundamental problems in nanotechnology, given in (Manikandan *el at.,* 2020). The first is the precise fabrication of nanomaterials (Wang *et al.,* 2020), the second one is the precise positioning of materials and repeated at ease for various manipulation strategies (James *et al.*, 2019). Thirdly, nanomaterials are interconnecting for functional devices fabrication (Karim *et al.,* 2019), and finally is the mass production of nano-based devices for commercialization (Selvaraju, Sundaresan, & Dharmar, 2019). Due to its benefits of being easily synthesized, being environmentally stable in air and water, and possessing high conductivity, polyaniline is an interesting conducting polymer (Zhao *et al.,* 2019). However, polyaniline (PANI) has its own significance in the materials for electrochemical capacitors, with a higher precise capacity, a higher energy capacitance, and a faster energy density than typical capacitors (Roy *et al.,* 2018; Mazzeu *et al.,* 2017).

It comprises one silicon atom and 2 atoms of oxygen that yields SiO_2 that contributes to today's technology revolution that is applied to computer plastics and provides the raw material for silicon chips (Roosz *et al.,* 2017b). The Silica is widely used and exists uncommonly in amorphous state and often in crystalline state (Roosz *et al.,* 2017a).

It is observed that 95% of the rice husk is made up of SiO_2, obtained at 800 °C (Liu *et al.*, 2020) known as silica RHACCl (Adam *et al.*, 2010). Several metal oxide nanoparticles (NPs) are attached to the conductive polymers to create nanocomposites (NCs) applied in the manufacture from PANI/ RHACCl (silica) nanocomposites in the presence of triethylamine (Et_3N) that has been synthesized by Et_3N. It is a new method for synthesizing silica (RHACCl) with polyaniline to form a nanocomposite used sol-gel.

The term polymer is widely used today to indicate the sense of "plastic" or "resin" (Roosz et al., 2017a). A polymer is a chemical compound where long repeating chains, molecules are bound together. Such polymers have specific properties and can be modified according to their intended purpose (Roosz *et al.*, 2017b). The backbone description of the polymer chain is categorized into two (inorganic and organic) polymers. The polymer whose backbone chain is composed of carbon atoms is referred to as an organic polymer, typically hydrogen, hydrogen, oxygen, oxygen, nitrogen, nitrogen, etc.) (Liu *et al.*, 2013). On the other hand, most plastic polymers are organic, with no carbon atom in the chain backbone. However, glass is referred to as inorganic polymers, while silicone rubber is an exception (Kim & Kang, 2013). It is also possible to classify organic polymers as conductors polyaniline (PANI) (Ruiz-Pérez *et al.*, 2020), polypyrrole (PPy), (Abolghasemi *et al.*, 2018), polythiophene, polyimide (Chen *et al.*, 2017) and non-conductive, such as polystyrene (Balint *et al.*, 2014).

One of the reasons for this study is the ease of linking polyaniline to the modified silica obtained from rice husks due to the ease of removing the chlorine atom from the modified silica and linking the polyaniline in place to form a nanocomposite. This is a new method for preparing nanocomposites. In addition to the electrical properties.(Chen *et al.*, 2017).

1.2 Polyaniline

Due to its unique characteristics like low cost, lightweight, environmental, height stability, dielectric properties, workability, resilience, thermal stability, oxidative stability, and sensitivity, polyaniline is recognized as being the most attractive conducting polyaniline (Chen *et al.*, 2017). Ease from synthesis, good electrical conductivity, including interesting redox properties is connected with the chain

nitrogen (Song & Choi, 2013). The Polyaniline (PANI) uniqueness defines its great impact to be used in technology such as material involved in electromagnetic shielding, anti-corrosion protection, sensor devices, anti-fouling protection, including antistatic materials, among others (Caldas *et al.*, 2017). The reversible tunable redox features define the electrical conductivity control with a great range over protonation and charge-transfer doping that are distinctive features of PANI compared to other conjugated polymers (Soares, 2018).

1.3 Silica Nanoparticles (SiO_2NPs)

Silica is a cluster of minerals made of silicon and oxygen i.e. two copious elements in the earth layer. Silica formula of SiO_2 that is pragmatic on earth since quartz which is greater than 10% by mass from the earth layer. The rapidly evolving nanotechnology fields and the versatile processes used to generate, manipulate and utilize nanomaterial, which is recently being emphasized leading to a promising era in bio-analytical, biotechnological, and biomedical applications (Ionita *et al.*, 2008). A round of work activity has focused on the preparation and functionalization of the nanoparticles (NPs), owing to their unique, chemical (catalytic), physical (structural, magnetic, optical and electronic), including electrochemical characteristics (Lee *et al.*, 2010).

Rice husk (RH) is fibre based on cellulose, ideal for recycling. Rice is grown in over 80 nations. The world's annual rice production ranges from four hundred to fifty-five hundred million metric tons, more than 10 % than rice husk because of its high silica content. Using this free raw material makes economic sense (Adam *et al.*, 2012). Rice husk consists of 38% cellulose, 20% ash, 18% pentose, 22% lignin, 2% water, and other organic ingredients. The burning of rice husk in nature has caused environmental pollution. Consequently, serious steps have been taken to burn the peels at reasonable heat and controlled pressure and these wastes are used to produce scientific materials that have commercial use within the recycling of materials (Shen, 2017). It is worth to note that rice husk charred produced the white ash, that porous silica by high specific surface area and this can be accumulated. This RHA comprises of over 95% silica (Wang *et al.*, 2019).

