

## Mechanical Properties and Water Absorption Behaviour of Durian Rind Cellulose Reinforced Poly(lactic acid) Biocomposites

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**Abstract**— Environmental concerns have resulted in replacing petrochemically derived polymer with biodegradable renewable resource. In this study, mechanical properties and water absorption behaviour of durian rind cellulose reinforced poly(lactic acid) biocomposites were investigated. Poly(lactic acid) was mixed with 25 and 35 wt. % of durian rind cellulose that was derived from durian consumption wastes. The biocomposites were melt-blended at 165 and 175 °C with 15 min using a Brabender internal mixer followed by a hot compression moulding technique. The results showed that impact strength and modulus of Young increased with increasing of cellulose content but decreased at higher mixing temperature. Water absorption behaviour of biocomposites as function of days was also investigated. It was found that the water absorption amount of biocomposites increased with increasing of cellulose content and exposure time.

**Keywords**— Poly(lactic acid); durian rind; cellulose; mechanical properties; water absorption.

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### I. INTRODUCTION

Over decades, the most polymers used are produced from non-biodegradable petrochemically based products [1]. These polymers exhibit very long time of environment decomposition, which represent a serious problem with long life of plastic waste, subsequently causing increases volume of commercial and industrial dumps in the landfills [2]. Moreover, the petroleum shortage is a real threat throughout the world. Considering these two problems, many countries are trying to replace the commodity synthetic polymer with non-petroleum resources called biodegradable polymer which is readily susceptible to microbial action. Biodegradable polymers are generally produced from renewable natural and abundant biodegradable polymeric materials such as polysaccharides, proteins, lipids, or combination of these components [3]. Cellulose, as one of the most abundant polymers, is renewable and natural and has been increased attention as reinforcing filler in biocomposites materials. Natural fibers, such as kenaf, ramie, flax, jute, and bamboo have been studied to reinforce biodegradable polymers to produce biocomposite materials [4]. The source of fiber is not only from plant but also agricultural waste such as peel, husk, and hull. Generally, these wastes are burnt or sent to landfills which cause

negative impacts in terms of environmental concern due to the increasing amount of pollutant disposal, and in terms of industrial sustainability due to the high costs related to their management [5]. Currently, more and more researchers are developing fully biodegradable composites which are composed of natural fibers and natural matrices or synthetic biodegradable matrix. Composites are usually fabricated with biodegradable polymers as matrix phase and natural fibers as enhancement phase. Poly(lactic acid) (PLA) is one of most commonly used as matrix phase of composites [6].

Poly(lactic acid) (PLA) is a linear polyester produced from renewable resources primarily corn starch and sugar canes [3]. The monomer, lactic acid is a compound that plays a significant role in several biochemical processes. It is mainly prepared by the bacterial fermentation of carbohydrates. The promising characteristics of PLA are good mechanical property, thermal stability, process ability, and low environment impact by being fully biodegradable [2]. Various studies of PLA reinforced with natural fiber have been reported [7-15]. Although a variety of natural fibers like kenaf, hemp, sisal, jute, abaca, wood etc. were investigated in detail; the use of durian rind cellulose as reinforcing material in PLA has not been widely explored yet.

Durian (*Durio zibethinus* Murray) is the most popular fruit in Southeast Asia, particularly in Thailand, Malaysia,

Indonesia, and Philippines [16]. It is well known as the “king of fruit”. However, only one-third of durian is edible, whereas the seeds (20–25%) and the shell are usually discarded [17] and become the cause of environment pollution. Durian rind, a byproduct of durian fruit consumption, provides one such good source for cellulose [18].

Generally, cellulose is poor resistance to water absorption due to hygroscopic characteristic. Both mechanical properties and dimensional stability of biocomposites will be subsequently deteriorated [6, 19]. In long-term, embrittlement linked to the degradation of the macromolecular skeleton by hydrolysis [6]. It can be said that water absorption is one of the most important physical characteristics of cellulose reinforced polymer composites exposed to environmental conditions that determine their end use applications. Therefore, the effect of cellulose content at different mixing temperatures on mechanical properties and water absorption behaviour of biocomposites based on PLA and durian rind cellulose was investigated in this study.

## II. MATERIALS AND METHOD

### A. Materials Preparation

Poly(lactic acid) (PLA) resin (Ingeo biopolymer 2003D food packaging-grade) was purchased from Natureworks® (USA). Durian rind was collected from Phatthalung Province, Thailand. Reagent-grade sodium hydroxide (NaOH) and acetic acid (CH<sub>3</sub>COOH), and technical-grade sodium chlorite (NaClO<sub>2</sub>) of 80% purity were purchased from Fisher Chemicals Sdn. Bhd. (Malaysia).

