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CHARACTERIZATION OF NANO COMPOSITE FILM BASED ON PLASTICISED POLYHYDROXYBUTYRATE AND POLYCAPROLACTONE BLENDS FILMS FOR PACKAGING APPLICATIONS

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ABSTRACT

This research demonstrates that improving the characterization of Polyhydroxy butyrate (PHB) with 20 wt% Polycaprolactone (PCL), 5 wt% mixed Plasticizer (i.e., monomeric plasticiser (Triacetin 99%) and Polymeric plasticiser (Ultramoll IV), and nanomaterials, i.e., 1 wt% nanocellulose and 6 wt% nano clay, can improve the PHB characteristics for packaging purpose. Biodegradable polymer films were developed using injection moulding and hot-pressing methods, and characterised by tensile properties, water vapor barrier properties, and biodegradation properties in compost and seawater medium. The properties of the nanocomposite films compared to plasticised PHB-PCL 20 films show improved tensile strength, water vapour barrier properties, and slightly higher biodegradation rate with nanomaterials, though with reduced elongation at break. The nanocomposite films were also compared to neat PHB, with 1% nanocellulose (nCell) and 6% nano clay (nClay) providing higher tensile elongation (168% & 146% higher), higher water vapor barrier properties / Lower water vapor transmission rate (46% & 58% lower), and higher biodegradation in home compost (13 % & 11% higher) and seawater medium (10% & 11% higher). The research outcome shows that the high-performance PHB nanocomposite blend with 1% nanocellulose and 6% nano clay.

Keywords: Polyhydroxy butyrate (PHB), Polycaprolactone (PCL), Nano composite, tensile properties, Water vapor barrier properties, Biodegradation properties.

1. INTRODUCTION

Polymers and plastics have been seen as necessary in our everyday lives. However, the excessive use of nondegradable plastics creates environmental pollution issues [1]. Hence, biodegradable polymers, which are eco-friendly, were used to ensure a sustainable future [2]. Biodegradable polymers are particularly used for packaging applications because packaging material significantly contributes to plastic waste. Poly(3-hydroxybutyrate) (PHB) is the most widespread and best-characterised Polyhydroxyalkanoates (PHA) polymer family member [3]. Although biodegradable polymers are used in the commercial market, many commercially available biodegradable polymers do not fully degrade within a reasonable time frame [4]. The primary motivation is that many biodegradable, commercially available products require industrial composting conditions exceeding 50°C for degradation [5]. Data on biodegradation under home compost and marine conditions at ambient temperature are not available for polymer composites. Most of the research data are available for industrial compost conditions. Our research motivation is to expand knowledge in the field of biodegradation, which will benefit future researchers. PHA are a family of biodegradable polyesters produced by bacteria [6]. PHAs are stored as granules in bacterial cells as carbon and energy storage materials. They are commercially produced by bacterial fermentation of renewable materials [7, 8] such as glucose and lipids, under imbalanced nutritional conditions, followed by extraction with solvents. PHB has tensile strength (40 MPa) similar to PP (Polypropylene), and low elongation at Break of 5 – 8%, high melting temperature (178°C), and the glass transition temperature is 4°C, which is higher than PP. The water vapor transmission rate (WVTR) is about 158 – 217 g m^{-2} day^{-1}, which is higher than that for the traditional polymer [9]. The PHB is fully biodegradable in both compost and marine conditions without forming any toxic products [3]. PHAs are reported to degrade in a wide range of temperatures, but not at ambient temperature. The degradation of nanocomposite films with PHB at ambient temperature was limited, despite several studies focusing on the nanocomposite, as shown below. Nano composites were prepared by blending PHB with adipate placticizer proviplast (Bis[2-(2-butoxyethoxy)ethyl] adipate) along with 2,5 and 10% cellulose fibers, and carried out studies on thermal and barrier properties (WVTR), and the studies shows that a reduction of melting temperature, crystallinity, and reduction in water barrier properties (increase in WVTR) with cellulose fibres [10]. A higher disintegration rate with plasticisers and a slower disintegration rate with nano-cellulose, which was investigated by nano-composite films based on PHB, incorporated with two types of nano-cellulose (2, 4 wt%), bacterial cellulose (BC) or cellulose nanocrystals (CNC), and plasticized with tributyrin or a polymeric plasticizer poly (adipate diethylene), and the studies involved several characterisation such as morphology, thermal properties, and disintegration under composting conditions [11].

