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Biopolymer nanocomposites: their mechanical, thermal, and gas barrier properties for food packaging

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Abstract. The food packaging industry is the largest sector of the plastic industry, accounting for 40% of the total (conventional) plastic demand. Conventional plastic packaging has significant practical advantages, in protecting packaged foods from contamination and the loss of food quality. Plastics are made up of repeating small units called 'monomers' that bind together to form the long chains called 'polymers'. However, conventional plastics have detrimental effects human health, wildlife, and the environment. Many developing countries are overwhelmed with the plastic waste emitted to the oceans. Indonesia is the second biggest emitter of plastic waste, after China. Biodegradable plastics (bioplastics, green plastics) are an alternative for saving our environment. Bioplastics or biopolymers, are made from at least 60% natural materials, mostly of plant based origin, but they have several drawbacks: (i) lower mechanical properties; (ii) bad thermal stability; (iii) relatively low gas barrier properties. This lab-based study investigates the enhancements to mechanical, thermal and gas barrier properties of bioplastics through addition of nanocomposites derived from clay minerals (nano clay). The resulting product was examined under Scanning Electron Microscope (SEM). This paper summarizes the clay concentrations and mixing temperature conditions that are required for the enhancement of bioplastics.

1. Introduction

The food packaging industry is the sector of the plastic industry, accounting for 40% of the total (conventional) plastic demand. Conventional based plastic packaging has practical advantages in protecting packaged foods from contamination and the loss of food quality. Plastics in general are made up of repeating small units being called 'monomers' that bind together to form long chains called 'polymers' (Figure 1). The most commonly used polymers in the field of food packaging are synthetic polymers *e.g.* polyethylene, polyethylene terephthalate (PET), polyvinylchloride (PVC), which are used for making grocery bags [1]. After their uses, it is extremely difficult to get rid of these plastics because they do not degrade easily or at all. These conventional plastics have detrimental effects on human health, wildlife, and the environment [2]. The most worrying ones are: i) they are health hazardous materials (carcinogenic substances); ii) they can break break into smaller pieces and to infiltrate food webs, as some living organisms are able to ingest micrometer sized plastic particles [3].



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Many developing countries are overwhelmed with the plastic wasteemitted to the oceans likewise in Indonesia. Indonesia is the second biggest plastic waste emitter to the seas, after China. Gross [4] estimated that less than one third of the plastic produced until 2015 was still in use, while the accumulated waste amounts to over 6,000 Million tons (Mt), which is more than enough to wrap our earth with a thin layer of plastic film.

Biodegradable plastics (*bioplastics*, green plastics) are an alternative that could save our environment (Figure 1). Bioplastics, or *biopolymers*, are made from at least 60% natural materials, mostly of plant based origins derived from a) biomass; including plant starch, cellulose, seaweeds, or plant-based protein; b) microorganisms; c) bio-derived *monomers* obtained from biotechnology that serve as the building blocks of *polymers*. Such long bio-polymers can be, converted into simpler organic compounds, which degrade in soil medium within one month. Some have been well recognized and explored commercially, *e. g.*: bio-based polyethylene (PE), bio-based *polyethylene terephthalate* (PET), and bio-based *polyamides* (PA). Polyethylene furanoate (PEF) is expected to enter the market in 2023 and has high potential as material for bottles in the beverages industries [5], [6].



Figure 1. Overview of this current study: (i) definition of '*monomers*' as the building blocks of *polymers*, (ii) the matrix of biopolymers reinforced with nano clay.

Nevertheless, bioplastics have several drawbacks, *e.g.*: (i) lower mechanical properties; (ii) bad thermal stability; (iii) relatively low gas barrier properties; in other words, high moisture absorption; (iv) consumed more time for those products obtained *via* genetically modified corn or potato. The bad thermal stability and high moisture absorption are particularly problematic for usage in the food packaging industry. Thus, reinforcement with materials made from both natural and non-natural fibers is necessary in order to enhance the mechanical stability and thermal properties (Figure 1). Natural fibers include sugar-cane, kenaf, ramie, pineapple leaf, *Oil Palm Empty Fruit Bunches* (OPEFB), hemicellulose based on wood-lignin, and nanocomposites derived from altered volcanic ash clay. Commonly used non-natural fibers are mica and silicate [2], [5].

