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# The Effect of Alkali Treatment and Microcrystalline Cellulose Addition on Density Value of Cantala Fiber Reinforced Unsaturated Polyester Composites

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### Abstract

Natural fiber reinforced composites is one material potentially developing in Indonesia. One of biggest problem with composites specimen is its void. One property to find out void of composites is composites density value. The objective of research is to investigate the effect of fiber alkali NaOH treatment and Microcrystalline Cellulose (MCC) addition on density value of cantala fiber reinforced unsaturated polyester composites. Firstly, cantala fibers was submerged into alkali NaOH 6% solution for 0, 3, 6, 9, and 12 hours. Furthermore, the fiber was washed using acetid acid and then using clean water to reach pH 7. Thereafter, cantala fiber was dried in the oven for 10 hours at temperature 60 °C. Composites was composed of cantala fiber, unsaturated polyester polymer matrix, and microcrystalline cellulose according to the composition with volume fraction 30%. Composites was casted using compression molding method with compressive strength of 10 MPa for 12 hours. The result of research showed that the longer the alkali treatment time and the more addition of Microcrystalline Cellulose (MCC) filler, the higher is the composites density. The higher density value of cantala fiber reinforced unsaturated polyester is alkali treatment six hours, which was 1.223 gr/cm<sup>3</sup>.

## 1 Introduction

Technology development increases the need for material. One of materials experience rapid increase is composites material [1]. Composites is a material consisting of two components, reinforcing filler and matrix. Composites has some strengths, light, resistant to corrosion, easy to fabricate, and more economical [2]. Escalated environmental problem, abundant waste, and greenhouse effect encourage material industry to use environment friendly and renewable composites [3]. In this decade, one of environment friendly and renewable composites is bio-composites. Bio-composites is the one composed of polymer matrix with natural fibers, like agave, kenaf, hemp, etc. [4]. Natural fibers are the following advantages compared with synthesis fibers, example available widely particularly in Indonesia, light, low-cost, non-corrosive, and having low density [5]. Polymer matrix often used in composites material is thermoplastic, like Polyethylene (PE), Polypropylene (PP), and Polyvinyl Chloride (PVC), and thermoset, like unsaturated polyester, epoxy, and phenol [6]. Natural fiber that can be applied to bio-composites material are cantala fiber.

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The cantala fiber comes from the leaves of the Agave cantala plant as shown in Figure 1. The characteristic of the Agave cantala plant are bluish-gray leaves, sharp spiny leaf edges, and growing on dry soil [7]. Cantala fiber has good mechanical properties is light, environmentally friendly, economical, and renewable, which can be seen Figure 2. Then, the composition of cantala fiber consists of 64.23% cellulose, 13.13% moisture, 4.98% ash, 5.91% lignin, and extractive 1.1% [8]. Unsaturated polyester matrix is a synthetic resin of straight chain resulting from the reaction between glycol and dysfunctional acid such as maleic acid and adipic acid [9]. This matrix has some strengths, high mechanic power, heat resistant, chemical resistant relatively low cost, and easy to fabricate. This matrix unsaturated polyester has been widely applied in the automotive and construction field [10].



**Figure 1.** Agave cantala plant



**Figure 2.** Cantala fiber

Mechanic properties of natural fiber reinforced polymer composites are largely affected by adhesiveness between fiber and matrix. The adhesiveness is due to natural fiber still containing high hemicellulose, pectin, and lignin, thereby decreasing the mechanic strength of composites [11]. One of methods used widely to remove hemicellulose, pectin, and lignin is alkali chemical treatment. Alkali treatment method or called mercerization utilizes aquades solution and NaOH crystal with concentration and time variables according to the chemical equation of alkali treatment in Equation 1 [12].



One of important variables in alkali NaOH chemical process is alkali treatment duration (time) as it affects the mechanic properties, particularly density value of fiber. Ariawan et al. [13] has conducted alkali treatment with time variable on density of zalacca fiber. This study found that the longer the duration of alkali treatment, the higher is the density value of fiber. Then, Raharjo et al. [14] have also examined the

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density value of cantala fiber with alkali treatment NaOH 2% solution for 12 hour and without alkali treatment. The result showed that the density of cantala fiber with alkali treatment was higher than those without alkali treatment.

The most influential natural fiber reinforced polymer composites is cellulose compound. One of problems with composites is the less even distribution of cellulose compound, so that cellulose compound particles should be lessened using microcrystalline cellulose in order to improve the cellulose distribution in composites [15]. Microcrystalline Cellulose (MCC) is a pure cellulose isolated from alpha cellulose using mineral acid with the following advantages, renewable, high mechanic property, non-toxic, and biocompatible [16]. Kiziltas et al. [17] has conducted a research on the effect of microcrystalline cellulose volume fraction on density value of nylon 6-microcrystalline cellulose composites. The result of research showed that the higher the volume fraction, the higher is the density value of composites.