The investigation of nano-composites of PHB incorporated with two types of cellulose (cellulose nanocrystals-CNC and bacterial cellulose-BC) along with two different plasticizers (glyceryl tributyrate-TB and a polymeric plasticizer poly[di(ethylene glycol) adipate) [12] shows that the plasticized PHB nanocomposites prepared by adding CNC and BC nanoparticles were macroscopically more homogeneous and that bacterial cellulose was not as uniformly dispersed as CNC, probably because of its large aspect ratio. The results showed that plasticised nanocomposites' tensile properties were not much different from those of pristine PHB due to the opposing influence of plasticiser and nanoparticles. The plasticised film shows that the addition of plasticisers reduced barrier properties, which was partly countered by the improved barrier properties with nano-cellulose. However, there was a net increase in WVTR compared to neat PHB [12]. Pracella et. al investigated the nanocomposites of polyhydroxybutyrate (PHB) and poly(hydroxybutyrate-co-hydroxyvalerate) (PHBV) with cellulose nanocrystals (CNCs), which were prepared by dispersion of CNCs into (aqueous) poly(vinyl acetate) (PVAc) emulsion or poly(ethylene glycol) (PEG) solution, followed by melt mixing of dried PVAc/CNC and PEG/CNC masterbatches with neat polyesters [13]. The results indicated that the polymer blending procedure of PHB and PHBV enhanced the CNC dispersion into the polymer matrices and modified the phase morphology and properties. Adding PVAc for PHB and PHBV nanocomposites resulted in higher tensile strength and elongation at break by increasing the CNC content.

The modified nano clay is also used to develop the nanocomposite films in several studies. Nano-composite films based on PHB and PHBV using different loadings of organo-modified montmorillonite (OMMT-Cloisite 10A) clay, and studied mechanical, thermal, and water vapour permeability properties [14]. This study shows that the composite films' properties were highly dependent on the concentration and dispersion of clay in the polymer matrix, and the results show that enhanced water vapour barrier properties/ reduction in WVTR by 41% for PHB-based composite films. They also observed an improvement in the mechanical properties, tensile strength and reported that the elongation at break increased by 152.3% and 77.4%, respectively, with 3% nano-clay [14]. Three different types of nano clays are used to prepare the nanocomposite of PHB, and the results indicate improvement in mechanical properties/Young's modulus (by about 40%) in nanocomposites, compared to the neat polymers. Biodegradation studies suggest that the decomposition rate varies with the type of nano-clay and surface hydroxyl groups in the clay [15]. Only a few studies have been conducted on nanomaterials, and there is a lack of detailed information about their morphology, barrier properties, and biodegradation properties.

Nanocellulose is an attractive biodegradable material for preparing nanocomposites of biodegradable polymer; however, little research has been done on nanocomposites of biodegradable polymers using nanocellulose. Most research has been done on organically modified nano clay to prepare the nanocomposite. Only minimal research has been done on nanocomposites based on plasticised PHB PCL blends with plasticisers. A comparative analysis of the effects of nanocellulose and nano clay on these polymer blends is lacking. It is necessary to analyze the biodegradation of plasticised polymer blends in both compost and marine environments under home composting conditions. Few studies have been reported on the biodegradation of PHB in marine conditions, and further research is needed to understand the biodegradation of plasticised polymer blends in the aquatic environment. The primary objective of this research is to develop nanocomposite blends with plasticised PHB–PCL and characterise the biodegradable polymer blend films for flexible packaging applications. The sub-objective of this research is to fabricate nanocomposite films by incorporating different loadings of nano-cellulose and nano-clay, and to compare the performance of the two nanomaterials based on several characteristics, such as tensile properties, water vapour barrier properties, and biodegradation properties, in both compost and marine conditions at ambient temperature. The present work used injection moulding and hot-pressing methods to fabricate the nano-composite based plasticised PHB-PCL blends. The study compares the performance of nano cellulose and nano clay on these plasticised polymer blends by tensile properties, water vapour barrier properties, and biodegradation properties in compost and seawater medium, at ambient temperature for 180 days, as per the American Society for Testing and Materials (ASTM) standards [16].

