

# APPLICATION OF NANO CRYSTALLINE CELLULOSE (NCC) FROM OIL PALM EMPTY FRUIT BUNCH AS REINFORCEMENT OF BIOPLASTIC NANOCOMPOSITE WITH POLIVYNIL ALCOHOL (PVA) MATRIX

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## ABSTRACT

The number of oil palm empty fruit bunches (OPEFB) in Indonesia is very abundant and has not been used optimally and becoming industrial waste. Indonesia is the second largest country that contributes to the volume of plastic waste in the oceans. Thus, we need a more environmentally friendly plastic alternative, called as bioplastics. Nano Crystalline Cellulose can improve the quality and physical properties of bioplastics. Bioplastic products from nanocellulose are transformed into plastic products that have high selling value. The purpose of this study was to determine the characteristics of bioplastics with nano crystalline cellulose (NCC) reinforcement and polyvinyl alcohol (PVA) matrix. Bioplastic composites have tensile strength ranged from 17.8–20.14 MPa, and have met the minimum standard of tensile strength values for bioplastic based on the Japanese Industrial Standard. The addition of NCC succeeded in reducing the value of the water vapor transmission rate and increasing the tensile strength of the bioplastic composites. Bioplastics can be completely degraded in the fourth week. The development of NCC applications in various fields of bioplastics, chemistry, food, pharmaceuticals, and others is highly recommended.

**Keywords:** NCC, OPEFB, composite, bioplastic

## INTRODUCTION

The focus of development of palm oil process technology is to achieve a level of efficiency and effectiveness of production with the concept of zero waste. Almost 100 percent of this biological resource can be processed into commercial products with high economic value, one of them is oil palm empty fruit bunches (OPEFB). OPEFB has not been optimally utilized and became industrial waste. The number of OPEFB in Indonesia is very abundant, estimated at 27.6 million tons per year. Only 10% of the OPEFB have been utilized. OPEFB contains about 37.5 percent cellulose (Herawan and Rivani, 2013). OPEFB cellulose has the potential to be developed as a raw material in the manufacture of various cellulose-based products, such as paper, rayon and bioplastics.

Utilization of OPEFB cellulose for bioplastic is to reduce the use of synthetic plastics which cause global environmental pollution issue. According to (Purwitasari, 2018), the potential of cellulose from OPEFB which can be used for bioplastic production was 2,097,225 million tons, assuming the yield of bioplastics obtained was 17.28%. In this case, Indonesia is the second largest country that contributes to the volume of plastic waste in the oceans of 187.2 million tons while China is the first that contributes 262.9 million tons of plastic waste (Kompas, 2020).

Currently, plastic consumption in Indonesia reaches 4.6 million tons per year with an average growth of 5% per year. The plastics used will then become waste that pollutes the environment. This is due to the characteristic of plastic which cannot be degraded naturally by the environment in a short time. Thus, it is very important to produce bioplastics by utilizing OPEFB waste. OPEFB cellulose has great potential for the development of bioplastic as the alternative of synthetic plastics.

Currently, the public demand for bioplastics is increasing in the market with a market share of 39.1 percent of the total volume of world plastic production. According to the European Bioplastic Association, the demand for bioplastics in the world increased to 7.8 million tons in 2019 and will increase every year. It is expected that this will enhance the development of bioplastics from palm oil waste (Kompas, 2020). The constraints in the development of bioplastics are that the physical, mechanical and thermal properties are generally lower than synthetic plastics. The increasing of the bioplastics strength can be pursued by using fillers as the reinforcing material (Wicaksono, et al, 2013). One of material that has the potential to be developed as a filler is natural fiber. Fiber source that has the potential to be explored is cellulose.

Cellulose is widely used as a reinforcing material in various polymer composites (Peng, 2011). Recently, composite materials with nano-sized reinforcement, called as nanocomposites, have been observed. The addition of nanoscale elements into the polymer matrix results in better properties (Ranby, 2018), such as high surface area, high modulus young, high tensile strength, and low coefficient of thermal expansion (Samir, 2012). Nanocellulose can improve the quality and physical properties of bioplastics. Bioplastic products from nanocellulose are transformed into plastic products that have high selling value. Bioplastics of these two materials are stronger and transparent.

