

Bioplastics based Crystalline Cellulose from Bamboo Fibre, Poly (Lactic Acid) and Poly (Butylene Succinate) Nanocomposites using Acid Hydrolysis Method

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Abstract

The synthesis and characterisation of bioplastic nanocomposites reinforced with crystalline cellulose (CC) derived from bamboo fibre using the acid hydrolysis process is the main goal of this work. The extracted CC was surface modified by acetylation and added to a combination of polylactic acid (PLA) and polybutylene succinate (PBS) to enhance compatibility with the polymer matrix. The effects of filler loading, surface modification, and CC extraction on the composites' chemical, morphological, thermal, and mechanical performance were examined. Successful CC acetylation was validated by Fourier Transform Infrared (FTIR) analysis, which showed decreased hydroxyl ($-OH$) intensity about 3330 cm^{-1} and ester carbonyl ($C=O$) peaks at about 1740 cm^{-1} , suggesting improved hydrophobicity and interfacial compatibility. Acetylated, bleached CC demonstrated a rod-like shape with particle sizes under 200 nm and consistent dispersion within the PLA/PBS matrix at low filler loading, according to Field Emission Scanning Electron Microscopy (FESEM). The nucleating impact of CC in the polymer mix was confirmed by Differential Scanning Calorimetry (DSC) findings, which showed an increase in crystallinity from 21.6% to 29.4% . Thermogravimetric analysis (TGA) revealed decreased weight loss at $600\text{ }^{\circ}\text{C}$ and increased thermal stability, with the maximum degradation temperature rising from $348\text{ }^{\circ}\text{C}$ to $371\text{ }^{\circ}\text{C}$. The composite containing 0.5 phr acetylated, bleached CC showed the most promising tensile performance due to its superior chemical compatibility, higher crystallinity, enhanced thermal stability, and homogeneous filler dispersion, despite sample constraints limiting quantitative tensile testing. In polymer composites, these features are known to limit stress concentration and encourage effective stress transmission. Overall, the results show that PLA/PBS bioplastics may be effectively reinforced with acetylated bamboo-derived CC at low loading, underscoring its potential for sustainable packaging applications.

1. Introduction

Bioplastics are less environmentally impact compared to traditional plastics, especially when considering their life cycle assessments (LCA) and what happened after they are no longer necessary or end of life (EOL). Switching to bioplastics can save around 13% of CO_2 emissions compared to conventional plastics (Dissanayake et al., 2024). However, the environmental benefits of bioplastics are slightly more significant and strongly

dependent on how they are produced, the cultivation of their feedstocks, and the way human manage their EOL. For example, the use of pesticides and fertilizers in plant growths of feedstocks can lead to eutrophication and other adverse environmental impacts. In addition, different bioplastics have different levels of energy consumption in resin production, particularly electricity; PBS, for instance, uses much less than PLA [1]. One of the most significant features of bioplastics is their biodegradability, which means that they can break down into water, carbon dioxide, and biomass in particular conditions, and this will reduce pollution as well as waste. But the rate of biodegradation and mode can be significantly different for different bioplastics as well as different environments. PBS and PBS-starch blends were also observed to degrade fairly quickly in soil at rates between 1% and 7% in 28 days, and up to 24% for the powder forms. PLA slowly degrades in soil, usually showing little or no alteration over the same duration [2]. The widespread use of petroleum-based plastics in packaging has caused serious environmental problems, such as persistent pollution, microplastic accumulation, and greenhouse gas emissions. In contrast, bioplastics such as PLA and PBS have attracted much attention as alternative polymers from sustainability point of view for the degradation to biodegradable products in the atmosphere and a renewable origin. However, the applications of PLA and PBS in practice are constrained by their poor performances. PLA has brittleness, low thermal stability, while PBS is more flexible with inferior mechanical behaviour and higher production cost. One strategy to tackle these problems is the addition of biobased additives such as PBS to PLA; however, resulting materials fall short in terms of mechanical and thermal properties. One promising way to overcome these problems is reinforcing PLA/PBS blend with CC derived from bamboo, having excellent mechanical properties and crystallinity. Bamboo is a fast-growing, regenerative material and possesses high cellulose content so it is suitable for ecologically friendly CC extraction. However high quality highly crystalline CC is yet difficult to extract from bamboo through acid hydrolysis, requiring careful optimization of process parameters to the yield and structural integrity required. When adding bamboo-derived CC to PLA/PBS matrices, there are additional hurdles, such as achieving uniform dispersion and high interfacial compatibility, as well as determining the ideal CC loading to maximise mechanical and thermal advantages while preserving processability and biodegradability. In this issue, several tests were conducted to examine their crystallinity, mechanical strength, form, and thermal stability. Furthermore, this study is about optimizing the extraction and uniform dispersion of bamboo-derived CC in PLA/PBS matrices to improve mechanical, thermal and biodegradation properties while ensuring strong interfacial compatibility and consistent composite performance.