2. MATERIALS AND METHODS

The PHB resin (ENMAT Y3000P PHB), having a melting temperature of 175-180°C, glass transition temperature(Tg) of 4°C, and Mw = 350000 g/mol, was obtained from TianAn Biologic Materials Co., China [16]. The polycaprolactone (Capa 6500) resin was purchased from Pertstrop, USA, which has a melting temperature of 60-62°C, glass transition température (Tg) of -60°C, and weight average molecular weight (Mw) of 84500 g/mol [17]. Plasticizer such as triacetin (99%) (GTA) was purchased from Sigma Aldrich, New Zealand, with a molecular weight 218.20. Ultramoll IV (UM) was supplied from Lanxess, Australia, a polymeric plasticiser based on an adipic acid polyester. Nanomaterials such as nanocellulose (cellulose nanofibrils, CNF), which have a 20-60 nm diameter and a length of a few micrometers, were purchased from a cellulose lab in Canada. Nano clay (Dellite 72T), an organically modified montmorillonite clay, was purchased from Laviosa Chimica Mineraria. The compost was purchased from Daltons, New Zealand (NZ), and seawater was collected from Browns Bay in Auckland, NZ [17].

TABLE 1: COMPOSITION OF NANOCOMPOSITE FILMS BASED ON PLASTICISED PHB-PCL BLEND.

Material designation	PHB content (wt%)	PCL content (wt%)	Plasticizer (wt%) With respect to PHB – PCL Blends	Nanomaterial (wt%) With respect to PHB – PCL Blends
PHB-PCL20- P3-nCell 1	80	20	5%*	1%**
PHB-PCL20-P3-nCell 3	80	20	5%*	3%**
PHB-PCL20-P3-nCell 6	80	20	5%*	6%**
PHB-PCL20- P3-nClay 1	80	20	5%*	1%***
PHB-PCL20- P3-nClay 3	80	20	5%*	3%***
PHB-PCL20-P3-nClay 6	80	20	5%*	6%***

*(GTA – UM (50/50)), **NANO CELLULOSE, ***NANO CLAY.

The PBS and PCL polymers were dried in an air oven at 50℃ for 12 hours to prevent hydrolysis during thermal processing. Polymer Blends with nanocomposites were prepared by melting the polymer resins in a hot blender using a slow-speed stirrer, [16] The blending temperature was maintained at about 120°C [18]. Nanocomposite blends were prepared with various compositions shown in table 1. Thick circular sheets of 2.5 mm thick nanocomposite blends were prepared by injection moulding and hot-blended resins at 120℃ [17]. Nanocomposite films (thickness of 0.25 mm/~ 10 mils) were prepared by the hot-pressing method from the thick polymer sheets prepared as per table 1 [17]. The thick sheets of polymer were taken into a mould, heated at 120℃, and then pressed with a precision temperature control device into a hydraulic press at a pressure of approximately 1MPa [16]. Hot pressing was performed for three minutes and then cooled to room temperature [16].

The surface morphologies of the nanocomposite samples were observed by Hitachi SU-70 scanning electron microscope instrument operated at 10Kv [16]. The Nanocomposite samples were coated with Platinum before starting the experiment [17]. A TA.XT PLUS tensile machine fitted with a 5 KN load cell was used for tensile testing [16]. The tensile test used a rectangular strip of nanocomposite films with dimensions 0.25mm in width* 10mm in length* 100mm in height [17]. The nanocomposite film sample's tensile Properties were established using the ASTM D882 method, with slight modifications [16]. The water vapor transmission rate (WVTR) was established by a gravimetric method called the cup method. This research followed the water cup method, as per ASTM E96 [17]. Biodegradation studies for nano composite films were carried out in compost medium as per ASTM D5338-15 method, with slight modifications such as temperature for testing. The studies were carried out in seawater medium, as per the ASTM D6991-17 method [19]. Both studies were conducted under aerobic conditions and ambient temperature. Whatman filter paper (cellulose) was used as a reference for biodegradation studies in compost and seawater medium [16]. The experimental setup for the biodegradation studies was similar to the carbon dioxide evolution test in both the compost and the seawater medium [20].

