MECHANICAL, THERMAL AND FLAMMABILITY PROPERTIES OF HYBRID RICE HUSK/NANOFILLERS FILLED POLYPROPYLENE NANOCOMPOSITES

EZENKWA, OBINNA EMMANUEL

UNIVERSITI TEKNOLOGI MALAYSIA

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EZENKWA OBINNA EMMANUEL

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ABSTRACT

This study investigated the mechanical, thermal, and flammability properties performance of hybrid rice husk / nanofiller filled polypropylene (PP) nanocomposite. With rice husk (RH) content at 15 wt.%, and graphene nanoplatelets (GNP) at increasing contents of 0.5, 1, 1.5, 2, 2.5, and 3 parts per hundred (phr) of PP/RH composite, the hybrid blends were melt blended in a twin-screw extruder and injection molded for tensile, flexural, impact, thermal and flammability tests. Before this hybrid combination, a comparative study on RH and rice husk ash (RHA), and different compatibilizers of PP grafted maleic anhydride (MAPP) and ethylene-acrylic ester maleic anhydride (E-AE-MA) was studied. The result showed that RH performed better than RHA with MAPP better than E-AE-MA in enhancing mechanical and thermal properties. Further comparative study between the nanofillers of GNP, graphene oxide (GO) and halloysite nanotubes in PP nanocomposite systems also showed that GNP exhibited better mechanical and thermal stability property. Hence the hybrid of GNP-RH filled PP nanocomposite was chosen for a synergistic probe. Among the hybrid blends, results showed that GNP optimum content was attained at 1phr with enhanced tensile strength (25%), tensile modulus (72%), flexural strength (10%), flexural modulus (0.3%), impact strength (29%), thermal stability (25 °C) and flame retardancy-LOI (5%), compared to single-filled PP/RH composite system. Microstructural analysis of the tensile fractured surfaces of the hybrid blends revealed good nanofiller dispersion was attained with 0.5 and 1phr GNP contents demonstrating good filler-matrix interfacial bonding quality aided by MAPP compatibilizer and, responsible for excellent composite strength performance. Based on the PP/RH/GNP/MAPP hybrid nanocomposite field emission scanning electron microscopy micrograph, an interaction model of RH and GNP fillers with PP matrix was suggested in which GNP nanosheets are observed to attach to the layers of RH fibers and filled possible gaps between PP and RH necessitating effective stress transfer from the matrix to the fillers. The void filling mechanism, large surface area, and exfoliation of GNP nanosheet within the hybrid nanocomposite system are believed to be contributory to the enhanced properties of the hybrid nanocomposite compared to the single-filler filled PP/RH composites. It can be noted that formulations involving single nanofillers-PP nanocomposite systems attained high stiffness-toughness balance with PP/GNP/MAPP nanocomposite achieving increased tensile strength (8%), tensile modulus (96%), elongation at break (29%), flexural strength (20%), flexural modulus (18%), impact strength (104%) and thermal stability (12 °C) compared to pristine PP. The outcome of this study suggests that synergistic incorporation of GNP and RH is beneficial to the enhancement of overall hybrid PP nanocomposite properties.

