Effect of Microcrystalline Cellulose from Paper Pulp on Poly(Lactic Acid) Properties as Reinforced Fiber in Biodegradable-Material Application

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Abstract. *This study investigates the application of thermal processing techniques in packaging, focusing on the development and characterization of a composite material comprising poly(lactic acid) (PLA) and cellulose derived from paper pulp (PP). Cellulose sourced from the paper industry is treated with sodium hydroxide and sulfuric acid under controlled temperature conditions. Fourier-transform infrared (FT-IR) spectroscopy reveals structural similarities between the synthesized cellulose and standard cellulose. X-ray diffraction (XRD) analysis indicates a potentially high crystalline structure in the microcellulose, about 81% from raw PP at 63%. In comparison, the commercial microcrystalline cellulose (MCC) was about 82%, and peaks were similar to those of MCC. Scanning electron microscopy (SEM) analysis reveals a reduction in the size distribution of cellulose after processing. The morphology of the polymer composite shows heterogeneous dispersion of cellulose within the PLA matrix. Differential scanning calorimetry (DSC) results indicate that the crystallization and melting enthalpies in the composite material are comparable to those of its components. Thermal stability shows a lower degradation temperature, about 10°C, making degradation easier. Adding microcrystalline cellulose enhances the tensile strength (50 to 67 MPa) and Young's modulus (2424 to 2898 MPa) of the PLA/cellulose composites. However, stability issues arise at a cellulose content of 5 phr compared to neat PLA. Furthermore, UV-vis spectroscopy demonstrates that the PLA/cellulose composites exhibit improved UV-blocking ability. Lastly, the materials show suitable results for biodegradable-material applications.*

Keywords: Poly(Lactic Acid), Microcrystalline Cellulose, Reinforced Fibers

1 Introduction

Plastic is widely used daily, but most plastic materials cannot be decomposed. It can be seen that plastic waste has a serious impact on the ecosystem, which will lead to global warming because it takes up to 500 years for most plastics to be degraded by landfill [1]. These problems motivate studying the biodegradable polymers extracted from natural sources. The famous is poly(lactic acid) (PLA), a biodegradable strong polymer. PLA has the advantages of high strength, transparency, and rapid degradation, only 180 days, but some mechanical properties are still low for some applications [2].

Cellulose $(C_6H_{10}O_5)_n$ is an organic compound that is a significant structural component of the primary cell wall of green plants, various forms of algae, and oomycetes. It is a non-toxic and biodegradable polymer with high tensile and compressive strength; it has widespread use in various fields, especially as a reinforced fiber in polymer composites [3]. Microcrystalline cellulose is a cellulose of micron size and high crystallinity. It can be synthesized and purified by eliminating the lignin and hemicellulose, which are amorphous phases [4]. Paper pulp (PP) is an industry product processed before forming into paper in various grades and sizes. However, paper is used less every year and is replaced by technologies such as computer tablets and digital files. As paper pulp is a raw material, microcrystalline cellulose adds value and creates new avenues of use [5]. Not only that, PP passing some plant treatment makes it easier to purify the amorphous phase, such as lignin and hemicellulose [6-8].

