International Journal of Current Science, Engineering & Technology
ISSN : 2581-4311
Original Research Article | Open Access | AMCT 2017 Malaysia | *Special Issue*

Effect of Supercritical Carbon Dioxide on Tensile Properties of Durian Skin Fibre Biocomposite

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ABSTRACT. The exploration of new biocomposites that are environmental friendly is gain attention due to depletion of petroleum resources and increasing global environmental concern. Therefore, the investigation of environmental friendly and sustainable materials requires to substitute the synthetic based materials. This project is conducted to investigate the effect of supercritical carbon dioxide on tensile properties of PLA/DSF biocomposite films. The PLA/DSF biocomposites are produced via solvent casting method. Then, the samples are treated under supercritical carbon dioxide ($SCCO_2$) at 40 °C and at 200 bar pressure. From tensile properties, it was found that the tensile strength and modulus for untreated and treated PLA/DSF biocomposite are higher compared to net PLA which are 24.9 MPa and 23.5 MPa. Thus, the addition of durian skin fiber improved the mechanical strength PLA polymer. But, the presence of foams and pore in treated PLA/DSF biocomposites decreased its tensile properties by 1.4 MPa less compared to untreated samples as confirmed by morphological observations under scanning electron microscope.

Keywords: *Durian skin fibre, Epoxidized palm oil, Tensile, Supercritical carbon dioxide;*

Received: 15.10.2017, Revised: 15.12.2017, Accepted: 30.02.2018, and Online: 20.03.2018;

DOI: *10.30967/ijcrset.1.S1.2018.197-202*

Selection and/or Peer-review under responsibility of Advanced Materials Characterization Techniques (AMCT 2017), Malaysia.

1. INTRODUCTION

Recently, due to unstable consumption of petroleum had gain an intensive awareness towards green environment in the society by performing an upmost strategy to produce biodegradable composites packaging materials as alternatives materials for the packaging industries. The environmental concern across the globe also are worrisome [1]. This problem can be seen throughout the average rate of wastage over the time. Department of Environment from Ministry of Natural Resources and Environment already testified the highest proportion of waste approximately 56% accumulated in Malaysia is conquered by wastage dumped into landfills to decompose rather than reused or recycled product. Thus, the growing environmental issues around the world encouraged the design of materials should compatible with environment. The introduction of the biobased product which are made from recycle resources can reproduce eco-friendly product that potentially compete with synthetic-based product [2]. Biodegradable polymer films packaging offer alternatives way to conventional packaging based on their great performance in biodegradability, compostability, processability and sustainability.

Polylactic acid (PLA) is the most generally used as biopolymer plastic due to its properties which are known as biodegradable polymer. It has great potential from an environmental point of view. Indirectly, PLA is most eligible as a candidate polymer for the advancement of biodegradable films according to it behaviour that can be readily cast into edible films via dissolving method. Despite these outstanding features, the brittle nature of PLA limits its application [1]. PLA polymer matrices has several drawbacks significantly in mechanical properties and cause unsatisfactory behaviour for the packaging application. The limitation of PLA however can be improved by the addition of reinforcement such as natural fiber [3]. Durian skin fiber (DSF) is chosen as natural fiber in this study where it have great properties in mechanical and also may reduce the material cost [4,5]. The improvement of biocomposite materials for food packaging applications by using PLA and DSF can be one of the alternatives to resolve the environmental issues. But the long period of experimental exposure of biocomposite film will affect the durability and brittleness of properties. Brittleness also affect the processability which can be modified with plasticizer. Plasticizer like epoxidized palm oil (EPO) was reported able to enhance the flexibility of polymer chain [6,7]. Natural-based plasticizer are more preferred which are categorized as non-tocixity and low migration rates [8].

The growing demand of packaging industries using natural-based additives as antimicrobial agent in food packaging applications has resulted through research exploration to improve the antimicrobial superiority on biocomposite plastic. Cinnamon essential oil (CEO) shows antimicrobial function and may assist as antimicrobial agents against food pathogen and microorganisms. It possesses a better performance in extending the shell life of preserved food and ensure security usage of food is covered [9,10]. Antimicrobial packaging system with the presence of antimicrobial properties has ability in maintaining flavour of food and reduce microorganism activities on perishable food. Furthermore, the supercritical carbon dioxide ($SCCO_2$) method are used due to many advantages in particle reduction and plasticization modification on polymer. The usage of carbon dioxide as a main solvent may offer several benefits from a safety, environmental, manufacturing and economical point of view [11]. According to Comin et al. [12], solvent of CO_2 is believed to acts as plasticizer and decrease the glass transition temperature.