ABSTRAK

Kajian ini menyiasat prestasi sifat mekanikal, terma dan kebolehbakaran nanokomposit polipropilena terisi sekam padi / nano pengisi hibrid. Dengan kandungan sekam padi (RH) pada 15 wt.%, dan nanoplatelet grafin (GNP) pada peningkatan kandungan 0.5, 1, 1.5, 2, 2.5 dan 3 bahagian per seratus (phr) komposit PP/RH, adunan hibrid telah diadun lebur ke dalam penyemperit skru berkembar dan acuan suntikan untuk ujian tegangan, lentur, hentaman, terma dan kebolehbakaran. Sebelum gabungan hibrid ini, satu kajian perbandingan antara RH dan abu sekam padi (RHA), dan penyerasi berbeza anhidrida maleik tercantum PP (MAPP) dan anhidrida maleik ester etilena akrilik (E-AE-MA) telah dikaji. Keputusan kajian menunjukkan bahawa RH berprestasi lebih baik daripada RHA dengan MAPP lebih baik daripada E-AE-MA dalam meningkatkan sifat mekanikal dan terma. Kajian lanjut perbandingan antara pengisi-nano GNP, grafin oksida dan tiub-nano hallosit dalam sistem nanokomposit PP juga menunjukkan bahawa GNP mempamerkan sifat mekanikal and kestabilan terma yang lebih baik. Oleh itu nanokomposit PP terisi hibrid GNP-RH telah dipilih untuk siasatan sinergistik. Antara kesemua campuran hibrid, keputusan menunjukkan bahawa kandungan optimum GNP adalah pada 1 phr dengan peningkatan kekuatan tegangan (25%), modulus tegangan (72%), kekuatan lentur (10%), modulus lentur (0.3%), kekuatan hentaman (29%), kestabilan terma (25° C) dan kalis api-LOI (5%), berbanding sistem komposit PP/RH terisi tunggal. Analisis mikrostruktur pada permukaan patah sampel tegangan bagi adunan hibrid menunjukkan penyebaran pengisi nano yang baik telah dicapai dengan 0.5 dan 1 phr kandungan GNP menunjukkan kualiti ikatan antara muka pengisi-matriks yang baik dibantu oleh penyerasi MAPP dan, bertanggungjawab untuk prestasi kekuatan komposit yang cemerlang. Berdasarkan mikrograf mikroskop elektron imbasan pelepasan medan bagi nanokomposit hibrid PP/RH/GNP/MAPP, model interaksi pengisi RH dan GNP dengan matriks PP telah dicadangkan di mana helaian nano GNP dilihat melekat pada lapisan gentian RH dan mengisi mugkin ruang kosong antara PP dan RH memerlukan pemindahan tegasan yang berkesan daripada matriks kepada pengisi. Mekanisme pengisian lompang, luas permukaan yang besar dan pengelupasan helaian nano GNP dalam sistem nanokomposit hibrid dipercayai menyumbang kepada peningkatan sifat nanokomposit hibrid berbanding komposit PP/RH terisi tunggal. Dapat diperhatikan sistem nanokomposit PP terisi tunggal mencapai keseimbangan kekakuan-kekukuhan yang tinggi dengan nanokomposit PP/GNP/MAPP meningkatkan kekuatan tegangan (8%), modulus tegangan (96%), pemanjangan semasa putus (29%), kekuatan lentur (20%), modulus lentur (18%), kekuatan hentaman (104%) dan kestabilan terma (12 ° C) berbanding PP tulen. Hasil kajian ini menunjukkan bahawa penggabungan sinergistik GNP dan RH adalah bermanfaat kepada peningkatan sifat nanokomposit PP hibrid keseluruhannya.

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LIST OF ABBREVIATIONS

0-D	-	zero dimensional
1-D	-	one dimensional
2-D	-	two dimensional
3R	-	reduce, recovery and reuse
ANOVA	-	analysis of variance
APP	-	ammonium polyphosphate
ASTM		American society for testing and materials
BBT	-	2,5-bis (2-benzoxazolyl) thiophene
BRHA	-	black rice husk ash
CM	-	compression moulding
CNF	-	carbon nanofiber
CNT	-	carbon nanotube
CVD	-	chemical vapour deposition
DMF	-	dimethylformamide
DSC	-	differential scanning calorimeter
EAEMA	-	ethylene acrylic ester maleic anhydride
EDXRF	-	energy dispersive x-ray fluorescence
EPDM	-	ethylene propylene diene elastomer
EPR	-	ethylene propylene rubber
FESEM	-	field emission scanning electron microscopy
FGS	-	functionalized graphene sheets
FIGRA	-	fire growth rate
FRPCs	-	fiber reinforced polymer composites
FTIR	-	fourier transform infrared spectroscopy
GBMs	-	graphene based materials
GNS	-	graphene nanosheets
GNPs	-	graphene nanoplatelets (exfoliated)
GO	-	graphene oxide
GQDs	-	graphene quantum dots
HDT	-	heat deflection temperature