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APPENDIX C:

LIST OF PUBLICATIONS

i. Mezan, S. O., Jabbar, A. H., Hamzah, M. Q., Tuama, A. N., Hasan, N. N., & Agam, M. A. (2018). Synthesis and Characterization of Zinc Sulphide (ZnS) Thin Film Nanoparticle for Optical Properties. *Journal of Global Pharma Technology, 10(07), 369-373*.

ii. Mezan, S. O., Hello, K. M., Jabbar, A. H., Hamzah, M. Q., Tuama, A., Roslan, M. S., & Agam, M. A. (2020). A review on synthesis of conducting with polyaniline rice husk ash silica nanocomposites and application. International Journal of Psychosocial Rehabilitation, 24(03). *International Journal of Psychosocial Rehabilitation, 24(03).*

iii. Mezan, S. O., Hello, K. M., Jabbar, A. H., Hamzah, M. Q., Tuama, A. N., Roslan, M. S., & Agam, M. A. (2020). Synthesis and Characterization of Enhanced Polyaniline Nanoparticles by Oxidizing Polymerization. *Solid State Technology*, *63*(1), 256-266.

iv. Mezan, S. O., Jabbar, A. H., Hamzah, M. Q., Tuama, A. N., Hasan, N. N., Roslan, M. S., & Agam, M. A. (2019, August). Synthesis, characterization, and properties of polystyrene/SiO_2 nanocomposite via sol-gel process. *In AIP Conference Proceedings (Vol. 2151, No. 1,p. 020034).*

v. Mezan, S. O., Al Absi, S. M., Jabbar, A. H., Roslan, M. S., & Agam, M. A. (2021). Synthesis and characterization of enhanced silica nanoparticle (SiO_2) prepared from rice husk ash immobilized of 3-(chloropropyl) triethoxysilanea. *Materials Today*: Proceedings.

APPENDIX D

LIST OF EQUIPMENT AND CHEMICAL USED

i. aniline monomer C_6H_7N.

ii. Ammonium peroxydisulfate (APS) $(NH_4)_2S_2O_8$.

iii. Saturated hydrochloric acid HCl.

iv. 3-(chloropropyl) triethoxysilane $C_9H_{23}NO_3Si$

v. Sodium hydroxide NaOH

vi. Rice Husk Ash RHA.

vii. Nitric acid HNO_3

viii. Aston CH_3COCH_3

ix. Deionized water (DI)

x. Polyaniline $([C_6H_4NH]_2[C_6H_4N]_2)n$

xi. 50- 60 ^oC Conventional Oven.

xii. Air dryer cupboard.

xiii. Toluene $C7H_8$ or $C_6H_5CH_3$

xiv. Ammonium hydroxide NH_4OH.

xv. Diode Laser Generator Power 1.265 W, 450 nm wavelength to expose nanocomposite of PANI and PANI/RHACCl (SiO_2) nanoparticles that have already been prepared.

xvi. Commonly used analytical glass devices in a laboratory experimental tests include 10 mL and 25 mL volume measurement glasses; 5mL, 10 mL, 50 Ml, and 700 mL fixed volume volumetric flasks; beaker glasses; 25 mL volume microbursts; and volumetric pipettes.

xvii. Micro 200 Hettich Zentrifugen D-78532 Mini Ultracentrifuge Computer fabricated in Germany.

xviii. Vortex homogenizer machine, Uzusio VTX-3000L, Made in Japan.

xix. Small Erlenmeyer flask

xx. Large dish of ice

xxi. Stirrer hotplate

Xxii. 1" stir bar

APPENDIX E

ANALYTICAL INSTRUMENTS

i. (0.0001)-gram sensitivity analytical balance.

ii. Magnetic Stirrer Hotplate Machine: IKA RCT basic safety control mode, temperature range up to 310 oC, motor rotation ranges up to 1500 rpm.

ii. The furnace at range 1200 ^{o}C.

iii. Driven Bench-Top Ultrasonic Cleaner Micro Process Made by

iv. Seoul, Korea, Hwashin Technology Co. Model: Powersonic 405, Input/output electrical energy: AC 230 volt, 50 Hz 350 watt for samples to agitate/irradiate.

VITA

The author was born in 3 April 1975 and completed his secondary school in Ibin Hian high school for boys, Al Muthanna, Iraq. He then joined the bachelor's degree in 1996 at the University of Qadisiyah - College of Education - Department of Physics. He has worked as a physics teacher in high schools since 2001 and until now.

He has considerable experience in the use and maintenance of computers and got several books of thanks from the Directorate-General for Education Muthanna. He obtains his Master's degree in Physics Department from the Osmania University, Hyderabad, and Telangana, INDIA. He started his PhD in October 2017 as a student at University Tun Hussein Onn Malaysia.