PREPARATION AND CHARACTERIZATION OF RAMIE CELLULOSE NANOFIBERS/CaCO3 UNSATURATED POLYESTER RESIN COMPOSITES

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PREPARATION AND CHARACTERIZATION OF RAMIE CELLULOSE NANOFIBERS/$CaCO_3$ UNSATURATED POLYESTER RESIN COMPOSITES

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ABSTRACT

In this study, Ramie Cellulose Nanofiber (CNF) and $CaCO_3$ were used as a reinforcement of composite with Unsaturated Polyester Resin (UPR) matrix. Ramie CNF was prepared by HEM (high energy milling) and dried in drying oven at 50 °C for 24 h. It mixed with UPR matrix by hand layup casting method. Methylethylketone peroxide (MEKPO) and $CaCO_3$ were added in the mixture solution with the amount of 1wt% and 2wt% respectively by stirring manually. The tensile test was carried out to know the mechanical properties of biocomposite. The fracture surface was observed by using Scanning Electron Microscopy (SEM). Fourier Transform Infrared (FT-IR) and Thermogravimetric Analysis (TGA) were also performed to know the functional group and thermal stability, respectively. The result shows that the addition of CNF and $CaCO_3$ able to increase thermal stability and mechanical properties of biocomposite.

Keywords: ramie, cellulose nanofibers, unsaturated polyester resin, composite.

1. INTRODUCTION

Composites are solid materials consisting of combination two or more materials in which each material retains its properties. Another definition of composites is the heterogeneous mixture of two or more heterogeneous phases bonded together [1]. The use of biocomposite materials in the industrial world has significant improvements due to its superiority such as biodegradability, good thermal properties, mechanical properties, low density, inexpensive and renewability. Some natural fillers can be used as polymer boosters to replace synthetic fibers such as rattan, kenaf, ramie, water hyacinth, coconut husk, limestone, clay, and others [2, 3, 4]. In recent years, many researchers focused on the use of some fillers into a matrix. Micro or nanometer fillers will be more advantageous as matrix reinforcement due to good interaction between filler-matrix. It is affected of larger surface contact area [5]. Several methods for synthesis CNF of natural fibers have been carried out by researchers. One of them is chemical-mechanical method by Alemdar *et al.* They studied about extraction of cellulosic nanofibers from agricultural residues such as wheat straw and soybean husk for reinforcement in biocomposite [6]. In that study, they obtained cellulosic nanofibers with a diameter 10-80 nm. The increasing crystallinity index of nanofibers wheat straw and soybean skin was 35% and 16 % respectively than untreated fiber. Generally, the fiber is suitable as a reinforcement in matrix composites as reported by Abral *et al.* [4]. Matrix are widely used for biocomposites such as rubber, polylactic acid, polyvinyl alcohol, acrylic latex, polyethylene, and thermoplastic starch. One of the most popular thermoset polymers is the Unsaturated Polyester Resin (UPR) which is widely applied to the motor vehicle body, ship, and aviation industry. UPR has some uniqueness among others. Its properties are transparency, water resistance, good thermal stability, and high elastic modulus and strength. However, UPR also has limitations such as low toughness, brittle and small elasticity [7]. Therefore, the ramie CNF was prepared by chemical-milling treatments to improve the properties of UPR matrix followed by the addition of $CaCO_3$. According to the best our knowledge, the hybrid material with ramie CNF and $CaCO_3$ as a filler in Unsaturated Polyester Resin matrix composites has been not reported yet. In this study, biocomposite materials were characterized by testing of mechanical, physical, and thermal properties. Scanning Electron Microscopy (SEM) was also carried out to know the morphological of fractured composites.

2. MATERIALS AND METHODS

2.1 Materials

Ramie is a stem fiber plant which obtained from the experimental garden of Agricultural State Polytechnic of Payakumbuh, Indonesia. The process of stem bark removal (decortication) from the main stem was done manually. The pro-analytical (PA) chemicals used were sodium hydroxide (98% NaOH of Merck Sigma-Aldrich), Potassium hydroxide (KOH Merck Millipore), sodium chlorite ($NaClO_2$ Merck Pubchem), acetic acid and $CaCO_3$ (397 nm), respectively.

