

EFFECTS OF NANOPARTICLE FILLER ON THERMO-PHYSICAL PROPERTIES OF RATTAN POWDER-FILLED POLYPROPYLENE COMPOSITES

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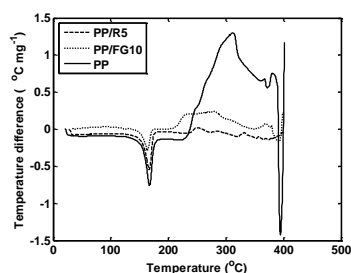
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Graphical abstract



Abstract

Poor bonding with its accompanying matrix remains the major factor that inhibits the widespread use of natural fiber as filler for composite materials. Recent literature has suggested that this matter can be addressed by applying fillers in nanometer size. Thus, the present study is carried out to investigate the effects of incorporating rattan powder of nanoparticle size into polypropylene (PP) on its thermo-physical properties. Production methodology of nanoparticle, particle analysis, morphological study, and thermal analysis were carried out and discussed. The composites were produced using the twin extrusion moulding. The production of nanoparticle was found optimal under 30 minutes of milling time, confirmed by means of particle analyzer and morphological inspection. Better thermal stability was found in PP reinforced with 5% rattan nanoparticle filler compared to the neat PP and PP with 10% glass fiber.

Keywords: Composites, nanoparticle size, natural fillers, polypropylene, thermal properties

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1.0 INTRODUCTION

Interest towards fabrication of natural filler composites and exploring their benefitting properties have been escalated in recent years [1-4]. Establishment of several new sets of regulation that are renewable resources-inclined push industries to manufacture and improve existing available composite materials to be more environmental-friendly. It is well-recognized rather recently that the

use of the natural filler reinforced composites have increasing significant advantages than traditional fillers such as glass and carbon fiber [1]. Not only are these composites low cost and low density [2], several studies have found that the natural filler reinforced composites exhibit improvement in thermal and mechanical properties in comparison to existing more commonly available types [5-7].

Several works had explored the use of natural fillers as new reinforcement agents in polymer-based

composite, the materials of which include Alfa [8], coir [9], banana [10], calcium carbonate [11], clay [12], graphene [13], and nut-shells [14]. Essabir et al. [14] observed an improvement in the Young's modulus when nut-shells of argan particles were used as filler compared to the neat polypropylene, with a gain of 42.65%, 26.7% and 2.9% for 20 wt.% particle loading. Arrakhiz et al. [8] had similarly found a Young's modulus enhancement in PP reinforced with Alfa fiber under esterification treatment, with a 35% rise when compared to alkali and etherification treatments.

Numerous studies had investigated effects of rattan filler loadings in several composites [15-18]. Muniandy et al. [15] found an increase in the stress at 100% elongation (M100) of rattan powder-filled natural rubber (NR) composites without the silane coupling agent. Balakrishna et al. [16] reported that the tensile strength, elongation at tensile failure, and impact strength decreased, while the stabilization torque, thermal stability, and water absorption increased by rising rattan filler loading between 0 and 40 phr in PP composites. Balakrishna et al. [17] observed the deterioration of the fatigue life of rattan powder-filled natural rubber composites by increasing the rattan powder loading. Recently, Balakrishna et al. [18] found that incorporating composite rattan powder and 10 phr kaolin fillers in polypropylene raise 12 % in the composite tensile modulus compared with 0, 2, 4, and 6 phr kaolin filler. Muniandy et al. [15] also found that the tensile strength, elongation at break (Eb), stress at 100% elongation (M100), and stress at 300% elongation (M300) of all composites increase corresponding to the rise in the carbon black (CB), mica, and calcium carbonate (CaCO₃) filler-loading ratios. While all the above studies examine the effects of rattan powder with microparticle size on the physical and mechanical composite properties, there is noticeably a lack of study on the influence of rattan powder in the nanoparticle range as filler on thermo-physical properties of composites. Therefore, the present study aims to investigate the effects of rattan powder nanoparticle on the thermo-physical properties of rattan powder-filled polypropylene composites. Knowledge of these properties is essential to the manufacturing of a better performing engineering material from a renewable resource.

