

PREPARATION OF POLYPYRROLE AND POLYPROPYLENE-GRAFTED-  
POLYPYRROLE BY USING ULTRASONIC IRRADIATION

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The Almighty of Allah, The Most Gracious and The Most Merciful

Love special dedicate to...

Special inspiring of my lovely husband, Mohammad Aidil Adam B. Hamidan,  
Special encouraging of my beloved parents, Elias B. Burhan and Zuliah Bt. Dalimi,  
Special amazing daughter and sons, Muhammad Aqil, Maria Masturah and  
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## ABSTRACT

Ultrasonic irradiation has been used to polymerize the nanosized polypyrrole (PPy) at frequency of 20 kHz, reaction temperature of 5 °C and reaction time of 30 minutes. The intensities of ultrasonic irradiation were varied from 20% to 100%. Effects of the intensities variations on the functional group, thermal transitions and degradation behaviours, morphologies and conductivity properties were investigated. Fourier transform infrared spectroscopy (FTIR) showed broad peaks of N-H, C-H and C=C aromatic with increasing the ultrasonic intensity. The glass transition temperature,  $T_g$ , appeared at 107.93 °C for PPy polymerized under 20% intensity and kept increased until 118.07 °C under 100% intensity, as indicated by the differential scanning calorimetry (DSC) graph. Meanwhile, the PPy samples were not fully decomposed even at higher temperature of 900 °C as shown by the thermogravimetric analysis (TGA) plots. The maximum temperature and final residue was increased as the ultrasonic intensity increased from 20% to 100%. Field emission scanning electron microscopy (FESEM) micrographs showed no significant changes in size of PPy particles at various ultrasonic intensities. The conductivities of the samples were also unaffected with increasing ultrasonic intensities. Subsequently, polypropylene-grafted polypyrrole (PP-g-PPy) was prepared by using ultrasonic assisted extrusion. It was to investigate the effects of PPy composition on the functional group, rheological, tensile, morphological, electrical conductivity and thermal properties of PP-g-PPy blends. The sample of PPy polymerized under 100% ultrasonic intensity was chosen to graft with PP. The sample formulation of PP/PPy in weight % were consist of 100/0, 95/5, 90/10, 85/15 and 80/20. 1% of dicumyl peroxide was used as the initiator. Frequency of 20 kHz with temperature of  $170 \pm 5$  °C were applied during the process of PP-g-PPy. The FTIR plots confirmed the appearance of PP-g-PPy peaks by shifting the NH stretching of PPy from  $3395 \text{ cm}^{-1}$  to  $3379 \text{ cm}^{-1}$ . TGA plots showed the thermal stability of PP significantly improved by grafting with PPy, and increased with the increment of PPy content. From DSC graph, the melting temperature, crystallization temperature and degree of crystallinity of the blends were lower than the pure PP, and decreased as the PPy content increased in PP-g-PPy. The dynamic mechanical analysis plots showed the storage modulus, loss modulus and  $\tan \delta$  of PP-g-PPy were higher than the pure PP and increased with PPy content increased, over the whole temperature range. The tensile strength and the elongation at break were decreased while the Young's modulus was increased as the amount of PPy increased. FESEM micrographs showed the tensile fractured surface of 5 wt% PPy content exhibit the most clear of fibrous structure, and getting lower and flat fracture surfaces were formed as the increment of PPy content. The conductivities of samples increased as the compositions of PPy were increased. All the blends and pure PP demonstrated pseudoplastic flow and shear thinning behaviour where the viscosity decreased with the increasing shear rate, as shown in rheology plots. The viscosity increased when PPy content was introduced into PP matrix, and increased with the PPy content. Based on the study conducted, the intensity of ultrasonic irradiation can effects the functional group, thermal transitions and degradation behaviours of prepared PPy and also PPy composition can influence the properties of PP-g-PPy blends.

