

## EFFECT OF RATTAN BARK NANOFIBER MICROSTRUCTURE WITH HIGH ENERGY MILLING METHOD TO QUALITY MECHANICAL BIOCOMPOSITES

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### ABSTRACT

Rattan bark fiber is waste of raw rattan production activities. It usually used for roofing houses and strap goods in the traditional markets. Rattan bark fiber containing about 40% cellulose, which was potential to be used as filler for composite applications. Nanotechnology based nanoparticles with High Energy Milling (HEM) has been applied in this research to find the optimum particle size to be implemented as automotive component filler. Three HEM has been applied (1 hour, 5 hours, and 10 hours), and without HEM (0 hours) as control. Measurable particle size is the size of the single particle. Data in the form of particle size distributions, intensity, number and volume of distribution, so it can be assumed to describe the overall condition of the sample. The result shows that 5 hours milling produce the smallest particle size (16.22 nm) and z averages (129.78 nm). Particle Size Analyzer (PSA) results was related to the Scanning Electron Microscopy (SEM) images of 5 hour milling, particle become smaller and look the same or homogeneous as balls. These results indicate that the cellulose in the form of three-dimensional nanoparticles is spherical, and the mechanical tests show that biocomposite rattan bark fiber performance was highest among the other natural composites (kenaf, acacia, water hyacinth, banana, coconut).

Keywords: Rattan bark fiber, biocomposites, nanoparticles, high energy milling

### ABSTRAK

Serat rotan adalah kulit kayu limbah dari kegiatan produksi rotan mentah. Ini biasanya digunakan untuk atap rumah dan barang tali di pasar tradisional. Rotan serat kulit kayu mengandung sekitar 40 % selulosa, yang berpotensi untuk digunakan sebagai pengisi untuk aplikasi komposit. Nanoteknologi berbasis nano partikel dengan Milling Energi Tinggi (HEM) telah digunakan dalam penelitian ini untuk menemukan ukuran partikel optimum untuk diimplementasikan sebagai komponen filler otomotif. Tiga HEM telah diterapkan (1 jam, 5 jam, dan 10 jam), dan tanpa HEM (0 jam) sebagai kontrol. Ukuran partikel yang terukur adalah ukuran partikel tunggal. Data dalam bentuk tiga distribusi yaitu distribusi ukuran partikel, intensitas, jumlah dan volume, sehingga dapat diasumsikan untuk menggambarkan kondisi keseluruhan sampel. Hasilnya menunjukkan bahwa 5 jam penggilingan menghasilkan ukuran partikel terkecil (16.22 nm) dan z rata-rata (129,78 nm). Ukuran partikel Analyer (PSA) hasil ini terkait dengan Scanning Electron Microscopy (SEM) selama 5 jam penggilingan, partikel menjadi lebih kecil dan homogen. Hasil ini menunjukkan bahwa selulosa dalam bentuk nanopartikel tiga dimensi berbentuk

*bola, dan uji mekanis menunjukkan bahwa biokomposit rotan kinerja serat kulit adalah tertinggi di antara komposit alam lainnya (kenaf, akasia, eceng gondok, pisang, kelapa )*

*Kata kunci: serat rotan, biokomposit, nanopartikel, high energy milling*

## INTRODUCTION

The use of synthetic fibers in the composite increased. It makes its own problems where waste from synthetic fibers can not be degraded naturally. In response to these problems, Europe and some Asian countries set up a regulatory requirement consumables (end of life). In this regard, the use of natural fibers is one of the best choices.<sup>1</sup> Rattan bark waste is a natural fibrous material containing an average of 45% cellulose and is available throughout the year from rattan forest.<sup>2</sup> Thus rattan bark fiber waste has the potential to be utilized.

Nanotechnology is important in the world of engineering material because people are trying to integrate a function or a job in a smaller scale. Integrating a function machines or products in smaller size means not only beautify but also minimize the energy required and speed up the process and save costs jobs. One of its applications is the synthesis of rattan bark waste in the form of nanoparticles on bionanocomposite applications using High Energy Milling (HEM).