To date, several studies have evaluated physical and mechanical properties of polypropylene composites using varying filler loadings [19-24], varying particle size loadings [25], and chemical treatments [26-27]. Beckermann and Pickering [27] found that hemp fiber reinforced polypropylene composites show increase in both tensile strength and Young's modulus when treated using alkali. Alternatively, Yao et al. [20] evaluated the effects of varying shell waste and modified powder intensities on the mechanical and thermal properties of composites. Aligning with this research trend, the present study therefore focuses on the effects of rattan nanoparticle filler loadings on thermo-physical

properties of rattan powder-filled polypropylene (PP) composites. For comparison, commercially available E-glass composites are included also in the current study. The paper is arranged as follows. Materials used and associated fabrication method are first described. Briefly, preparation methods for the filler and composite are offered. This is then followed by the exploration methodologies and findings for particle analysis, morphology study, and thermal properties. This paper ends with a concluding section highlighting the main findings.

2.0 MATERIALS AND METHODS

2.1 Materials

In this study, rattan pole wastes (see Figure 1) were collected from Pontianak, West Kalimantan, Indonesia. Rattans are classified under the palm family (Palmae or Arecaceae). There exist currently 610 different species under 13 genera. They are chiefly available in the Southeast Asia region. Polypropylene used in this study has a melt flow index of 4.5 g/minute at 230 °C. It has a density of 0.95 g cm⁻³. Synthetic glass fiber (E-Glass) used in the comparison has a density of 2.58 g cc⁻¹, refractive index of 1.558, softening point of 771 °C, and annealing point of 657 °C.



Figure 1 Rattan pole wastes

2.2 Preparation of Filler

The rattan pole wastes were first cleaned and then cut into small sizes using knife. The rattan pole wastes were then dried in the oven at 100 °C for 15 minutes to eliminate impurity and excess moisture. Furthermore, milling and shaking were performed on the rattan pole wastes, followed by sieving to an average particle size within 75 (sieve 200 mesh). Nanoparticle production using hammer mill with the

rate of 25000 rpm was carried out to obtain particle sizes < 100 nm under three time variations of 15, 30, and 45 minutes. Hammer milling is a technical treatment to minimize particle size with the combined actions of collision, shaking, and milling.

2.3 Preparation of Nanocomposites

For the composites, polypropylene as matrix was reinforced by rattan nanoparticles, which act as the filler, in the company of the coupling agent maleic anhydride (MA). The composition of composite was 5% filler, 3% MA, and 92 % matrix. For comparison purpose, synthetic composite with the composition of 10% glass fiber, 1% MA, and 89% PP was fabricated against the currently studied composites. The designation for materials studied as well as their filler composition are listed in Table 1. All samples were fabricated with the twin extrusion moulding machine at 190 °C, with a mixing speed of 45 rpm for 3 hours.

Table 1 Composition of fillers in composites

Sample Designation	Filler
PP/R5	Rattan 5%
PP/FG10	Glass fiber 10%
PP	-

2.4 Particle Size Analyzer (PSA)

Examination of particle size of rattan powder was done using the Particle Size Analyzer (PSA), VASCO. VASCO is a dynamic light scattering measurement-based and fully agile nanoparticle size analyzer. The rattan powder was put into the sample holder. This examination was performed for three sample types produced by 15, 30, and 45 minutes milling processes.

2.5 Morphological Study

Examinations of the morphology and the composition of rattan fibers were carried out by means of Scanning Electron Microscopy (SEM) using JEOL JED model 2300 at an accelerating voltage of 20 kV. SEM micrographes were taken at 1000 magnification. Sputter coated with gold was used on the fracture surfaces to avoid electrostatic charging and low image quality during examinations.