3. RESULTS AND DISCUSSION

A review of a previous study [17] is summarised in Figure 1 and showed that PHB with 20% of PCL has tensile elongation of 68%, and the water vapour barrier properties of 26.8% more than pure blends of PHB. The biodegradation rate in compost medium was increased by 6.4%, and the biodegradation rate in seawater medium was increased by 7.5%, even though there was a slight decrease in tensile strength. In conclusion, the polymer blends with 20 wt% of PCL provide overall improved polymer properties [17]. The previous investigation about pure PHB and PHB–PCL blends helps to compare the characteristics with the nanocomposite film.

Previous research shows that compared to plasticised PHB with 20 wt% PCL blends, with polymers blends PHB, with 20 wt% of PCL, there was a significant increase in tensile elongation of 76.6% and an increase in biodegradability by 3.6% in compost medium and 1.8% in seawater medium, respectively, with the addition of all three 5wt% plasticisers (Ultramoll). However, the tensile strength and water vapour properties were decreased. The plasticiser study shows that plasticised polymer blends using mixed plasticiser (P3) enhance overall performance [16]. The previous investigation about plasticised PHB–PCL blends helps to compare the characteristics with the nanocomposite film.

This section focuses mainly on nanocomposites of plasticised PHB-PCL20 blend films prepared using nanocellulose (nCell) and nano clay (nClay) with different loadings of 1, 3, and 6 wt% with respect to the PHB-PCL 20 blend. It also analyses several characteristics of nanocomposite films, such as mechanical properties, water barrier properties, and biodegradation properties in both compost and seawater medium at ambient temperature. The properties of the nanocomposite of plasticised PHB-PCL 20 blend films were compared to plasticised PHB-PCL 20 Films [16]. Finally, the overall performance is compared to the properties of the nanocomposite films to pure PHB film [17]. Figure 1 shows photographs of the nanocomposite of plasticised PHB-PCL20, which shows 1, 3, and 6% of nanocellulose and nano clay.

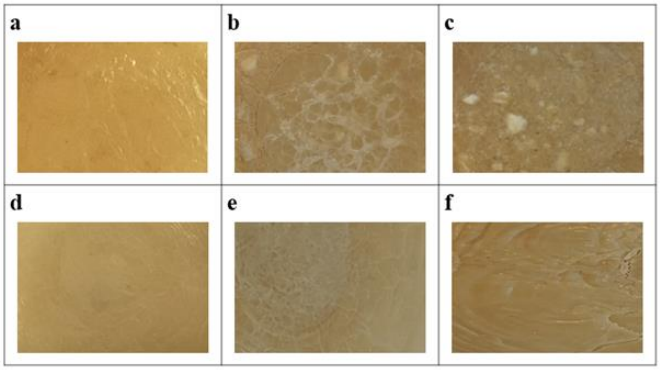

Figure 1: PHOTOGRAPHS OF NANO-COMPOSITE FILMS BASED ON PHB-PCL a) PHB-PCL20- P3-NCELL 1, b) PHB-PCL20- P3-NCELL 3, c) PHB-PCL20- P3-NCELL 6, d) PHB-PCL20- P3-NCLAY 1, e) PHB-PCL20- P3-NCLAY 3, f) PHB-PCL20- P3-NCLAY 6.

3.1 Mechanical Properties of Nanocomposites:

Nanocomposites of a plasticised PHB-PCL20 blend, with different nanocellulose and nano clay loadings, were prepared to improve barrier properties. However, the effect of these nanomaterials on tensile properties was evaluated, and the test results are presented in figures 2 and 3. A nanocomposite based on nano-cellulose exhibited a slight increase in tensile strength with the addition of nanomaterials. Still, the tensile strength was higher with 1 wt% of nanocellulose, and in the case of the nano clay, the highest tensile strength was obtained with the highest addition of nano clay. The slight increase in tensile strength with the addition of nano materials is due to the reinforcement of the polymer chains with nanomaterial particles [21].