## ABSTRAK

Radiasi ultrasonik telah digunakan untuk pempolimeran polipirol (PPy) bersaiz nano pada frekuensi 20 kHz, suhu tindak balas pada 5 °C dan masa tindak balas selama 30 minit. Keamatan radiasi ultrasonik berbeza dari 20% hingga 100%. Kesan perbezaan keamatan terhadap kumpulan berfungsi, peralihan haba, kelakuan perosotan, ciri-ciri morfologi dan kekonduksian telah disiasat. Spektroskopi inframerah transformasi Fourier (FTIR) menunjukkan puncak-puncak lebar N-H, C-H dan aromatik C=C dengan peningkatan keamatan ultrasonik. Suhu peralihan kaca,  $T_g$ , muncul pada 107.93 °C untuk pempolimeran PPy dibawah keamatan 20% dan terus meningkat sehingga 118.07 °C dibawah keamatan 100%, seperti yang ditunjukkan oleh graf kalorimetri pengimbasan pembezaan (DSC). Sementara itu, sampel-sampel PPy tidak terurai sepenuhnya walaupun pada suhu yang lebih tinggi iaitu 900 °C seperti yang ditunjukkan oleh plot pengujian analisis termogravimetri (TGA). Suhu maksimum dan baki akhir adalah meningkat dengan peningkatan keamatan ultrasonik dari 20% - 100%. Mikrograf mikroskopi elektron imbasan pancaran medan (FESEM) menunjukkan tiada perubahan ketara dalam saiz zarah PPy pada pelbagai keamatan ultrasonik. Kekonduksian sampel-sampel juga tidak terjejas dengan kenaikan keamatan ultrasonik. Selepas itu, polipropilena-tercangkuk-polipirol (PP-g-PPy) telah disediakan dengan menggunakan penyemperitan berbantu ultrasonik. Ianya untuk mengkaji kesan komposisi PPy terhadap kumpulan berfungsi, reologi, regangan, morfologi, kekonduksian elektrik dan sifat-sifat terma adunan PP-g-PPy. Sampel pempolimeran PPy dibawah keamatan ultrasonik 100% telah dipilih untuk dicangkuk pada PP. Formulasi sampel PP/PPy dalam peratusan berat adalah terdiri daripada 100/0, 95/5, 90/10, 85/15 dan 80/20. 1% dicumil peroksida telah digunakan sebagai pemula. Frekuensi 20 kHz dengan suhu  $170 \pm 5$  °C telah digunakan semasa proses PP-g-PPy. Plot FTIR mengesahkan kewujudan puncak PP-g-PPy dengan pengalihan NH meregang pada PPy dari  $3395 \text{ cm}^{-1}$  kepada  $3379 \text{ cm}^{-1}$ . Plot TGA menunjukkan kestabilan terma PP bertambah baik dengan ketara apabila tercangkuk dengan PPy, dan bertambah dengan kenaikan kandungan PPy. Dari graf DSC, suhu lebur, suhu penghabluran dan darjah kehabluran pada adunan adalah lebih rendah daripada PP tulen, dan menurun apabila kandungan PPy meningkat didalam PP-g-PPy. Plot penganalisis mekanikal dinamik menunjukkan modulus simpan, modulus kehilangan dan  $\tan \delta$  PP-g-PPy adalah lebih tinggi daripada PP tulen dan bertambah apabila kandungan PPy bertambah, sepanjang julat suhu keseluruhan. Kekuatan tegangan dan pemanjangan pada takat putus telah menurun manakala modulus Young telah meningkat apabila jumlah PPy meningkat. Mikrograf FESEM menunjukkan permukaan patah regangan untuk peratusan berat 5 wt% PPy menunjukkan yang paling jelas struktur berserabut, dan permukaan patah regangan semakin rendah dan rata terbentuk apabila kandungan PPy bertambah. Kekonduksian sampel-sampel bertambah apabila komposisi PPy meningkat. Semua adunan dan PP tulen menunjukkan aliran pseudoplastik dan sifat penipisan ricih dimana kelikatan menurun dengan kenaikan kadar ricih, seperti yang ditunjukkan dalam plot reologi. Kelikatan bertambah apabila kandungan PPy dimasukkan kedalam matriks PP, dan bertambah dengan kandungan PPy. Berdasarkan kajian yang dijalankan, keamatan radiasi ultrasonik boleh memberi kesan pada kumpulan berfungsi, peralihan haba dan kelakuan perosotan terhadap PPy yang telah disediakan dan juga komposisi PPy boleh mempengaruhi sifat-sifat adunan PP-g-PPy.