2.6 Thermal Properties Inspection

Thermogravimetric analysis (TGA) was done using the Shimadzu DTG-60 TGA analyzer. All the sample weights used in this study were in the range of ± 5 mg. The samples were assessed with a thermal scan in the presence of heat from a temperature of 20 °C to 402 °C at a heating rate of 10 °C min⁻¹ under air atmosphere. In the process, weight loss versus temperature change was monitored.

3.0 RESULTS AND DISCUSSION

3.1 Sizing

Figures 2a, 2b, and 2c show the PSA outcomes of rattan powder milled under 15, 30, and 45 minutes time spans, respectively. As shown in Figure 2a, the distribution of particle is found to be concentrated within the region of 56-467 nm. This is attributed to a short exposure of milling period, which is about 15 minutes in the total time span. When the milling period was increased to 30 minutes, the distribution became more condensed with reduced particle sizes (about 15-48 nm). When milling period increases, more force is provided. As a result, greater energy is transferred into the particles. The rise in the transferred energy induces more cavitation so that the sizes are reduced and the particles created fall in the range of smaller than 100 nm. It is well known that each material has a maximum elasticity under certain deformation. Hammer mill with various given time variations cause different deformation of particles, which will in principle exceed the maximum elasticity after certain time spans threshold, so that the particles can be ruptured. Therefore, smaller sizes can be obtained after a longer milling time.

Interestingly, the particle sizes distribution is observed to increase corresponding to 45 minutes milling time not only in their spreading but also in terms of sizes (see Figure 2c). This finding is unexpected and suggests that the distribution of particle is a function of milling time up to a certain threshold period for the production of an optimal set of nanoparticle sizes. This observation occurred because an increase in milling time caused a rise in temperature in the holder. Consequently, atoms of the sample initiated specific self-merging owing to the combined actions of vibration, lattice void creation, and disorder induced by the temperature enhancement (recrystallisation). The results imply that after an optimal milling time, the particle sizes increase accordingly. Several studies had shown that there exists a strong correlation between the milling time and the resulted particle size [28-29]. The change observed in the present study is in agreement with [28] and [29] findings, which showed that the variation in milling time caused the spatial fluctuation in powder particle size. Note that the current study aims to demonstrate such trend although further study is required to identify a better milling time. This task may be considered as the subject of future work. Nevertheless, 30 minutes milling time can be considered as optimal for the time variations explored in the current study.

3.2 Morphological Discussion

Figures 3a, 3b, and 3c show the morphology of the rattan powder under milling time variations. As mentioned in the discussion of outcomes from the PSA analysis, SEM-based morphology of rattan