Thus, the objective of this study is to:

- To characterize the effect of acid hydrolysis process towards extracted CC by using FTIR and FESEM
- To investigate the effect of acetylation CC on mechanical, chemical and thermal properties.
- To evaluate the chemical and thermal properties of CC-reinforced PLA/PBS nanocomposites.
- To analyse the morphology and dispersion of CC in PLA/PBS matrix and evaluate its influence on the overall performance of bioplastic nanocomposites.

2. Methodology

This chapter included further discussion on the experiment's timeline and methodology. Additionally, the sample's characterisation was discussed.

2.1 Extraction of Cellulose from Bamboo Fibre

Bamboo fibre was purchased from local company called HangTerra Bamboo Malaysia. The bamboo fibre was harvested after reach 4-5 years. To maximise the pre-treatment process's overall surface area and efficiency, the bamboo fibre was first turned into bamboo pulp (BP). The bamboo fibrils were then ground into particles using a planetary ball mill set at 450 rpm for ten minutes. A sieve measuring 75 μm was used to filter the resulting bamboo pulp. For the pre-treatment process, bamboo pulp (BP) were soaked using 20% (w/v) of NaOH solution. After that, under the continuous stirring, the mixture was heated at 90 °C for 4 hours. After 4 hours, there are solid residue formed and washed by using distilled water until the pH became neutral (7). Then, by using oven at 50 °C, the solid residue was left overnight. In bleaching process, the solid residue that has been dried overnight was soaked by using 4% (w/v) of NaOH. In the meantime, H_2O_2 was added dropwise to the mixture with the same amount of NaOH. This process will be in continuous stirring for 60 minutes. The pre-treated bamboo pulp (PBP) was washed by using distilled water until it became neutral. The PBP then dried overnight at 50 °C [3].

2.2 Acid Hydrolysis Method to Obtain Crystalline Cellulose

By undergo acid hydrolysis process, CC can be isolated from the cellulose of BP. This process was carried out with 65 wt% concentrations of H_2SO_4 at constant temperature and time. The temperature used for the hydrolysis is 45 °C at 60 minutes [4]. The ratio for PBP mass to H_2SO_4 volume was 1:20. The excess cold distilled water was added to the mixture after the hydrolysis process has been completed. The centrifuge process for suspension at 6000 rpm for 10 minutes is to remove the acid solution. The distilled water was used to wash the precipitate to neutralize it. After the bamboo pulp has dried, the analysing of functional group has been done by using FTIR [5].

2.3 Crystalline Cellulose Surface Modification by Using Acetylation

Unmodified crystalline cellulose was first mixed with glacial acetic acid while continuously stirring with a magnetic stirrer for 45 min at 35 °C. The reaction mixture was then shaken for an hour at 35 °C after which an additional more glacial acetic acid and sulfuric acid (H_2SO_4) were added as catalysts [6]. After some cooling, acetic anhydride and additional sulfuric acid were added to the mixture. To complete the esterification process, the temperature was raised to 50 °C and run for three hours after the reaction was kept at room temperature for fifteen minutes. After completion, the reaction mixture was allowed to cool before being washed periodically with distilled water, filtered, and dried by using oven at 60°C until it completely dried.