HNTs	-	halloysite nanotubes
HRR	-	heat release rate
iPP	-	isotactic polypropylene
ISO	-	international standards organization
KF	-	kenaf fiber
KBr	-	Potassium bromide
KPM	-	kenaf polypropylene mapp compatibilized composite
KPMG	-	kenaf polypropylene mapp compatibilized graphene hybrid nanocomposite
LOI	-	limiting oxygen index
MAPP	-	polypropylene grafted maleic anhydride
NG	-	nano graphene
PAA	-	poly acrylic acid
PALF	-	pineapple leaf fiber
PEN	-	poly(ethylene-2,6-naphthanate)
phr	-	parts per hundred
pHRR	-	peak heat release rate
PMMA	-	Poly methyl methacrylate
PP	-	polypropylene
PVC	-	poly vinyl chloride
QM	-	quaternary ammonium salt
RH	-	rice husk
RHA	-	rice husk ash
SDG	-	sustainable development goals
SEM	-	scanning electron microscopy
T_c	-	crystallization temperature
TGA	-	thermogravimetric analysis
THR	-	total heat release
T_{max}	-	temperature at maximum decomposition
T_m	-	melting temperature
T_{onset}	-	onset temperature of decomposition
VSP	-	vicat softening point
WAIM	-	water assisted injection molding

WRHA	-	white rice husk ash
Wt.%	-	weight percent

LIST OF SYMBOLS

ΔH_f	-	fusion enthalpy of polymer sample in a composite
ΔH_f^o	-	fusion enthalpy for 100% crystallinity polymer

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CHAPTER 1

INTRODUCTION

1.1 Background

Composites generally consist of at least two distinct materials whereby one is reinforcement and the second is the matrix (Afzal & Nawab, 2021). In composites, there are three main phases: the matrix, the reinforcement, and the interface region (Šupová *et al.*, 2011). The matrix system in a composite is either metallic, ceramic, or polymeric (Altenbach *et al.*, 2018). A Polymer composite is a multi-phase material in which the reinforcing fibers or fillers are integrated with a polymer as a matrix resulting in enhanced mechanical properties that cannot be achieved from a single component (Wang *et al.*, 2017). Polymeric composites represent about 75% of composite materials production (Todor *et al.*, 2018). Fiber reinforced polymer composites offer high strength to weight ratio, high durability, stiffness, damping property, flexural strength and resistant to wear (Rajak *et al*., 2019). In fiber reinforced polymer composite structure, the interfacial region in the composite structure is responsible for 'communication' between the matrix and the filler and has properties different from the bulk matrix because of its proximity to the surface of the filler (Šupová *et al.*, 2011). Natural fiber-reinforced polymer composites are known as bio-composites and further categorized as complete or partial green composites (Khalid *et al.*, 2021). The term bio-composites refers to composites in which plant based natural fibers such as kenaf, sisal, jute, rice husk and/or coconut fiber are reinforced with either biodegradable or non-biodegradable matrices (Militky & Jabbar, 2015).