2.2 Preparation of ramie CNF

The stems were cut into 10-20 mm long and dried under the sun for 3 days (water content 9-10%). Lignin and hemicellulose of ramie fibers were removed by a pulping process in the digester (high-pressure reactor) with 18% NaOH solution at temperature 170 °C and pressure 7-9 kg/cm^2 for 2 h [8, 20]. The pulp fiber was washed with distilled water until pH 7 (neutral) and continued by bleaching process 1 with mixing 5% sodium chlorite

($NaClO_2$) and acetic acid at temperature 70°C for 2 h. The result of bleaching process 1 was neutralized with aquadest. Then, the bleaching process 2 was done with 4% KOH at 80 °C for 1 h to reduce the residual non-cellulose content [9]. It was rinsed with aquadest until pH 7 and dried in a bed dryer until become granular. The ramie fiber granular was treated by HEM (high energy milling) for 60 min to obtain cellulose nanofibers ramie (CNF ramie).

2.3 Fabrication of ramie CNF/$CaCO_3$-UPR composites

Ramie CNF/$CaCO_3$-UPR composites was prepared by hand lay-up techniques. Unsaturated Polyester Resin (UPR) was prepared as much as 100 ml and added ramie CNF, $CaCO_3$, catalyst with composition in Table-1.

Table-1. The composition of Ramie CNF/$CaCO_3$-UPR composites.

Control	Polyester (ml)	Ramie CNF (gram)	$CaCO_3$ (gram)	MEKPO (%)
Control / UPR	100	0	0	1
UPR + 2CNF	100	2	0	1
UPR + 4CNF	100	4	0	1
UPR + 6 CNF	100	6	0	1
UPR + 2 $CaCO_3$	100	0	2	1
UPR + 2 CNF 2 $CaCO_3$	100	2	2	1

Ramie CNF and $CaCO_3$ were added in UPR while stirring manually, then placed into an ultrasonic bath in other to uniformly distributed of filler in the UPR matrix. After 30 min, the MEKPO catalyst was poured into the mixing solution while stirring until evenly distributed. The mixing solution was poured into a glass mold with dimensions 200 mm x 200 mm x 3 mm. To overcome the samples sticky on the mold, then the mirror glaze was applied on the surface of the mold. Let it dry before being used for the mold. The drying process was carried out at room temperature for 24 h. The sample was released from the mold and prepared accordance with standard which become reference before its characterization.

2.4 Particle size analyzer (PSA)

Ramie cellulose nanofiber particle was measured by using Beckman Coulter Delsa Nano C instrument. Spectroscopy of photon correlation (PCS) was used to determine the particle size by calculating the degree of fluctuation in the intensity of the laser beam spread by particles that spread through the liquid. The heavily preheated CNF fluid was fed into the centrifuge tube of the appliance. The condition of test was conducted at temperature 25 °C, viscosity 0.88 cP, scattering intensity (SI): 9581 cps, refractive index (RI): 1.3332. The particle measurement data was processed by using Delsa Nano software.

2.5 Tensile test

Tensile testing machine with Brand Com-Ten Series 95T was used to measure tensile strength, elastic modulus, and elongation at break of ramie CNF/$CaCO_3$-UPR composite samples. The speed and temperature for testing were 2 mm/min and room temperature, respectively. American Society for Testing Materials (ASTM) D 638-03 (type III) standard was used to determine the tensile test specimen. The specimen dimensions was presented in Table-2 and Figure-1.

2.6 Fourier transform infrared (FT-IR)

Fourier Transform Infrared (FT-IR) analysis was performed to detect the presence of functional groups on UPR and some filler variations. The FTIR spectrum was recorded by using Perkin Elmer FT-IR frontier spectrometer in the measurement range 400-4000 cm^{-1}.

Table-2. The size of tensile test specimens [11].