To date, there is no study reported on $SCCO_2$ aided plasticized PLA biocomposite. Therefore, this study explores the effect of supercritical carbon dioxide ($SCCO_2$) on the tensile properties of PLA/DSF biocomposite.

2. MATERIALS AND METHODS

2.1 Materials. Polylactic acid (PLA) was purchased from NatureWorks®, China in pellets form. Durian skin waste was collected from night market in Gombak, Selangor. Epoxidized palm oil (EPO) was obtained from Advanced Oleochemical Technology Division (AOTD), Malaysian Palm Oil Board (MPOB) in Bangi, Selangor. Chloroform was bought from Merck and Cinnamon oil essential (CEO) was supplied by local supplier from Best Formula Company.

2.2 Samples Preparation. The PLA/DSF biocomposite were produced via solvent casting method. PLA and DSF were kept in an oven at temperature 70 ^{o}C for 24h day to remove moisture. The composition of biocomposite prepared is shown in Table 1.

Table 1 The amount of composition in PLA/DSF biocomposite

Sample	PLA (wt%)	DSF (wt%)	EPO (ml)	CEO (ml)	Chloroform (ml)
PLA	100 wt%	-	-	-	51.00
PLA/DSF/EPO/CEO	92 wt%	3 wt%	5 wt%	1 wt%	51.00

The solvent mixture was then stirred with strong agitation via magnetic stirrer on hot plate until it dissolved uniformly. Dried PLA/DSF biocomposite films were peeled off from mould after left for 24 h at room temperature. Preparation steps of PLA/DSF biocomposite films were shown in Fig. 1. Note that treated is referred to PLA/DSF biocomposite underwent $SCCO_2$ process. While untreated is referred to PLA/DSF biocomposite without $SCCO_2$ process.

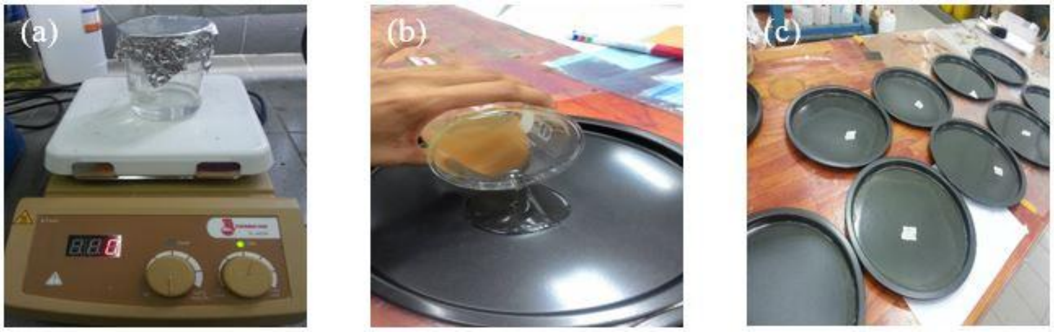

Fig. 1 The sample preparation of PLA/DSF biocomposite films (a) stirred process with magnetic agitation on hot plate; (b) poured the sample onto flat mould and (c) left the samples for drying process

2.3 Supercritical Carbon Dioxide Process. The PLA biocomposite was then underwent supercritical carbon dioxide process at temperature 40 °C at 200 bar for 2 hours. The CO_2 was charged into the biocomposite until the desired pressure was reached. The sample were treated through a supercritical carbon dioxide equipment with model Waters (TAP SFE Biobotanical system, IPM) at International Institute for Halal Research and Training (INHART), IIUM. The schematic diagram of supercritical carbon dioxide treatment system used in this study is exposed in Fig. 2. The tensile test was conducted based on ASTM D882 using Universal Testing Material, Shimadzu Autograph AGS-X series equipped with a 5kN load and 20 mm/min cross head speed. The biocomposites were cut into dimension according to ASTM D882. Anuar et al. (2016) reported that the thickness of sample at 0.1 ± 0.05 mm for tensile test was accepted. The surface and tensile fracture surface of PLA biocomposite was observed under scanning electron microscope (SEM) for morphological observation.