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Nanocomposites have been discussed in material science since the early 1990's; they are materials with nanoscale reinforcement with particle sizes less than 50 nanometers. They are used in the plastic industries to improve mechanical properties, increase thermal stabilities, and decrease gas permeability, which is valued in the packaging industry [1], [5]. *Nano-clays* contain carbon nanotubes, which are *intercalated* (inserted) between the existing layers in the polymer matrix after the mixture (Figure 2). Researchers in Toyota Central Research Lab. in Japan invented the *nylon-6*-nanocomposites, which marked a revolution in material science. Nowadays, research in nanocomposites has been applied to a wide array of applications, including agrochemicals, cosmetics, coating technologies, food sheet packaging, and structural materials for buildings. The market for nanocomposites in Europe is currently valued at \$20 billion and expected to grow substantially. Production numbers are projected to grow from 2.1 Mt to 2.4 Mt from 2019 to 2024; a lot of that growth is attributed to the food packaging industries [8].

This study aims to address some of the drawbacks of bioplastics, therefore we investigated the mechanical and thermal enhancement, as well as gas barrier properties of biopolymers enhanced with nanocomposites (5-7 % w/w) derived from clay minerals. The morphology of the products was closely examined under *Scanning Electron Microscope* (SEM) in order to see how fine they were dispersed on the biopolymer matrix.

2. Materials and methods

2.1. Materials

Two kinds of commercial bioplastics were applied in this experiment: Bioceta[©], which is derived from cellulose based material (2.1.1), and 'Mater-BiTM ZF 03U/A', which is a cassava starch based polymer (2.1.2). The nano clay applied in this experiment was I.30 TC (2.1.3).



Figure 3. Chemical structure of cellulose diacetate or Bioceta.

2.1.1. Bioceta. Bioceta is an abbreviation for Biodegradable-Cellulose-Diacetate (Figure 3). The cellulose diacetate originates from wood or cotton linters with a chemical composition of C= 50.5%; H= 6.4%; O= 43.1%. Its incineration produces only non-toxic carbonic gases and water; with a speed of degradation comparable with an oak leaf. It degrades easily after contact with the soil bacteria, soil humus, and mushrooms. It is mostly used in food packaging films, due to its transparency as thermoplastic material, non-hazardous biofilm with good dimensional stability up to 70 °C. However, Bioceta has not been adopted widely, because of its high price and low thermal properties. Due to the limited capacity of the kneading batch machine's mixing container, we applied two 200 g samples of Bioceta.

2.1.2. Mater-Bi[©] ZF 03U/A or referred here as Mater-Bi. Materbi is actually polycaprolactone (PCL), which is derived from (cassava) starch and produced by Novamont (Figure 4). It is hydrophilic and tends to absorb or lose moisture depending on the environmental condition. Thus, it is highly recommended for moisture barrier packaging especially for shipment or for plastic bags that are easy to recycle. Nevertheless, the weak mechanical properties of Mater-Bi need to be improved for a wider utilization in the food packaging industry. Due to the limited capacity of the kneading batch machine's mixing container, we applied two 200 g samples of Mater-Bi.



Figure 4. Chemical structure of polycaprolactone or PCL.