Diez Pascal et al., (2014) research that the nanoparticles interfere with chain regularity and nucleation kinetics, generating more small and less perfect crystals, which contribute to energy dissipation under mechanical stress [22]. In polymer films, molecular transport takes place almost exclusively in the amorphous region due to its less defined molecular structure and accordingly higher free volume than the crystalline phase, leading to a decrease in barrier properties of the material. Therefore, there is a delicate balance between barrier properties and mechanical properties [23]. Mohapatra et al., in PHB / Cloisite nanocomposites, blending led to reduced crystallinity and altered lamellar structures. This disruption in the crystalline regions resulted in enhanced toughness and ductility at the expense of tensile strength, mainly due to the more amorphous character of the polymer matrix and better dispersion of nanofillers, which hinder crystal growth and perfection [24].

The elongation at break of nanocomposites, nano-cellulose and nano-clay addition resulted in a decrease in elongation at break, with both the nanomaterials, the initial addition of 14% of the nanomaterials, resulted in a very slight reduction in elongation at break, only, the values were very near to that of the blend without nanomaterials. A greater decrease in elongation at break was observed with nano-cellulose compared to nano-clay at higher loadings. A reduction in tensile elongation with the addition of nanomaterials may be due to decreased polymer chain mobility due to the presence of nanomaterials, and a more considerable decrease at higher concentrations may be due to the non-uniform distribution of nanoparticles [25]. Figure 4, shows SEM Images of the film without nanomaterials, and the nanocomposite film with 1 and 6 wt% of nanocellulose and nano clay. Though difficult to interpret, the SEM images of nanocellulose with 6 wt% show a non-uniform distribution with more phase separation, correlating with the observed lesser ductility.

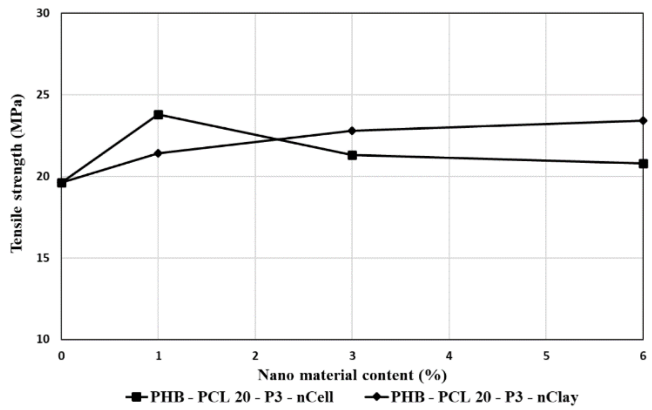

FIGURE 2: TENSILE STRENGTH OF NANOCOMPOSITE FILMS BASED ON PHB–PCL

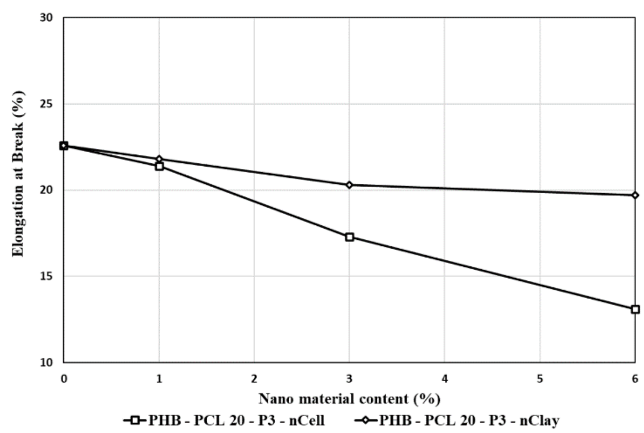

FIGURE 3: ELONGATION AT BREAK OF NANOCOMPOSITE FILMS BASED ON PHB–PCL.

