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Tensile Properties of Sago Starch Biocomposites Reinforced with Nanocrystalline Cellulose from Rattan Biomass

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Abstract. The biomass of rattan is one of natural waste that contains 37.6% of cellulose contents, which is potential to become a source of nanocrystalline cellulose as a filler in biocomposites. There are three important steps to produce alpha cellulose: delignification, alkalization, and bleaching. Nanocrystalline cellulose was extracted through the hydrolysis of alpha cellulose by using sulfuric acid and followed by ultrasonication, centrifugation, and filtration steps. Sago starch biocomposite has been made by casting method. The tensile properties of biocomposite were analyzed and the characterization was done by scanning electron microscopy (SEM). Results have shown that nanocrystalline cellulose has made a strong interfacial interaction with starch that can be improved the tensile strength of biocomposites. SEM have shown smoother and more coherent surface and supported the tensile properties result.

1. Introduction

Sago can be classified as a natural ingredient that has been known as a promising renewable material due to its cheap price, accessibility, and biodegradability. Sago starch can be safely degraded. Sago is one of natural plant which has a high starch content and productivity in Indonesia and it could be modified to become a natural product like bioplastic by the temperature and shearing control [1].

The biodegradable film based on starch exhibit poor mechanical properties. To fix the weak mechanical properties of biodegradable film based on starch, additive material is like plasticizer could be added to biodegradable starch film. The amount of additives such as biomass of rattan as filler, glycerol as plasticizer and acetic acid as co-plasticizer are regularly used to reduce the rigidity of biodegradable film [2]. Plasticizer and additive have been known for gelatinization of starch under thermal processing. Glycerol as plasticizer more commonly used to increased tensile properties of biodegradable film based on starch. Additionally, there is still one other plasticizer such as acetic acid. It was examined to enhance the processing ability properties of biodegradable starch film. Acetic acid is known to be able to weaken the non linear chain of starch [1]. Tawakaltu, et.al have reported the application of citric acid and acetic acid as co-plasticizer on biocomposite films [3].

The amount of natural fillers are usually added to increase the tensile properties of the bioplastic. The application of natural fibers as filler is the best option to fix tensile properties of the biodegradable film based on starch. The similiar structures between starch and natural fiber lead to higher interaction between the materials. Nasution, et.al (2017) and Harahap (2016) have reported that microcrystalline cellulose (MCC) and nanocrystalline cellulose (NCC) can be produced from natural biomass such as



empty bunches of oil palm and sugarcane bagasse, where both of them have advantages as filler in biocomposite [4,5].

Rattan is one of the natural resources that could be modified to natural filler with some modifications. The high rattan productivity makes it usable as one of natural resources which could be processed to innovation item by the utilizing of nano equipments [6]. Rattan comprises of three important components, such as 40-50% of cellulose contents, 30-40% of hemicellulose content and 20-30% of lignin contents [7]. Cellulose has some outstanding characteristics like low density, rigidity and can be degraded naturally. In light of cellulose characteristics make it as a hopeful material as natural filler. Cellulose substance of rattan can be separated to produce nanocrystal cellulose [8].

Nanocrystalline cellulose has some advantage points, e.g. the nano scale, high particular quality and high surface zone. NCC could be produced using strong acid hydrolysis method under the effect of temperature and time control. Nanocrystalline cellulose (NCC) was usually produced by sulfuric acid to remove the amorphous region of cellulose in order to produce cellulose crystals with a diameter around 10-100 nm [9].

Based on those explanations, the tensile properties of nanocrystalline cellulose (NCC) from rattan biomass reinforced sago starch biocomposites will be investigated.

2. Method

2.1 Material

The biomass of rattan was taken from home furniture industry, Medan, Indonesia. Sago was bought from nearby market and screened into a size of 74 microns. Other chemicals were supplied by MERCK.

2.2 Preparation of NCC

The biomass of rattan was made into a small pieces and delignificated by 3.5% HNO₃ at 90 °C and digested with NaNO₂ and 2% w/v NaOH. The primary bleaching step with 1.75% NaOCl treatment was performed at 101 °C for 30 minutes. The second process (alkalization process) was performed by 17.5% NaOH at 80 °C for 30 minutes. 10% H₂O₂ was used as the last bleaching treatment which was performed at 60 °C for 1h. All treatments were filtered by using distilled water to eliminate the alkali components from sample. Sulfuric acid was used to remove the amorphous region at 45 °C with and warmed for 45 minutes under mechanical mixing. The cellulose that has been hydrolyzed was centrifuged at 10000 rpm for 10 minutes and precipitated. The nanocrystalline cellulose's precipitate was washed by using distilled water and after that it is ultrasonicated by ultrasonication equipment for 10 minutes. The last step is the sample was dialyzed by using distilled water for 4-5 days until reach a neutral pH.

