# Processing, Mechanical, and Thermal Properties of Polypropylene/Rattan Powder/Talc Hybrid Composites

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Talc was incorporated as filler alongside rattan powder in polypropylene (PP) matrix to produce a hybrid composite. 20 phr of rattan powder was used for all samples. Talc filler loadings were varied from 0 to 10 phr. The composite was manufactured using a Polydrive Thermo Haake internal mixer, which provided processing characteristics for the composite. Peak and stabilization torques gradually increased with increasing talc filler loading. Tensile properties of the hybrid composites showed an increase in tensile modulus and a decline in elongation at break with increasing talc loading. The tensile strength was enhanced with the addition of talc up to a loading of 4 phr and then decreased with further talc filler loading. A water absorption test was carried out, whereby the water uptake of the hybrid composites was reduced with higher talc filler loading. Thermal degradation of the composites was analyzed by studying thermogravimetric analysis (TGA) thermograms and derivative thermogravimetry (DTG) curves, which showed improvement in the thermal stability of the composite with higher talc content. The morphological studies carried out on tensile-fractured surfaces of the hybrid composites explained the enhancement and deterioration of tensile properties with regard to different filler loadings.

Keywords: Rattan filler; Talc; Polypropylene; Hybrid composite; Processing torque; Mechanical properties; Water absorption; SEM; TGA

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## INTRODUCTION

The plastics industry is by far the largest of the main polymer-based industries, with a high growth rate in consumption. Plastic composites first gained recognition during the 1930s with the discovery and production of fiberglass, as well as the patent for unsaturated polyester resin. The fibre-reinforced plastics industry started to develop during World War II in electronics and aircraft structures (Kulshreshtha 2002). These developments sparked a strong interest in producing strong lightweight materials; thus, plastic composites were recognized as an ideal material in many structural and household items. Today, the world consumption value of plastic composites has surpassed the consumption of metals in terms of volume (Biron 2013).

Industrial crop and wood wastes are abundantly present in developing countries. In Malaysia, the large plantation areas of such crops as oil palm and coir can generate large quantities of vegetable waste (Khalil *et al.* 2006). Forest-based materials that contribute to the building and furniture industries also produce large amounts of waste. Consequently, the practice of waste management is needed to prevent the accumulation of these wastes. Many have taken the initiative to transform these wastes into products that can be marketed. The well-known wood-waste market has seen much activity in the panel board and biomass industries. Agro-wastes have been constantly researched and are presently in competition with synthetic fibres and reinforcements (Tajvidi and Ebrahimi 2003). A suitable addition to these waste markets is the group of widely used materials of non-wood origin that are preferably naturally abundant. One example is bamboo, which is commonly found across East Asia. In Malaysia, a suitable option would be rattan.

Amongst the common mineral fillers, talc deposits, which are processable, can be found worldwide, although they are not as widespread as calcium carbonate or clay. Talc is largely produced as an impure product. The chemical composition of talc is  $Mg_3(Si_4O_{10})(OH)_2$ . Pure talc is the softest known mineral, with a hardness of 1 on the Mohs hardness scale, thus causing less abrasion and easier dispersion than other minerals. The early uses of talc involved the cosmetics industry in the 1960s, and it went on to be used in paint as an extender and filler pigment. Talc is also utilized in paper production for coating and filling due to its fine particle size and white colour. Talcs have been used in ceramics due to their uniform chemical composition and constant amount of shrinkage over a wide temperature range (Hancock and Rothon 2003; Lefond 1983). Domestic appliances also benefit from talc-filled composites, where examples of products are washing machine soap dispensers and disposable food packaging (DeArmitt and Hancock 2003). The applications chosen for talc-filled materials are often those that do not require toughness or high strain because of the rigidity of the composite, which may lead to brittleness.

In polymer composites, natural fibres from forest products as well as agricultural crops are popular in terms of transforming the composite into an environmentally friendly material (Khalil *et al.* 2001; Nawang *et al.* 2001; Rozman *et al.* 2001; Salmah and Ismail 2008). Although they may be inconsistent in performance due to their dependence on cultivation conditions as opposed to synthetic fillers, many processing and treatment methods have been researched to enhance their properties. Therefore, the production of biocomposites with acceptable performance is becoming more feasible.

Minerals are also used in polymer composites as fillers to reduce the cost of composites or to improve the performance of the polymer. For example, they may increase the stiffness, tensile strength, hardness, and temperature performance, or they may modify optical properties such as colour. Mineral fillers that are based on silicates and carbonates are widely used because of their abundance and intrinsic properties (Hancock and Rothon 2003). Examples of such fillers include calcium carbonate and mica. Other salt-like compounds, metals, or compounds based on an extended structure of covalently bound atoms are also being used (Caseri 2007). Both natural fibres and mineral fillers have gained their own reputations in producing polymer composites for different reasons; thus, the interest in the potential of their synergistic values in composites is high.

Previous research has studied the hybridisation of natural and synthetic fillers in plastic composites, such as the use of glass fibres, silica, and clay with various natural fibres such as kenaf, rice husk, and wood fibre (Rozman *et al.* 2000; Premalal *et al.* 2002; Ismail *et al.* 2003a; Ismail *et al.* 2003b; Gwon *et al.* 2012, Ismail *et al.* 2013). Rattan powder is a viable filler with little prior research; in this study, rattan powder was hybridised with talc to investigate its performance as a hybrid polypropylene (PP) composite.