2.1.3. Nano clay: I.30 TC. Nano clay with a phylosillicate or sheet structure was applied as an additive. It has a 2:1 layered structure; each layered sheet is slightly < 1 nanometer (nm). The spacing between each sheet is termed as 'gallery' and has a size of 3*5 Angstrom (Å). Clays are found in white or yellow powder or fine-grained material. They are able to absorb water and the water molecules can readily enter the gallery structure. Concentrations of hydrated sodium calcium aluminum silicate [(Na, Ca)(Al, Mg)₆(SiO₁₀)₃ (OH)₆- n H₂O] of 3.5% and 7% (w/w) were applied. In order to compare the efficacy of nano clay, chalk (CaCO₂) was also applied and added in the Bioceta and served as blank sample or standard [9].

2.2. Methodology

The production of biopolymer nanocomposites was conducted at the Wageningen University and Research (WUR), in the Netherlands at the Research & Development Unit for Polymer led by Ir. G.J. Schennink. Four steps applied: (i) mixing the two major components into granules; (ii) forming or molding the materials into desired forms suitable for further tests; (iii) testing the materials based on their mechanical, thermal, and gas barrier properties [9]; (iv) analyzing their morphology under the *Scanning Electron Microscope* (JSM 5600 LV-SEM, JEOL) (Figure 5). Each sample was prepared in replicates that was served for the mechanical, thermal, and gas barrier properties; *except* for the Scanning Electron Microscope (SEM) due to the high cost for preparing one sample, as a coverage of pure gold (**Au**) on the sample was necessary (further explanation: Point 2.2.5.).



Figure 5. The four steps applied in this study.

2.2.1. Mixing bioplastics with nano-clay. Nano-clay with 3.5% and 7.0% w/w was mixed with the 200 g of Bioceta and Materbi samples, this corresponds to 7.25 g and 15.05 g of nanoclay [9] (Table 1). First, the kneading machine was heated up to 50-75 °C. After the pre-heating, the bioplastics (one of each) were put in the 'batch kneading machine' (Haake, Rheomix 3000p – Poly Lab. System) and kneaded for five minutes. After five minutes, the clay was added and mixed for another two minutes in the matrix of biopolymers in order to alter the properties of the materials and prevent degradation of the polymer during processing.

Table 1. Ingredients of bioplastics, weight of nano-clay, shear force, time of mixing.

No.	Clay ^š Concentration (%w/w)	Temj (perature (°C)	Shear Force – (rpm¶)	Time of mixing (min.)		
	-	Bioceta	Materbi**				
1.	0	190	125	75	7.0		
2.	3.5	170	105	75	7.0		
3.	3.5	190	125	75	7.0		
4.	3.5	210	145	75	7.0		
5.	7.0	190	125	75	7.0		

[§]Clay: I.30 TC, ¶rpm= rotation per minute, ** Materbi required much lower temperatures than Bioceta, because it easily gelatinizes under temperatures above 170 °C; we found this through trial and error conducted prior to the major experiment. Therefore, a uniform condition between the two samples was not possible [9].

2.2.2. Forming the granules into bars or foils/sheets. The materials were cooled down to room temperature (T=25 °C), afterwards they were granulated with a granule-machine (Lenze, IP 54, Lovato). The granules were reserved for further mechanical, thermal and WVP Property Tests.

2.2.3. Mechanical and thermal property test. First, 'dumb-bell' type or plastic bars with a size of 6.5 cm length; 0.5 cm width; 2 mm thick were made from the granules after they were injected in the injection-molding machine (Elenac, Batch No. 9/ 43/ 993184/ 227: 9851 001). These were intended for further testing of mechanical and thermal properties.

The first test we conducted was the mechanical one, which was performed on a *Zwick* test machine (Z 0101). The equipment was directly integrated with a personal computer and connected to a printer. The bars were subjected to an *axial force* until breakage according to UNE-EN ISO 527-2 standard [9]. The tests were conducted in a climatized chamber with a constant temperature (T= 20 °C) using an extensional rate of 10 mm/ min. The equipment detects when the plastic bars break and immediately presents the values of maximum tensile stress [σ], strain at the break [\in] and the Young's Modulus [E= σ / ϵ] on the monitor (Figure 6). The measurement unit for 'E' is noted in Mega Pascale (MPa) or N mm⁻²[9]. The second test was the 'thermal property test', also known as *Heat Deflection Temperature*

or DMTA. There are some distinct differences between the DMTA and mechanical test: i) DMTA is a non-destructive test, ii) very small strain is involved, iii) the small strain involved does not exceed the maximum tensile strength of the samples, iv) temperature scanning method is applied. The equipment reserved for this purpose was Rheomix Luc. RS II, which enabled us to detect the melting temperature of material (Tg). The Rheomix Luc. was also directly integrated with a personal computer and connected to a printer.



