

MECHANICAL AND PHYSICAL PROPERTIES OF KENAF-DERIVED CELLULOSE (KDC)-FILLED POLYLACTIC ACID (PLA) COMPOSITES

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Kenaf-derived cellulose (KDC)-filled poly(lactic acid) (PLA) composites were prepared via melt blending and compression molding to improve the properties of PLA by introducing a natural cellulose that was chemically derived (chlorination and mercerization processes) from plant-based kenaf bast fibers. The effect of KDC content (0-60 wt.%) on the tensile elongation at the break point and during flexural and impact testing and on the water absorption and density of the composites was investigated, while the neat PLA polymer (without the addition of cellulose) served as a reference for the composites. The elongation at the break point of the composites was 9% on average, making it less elastic than the neat PLA. The flexural strength and modulus also increased by 36% and 54%, respectively. The impact strength of the composites was improved at KDC contents below 40 wt.%, but the impact strength was reduced above 40 wt.%. The composite containing the highest amount of KDC (60 wt.%) was denser than the neat PLA and had a water uptake of approximately 12%, which is notably low for a biocomposite system.

Keywords: Polylactic acid; Kenaf; Derived cellulose; Composite; Mechanical properties

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INTRODUCTION

The use of lignocellulosic fibers from plants to develop new composite materials has a low impact on the environment because of the materials' biodegradability. Specifically, plant fibers are carbon-neutral because they bind as much carbon dioxide during growth as is emitted during combustion (Mohanty et al. 2005). Moreover, natural fibers have a low density, low equipment abrasion, a relatively high specific strength and stiffness, low cost, can be easily surface modified, and are renewable (Wambua et al. 2003; Mohanty et al. 2005). Thus, these fibers have generated increasing interest as reinforcing fillers in composite materials because, although they are not as strong as glass fiber-reinforced composites, their properties are suitable for such applications as packaging, medicine, furniture, and automotive components.

Natural fibers, such as kenaf, ramie, flax, jute, and bamboo, have been used to

reinforce biodegradable polymers to produce biocomposite materials (Canigueral et al. 2009; Yu et al. 2010; Okubo et al. 2009).

Cellulose is the major component in lignocellulosic fibers. The cellulose can be obtained from pure cellulose fibers (e.g., cotton, which contains 98-99% cellulose) or can be derived from other lignocellulosic materials. For instance, α -cellulose is widely used as a starting material for the production of other valuable products, such as rayon, microcrystalline cellulose (MCC), carboxymethyl cellulose (CMC), and cellulose derivatives (Sixta 2006). This material can be derived from natural fibers by mercerization or alkali treatment (Soom et al. 2006; Goda et al. 2006). Mercerization is a chemical process that subjects natural fibers to a relatively strong basic aqueous solution to induce sufficient swelling to removing waxy materials, lignin, hemicelluloses, and impurities (Goda et al. 2006). Recently, α -cellulose has been derived from different natural fiber sources, namely, kenaf fibers (Khalid et al. 2008) and empty oil-palm fruit-bunch fibers (Tajeddin et al. 2009) to improve the stiffness and strength of different polymeric matrices.

In Malaysia, the government has identified kenaf (*Hibiscus cannabinus* L.) as a potential crop replacement for tobacco. From 2005 to 2011, the kenaf planting area expanded from 42.5 to 400 hectares (Anonymous 2011). Kenaf fibers (core and bast) have been used to make biocomposites for the building industry, car components, furniture, and food packaging (Saraswati and Mahanum 2008). Long kenaf fibers (bast) have potential for reinforcing polymers, due to their high strength properties (Aji et al. 2009). Kenaf bast fibers have a higher content of cellulose, ranging up to 69.2%, compared to the core fiber and the other abundantly natural fibers in Malaysia, such as empty oil-palm fruit-bunch fibers (Akil et al. 2011; Khalid et al. 2008).

PLA is a well-known biodegradable polymer used for making packaging materials and has comparable mechanical properties to many other petroleum-based polymers, such as polyethylene (PE), polystyrene (PS), polypropylene (PP), and polyethylene terephthalate (PET) (Auras et al. 2004). Additionally, PLA has several advantages, such as its biodegradability, its ability to be sealed at low temperatures, its low gas emission, and the renewability of its raw materials.