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**LIST OF SYMBOLS**

|                     |   |                                   |
|---------------------|---|-----------------------------------|
| %                   | - | Percentage                        |
| °C                  | - | Degree celcius                    |
| Atm                 | - | atmosphere                        |
| cm <sup>2</sup> /Vs | - | Square centimetre per volt second |
| eV                  | - | Electronvolt                      |
| g/mol               | - | gram per mol                      |
| g/ml                | - | gram per mililiter                |
| G                   | - | Weight change (WVTR analysis)     |
| kHz                 | - | Kilo herz                         |
| kGy/h               | - | kilogray per hour                 |
| K/s                 | - | Kelvin per second                 |
| MHz                 | - | Mega herz                         |
| mW/mg               | - | mili watt per mili gram           |
| nm                  | - | nanometer                         |
| pC/N                | - | Picocoulombs per newton           |
| rpm                 | - | revolution per minute             |
| S.cm <sup>-1</sup>  | - | Siemens per centimeter            |
| t                   | - | Time                              |
| V/V                 | - | volume per volume                 |
| W/cm <sup>2</sup>   | - | Watt per sentimeter square        |
| w/v                 | - | Weight per volume                 |
| wt%                 | - | weight percentage                 |

## LIST OF ABBREVIATIONS

|      |   |   |
|------|---|---|
| ASTM | - | American Society of Testing and Materials |
| DSC  | - | Different scanning analysis               |
| DBSA | - | Dodecyl benzene sulphonic acid            |
| FTIR | - | Fourier transform infra-red               |
| KBr  | - | Potassium bromide                         |
| NMR  | - | Nuclear magnetic resonance                |
| PEW  | - | Polyethylene wax                          |
| PP   | - | Polypropylene                             |
| SEM  | - | Scanning electron microscopy              |
| SDS  | - | Sodium dodecyl sulphate                   |
| TGA  | - | Thermogravimetric analysis                |

## CHAPTER 1

### INTRODUCTION

#### 1.1 Background of the Study

Over the past three decades, there has been increasing interest in the conducting polymers field due to their interesting electrical properties. Conjugated polymers are of interest that lead them to the wide range of useful applications include molecular electronics, transistors, solar cells, sensors, batteries, polymer light emitting diodes, antistatic coatings, energy storage devices and electromagnetic interference shielding (Han, 2009; Hai *et al.*, 2017). The conducting polymers have main chain of  $\pi$ -conjugation, which possess electronic properties of metals, while retaining the mechanical properties and processibility of conventional polymer (Vernitskaya and Efimov, 1997). The transport of electrons can be assumed to occur by the motion of delocalized electron through the conjugated systems (Cho *et al.*, 2006). This condition makes the conductive polymers exhibit highly reversible redox behaviour and the unusual combination of metal and plastics properties (Higazy *et al.*, 2006). Normally, conducting polymer will aggregates in doped state. There are two possible causes of aggregates: (1) an increase in rigidity of the polymer chain on doping and (2) increased polar interactions between the polymeric chains (Bhattacharya and Misra, 2004).

The conducting polymers that have been widely studied and have great interest for being candidates in various electronic devices included polyaniline (PANI), polypyrrole (PPy) and polythiophene (PTH), as compared with activated carbon and transition metal oxides (Wu *et al.*, 2007; Frackowiak and Beguin, 2002). Among the conducting polymer, polypyrrole (PPy) is one of the most attractive polymer for

commercial applications due to the high conductivity, redox property, good environmental stability and ease to synthesize (Chen *et al.*, 2008; Baytekin, 2009). PPy and its derivatives usually prepared by either electrochemical or chemical polymerisation. Electrochemical polymerisation can be performed in either aqueous or organic media. Wei *et al.* (1991) stated that the applied potentials are required to be higher than the oxidation potentials of monomers in order to achieve a fast rate polymerisation. However, at high potential, the polymers might undergo undesirable side reactions such as crosslinking and degradation resulting from over-oxidation. It also might lead to substandard quality of the polymer including irreversible loss of electroactivity.