Polypropylene (PP), petroleum-derived thermoplastic polymer has been widely used as a matrix in composite fabrication due to its excellent and desirable physical, mechanical and thermal properties (Kumar *et al.*, 2010). However, due to its aliphatic carbon structure, PP burns rapidly without leaving a char residue with a low self-ignition temperature level of 357 °C (Motahari *et al.*, 2015). Furthermore, PP has a high melting point of ~160 °C and low glass transition temperature at ~ -25 °C, limiting the polymer's applicability in the low-temperature range due to its low temperature poor impact-toughness (Greene, 2021). In order to circumvent PP's problem of embrittlement at subzero environment, copolymerization with ethylene block copolymers such as ethylene propylene rubber (EPR) is often adopted (Shirvanimoghaddam *et al.*, 2021). Although, the copolymerization of PP with EPR improves the impact strength and toughness of PP, a decrease in stiffness of the PP copolymer is observed at high content of EPR (Gahleitner *et al.*, 2013; Shirvanimoghaddam *et al.*, 2021). To improve the stiffness of polymers, fibers and fillers are often incorporated (Deng *et al.*, 2019; Raghavendra *et al.*, 2013; Raghu *et al.*, 2018; Senthilkumar *et al.*, 2018). However, achieving a balance of improved stiffness and toughness is paramount in enhancing the application needs of PP especially in packaging and automotive exterior parts (Gahleitner *et al.*, 2013).

Among fibers and fillers for reinforcement in PP composites are rice husk. Rice husk (RH) natural fiber belongs to the 'stalk' vegetable class of fibers (Kumar *et al.*, 2018). Rice husk is a by-product of the industrial processing of paddy rice and accounts for approximately 20 wt.% of the bulk grain weight (Hossain *et al.*, 2018). The main constituents of rice husk are organic substances such as cellulose, hemicellulose, lignin (70-80%), and mineralogical components such as silica, alkalis, and trace elements (20-30 wt.%) (Sarangi *et al.*, 2009). The composition of rice husk, however, depends on plant variety, climatic conditions, and geographic locations (Hossain *et al.*, 2018). Rice husk as an agricultural waste poses a disposal problem, but when incorporated as a filler into polymer composites, increases the flexural strength, leading to a low-cost product with high strength and good performance (Arumugaprabu & Pragatheeswaran, 2019). In Malaysia, over 800,000 tonnes of RH and stalk waste are produced annually (Suhot *et al.* 2021), which portends a high availability of raw materials as reinforcing fibers in polymer composites. Application of rice husk in polymer composite fabrication contributes immensely to environmental and waste management compared to other natural fibers. As a filler in polymer composites, rice husk can be applied in its raw form (RH) or rice husk ash form (RHA) which is a product of calcined raw rice husk. In a raw form, RH is relatively high (above 90%) in silica when extracted with acid or alkali (Hossain *et al.* 2018). Rice husk ash is composed majorly of silica at high contents of about 95% and 54% for the white ash and black ash grades respectively (Pongdong *et al.*, 2018).

Molecular interaction and adhesion between RH and RHA and non-polar polymers such as polypropylene are often a challenge due to their incompatibility. Like many lignocellulosic fibers, RH and RHA are hydrophilic. Incorporation of RH or RHA as fillers with non-polar polypropylene often results in composite with high stiffness and low impact strength. The resulting poor impact property of PP filled RH or RHA composites has been attributed to poor interfacial bonding between the hydrophilic fillers and non-polar matrix, inducing micro spaces and numerous micro-cracks during impact which results in easy crack propagation and decreased impact strength of the composite (Yang *et al.*, 2004). In previous study (Premalal *et al.* 2002), RH loading beyond 15 wt.% was found to result in decreasing RH/PP composite tensile strength mainly due to the occurrence of interfacial discontinuities. Consequently, filler-matrix interfacial modification is often applied to improve interfacial interactions between the composite constituents.