However, several drawbacks, such as brittleness, low thermal stability, medium gas barrier properties, and the low water resistance of the PLA, limit its use in certain packaging applications (Cabedo et al. 2006).

Natural fibers have been added as fillers to PLA to enhance its toughness and thermal stability and to reduce its brittleness (Huda et al. 2006; Bax and Mussing 2008; Huda et al. 2008; Talib et al. 2011). Furthermore, the addition of alkali-treated fibers can increase the interfacial adhesion between the filler and polymer matrix to produce composites having high strength properties (Huda et al. 2008; Khalid et al. 2008). Recently, α -cellulose (single fiber with low lignin content) has been successfully derived from kenaf bast fibers and has demonstrated better tensile strength and stiffness, as well as thermal stability when added to PLA composites (Talib et al. 2011). The goal of this study is to investigate the effect of adding KDC (0 to 60 wt.%) on the mechanical and physical properties of the biocomposites.

EXPERIMENTAL

Materials

PLA 2002D food packaging-grade resin was purchased from NatureWorks LLC, USA, and its properties are listed in Table 1. Kenaf bast fiber was generously supplied by the Institute of Tropical Forestry and Forest Products (INTROP), Malaysia. Reagent-grade sodium hydroxide (NaOH), acetic acid (CH₃COOH), and technical grade sodium chlorite (NaClO₂) (80% pure) were purchased from Fisher Chemicals Sdn. Bhd., Malaysia.

Table 1. Properties of PLA Resin

Properties	PLA Polymer 2002D
Molecular Weight, g/mol	204453
Melting Point, °C	150-160
Glass Transition Temperature, °C	58
Specific Gravity	1.24
Melt Index, g/10 min (260°C /2.16 kg)	5.0 - 7.0
Tensile Yield Strength, MPa	60
Tensile Modulus, GPa	3.5
Tensile Elongation, %	6.0
Notched Izod Impact, J/m	12.81

Preparation of KDC

α -cellulose and KDC were prepared using two steps: the first step was to produce holocellulose via chlorination or a bleaching process according to ASTM D1104. First, the lignin was removed (delignification) from the kenaf fiber. Specifically, 2.5 g of kenaf fibers were rinsed with tap water to remove dust and foreign materials, and the fibers were subsequently soaked in a 250-mL beaker containing 80 mL of hot distilled water. The beaker was transferred to a 70 °C water bath. Next, 0.5 mL of acetic acid and 1 g of sodium chlorite were consecutively added to the beaker every hour for 5 hours. The delignification process was tracked by the changing color of the kenaf bast fibers, which shifted from light brown to white. Finally, the holocellulose was washed and rinsed with tap water. The second step of this process was to convert the holocellulose to cellulose by mercerization.

The α -cellulose was prepared according to ASTM D1103. The holocellulose was added to a beaker with 10 mL of 17.5% NaOH solution and was heated at 20 °C in a water bath. After 5 min, 5 mL of 17.5% NaOH was added to the mixture 3 times at 5 min intervals and was incubated for a final 30 min at the end of the process. Next, 30 mL of distilled water was added to the mixture for 1 h via filtration and rinsing with tap water, 100 mL of 8.3% NaOH was added to the cellulose for 5 min. The alkali cellulose was neutralized by adding 10% acetic acid for 5 min. Finally, the cellulose was filtered, washed, and rinsed with distilled water, until the cellulose residue was free from acid, as indicated by the pH meter. The cellulose was dried overnight in a vacuum oven at 100 to 105 °C.

Characteristics of KDC

Each KDC fiber had a rod-like shape with a relatively non-uniform diameter and length (Fig. 1). Individual KDC fibers were measured by images projected from a microscope. The ESEM was used to obtain the printed image of the KDC. The printed images were examined under an image analyzer to determine the length and width of the individual KDC fibers. The aspect ratio (L/D) of the KDC was approximately 25 with an average length of 326 μm and an average diameter of 13 μm . The bulk density of the KDC was determined using the Bulk Density Apparatus according to ISO 60. The KDC was poured through a funnel into a measuring cup of 100 cm^3 . By weighing the empty measuring cup and subsequently filling the cup with cellulose, the bulk density was calculated. The bulk density of the KDC was 0.04 g/cm^3 .