Chemical polymerisation has become the choice of many industries since it has a simple preparation, short reaction time and can produce a bulk production, although the resulted polymers are poor quality and low electroconductivity compared with electrochemical polymerisation (Baytekin, 2009). For example, a report by Jang *et al.* (2002) synthesized PPy nanoparticles as small as 2 nm in diameter at low temperature of 3 °C by microemulsion polymerisation with various surfactant. Wang *et al.* (2005) also used microemulsion polymerisation in synthesizing PPy nanoparticles for about 3 hours. Liu *et al.* (2006) synthesized PPy nanoparticles by using alcohol-assisted microemulsion polymerisation in order to adjust the inner structure. Zhang and Manohar (2005) synthesized narrow pore-diameter polypyrrole nanotubes by chemical oxidative polymerisation using vanadium pentoxide, V<sub>2</sub>O<sub>5</sub> nanofibers as the sacrificial template.

Thermoplastic is an attractive material in the polymer industry due to their good processability (Legge *et al.*, 1989), particularly interest is the non-polar thermoplastic polyolefin, namely polypropylene (PP). PP is in commercial importance because of its cost-effectiveness as well as intrinsic properties such as low density, high stiffness, good tensile strength and inertness toward acids, alkalis and solvents (Pasquini, 2005). Thus, PP has been used in a wide range of applications including packaging, textiles, automotive components and so on (Zare *et al.*, 2014). The functionalized of PP through grafting polar monomers such as maleic anhydride and acrylic acid because in many cases the grafted monomer functionality is able to react



with  $-OH$ ,  $-COOH$  and  $NH_2$  (Flores-Gallardo *et al.*, 2001). Several literatures have been reported on grafting PP as a matrix such as the work by Flores-Gallardo *et al.* (2001) have prepared PP-grafted acrylic acid blends by using Brabender mixing chamber and Ni *et al.* (2011) who prepared PP-grafted maleic anhydride by using extruder. In addition, Oramiehie *et al.* (2014) also grafting maleic anhydride onto PP as the matrix. Besides that, Hai *et al.* (2017) who grafted poly(3-hexylthiophene) onto the PP surface by chemical oxidative polymerisation

Nowadays, modification is an interesting field in both basic and applied research. Several methods for modifying have been reported in the literature that included blending, curing and grafting. Blending polymers is the physical mixture of two or more polymers to obtain the required properties (Bhattacharya and Misra, 2004). The thermodynamically incompatible is the reason of blending technique was applied (Chen and Li, 2005). Due to the blends, mechanical properties greatly rely on the morphology, and are normally very poor due to the lack of physical or chemical interactions between different phases (Hope and Folkes, 1993). In the practical blend processing, it is hard to get a better dispersion if viscosity of the disperse phase is much higher than that of the matrix (Chen and Li, 2005). Meanwhile, curing is a polymerization of oligomer mixture forms a coating which adheres to the substrate by physical forces. On the other hand, grafting is a method wherein monomers are covalently bonded onto the polymer chain and two or more different monomers are grafted side-by-side to obtain the necessary property. Grafting can reduce the rigidity of the polymer chain and hence, provides a potential technique to overcome this limitation (rigidity) so that can improve the processability of the conducting polymer and to obtain the particular use properties required (Bhattacharya and Misra, 2004). Stanke *et al.* (1995) successfully grafted the pyrrole onto a poly(methyl methacrylate (PMMA) backbone. The graft copolymers of pyrrole/ poly(methyl methacrylate) (Py/PMMA) with very low Py amounts were soluble but rapidly insoluble with the increasing Py due to the formation of long Py sequences. Bae *et al.* (2005) have prepared the PPy graf copolymer/ clay by in situ polymerization of pyrrole onto pre-exfoliated water-soluble poly(styrenesulfonic acid-co-pyrrolylmethyl styrene) (P(SSA-co-PMS)/ clay nanocomposite or by simple blending of poly(styrene sulfonic acid-g-pyrrole) (PSAA-g-PPy) with clay.