### B. Preparation of Cellulose from Durian Rind

Durian rind was first sun-dried and was then ground as in Fig. 1a and 1b. Cellulose was prepared using a two-step process. The first step is the preparation of holocellulose by chlorination method (ASTM D1104). Then, the cellulose was further treated by mercerisation method (ASTM D1103) in order to activate the hydroxyl groups of the cellulose. The detail of the procedure was reported elsewhere [4]. Finally, cellulose was then dried overnight at 80 °C. The cellulose as Fig. 1c was kept in an air tight container at room temperature until analysis.

### C. Preparation of Biocomposites

The cellulose was ground using grinder and then passed through a sieve (Retsch, AS 200 digit, Germany). The cellulose of size between 250 to 125 μm was collected. The PLA was mixed with two different weight fractions of cellulose; 25 and 35 wt. % using an internal mixer (Brabender Plastograph EC, Germany) for 15 min at a 50 rpm screw speed with two different mixing temperatures; 165 and 175 °C. Before further step, the biocomposites were dried at 80 °C for 16 h to avoid pores' formation. Next, the moulded sheets were then prepared using a hot press machine at 160 °C. This process involved 5 min of preheating and 5 min of pressing, followed by 2 min of cooling for tensile testing specimens with 1-mm thickness. Eight minutes of preheating, 7 min of pressing, and 3 min of cooling were used to produce samples for impact testing

with 3-mm thicknesses. All biocomposites were packaged in air tight containers and stored at room temperature until analysis.

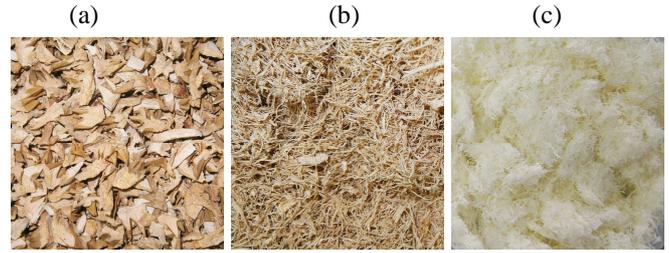


Fig. 1 Macroscopic image of (a) rind, (b) ground durian rind, and (c) cellulose [20]

### D. Characterization

1) *Morphological Analysis*: The SEM [S-3400N, Hitachi, Japan] images were taken to elucidate the morphologies of the untreated durian rind and cellulose. Samples were mounted on aluminum studs and coated with gold in a vacuum before being observed using a SEM.

2) *Mechanical Testing*: The modulus of Young was determined using an Instron Universal Testing Machine (Model 5566; USA) at a cross speed of 5 mm/min on specimens with dimensions of 12.7 mm × 63 mm × 1 mm according to ASTM 1882L, until tensile failure was detected. Five samples of each biocomposites were tested. The impact test was performed using an Impact Pendulum Tester (Ceast Model 9050) on 2.5-mm notched rectangular specimens with dimensions of 12.7 mm × 63 mm × 3 mm according to ASTM D256. The Izod method was conducted with a 0.5-J hammer. Seven samples of each biocomposite were tested.

3) *Water Absorption Behaviour*: In order to measure water absorption, the rectangular specimens having dimensions of 30 mm × 12.7 mm × 3 mm were prepared. The specimens were dried in an oven at 80 °C for 24 h, cooled in a desiccator to a constant weight, and immediately weighed. The water absorption tests were carried out by immersing the specimens in distilled water at room temperature from 1 to 50 days (12,000 hours). At regular time intervals, each specimen then was removed from the water immersion and wiped with tissue paper to remove surface water and subsequently weighed by electronic balance.

The water absorption was calculated and reported as the percentage increase of the initial weight as eq. (1) [21]

$$Mt (\%) = [(Wt - Wo)/Wo] \times 100 \quad (1)$$

where Wt is the weight of sample at time and Wo is the initial weight of sample. All data from five repeated tests were averaged.

The analysis of diffusion mechanism and kinetics was performed based on Fick's theory and fitting the experimental values: [19]

$$\log(Mt/M\alpha) = \log(k) + n \log(t) \quad (2)$$

where  $M_t$  is the water absorption at time  $t$ ; and  $M_\infty$  is the water absorption at the saturation point. The coefficient  $k$  and  $n$  are constants that are calculated from slope and intercept of the log plot of  $(M_t/M_\infty)$  versus time ( $h$ ), which can be drawn from experimental data.