2.4 Preparation of Mixing CC with PLA/PBS

The weight ratio of PLA/PBS blend were created: 70/30 wt%. In this blending, CC of 0.5 and 1.0 phr were added to polymer mixtures. In order to reduce moisture, CC was dried at 110°C for 24 hours prior to compounding, while PLA and PBS pellets were dried at 50°C for 24 hours in an air-circulating oven. After being dried, the PLA and PBS were added into the internal mixer and waited until it completely melted. Then, CC was added to the PLA and PBS that has been completely melted and let the mixture blending well. Using a crusher, the PLA, PBS, and CC mixture was broken up before being hot press. A hot press set at 180°C and 150 MPa of pressing pressure was used to create compressed sheets with a 0.25 mm thickness after warming for five minutes and pressing for four minutes. Then, before additional testing, the compressed sheets were placed in a desiccator and dried for 24 hours at 50°C in an oven [7].

2.5 Performance Evaluation

FTIR is basically a technique that being used to check the chemical binds in a specific material by analysing how it absorbs infrared (IR) light. A popular and practical technique for identifying and examining the chemical groups and interactions in bioplastic nanocomposites, like those composed of crystalline cellulose (CC) extracted from bamboo fibres mixed with (PLA) and (PBS) by using FTIR. By doing this testing, the presence of cellulose can be determined by looking at the spectra that produced by the FTIR [5][6]. Field emission scanning electron microscopy (FESEM) was used to determine the crystallinity of the sample. The Perkin-Elmer TGA7 (Wellesley, MA, USA) was used to assess the thermal stability of the nanocomposite films. About 10 mg of samples were subjected to TGA analysis at a heating rate of 10 °C/min from 0 to 600 °C while a nitrogen environment was present. Next, it was noted how the samples' weight ratio changed in relation to their temperature. The Perkin-Elmer DSC7 (Wellesley, MA, USA) was used to perform DSC analysis on samples weighing around 3–5 mg. The samples were put in a sample pan, and a temperature scan was performed at a rate of 10 °C/min (heating rate) in the temperature range of 30–200 °C. A Dog Bone Tensile Testing Machine was used to examine the tensile characteristics of compressed samples in compliance with ASTM-D882. Prior to testing, the test specimens of clean PLA, clean PBS, PLA/PBS blends, and PBS/PLA mixes with CC were allowed to cure for 24 hours at room temperature. The average of seven measurements was recorded after the test was conducted at room temperature with a crosshead speed of 50 mm/min.

3. Results and Discussion

This chapter discussed regarding the finding for this study. The comparison on the best mixing polymer with the characteristics of the crystalline cellulose is chosen based on the testing made on the surface and the thermal degradation.

3.1 Morphology of Crystalline Analysis by Using FESEM

The FESEM micrographs illustrate how chemical treatments transform bamboo fibres. Alkaline pretreatment with NaOH significantly alters the fibres by creating rougher, more porous surfaces and partially separating bundles, which indicates effective hemicellulose removal. This process increases the cellulose surface area, enhancing its accessibility for acid hydrolysis. After hydrolysis, the fibres fragment into smaller particles, revealing the breakdown of amorphous cellulose, while some lignin and contaminants remain, potentially affecting composite quality. Bleaching prior to hydrolysis refines the cellulose, resulting in smoother, finer particles as remaining impurities are removed. In contrast, untreated acetylated samples exhibit uneven surfaces and clustering, showing poor purification. The study emphasizes that effective chemical modifications hinge on contaminant removal to achieve uniform particle sizes and improve material properties for bioplastic composites. The optimal results were found in bleached acetylated samples, which displayed a consistent morphology, indicating effective modification of cellulose's hydroxyl groups for better dispersion and adhesion in composites, especially with hydrophobic polymers like PLA and PBS [8]. Comparisons between bleached and non-bleached cellulose/copolymer composites reveal that using non-bleached cellulose at 0.5 phr results in rough surfaces and poor adhesion due to residual impurities. Increasing this to 1 phr leads to agglomeration, hindering polymer matrix interaction and compromising composite integrity. Conversely, a composite with 0.5 phr bleached crystalline cellulose exhibits a smoother structure, benefiting from the removal of lignin and hemicellulose. Optimal loading at 0.5 phr enhances composite performance, while higher concentrations create irregularities due to agglomeration and voids, illustrating that the polymer matrix can become overloaded at excessive filler loads [9].

