UNIVERSITI PUTRA MALAYSIA

CELLULOSE NANOFIBER AS NUCLEATING AGENT AND REINFORCEMENT MATERIAL IN IMPROVING CRYSTALLIZATION AND MECHANICAL PROPERTIES OF POLYLACTIC ACID NANOCOMPOSITES

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By

SITI SHAZRA SHAZLEEN SHAMSUDIN

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Polylactic acid (PLA) is one of the most utilized biodegradable polymer to replace petroleum-based polymer. However, PLA has a slow crystallization rate which has a negative impact on its melt processing, hence limiting the application of PLA in industry. The reinforcement of cellulose nanofiber (CNF) within the PLA matrix can enhance the mechanical properties of nanocomposites, but the nucleation effect of CNF on the crystallization behavior, particularly the crystallization rate, remains unclear. In this study, PLA nanocomposites consisted of 1 – 6 wt% CNF (PLA/CNF1-PLA/CNF6) were prepared by melt blending method, and the crystallization kinetic behavior of the PLA and PLA/CNF nanocomposites were determined by DSC analysis. In the non-isothermal crystallization study, it was found that PLA/CNF3 exhibited the highest crystallization onset temperature and enthalpy among all the PLA/CNF nanocomposites. PLA/CNF3 also had the highest crystallinity of 44.2% with an almost 95% increment than neat PLA. The highest crystallization rate of 0.716 min^{-1} was achieved when PLA/CNF3 was isothermally melt crystallized at 100°C. The crystallization rate was 65-fold higher as compared to the neat PLA (0.011 min^{-1}). At CNF wt% higher than 3%, the crystallization rate reduced, suggesting the occurrence of agglomeration at higher CNF loading. PLA-*g*-MA was used as compatibilizer to improve interfacial adhesion between CNF and PLA. Results showed that the PLA-*g*-MA has some effect on nucleation, in which the crystallization half time for PLA-*g*-MA reduced to 33.2 min compared to neat PLA when isothermally melt crystallized at 100°C. Nevertheless, the presence of PLA-*g*-MA in PLA/PLA-*g*-MA/CNF3 nanocomposites did not improve the crystallization rate as compared to PLA/CNF3, indicating that the use of PLA-*g*-MA as compatibilizer may not be necessary in order to improve the crystallization kinetic of PLA nanocomposites. Tensile strength and Young’s modulus of the PLA/CNF nanocomposites increased with CNF incorporation up to 5 wt%, without the use of any compatibilizer. The highest tensile strength and Young’s modulus of 76.1 MPa and 3.3 GPa, respectively, were recorded at 4 wt% CNF.

These were higher than those of neat PLA (70.6 MPa and 2.9 GPa). This shows that CNF is an effective reinforcement material for PLA. When PLA-*g*-MA was used, the tensile strength was lower compared to the nanocomposite without compatibilizer. The tensile strength was found reduced with the increased amount of PLA-*g*-MA. On the other hand, Young's modulus increased drastically to 11 GPa in the presence of compatibilizer, suggesting the rigidity of the nanocomposites when PLA-*g*-MA was used. The combination of CNF nucleation and PLA-*g*-MA compatibilization did not influence the crystallite size of the PLA nanocomposites. The addition of CNF and PLA-*g*-MA into PLA also did not have much effect on the thermal stability, despite of slight reduction in thermal degradation temperature. It was evident that the presence of PLA-*g*-MA in PLA/CNF nanocomposites did not improve the crystallization kinetic and mechanical properties. These findings affirm the role of CNF as an effective nucleating agent and simultaneously act as a nano-reinforcement material in enhancing the crystallization and mechanical properties of PLA. Thus, it is possible to manufacture higher quality biodegradable nanocomposites that is eco-friendlier and more sustainable in the future.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia
sebagai memenuhi keperluan untuk Ijazah Sarjana Master Sains

NANOFIBER SELULOSA SEBAGAI BAHAN NUKLEASI DAN BAHAN PENGUAT UNTUK MEMPERBAIKI SIFAT PENGHABLURAN DAN MEKANIKAL NANOKOMPOSIT POLILAKTIK ASID