At early stages and small times (typically  $M_t/M_\infty \leq 0.5$ ), the diffusion process is presented as follows [22]:

$$M_t/M_\infty = 4(Dt/\pi L^2)^{1/2} \quad (3)$$

where  $L$  is the thickness of the specimen and  $D$  is the diffusion coefficient.

### III. RESULTS AND DISCUSSION

#### A. Morphological Analysis

Fig. 2 compares the micrographs of untreated durian rind and its cellulose. The micrograph of untreated durian rind shows the amount of non-cellulosic components; pectin, lignin and hemicellulose scattered over the surface, which provide the bigger diameter than cellulose. These components were then removed after delignification and alkali treatment [23].

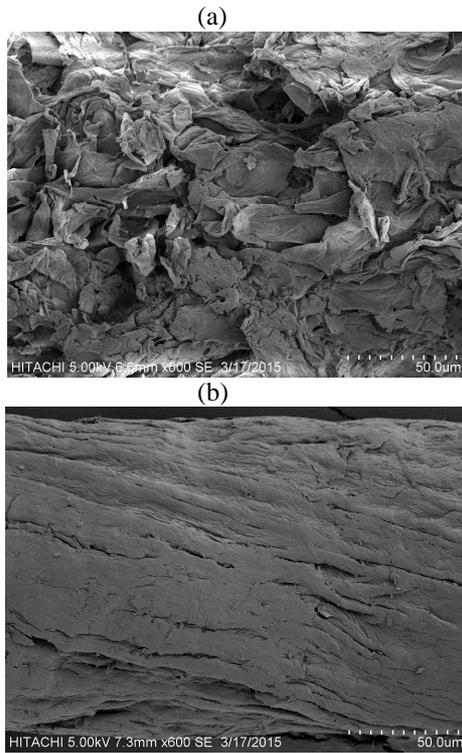


Fig. 2 Scanning electron micrograph of (a) untreated durian rind and (b) cellulose

The alkali solution causes fiber cell wall to swell and become round, then increasing the tensile strength and absorbency [24]. In addition, the removal non-cellulosic components provide crystalline cellulose which improve its density and also increase the strength due to regulated and dense molecular structure [25]. The important consequence of diameter reduction was higher reinforcing ability of the cellulose for composite application because the increasing of aspect ratio ( $L/d$ ,  $L$  is length and  $d$  is diameter) [25] which is

the major factor affecting on the mechanical properties of the composites for short fiber-reinforced polymers [26]. In our previous paper, the aspect ratio of cellulose from durian rind was investigated and found that the most particles displayed aspect ratio in the range of 20- 25 [27]. Generally, the minimum aspect ratio for good strength transmission for any reinforcement has to be superior to 10 [28-30], therefore, the cellulose from durian rind used for this present research has efficiency to be reinforcement material for composites due to its aspect ratio was superior to minimum value.

#### B. Mechanical Properties

Fig. 3 presents the modulus of Young as a function of cellulose content and mixing temperature. It was observed that modulus of Young for the biocomposites increased with increasing cellulose content. This indicated that biocomposites become stiffer at higher cellulose content. Generally, natural fibers have a higher modulus of Young thereby contributing to higher stiffness of the composites [9, 31]. A similar trend in mechanical properties with Nam et al. [32] which reported for modulus of jute fiber reinforced poly(butylene succinate). There is no significantly effect from mixing temperature on modulus of Young of biocomposites. Way et al. [33] reported that with addition of fiber, the modulus of Young of biocomposites will be increased due to modulus of fiber is much higher than that of PLA matrix. In addition, the results showed slightly decreased with higher mixing temperature for both different cellulose contents.