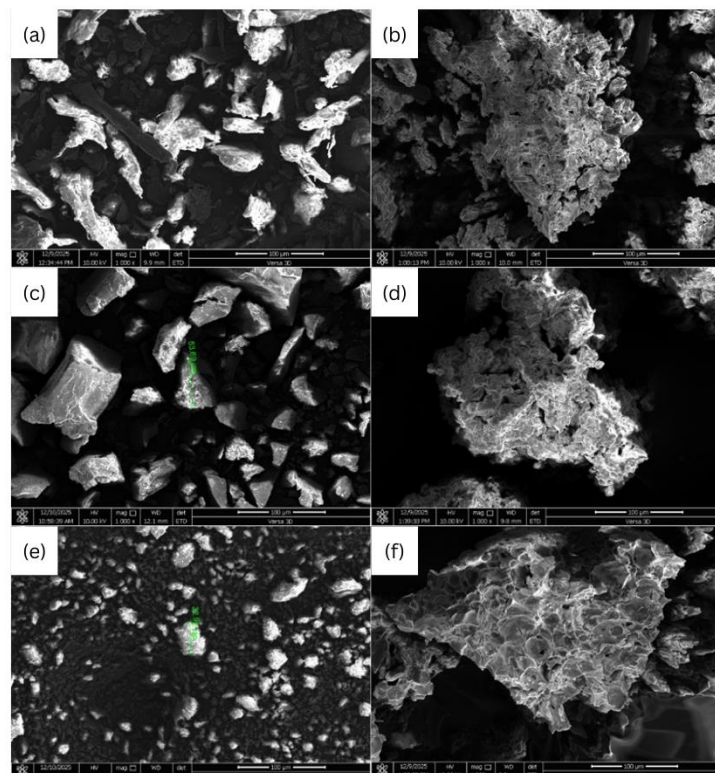


Fig. 1 The images by using FESEM with magnification 1000× (a) Raw Bamboo, (b) Pretreated Bamboo with NaOH, (c) Acid hydrolysis without bleaching, (d) Acid hydrolysis with bleaching, (e) Acetylation without bleaching and (f) Acetylation with bleaching

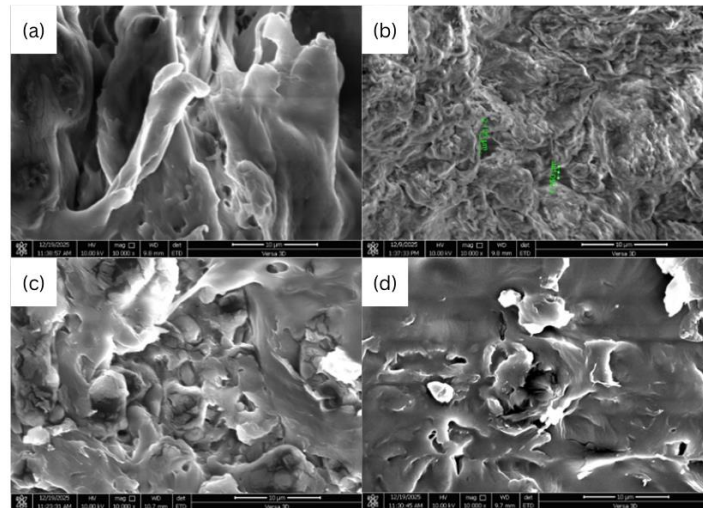


Fig. 2 The images of crystalline cellulose that has been mixed with different ratio (a) 0.5 phr Non-Bleach CC mix with polymer, (b) 1 phr Non-Bleach CC mix with polymer, (c) 0.5 phr Bleach CC mix with polymer and (d) 1 phr Bleach CC mix with polymer