To produce bionanocomposite with physical and mechanical properties of the optimal, required input data (frame of reference) relating to the microstructure and mechanical properties of the constituent elements that can answer the need for composites.

## EXPERIMENTAL

The material used in this study were rattan (*c.scipionum* Burr), polypropylene, PPMA, ethanol. The tools used are disk mill, electromagnetic shaker, High Energy Milling E-3D, Scanning Electron Microscopy, Particle Size Analyzer, single screw extruder, compression molding, tool ASTM D638 tensile test.

The research began with the stage of the synthesis and characterization of nanoparticles rattan bark, sample preparation and synthesis of nanoparticles with HEM. Rattan bark that has been selected, then boiled and dried to ensure rattan really dry and soft, then milling and sheaking using a disk mill and electromagnetic shaker to get the size of <75  $\mu\text{m}$ . Furthermore, milling sample size of <75  $\mu\text{m}$  using HEM with time of 1 hour, 5 hours and 10 hours.

Characterization of nanoparticles using Particle Size Analyzer (PSA) to determine the size of the nanoparticles, the characterization of Scanning Electron Microscopy (SEM) for surface morphology analysis.

At the stage of the synthesis and characterization bionanocomposite, polypropylene polymers used as matrices and rattan bark fibers as filler.

Composition of bionanocomposite comprise of polypropylene 93%wt, 5%wt rattan bark fiber and 2%wt PPMA. Samples were prepared using extrusion equipment to mix material then Compression Molding and Punching to get the size of the specimen in accordance with standard mechanical characterization (tensile test).

## RESULTS AND DISCUSSION

Cellulose  $(C_6H_{10}O_5)_x$  is a major part of woody plants that make up the component pieces lengthwise. The separation of the good fiber and guarantee the optimal condition and original content of fiber can be maintained. The basic principle of the separation of the fiber is separated from the building blocks of non-cellulose fibers allowing the fibers can be mechanically extracted after drying.<sup>3</sup>

Figure 1 shows that the extraction of cellulose bark of rattan, which is produced with a disk mill produces fibers on the size of the stages are long fiber, 5 cm, 1 mm, 500  $\mu\text{m}$  to  $<75 \mu\text{m}$ . Fiber size order of  $<75 \mu\text{m}$  was followed by using HEM tool to obtain the order of nanometer size particles. The purpose of the preparation of cellulose rattan bark is to simplify the optimization process of cavitation HEM.

Figure 2 shows that during the HEM, collisions between balls with rattan bark fibers with speed 1400 rpm vial can destroy the fibers and soften up the nanometer size, cavitation process marked after milling rattan fiber bark smoother and shrink.

HEM using three-dimensional motion and rotation in the vial so that the mechanism of nanoparticle formation process amorfisasi and more quickly and effectively. HEM can be used for mixing, homogenizing, mechanical milling, mechanical alloying, and make the emulsion. The advantage is manufacturing nanoparticles without the addition of chemicals, smoothing material to the nanometer scale, to mixing (mixing) and uniformity (homogenization), making alloys making mechanical-chemical reactions.<sup>4</sup>

To determine the particle size rattan bark fibers used HEM results of the two approaches, namely using the PSA analysis and SEM. Characterization of PSA, where the particles dispersed in a liquid medium so that the particles are not mutually agglomeration. Measurable particle size is the size of a single particle. Data obtained in the form of three particle size distributions, namely intensity, number and volume of distribution, which can be assumed to describe the overall condition of the sample.

Table 1 indicates the results of HEM from 1-10 hours. 1 hour of milling has a minimum particle size of 81.30 nm and then at 5 hours is optimum HEM and produce a homogeneous particle cavitation until 16.22 nm but the size of the rise time is 10 hours milling 22.39 nm. This is due to the filler is an organic material, so has the nature of a special sensitivity to temperature compared with the inorganic material/synthetic.