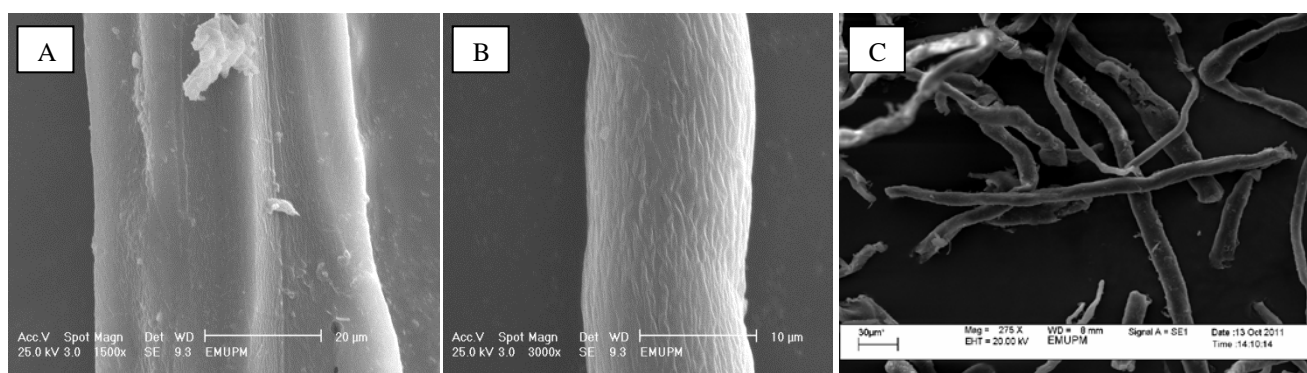


Fig. 1. The ESEM micrographs of A) Untreated kenaf fibre at 1500x magnification, KDC at 3000x magnification B), and C) Rod-like structure of KDC at 275x magnification.

Preparation of Composites

The KDC was ground and passed through a sieve. The KDC and PLA were mixed via melt blending using an internal mixer (Brabender Plastograph) at a fixed screw speed of 50 rpm and a temperature of 170 $^{\circ}\text{C}$ for 30 min. Molded composite sheets were subsequently produced at 160 $^{\circ}\text{C}$ for 10 min using a Model LP-50 Hot Press Machine. The sheet had a thickness of 3 mm for the flexural and impact tests and a 1-mm thickness for the tensile test.

Mechanical Tests

The tensile strength and flexural properties of the composites were measured using a Model 4301 Instron Universal Testing Machine with a load cell of 1 kN. Each tensile test was performed at a cross-head speed of 5 mm/min on specimens with dimensions of 12.7 mm \times 63 mm \times 1 mm according to ASTM 1882L, until tensile failure was detected. The percent elongation at the break point was also calculated. The three-point bending flexural test was performed at a cross-head speed of 1.3 mm/min on specimens with dimensions 12.7 of mm \times 130 mm \times 3 mm according to ASTM D790. The specimen was deflected, until rupture occurred. Meanwhile, the impact test was conducted on a notched rectangular specimen (12.7 mm \times 63 mm \times 3 mm) according to

ASTM D256. The Izod method was conducted using a Ceast CE UM-636 Impact Pendulum Tester with a 4 J hammer. Seven specimens of each sample were tested.

Physical Tests

The specimens used for the water absorption test had dimensions of 25 mm × 1.5 mm × 3 mm. The specimens were dried in an oven at 50 °C for 24 hours and were subsequently cooled in a desiccator for at least 30 min. The initial weight of the dried specimens was measured using an electronic balance. The specimens were then placed in a petri dish and immersed in distilled water at room temperature (~25 °C). The weight of the immersed specimens was measured daily for 60 days. A minimum of five specimens for each condition were tested. The densities of the composites were measured using an MD-200S Electronic Densimeter by the water displacement method. Three rectangular bar shape composites (20 mm × 12 mm × 3 mm) were tested to determine the average value.

$$\text{Water absorption} = \frac{W_w - W_d}{W_d} \times 100 \quad (1)$$

Scanning Electron Microscopy

Scanning electron microscopy was used to determine the dispersion of the KDC in the PLA matrix and the adhesion between the KDC and PLA matrix. The fracture surface of the composite specimens after impact testing was studied using a Philips XL30 Environmental Scanning Electron Microscope under an acceleration voltage of 20 kV. Before scanning, each sample was coated with gold for 30 min.