Nano Crystalline Cellulose (NCC) is a pure crystalline cellulose derivative that has particles shaped like rods with a diameter of 1-100 nm and a particle length of a few micrometers (Brinchi, 2013). Nanocrystalline cellulose with a particle size of 160-400 nm (length) and 20-30 nm (diameter) has a relatively low aspect ratio (Sofla, et. All, 2016). Nanocrystalline cellulose consists of 100% cellulose with a crystalline content of 54-88% (R. Moon, 2011).

Nano Crystalline Cellulose is biocompatible and biodegradable, thus it is a potential material for research and development. Nano Crystalline Cellulose can be used in various applications because it has superior characteristics such as high tensile strength, large surface area, low density, non-toxicity, and electromagnetic response (Tang, et al, 2015). According to (Ioelovich, 2012), nanocellulose is a new type of cellulose material characterized by an increase in crystallinity, aspect ratio, surface area, and an increase

in dispersion and biodegradability. Due to this ability, nanocellulose particles can be used as polymer reinforcing fillers, additives for biodegradable products, membrane reinforcement, thickener for dispersion, and drug carrier media and implants. Nanocellulose can be used in various fields such as paper industry, packaging industry, food industry and pharmaceutical industry.

Composites have two parts, matrix and filler. Filler functioned to strengthen or harden the material of a composite (reinforcement). The use of nanoscale reinforcing materials shows better improvements in physical and mechanical properties such as tensile strength and thermal stability compared to other conventional materials. Some types of reinforcing material are fiber and particles. Natural fibers are used as fillers in composite polymers to increase the strength of composites. The matrix is used to keep the filler in the structure, protect the filament, assist in load distribution, and carry interlaminatory strain (Fajar, 2013).

Polyvinyl alcohol (PVA) is a biodegradable synthetic material that can be used as for making biodegradable plastics. PVA is widely used as a promising alternative packaging material because of its excellent properties in packaging formation, resistance to oil and grease, high tensile strength and flexibility. PVA has good compatibility if a filler in the form of nanocellulose is added thus it can produce environmentally friendly composite products (Rohani et al, 2008). PVA has the potential ability to interact with the hydrophilic surface of the biomaterial due to its strong hydrogen bonds formed in suitable combinations with cellulose or nanocellulose to produce environmentally friendly nanocomposites (Li et al, 2012).

Some of the advantages of natural fiber fillers are renewable, biodegradable, and abundant availability. The research of bioplastic composites from starch with fillers of various cellulose fibers such as OPEFB (N. A. and S. K., 2017), sugar palm fiber (Fahma et al, 2016), bamboo (Susanti et al, 2015) and water hyacinth fibers (Asrofi, 2018) have been carried out. Research on bioplastics reinforced by cellulose micro fibers (CMF) from natural hemp fiber had been carried out by (Syafri et al, 2018). Maryam et al (2019) have also conducted micro-synthesis of bacterial cellulose as reinforcement for bioplastic composites with a polyvinyl alcohol (PVA) matrix. Research on bioplastics with PVA matrix with nano cellulose fiber filler (Kakroodi et al, 2014) and bioplastics with PVA matrix with pineapple fiber nanocellulose filler (Iriani et al, 2015) were carried out. Also, several other studies such as bioplastics with a PLA matrix and filler precipitated  $\text{CaCO}_3$  (Baek et al, 2014); bioplastics with PCL matrix and filler of  $\text{CaCO}_3$  nanoparticles; bioplastics with a matrix of tapioca starch nanoparticles (Mulyono et al, 2015); bioplastics with tapioca starch matrix and clay nanoparticle filler (Souza et al, 2012) bioplastics with aloe vera starch matrix and chitosan filler (Utomo et al, 2013); and bioplastics with a matrix of sweet potato starch and cellulose filler of seaweed (Darni et al, 2011) were conducted. Based on the results of the literature review conducted, the novelty of this study is the development of a bioplastic composite with a PVA matrix and NCC filler from OPEFB. NCC from OPEFB was produced by bioprocess technology.

The purpose of this study was to determine the characteristics of the bioplastics developed with the addition of nano crystalline cellulose filler from OPEFB. This research aimed to tackle the issue regarding the use of non-degradable synthetic plastics and to overcome issues in the development of bioplastics. The disadvantage of bioplastics is the lower physical and mechanical characteristics compared to synthetic plastics. NCC can be used as a new biomaterial which is expected to be a solution to improve the characteristics of bioplastics.

## EXPERIMENTAL SECTION

### Materials

The materials used in this study were polyvinyl alcohol (PVA), nano crystalline cellulose from OPEFB, glycerol, distilled water.