From elaboration above, the research on the effect of alkali treatment and Microcrystalline Cellulose (MCC) on density value of cantala fiber reinforced unsaturated polyester composites needs to be conducted. Alkali treatment using NaOH at concentration of 6% with time variable of 0, 3, 6, 9, and 12 hours. The composites were molded using compression molding with volume fraction 30% and short random cantala fiber orientation.

## 2 Experimental Methods

The designed composite in this work consist of the following materials, i.e., cantala fiber, unsaturated polyester, Methyl-Ethyl-Ketone-Peroxide (MEKPO) catalyst, aquades, NaOH crystal, and acetid acid. Cantala fiber is submerged into alkali solution, the combination of aquadest solution and NaOH crystal at concentration of 6% for 0, 3, 6, 9, and 12 hours, which can be seen Figure 3. Thereafter, cantala fiber is taken and submerged into acetic acid solution at 1% concentration, and then fiber is rinsed using distilled water until it is clean and reaches pH of 7. The next step is to dry cantala fiber in the oven for 10 hours at 60 °C. The dried fiber is then sliced in 10 mm.



**Figure 3.** Alkali treatment process

The process of preparing composites is started with putting all compositions-cantala fiber, unsaturated polyester matrix, MEKPO catalyst 1% of total unsaturated polyester volume fraction and MCC 5% into mold. Then, cast the composites using compression molding method with pressure of 10 MPa for 12 hours at room temperature, which can be seen Figure 5. Thereafter, put the composites into the oven to undertake post cure process for two hours at 60 °C. And then, slice the cantala fiber-reinforced unsaturated polyester composites according to standard American Society Testing and Material (ASTM) D792 was used composites density test. There are seven composites specimen variations, i.e.:

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1. UPRs (neat unsaturated polyester)
2. UPRs MCC (unsaturated polyester + MCC)
3. AL0 MCC (non-alkali fiber + MCC)
4. AL3 MCC (alkali fiber 3 hours + MCC)
5. AL6 MCC (alkali fiber 6 hours + MCC)
6. AL9 MCC (alkali fiber 9 hours + MCC)
7. AL12 MCC (alkali fiber 12 hours + MCC)

Density test is the measurement of mass unit per volume unit. Density value of polymer composites material with standard ASTM D792 is actually as shown in Equation 2 [13]:

$$\rho = \frac{m_u}{m_a + m_u} \times \rho_a \quad (2)$$

where:

- $\rho$  = Composites density (kg/m<sup>3</sup>)
- $m_u$  = Composites mass in the air (kg)
- $m_a$  = Composites mass in the water (kg)
- $\rho_a$  = Water density at room temperature (kg/m<sup>3</sup>)

Considering the Rule of Mixture (ROM), the density of natural fiber reinforced composites can be calculated using the following Equation 3 [18].

$$\rho_c = Vf \cdot \rho_f + (1 - Vf) \cdot \rho_m \quad (3)$$

where:

- $\rho_c$  = composites density (kg/m<sup>3</sup>)
- $Vf$  = fiber volume fraction
- $\rho_f$  = fiber density (kg/m<sup>3</sup>)
- $\rho_m$  = matrix density (kg/m<sup>3</sup>)