RESULTS AND DISCUSSION

Flexural Properties

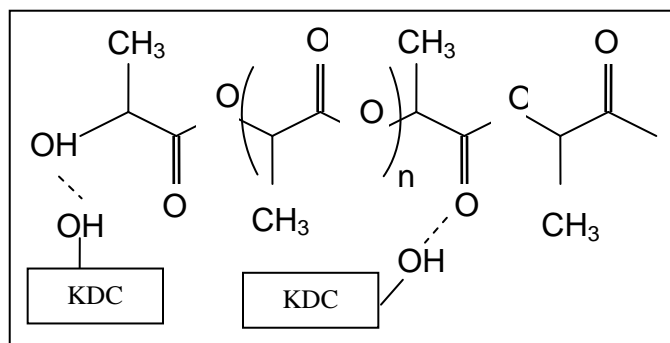
The effect of different amounts of KDC on the flexural strength is tabulated in Table 2. The neat PLA polymer exhibited the lowest flexural strength (63.4 MPa). The flexural strength increased by 36% up to 20% KDC, where it reached its maximum value (98.8 MPa). According to Shibata et al. (2003), the high flexural strength of PLA makes it difficult to improve the flexural strength in PLA composites. However, the flexural strength of these composites was improved compared to the neat PLA, which can be attributed to a well-formed interface that allows better stress transfer from the matrix to the fiber. The rough surface topography of the KDC seen by ESEM (Fig. 1) may improve the adhesive characteristics and thus improve the stress transfer during an applied load (Avella et al. 2009) by improving the mechanical interlocking between the KDC and matrix. Edeerozey et al. (2007) observed a rough surface topography and an absence of impurities from the kenaf fiber surface via SEM micrograph after treating with 9% NaOH. Additionally, use of high alkali concentrations (i.e., from 10 to 30% NaOH) could rupture the fiber's cell wall and remove the cementing substances, such as lignin and hemicellulose, generating a rough surface (Sreekala et al. 2000).

Table 2. Mechanical Properties of KDC/PLA Composites

Material	Density (g/cm ³)	Flexural Strength (MPa)	Flexural Modulus (GPa)	Impact Strength (J/m)	Elongation at break (%)
Neat PLA	1.26	63.4 (7.7)	3.9 (0.0)	29.9 (1.3)	12.3 (0.8)
10%	1.27	91.8 (0.3)	3.9 (0.1)	33.4 (1.8)	9.0 (0.4)
20%	1.30	98.8 (1.9)	5.5 (0.1)	33.9 (2.3)	8.7 (0.4)
30%	1.32	95.3 (3.0)	5.4 (0.1)	35.3 (0.7)	8.4 (0.2)
40%	1.35	97.2 (2.7)	6.6 (0.1)	34.8 (0.7)	9.0 (0.2)
50%	1.39	77.3 (2.9)	6.4 (0.1)	29.5 (0.7)	8.7 (0.2)
60%	1.41	68.2 (5.0)	8.4 (0.2)	27.4 (0.6)	9.1 (0.2)

Standard deviation in parenthesis ()

Moreover, good interfacial adhesion between the KDC and PLA was also expected, due to chemical interactions, such as hydrogen bonds. Hydrogen bonds are present in the composite between the hydroxyl groups in the kenaf fibers with the terminal hydroxyl groups (Bax and Mussing 2008) and the carbonyl groups of the ester groups of PLA (Garlotta et al. 2003) (Fig. 2).

**Fig. 2.** Possible interactions between KDC and PLA (Taib et al., 2009)

There was an appreciable rise in the flexural strength of the composites from 10 to 60 wt.% KDC compared to that of neat PLA, but above 50 wt.% KDC, the flexural strength decreased with increasing KDC loading. The flexural strength results demonstrate that incorporating KDC (20-40 wt.%) into the PLA matrix did not further enhance the bending strength of the composites and caused the flexural strength to decrease at higher concentrations. Huda and co-workers found a similar trend in the flexural strength of wood fiber/PLA composites at 20-40 wt.% filler, but no concrete explanation was given for this particular phenomenon (Huda et al. 2006). The non-uniform diameter and length, as well as the low aspect ratio of the KDC, were assumed to contribute to their ability to support stress transmitted from the PLA matrix. This result agrees with the

findings by Rozman and co-workers (2003), where the irregular shape and aspect ratio of the empty oil-palm fruit-bunch (EFB) fibers at different mesh sizes influenced the stress transfer during bending between the PP matrix and fibers. These researchers found that at smaller fiber mesh sizes (60 mesh), the flexural strength decreased with increasing fiber content (between 20 and 60 wt.%).