Figure 6. The three pictures present the explanation of 'Young Modulus' or 'E' presented with the typical stress-strain diagram at the right side; while the definition of E is stress divided by strain (stress is indicated with the blue star, strain is indicated with the red one); source [9].

2.2.4. Water vapor permeability (WVP) Test. Prior to the WVP conducted, we made sheets or foils first derived from the granules at the beginning. They were subsequently put into a press-molding machine (PHI, Serie No. : 93-07-010; model No.: 75 UI 2095 CJCS J-Y2-S5-7) and then square shaped foils with the following dimensions we produced: length*width= 7.5 cm* 7.5 cm and 2 mm of thickness.

The WVP test aimed to determine the gas barrier property and was conducted with this method: i) approximately 17 g of distilled water (H₂O) was filled in a special designed acrylic cup and then, the full weight (cup and H₂O) was weighed on a digital scale (two digits after comma, Sartorius, Made in Germany) and noticed as *W1* in grams (g); ii) foils were put on the acrylic cup and fitted with four special screws placed at the four corner; iii) cups were evaporated for 7 days in an oven [T= 20 °C, Relative Humidity (RH)= 60%], iv) the left-over distilled water, the foil, and the cup were weighed and noted as '*W2*' in (g) (equation 1). The water loss was indicated in [%] (see: equation 1). Thus, the final standard unit for WVP was determined in g. (ms.Pa) ⁻¹. Each sample was made in replicates, while the average of two values served for further statistical analysis.

WVP (in %) =
$$\frac{W1 - W2}{W1}$$
% (1)

2.2.5. Scanning electron microscope (SEM). The morphology was analyzed with a Scanning Electron Microscope (SEM) [JEOL, Code JSM 5600 LV]. It is worth noting that prior to observation, the samples were first subjected to sputtering treatment with palladium or pure gold in order to enhance the electrical conductivity of the nanocomposites as well as the quality of micrographs [9]. The lowest magnification of 2,500x was first applied, then continued with stratified and stronger magnifications [5,000x, 10,000, and 20,000x (x= times)] to examine the dispersion of the clay within the biopolymer matrix. Here, the clay was indicated as small white dots on the matrix (Figure 7, 8).

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2.3. Statistical analysis

Simple statistical analysis was conducted *via* Microsoft Excel for calculating the mean values (averages), standard deviation (STD), and percentage (%).

3. Results and discussion

Advancement in material technologies have become more sophisticated with a trend toward refinement of features and functions at the nanoscale level. Nanotechnology has replaced conventional 'macroscopic' matrix fillers, such as silicate, mica, and other short fiber technology, because of their advantages in terms of mechanical, thermal, and gas barrier properties [10]. This lab.-based study shows the possibility of nano composites as reinforcement material in the matrix of bioplastics, which are used in the packaging industry, for wrapping, compostable plastic grocery bags, and for bottles in many beverages (particularly in the Netherlands).

Table 2 presents the mechanical, thermal, and gas barrier property tests results from our experiment. The experiment with the injection molding shows that bioplastics can be directly processed with molding's temperature of 70 °C but not lower than this. Lower temperature condition is more suitable for cassava starch based biopolymer (Materbi) with an average of Tg at 66,60 °C [Standard Deviation (STD) = 3 °C]. The average of *Young Modulus* (E_{value}) is 176.60 Nmm⁻² (STD= 10 Nmm⁻²), which is relatively low. The reinforced Bioceta materials have 7.4x and 8.3x E_{value} than the Materbi.