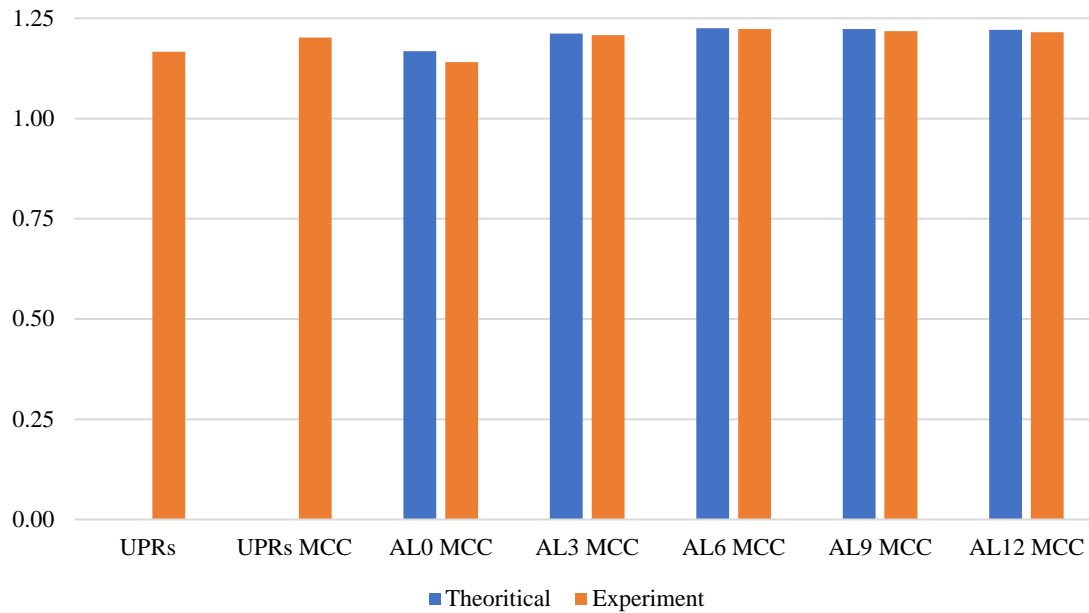
### 3 Results and Discussion

Density value of composites unsaturated polyester-cantala can be seen from Figure 4. The theoretical result value of unsaturated polyester composites reinforced cantala fiber indicates the higher result compared with the experiment density value. It is because of an assumption that rule of mixture is no porosity of natural fiber in polymer composites [19]. The biggest error is 2.31% occurring in cantala fiber-reinforced composites without alkali treatment as it still has many amorphous areas [14]. The increasing density value indicates the improved fiber structure leading to the improved mechanic strength, while the decreasing one indicates the damage of natural fiber's structure [20].

Density value of neat Unsaturated Polyester (UPRs) is 1.167 gr/cm<sup>3</sup>, while density value of Microcrystalline Cellulose Reinforced Unsaturated Polyester composites (UPRs MCC) is 1.202 gr/cm<sup>3</sup>. Then, the cantala fiber reinforced unsaturated polyester composites with microcrystalline cellulose addition without alkali treatment (AL0 MCC) has theoretical and experimental density values (1.168; 1.141) gr/cm<sup>3</sup>. Furthermore, the composites with varying fiber alkali treatment (AL3 MCC), (AL6 MCC), (AL9 MCC), and (AL12 MCC) has the following theoretical and experimental density value of composites (1.212; 1.208), (1.225; 1.223), (1.223; 1.218), and (1.221; 1.215) gr/cm<sup>3</sup>.



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**Figure 4.** Density UPRs – cantala composites

Figure 5 indicates the density value of UPRs MCC higher (2.17%) than neat UPRs. It is because density value of MCC is higher than that of unsaturated polyester. The addition of MCC will reduce void area significantly making the vacuum of composites reduced, so that density value increases [21]. Kiziltas et al. [17] also conducts a study comparing density value of neat nylon 6 and that of MCC reinforced nylon 6 polymer composites. The result of research indicates the density value of microcrystalline cellulose reinforced nylon 6 polymer composites is higher than that of neat nylon 6.

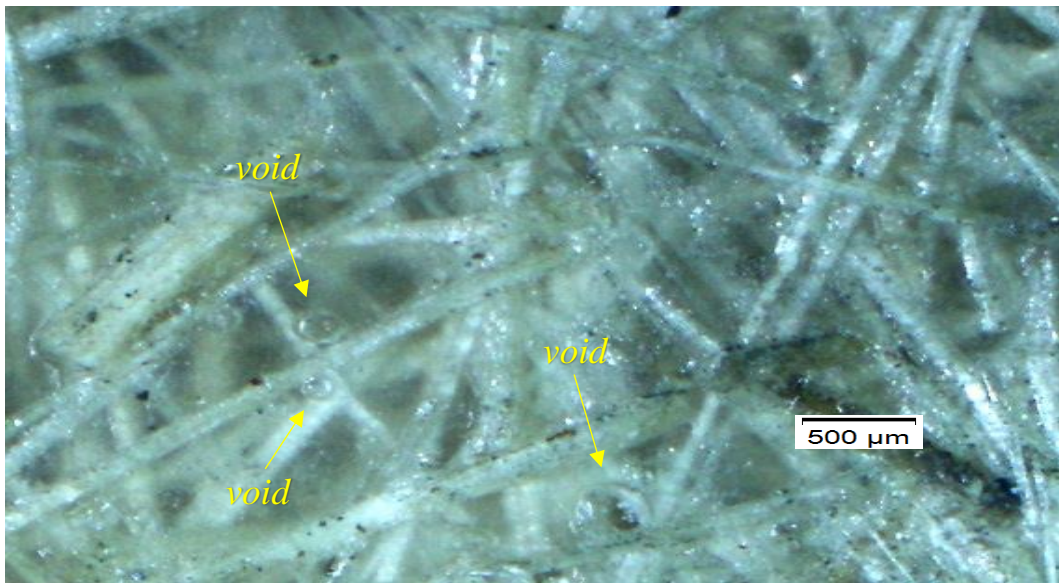
The density of cantala fiber reinforced unsaturated polyester composites increases not too significantly along with the increased duration of alkali NaOH treatment. It is because of the eroded amorphous compound like lignin and hemicellulose in cantala fiber making the crystalline area increasing. In addition, the eroded amorphous area will increase the composites density along with the reduced composites volume and the increased composites mass [14]. However, UPRs-cantala composites with alkali treatment for 9 and 12 hours decreases because of polymorphic transformation from cellulose I to cellulose II so that crystalline area of fiber decreases, leading to the decreased density value [22].