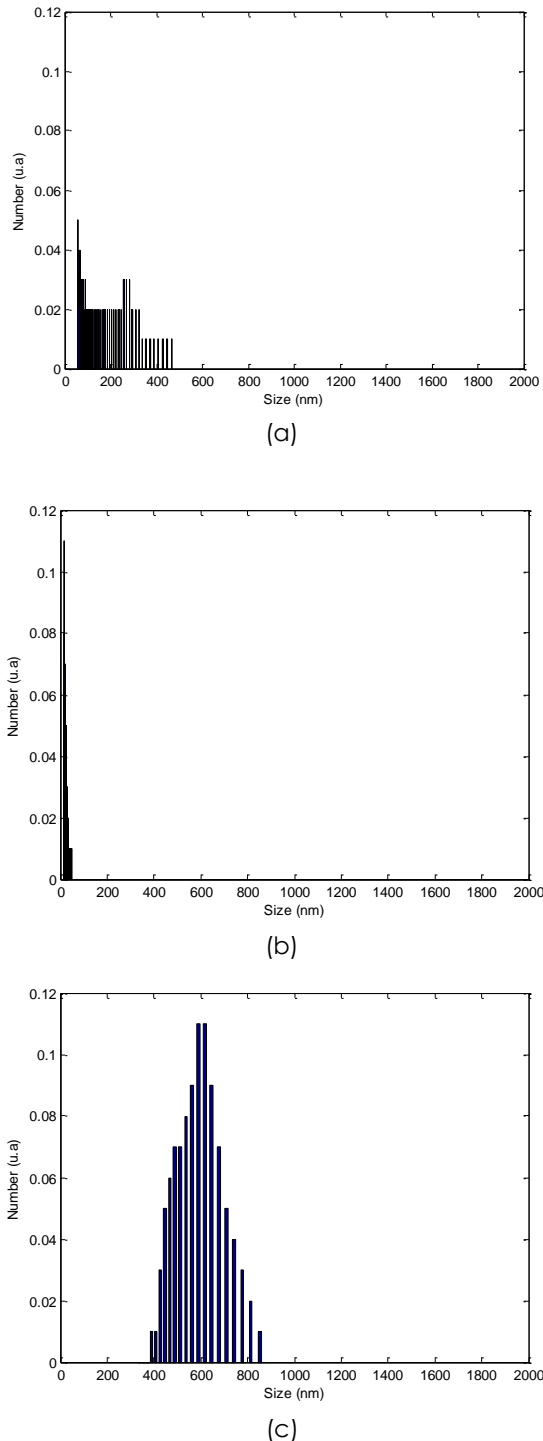


Figure 2 The distribution of rattan particles after (a) 15 minutes milling time, (b) 30 minutes milling time, and (c) 45 minutes milling time

powder shows the particle size distribution of the samples decrease with milling time. The micrographes show obviously that the particles decreased in size to an average of 75 when 30 minutes were elapsed (see Figure 3b). These results agree consistently with the particle analyzer examination. There exist greater irregularities in size,

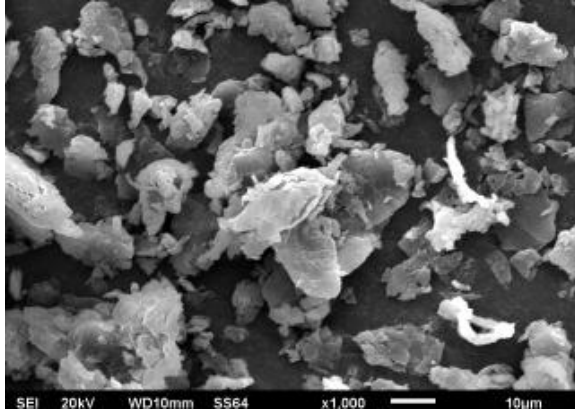
shape, and length for particles under 15 minutes milling time as shown in Figure 3a compared to others. Poor bonding has been one of the principal issues demoting the widespread use of natural fibers as reinforcement in composites. Therefore, it is essential that the distribution of particles including their shapes and sizes can offer more surface area for accommodating favorable adhesion with the matrix used. Particles in Figure 3c exhibit majorly larger size than those shown in Figure 3a, confirmed by Figure 2c where the greatest distribution in terms of number is concentrated around 600 nm, about twice as high against the maximum number in Figure 2a. These results seem to be consistent with other works, which found that increasing the milling time caused the average particle size to decrease significantly [28–31]. The smallest particle size was not obtained from the longest milling time. Homogeneity of the nanomaterial production is an important and desired result to produce optimal thermo-mechanical properties and characteristics. A few studies had investigated the effect of particle size in several composites [2,12,31–33]. Saha *et al.* [32] suggested that nanoparticles, i.e., sizes smaller than those considered in most existing studies generally possess more favorable mechanical and thermal properties.