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Polilaktik asid (PLA) adalah salah satu polimer biodegradasi yang paling banyak digunakan untuk menggantikan polimer berasaskan petroleum. Walau bagaimanapun, PLA mempunyai kadar penghabluran perlahan yang memberi kesan negatif terhadap pemprosesan leburnya, sehingga menghadkan penggunaan PLA dalam industri. Pengukuhan selulosa nanofiber (CNF) dalam matriks PLA dapat meningkatkan sifat mekanikal nanokomposit tetapi kesan nukleasi CNF terhadap tingkah laku penghabluran, terutamanya kadar penghabluran, masih belum jelas. Dalam kajian ini, nanokomposit PLA terdiri dari 1 – 6% berat CNF (PLA/CNF1-PLA/CNF6) dibuat dengan kaedah pencampuran lebur, dan tingkah laku kinetik penghabluran nanokomposit PLA dan PLA/CNF ditentukan oleh analisis DSC. Dalam kajian penghabluran bukan isoterma, didapati bahawa PLA/CNF3 menunjukkan suhu permulaan dan entalpi penghabluran tertinggi di antara semua nanokomposit PLA/CNF. PLA/CNF3 juga mempunyai darjah kehabluran tertinggi 44.2% dengan kenaikan hampir 95% daripada PLA. Kadar penghabluran tertinggi 0.716 min^{-1} dicapai apabila PLA/CNF3 dicairkan secara isoterma pada suhu 100°C. Kadar penghabluran adalah 65 kali lebih tinggi berbanding dengan PLA (0.011 min^{-1}). Pada CNF berat % lebih tinggi daripada 3%, kadar penghabluran berkurang, menunjukkan berlakunya pengagregatan pada pemuatan CNF yang lebih tinggi. PLA-*g*-MA digunakan sebagai penserasi untuk meningkatkan lekatan antara muka antara CNF dan PLA. Hasil kajian menunjukkan bahawa PLA-*g*-MA berpengaruh pada nukleasi, di mana waktu penghabluran separuh masa untuk PLA-*g*-MA berkurang menjadi 33.2 min berbanding dengan PLA ketika dicairkan secara isoterma yang dihablur pada 100°C. Walaupun begitu, kehadiran PLA-*g*-MA dalam nanokomposit PLA/PLA-*g*-MA/CNF3 tidak meningkatkan kadar penghabluran berbanding dengan PLA/CNF3, yang menunjukkan bahawa penggunaan PLA-*g*-MA sebagai penserasi mungkin tidak diperlukan untuk meningkatkan kinetik penghabluran nanokomposit PLA. Kekuatan tegangan dan modulus Young dari nanokomposit PLA/CNF meningkat dengan penggabungan

CNF hingga 5% berat, tanpa penggunaan penserasi. Kekuatan tegangan tertinggi dan modulus Young masing-masing 76.1 MPa dan 3.3 GPa, dicatatkan pada CNF 4%. Ini lebih tinggi daripada PLA (70.8 MPa dan 2.9 GPa). Ini menunjukkan bahawa CNF adalah bahan penguat yang berkesan untuk PLA. Semasa PLA-*g*-MA digunakan, kekuatan tegangan lebih rendah berbanding dengan nanokomposit tanpa penserasi. Kekuatan tegangan didapati berkurang dengan peningkatan jumlah PLA-*g*-MA. Sebaliknya, modulus Young meningkat secara drastik menjadi 11 GPa dengan adanya penserasian, menunjukkan kekakuan nanokomposit ketika PLA-*g*-MA digunakan. Gabungan nukleasi CNF dan keserasian PLA-*g*-MA tidak mempengaruhi ukuran kristal nanokomposit PLA. Penambahan CNF dan PLA-*g*-MA ke dalam PLA juga tidak banyak mempengaruhi kestabilan terma, walaupun terdapat sedikit penurunan suhu degradasi termal. Telah terbukti bahawa kehadiran PLA-*g*-MA dalam nanokomposit PLA/CNF tidak meningkatkan sifat penghabluran kinetik dan mekanikal. Penemuan ini mengesahkan peranan CNF sebagai agen nukleasi yang berkesan dan sekaligus bertindak sebagai bahan penguat nano dalam meningkatkan penghabluran dan sifat mekanik PLA. Oleh itu, adalah mungkin untuk menghasilkan nanokomposit biodegradasi berkualiti tinggi yang lebih mesra alam dan lebih lestari di masa depan.