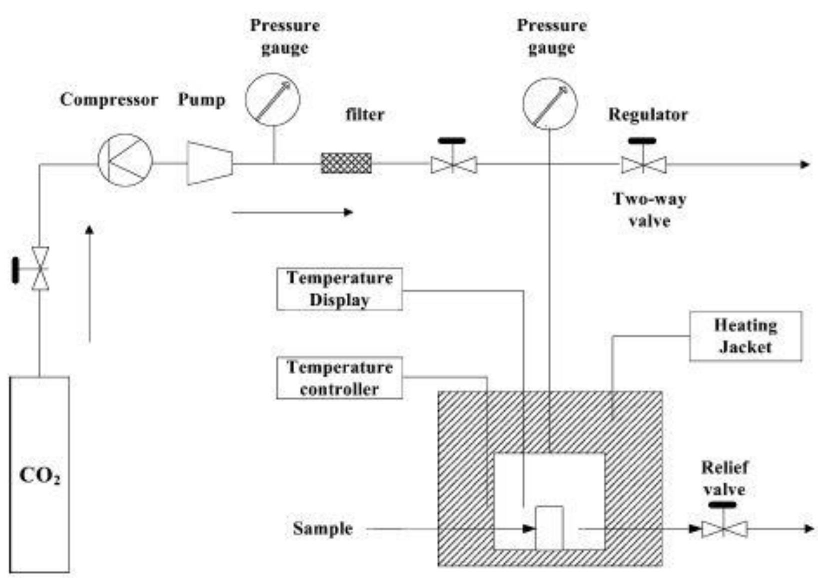

Fig. 2 The schematic diagram of supercritical carbon dioxide equipment [13]

3. RESULTS AND DISCUSSION

The effect of supercritical carbon dioxide treatment on tensile properties of PLA/DSF biocomposite was studied and the results were compared with PLA and PLA/DSF biocomposite without treatment. The tensile strength and tensile modulus of PLA, PLA/DSF/EPO/CEO biocomposite with and without $SCCO_2$ treatment are summarized in Fig. 3. It was found that the tensile strength for untreated PLA/DSF biocomposite and treated PLA/DSF biocomposite are higher than PLA by 2.57 and 1.2 MPa. The value attained for PLA was 22.3 MPa.

The tensile properties of PLA/DSF biocomposite was the highest due to strong interaction of interfacial bonding between PLA and DSF [3]. Tensile properties will only rise when the load transfer between matrix and fiber is sufficient [14]. However, the interaction between matrix and reinforcement still poor due to hydrophobicity behaviour of PLA and hydrophilicity of DSF [15]. Therefore, the presence of epoxidized palm oil (EPO) are believed to improve interfacial adhesion and brittleness of PLA/DSF bicomposites. The improvement of tensile strength of PLA biocomposite were also reported by Ali et al. [6], Anuar et al. [16] and Chieng et al. [17]. This is because the intermolecular forces are reduced and polymer chain interaction increased with the addition of EPO which are enhance flexibility and extensibility.

The tensile properties treated PLA/DSF biocomposite with $SCCO_2$ is slightly low compared to untreated biocomposite. The behaviour of CO_2 tend to expand and forming the pores in biocomposite polymer [18]. It can be seen in Fig. 4 the present of foam in the PLA biocomposite. From Fig 2(b) and (d), the treated PLA biocomposite seems to have pores size compared to untreated sample based on SEM micrograph on the surface of sample. Similar morphological structures were also studied by Biani et al. [11] and Nguyen and Baird [19] on other polymer-based composites systems. According to their findings, when supercritical carbon dioxide pressure was increased, the cell sizes also tend to increase. Meanwhile, cell density of composite was slightly decreased. The low density of foam can reduce the mechanical strength of sample. Lobos and Velankar et al. [20] revealed in their investigation, the foams strength of composites may be increased by reducing the expansion. The foamed expansion occurred due to depressurize rate during releasing of carbon dioxide gas in a rapid and non-constant technique after $SCCO_2$ process was conducted. Hence, these was the main factor affected the tensile properties by treated PLA/DSF biocomposite. It is evidenced by SEM analysis on tensile fracture surface in Fig. 4 (e) and (f), the samples treated PLA/DSF biocomposites appears to have more expansion of foam and pore size compared to untreated samples.