Polymer composite is a technique for increasing the properties of a polymer by adding a particle into the polymer matrix. It can be used for specific applications, such as carbon fiber for electrical conductivity [9] or adding chitosan for antibacterial applications [10]. Some composites were investigated to increase mechanical and thermal properties for various applications [11]. Cellulose composites are famous for being used as a reinforcement to increase the polymer's strength and rigidity. In addition, some research shows that polymers have increasing thermal properties, hydrophilicity, and crystallinity [12-16]. For example, Sonia and Dasan [14] prepared the composites of celluloses microfibers (CMF)/poly (ethylene-co-vinyl acetate) (EVA) for food packaging applications. They reported that the composite showed an increased barrier property with CMF loading. SEM and optical microscopic photographs showed a uniform distribution of CMF in EVA. XRD and DSC results indicated increased crystallinity of composites with CMF loading. Moreover, the CMF incorporation improved the biodegradation of composites when studying using A. Niger. Frollini et al. [15] prepared composites of poly(butylene succinate) (PBS) reinforced with different lignocellulosic fibers. They found that sisal and curaua fibers have significant potential as reinforcing agents of PBS due to their superior chemical compatibility with the aliphatic matrix. Sisal/PBS and curaua/PBS composites demonstrated greater water absorption resistance than coconut/PBS and sugarcane bagasse/PBS composites. Luzi et al. [16] prepared poly(lactic acid) (PLA) and poly(butylene succinate) (PBS) blends and nanocomposite films by solvent casting method incorporating 1 or 3 wt% of cellulose nanocrystals extracted from Carmag-Nola carded hemp fibers. Both unmodified (CNC) and surfactant-modified (s-CNC) cellulose nanocrystals were added to the PLA matrix or the PLA/PBS blend to produce binary or ternary formulations. They reported that the mechanical analysis showed an increase in Young's modulus in binary and ternary formulations, which was more

evident in the CNC-based formulations. The presence of CNC and s-CNC and the addition of PBS to the PLA matrix improved barrier properties.

In this research, cellulose synthesis using paper pulp was studied and characterized in terms of morphology, functional groups, and crystallinity. Additionally, cellulose was investigated as a reinforcement composite to enhance the mechanical properties of PLA, which was the primary focus of this study. The resulting composites were characterized based on morphology, UV absorption, mechanical properties, and thermal properties, aiming to develop high-strength materials with desirable properties for various applications. The knowledge gained from this work can be applied to develop the preparation of microcrystalline cellulose from paper pulp and use it as a modifier in polymers.

2. Experimental

2.1 Materials

Poly(lactic acid) (Ingeo™ Biopolymer 2003D) with a melt flow index of 6 g/10 min and a specific gravity of 1.24 g/cm³ was supplied by NatureWorks LLC, USA. Cellulose was prepared from the final pulp product derived from Eucalyptus tissue, which underwent industrial treatment before being processed into a paper sheet. The final pulp was provided in collaboration with Thai Paper Company Ltd. The chemicals used included sodium hydroxide (NaOH, 98%, AR grade) and sulfuric acid (H_2SO_4, 98%, AR grade), both purchased from LOBA CHEMIE PVT.

2.2 Cellulose synthesis

Ten grams of paper pulp was cleaned with 500 mL of deionized (DI) water at 60°C for one hour, then separated and dried in an oven at 60°C for 8 hours. After drying, 10 g of the cleaned pulp was added to 500 mL of a 4% w/v NaOH solution at 60°C for 4 hours for purification. Following filtration and pH adjustment, the pulp was stirred in 500 mL of a 20% v/v H_2SO_4 solution at 60°C for 4 hours to reduce particle size through acid hydrolysis. The mixture was then filtered, and the pH was adjusted to neutral using DI water. Finally, the material was dried in an oven at 60°C for 12 hours, crushed, and sieved. The cellulose obtained from the paper pulp is designated as CPP.

2.3 Preparation of polymer composites

PLA was dried in an oven at 80°C for 4 hours before blending, while cellulose was dried at 60°C for 24 hours. The PLA was then melted in an internal mixer at 170°C with a rotor speed of 50 rpm for 15 minutes. CPP was added at 1, 3, and 5 parts per hundred resins (phr) based on the weight of PLA. Prior to compression molding, all samples from the internal mixer were dried in an oven at 60°C for 1 hour. Finally, the PLA and polymer composites were molded using hot press compression molding at 170°C and 4000 bar for 15 minutes to form them into specific shapes for characterization.