These two parameters confirm the drawback of starch based plastics (in this case Materbi) compared to conventional ones in terms of mechanical and thermal properties. Interestingly, the starch based plastics had almost up to nine times higher values of *Length (L) at stress max* (31.98 %) and *Strain at fraction* (198.52 %) compared to the Bioceta reinforced with clay and chalk. Moreover, the average value of WVP was 4.2 times higher. This result implies that the starch based material persists the stress exerted on the bars, and are more *viscoelastic* especially at high temperature (above 70 °C) compared to the reinforced Bioceta. Among all parameters applied, we can also conclude that an addition of 3.5% (w/w) clay with a mixing temperature of 105 °C shows a better performance of mechanical property. Meanwhile, a higher concentration (7.5% w/w) and a mixing temperature of 125 °C could enhance the thermal performance and the gas barrier properties as indicated by the highest values of *Tg* and WVP (Table 2). High WVP value confirms the superiority of nanocomposites as novel material applied with high gas barrier properties. This characteristic supports the application of nanomaterials as reinforcement material in Materbi for food packaging under controlled condition [1].

Previous references mentioned that nanocomposites are a promising key to overcome many of the drawbacks of the bioplastics [7]. Although our results do not completely support this statement in terms of cellulose based matrix (Bioceta), the addition of nano clay (I.30 TC) slightly increased the average E_{value} up to 12-13% compared to the value performed by Bioceta and chalk. Interestingly, chalk addition in Bioceta shows higher values in three parameters: *Stress max.*, *L at stress max.*, *Strain at fraction*, and *Tg* values. The last one was in accordance with a previous result [7], which reported the advantage of nano clay in improving a set of thermal properties. General, the addition of nano clay in the cellulose based material (Bioceta) did not improve the mechanical, thermal and gas barrier properties, unless a higher mixing temperature condition of more than > 190 °C was applied (Table 2). Two reasons could be proposed due to the *no-reinforcing* effects: (i) the thermal decomposition of 'Bioceta' is above 230 °C , while (ii) the mixing temperature in the experiment was lower. Such conditions might lead to the inhomogeneous molecular distribution within the biopolymer matrix [11]. From this result, we could propose that nano clay concentration of 3.5, 5.0, and 7.0 % (w/w) with temperatures over 200 °C should be further investigated.

Scanning Electron Microscope (SEM) imaging was performed in various stratified magnifications (Figure 7, 8). Based on the few pictures taken, they showed that nano clay (white dots) was still dispersed at the surface of the matrix and it did *not yet* reach the intercalated phase (Figure 2). Therefore, we

presumed an imperfect homogenous mixture between bioplastics and clay was occurred. Other alternative methodologies: a more homogenous mix, altering the temperature condition applied to both materials, using a bigger container size in the batch kneading machine, would be highly advised in order to reach the a more homogenously dispersed condition.

This work opens the possibility of nanocomposites' application in biopolymer targetted in the food packaging industry. However, the cost increase of around 0.50-0.70 Euro per item compared to conventional plastics is an impedance to the wide acceptance of bioplastics in the global market, especially in food packaging industries [7]. Thus, it is worth to highlighting the transition towards a *greener economy*; in this respect the promotion of bioplastics and compostable nanocomposites is a door-opening research opportunity that might be well suited in addressing the waste problems in many developing countries, including in Indonesia. Biopolymer nanocomposite could act as a catalysator of a dramatic shift towards a greener economy for a more sustainable environment [8].

Table 2. Summary of mechanical, thermal, gas barrier property tests belong to mixture of nanocomposites (biopolymer and nano clay (I.30 TC).