Table 2 shows that the flexural modulus of the KDC/PLA composites linearly increased with increasing (0-60 wt.%) KDC content. The stiffness of the KDC, combined with a higher KDC content, could explain the significantly enhanced flexural modulus of the KDC/PLA composites (Huda et al. 2006). The KDC might restrict the mobility of the polymer chain and thus hinder the ability of the polymer to deform. As reported by Karmakar et al. (2007), the increased composite stiffness depends on the filler content and the uniformity of the filler dispersion. These observations are supported by Canigueral et al. (2009), who concluded that a good fiber dispersion in the composite system could be confirmed by observing the linear increase in the composite's modulus. Figure 3 shows an even distribution of KDC fibers in the 30 wt.% KDC/PLA composite by ESEM. The KDC fibers were randomly distributed across a unit area and were separated from each other without the presence of clumps or agglomerates.

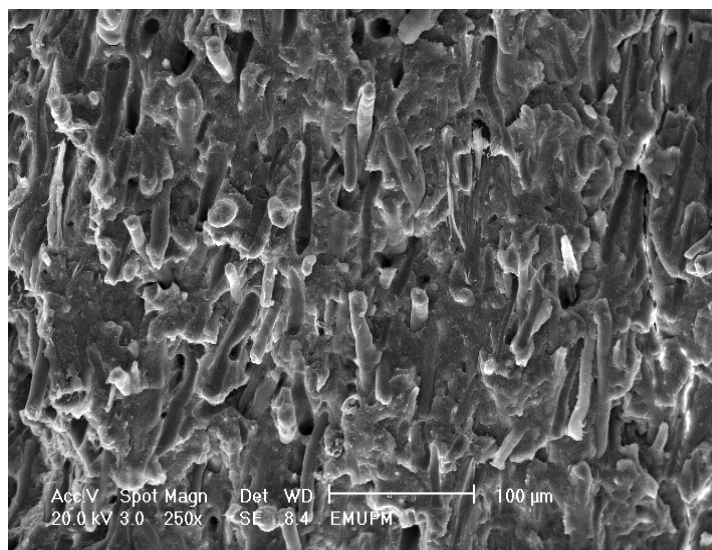


Fig. 3. The ESEM micrograph image of the KDC distribution of the 30 wt.% KDC/PLA composite

Table 2 shows the elongation at break for the different KDC/PLA composites (0-60 wt.%). The neat PLA had a higher percent elongation at break compared to the KDC/PLA composites, which contradicts the flexural strength and modulus data. However, a reduction in strain at break with increasing fiber content in a polymer matrix is common in biocomposites (Mathew et al. 2005). The neat PLA matrix can elongate more, due to its elasticity. As the KDC content in the PLA increased, the elasticity of the PLA was suppressed, due to the derived cellulose. Rozman et al. (2000) showed that a rigid interface between the fibers and the matrix material reduces the deformability of the composites.

PLA is naturally brittle; adding more fibers to the composite should lower the strain at break and create stress-concentrated areas, especially at the cellulose ends, resulting in lower strength. However, in this case, by adding KDC to the PLA matrix, the strain at break of the composites was minimized without reducing the tensile strength or modulus (Tawakkal et al. 2010) of the composites.

Impact Strength

Table 2 also presents the effects of various KDC contents on the notched Izod impact strength of the KDC/PLA composites. Adding KDC to the PLA matrix did not significantly contribute to the total impact strength of the composites. However, the 30 wt.% KDC/PLA composite achieved the highest impact strength (35.3 J/m) with a 15% increment. Overcoming the brittleness of PLA improves its impact resistance properties. Taib et al. (2009) used a plasticizer (PEG) to improve the impact properties of kenaf/PLA composites but did not observe positive results, while in this study, 40 wt.% kenaf/PLA composite exhibited a 60% reduction in impact strength compared to the neat PLA.