3.2 Fourier Transform Infrared (FTIR) Analysis on Intermolecular Interactions

The study examines the impact of various treatments on cellulose's properties, particularly focusing on O-H stretching vibrations linked to its hydroxyl groups. A broad absorption band in the $3200\text{--}3600\text{ cm}^{-1}$ range indicates significant hydrogen bonding in acid-hydrolysed non-bleached cellulose. While moisture absorption persists, the intensity of O-H stretching decreases with increased polymer concentration, suggesting partial hydrogen bonding. Bleaching effectively removes lignin and hemicellulose, enhancing cellulose purity and reducing O-H stretching intensity. Notably, altered C=O stretching peaks in bleached cellulose improve interfacial adhesion with polymers. The successful acetylation of cellulose is evidenced by decreased O-H stretching intensity and enhanced compatibility due to increased hydrophobicity. However, higher filler loadings weaken dispersion efficiency [9]. The optimal formulation includes 0.5 phr of bleached acetylated cellulose, achieving superior mechanical properties and thermal stability, although excessive CC loading can lead to diminished composite performance. Ultimately, combining bleaching and acetylation significantly boosts cellulose-polymer compatibility and overall performance.

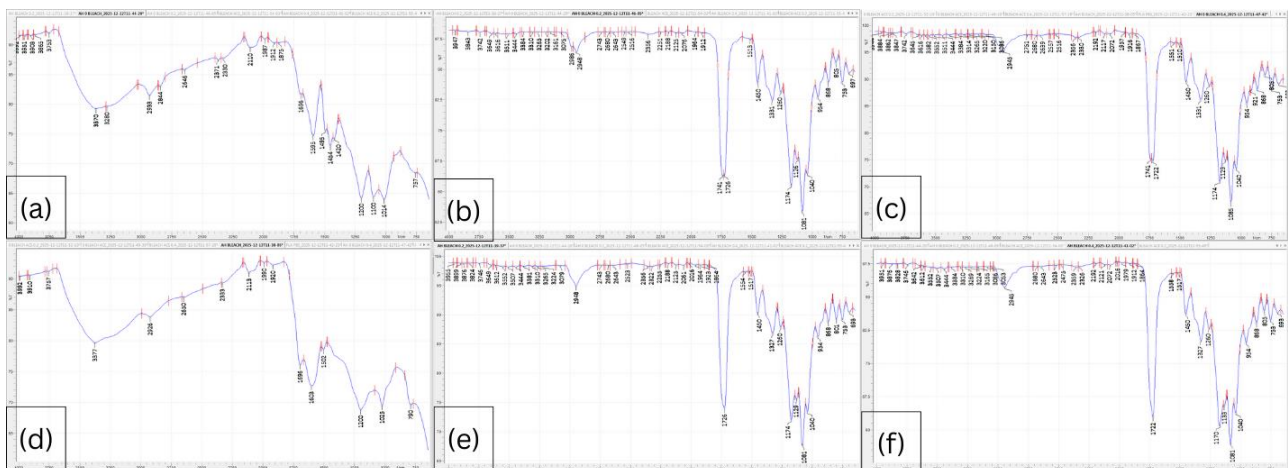


Fig. 3 The FTIR spectrum (a) non bleach acid hydrolysis CC (b) 0.5 phr of non-bleach acid hydrolysis CC mix with polymer (c) 1 phr of non-bleach acid hydrolysis CC mix with polymer (d) bleach acid hydrolysis polymer (e) 0.5 phr of bleach acid hydrolysis CC mix with polymer (f) 1 phr of bleach acid hydrolysis CC mix with polymer