### Instrumentation

The equipments used were a hotplate stirrer, glassware, thermometer, plastic mold, oven, vacuum pump and analytical balance.

### Procedure

The matrix used were polyvinyl alcohol with the addition of NCC (0%, 1%, 3%, 5%) with the casting method. Bioplastics were made by dissolving x% (according to treatment) NCC into 100 mL of aquadest and 3 mL of glycerol. Then 10 g of PVA was added and stirred using a hotplate stirrer for 1.5 hours at 80-85 °C. The solution was vacuumed to remove air bubbles. The bioplastic solution was poured into a glass plate mold with the size of 250 mm x 250 mm x 3 mm, and dried for 2 days in air after being dried in oven at 50°C for 24 hours (Maryam et al, 2019). Characterization of bioplastic composites were tensile strength, biodegradation and water vapor transmission rates.

### *The Tensile Strength (ASTM D 882-02, 2006)*

The tensile strength was carried out using a Universal Testing Machine (UTM) Com-ten 95T. The samples were put in a climatic chamber at a standard relative humidity temperature (29°C, 50%) for 48 hours. The measurement results were the average measurement result of 5 samples. The test was carried out based on ASTM 88-02 standard at a speed of 5 mm / minute. The tensile strength of plastic was calculated by the following equation:

$$=F_{\max} / A$$

where:

= tensile strength (Mpa)

F<sub>max</sub>= maximum force (N)

A = unit area (mm<sup>2</sup>)

***The test of water vapor transmission rate and permeability of bioplastics on water vapor (Latriyanto et al, 2007)***

The cup containing dry silica gel was tightly covered with a film. The cup was put in an airtight container contained a saturated NaCl solution to create  $\pm 75$  percent relative humidity conditions. Water vapor diffused through the film will be absorbed by the silica gel and will increase the weight of the silica gel. Changes in cup weight were recorded every hour until the seventh hour. The data obtained was made into a linear regression equation, in order to obtain a slope. The water vapor transmission rate (WVTR) was calculated by the formula:

$$\text{WVTR} = \frac{\text{Slope of regression line of sample weight vs time } (\frac{g}{\text{hour}})}{\text{area of bioplastic (m}^2\text{)}}$$

***Biodegradability Test***

Biodegradability is a susceptibility of a compound (organic or inorganic) on changes in material due to the activities of microorganisms. The test referred to (Tokiwa et al, 1994) used soil or the soil burial test method by burying 4 x 1 cm<sup>2</sup> sample in a pot filled with soil and leaving the sample exposed to air. Observations were carried out once a week until the sample was completely degraded or the bioplastic sheet was disappeared.

**RESULTS AND DISCUSSION**

***Bioplastic Nanocomposite***

Composite materials generally consist of two or more different materials, filler and binder, called as matrix. Filler is a component inserted into the matrix which functions as a receiver or a major load bearing (reinforcement). The matrix is a part of the composite that surrounds the composing particles of the composite, which functions as a binder for the particles and helps forming the physical structure of the composite. Cellulose from OPEFB has the potential to be used as a reinforcement material in bioplastic nanocomposites and to increase the added value of OPEFB. The promising properties of nano crystalline cellulose are biodegradability, sustainability, high biocompatibility and most significantly for the reinforcement of polymer matrices. The EFB waste represents a renewable source of lignocellulose as an abundant, inexpensive, and readily available biomass that can be used in various applications such as reinforcement of nanocomposites.

The nanocomposites in this study consisted of PVA which functions as a matrix and nano crystalline cellulose (NCC) from OPEFB as the reinforcing material. The NCC used in the manufacture of bioplastics has an average size of 224.40 nm with a crystallinity index of 60.15% (Maryam et al, 2019). The nano crystalline cellulose (NCC) used can be seen in Fig. 1. The making of bioplastic nanocomposites was carried out by the casting method with the addition of NCC. Bioplastic composites can be seen in Fig. 2.