Further addition of KDC (from 40 to 60 wt.%) slightly reduced the strength of the composites by up to 8.4%. This result suggests that the presence of the KDC did not significantly contribute to the capacity to absorb energy under impact or to the final composite toughness because the incorporation of the KDC into the PLA matrix might result in stress concentration regions that require less energy to initiate cracks. This stress concentration could be generated from the fibers' ends, composite defects, and a lack of adhesion between the fibers and the matrix at the interface region (Huda et al. 2006).

The decrease in impact strength indicates that the amount of matrix is probably insufficient to transfer the stress effectively during a sudden impact in combination with the lower absorption characteristics of the KDC. During an applied load, the stress is transferred from the PLA matrix to the KDC, which can act as a barrier to transfer the impact stress load from this small portion of the PLA matrix. Additionally, less energy is absorbed, due to higher crack propagation and crack initiation (Huda et al. 2006).

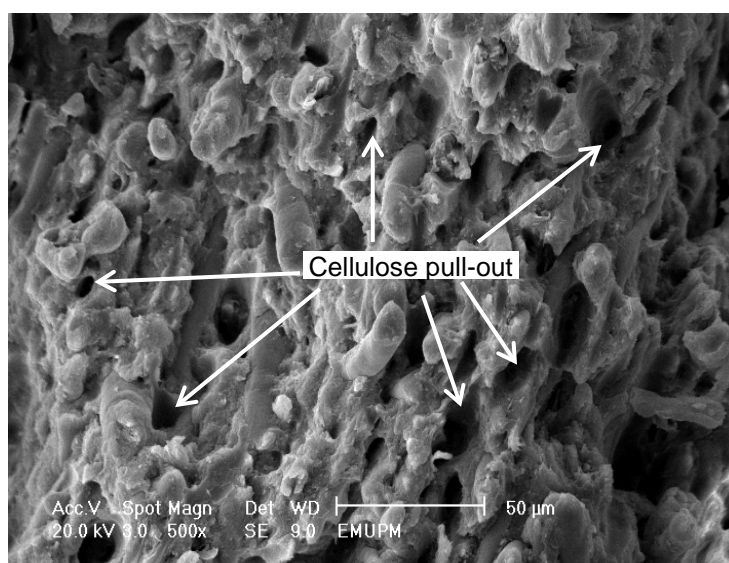


Fig. 4. The ESEM micrograph image of the KDC distribution on the impact fracture surface of 60 wt-% KDC/PLA composite.

Figure 4 shows an ESEM micrograph indicating the presence of cellulose pull-out in the 60 wt.% KDC/PLA composites on the impact fracture surface. This smooth cellulose pull-out reveals that the crack may propagate along the cellulose-PLA interface, rather than in the interior of the cellulose. In this condition, less energy will be absorbed at higher KDC contents.

Physical Properties

Figure 5 presents the water absorption of the neat PLA and KDC/PLA composites at various KDC contents as a percentage of dry weight after continuous immersion in water for 60 days. The results demonstrate that the water absorption of the neat PLA was lower than that of the KDC/PLA composites. The commercial PLA polymer absorbed less than 1% water, which is similar to most petroleum-based polymers, while the 60 wt.% KDC/PLA composites absorbed up to 12% water. The water absorption of the composites was found to increase with increased KDC content. The first 10 days of immersion showed a rapid increase in the level of water absorption of the KDC/PLA composites. The next 10 days showed a gradual increase in the water uptake, and the samples finally reached a plateau for the rest of the testing period. Additionally, the water absorption increased with soaking time, but the rate of absorption decreased over time for all of the KDC/PLA composites.