Dimensions (see drawings)	Thickness 7(0.28) or under, mm (in)		Thickness > 7 to 14 (0.28 to 0.55), mm (in)
	Type I	Type II	Type III
W-Width of narrow section EF	13 (0.50)	6 (0.25)	19 (0.75)
L-Length of narrow section	57 (2.25)	57 (2.25)	57 (2.25)
WO-Width overall, min G	19 (0.75)	19 (0.75)	29 (1.13)
LO-Length overall, min H	165 (6.50)	183 (7.20)	50 (2.00)
D-Distance between grips	115 (4.50)	135(5.30)	115 (4.50)
RO-Radius of fillet	76 (3.00)	76 (3.00)	76 (3.00)

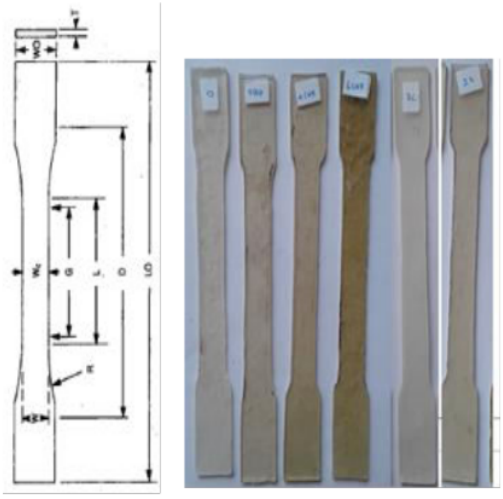

Figure-1. The shape tensile test specimens [11].

2.7 Scanning electron microscopy (SEM)

The fracture surface of all samples tested was observed using SEM (Hitachi series 3400 N) equipment. The operation voltage was 10 kV. The observation of sample was limited in the micrometer area.

2.8 Thermogravimetric analysis (TGA)

Thermogravimetric Analysis (TGA) was used to determine the characteristics of the all samples tested. The TGA4000 equipment (a product of Perkin Elmer) was used in this study. The heat rate was 10°C per min under a nitrogen atmosphere in range temperature from 50 to 500 °C.

3. RESULTS AND DISCUSSIONS

3.1 Particle size distribution of ramie CNF

The High Energy Milling (HEM) process was performed to reduce the size of the ramie fiber particles that have undergone chemical processes. The initial particles were 11-15 µm. After milling process was done for 60 min, the average size of ramie CNF was 296 nm. The distribution size of ramie CNF can be seen in Figure-2. In the process of milling, there has been a cavitation phenomenon that breaks the micro to nanoparticles due to the influence of friction and collisions between particles. Homogeneity process in generating nanomaterials was desirable due to it is highly influential on the properties and characteristics (heat, electricity, and mechanics) that are optimal and stable in its application. The force in milling process able to produce the energy transferred to the particles and break up to nanometer in size. The heat also present while milling process. It can be influenced by milling time. Recombination boundary of ramie fiber formed after experiencing vibrations, lattice voids and irregularities due to temperature effect. This phenomenon was called recrystallization [10].

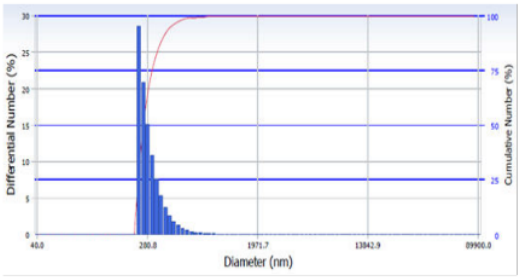

Figure-2. Particle size distribution of Ramie CNF.

3.2 Tensile strength analysis

Figure-3 shows the effect of filler content on tensile strength (σ) ramie CNF / $CaCO_3$-UPR composites. Maximum tensile strength was obtained at 44.44 MPa without addition filler (UPR). The addition of 2, 4 and 6% ramie CNF decreased in tensile strength 39.34, 34.70, and 32.25 MPa, respectively. It was indicated due to bad adhesion bonding between ramie filler (hydrophilic) and the polyester matrix (hydrophobic).

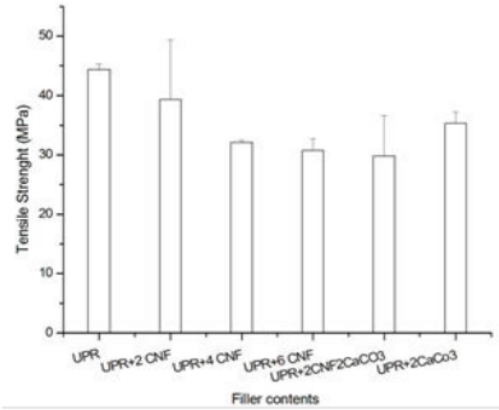

Figure-3. Tensile strength of Ramie CNF/$CaCO_3$ UPR composites.