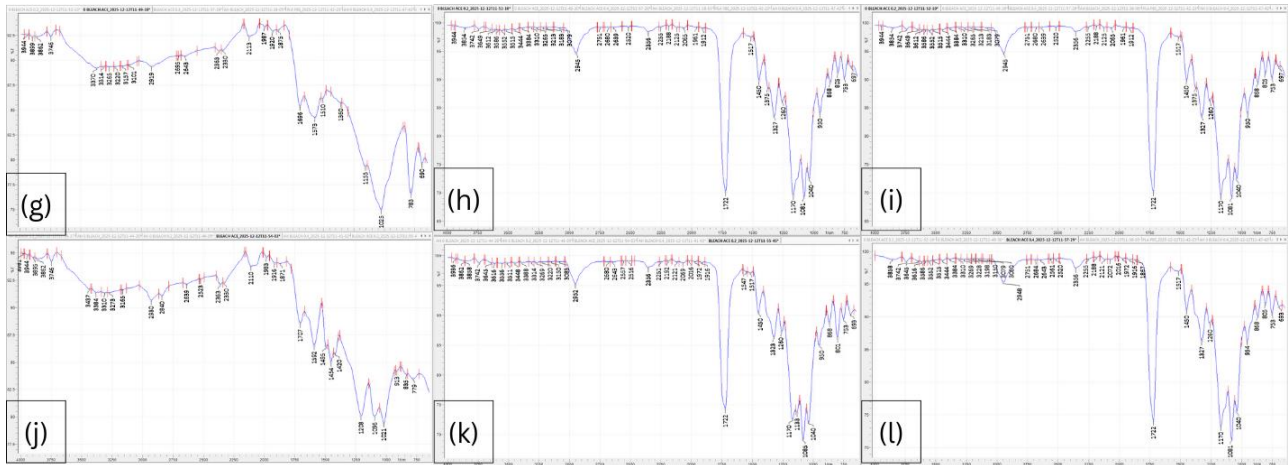


Fig.4 The FTIR spectrum (g) non bleach acetylation CC(b) 0.5 phr of non-bleach acetylation CC mix with polymer (c) 1 phr of non-bleach acetylation CC mix with polymer (d) bleach acetylation polymer (e) 0.5 phr of bleach acetylation CC mix with polymer (f) 1 phr of bleach acetylation CC mix with polymer

3.3 Thermogravimetric Analysis (TGA) towards Thermal Degradation

Thermogravimetric analysis (TGA) was utilized to investigate the thermal stability of raw bamboo and variously treated cellulose samples. Key metrics analyzed included maximum weight loss, char residue, and critical degradation temperatures. Raw bamboo exhibited poor thermal stability with a 97.883% weight loss at 600 °C and only 2.117% char residue, due to the presence of hemicellulose and lignin. In contrast, bleached bamboo demonstrated better resilience, losing 78.628% of its weight and producing 21.372% char residue thanks to its cellulose-rich structure. Acid hydrolysis improved thermal stability for both bleached and unbleached celluloses, retaining over 43% char residue. Notably, acetylated cellulose showed the highest thermal stability, with weight losses of 54.707% for unbleached and 52.982% for bleached versions, attributed to reduced hydrogen bonding. Further analysis of bleached acetylated cellulose at different filler levels revealed that a 0.5 phr sample had excellent heat resistance, while a 1 phr sample suffered from particle agglomeration leading to substantial weight loss. Overall, the findings underscore the significant impact of chemical modifications on the thermal degradation behavior of bamboo-derived cellulose [3]. Acetylation enhances thermal resistance by reducing unstable functional groups and promoting char formation, while acid hydrolysis boosts thermal stability through increased crystallinity. Notably, the 0.5 phr bleached acetylated cellulose demonstrated superior thermal performance, making it a promising candidate for bioplastic composite reinforcement.

3.4 Differential Scanning Calorimetry (DSC) towards Degree of Crystallinity

The study evaluated the thermal behavior and crystalline structure of untreated and chemically treated bamboo using Differential Scanning Calorimetry (DSC). The degree of crystallinity (%X_c) was determined from the melting enthalpy (ΔH_m) obtained from DSC thermograms. The degree of crystallinity can be calculated by using Equation 1 by using the data from melting enthalpy. Chemical treatments such as alkali treatment, bleaching, hydrolysis, and acetylation were found to alter the cellulose structure and affect crystallization. The results indicated that raw bamboo exhibited the lowest crystallinity at 13.5% and a melting enthalpy of 18.2 J/g, primarily due to high levels of lignin and hemicellulose. NaOH treatment improved crystallinity to 19.9% and ΔH_m to 26.8 J/g by enhancing cellulose exposure and aligning chains, while bleaching further increased these values to 23.3% and 31.5 J/g, respectively, by effectively removing lignin and allowing for more efficient packing of cellulose chains. Despite improvements, the crystallinity remained moderate, indicating a need for further structural modifications to achieve optimal ordering [10].