Fiber surface treatment techniques such as mercerization (Chanda *et al.*, 2015; Liu *et al.*, 2004; Mina *et al.*, 2018), acetylation (Fávaro et al., 2010), electron irradiation (Ahmad *et al.*, 2012; Chong *et al.*, 2010; Rajendran Royan *et al.*, 2018), plasma (Nguyen *et al.*, 2011) and use of interfacial modifier such as compatibilizer/coupling agents (Bilal *et al.*, 2014; Huner, 2017a; Majeed *et al.*, 2014; Saidi *et al.*, 2018), have been variously employed to improve adhesion between the hydrophilic fiber or filler and non-polar polymer matrix. Although mercerization has seen wide application in fiber surface modification, it is challenged by its effect on the fiber material integrity under conditions above optimum. Studies involving fiber/filler surface modification by plasma and electron beam irradiation techniques (Chong *et al.*, 2010; Nguyen *et al.*, 2011; Rajendran Royan *et al.*, 2018) have shown their high effectiveness in enhancing polymer composites properties and retains fiber material integrity. However, its complexity and high cost compared to other techniques affect its wide application. Compatibilization and coupling though retain fiber material integrity, its effectiveness is dependent on the compatibility of the base polymer with the matrix polymer. Nevertheless, the compatibilization technique has seen wider application in rice husk-filled polymer composite fabrication compared to other techniques majorly due to its ease of application and observed mechanical properties enhancement. Compatibilizers such as PP grafted maleic anhydride (MAPP) and ethylene-acrylic ester maleic anhydride (E-AE-MA), function as interfacial modifiers by interacting with the hydroxyl groups of the natural fiber at its maleic anhydride group sites, forming ester linkages/hydrogen bonding and further entangling with the polymer matrix through its hydrophobic polymer base, thus establishing a fiber-matrix interfacial bonding (Mutjé *et al.*, 2006).

In recent times, nanotechnology is been employed in polymer composites fabrication. It combines the advantages of polymers and nanoparticles for creating high-performance materials known as polymer nanocomposites (Kim, *et al.*, 2010). Various nanofillers in use include but are not limited to nano-clay, carbon nanotubes, carbon nanofibers, and graphene-based materials. Of most trending among the nanofillers is graphene. Graphene is a graphite-based nanofillers. Its unique properties include high electrical conductivity of 2 x 10^3 S/cm, high thermal stability up to 601 °C (Wu *et al.*, 2009), the high elastic modulus of 1 TPa (Lee *et al.*, 2008), the high thermal conductivity of 5300 W/Mk (Balandin *et al.*, 2008), large surface area (theoretical) of 2600 m^2/g (Rao *et al.*, 2010), ultra-high electron mobility of 200,000 Cm^2 V^{-1} S^{-1} (Bolotin *et al.*, 2008), etc. Graphene exists in various forms such as graphene nanoplatelets (GNP), graphene oxide (GO), reduced graphene oxide (rGO) (Goenka *et al.*, 2014), graphene nanoribbons (GNR), and graphene quantum dots (GQDs) (Pan *et al.*, 2010). These graphene base materials (GBMs) have different techniques of synthesis and differ from one another in both physical and chemical characteristics. Although each form of graphene has excellent qualities and can be tailored as per user requirement, GNP and GO have attracted massive attention as reinforcing nanofillers in polymer composites due to ease and good scalability of techniques of synthesis.

Halloysite nanotubes (HNTs) are an example of nano-clay-based nanofiller. HNTs are similar to CNT in morphology and are environmentally friendly (Liu *et al.*, 2014; Lvov & Abdullayev, 2013). Similar to GNP and GO, HNTs are characterized by a large aspect ratio, high mechanical strength, high availability, and ease of functionalization (Lvov *et al.*, 2016). Recent studies have shown that incorporation of nanofillers of GNP, GO and HNTs singularly, in PP based nanocomposites system with an interfacial modifier, enhanced the mechanical, thermal, and flammability properties of the nanocomposites compared to pure PP (Ajorloo *et al.*, 2019; Chen *et al.*, 2018; Duguay *et al.*, 2013; Inuwa *et al.*, 2014; Shin *et al.*, 2012; Yetkİn *et al.*, 2017). A combination of the advantages of different nanofillers and natural fibers in hybrid nanocomposites has been reported (Idumah & Hassan, 2017; Sheshmani & Amini, 2013; Subasinghe *et al.*, 2016). In hybrid polymer nanocomposite fabrication, GNP incorporated into wood/PP composite, as low as 0.8 %, highly increased the tensile strength, impact strength, flexural strength, with a decrease in water uptake property of the nanocomposite (Sheshmani & Amini, 2013). Thermal stability and flame retardancy of GNP incorporated hybrid PP filled kenaf fiber composite was found to enhance significantly compared to pure PP and PP-Kenaf composite (Idumah & Hassan, 2017). The good thermal transport property of graphene has also endeared its use as thermal interface material in polymer matrix for thermal management in electronic equipment (Mahanta & Abramson, 2012). A hybrid of GO/SiO_2 at 0.25 wt.% was found to enhance the mechanical and thermal properties of PP-based GO/SiO_2 hybrid nanocomposites (Bian *et al.*, 2017). Incorporation of 2 vol% of HNTs into PP-kenaf composite was also found to improve mechanical and thermal properties of the hybrid nanocomposites compared to pure PP and when compared to only PP-kenaf composite (Franciszczak *et al.*, 2020).

