CHARACTERIZATION OF FUSED DEPOSITION MODELLING PRINTED KENAF FIBRE REINFORCED POLY-LACTIC ACID COMPOSITES

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A thesis submitted
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DECLARATION

I declare that this thesis entitled “Characterization of Fused Deposition Modelling Printed Kenaf Fibre Reinforced Poly-Lactic Acid Composites” is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature : 

Name : AIDA HARYATI BINTI JAMADI

Date : 06/03/2023

APPROVAL

I hereby declare that I have read this thesis and in my opinion, this thesis is sufficient in terms of scope and quality for the award of the degree of Master of Science in Mechanical Engineering.

Signature : 

Supervisor Name : DR. NADLENE BINTI RAZALI

Date : 06/03/2023

DEDICATION

In the name of Allah SWT and to my beloved mother and father

ABSTRACT

Natural fibre as a reinforcing agent has been widely used in many industrial applications. However, the reinforcing agent devotes a better strength when embedded with a polymer matrix. Nevertheless, the characteristic of natural fibre and polymer matrix are in contrast, as natural fibre is hydrophilic, while polymer is hydrophobic in nature. Natural fibre is highly hydrophilic due to the presence of a hydroxyl group (-OH), while polymer matrix has an inherent hydrophobic characteristic which repels water. This issue has been fixed by modifying the natural fibre's surface using a chemical treatment combining an alkaline treatment and a silane coupling agent. This modifying process of natural fibre might reduce the attraction of water and moisture content and increase natural fibre surface roughness, which improves the interfacial bonding between these two phases. Fused Deposition Modelling (FDM) gets the most attention in development and manufacturing industries. The demand for FDM in industries increases gradually over time and attracts many researchers to enhance the quality of the FDM's fillers. To overcome the issue and replace the current fibres and achieve the bio-composites fibre, researchers suggested using natural fibre to replace the synthetic and carbon fibres as the reinforcement, which is also combined with bio-polymer matrix such as thermoplastics as the polymer matrix in FDM's industries. The effect of alkaline and silane treatment has been proven by performing the mechanical test, 1.0% silane treatment displayed better strength performance (57.846MPa) and tensile modulus (1.174GPa) as compared to other composites, which was proven by performing Scanning Electron Micrograph (SEM). The composites properties have been obtained by performing Differential Scanning Calorimetry (DSC), Thermogravimetric Analysis (TGA) and capillary rheological tests. By claiming 1.0% as the optimum silane concentration that perform better in mechanical properties, several factors need to be considered such as size and dimensional of fibre in binding. This factor has been studied by variying the size of natural fibre as the responding variables with a fix weight percentage of fillers. This modifying process of natural fibre might increase the dispersion of filler towards polymer matrix and increase the internal strength of composites, subsequently improving the interfacial bonding between these two phases. As the results tensile test indicateds that ≤100μm have hisghest strength (45.578MPa) with highest tensile modulus (1.096GPa) which also has been proven under SEM. The surface quality has been visualize by using 3D profilometer As a conclusion, by reducing the size of fillers, kenaf fibre composites could develop high strength performance in industry applications.

