EFFECTS OF LIGNIN IN NANOCRYSTALLINE CELLULOSE ON MECHANICAL, MORPHOLOGICAL AND THERMAL PROPERTIES OF KENAF CORE-REINFORCED POLYPROPYLENE COMPOSITES

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IPTPH 2019 10

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By

FATIMAH ATHIYAH BINTI SABARUDDIN

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirement for the Degree of Doctor of Philosophy

April 2019

Bismillahirrahmanirrahim

To my beloved parent, family, teachers and friends

Abstract of the thesis to Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Doctor of Philosophy

EFFECTS OF LIGNIN IN NANOCRYSTALLINE CELLULOSE ON MECHANICAL, MORPHOLOGICAL AND THERMAL PROPERTIES OF KENAF CORE-REINFORCED POLYPROPYLENE COMPOSITES

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April 2019

Chairman : Professor Paridah Md Tahir, PhD
Institute : Tropical Forestry and Forest Products

The excessive application of chemicals during the NCC production led to harmful chemical discharge that can damage the environment. The process of NCC production can be shorten by excluding the bleaching process and retain partial of lignin within NCC. Lignin was found to be high in thermal stability as it was able to withstand high temperature up to 900 ˚C. Lignin also possess polar and non-polar groups in its chain network which make lignin as good compatibilizer. The implication of lignin in NCC surface has been reported, however its effect on the nanocomposites has not been discussed yet. Hence, if lignin can be retained in NCC and posing no significant adverse effect on the composites, it would give a new perspective in the NCC application. Therefore, in this study, NCC from KC was produced through the soda-AQ pulping process and the lignin content in NCC were varied via different sequence of bleaching. Four different pulps were produced from the bleaching process denoted as UB (unbleached pulps), B1 (bleached up to stage D1), B2 (bleached up to stage D2), and B3 (bleached up to stage D3). All pulps were proceeded with acid hydrolysis treatment to produce the NCCs. All NCCs were analysed having different lignin content which confirmed via chemical analysis and kappa number test. The thermal analysis shows that, the NCC of high lignin content was not able to resist early thermal degradation temperature but was able to withstand a temperature up to 700 ℃ with highest amount of residual char. The NCCs (0.5 and 1 wt. %) then were incorporated within kenaf core reinforced polypropylene (PP/KC) composites with addition of 1 wt % of maleic anhydride grafted polypropylene (MAPP) and its effect on physical, chemical, mechanical, and thermal properties were determined. Overall, the presence of lignin in NCC help to improve the thermal stability of the nanocomposites with marginal effect on the mechanical properties. Furthermore, post alkaline treatment were done upon NCC having different bleaching condition to remove the remaining sulphates as by-product of acid hydrolysis process. However, post alkaline treatment were analysed to be unnecessary for unbleached NCC as it did not give any significant effect to the properties of the NCC and the nanocomposites.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

KESAN KANDUNGAN LIGNIN DALAM SELULOSA NANOKRISTAL TERHADAP SIFAT-SIFAT MEKANIKAL, MORFOLOGI DAN HABA TERHADAP TERAS KENAF DIPERKUAT KOMPOSIT POLIPROPALINA