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

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I hereby confirm that:

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

BNC	Bacterial nanocellulose
CNC	Cellulose nanocrystal
CNF	Cellulose nanofiber
DBPO	Dibenzoyl peroxide
DSC	Differential scanning calorimetry
FESEM	Field emission scanning electron microscopy
MA	Maleic anhydride
NMR	Nuclear magnetic resonance
PLA	Polylactic acid
PLA-g-MA	Polylactic acid grafted maleic anhydride/ maleated polylactic acid
TGA	Thermogravimetric analyser
XRD	X-ray diffractometer

CHAPTER 1

INTRODUCTION

1.1 Overview

Today, most plastics are designed to be discarded once used, leading to the accumulation of single-use disposable plastic waste that ends up in landfills, dumps or in the open environment. Commonly, single-use plastics are used for packaging and carry bags. Plastics are mainly composed of polymers and other chemicals such as stabilizers, colourants and processing aids, in which the quantity and type will depend on the processing method and targeted applications (Siracusa *et al*., 2008). Plastics are generally made by either condensation polymerization or addition polymerization of petroleum-based polymers which results in the production of synthetic plastics that cannot be degraded (Tang *et al*., 2012). This has become a major concern since according to a research report on plastic waste inputs from land into the ocean by Jambeck *et al*. (2015), Malaysia has been ranked 8th among the top 10 countries with poorly managed plastics waste worldwide. With the country producing 0.94 million metric tons of plastic annually, it has become a major environmental waste issue. Therefore, considering the magnitude of this issue, researchers have been encouraged to develop biodegradable plastics from renewable resources to replace single-use plastics.

Biodegradable plastic refers to a biologically derived plastic and they can be decomposed by bacteria into biomass, carbon dioxide, and water without introducing toxicity to the environment (Luyt & Malik, 2019). Having a great potential to replace petroleum-based plastics, considerable effort has been made to develop bioplastic/biopolymer from biodegradable resources. Studied on this matter have led to the production of polylactic acid (PLA) which can be mass-produced through fermentation of agricultural sources into lactic acid. Even though PLA is an attractive biodegradable alternative to conventional petroleum-based polymers, it is a slowly crystallizing polymer and brittle compared to conventional plastics, therefore, limiting the usefulness of PLA as general use plastic significantly (Frone *et al*., 2013; Picard *et al*., 2011). For instance, it was found that the elongation at a break and the impact strength of PLA were lower than polypropylene (PP), polyethylene (PE), poly(ethylene terephthalate) PET and poly amide (PA) (Hamad *et al*., 2015). This limitation then led researchers to add a reinforcement material into PLA which can also act as a nucleating agent to improve the crystallization and mechanical properties of PLA. Several studies have been conducted to identify the efficient reinforcement material for PLA and graphene (Chen *et al*., 2013; Yang *et al*., 2019), talc (Cipriano *et al*., 2014; Fowlks & Narayan, 2010; Refaa *et al*., 2014) as well as natural fibers (Jia *et al*., 2014; Wu, 2009; Yu *et al*., 2014; Zhang *et al*., 2012) have been proven can act as nucleating agents to PLA, as they can enhance both mechanical and crystallization kinetics of PLA.

Recently, studies have focused on the use of cellulose nanofiber (CNF) to promote crystallization in PLA by acting as a heterogeneous crystal nucleating agent (Kose & Kondo, 2013). CNF can accelerate the process of crystallization by increasing the number of crystal nuclei and reducing the crystallization half-time (Cipriano *et al*., 2014). In fact, CNF has been utilized globally in industrial application owing to its outstanding properties; high flexibility, high in mechanical strength, high crystallinity, high aspect ratio which are advantageous to enhance the mechanical properties of polymers.