PENCIRIAN PENCETAKAN PEMODELAN PEMENDAPAN BERCANTUM KOMPOSIT ASID POLI LAKTIK DIPERKUAT GENTIAN KENAF

ABSTRAK

Serat asli sebagai agen penguat telah digunakan secara meluas dalam banyak industri pada era ini. Walau bagaimanapun, agen penguat memperincikan kekuatan yang lebih baik apabila dicampurkan dengan matriks polimer. Namun begitu, ciri gentian asli dan matriks polimer adalah berbeza, kerana gentian semula jadi adalah bersifat hidrofilik, manakala polimer bersifat hidrofobik. Serat semulajadi bersifat sangat hidrofilik kerana kehadiran kumpulan hidroksil (-OH), manakala matriks polimer mempunyai ciri hidrofobik yang melekat dan menolak air. Isu ini telah diselesaikan dengan mengubah suai permukaan gentian semula jadi menggunakan rawatan kimia yang menggabungkan rawatan alkali dan agen gandingan silane. Proses pengubahsuaian gentian semula jadi ini mungkin mengurangkan daya tarikan air dan kandungan lembapan dan meningkatkan kekasaran permukaan gentian semula jadi, yang meningkatkan ikatan antara muka antara dua fasa ini. Pemodelan Pemendapan Bercantum (FDM) mendapat perhatian ketara dalam pembangunan dan industri pembuatan. Permintaan untuk FDM dalam industri meningkat secara beransur-ansur dari semasa ke semasa dan menarik ramai penyelidik untuk meningkatkan kualiti pengisi FDM. Untuk mengatasi isu ini dengan mencapai pengisi biokomposit, penyelidik mencadangkan menggunakan gentian semula jadi untuk menggantikan gentian sintetik dan karbon sebagai tetulang, yang juga digabungkan dengan matriks bio-polimer seperti termoplastik sebagai matriks polimer dalam industri FDM. Kesan rawatan alkali dan silane telah dibuktikan dengan melakukan ujian mekanikal. Rawatan silane 1.0% menunjukkan prestasi kekuatan yang lebih baik dengan mencapai kekuatan tegangan sebanyak (57.846MPa) dan tegangan modulus (1.174GPa) berbanding dengan komposit lain, yang dibuktikan dengan melakukan Pengimbasan mikroskop elektron (SEM). Sifat komposit telah diperolehi dengan melakukan ujian Perbezaan Pengimbasan Kalorimetri (DSC), Analisis Termogravimetrik (TGA) dan kapilari reometer. Dengan mendakwa 1.0% sebagai kepekatan silan optimum yang berprestasi lebih baik dalam sifat mekanikal, beberapa faktor perlu dipertimbangkan seperti saiz dan dimensi pengisi dalam pengikatan. Faktor ini telah dikaji dengan mengubah saiz gentian semula jadi sebagai pembolehubah bergerak balas dengan peratusan berat yang tetap. Proses pengubahsuaian gentian asli ini mungkin meningkatkan isipadu pengisi ke dalam matriks polimer dan meningkatkan kekuatan komposit, dan meningkatkan ikatan antara dua fasa ini. Keputusan ketegangan kekuatan menunjukkan bahawa ia mempunyai daya kekuatan yang lebih tinggi (45.578MPa) ddengan kekuatan modulus (1.096GPa) yang telah dibuktikan di bawah SEM. Kualiti permukaan telah digambarkan dengan menggunakan profilometer 3D. Kesimpulannya, saiz gentian ≤100μm menunjukkan prestasi kekuatan yang lebih baik berbanding dengan komposit lain.

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LIST OF SYMBOLS AND ABBREVIATIONS

3D	-	Three dimensional
APS	-	Aminopropyltriethoxysilane
FFF	-	Fused Filament Fabrication
CNT	-	Carbon Nano Tube
ASTM	-	American Society for Testing and Materials
Pa	-	Pascal
N	-	Newton
mm	-	milimeter
cm	-	centimeter
wt%	-	Weight percent
˚C	-	Degree celcius
rpm	-	Revolutions per minute
µm	-	Micron meter
MPa	-	Mega Pascal
FDM	-	Fused Deposition Modeling
AM	-	Additive Manufacturing

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LIST OF PUBLICATIONS

Journal with Impact Factor

H. J. Aida, R. Nadlene, M.T. Mastura, L. Yusriah, D. Sivakumar and R. A. Ilyas (2021) 'Natural fibre filament for Fused Deposition Modelling (FDM): a review Natural fibre filament for Fused Deposition Modelling (FDM): a review', *International Journal of Sustainable Engineering. Taylor & Fran*cis, 00(00), pp. 1–21. doi: 10.1080/19397038.2021.1962426. (ISI indexed, Q2, IF= 2.30 (2021)

Aida Haryati Jamadi, Nadlene Razali, L. Michal Petr ˚u, Mastura Mohammad Taha, Noryani Muhammad and Rushdan Ahmad Ilyas (2021) 'Effect of Chemically Treated Kenaf Fibre on Mechanical and Thermal Properties of PLA Composites Prepared through Fused', *MDPI Polymer*, 13(19), p. 20. doi: https:// doi.org/10.3390/polym13193299. (ISI indexed, Q1, IF= 4.329 (2020)

Conference Proceedings

Noryani, M., Aida, H. J., Nadlene, R., Mastura, M. T., and Shaharuzaman, M. A. (2021). Materials Today : Proceedings Correlation study on physical properties and mechanical properties of kenaf fibre composites. *Materials Today: Proceedings,* (2020). https://doi.org/10.1016/j.matpr.2021.10.381

Aida H. J, R. Nadlene, M. T Mastura, M. Noryani, S. H. S. M. Fadzullah, A. A. Kamarolzaman, M. A. Salim and Bushra Rashid (2022). The impact of Alkaline Treatment On The Mechanical and Physical Properties of Kenaf Core Fibre Reinforced Vinyl Ester Composites. *International Journal of Nanoelectrics and Materials: Proceedings,* (2021).