Figure 1 Stages milling rattan bark fiber long fiber (a), particles  $<75 \mu\text{m}$  (b), and nanoparticles (c)



Figure 2 High Energy Milling Process rattan bark fibers fiber filled vial and balls (a), after the milling process (fibers shrink) (b), fiber and milling yield (c)

Table 1 Comparison of PSA results, HEM 1 hour, 5 hours and 10 minutes

Time	Minimum (nm)	Modus (nm)
1 hour	81.30	269.22-309.11
5 hour	16.22	16.22
10 hour	22.39	22.39

Besides, the longer the time HEM, heat during the process also increased and the constituent atoms of the sample has a margin of arrangement or amalgamation themselves up after a vibration, lattice vacancies and disorder caused by the temperature.

SEM data processing based on the detection of secondary electrons (reflection) of surface samples. Electrons do not penetrate the footage but only reflected the result of the collision of electrons with the surface of the footage captured by the detector and processed into an image that has been magnified object structure.

Figure 3a shows the SEM image before the HEM, which form elongated fibers with a diameter of  $<50 \mu\text{m}$ . In this SEM image looks a black spot organic components (trakeid), where the pores are connected to one another with a substance lignin. This is what distinguishes between natural fibers with synthetic fibers. Synthetic fibers are made from inorganic materials with specific chemical composition that can be set according to application needs, so that the nature and relatively uniform size and strength of the fiber can be pursued together along the fiber.

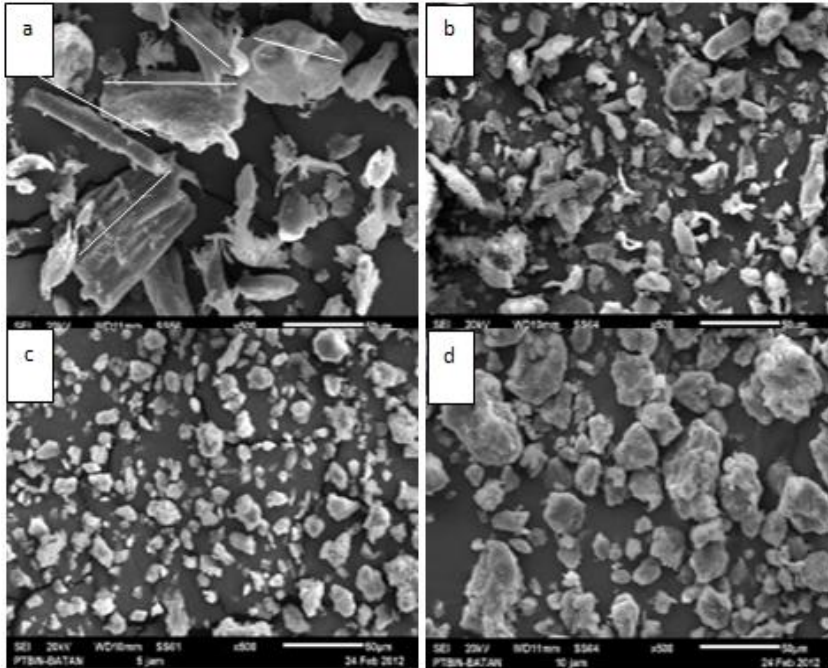


Figure 3 SEM image at 500x magnification variation time 0 hour (a), 1 Hour HEM (b), 5 Hours HEM (c), and 10 Hours HEM (d)

SEM image of Figure 3b 1 hour HEM showed a smaller fiber size dbandingkan without HEM process. While Figure 3c SEM image showing 5 hours HEM particles become smaller and look alike or homogeneous as balls. While the SEM image of Figure 3d 10 hour HEM nearly equal to 5 hours of milling but different sizes because some particles are agglomeraton making it look bigger. The growth rate depends on the temperature. Increased temperatures increase the thermal vibrational energy, which includes atoms accelerate grain boundary diffusion of small grain into large grains.<sup>5</sup>

Extrusion is the process of treatment is a combination of mixing (mixing), pengulenan (kneading), agitation (shearing), heating (heating), cooling (cooling), printing (shaping). The principle of operation is the same for all extruder. The raw material is inserted and passed along the extruder. When moving along the extruder, die small volume limit and hinder the movement of materials. As a result of high pressure materials. During moving along the extruder, screw rotate materials (kneading) and turn it into a semisolid which is plastic. Mixed polypropylene, PPMA and rattan bark fibers before they are printed through the compression performed using a single screw extrusion blending four replications with a speed of 45 rpm.<sup>6</sup> Results obtained in the form of spherical dots (Fig. 4 a). Once that is done the Compression colin P 300P. And continued with the cutting line with a mechanical test (Fig. 4 b).