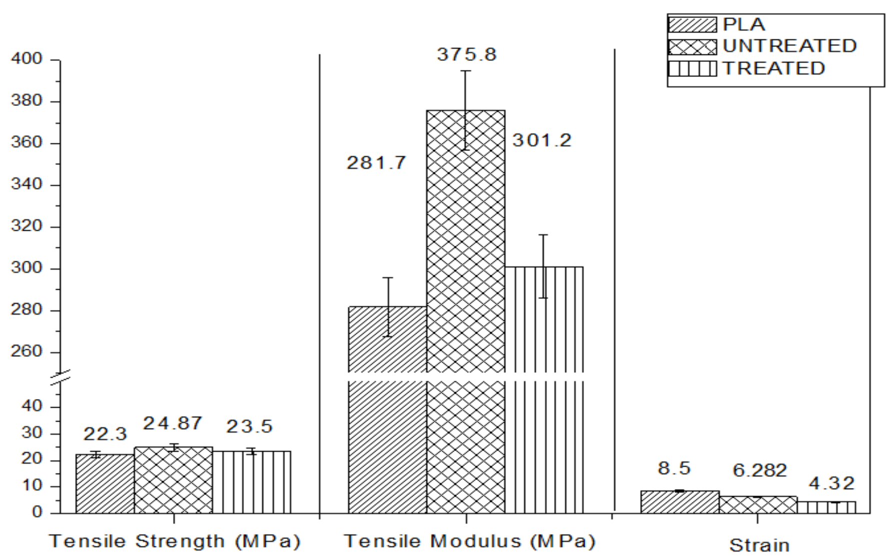

Fig. 3 Tensile strength, tensile modulus and strain for PLA, untreated PLA/DSF biocomposites and treated PLA/DSF biocomposite

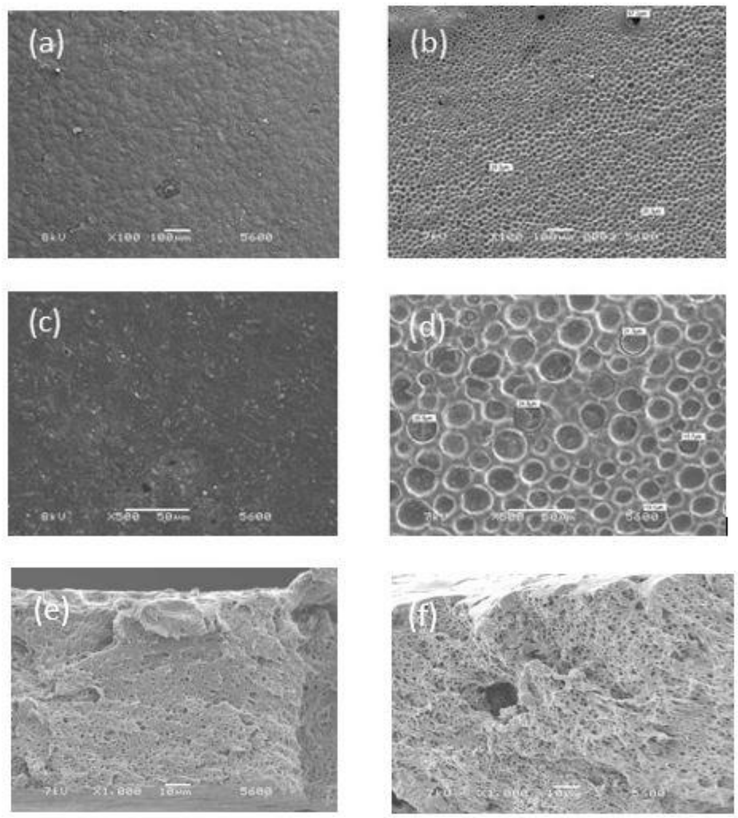

Fig. 4 SEM migrographs of (a) and (c) untreated PLA/DSF biocomposite; (b) and (d) treated PLA/DSF biocomposites on surface at 100x magnification and SEM micrograph of (e) untreated PLA/DSF biocomposite; (f) treated PLA/DSF biocomposite on tensile fracture surface at 1000x magnification

4. SUMMARY

As a conclusion, untreated and $SCCO_2$ treated PLA/DSF biocomposite are both potential for application in packaging industries based on tensile properties obtained. $SCCO_2$ treatment could represent an effective way to prepare PLA/DSF biocomposite which are characterized among high tensile properties with respect to food packaging applications.

ACKNOWLEDGEMENT

The authors wish to thank Ministry of Education Malaysia (MOE) for providing funding and financial support for this research via Fundamental Research Grant Scheme (FRGS16-003-0502) and International Islamic University Malaysia (IIUM) for the equipment and facilities provided in making this research a success.

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