3.3 Thermal Behavior

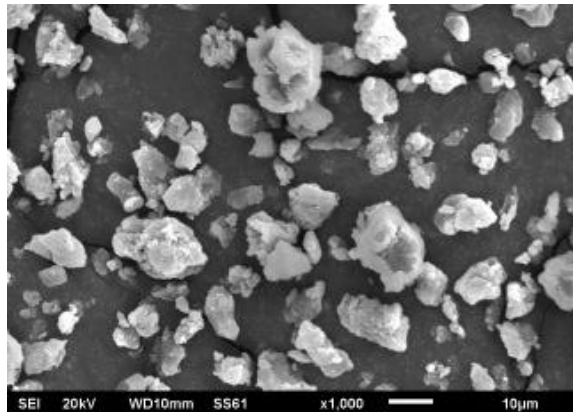
Figures 4 and 5 show respectively the sample weight-temperature and DTA-temperature relationships obtained from the thermogravimetric analyses of PP composites with various fillers. All sample weights remained consistently constant for certain ranges of temperature, after which weights were lost considerably. The slopes of the curves after weight loss are different for PP/R5, PP/FG10, and PP. As noticed in Figure 4, a one-step degradation process is observed for all samples. The degradation process of PP/R5 and PP/FG10 initiated at 290 °C and 279 °C, respectively whereas the weight degradation of PP occurred at the lowest temperature at 262 °C, indicating that the thermal stability superiority can be arranged as follows PP/R5 > PP/FG10 > PP. This is because it takes a higher temperature to degrade PP/R5 compared to others.

The remaining residues at the end of the degradation test for different samples are listed in Table 2. Table 2 indicates that PP/R5 has the highest amount of residue compared to PP/FG10 and PP. This result is supported by the curves in Figure 4 where PP/R5 had a much faster, greater degradation rate as well as took a greater temperature for the degrading initiation compared to PP/FG10 and PP. These results imply that PP/R5 composites exhibit a better thermal properties or more thermally stable compared to PP/FG and PP. These results find similarity with the outcome reported by [16] where the residue of their sample increased with the addition of rattan as filler in the hybrid composite compared to only the neat PP. Recently, natural fillers, especially rattan powder [15–18, 33], have

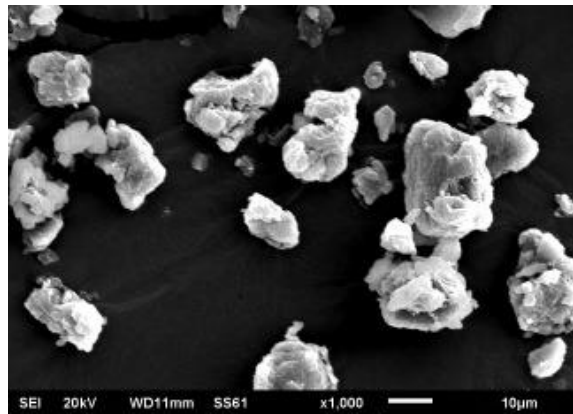
become one of the most popular additives in composites. It should be noted nonetheless that the above mentioned studies used filler in the micrometer range. This is in contrast with the nanoparticle filler used in the present study, which explores in addition an optimal milling time to achieve the desired size range.



(a)



(b)



(c)

Figure 3 The morphology of rattan particles after (a) 15 minutes milling time, (a) 30 minutes milling time, (a) and 45 minutes milling time

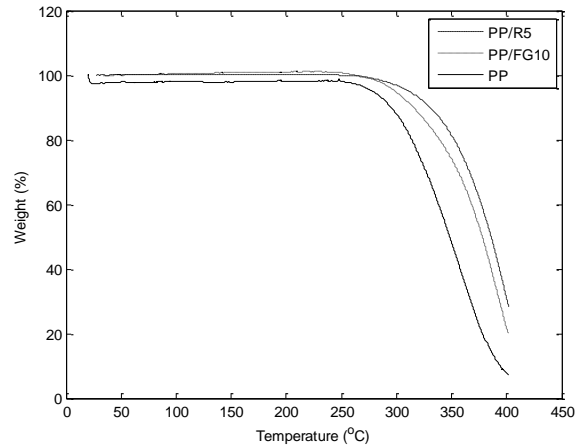


Figure 4 Weight-temperature curves of polypropylene reinforced with various fillers and that without

Table 2 Percentage of residue weight of samples

Sample Designation	T_{max} (°C)	Residue (%)
PP/R5	401.8	28.1
PP/FG10	401.8	20.1
PP	401.8	7.3

Figure 5 shows DTA curves of composites with varying fillers and that without. The obvious drops for all three curves around the same temperature illustrate the decomposition of PP, the substance shared by all three samples. The amount of drop is the least for PP/FG10 since it contains lesser PP that other samples.