Ultrasound is a type of elastic mechanic wave of frequency  $10^4 - 10^8$  Hz that can influence a medium through which the waves propagate by sound streaming action, shatter, cavitation and etc. (Chen and Li, 2005). The application of ultrasonic irradiation has been widely used in chemical reaction such as organic synthesis and polymerisation. This is due to the ultrasonic cavitation that can produce temperatures until 5000K and pressures until 500 atm, with heating and cooling rates greater than 109 K/s (Haldorai *et al.*, 2011). Kuijpers *et al.* (2004) stated that the ultrasound-induced the polymer breakage is a direct effect of the cavitation. The breakage of the C-C bond by ultrasound generally leads to the development of long-chain radicals in polymer solutions, which can cause the formation of copolymer by coupling of those long-chain radicals from different polymers (Lebovitz *et al.*, 2003). This technique is a useful for preparing many materials with improved properties (Haldorai *et al.*, 2011). Several studies have shown that the employment of ultrasonic on polymer liquid processing will give the physical changes such as rapid mixing and high yield of final product (Watanabe *et al.*, 1993). Sonication process also can help in the dispersion quality (Lim *et al.*, 2003) and give well-crystallized of polymer (Liu *et al.*, 2002). Polymer prepared by ultrasonication gives more thermally stable than prepared by conventional method (Ginic-Markovic *et al.*, 2006). For conducting polymer, the used of ultrasonic irradiation contributes to the increase in conductivity properties of the final product (Xia and Wang, 2002). Ultrasound is also known to be useful for initiating reactions and enhancing reaction rates (Kobayashi *et al.*, 2013).

According to Zhou *et al.* (2008), the ultrasonic irradiation can partly broke down the agglomerates of fillers which then provides the new reactive spaces for chemical bonding with other polymers. Nevertheless, Wenduan and Xi (1983) have used the ultrasonic irradiation on grafting of polyvinyl acetate (PVAc) with polyethylene oxide (PEO). PEO and PVAc in the benzene solution were degraded through the strong impulsive waves produced by the ultrasonic cavitation. During the irradiation, the desired copolymer was formed by the recombination of the different macroradicals or by the chain transfer reactions between a macroradical and another macromolecular chain.

Extruders capable of efficient devolatilizing are required for use in the preparation of bulk thermoplastic. Normally, the volatile components, monomers and low molecular weight components are more challenging to be removed and contributed to the large part of the remaining volatile components in the final extruded bulk products (Li *et al.*, 1995). The acoustic treatments are introduced into the devolatilizing sections in order to increase the efficiency of extrusion devolatilizing performance (Tukachinsky *et al.*, 1993). Recently, ultrasound has been applied to the melt state polymer during processing. Chen and Li (2005) have used a single-screw extruder with ultrasonic-assisted in preparing PP/ethylene-propylene-diene terpolymer (EPDM) blends. They found that the blends prepared under ultrasonic irradiation become more homogenous than the blends prepared with the conventional method. Oh *et al.* (2003) and Kim and Lee (2002) found that the utilization of ultrasonic irradiation can improve the interfacial adhesion and compatibilisation between different phases of immiscible polymer blends.

In the present study, ultrasonic irradiation was employed to PPy polymerisation. Ferric chloride hexahydrate,  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  was used as the oxidant, which helps in the formation of the conductivity of PPy. Sodium dodecylsulfate (SDS) was used as a surfactant that help in stability of PPy in organic solvent. The PPy was then characterized by fourier transform infrared spectroscopy (FTIR), thermogravimetric analysis (TGA), differential scanning calorimeter (DSC), field emission scanning electron microscopy (FESEM) and electrical conductivity test. After that, the different composition of PPy was grafted with PP as the matrix. Nevertheless, PP is abundantly available, easily processed and economically affordable. The single screw extruder with ultrasonic-assisted was employed to produce PP-g-PPy. Dicumyl peroxide (DCP) was used as the initiator to form the free radical.