2.4 Cellulose and polymer composites characterization

The surface morphology of the cellulose and polymer composites was observed by a scanning electron microscope (SEM) (MIRA3, TESCAN, Czech Republic). The cellulose in each step was dispersed in RO water and dropped on the stuff. The tensile fracture surfaces of the polymer composites were examined using SEM. Fourier transform infrared (FT-IR) spectroscopy (Bruker ALPHA II) was employed to verify the chemical structures of dry cellulose particles and polymer composite film sheets. Crystallinity was analyzed using X-ray diffraction (XRD), a method based on Bragg's law, with a wavelength of 0.154 nm, operated at 40 kV, and a scanning range of 2θ from 1° to 80°. Mechanical properties were evaluated using a universal testing machine (UTM) (ASTM D 638-00, EZ Test, EZ-LX/EZ-SX Series) at room temperature with a crosshead speed of 50 mm/min. Thermal properties were characterized using thermogravimetric analysis and differential scanning calorimetry (TGA-DSC) (SDT Q600, TA Instruments). Samples weighing between 5–10 mg were measured in a nitrogen atmosphere, with temperatures ranging from 50°C to 600°C and a heating of 10°C/min. UV-Vis absorbency of the films was recorded in the 200–800 nm range using a UV-Vis spectrophotometer (VARIAN, Cary 5000) to investigate UV-Vis adsorption.

3. Results and Discussion

3.1 Characterization of cellulose from paper pulp

3.1.1 Morphology

The morphology of raw PP, PP after alkaline treatment, and CPP after acid hydrolysis was observed, as shown in Fig. 1. Fig. 1(a) shows the SEM image of the raw PP. After stirring with water, the fracture surface of the paper pulp revealed that the main structural fibers were long and raft-like. However, the surface had some layers covered by the main fibers. Ruigang Liu et al. describe it as a hemicellulose with some lignin, which has a non-crystalline structure [8]. After stirring with NaOH, the morphology, as shown in Fig. 1(b), still consisted of long fibers and raft-like surfaces. However, the thin layer covering the main fibers disappeared. The amorphous phases were almost entirely removed by using only 4% NaOH, resulting in a more purified fiber with a higher percentage of crystallinity. Fig. 1(c) shows the cellulose from paper pulp (CPP) after stirring with H_2SO_4. The morphology revealed short fibers and raft-like surfaces. The particle size was approximately 136 ± 24 μm in length and 10 ± 2 μm in width. Compared to the commercial MCC, which is 20-100 μm, the PPC is similar because it is still in a micron size range. [17]. This indicates that acid hydrolysis using H_2SO_4 can effectively reduce the size of the fibers. By the way, some limitations of the acid used are limited from the PP raw material. The eucalyptus is a softwood tissue that cannot be cut with a stronger acid to produce nano-sized fibers [18]. Lastly, this condition shows that the micron size of cellulose can be synthesized from this process and conditions.

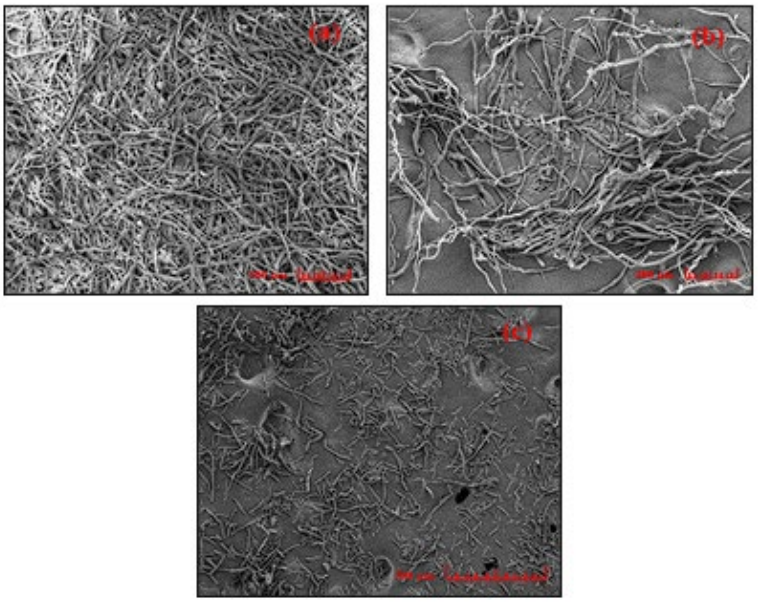

Figure 1. SEM micrographs of cellulose: (a) Paper pulp, (b) Alkali treatment by 4% w/v NaOH, and (c) Acid hydrolysis by 20% v/v H_2SO_4 at 150X