Oleh

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Pengerusi : Profesor Paridah Md Tahir, PhD
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Penggunaan bahan kimia yang berlebihan semasa pembuatan NCC menyebabkan penghasilan bahan kimia berbahaya yang boleh merosakkan alam sekitar. Proses pembuatan NCC dapat dipendekkan dengan pengecualian proses pelunturan dan mengekalkan sebahagian daripada lignin didalam NCC. Lignin didapati mempunyai kestabilan terma yang tinggi kerana dapat menahan suhu tinggi sehingga 900 ˚C. Lignin juga mempunyai kumpulan kutub dan bukan kutub dalam rangkaian rantaiannya yang menjadikan lignin sebagai penserasi yang baik. Kesan lignin di permukaan NCC telah pun dibentangkan tetapi kesannya terhadap nano-komposit masih belum dibincangkan. Oleh itu, jika lignin dapat dikekalkan di permukaan NCC dan tidak memeberikan kesan yang tidak baik terhadap komposit, ia dapat memberi perspektif baru terhadap penggunaan NCC. Oleh itu, dalam kajian ini, NCC dari teras kenaf dihasilkan melalui proses pempulpaan soda-AQ dan kandungan lignin dalam NCC divariasikan melalui urutan prosess pelunturan. Empat pulpa berlainan dihasilkan dari proses pelunturan yang dikenali sebagai UB (pulpa yang tidak diluntur), B1 (diluntur hingga ke tahap D1), B2 (diluntur hingga ke tahap D2), dan B3 (diluntur hingga tahap D3). Semua pulpa kemudiannya diteruskan dengan proses hidrolisis asid untuk menghasilkan NCC. Semua NCC dianalisis mempunyai kandungan lignin yang berbeza dan disahkan melalui analisis kimia dan ujian nombor kappa. Analisis terma menunjukkan bahawa NCC dengan kandungan lignin tinggi tidak dapat menahan degradasi pada suhu awal tetapi dapat bertahan sehingga suhu 700 ℃. NCC (0.5 dan 1 wt.%) kemudiannya dimasukkan dalam komposit teras kenaf diperkuat polipropilena (PP / KC) dengan penambahan 1% berat maleik anhidrida dicantum polipropilena (MAPP) dan kesannya terhadap fizikal, kimia, mekanikal dan terma ditentukan. Secara keseluruhannya, kehadiran lignin di dalam NCC membantu meningkatkan kestabilan haba nanokomposit dengan memberi kesan kecil pada sifat-sifat mekanik. Tambahan pula, rawatan pasca-alkali telah dilakukan apabila NCC dengan sifat kelunturan yang berbeza untuk mengeluarkan sisa sulfat kesan hasil sampingan proses hidrolisis asid. Walau bagaimanapun, rawatan pasca-alkali telah dianalisis untuk tidak diperlukan untuk NCC yang tidak diluntur kerana ia tidak memberi kesan kepada sifat-sifat NCC dan nanokomposit.

ACKNOWLEDGEMENTS

In the name of Allah, Most Gracious, Most Merciful and Praise be to Allah, the Cherisher and Sustainer of the worlds for giving me chance to complete this work. I would also like to convey my sincere salam to His messenger and our prophet, Nabi Muhammad (SAW). I wish to express my sincere appreciation and gratitude to my supervisor Prof Dr Paridah Md Tahir, for her extremely helpful guidance, generous support and continuous encouragement with her wisdom, knowledge and experience. I would also thanks my co-supervisor, Dr Khalina Abdan, Dr Norkhairunnisa and Dr Adlin Sabrina for their kind support and meaningful contribution.

My appreciations also extends to all staffs of Institute of Tropical Forestry and Forest Products (INTROP) who have help me a lot in accomplishing my study. My gratitude also goes to my research mates, Naziratul Asikin, Zakiah Sobri, Nur Liyana Mohd Hafiz, Lee Chuan Li, Mohd Hizami, Norwahyuni, Redzuan and Aisyah Humairah for their help, encouragement and support throughout this research. My further appreciation goes to my post doctorate and fellow researcher; Dr Mohammad Asim Khan and Dr Lee Seng Hua for their help in contributing and accomplishing my research.

Finally, I would particularly like to thanks and acknowledge my parents, siblings and nephews for their countless help on giving me their affection and moral support. I will always be in their debt.

Thank you very much, only Allah can repay everything.