CHAPTER 1

INTRODUCTION

1.1 Background

Nowadays, attention of engineers and professionals has been triggered regarding the increased consumption of petroleum and the depletion of these sources. In addition, the emission of harmful gas into the environment and the greenhouse effect during incineration produced an alternative in the development and sustainability of natural polymer composites (Fiore, Di Bella, and Valenza, 2015; Yucheng Liu, Lv et al., 2019). Aerospace, automotive, and construction industries have widely used advanced polymer composites, which contain carbon and glass fibre as the primary materials (Lee, Kim, Lee, Kim, Dorgan, 2009). It was found that these primary materials are hardly reusable and reutilised (Lee et al., 2009). Therefore, natural fibre has been introduced to replace the consumption of petroleum-based and synthetic fibres. Other than that, the characteristics between natural fibres and synthetic fibres are quite similar, such as low density, high stiffness, and good mechanical properties (Yucheng Liu, Lv et al., 2019). In comparison to the characteristics of other fibres such as synthetic, glass, and carbon, natural fibre (Van de Weyenberg et al., 2003) shows an advantage in biodegradability, renewability, non-toxicity, CO_2 neutral life cycle, degradablity, sustainability, and environmentally friendliness (Edeerozey, Akil, Azhar, and Ariffin, 2007; Lee et al., 2009; V. S. Sreenivasan, Ravindran, Manikandan, & Narayanasamy, 2012; Asim, Jawaid, Abdan, and Ishak, 2016; Fiore et al., 2015; Yucheng Liu, Xie et al., 2019; Oushabi et al., 2017; Sreenivasan, Sujith, & Rajesh, 2019).

In advanced applications, thermoplastic polymers are widely used, but due to their disadvantages which are lower in thermal stability and strength, some applications might not be applicable (Manral and Bajpai, 2020). One of the renewable and biodegradable base polymers in the polyester group is Polylactic Acid (PLA) (Huda et al., 2008; Asumani et al., 2012; Ghaffar, Madyan, Fan, and Corker, 2018; Mazzanti, Salzano de Luna, Pariante, Mollica, and Filippone, 2020), which emits less CO_2 gas and shows that this material is not harmful to the greenhouse, humans, and animals (Huda et al., 2008). Overall, the production of natural fibre is a new issue that has been introduced by many researchers. Natural fibre-reinforced polymer biocomposites using environmentally friendly Fused Deposition Modelling (FDM) technology has attracted many industries and researchers. The implementation of natural fibres in the filament of FDM to replace the current fillers has attracted many competitors and market platforms (Milosevic, Stoof, and Pickering, 2020). However, the use of a thermoplastics polymer as the main material for FDM is still not recommended. The important elements of a polymer are its mechanical properties, which are strength and stiffness. As previously stated, the mechanical aspects of many bio-based polymers have been investigated to enhance the technology of FDM. Acrylonitrile butadiene styrene (ABS) and polylactic acid (PLA) are popular because they are stable. The most frequent thermoplastic that had been produced in this technology is PLA. The advantages of using PLA are that it is recyclable, biodegradable and has a temperature of 145–160 ◦C (Petchwattana et el., 2019). The fibre loading optimisation and also the chemical treatment of the reinforcement can affect the mechanical properties of the product. Therefore, the natural fibre that combines with the PLA is firm and requires dried feedstock and storage (Mazzanti et al., 2020).

1.2 Problem Statement

The usage of natural fibre in industry is a way to replace glass and also carbon fibre which have many disadvantages such as the price is high and it is not environmentally friendly. In other words, carbon and glass fibre can donate to pollution. The reason natural fibre is emphasized in production because the price range of the fibre is low compare to the glass and carbon fibre, low in weight, environmentally-friendly, easy to decompose, easy to manufactured and the properties of strength quite impressive.