Currently, much of the development on natural fiber-based hybrid nanocomposites is centered around commercially viable natural fibers. The incorporation of graphene as reinforcement enhancing agents in PP filled RH/RHA composites is scant and where it exists (Chang *et al.*, 2017), is replete with poor mechanical properties due to a high degree of filler agglomeration. The utilization of rice husk in polymer composites fabrication has not been advanced in industrial applications as compared to wood, jute, sisal, kenaf, and so on. Nevertheless, its high global natural abundance in the tune of 167.1 million tonnes annually, promises a high economic advantage for the advancement of rice husk natural fiber-based polymer composites. Up to date, no study has reported on mechanical, thermal, and flammability properties of hybrid rice husk-graphene nanofiller reinforced polypropylene nanocomposite. Hence, enormous opportunities for further research and development of nanofiller-based hybrid rice husk-filled polypropylene nanocomposites abound which can be demonstrated in this present work.

1.2 Problem Statement

PP (impact copolymer grade) with high content of elastomeric phase is known to have good toughness but low stiffness (Gahleitner *et al.*, 2013; Shirvanimoghaddam *et al.*, 2021). Incorporation of rice husk fillers in PP results in polymer composites with improved stiffness and decreased impact toughness (Yang *et al.* 2004; Erdogan & Huner, 2018; Raghu *et al.*, 2018; Yiga *et al.*, 2019). This stiffness-toughness trade-off is a fundamental drawback to the industrial application needs of rice husk-filled PP composites. Achieving increased stiffness with the improved impact strength of PP composites will improve its application needs in packaging and automotive exterior parts where a balance of rigidity and toughness is especially required (Gahleitner *et al.*, 2013). Another challenge of PP copolymer is its high flammability characteristics. Due to its aliphatic structure, PP burns readily and completely with a high degradation rate (Motahari *et al.*, 2015). At a limiting oxygen index (LOI) of 17.9 % (Younis, 2017), PP burns readily at a short time with an increased burning rate. The incorporation of rice husk fillers often decreases the thermal stability of PP composite mainly due to the diluent nature of rice husk fillers (Amash & Zugenmaier, 2000; Arjmandi *et al.*, 2017). Chang *et al.* (2017) investigated the flame retardancy of PP/RH composite using GNP and metal hydroxide flame retardants and reported improved flame retardancy of the hybrid nanocomposite at the expense of mechanical properties mainly due to non-use of interfacial modifiers.