This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

ΔHf	Enthalpy of Fusion
ASTM	American Society for Testing and Materials
DMA	Dynamic Mechanical Analysis
DTG	Derivative Thermogravimetry
E'	Storage Modulus
E''	Loss Modulus
FTIR	Fourier Transform Infrared Spectroscopy
HDPE	High Density Polyethylene
KC	Kenaf Core
LDPE	Low Density Polyethylene
MAPP	Maleic Anhydride Grafted Polypropylene
MC	Moisture Content
NCC	Nanocrystalline Cellulose
NFRPC	Natural Fibre Reinforced Polymer Composites
OMMT	Organically Modified MMT
PLA	Poly Lactic Acid
PP	Polypropylene
PP	Polypropylene
PU	Polyurethane
PVC	Poly Vinyl Chloride
SEM	Scanning Electron Microscopy
Tan δ	Tan Delta
T_c	Crystallization Temperature
T_g	Glass Transition Temperature
TGA	Thermogravimetric Analysis
UPM	Universiti Putra Malaysia

CHAPTER 1

INTRODUCTION

1.1 Overview

The issue regarding limited petroleum source has affected many parties particularly in finding other alternatives on petroleum-based products. Recent studies reported that cellulose in plants is found to be the most common organic polymer and considered as almost infinite source of raw material to fulfil the increasing request for eco-friendly and bio-compatible products. It represents about 1.5 billion tonnes of the total annual biomass production (Klemm et al., 2009). Generally, the application of natural fibres and biomass crops impart more positive than negative effects on the environment (Frenando, 2013). It is important that at the end of materials' life cycle, the products should be reused, recycled or disposed. Hence, it is vital that disposable materials or products should be compostable in a short time. Natural fibres composites have the advantages over petroleum-based composites since the latter take much longer time to be composted (Kim et al., 2015). Therefore, the potential of exploiting the use of renewable material suddenly had become more interesting and equally important (Moon, Schueneman, & Simonsen, 2016).

Fibre-reinforced polymer composites (FRPCs) consist of a polymeric medium for example polypropylene (PP), polyester, epoxy, or poly-lactic acid, and a fibrous material such as carbon fibre, glass fibre, aramid fibre or natural fibre. The denotation of the composites is usually defined based on the fibre types, e.g., carbon fibre-reinforced polymer, glass fibre-reinforced polymer, and natural fibre-reinforced polymer composite (NFRPC). The mixture of two material types which known as "composite" material imparts better properties in both form and function to that of the constituent materials in isolation. In general, the fibres provide the strength while the polymeralso known as the matrix, provides dimensional stability and the transfer of shear stresses between the fibres. As a result, it produces a strong functional material which can be easily customised to deal with a wide variety of uses.

1.1.1 Advantages in Using NFRPC

Research and development have shown that there is a various range of potential application for NFRPC in the industry (Mwaikambo & Ansell, 2003; Mohanty, 2009). The most successful area for NFRPC is in non-structural architectural and functional applications for example doors, door frames, skirting boards, and other decorative features. They can be architecturally and aesthetically pleasing which have potential in a wide range of applications and unique products. Their proven durability in research suggests that NFRPCs are well suited to outdoor applications such as window frames or decking (Sharba et al., 2016).

Furthermore, natural fibres have imparted good thermal properties which the application of NFRPC might be useful in situations where thermal bridging could be an issue. Natural fibres also have good acoustic dampening properties although more research is needed to verify this. Preconceptions about the inherent weaknesses of natural fibres can obscure their potential as a serious alternative to glass fibre. Researches have shown that many of the mechanical issues associated with natural fibres that can be overcome with chemical modification and that natural fibres have similar properties to glass fibres. Furthermore, NFRPCs can be easily formed into structural components such as beams, reinforcing bars, and board materials that could potentially be used in racking panels.

1.1.2 Nanocellulose as New Source of Reinforcement in NFRPC

The versatility of nanocellulose as new reinforcement materials make rooms for consideration in a variety of possible applications. At the same time, the production of different grades of nanocellulose provides the opportunity for product enhancement, flexible applications, and new materials creation. Recently, researchers are focusing on the utilisation of nanocellulose in low volume and high performance applications (Salas et al., 2014).

Nanocellulose has unique combination of properties: high mechanical properties, low thermal expansion, high thermal stability, and relatively low density compared to inorganics and metals (Moon et al., 2016). High surface-area-to-volume ratio of nanocellulose allows them to exert a large effect on their local surrounding while acting as a stiff phase in suspension and composites. Its application can be varied such as a reinforcement in polymer composites, an additive in paper-based products, food packaging products, an additive in cement-based materials biomedical applications, etc. (Moon et al., 2016).