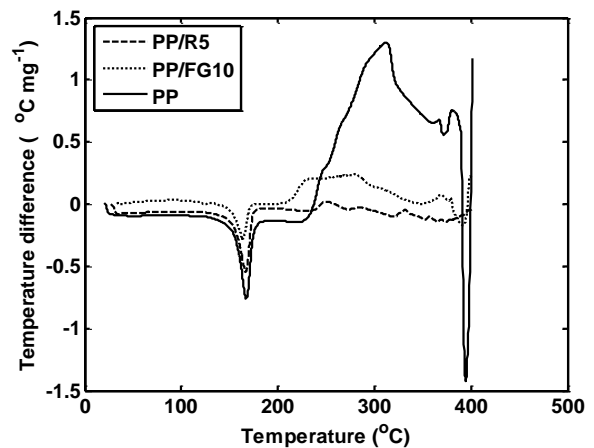


Figure 5 DTA curves of polypropylene reinforced with various fillers and that without

The first melting point of PP/R5 occurred at 166.34 °C (endotherm peak), at which the sample was still in a gelling condition. The end melting point of PP/R5 occurred at 177.59 °C. When an organic or inorganic

material is heated, the kinetic energy of molecules is increased as a result of their vibration, rotation, and interatomic collision. PP/R5 has five exotherm peaks at 250 °C, 282 °C, 332 °C, 356 °C, and 398 °C. The first to fourth exotherm peaks indicate the evaporation and carbonization processes followed by the disintegration of carbon bonds. The fifth exotherm peak corresponds to the termination of disintegration of carbon bonds by means of decomposition of mass. PP/FG10 has an endotherm peak at 163 °C. It has four exothermic peaks at 229 °C, 281 °C, 369 °C, and 398 °C. On the contrary, the neat PP has an endotherm peak at 168 °C. At this temperature, the sample is soft due to the existence of crystal and amorphous. PP has four exotherm peaks at 312 °C, 367 °C, 381 °C, and 401 °C.

It is apparent from the plots that PP/R5 has a much higher melting point than PP/FG10, exhibiting composite reinforced with natural filler (rattan) has an increased thermal properties. Principally, composites are easy to be reinforced with glass fiber according to application purposes. However, glass fiber is not naturally easy to degrade as opposed to natural filler used in this study, which is more suitable and friendly to surrounding environment in terms of applications in the long run. It is also worthwhile to note that several recent studies have been aimed towards the sustainable and advanced materials as well as structural elements [34-40].

4.0 CONCLUSION

This paper examines the effects of rattan filler nanoparticle loadings on the thermo-physical properties of rattan powder-filled polypropylene composites. It was found that the best distribution of particle to reach the definition of nanoparticle was obtained in the 30 minutes milling time process. This observation was further confirmed with the morphological study obtained from the SEM analysis. Also, the thermogravimetric analyses have demonstrated that, in comparison to PP/FG and neat PP, PP/R5 leaves the highest amount of residue after an exposure to high elevated temperature, indicating that it is more thermally stable.

This research provides a framework for the exploration of natural filler to replace glass fiber filler, especially to achieve an environmentally friendly material for numerous applications. The current study has therefore managed to show the advantages of using the natural filler as a solution to improve thermo-physical properties of composites especially in terms of those polymer-based. It would be interesting to further assess the effects of the inclusion of rattan powder filler on the mechanical properties of composites including their performance in some practical components used in various industries. In addition, a greater range of variation in milling time and related effects could be considered in the future for wider exploration of optimization.

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