Previously, Anzaldo et al. [23] has conducted a research on the effect of alkali treatment on density value of agave cantala fiber reinforced Linear Low Density Polyethylene (LLDPE) composites. The study found that the density value of such composites is less (16.02%) than that of cantala fiber reinforced unsaturated polyester composites with microcrystalline cellulose addition. It may occur because the addition of microcrystalline cellulose will improve the distribution of micro-sized cellulose more broadly, so that the density of material will increase and the vacuum or void will be reduced, and density value will increase [21,24].

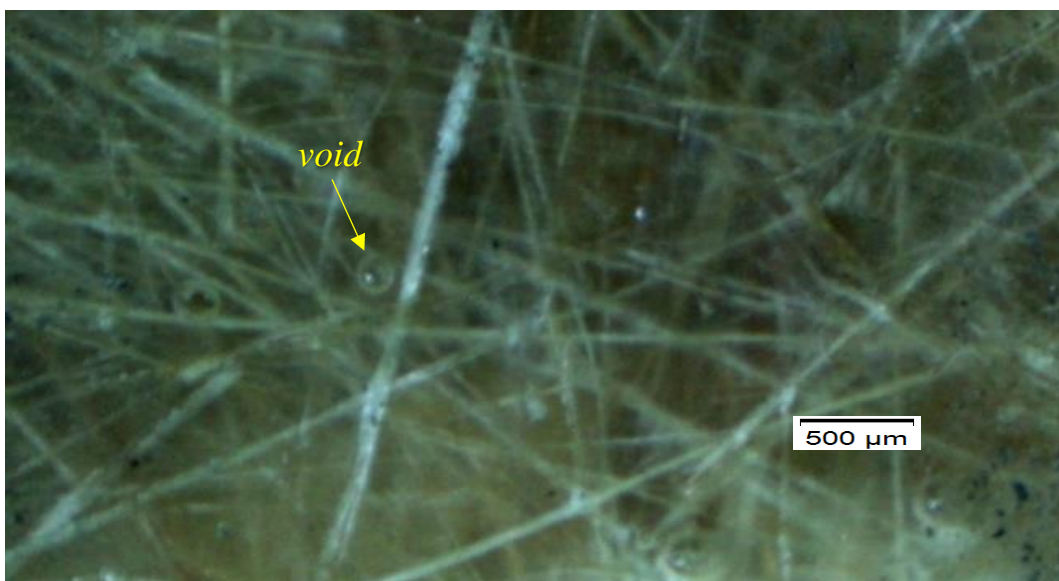
Figures 5 and 6 show the macro photograph of unsaturated polyester composites surface with cantala fiber reinforcement without alkali treatment and cantala fiber reinforced unsaturated polyester with alkali treatment for six hours. The fiber composites with alkali treatment for six hours seem to have better adhesiveness between fiber and matrix compared with the fiber composites without alkali treatment NaOH. It is because the fiber with alkali treatment for six hours can reduce amorphous area including hemicelluloses, lignin compound, and other dirty substances, leading the fiber to be coarser [25]. The coarser surface of fiber will increase the adhesiveness of fiber and matrix and decrease the void, thereby increasing the density value. Density and void has inversely proportional value, meaning that the lower the void of composites, the higher is the density value of composites [26]. In addition, fiber alkali treatment

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and MCC addition will increase the crystalline-phase area in the composites, leading to the higher density value. It can be seen that crystalline phase has higher density value than amorphous phase [27].



**Figure 5.** UPRs – cantala composites without alkali treatment (AL0 MCC)



**Figure 6.** UPRs – cantala composites with alkali treatment six hours (AL6 MCC)

#### 4 Conclusions

From the result of research and discussion on the density value of cantala fiber reinforced unsaturated polyester composites, the following conclusions can be drawn:

1. The longer the duration of alkali NaOH treatment and MCC addition into cantala fiber reinforced unsaturated polyester composites, the higher will be the density value. The alkali treatment will decrease the interphase of matrix fibers so that void also decrease. Decreasing void will increase the density value of composites.
2. The highest density value of UPRs-cantala composites is  $1.223 \text{ g/cm}^3$ , found in alkali treatment for six hours and the addition of microcrystalline cellulose filler.

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