Hydrophilic means the tendency to absorb more water which specifically define as one of the disadvantages of using natural fibre. Major effect that can show from this problem such as wetting problem of the composites, the tendency of water absorption, instability of composites and also the changing of dimension. However, this issue can handle by modify the surface of the fibres by using pre-treatment process. Alkalization or known as mercerization is one of the famous pre-treatments that widely used onto the natural fibre. Many researchers claimed that alkaline treatment can enhance the bonding between fibre and polymer matrix. By using alkali solution and immersion of natural fibre with certain value of concentration and time, in can increase the aspect ratio and roughens the surface of natural fibre which can have better result in mechanical properties. After the alkaline treatment, the impurities on the fibre might been reduced. Specifically, by doing this treatment, the bonding between two phases can be enhanced instead the interfacial bonding can be improved. In addition with several pre-treatment process, by adding the silane coupling agent steps also can increase the enhancement bonding between fibre and polymer matrix (Xie et al., 2010).

Other critical issues of developing filament polymer composites are the uniformity of the mixing between fibre and polymers. From the literature reviews, the fibre size needs

to control in order to improve the filament dimension stability (Ferreira, Capela and Costa, 2010, Ramanaiah, Prasad and Chandra, 2013, Sapuan, Jawaid and Al-shuja, 2014, Pandian et al., 2014). It is important since the nozzle of FDM is sensitive on the diameter of the filament that can cause the clogged of the nozzle and effect the finishing of products. In natural fibre reinforced composites or known as NFRP, fillers such as short, particles nor nano fibre are widely used in way to improve the mechanical properties of printed product. Next, the mixture of fibres and matrix is more at ease when use the smaller size of fibre (Rahim, Abdullah, & Md Akil, 2019). It made the blend process become more easier and also save much time in fabricating the composites. So, many experimental used nano size, particles or powder state in producing the 3D printing filament for extrusion process.

In this study, fibres is uniquely treated with silane coupling agent. The novelty of using silane coupling agent treatment towards kenaf fibre and PLA polymer composite were explored in order to observe the mechanical and physical properties of kenaf fibres. Other than that, by achieve a good result towards kenaf composites, the effect of fibre sieze toward composites also has been studied to discover the strength and surface finishing of the data samples. The motivation of this research is to establish the development of degradable filament since the information through the research publication is very limited.

1.3 Research Objective

Kenaf fibre reinforced PLA composite's filament has been extruded regarding this study. Therefore, there are two objectives that need to consider in while the experiment is being operate.

The first is to evaluate the optimum concentration of of silane coupling agent with three different concentration (0.5 wt%, 1.0 wt% and 2.0 wt%) towards the mechanical,

thermal, rheological and physical properties of kenaf fibre reinforced PLA composites fabricated bu using FDM method. By refering the optimum of silane concentration obtained from first objective, the second objective to study the effect of varying fibre size (≤100µm, 100µm, 250µm and 300µm) towards mechanical properties and surface roughness.

1.4 Scope of Research

Two types of cleaning process that had been done in this experiment which are by using alkaline treatment with constant concentration of alkali solution is 6% at 24 hours in room temperature, followed by silane treatment with three different weight percentage. 0.5 wt%, 1.0 wt% and 2.0 wt% of silane was dissolve in 30 wt% of water and 70 wt% of ethanol and fibres were soaked at three hours in room temperature. The mechanical properties covered tensile test and flexural test to check the bonding between fibre and matrix, rheological test to check the viscosity of the composites and thermal test to check the glass temperature (T_g) of the composites in various types of environment. Next by obtaining the optimum concentration from the previous experiment, new samples have been subjected with fibre loading 2.5 wt% with 4 different size of fibre which are ≤100µm, 100µm, 250µm and 300µm.

Both experimental process used FDM method to produce the testing sample. The filament was extruded by using twin screw extruder with specific parameter machine extrusion. Data analysis then has been interpret to obtain the results for further discussion.

1.5 Contribution of Study

The contribution of these study is to develop a composites that can be commercialized and also environmental friendly. On the other hand, in way to produce an applicapable product, many factor need to consider.