$$X_c(\%) = \left(\frac{\Delta H_m}{\Delta H_{100\%}(1 - \theta)} \right) \times 100 \quad (1)$$

3.5 Tensile Testing on Bio Composites Film

Tensile testing is a critical method for evaluating the mechanical properties of polymeric materials, specifically looking at strength and stiffness. In this case, PLA/PBS composites reinforced with CC from bamboo were intended for testing, but due to limited raw materials and insufficient film production, experimental testing couldn't be performed. However, existing morphological, chemical, and thermal data can provide insights into the tensile behavior of these nanocomposites, aligning with current literature [7][8]. Research indicates that acetylated CC shows improved dispersion within the PLA/PBS matrix, enhancing interfacial compatibility and reducing filler agglomeration, which can negatively impact tensile behavior. The resulting well-dispersed nanofillers are likely to improve tensile strength and Young's modulus, suggesting that composites with acetylated CC will outperform those with unmodified CC. The findings indicate that acetylation of CC enhances its bonding with the PLA/PBS matrix, leading to improved tensile strength through better interfacial adhesion. Low loadings of surface-modified cellulose nanofillers [6][8], typically less than 1 wt%, significantly enhance tensile characteristics without compromising ductility. This study suggests optimal performance occurs with CC loading 0.5 phr, maximizing reinforcement effectiveness. Thermal analyses further support these findings, showing that increased crystallinity in CC-reinforced PLA/PBS composites contributes to higher tensile modulus and strength due to enhanced load-bearing capacity and reduced molecular mobility. The results align with existing literature, demonstrating that acetylated cellulose can increase tensile strength and stiffness by 20–35% while maintaining flexibility, thus supporting the development of biodegradable materials with improved properties.

4. Conclusion

This research focused on creating and characterizing a bioplastic nanocomposite using crystalline cellulose (CC) extracted from bamboo fibre, which was reinforced into a poly(lactic acid) (PLA)/poly(butylene succinate) (PBS) matrix through acid hydrolysis. The study employed systematic chemical, morphological, mechanical, and thermal characterization techniques. The first goal was achieved by using FTIR and FESEM to characterize the effects of acid hydrolysis on the extracted CC, confirming the successful removal of amorphous components such as hemicellulose and lignin. The analysis revealed well-defined, rod-like CC structures, indicating improved cellulose crystallinity. The second objective addressed the impact of CC acetylation on the material's properties, showing that substituting hydroxyl groups with acetyl groups enhanced compatibility with the PLA/PBS matrix, reduced hydrophilicity, and improved mechanical and thermal properties. The results illustrate the potential of acetylated CC in enhancing the performance of biopolymer composites, aligning with findings from previous studies. The study evaluated the chemical and thermal properties of CC-reinforced PLA/PBS nanocomposites using FTIR, DSC, and TGA techniques. FTIR confirmed the compatibility of CC with the PLA/PBS matrix, while DSC indicated that CC acted as an efficient nucleating agent, enhancing crystallinity and improving dimensional stability and thermal resistance. TGA results showed that CC-loaded composites exhibited better thermal properties. FESEM images revealed that acetylated CC was well dispersed in the matrix, contributing to improved mechanical performance. However, higher CC concentrations led to particle aggregation, negatively affecting tensile strength, underscoring the importance of optimizing CC levels. The research demonstrates that cellulose from bamboo is a sustainable reinforcement for PLA/PBS bioplastics, significantly enhancing their mechanical and thermal properties while retaining biodegradability. The findings support the use of these nanocomposites in sustainable packaging applications.

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Conflict of Interest

Authors declare that there is no conflict of interests regarding the publication of the paper.

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