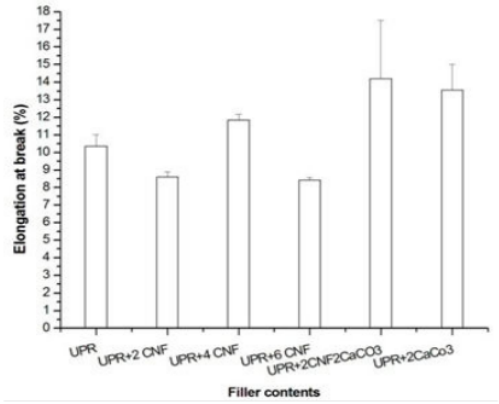

Figure-4. Elongation at break Ramie CNF/$CaCO_3$ UPR composites.

The similar study was previously reported, that the use of fillers with grain size greater than 80 nm in the composite able to decrease in tensile strength (σ) as increasing filler concentration in the matrix [11, 12]. Other studies by Fan *et al.* also stated the different case. They investigate the tensile strength of polypropylene-montmorillonite composites by addition of clay content [13]. They reported that addition of clay in matrix decreased a tensile strength. This case due to of the unfavorable bonding interface between ramie CNF and UPR matrix, so it can presence air cavity. The tensile strength of 2% $CaCO_3$ in UPR matrix was 35.32 MPa. This value was higher than compared with the addition of 2% ramie CNF. It was indicated due to the uniformly distribution size of $CaCO_3$ than ramie CNF. Figure-4 shows elongation at break of ramie CNF/$CaCO_3$ UPR composites. The addition of 2$CaCO_3$ and 2CNF2$CaCO_3$

increase the elongation at break of UPR matrix by 13.56% and 14.20% compared to the addition of 2CNF, respectively. It can be said, that the $2CaCO_3$ filler and $2CNF2CaCO_3$ able to improve the elasticity of the composite.

3.3 Fracture surface observation

Fracture surface of composite specimens was observed by SEM at Figure-5.

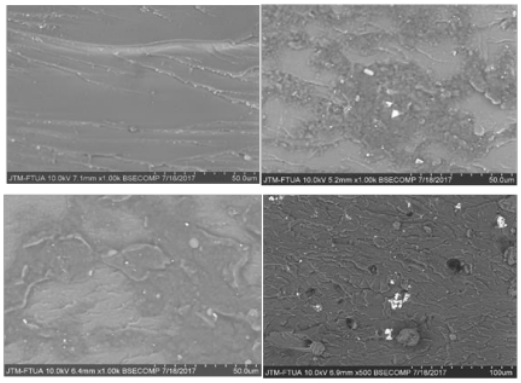

Figure-5. SEM images of fracture surface composites a) UPR, b) UPR+$2CaCO_3$, c) UPR+2CNF, d) UPR+$2CNF2CaCO_3$.

In Figure-5b and 5d there was a clear separation between polyester and $CaCO_3$, but for lamin cellulose fibers was not clearly visible. Cellulose that has undergone the process of pulping and bleaching significantly increase the compatibility between matrix and fiber. It affected the better adhesion bonding between matrix and fiber. This phenomenon was also the same with Mulinari *et al.* (2009). They studied with sugarcane bagasse as filler. Cellulose modification improves the compatibility between HDPE and more fibers than unmodified cellulose [14].