2.3 Biocomposite Production

Biocomposite was prepared by casting a blend of sago starch, NCC, glycerol as plasticizer, co-plasticizer such as acetic acid and distilled water. The amounts of filler (1, 2, 3, 4 wt% of starch) were dispersed by using 95 ml of aquadest at 1000 rpm for 15 minutes. A quantity of 10 g of starch, 30 wt% of glycerol and co-plasticizer with the amounts (10, 20, 30, 40 wt% of starch) were mixed into dissolution starch. The mixture was heated at 70 °C under mechanical stirring during 30 min until gelatinized was reached. The blend was casted in a plastic mold and evaporated at room temperature overnight.

2.4 Tensile Properties

Tensile test was performed by Instron Universal Testing Machine based on the ASTM D882-02.

2.5 Morphological Characteristic.

The morphology of biocomposite was tested by using scanning electron microscopy (EVO MA 10 ZEISS).

3. Discussion

Effect of NCC and acetic acid compositions on the tensile strength of biocomposites are shown in Fig. 1.

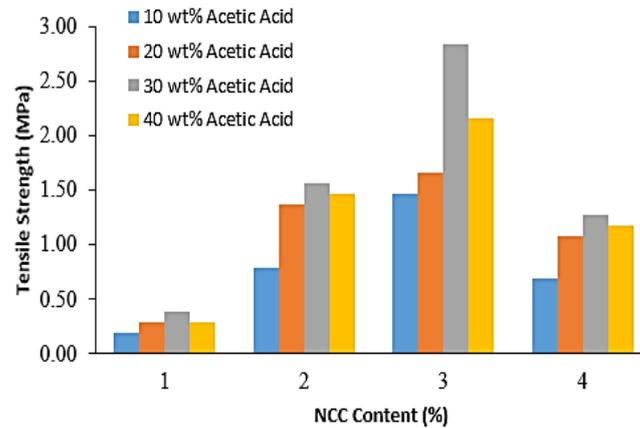


Figure 1. Effect of NCC and Acetic Acid Compositions on the Tensile Strength of Biocomposites

The higher of filler and co-plasticizer content will increase the tensile strengths of biocomposite, until reach an optimum value at 3 wt% of NCC with 30 wt% of acetic acid. It was demonstrating an ideal value of 2.84 MPa. The improvement was indicated by a strong interfacial interaction existed between starch and cellulose where NCC was embedded to the matrix, causing to efficient stress transfer. The result was supported by scanning electron microscopy (SEM) in Fig. 4. At 4 wt% NCC composition, there is a reduce in (or stabilization of) the tensile strength was noticed, which was most possibly caused by the accumulation of the filler in the matrix. The stress was caused by aggregation of the NCC in the matrix phase could not be distributed uniformly and gave rise to less efficient stress transfer [10]. The biocomposite tensile strengths were affected by the acetic acid substance until reach an optimum value at 30 wt%. The acetic acid will have a role as a cross-linking agent and as co-plasticizer in the biocomposites. These functions of acetic acid were noticed by the concentration of acetic acid. Acetic acid could possibly break the spread chains of starch particle that incite arrangement of profoundly straight structure which thus permit framing more hydroxyl chains among the chains of starch and expanding the tensile strength [11]. Acetic acid will form hydrogen bonds with cellulose as fillers, as shown in the following Fig. 2.

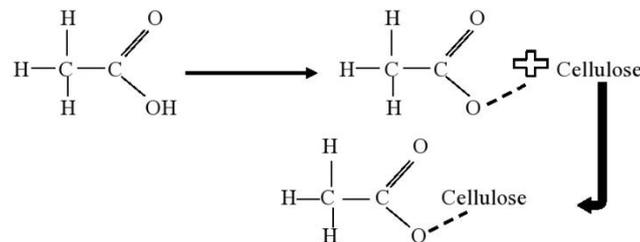


Figure 2. The Scheme of Acetic Acid and Cellulose Interactions [12]

However, the higher of acetic acid content that was reached at 40 wt%, the higher residual acetic acid in the biocomposite could be assumed a part as the plasticizer that was similar like glycerol and break the macromolecule chains. That was affecting the decrease of the biocomposite tensile strength.

Effect of NCC and acetic acid composition on the elongation at break of biocomposites are shown in Fig. 3.