Nanocellulose can be produced by extracting cellulose from plants via purification involving chemical pretreatments which consists of alkali extraction and series of bleaching process. As reported by Dufresne (2013), the Young's modulus of nanocellulose with density for crystalline cellulose of around 1.5−1.6 g/cm^3 is much higher than that of the glass fibre, around 70 GPa with a density around 2.6 g/cm^3. Nanocellulose also has greater axial elastic modulus than Kevlar and potentially stronger than steel. Therefore, outstanding mechanical properties can be obtained by mixing nanocellulose with a polymer matrix even at low amount of filler loading. Such properties originate from the high stiffness of crystalline cellulose, the nanoscale dimension, and high aspect ratio that provide the strength to the nanocomposites (Moon et al., 2011; Dufresne, 2013). Those great properties make nanocellulose the most suitable additive to produce high performance and environmental friendly composites materials for the future.

1.2 Problem Statement and Justification

Nowadays, apart from the mechanical properties, thermal stability has become increasingly important to comply with the safety requirements during application (Rault et al., 2015). Thus, the needs for high thermal stable composites are increasing. Polymer matrices such as polypropylene (PP) and polyethylene (PE), have high ability to burn easily because it is mainly composed of carbon and hydrogen. When supported with high temperature, it will easily ignite once it is mixed with the oxygen in air, and melt rapidly due to its wholly aliphatic hydrocarbon structure (Loutid et al., 2009). It becomes more crucial when the composites are added with natural fibres as it increases sensitivity of the composite to burn. In addition, most natural fibres that are used as a reinforcement are thermally unstable above 200 °C (Pickering et al., 2016). The selection of polymer matrix also limited to those with lower melting temperature at which natural fibre degrades. Therefore, only thermoplastic that softens below this temperature such as PE, PP, polyolefin, polyvinyl chloride, and polystyrene as well as thermoset are usable as matrix (Summerscales et al., 2010; Ismail et al., 2013; Pickering et al., 2016).

The low tolerance to heat made the NFRPC less desirable. Hence, the enhancement of thermal properties of NFRPC is crucial and this can be achieved by adding NCC in the matrix. However, the properties of NCC requires chemical bleaching process which is mainly to remove the lignin. In the process, the pulps undergo several steps with different chemicals. Hence, if the bleaching process can be excluded, the production cost can be reduced and the application of chemical pollutants can be avoided. Nevertherless, to exclude bleaching means some amount of lignin in the NCC will remain. Studies have shown that the presence of lignin in NCC provides good thermal resistant property and improves the thermal degradation of NFRPC even further. The amount of lignin in wood for example kenaf, flax, pine straw, wheat straw, and alfafa ranges from 12% to 39% when the amount is determined based on Klason lignin analysis (Hatakeyama & Hatakeyama, 2009; Watkins et al., 2015). According to Rozman et al., (2000), lignin also has the potential to act as a compatibiliser since it contains both polar hydroxyl groups and non-polar hydrocarbon as wel as benzene rings. Thus, it is eligible to play a role as a compatibility enhancer between matrix and reinforcement and improves the mechanical properties of a composite. To determine these effects, the NCCs prepared in this study were subjected to different bleaching sequences to obtain different amount of lignin that were retained in the NCC. These NCCs were then added into the composites and its effect on the performance of resulting composites were evaluated.

1.3 Research Objectives

The aim of this study is to produce high mechanical and thermal stable NFRPC using kenaf core (KC) as the main source of natural fibre. The KC was used in two forms: micro-sized powder and NCC. The NCC was applied in PP/KC composites at different lignin percentage. The specific objectives of this study are:

- To characterise the NCC of different lignin content from KC
- To evaluate the effect of kenaf fibre loading PP composites as the control samples
- To evaluate the effect of NCC of different lignin contents addition on the physical, chemical, morphological and mechanical properties of PP/KC composites.
- To examine the thermal properties of PP/KC composites added with NCC of different lignin contents.
- To determine the effect of alkaline post treatment on NCC and the properties of PP/KC/MAPP nanocomposites.