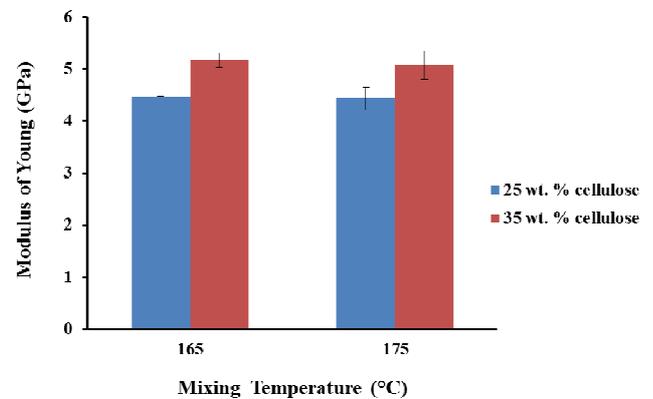


Fig. 3 Effect of cellulose content and mixing temperature on modulus of Young

The overall material toughness can be indicated by impact strength. Impact strength of fiber reinforced polymer is governed by interaction between fiber and matrix. When the composites undergo a sudden force, the impact energy is dissipated by the combination of fiber pull outs, fiber fracture and matrix deformation. Normally, impact strength of the composites increases with adding of fiber due to the increase in fiber pull out and fiber breakage [34]. Fig. 4 presents the impact strength of biocomposites as a function of cellulose content and mixing temperature. It was found that impact strength increased with increasing of cellulose content. The massive increase in impact performance for all man-made cellulose composites is due to smaller diameter and smoother surface, which affects the fiber/matrix

interaction and therefore allowing for pull-out to occur [9, 10] The cellulose used in this study was extracted to remove non-cellulosic components scattered over the surface and resulted in a decreased diameter and smoother surface as Fig. 1(a). Supposedly, for this reason, the path length of the propagated crack is enlarged, increasing the amount of energy needed to break the sample [10].

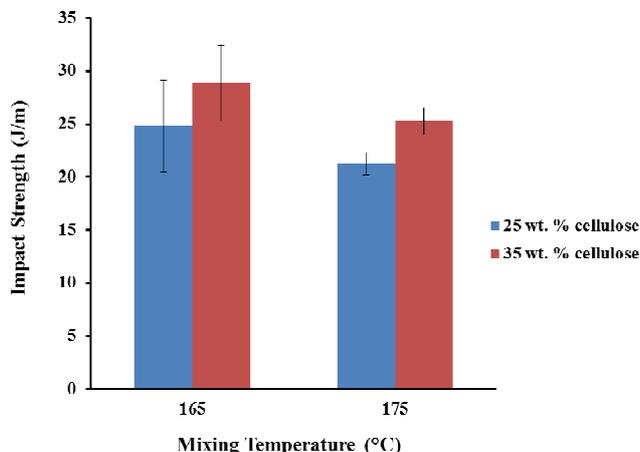


Fig. 4 Effect of cellulose content and mixing temperature on impact strength

Although natural fibers have been used as reinforcement material in PLA to improve mechanical properties and to reduce its brittleness [4] which is generated by hydrogen bond between hydroxyl groups in fiber and carbonyl groups in PLA [4], however, some research papers reported that with high cellulose loading resulting in lower mechanical properties if compare to neat PLA. Ochi [7] studied on mechanical properties of kenaf/PLA composites and found that the fiber content used for fabricating kenaf fiber-reinforced biocomposites should be kept less than 70 wt.%. In addition, Tawakkal et al. [4] reported that adding of 30 wt.% kenaf derived cellulose (KDC) to the PLA matrix provided the highest impact strength and enhanced 15% compared to the neat PLA. However, the adding of 40 wt.% KDC caused the reduction of 60% impact strength and also found that KDC loading 40 to 60 wt.% to the PLA matrix slightly decreased the strength of composites up to 8.4%. This may be affected by lack of good adhesion at the interface region of the fibers and the matrix [35 and 36].

Dhakal et al. [37] explained that when fibers are added to polymer matrix, there is always a chance of fiber entanglement randomly oriented fiber reinforced composites due to their irregularly shaped. This fiber entanglement can contribute to the formation of voids and porosity which act as stress concentrators leading to failure of composite samples. They also confirmed that the fiber volume fraction of hemp reinforced composite sample increases the void content also increases then resulting in lower mechanical properties.

### C. Water absorption

Generally, there are three mechanisms of diffusion of water in polymeric composites [37]; the first is diffusion of water molecules inside the micro gaps between polymer chains. The second is capillary transport into the gaps and flaws at the interfaces between fiber and the polymer matrix. The third is transport of microcracks in the matrix arising from the swelling of fibres especially in the case of natural fiber composites. Two types of water absorbed in polymers called free water and bound water as Fig. 5 [38]; free water is the water molecules which are relatively free to travel through the microvoids and pores, meanwhile bound water is the water molecules are dispersed in the polymer matrix and attached to the polar groups of the polymer [39].