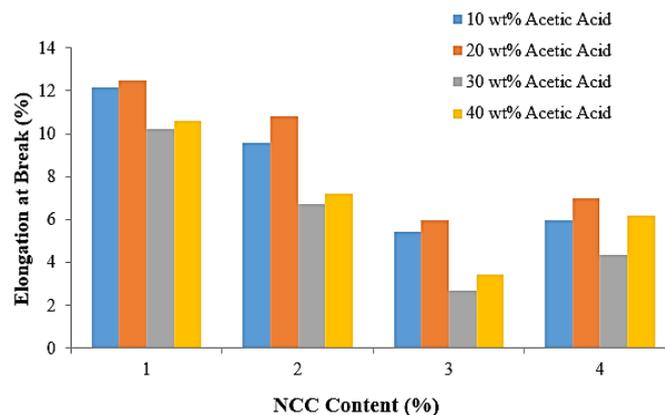


Figure 3. The Role of NCC and Acetic Acid Composition on The Elongation at Break of Biocomposites

As appeared in Fig. 3, the higher of NCC composition, the lower elongation at break until reach an optimum value at 3 wt% of NCC. Great interfacial tension attachment among matrix and NCC are common. Here, both of starch and NCC contain hydroxyl groups where the hydrogen chains among interfaces could be formed. The solid hydrogen interactions between NCC and matrix results in the development of an inflexible system of NCC that will be causing the reduceness support of the matrix [13]. It has been known that NCC has rigidity properties, the value of elongation at break will decrease. Acetic acid addition was known to expand the elongation at break.

The higher of acetic acid composition until reach 30 wt% acetic acid composition, the lower elongation at break. Acetic acid can most likely break the extended chains of starch particle that changed the arrangement of highly linear structure to turn allow the formation of hydroxyl chains among the matrix chains and decreasing the elongation at break value [11]. When the branching group (1.6 glycosidic) has completed, acetic acid is not only break the amylopectin chains, but will also break the linear chains e.g. (1.4 glycosidic) to form a shorter chain of amylose.

This properties were supported by the SEM results of biocomposites that were shown in Fig. 4(a) and 4(b). In SEM investigation results by 1 wt% of nanocrystalline cellulose (NCC) and 10 wt% acetic acid (Fig. 4a) composition show the matrix morfology is not smooth and flat. It was caused by the insoluble starch that was resulting in the formation of an empty fraction and agglomeration. In addition to 1 wt% nanocrystal cellulose (NCC) and 10 wt% acetic acid also showed poor interface adhesion between nanocrystalline cellulose (NCC) and sago starch matrix. The poor interface adhesion between matrix and filler is due to the accumulation of nanocrystalline cellulose (NCC) which would be able to reduce the strength of its appeal [2].

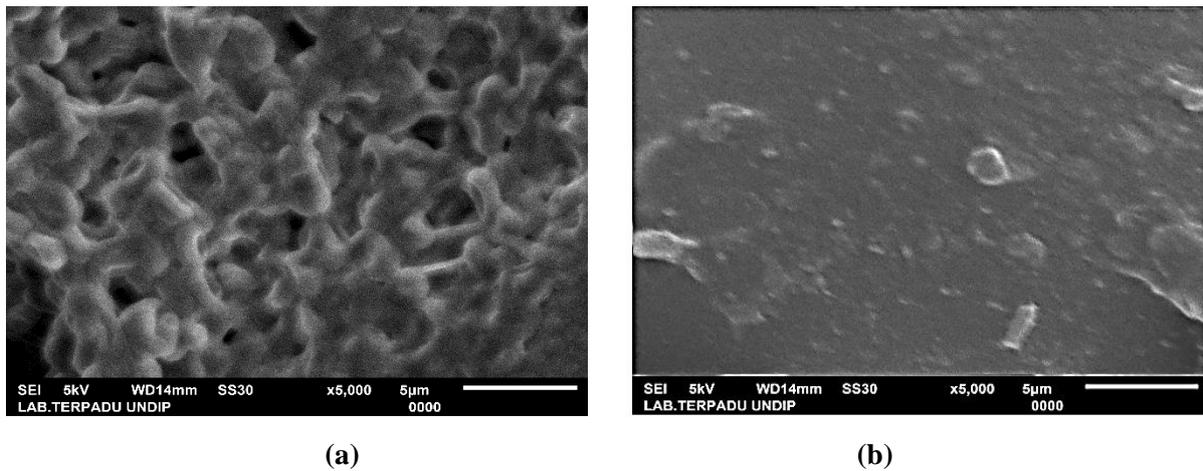


Figure 4. Morphology of Biocomposite by 1 wt% NCC and 10 wt% Acetic Acid Compositions (a) and Biocomposite by 3 wt% NCC and 30 wt% Acetic Acid Compositions (b)

Fig. 4(b) which is a biocomposite by 3 wt% nanocrystalline cellulose (NCC) and 30 wt% acetic acid compositions, the surface area of the biocomposite is smoother. It was shown that nanocrystalline cellulose (NCC) consistently circulated and implanted on the matrix is showing a great adhesion. It was caused by the uniformly dispersed of nanocrystalline cellulose (NCC) that has been derived from relatively little nanocrystalline cellulose (NCC) sizes and strong hydroxyl interaction among cellulose and starch [13].

4. Conclusion

Tensile strength of biocomposite was affected by NCC and acetic acid additions which show that biocomposite by 3 wt% NCC and 30 wt% acetic acid composition has smoother and more coherent surface to biocomposite by 1 wt% NCC and 10 wt% acetic acid composition.

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