3.1.2 Chemical structure

The Fourier transform infrared (FTIR) spectroscopy was used to study the chemical structure and purity of CPP. The spectra of PP and CPP samples in various steps are shown in Fig 2. The band at 1027 cm^{-1} is assigned to the CH_2 group oscillation of the cellulose structure. The prominent at 1428 cm^{-1} is associated with lignin C-H aromatic skeletal vibration. The band at 1639 cm^{-1} is attributed to the carboxylate groups stretching vibrations in hemicellulose and is prominent at 3330 cm^{-1}, observed in all spectra, and represents the free OH group [6].

The area normalization was calculated to study the purity of synthesis, as shown in Table 1. The raw paper pulp material was preferred to 100% of lignin and hemicellulose, and for all calculations, the area of 1027 cm^{-1} was a standard band area with Gaussian Lorentzian Models. The calculations show that the alkaline treatment can remove half of the hemicellulose and some of the lignin. However, the acid hydrolysis significantly decreased the lignin part to 32% remaining and the hemicellulose to 6%. Thus, the acid hydrolysis with H_2SO_4 reduced the size and increased the purity of the CPP. It corresponds with the SEM micrographs that the hemicellulose layers disappeared after passing the process [5, 19]. Also, the %yield of synthesis was calculated from the weight difference in each condition and showed a very high value. However, it passes the acid hydrolysis, which means the PP has a very high ratio of cellulose inside. The PP has a high potential to be used as a raw material for cellulose synthesis, and the synthesis method is also high quality to purify the cellulose.

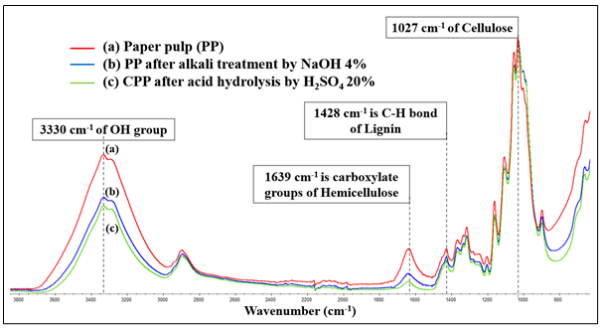

Figure 2. FTIR spectra of cellulose: (a) Paper pulp, (b) Alkali treatment by 4% w/v of NaOH, and (c) Acid hydrolysis by 20% v/v of H_2SO_4

Table 1 Summary of comparison of mechanical properties of polymer blends at different composition and sequence mixing.

Sample	%remaining of lignin	%remaining of hemicellulose	%Yield
Paper pulp (PP)	100	100	100
PP after alkali treatment	79	55	82
CPP after acid hydrolysis	32	6	64

3.1.3 Crystallinity

XRD analysis characterized the crystallinity. The XRD patterns of PP and CPP (Fig. 3) also show the three crystallization peaks at 2θ = 15.7°, 22.7°, and 34.8 °, which correspond to the typical diffraction of the planes of (110), (200), and (004), respectively [20]. It is a raw paper pulp with a crystalline structure similar to the last product. Hussein Oubani et al. [21] described the pattern of this position is the band of microcrystalline cellulose (MCC), but the shape of the peak of PP is broader than the others. The shape of CPP and MCC from the reference is sharper than that of PP, which is similar. So, the significant difference is the %crystallinity, which is calculated from the Eq. (1).

$$\%\text{crystallinity} = \frac{A_{crystal}}{A_{total}} \times 100$$

When $A_{crystal}$ and A_{total} are the diffraction peak areas of the crystalline region and the total region, the results show that the raw PP is 63%, which is lower than the MCC commercial. However, after passing the process, it can be increased to 81%, which is the same as the MCC commercial at 82% [22]. It can be confirmed that the CPP is too close to the MCC in terms of size, purity, and crystallinity as well.