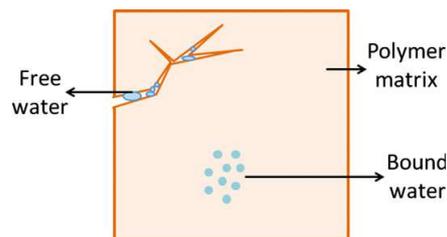


Fig. 5 Water absorbed in polymer matrix [38]

Fig. 6 shows the water absorption of biocomposites at two different cellulose contents and mixing temperatures as a percentage of dry weight after continuous immersion in water for 50 days. It was found that the water absorption amount of the composites increased with increasing the cellulose content. The first 400 hours (16 days) of soaking presented a rapid increase the level of water absorption then demonstrated a gradual increase in the water uptake then the composites finally reached an equilibrium point, which is no more water uptake into biocomposites and the weight of biocomposites remains constant [19]. This means that with increasing cellulose content, the number of hydroxyl (OH) group in the composites increased resulting in increasing the water absorption [4, 31] due to these free OH groups come in contact with water and form hydrogen bonding, which results in weight gain in the biocomposites. Also the biocomposites with lower cellulose content reach the equilibrium moisture content more quickly [40].

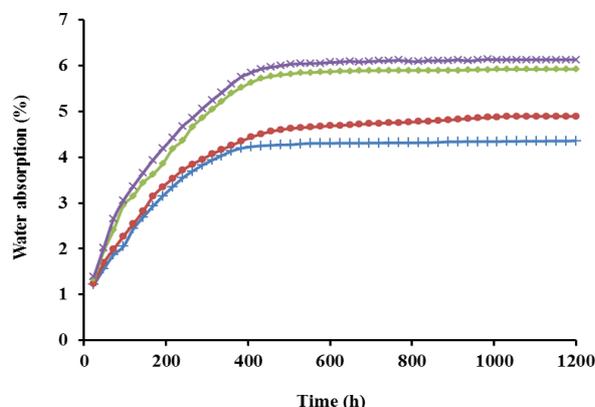


Fig. 6 Effect of cellulose content and mixing temperature on water absorption: 25% cellulose 165°C (blue line); 25% cellulose 175°C (red line); 35% cellulose 165°C (green line) and 35% cellulose 175°C (purple line)

The result showed a similar trend in PLA/kenaf-derived cellulose composites [4], recycled thermoplastics reinforced with *Pinus radiata* sawdust [40]. Further increase mixing temperature from 165°C to 175 °C slightly increased the water absorption. This can be explained that at higher mixing temperature affecting thermal degradation of cellulose and then occur of the gaps between cellulose and polymer which are cause of increasing of penetration of water into composites [6]. However, the results suggest that the higher temperature did not significantly contribute to absorption behaviour.

Fig. 7 shows the condition of moisture in a polymer matrix. When natural fiber composites are exposed to wet condition, water molecules penetrate into composites through micro-cracks then affecting adhesion between fiber and matrix. This causes swelling of the fibers, which may generate micro-cracks in the matrix and may eventually lead to debonding of fiber - polymer matrix [38]. A schematic illustration of this process is presented in Fig. 7.

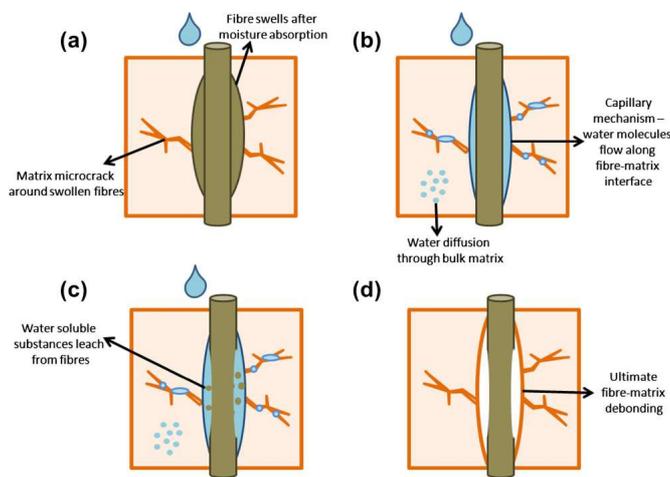


Fig. 7 Effect of water on fiber–matrix interface [38]

In general, there are three known mechanisms for water transport in polymer composites which are: Fickian diffusion, relaxation controlled, and non-Fickian or anomalous. The dominant mechanism depends on factors such as chemical structure of the polymer, dimensions and morphology of the wood filler and polymer–filler interfacial adhesion. In general, there are three known mechanisms for water transport in polymer composites which are: Fickian diffusion, relaxation controlled, and non-Fickian or anomalous. The dominant mechanism depends on factors such as chemical structure of the polymer, dimensions and morphology of the wood filler and polymer–filler interfacial adhesion [22 and 41].