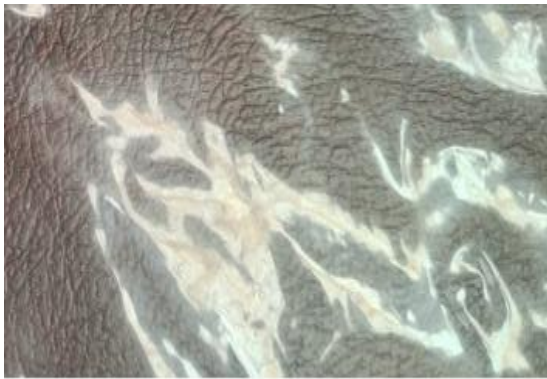


(1)



(2)

**Fig. 1.** Nano crystalline cellulose, (1) Solution; (2) Powder



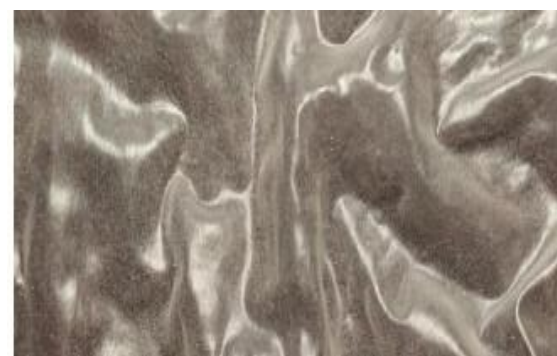
(1)



(2)



(3)



(4)

**Fig 2.** Bioplastic Composite (1) control; (2) addition of NCC 1%; (3) addition of NCC 3%; (4) addition of NCC 5%

**Properties of Bioplastic Nanocomposite**

Result of Bioplastic Nanocomposite Characterisation can be seen in Table 1.

**Table 1.** Characterisation of Bioplastic Nanocomposite

No	Addition of NCC	Elongation (%)	Tensile Strength (MPa)	Modulus Young (MPa)	Water vapor transmission rate (%)
1	Addition of 0%	30,12	15,06	0,50	13,76
2	Addition of 1%	25,47	17,83	0,70	8,17
3	Addition of 3%	20,39	18,35	0,90	6,86
4	Addition of 5%	16,78	20,14	1,20	5,65

The result showed that the tensile strength value of bioplastic nanocomposites increased with the addition of nano crystalline cellulose compared to standard bioplastic composites. The increase in tensile strength of the resulting nanocomposites was due to the stiffness of the chains attached to the nano crystalline cellulose, due to the strength of the inter and intra molecular hydrogen bonds on the cellulose itself and the homogeneous distribution of nano crystalline cellulose on the polymer and the high compatibility between the fibers and the matrix aided by the high interfacial surface area. Hydrogen bonding between nano crystalline cellulose and the PVA matrix caused an increase in the tensile strength of the resulting nanocomposites (Indria and Gea, 2017).

The strong compatibility and the good dispersion of fillers can strengthen the composite. The presence of cellulose nanocrystalline fillers can form hydrogen bonds due to the presence of hydroxyl group. Both can form a rigid network that can strengthen PVA composites. Due to the stiff nature of the resulting composite film, the elongation results at break will decrease. The addition of fillers to this composite can reduce the value of its elasticity. These results can be seen from the elongation value in Table 1. The more amount of NCC added resulted in the lower percentage value of elongation at break. The filler dispersion and the interface adhesion force of the filler and matrix greatly affected the results of the composite tensile strength. The strong hydrogen bond interfaces lead to the resulting improved matrix. In addition, the density of the composite (filler and matrix) also affected the tensile strength value.

The tensile strength value was also influenced by plasticizers which reduce the hydrogen bonds in the bioplastic composites, thus increasing the flexibility of the composites. The increased flexibility decreased the tensile strength of the composites. In addition, the addition of glycerol reduced the intermolecular forces of the polysaccharide chain thus the composite structure becomes more flexible (Cao, 2007). The tensile strength also increased with the addition of the nanocrystalline cellulose concentration in the composite. NCC fills the composite matrix and makes the composite more resistant to applied forces. It can be seen that the elongation of the composites is getting lower with the addition of higher concentrations of nanocellulose. The addition of glycerol can reduce the stiffness of the composite, so that

the composite becomes more flexible and plastic. The results showed that the resulting tensile strength ranged from 17.83–20.14 Mpa. The tensile strength value of bioplastics has met the minimum standard for the tensile strength of edible film (bioplastic) based on the Japanese Industrial Standard, (Japanese Industrial Standard) 3.92 Mpa. The bioplastic had a higher tensile strength value compared to bioplastics with the bacterial cellulose NCC filler from a study conducted by (Maryam et al, 2019), 15.72 MPa in an additional of 2%.

The elasticity value or modulus young was directly proportional to tensile strength value and inversely proportional to elongation. The greater modulus young composite value resulted in the smaller elastic stretch produced or the less elastic composite. The addition of NCC resulted in a greater modulus young value or a lower elasticity value. According to (Monara, 2016), the addition of NCC had no significant effect on the modulus young value.