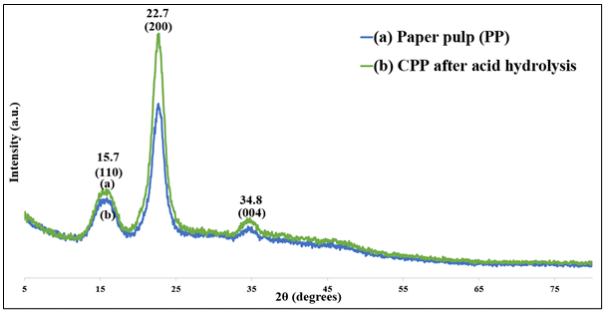

Figure 3. X-ray diffraction patterns of (a) raw PP and (b) CPP after pass alkali treatment and acid hydrolysis

3.2 Characterization of Polymer composites

3.2.1 Morphology

The cross-section morphology of neat PLA and PLA/CPP is observed in Fig 4. The neat PLA in Fig 4 (a) exhibits the smooth surface, indicating the material's rigidity and lack of toughness. In Fig 4 (b-d), cellulose is present in distinct phases, as evidenced by the visible cellulose particles. At a magnification of 300X, very small cellulose particles are observed to be well-dispersed within the polymer matrix. At a higher magnification of 5000X, fractures between the phases can be seen, revealing long particles within the matrix that appear to have been pulled out due to tensile forces. In some cases (Fig 4(b)), the side view of long cellulose fibers embedded within the PLA matrix is visible, showing no voids between them. The two matrices appear to be well-integrated and cohesive. These results are similar to the Somnuake P et al., that show cellulose's pull-out feature [5]. Additionally, cracks are observed to align and increase in number with higher cellulose ratios. The comparison between PLA and PLA/CPP composites revealed a higher tendency for crack formation with increasing cellulose content.

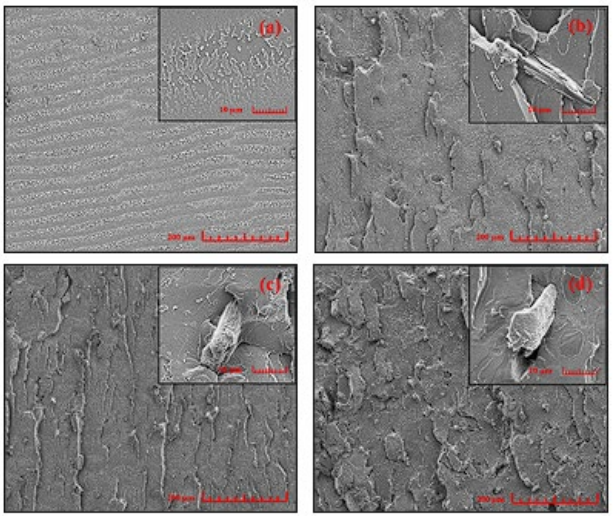

Figure 4. SEM micrographs of the cross-section fractured of (a) neat PLA (b) PLA/CPP 1 phr, (c) PLA/CPP 3 phr and (d) PLA/CPP 5 phr at 300X and 5000X

3.2.2 Mechanical Properties

The mechanical properties of neat PLA and PLA/CPP composites were investigated using tensile testing. The results, including tensile strength, Young's modulus, stress at break, and elongation, are summarized in Table 2. The results showed that the addition of CPP increased the strength and rigidity of the materials while maintaining unchanged break elongation. It can be inferred that cellulose acted as a reinforcing fiber for the biodegradable material, making it suitable for various applications such as food packaging and 3D printing. However, when CPP was added at 5 phr, all properties remained stable compared to the 3 phr addition. Based on these findings, it can be concluded that 3 phr of CPP is the optimal ratio for the composites. The dispersion of CPP in the polymer matrix is influenced by the non-homogeneous phase and the differing polarities of PLA and CPP. Lee Woojin et al. [23, 24] explained that the decrease in mechanical properties results from brittle fracture behavior, which is also observed in these material composites. Their studies demonstrated that the mechanical properties initially remain stable but decrease when particles are added beyond the necessary amount. Based on the results, adding 3 phr of CPP to the composites is the optimal condition for improving mechanical properties from the high properties. Although the 5 phr addition shows slightly higher values, the error bars indicate a non-significant change, and it requires more cellulose compared to the 3 phr addition.