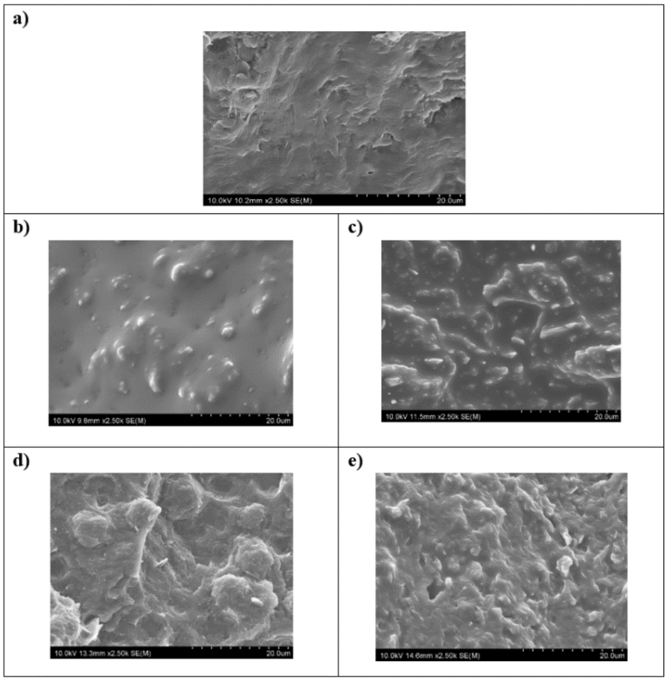

FIGURE 4: MORPHOLOGY/ SEM OF NANO COMPOSITE FILMS BASED ON PHB–PCL a) PHB-PCL20-P3, b) PHB-PCL20-P3-NCELL1, c) PHB-PCL20-P3-NCELL6, d) PHB-PCL20-P3-NCLAY1, e) PHB-PCL20-P3-NCLAY6.

3.2 Water Vapour Transmission Rate (WVTR) of Nano Composite Films Based on PHB PCL:

Nanocomposites of PHB-PCL were prepared, mainly to improve the water barrier properties, and WVTR tests were carried out to understand the effect of nanomaterials and the results obtained with 1 3 and 6 wt% of nano-cellulose and nano-clay are presented in Figure 5.

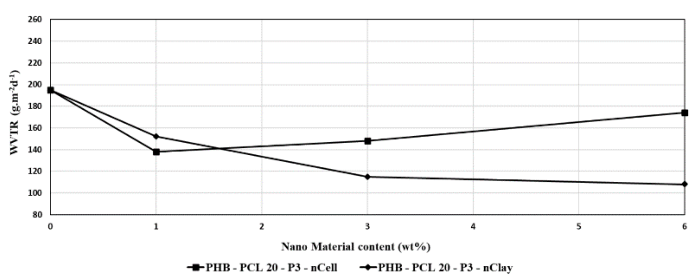

FIGURE 5: WVTR OF NANO COMPOSITES FILMS BASED ON PHB-PCL, AT 25℃, 100% RH, 25μM FILM THICKNESS

Comparison of the WVTR of nanocellulose and nano clay indicates that, though the lowest WVTR was obtained with nano-clay (with 6 wt% loadings), the value of WVTR obtained with a lower dose (1%) of nanomaterial was lower in the case of nano-cellulose. In other words, nano-clay provided a better water barrier property than nano-cellulose at a lower dose of nanomaterial. The higher barrier property/ lower WVTR obtained with nanocomposites is due to the creation of more torturous pathways [26]. Although an excellent barrier property was obtained in the case of nanocellulose, the decrease in barrier property observed at higher concentrations may be due to the agglomeration of nanocellulose and the creation of microchannels [27].

3.3 Biodegradation Characteristics of Nanocomposite Films Based on PHB–PCL:

The biodegradation test results of PHB-PCL nanocomposites with 1 wt% of nanocellulose and 6 wt% of nano clay in home compost are shown in Figure 6 and seawater medium in Figure 7. The test results indicate a slight increase in the biodegradation rate with the nanomaterials in both compost and seawater medium. The biodegradation rate of the nanocomposite with nano-cellulose (1 wt%) was higher than that of the nanocomposite with nano-clay (6 wt%) in the compost medium. Compared to the blend without nanomaterials, the results show a slight increase or almost the same biodegradation of nanocomposites in seawater medium. Thus, in the compost medium and seawater medium, only a minimal increase or no increase in biodegradation rate was observed in the case of PHB based nanocomposites, which may be due to the action of two opposing factors, increased surface area due to the nanocomposites may be promoting faster biodegradation, while increased barrier properties (lower WVTR) of nanocomposites may be retarding the biodegradation. As a result, there was a slight increase or no increase in biodegradation for PHB-based nanocomposites.