1.4 Scope of Study

This research was divided into four sections: (i) preparation of NCC from KC, (ii) production of NCC of different lignin contents, (iii) evaluation of NCC loading effects on the properties of PP/KC composites, and (iv) the production of post alkaline treated NCC and its effect on PP/KC composites.

1.4.1 Development of NCC with Different Lignin Percentage from KC

In this section, the isolation and extraction processes were done upon KC as a main source of fibre to produce nanocellulose. KC was reported to have high hemicellulose (30%) and cellulose (45%) which makes it a suitable biochemical source of cellulose derivatives such as nanocellulose (Jonoobi et al., 2010). The KC was pulped using alkali treatment or pulping process and then bleached to produce different levels of lignin content. Then, the pulps were then chemically treated through acid hydrolysis process to produce NCC. Following this, the NCC were freeze dried prior to the nanocomposite production.

1.4.2 Production of PP/KC Composites Reinforced with NCC of Different Lignin Content

The optimisation of PP/KC formulation was determined by varying the KC loading by 10, 20, 30, and 40 wt %. The furnish samples were added with MAPP, a coupling agent that enhances the interfacial adhesion between the polymer matrix and natural fibres. Then, the optimum mixture was chosen according to its mechanical and thermal properties with the support of morphological tests. The sample with the optimum results was chosen to be added with different types of NCC filler. The amount of NCCs loaded in the PP/KC composites were set to be 0.5 and 1 wt %. The mixture of PP, KC with and without NCC were compounded via melt mixing process and hot pressed according to the desired mould, conditioned and tested.

1.4.3 Evaluation on the Effects of NCC Loading on PP/KC Composites

The effect of KC loading on its mechanical and physical properties were evaluated by the addition of different KC loading (10 % wt., 20% wt., 30% wt. and 40% wt.) with and without addition of MAPP. Then, the effect of PP/KC and different types of NCCs were evaluated via mechanical, physio-chemical, and thermal properties. The analysis on mechanical properties of the nanocomposites were proven by the morphological analysis done via SEM.

1.4.4 Production of Post Alkaline Treated NCC and its Effect on PP/KC Composites

In order to remove the sulphates by-products in NCC from acid hydrolysis process, the NCC suspension undergo a post alkaline treatment using a strong alkali. In this part, instead of rinsing the NCCs via centrifugation and dialysis using membrane cellulose, the NCCs were rinsed using NaOH until the pH is neutral. Following this, the NCC were freeze dried prior to the nanocomposite production. The treated NCC of different lignin content then incorporated in PP/KC/MAPP composites to observe the effect on its physical, chemical, mechanical and thermal properties.

1.5 Research Novelty

This research mainly focuses on the production and application of NCC of different lignin content that was prepared from KC and its effect on the properties of NFRPC. Lignin contains both polar hydroxyl groups and non-polar hydrocarbon and the benzene ring. It was believed can contribute to the enhancement of compatibility between the polymer matrix and fibre reinforcement (Rozman et al., 2000; Hatakeyama & Hatakeyama, 2010). The uniqueness of this study is the variation on lignin content in the NCC which were achieved by controlling the bleaching sequence. The effect of the lignin on the properties of composites with NCC was evaluated. This study also applied two sizes of filler: KC in the form of NCC and microsized powder which also known as bimodal filler system, where the smaller particles (NCC) help to fill the interstitial space between larger particles (microsized powder) to give an increase in packing density of the fillers resulting in the improvement of composites properties (Hong et al., 2011).

1.6 Summary

This chapter was initiated by overviewing the application of the NFRPC and its advantages over the conventional composites. The impact of nanocellulose as the new type of reinforcement was also highlighted in this chapter. However, there are some problems that needs to be countered which become the main focus of this study. The objective of this study was listed and elaborated in the scope of research section. Finally, the novelty of this study was stated through the explanation on its uniqueness over other studies.

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