Studies have shown that incorporation of nanoparticles can simultaneously improve the stiffness and ductility of nanocomposites (Maillard *et al.*, 2012), thereby offsetting the impact strength trade-off often associated with rice husk filler incorporation. Also, the incorporation of nanofillers such as graphene nanoplatelets (GNP) creates an interconnected network of exfoliated graphene sheets, with strong filler-matrix interfacial bonding which improves the thermal stability of polymer nanocomposites (Idumah & Hassan, 2017). In the same vein, the charring effect of nanofillers during thermal decomposition of nanocomposites offers a blanket shield that prevents easy transport of combustible products and delays the rate of nanocomposite decomposition. Despite this nanofiller advantage in polymer composite technology, several studies have reported poor compatibility of nanofillers GNP, GO, and HNTs with non-polar PP (Altay et al., 2019; Bakhtiari *et al.*, 2020; Sánchez-Valdes *et al.*, 2018). Ideally, good dispersion of nanoparticles in a nanocomposite system induces a spatially homogenous cavitation process in which there is the slow growth of voids leading to a slow cavitation process (Shi *et al.*, 2019). It is also observed that under well-dispersed nanoparticle conditions, instead of a fast craze deformation process during tensile deformation or fracture, a transition to a more shear-deformation-like process, which is also known as brittle-ductile transition, occurs (Margolina & Wu, 1988).

This present study investigates the effect of hybrid rice husk natural fiber/ nanofiller on the mechanical, thermal, and flammability properties of compatibilized PP nanocomposite. Before this hybrid combination, a comparative effect of two forms of rice husk fillers viz raw rice husk (RH) and rice husk ash (RHA), and two compatibilizers of PP-grafted maleic anhydride (MAPP) and ethylene-acrylic ester-maleic anhydride (E-AE-MA) has been investigated. Very limited study has been done comparing RH and RHA PP-based composites in a single study, and none has compared their flexural and flammability properties (Aprilla *et al.*, 2019; Moreno *et al.*, 2020). Also, no study has been done comparing MAPP and E-AE-MA compatibilizers in PP/RH composite systems. Again, reinforcing effects of graphene nanoplatelets (GNP), graphene oxide (GO) and halloysite nanotubes (HNTs) in PP-based nanocomposites has been compared. To date, no study has been done comparing the reinforcing effects of GNP, GO, and HNTs in PP-based nanocomposites' systems in a single study. Single study-based comparisons systematically guide towards the appropriate choice of polymer nanocomposites constituents and achievement of targeted properties of nanocomposites. Hence, a hybrid combination was based on observed better reinforcing rice husk filler, of mixed particle size range (100-900 µm), and nanofiller, and compatibilizer. To date, very limited study has been done on hybrid rice husk/nanofillers of GNP, GO, and HNTs in a PP-based nanocomposite system. The choice of a maleic anhydride-based compatibilizer for this study is such that the polar groups of the compatibilizer interact with the surface moieties of the fillers, lowering their surface energies and enhancing dispersion in the PP matrix (Sánchez-Valdes *et al.*, 2018). It is therefore a great interest in this study to investigate the effects nanofillers and compatibilizer as an interfacial modifier in rice husk-filled PP composites, concerning mechanical, thermal, and flammability properties of the resulting hybrid PP nanocomposites.

1.3 Research Objectives

The overall objective of this study is to fabricate a hybrid rice husk/nanofiller filled PP nanocomposites with improved properties of mechanical, thermal, and flammability. The specific objectives of this study, therefore, are:

(a) To examine the effects of types of rice husk (RH and RHA) as fillers and types of compatibilizers (MAPP and E-AE-MA) on mechanical, thermal and flammability properties of RH and RHA filled PP composites.

(b) To compare the effects of nanofillers of graphene nanoplatelets (GNP), graphene oxide (GO), and halloysite nanotubes (HNTs) on mechanical and thermal properties of PP based nanocomposites.

(c) To determine the hybrid effect of rice husk/nanofiller on the mechanical, thermal, and flammability properties of PP-based hybrid nanocomposites.

1.4 Research Scope

Based on objective 1, 15 wt.% of RH and 15 wt.% of RHA was incorporated into 85 wt.% of PP. In another formulation, 4 phr MAPP and E-AE-MA each, was added into PP/RH and PP/RHA to fabricate PP/RH/MAPP, PP/RH/E-AE-MA, PP/RHA/MAPP and PP/RHA/E-AE-MA composites by melt extrusion and injection moulding techniques. Neat PP at 100 wt.% was used as control. The obtained composite samples were characterized by mechanical (tensile, flexural and impact), thermal (TGA and DSC), and flammability (LOI) tests/analysis.