### ***Water vapor transmission rate***

The value of the water vapor transmission rate can be used to determine the permeability value of a material on water vapor. The bioplastic nanocomposites have a thickness of about 0.1 mm to 0.2 mm. The value of the bioplastic water vapor transmission rate ranged from 8.17-5.65%. Table 1 showed the addition of NCC increased the ability of bioplastic nanocomposites to hold water vapor or decreased the value of the water vapor transmission rate. The water vapor transmission rate of control bioplastic was 13.76%, and decreased with the addition of NCC of OPEFB. This was due to the formation of a network structure between the NCC particles and the PVA component which increases the resistance of the composite on water. Nano crystalline cellulose changes the direct diffusion path of water molecules into the composite to become tortuous path, preventing water vapor from passing through the film (Lani et al, 2014). According to (Suyatma et al, 2005), adding a hydrophilic plasticizer can reduce its hydrophobic properties and increase the hygroscopic properties of composites. Hygroscopic properties are properties where a material can easily absorb water from the air, thereby increasing its water vapor transmission rate. Glycerol is a hydrophilic plasticizer that can improve its hygroscopic properties hence it has the ability to absorb water from the air around the material and increase the water vapor transmission rate of the material. The water vapor transmission rate is also a parameter that shows the quality of the bioplastic in maintaining the quality of the product which it will pack. High water vapor transmission rate values indicate that the bioplastic has large pores so that water vapor can easily pass through the composite matrix.

Polyvinyl alcohol (PVA) is a biodegradable synthetic polymer material. PVA has a molecular weight of 26,300-30,000, a melting point of 180-190°C, degree of hydrolysis of 86.5-89%, and can be degraded naturally. This has led to PVA being widely used as a promising alternative packaging material due to its excellent properties in packaging formation, resistance to oil and grease, high tensile strength and flexibility. However, this property is highly dependent on humidity where the higher humidity resulted in the more water absorbed from the surrounding environment, causing the PVA composite decrease the



tensile and tear strength, and increase the elongation. According to (Roohani et al, 2008), PVA has good compatibility, if a filler in the form of NCC is added thus it can produce environmentally friendly nanocomposite products. Thus, the addition of NCC to PVA-based bioplastics is expected to increase and improve the mechanical properties of the resulting PVA composites. The addition of polyvinyl alcohol, glycerol and cellulose nanofibers as reinforcement material resulted in composites with good physical and mechanical properties. The results showed that the addition of NCC increased the tensile strength and modulus young, but decreased the elongation and water vapor transmission rate of the composites. Composites can be degraded naturally and environmental friendly.

### ***Biodegradability***

Biodegradability is the durability of biodegradable bioplastic products against decomposing microbes, soil moisture and chemical factors contained in the soil. The process of biochemical decomposition of organic molecules by microorganisms is known as biodegradation, which converts C, N, S, and P (organic compound content) into inorganic products (Robertson, 2013). The film biodegradability test used the soil burial test method (Tokiwa et al, 1994) by implanting biodegradable bioplastic sheets into pots filled with soil and observed until the sheets were lost due to degraded by microbes. The results showed that the biodegradable composites decomposed completely at the fourth week. Biodegradable bioplastics with raw materials from PVA, NCC and glycerol are easily biodegradable because the raw materials used are easy to interact with water and microorganisms and are sensitive to physicochemical effects (Tan et al, 2016). Pratomo and Rohaeti (2011) showed that in the biodegradable biodegradation process of nata de cassava, there was a breaking of the bonds in the  $\beta$ -1,4-glycosidic bonds hence the cellulose molecules break down into glucose molecules gradually. Degradation of polymers is used to express the physical changes resulting from chemical reactions that include breaking bonds in the backbone of the macro-molecules. Chemical degradation reactions in linear polymers cause a decrease in chemical degradation reactions in linear polymers which result in the decreasing in molecular weight or a shortening of the chain length (Surdia, 2000).

### **CONCLUSION**

The resulting bioplastic composites have tensile strength of 17.83 to 20.14 MPa, and have met the minimum standard of tensile strength values for edible film (bioplastic) based on the Japanese Industrial Standard. The value of the bioplastic water vapor transmission rate was ranged from 8.17-5.65%. Bioplastics were completely degraded at the fourth week. The addition of OPEFB NCC was successful in increasing the tensile strength and degradation of bioplastics and was able to reduce the water vapor transmission rate.

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