The value of  $n$  is different for the cases as follows: in Fickian diffusion  $n = 0.5$ , relaxation  $n > 0.5$ , and anomalous transport  $0.5 < n < 1$  [22 and 41].

The fitting of the experimental data is given in Fig. 8 and the values of  $k$  and  $n$  resulting from the fitting of all formulations are shown in Table 1.

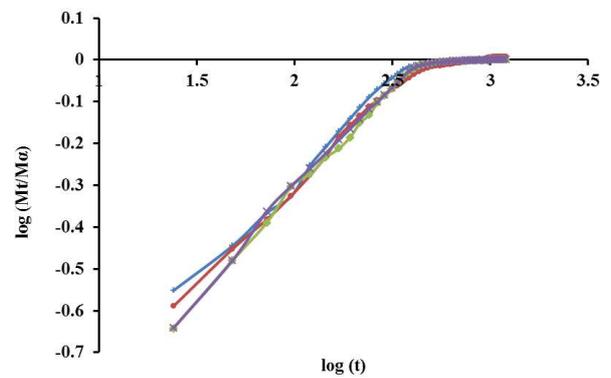


Fig. 8. Diffusion curve fitting for biocomposites: 25% cellulose 165°C (blue line); 25% cellulose 175°C (red line); 35% cellulose 165°C (green line) and 35% cellulose 175°C (purple line)

TABLE I  
DIFFUSION CURVE FITTING PARAMETERS

Sample	Water absorption	
	N	k (h <sup>2</sup> )
25% cellulose 165°C	0.48	0.0578
25% cellulose 175°C	0.47	0.0571
35% cellulose 165°C	0.50	0.0472
35% cellulose 175°C	0.50	0.0488

The result shows that  $n$  values are similar for all formulations and close to the value of  $n$  close to 0.5. Therefore, it can be concluded that the water and moisture absorption of all formulations approach the Fickian diffusion case [6].

TABLE II  
MAXIMUM WATER ABSORPTION AND WATER DIFFUSION COEFFICIENTS

Sample	Maximum water absorption (%)	Water Diffusion Coefficient ( $\times 10^{-12} \text{m}^2 \text{s}^{-1}$ )
25% cellulose 165°C	4.35	1.03
25% cellulose 175°C	4.80	1.15
35% cellulose 165°C	5.91	1.27
35% cellulose 175°C	6.12	1.27

The diffusion coefficient is the most important parameter of the Fick's model and shows the ability of water molecules to penetrate inside the composite structures.

Table 2 shows the water diffusion coefficients for all formulations. It is evident that the water diffusion coefficients increase with cellulose biocomposites. The similar trends were observed in wood flour prepared from saw dust reinforced recycled plastics (HDPE and PP) [6] and recycled cellulose fiber reinforced epoxy composites [22].

Although natural fiber provide many advantages in composites; low density, harmless, high toughness, renewable and biodegradable [42], however, a major limitation of natural fiber is difference of surface between the fiber and the matrix due to the fiber surface consists of high polar of hydroxyl but the matrix has high hydrophilic behaviour the resulting in poor of adhesion between the two materials [35 and 36]. As mentioned above, one of the common mechanisms of water penetration into composite materials is capillary transport into the gaps and flaws at the

interfaces between fibers and polymer matrix because of incomplete wettability and impregnation [37]. Thus, the higher water absorption and diffusion coefficients of higher cellulose content have caused more gaps and flaws at the interfaces between fiber and matrix.

#### IV. CONCLUSIONS

The biocomposites showed that increasing of cellulose content resulting in higher young of Modulus and impact strength that can be conclude that cellulose can act as reinforced material to improve mechanical properties of biocomposite. The water absorption of higher cellulose content is generally higher than lower cellulose content which will be affecting mechanical properties weaken during usage. Increasing in mixing temperature did not significantly contribute to mechanical properties and water absorption behavior. As a conclusion it seems necessary to use coupling agent to improve quality of adhesion between cellulose and PLA to reduce the gaps.

#### ACKNOWLEDGMENT

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