In objective 2, nanofillers of GNPs, GO and HNTs were incorporated into PP (100 wt.%) at varying contents of 1, 2, 3, and 4phr of PP, respectively, with MAPP fixed at 4phr of PP in each sample formulation to fabricate PP/GNP1/MAPP, PP/GNP2/MAPP, PP/GNP3/MAPP, PP/GNP4/MAPP, PP/GO1/MAPP, PP/GO2/MAPP, PP/GO3/MAPP, PP/GO4/MAPP, PP/HNTs1/MAPP, PP/HNTs2/MAPP, PP/HNTs3/MAPP and PP/HNTs4/MAPP nanocomposites by melt extrusion and injection moulding techniques. Neat PP at 100 wt.% was used as control. The fabricated nanocomposite samples were characterized by mechanical (tensile, flexural and impact) and thermal stability (TGA) tests/analysis.

The formulation for objective 3 was based on observed performance of the composite blends in objective 1 and 2. Hence, RH filler, MAPP compatibilizer, and GNP nanofiller were selected. Thus, hybrid of RH and GNP was chosen for synergistic probe in PP nanocomposite system. The formulation for the hybrid nanocomposites involved PP (85 wt.%), RH (15 wt.%), MAPP (4phr of PP/RH composite), and GNP at varying contents of 0.5, 1, 1.5, 2, 2.5 and 3phr of PP/RH composite, and were melt blended via melt extrusion and injection moulding techniques. The obtained hybrid nanocomposite blends were characterized by mechanical (tensile, flexural and impact), thermal (TGA and DSC), and flammability (LOI) tests/analysis. Neat PP (100 wt.%) and PP/RH (85/15 wt.%) were used as control.

1.5 Significance of Study

This study expects to fabricate PP based rice husk/nanofiller hybrid nanocomposite with improved mechanical, thermal, and flammability properties for improved application needs of PP. The utilization of rice husk agricultural waste promotes environmental sustainability and natural resource preservation through waste reduction, recovery, and reuse (3R). This study is also expected to contribute to the economies of scale in various areas of PP applications, as well as, promote the attainment of United Nations' Sustainable Development Goal (SDG) Goal 12.5: By 2030, which is to substantially reduce waste generation through prevention, reduction, recycling, and reuse.

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LIST OF PUBLICATIONS

Publications

1. **Ezenkwa, O. E**., Hassan, A. and Samsudin, S. A. (2021) Influence of Different Surface Treatments on Properties of Rice Husk Incorporated Polymer Composites, *Rev. Chem. Eng.*, 37, 907 (**Impact Factor: 6.299, Q1**)

2. **Ezenkwa, O. E.**, Hassan, A. and Samsudin, S. A. (2021) Tensile and Impact Properties of Rice Husk Filled Ethylene-Acrylic Ester Maleic Anhydride Compatibilized Polypropylene Composites, *Chem. Eng. Trans.*, 83, 511-516.

3. **Ezenkwa, O. E**.. Hassan, A. and Samsudin, S. A. (2022) Mechanical Properties of Rice Husk and Rice Husk Ash Filled Maleated Polymer Compatibilized Polypropylene Composites, *J. Appl. Polym. Sci.*, 139, 51702 **(Impact Factor: 3.125, Q2)**

4. **Ezenkwa, O. E.**, Hassan, A. and Samsudin, S. A. (2022) Comparison of Mechanical Properties and Thermal Stability of Graphene-Based Materials and Halloysite Nanotubes Reinforced Maleated Polymer Compatibilized Polypropylene Nanocomposites, *Polym. Compos.*, 43, 1852 **(Impact Factor: 3.171, Q2).**