3.2.3 Thermal Properties

The thermal properties and thermal stabilities of neat PLA and PLA/CPP composites were investigated using DSC and TGA analyses. DSC thermograms are presented in Fig 5 and the results, including the degradation temperature (T_d) obtained from TGA characterization, are summarized in Table 3. The glass transition temperature (T_g) and melting temperature (T_m) exhibit no significant change upon the addition of CPP to the polymer matrix. It can be described that the cellulose is not affected by the amorphous form [25] and the purity of the crystalline [26]. However, a slight shift in the co-crystallization temperature (T_c) is observed, which follows a trend as more CPP is incorporated into the PLA matrix. It can be observed that cellulose enhances crystallization and facilitates co-crystallization. Not only that, the enthalpy of melting (ΔH_m) and enthalpy of co-crystalline (ΔH_c) were used to calculate the %crystallinity (%X_c), as follows the Eq. (2).

$$\%X_c = \frac{\Delta H_m - \Delta H_c}{\Delta H_{m0}} \times 100$$

While ΔH_{m0} is the equilibrium enthalpy of melting (theoretical value of the polymer supposed to be 100% crystalline) of PLA is 93.7 J/g [27].

Table 2 Summary of comparison of mechanical properties of PLA/CPP composites.

Sample	Tensile strength (MPa)	Young's modulus (MPa)	Elongation at break (%)
Neat PLA	50.2 ± 1.9	2424.5 ± 36.5	2.9 ± 0.1
PLA/CPP 1 phr	55.9 ± 3.3	2441.3 ± 70.0	3.0 ± 0.2
PLA/CPP 3 phr	65.7 ± 1.9	2844.8 ± 110.3	2.9 ± 0.1
PLA/CPP 5 phr	66.0 ± 4.2	2898.3 ± 142.7	3.3 ± 0.3

The crystallinity (%X_c) of the polymer composites of the polymer composites increases with the addition of CPP. Additionally, the lower T_c indicates that CPP enhances the crystallinity of PLA. Furthermore, another advantage of adding CPP is the observed reduction in thermal degradation temperatures (T_{d10} and T_{d50}), which suggests lower energy consumption is required to degrade the materials. It is because the effected of cellulose which shows the T_{d10} at 284°C and T_{d50} at 316°C. So that, it will be decreased thermal stability of PLA composites from the lower stabilization of microcrystalline cellulose [28]. Yang et al. [29] describes that the degradation of the cellulose is about 200 – 300°C and it depends on the composition of the cellulose. Cellulose which obtains from wood pulp (in this case is eucalyptus pulp) contain normally cellulose, hemicellulose and lignin in varying amounts. These components have different decomposition temperatures: cellulose starts degrading (T_{d10}) about 315°C, the hemicellulose at 220°C and the lignin at 160°C [30]. It is evident that the cellulose undergoes degradation at lower temperatures, and the CPP exhibits lower performance compared to Yang et al.'s work due to the presence of approximately 32% unwashed lignin in the CPP. Another notable observation is that the PLA/CPP composite with 3 phr cellulose demonstrates mechanical properties comparable to the 5 phr composite, despite having a lower crystallinity (%X_c) and utilizing less cellulose. These mechanical improvements are attributed to the effective dispersion of cellulose, which maintains stress distribution, rather than minor changes in the homocrystalline structure of PLA.