				Time of			L at Stress	Stress at	Strain at			Tg for
	Clay Conc.	Shear	Temp.	Mixing	E	Stress Max.	Max.	fraction	fraction	F max	WVP	DMTA Test
No.	(%)	(rpm)	(oC)	(minutes)	(N mm-2)	(N mm-2)	(%)	(N mm-2)	(%)	(N)	g (ms.Pa) -1	(0C)
A.	A. Bioceta with I.30 TC											
1	0,00	75	190	7	1.223,00	25,40	3,60	21,20	32,00	253,80	2,61	113,36
2	3,50	75	170	7	1.438,00	27,00	3,70	22,20	22,80	270,40	2,38	116,52
3	3,50	75	190	7	1.482,00	27,10	3,70	24,80	23,50	270,70	2,38	121,60
4	3,50	75	210	7	1.512,00	26,30	3,60	23,90	19,90	263,10	3,12	113,30
5	7,00	75	190	7	1.636,00	26,50	4,20	24,30	16,20	265,00	1,64	114,46
				Average	1.458,20	26,46	3,76	23,28	22,88	264,60	2,43	115,85
				STD	150,67	0,68	0,25	1,52	5,85	6,89	0,53	3,47
B.						Bioceta wit	h Chalk (Place	bo)				
1	0,00	75	190	7	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
2	3,50	75	190	7	1.212,00	26,30	13,50	24,10	27,30	263,30	n.a.	121,05
5	7,00	75	190	7	1.388,00	27,80	10,40	23,60	21,50	278,40	n.a.	119,13
	Average 1.300,00					27,05	11,95	23,85	24,40	270,85		120,09
				STD	124,45	1,06	2,19	0,35	4,10	10,68	n.a.	1,36
C.						Materb	i with I.30 TC					
1	0,00	75	125	7	162,00	10,10	31,90	3,40	175,70	100,80	8,24	62,99
2	3,50	75	105	7	188,00	10,60	33,90	3,40	173,40	105,50	11,36	65,79
3	3,50	75	125	7	174,00	10,40	35,40	3,50	229,90	104,00	9,57	68,84
4	3,50	75	145	7	183,00	10,70	30,10	6,10	372,10	106,90	7,73	65,45
5	7,00	75	125	7	176,00	10,80	28,60	4,00	41,50	108,00	13,92	69,95
				Average	176,60	10,52	31,98	4,08	198,52	105,04	10,16	66,60
				STD	10	0	3	1	119	3	3	3

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Figure 7. SEM of cellulose based material (*Bioceta*) served as matrix. *Bioceta* added with chalk as additive (picture: a, b), while the small white dots indicated the nano clay dispersed on the cellulose matrix (c) magnification at 10,000 times, (d) magnification at 20,000 times



Figure 8. SEM of cassava starch based material (*Materbi*) served as matrix while the white dots indicated the nano clay, (a) magnification at 10,000 times , (b) magnification at 20,000 times

4. Conclusion

Biodegradable polymers derived from natural plant materials, e.g. starch and cellulose based polymer matrix could serve as alternatives to overcome environmental problems and sustainability issues of plastics. In this pilot lab. scale study, we succeeded in producing biopolymer nanocomposites by applying two kinds of biopolymer matrix: (i) Bioceta, (ii) Materbi, nano clay was applied as reinforcement material (I.30 TC) in two stratified concentrations: 3.5% and 7% (w/w). Clay addition of 3.5% (w/w) and mixing temperature of 105 °C was shown to be the best parameter for mechanical property enhancement in Materbi. Meanwhile, clay addition of 7.5% (w/w) at 125 °C could enhance both the thermal performance and gas barrier enhancement. This result confirms the suitability of nanocomposites for food packaging with improved gas barrier property indicated by the high WVP values. Interestingly, the addition of nano clay in the cellulose based material (Bioceta) showed noreinforcing effect in terms of mechanical, thermal and gas barrier properties, as this might require higher mixing temperature (over 190 °C) to homogenously disperse the clay molecule. SEM imaging was performed in order to complete the morphological examination of the novel material.

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