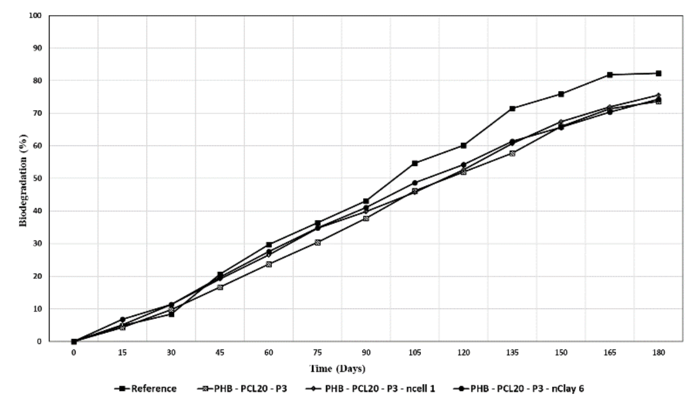

FIGURE 6: BIODEGRADATION CURVE OF NANOCOMPOSITE FILMS BASED ON PHB-PCL, IN COMPOST AT 25℃.

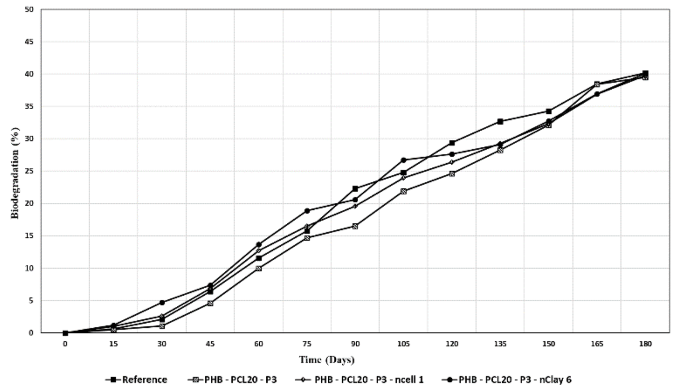

FIGURE 7: BIODEGRADATION CURVE OF NANOCOMPOSITE FILMS BASED ON PHB-PCL, IN WATER AT 25°C.

The current research shows several features, such as

When compared to the plasticised PHB PCL20 blends [16], PHB nanocomposites demonstrated improved tensile strength, improved water vapour barrier properties (decrease in WVTR), and a slightly higher biodegradation rate with nanomaterials, though with reduced elongation at break.

When compared to neat PHB [17], nanocomposite films with 1% nanocellulose (nCell) and 6% nano clay(nClay) provide higher tensile elongation, higher water vapour barrier properties / lower water vapour transmission rate, and higher biodegradation in home compost and seawater medium.

The studies also indicate that the best overall performance was obtained with 1 wt% nanocellulose and 6 wt% nano clay loading for the nanocomposites.

3.4 Summary:

This paper mainly focuses on improving the properties of the plasticised PHB–PCL blends by blending them with (1, 3, and 6 wt%) nanomaterials such as nano cellulose and nano clay. PHB nanocomposite blend films (thickness 0.25mm) were prepared by injection moulding and hot pressing.

The properties of the nanocomposite of plasticised PHB-PCL 20 blend films were compared to plasticised PHB-PCL 20 Films [16], which shows improved tensile strength, improved water vapour barrier properties (decrease in WVTR), and a slightly higher biodegradation rate with nanomaterials, though with reduced elongation at break.

Finally, the properties of the PHB nanocomposite films were compared to those of neat PHB [17], and find that nanocomposite films with 1% nanocellulose (nCell) and 6% nano clay(nClay) provide higher tensile elongation, higher water vapour barrier properties / lower water vapour transmission rate, and higher biodegradation in home compost and seawater medium.

The PHB nanocomposite films with 1% nanocellulose (nCell) and 6% nano clay(nClay) have the best characteristics in all properties.

4. CONCLUSION

The present investigation shows that the overall performance improvement of polymer PHB through PLC blending, plasticization, and the addition of nanomaterials. Additionally, the research outcome suggests that PHB nanocomposites, specifically those with compositions PHB-PCL20-P3-nCell1/nClay6, exhibit high-performance characteristics for flexible packaging applications, potentially after minor improvements. In this present work, however, oxygen and carbon dioxide barrier /permeability characteristics are also essential for packaging films, polymer film barrier properties for water vapor transmission rate only were studied due to time limitations, the future investigation may consider using the surface-modified nano-cellulose to improve the dispersion / reduce the agglomeration for enhanced performance with nano-cellulose.

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