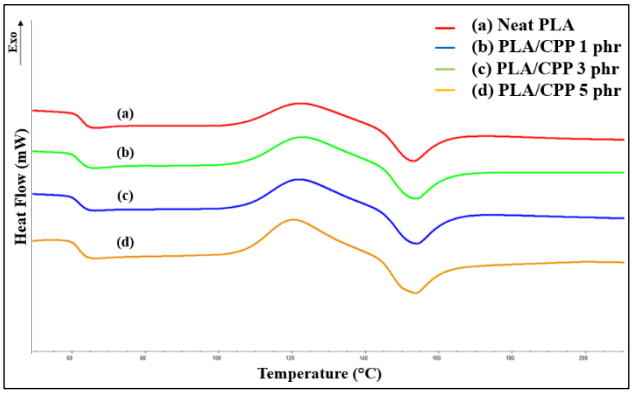

Figure 5. DSC Thermograms at first scan of neat PLA and PLA/CPP composites at 1, 3, 5 phr

Table 3 Thermal properties and %Crystallinity of neat PLA and PLA/CPP composites.

Sample	T_g (°C)	T_c (°C)	T_m (°C)	ΔH_m-ΔH_c	%X_c (%)	T_{d10} (°C)	T_{d50} (°C)
Neat PLA	62	122	153	28.7	30.6	335	360
PLA/CPP 1 phr	65	123	155	33.9	36.3	331	357
PLA/CPP 3 phr	63	122	153	32.2	34.3	332	353
PLA/CPP 5 phr	62	120	154	37.7	40.3	331	349

3.2.4 Optical Properties

UV absorption is an important property for the packaging. The light transmittance of PLA was studied in two wavelength ranges: visible (400-800 nm) and UV (250-400 nm). Fig. 6 displays the UV-Vis transmittance spectra of neat PLA and PLA/CPP composites at different ratios. The UV-Vis spectrum of PLA/CPP demonstrates improved absorption compared to neat PLA. Due to the inherent properties of PLA, it is transparent and cannot absorb light. However, when CPP is added, the material exhibits enhanced UV-blocking ability. This is because the addition of CPP increases crystallinity or introduces cloudiness, which enhances light absorption. As a result, it can absorb some light. Therefore, it can be concluded that increasing the ratio of CPP can prevent UV transmission in PLA/ CPP composites [11, 19].

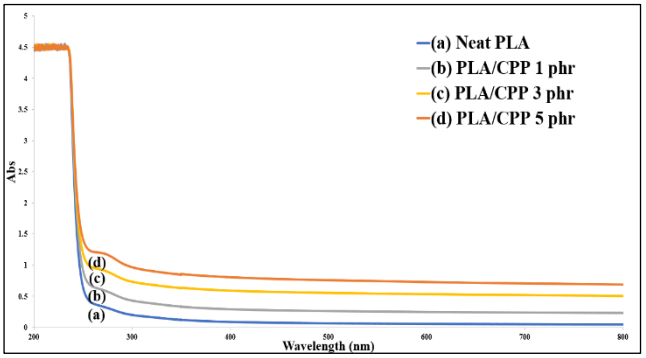

Figure 6. UV-Vis transmittance spectra of neat PLA and PLA/CPP composites at 1, 3, 5 phr

4. Conclusion

This work aims to study the value added of PP by synthesizing CPP and using it to develop the PLA by adding CPP as composites. The purity and crystallinity were increased after the alkali and acid hydrolysis treatment was passed. Then, blending the composites was focused on mechanical properties, and it was found that adding CPP increased the strength and rigidity of the polymer composites while saving the amount of cellulose used at CPP 3 phr, which is an optimum condition. The addition of paper pulp to PLA did not significantly affect the thermal properties of T_g and T_m but slightly decreased the T_c for easy crystallization. It was also found that the crystalline ($\%X_c$) increased with the same trends when CPP was added. The UV-Vis shows that the light blocking in this ratio is also very good, which is the main purpose of this work. In conclusion, PP shows the high quality of synthesizing MCC and PLA/CPP composites and the effective performance of applying in UV protection packaging applications.

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