Research on composite polypropylene filled with natural ligno-cellulosic fibers have been reported to the researchers<sup>6,7,8</sup>, but the research on composite polypropylene filled with rattan bark using HEM has not been done before. The results of mechanical testing rattan bark fiber biocomposite and other biocomposites can be seen in Table 2.

As a result of the fibrous structure and strong hydrogen bonds. Cellulose has a high tensile strength. Thereby, greater the higher the cellulose content of the fiber tensile strength. Fiber-reinforced composites rattan bark and kenaf fibers exhibit high tensile strength compared with acacia fiber, coconut fiber. Because the cellulose fiber content of rattan is also high ranging between 37-44% and 45-60% ranges kenaf fiber (Table 3).

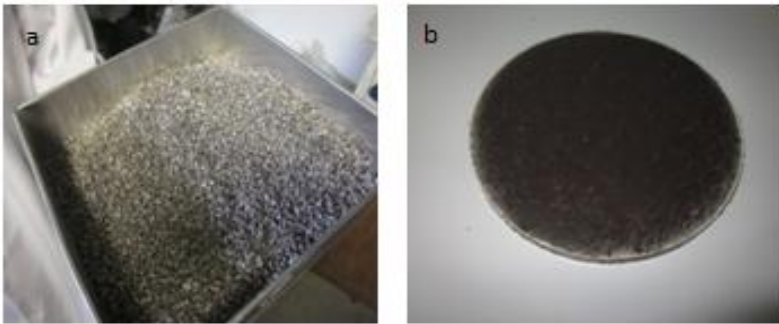


Figure 4 Results of extrusion (a) and compression (b)

The addition of rattan fibers into the composite lower elongation at break its properties compared with hemp fiber. This is due to the hydrophilic nature of the fiber that can absorb water. This condition will result in reduced effects of physical bonding between the fiber and polypropylene surfaces<sup>9</sup>. Decrease in elongation at break properties on the bark of rattan fiber biocomposites showed that the bark of rattan fibers more rigid than the flax fibers.

Hydrophilic nature of the fiber rattan bark because it contains cellulose and lignin, both ingredients are rich in hydroxyl groups are very easy to absorb water through hydrogen bonds within the cell wall<sup>10</sup>. The content of cellulose and lignin in the rattan fiber is 37.36% and 22.19%.

Table 2 Mechanical properties of biocomposites comparisons Rattan Bark Fiber and Natural Fiber

Biocomposite filler	Tensile Strength (Mpa)	Elongation at break (%)
Kenaf fiber	16.85	2.09
Acacia fiber	13.03	1.56
Water hyacinth fiber	14.72	1.75
Banana fiber	16.18	1.85
Coconut fiber	13.61	1.79
Flax fiber	17.25	4.50
Rattan bark fiber	18.32	3.93

Tabel 3 Several Natural Cellulose Fiber Content.<sup>6,11-13</sup>

Fiber	cellulose (%)
Kenaf	45-60
Banana	45-50
Rattan	37-44
Acasia	15-30
Coconut	14-20

## CONCLUSIONS

Use of High Energy Milling (HEM) after a disk mill to produce rattan bark fibers were homogeneous particle size until 16:22 nm with an optimum time of 5 hours. The results of mechanical testing tensile strength, fiber bionanocomposite rattan skin was higher among the other natural composites (kenaf fiber, acacia, water hyacinth, banana, oil reservoir, hemp) but the value of elongation at break is still under biocomposite hemp. To produce the mechanical properties of the bark of rattan fiber bionanocomposite optimum further research is needed related to the method, the synthesis and use of appropriate coupling agent.

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