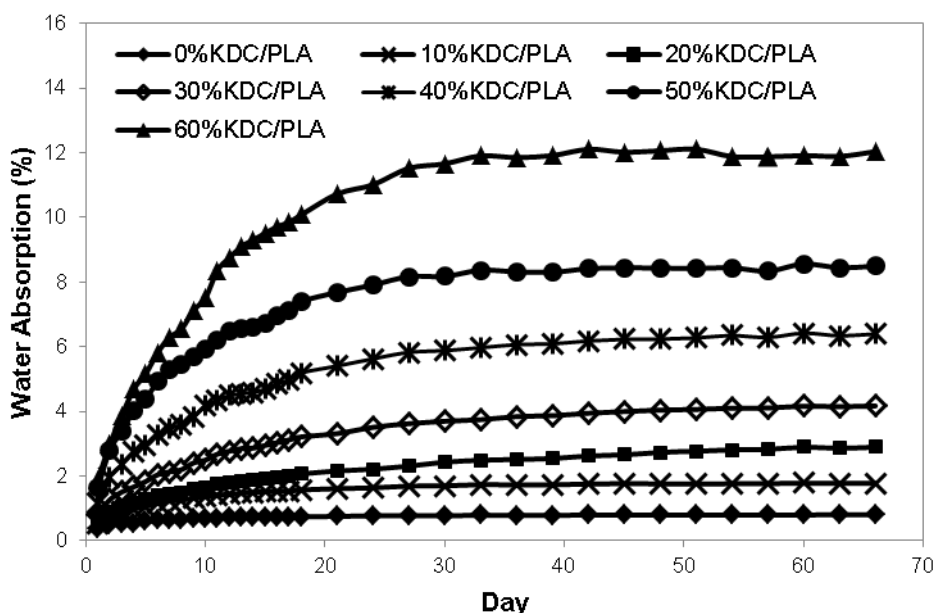


Fig. 5. The effect of KDC loading on moisture absorption of KDC/PLA composites

Several researchers have reported that PLA can absorb up to 1% water (Finkenstadt et al. 2007), while the KDC/PLA composites absorbed more water, due to the hydrophilic nature of the KDC that allowed hydrogen bonding to occur between the free hydroxyl groups of the cellulosic molecules with water molecules. During the first 20 days, the water caused the fiber's cell walls to swell and caused the fibers to expand, until the cell wall became saturated with water. At later times, the composites reached a constant water uptake, beyond which water exists as free water in the void structure and

does not contribute to further expansion (Rowell et al. 1998). At higher KDC contents, however, there were more free hydroxyl groups to interact and absorb water compared to composites with lower KDC contents.

Alkali-treatment of the grass fibers increased their hydrophilic characteristics by increasing the accessibility of the free hydroxyl groups, as revealed by the FTIR spectrum (Lui et al. 2004). Nevertheless, a considerable amount of water absorption by KDC/PLA composites was seen in this study compared with other untreated kenaf fiber-reinforced PLA that achieved higher water uptake (25%) at 40 wt.% kenaf loading (Taib et al. 2009). The mercerized fibers probably increased the accessibility of the hydroxyl group on the fibers' surface to generate a greater number of hydrogen bonds between the fibers and the water molecules. The void volume might be reduced in the KDC/PLA composites due to the strong intermolecular fiber-matrix bonding, restricting water penetration and storage through the interface.

Table 2 also tabulates the effect of various KDC contents on the composites' densities. Incorporating KDC into the PLA matrix changed the density of the PLA. Neat PLA (1.26 g/cm^3) was less dense compared to the KDC/PLA composites. The density gradually increased from 0 to 60 wt.% KDC content at a 12% increment. The 60 wt.% composite achieved the highest density (1.41 g/cm^3). The density of the composite increased with increasing KDC content during the molding process via compression.

This increase in density is accompanied by the formation of more free volume when a larger amount of KDC is incorporated into the polymer mass. The low bulk density (0.04 g/cm^3) of the KDC, before compounding, implies substantial void volume, and not all of the void volume disappears upon compounding. Thus it was observed that KDC had created more voids at the PLA matrix interface and initially had increased the volume per unit mass of the composite. At higher KDC contents, a lower density of the composite was initially formed in the hot press molding cast. Possibly, such voids will tend to close up when pressure is applied. Given the fact that the inherent density of cellulose is higher than the PLA, the net effect is a higher density as the amount of fiber component is increased, although an increased void volume may also be present.

CONCLUSIONS

The influence of KDC content on the mechanical and physical properties of KDC-filled PLA composites was investigated and resulted in the following conclusions:

1. The flexural properties of the KDC/PLA composites were improved compared to those of the commercial neat PLA polymer.
2. Although a considerable percentage of water was taken up by the KDC/PLA composites, it is considerably lower compared to that seen in many other natural fiber composites.
3. The density of the composite increased with KDC content, which may limit its potential applications. However, if density is not important or is the least important criterion, this material has advantageous properties, such as being a fully sustainable material and exhibiting reduced PLA content in the final product.

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