Ariffin *et al.* (2017) revealed that the addition of 3 wt% of CNF to PLA can increase both tensile strength and Young's modulus by 13 and 37% respectively compared to the neat PLA. The improvement in mechanical properties of the PLA/CNF could be related to the high crystallinity of CNF, as they also revealed that the addition of 3 wt% CNF was able to increase the crystallinity index of nanocomposite by 14% compared to the neat PLA. Similar finding were also reported by Jonoobi *et al*. (2010), Nguyen *et al*. (2019) and Norrrahim *et al*. (2018).

1.2. Problem Statements

Even though PLA has the advantage of being biodegradable and compostable, it has lower crystallization rate and crystallinity than the synthetic polymers, making its products especially produced from injection moulding become amorphous (Nanthananon *et al*., 2015). This creates technical issues in melt processing (e.g. extrusion and injection molding) where rapid crystallization is required as slow crystallization results in longer molding cycle time. Therefore, this indirectly contributes to low productivity and high energy consumption for the overall processing, hence becomes a major hurdle for large scale production (Suryanegara *et al*., 2011; Yang *et al*., 2019).

In regard to this matter, the addition of a nucleating agent is seen as one of the effective approaches to address embrittlement and reduce solidification time after thermal processing. Findings have revealed that CNF can be a great nucleating agent to PLA, as it was able to enhance the crystallization and mechanical properties of PLA (Suryanegara *et al*., 2009). However, it was also observed that the CNF tends to agglomerate at a certain amount notably at higher loading. Hence, the use of maleic anhydride (MA) was seen as one of the alternatives to improve the compatibility between PLA and CNF, and the effectiveness of MA has been proven for other thermoplastics/CNF composites such as polyethylene (PE) (Yasim-Anuar *et al*., 2018; 2020) and polypropylene (PP) (Peng *et al*., 2014).

To date, despite having numerous studies on CNF as a nucleating agent to PLA, the focus was mainly given on the roles of CNF in enhancing the mechanical properties of PLA. The interaction between its effect as nucleating agent and reinforcement material in PLA is unknown. Moreover, the correlation between nucleation and compatibilization effect of CNF and PLA-*g*-MA on the crystallization kinetics of PLA remains unclear. Hence, this study attempted to investigate the dual role of CNF as nucleating agent and reinforcement material in PLA. The function of PLA-*g*-MA as compatibilizer on the crystallization kinetics of PLA as well as the characteristics of PLA/CNF and PLA/PLA-*g*-MA/CNF nanocomposites is also clarified.

1.3. Objectives

The general objectives of this study were to determine the dual role of cellulose nanofiber as nucleating agent and reinforcement material for PLA, as well as to clarify the function of the PLA-*g*-MA in the crystallization kinetic of PLA.

The specific objectives of this study are as outlined below:

1. to investigate the effect of cellulose nanofiber on the crystallization kinetic of polylactic acid and determine its functionality as a nucleating agent to improve the crystallization rate of polylactic acid nanocomposite.
2. to determine the influence of maleated polylactic acid on the crystallization kinetic of polylactic acid/cellulose nanofiber nanocomposites.
3. to evaluate the mechanical characteristics of polylactic acid/cellulose nanofiber and polylactic acid/maleated polylactic acid/cellulose nanofiber nanocomposites.
4. to characterize the polylactic nanocomposites for their spherulite size, morphological, thermal and surface wettability properties.

1.4. Significance of the Study

This study provides an important opportunity to advance the understanding of the correlation between the amount of CNF in PLA that promotes its functionality as reinforcement material, and its effect on PLA nucleation for improving the crystallization rate. In the meantime, this particular study explores, for the first time the combination effects between CNF nucleation and PLA-*g*-MA compatibilization towards crystallization properties of PLA. Moreover, it is evident from the literature review that no research has been conducted on the combination effects of nucleation and compatibilization between CNF and PLA-g-MA and their correlation to improve the crystallization and mechanical properties of PLA.

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