3.4 Functional group determination

FTIR spectrum was used to know the physical structure and functional groups of UPR reinforced by ramie fibers and $CaCO_3$ at Figure-6. The widespread peak for wave uptake occurs at 3400-3300 cm^{-1} was due to the OH bond between cellulose and water absorbed [15]. The peak around 2900 cm^{-1} was caused by the vibration of the C-H stretching of cellulose [15]. The peak at 1630-1640 was caused by bending vibration of O-H from the water absorbed [16]. The wave absorption at the peak of about 1719 cm^{-1} was indicated the stretching of the C=O group of esters. Calcium carbonate appears visible on the peaks with wavelength 875 - 712 cm^{-1} [17]. The peak concentrated at 1256 cm^{-1} shows the C-C group of polyesters. Since the ramie fiber contain large amounts of cellulosic hydroxyl groups in their structure, they able to interact with the polar ester linkages, to obtain a strong adhesion between the fiber and its matrix. In general, the FTIR graph shows little change due to the rise of wave absorption by $CaCO_3$ and cellulose.

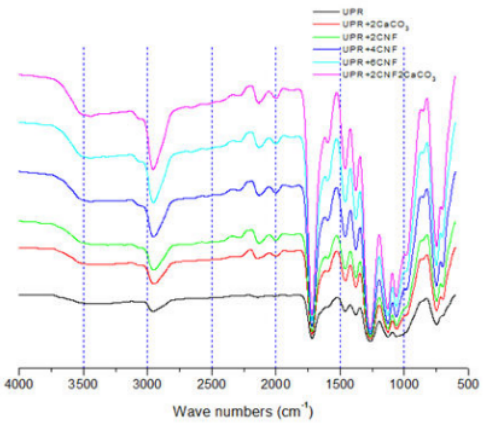

Figure-6. FTIR of Ramie CNF/$CaCO_3$ UPR composites.

3.5 Thermal stability analysis

Thermogravimetric analysis has been performed to indicate the thermal stability of some variations in the addition of ramie CNF and $CaCO_3$. Figure-7 shows the physical change of material due to weight loss with rising of temperature. It can be seen, that there were three steps of degradation area. First step, the weight loss at 90 – 145 °C. In this step, the missing component was moisture uptake as reported by Abral *et al.* [18]. At the second step, the extreme degradation occurs at 370-450 °C corresponds to cellulose and UPR matrix decomposition [7]. Thermal stability decreases with the addition of ramie CNF due to the organic material was easily decomposed by high temperature. The thermal stability of $CaCO_3$ was better than ramie cellulose nanofiber. The $CaCO_3$ filler can improve the thermal stability of the composite material [7]. This phenomenon able to influence and increase the thermal stability of composites. The improvement thermal stability in composites due to strong interaction between UPR matrix and CNF + $CaCO_3$ filler. The strong matrix filler interactions may be another reason for the shift of the main decomposition peak to higher temperatures compared to not added $CaCO_3$. The third step occurs at > 475 °C which indicated decomposition of all samples. In this step, all samples was become ash carbon as reported by previously study [19]. It can be conclude, that the stronger engagement between matrix and filler make composites more difficult to be decomposed.

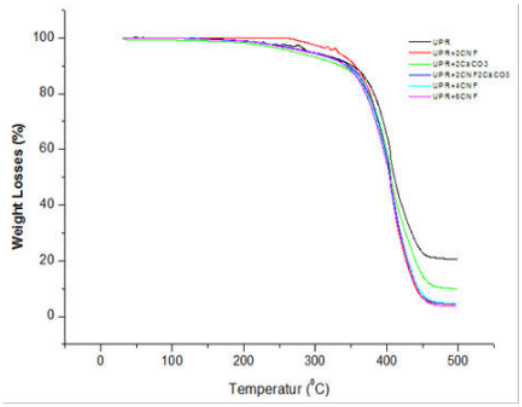

Figure-7. TGA Ramie CNF/$CaCO_3$ UPR composites.

4. CONCLUSIONS

From this research, we can take some conclusions. The size of ramie CNF and $CaCO_3$ as filler content were 296 nm and 397 nm, respectively. The tensile strengths for CNF fillers and $CaCO_3$ UPR composites decreased with increasing concentration of filler addition. Decreasing strength is due to the unfavorable bond between the filler and the matrix as well as the presence of voids / air cavities trapped in the UPR matrix composite. This phenomenon was agreement with SEM observation. SEM shows clear separation between matrix and filler. The addition of $CaCO_3$ able to improve the thermal stability of ramie CNF / $CaCO_3$ polyester composite. For functional group, all samples have a similar peak (did not change the chemical structure).

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