## 1.2 Problem Statement

Conducting polymers such as PPy has been studied during the last decade due to high conductivity, good environmental stability and simple preparation (Baytekin, 2009). Generally, PPy can be prepared by either chemical or electrochemical polymerisation. There are some problems associated with the PPy polymerisation from the literature such as long period needed to produce PPy and the temperature used was also quite high that will influenced the size of resulted particles. For example, Liu *et al.* (2006) have synthesized PPy by using chemical polymerisation for 12 hours while Sevil and Zuhail (2010) synthesized PPy by microemulsion polymerisation for 3 hours at room temperature. Fortunately, Kobayashi *et al.* (2013) have utilized the ultrasonication for PPy polymerisation in 1 hour with the varied frequencies at 22.8, 490, 940 and 1600 kHz. However, no study has been reported yet on the effect of different doses of ultrasonic intensity on PPy polymerisation while fixed the frequency. The ultrasonic intensity is one of the important parameters that plays a leading role in any reaction (Sivakumar *et al.*, 2006). Therefore, it is reasonable to expect a change in magnitude of a reaction with a change in magnitude of the ultrasonic intensity.

PP has been chosen as matrix in this study as PP is one of the recognized polymers in wide variety of fields such as packaging, textiles and automotive materials (Mazov *et al.*, 2014). PP also is an interesting material as compared with the other polymer because of its cheap, water resistant, transparent, light weight, low density, good mechanical property, chemical resistance and the ability to be shaped in various forms (Jin *et al.*, 2011). The combination of PP with PPy is one of the alternative to improve the properties of PP due to its highly non-polar nature and also to improve the mechanical properties of PPy at the same time. However, during the production that involved PP, the surface of polymer can charged easily which result in the serious problems such as damage to sensitive electronic equipment (Hai *et al.*, 2017). Therefore, modification by grafting of conjugated polymer on the PP may improve the antistatic properties of PP because of the conjugated structure can transfer the static charges (Hai *et al.*, 2017). The aim of this study is to identify the characteristics trends

that might be useful for investigation of their potential application in various electronic devices.

### **1.3 Objective of the Study**

The objectives of this study are as follows:

1. To investigate the effect of different doses of ultrasonic intensity on PPy from 20 – 100% on the functional group, thermal and thermal degradation, morphological and conductivity properties.
2. To investigate the effect of PPy composition on the functional group, rheological, mechanical, morphological, electrical conductivity and thermal properties of PP-g-PPy blends.

### **1.4 Scope of the Study**

In this study, the first task was to synthesize PPy from Pyrrole monomer under ultrasonication with fixed frequency wave of 20 kHz and varying the ultrasonic intensity from 20 – 100%. The model of the ultrasonic equipment used was a BILON-1200Y ultrasonicator with the power supply was 1200 watt and the voltage was 220 volt. The temperature of the water bath was maintained at 5 °C and the polymerization process was run for 30 minutes. The oxidant and the surfactant used was ferric chloride hexahydrate and sodium dodecylsulphate, respectively. After polymerization, the yield of PPy was calculated and then tested with fourier transform infrared spectroscopy (FTIR), differential scanning calorimeter (DSC), thermogravimetric analyzer (TGA), field emission scanning electron microscopy (FESEM), and two point probe tester.

In the second task of the PP-g-PPy preparation, the PPy sample underwent the grafting process with the thermoplastic of PP by using the ultrasonic single screw extruder & injection moulding. During the process, the samples were prepared with

varied PP/PPy content of 100/0, 95/5, 90/10, 85/15 and 80/20 wt%. Characterization of the functional groups and the internal networks structure of PP-g-PPy samples using FTIR and FESEM respectively. Mechanical properties of PP-g-PPy were studied by using tensile test. Using the dynamic mechanical analysis (DMA), PP-g-PPy were characterized to study the damping factor, storage and loss modulus of the samples. The thermal analysis was done by using DSC for the compatibility investigation. TGA was used to study the thermal stability in terms of temperature decomposition. The viscosity of the samples also were characterized by rheological test. Finally, the electrical conductivity properties were measured by the two point probe testing.

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