## EXPERIMENTAL

#### Materials

Polypropylene (TITANPRO homopolymer grade 6331) was supplied by Titan Petchem (M) Sdn. Bhd. (Johor, Malaysia), having a melt flow index of 14 g/10 min at 230 °C and a density of 0.9 g/cm<sup>3</sup>. Rattan (*Calamus manan*) pole wastes were collected from Seng Huat Sdn. Bhd. (Nibong Tebal, Penang, Malaysia). Talc was obtained from Ipoh Ceramic Sdn. Bhd. (Ipoh, Malaysia).

## **Composite Preparation**

Rattan pole wastes were ground and sieved to an average particle size below 180  $\mu$ m. Both rattan powder and talc filler were dried in a vacuum oven at 80 °C for 24 h to remove excess moisture. As shown in Table 1, different loads of talc filler were used in this study. The melt mixing of the hybrid composites was carried out at 180 °C with a rotor speed of 50 rpm using a Haake Rheomix Polydrive R 600/610 internal mixer. Polypropylene was loaded into the mixing chamber and allowed to melt for 2.5 min, after which rattan powder was added within a period of 30 s. Talc filler was added when a fairly homogenized mixture was obtained after 4.5 min. Mixing was continued up to a total mixing period of 7 min. The collected compounded material was then moulded into a 1-mm-thick sheet using a Go-Tech compression moulding machine type KT-7014-A. The upper and lower moulds were set to 180 °C. Composites were preheated for 7 min and compressed under a hydraulic pressure of 1000 psi for 3 min, followed by cooling at 1000 psi for 2 min. The samples were cut into shape according to the requirements for the tests conducted in this study.

Sample	Designation	PP	Rattan Powder	Talc Filler (phr)
		(phr)	(phr)	
1	PP/R20	100	20	0
2	PP/R20/T2	100	20	2
3	PP/R20/T4	100	20	4
4	PP/R20/T6	100	20	6
5	PP/R20/T10	100	20	10

 Table 1. Composition of Polypropylene/Rattan Powder/Talc Hybrid Composites

## **Tensile Test**

Tensile properties of the composites were measured in accordance with the American Society for Testing Materials (ASTM) D 638 standard. The test was performed using a Universal Testing Machine (UTM) Instron Model 3366 with a cross-head speed of 50 mm/min at  $25 \pm 3$  °C. Dumbbell-shaped specimens were prepared by cutting samples using a Wallace die cutter from the compression-moulded sheets. Tensile strength, tensile modulus, and elongation at break were obtained from the average results of five specimens.

# **Morphological Study**

Fracture surfaces of tensile test specimens were examined using a scanning electron microscope (SEM) (Zeiss Supra 35vp, Germany). The fracture surfaces were sputter-coated with gold prior to SEM observations to avoid electrostatic charging during examinations. SEM micrographs were taken at various magnifications.

## Water Absorption

Specimens were dried to a constant weight in a vacuum oven for 24 h at 80 °C. Samples were weighed to the nearest 0.001 g prior to immersion in distilled water. Weighing was repeated periodically using a Radwag balance model AS 310/X with a precision of 0.1 mg. The percentage of moisture content at time *t*,  $W_t$  (%) was calculated using Eq. 1 as follows,

$$W_t(\%) = 100 \times (w_w - w_i)/w_i$$
(1)

where  $w_i$  and  $w_w$  are the initial dry weight and wet weight after immersion at time *t*, respectively.

#### Thermogravimetric Analysis

Thermogravimetry analysis (TGA) was carried out using a Perkin Elmer Pyris 6 TGA analyzer. Dynamic TGA experiments were conducted to assess thermal degradation using a heating rate of 20 °C /min from 30 °C to 600 °C. Tests were done under a nitrogen flow of 50 mL/min, and the samples used had masses of 5 to 10 mg.

## **RESULTS AND DISCUSSION**

#### Processing Torque

The mixing torque development for PP/rattan powder/talc hybrid composites in Fig. 1 shows torque *versus* time curves for various talc filler loadings. From the initial observation, the processing curves of each talc filler loading showed minor differences, except for the composite without talc.



**Fig. 1.** Mixing torque *versus* time data for PP/rattan powder/talc hybrid composites with increasing talc filler loading during processing

The initial 2 min of mixing involved the shearing of solid PP granules with the application of heat, contributing to a high torque value. This continuous shearing action caused PP to melt and provided less resistance towards the shearing force of the rotor. This led to a lower torque value, which signified that the molten PP was stabilised enough for filler addition. Blending of rattan powder took place at 2.5 to 4.5 min, with a small peak at 3 min because of the dispersion of rattan powder in the mixture. Another peak at a slightly lower torque value was observed at 5 min into mixing, corresponding to the addition of talc filler into the mixture. The dispersion of talc required less force compared with rattan powder because of the fibrous form and hydrophilic nature of rattan powder, leading to a higher tendency for rattan powder particles to adhere to each other during compounding. The torque values decreased steadily as both rattan and talc filler gradually dispersed into the mixture and progressed to a stabilised state. On the whole, the mixing process took place over a period of 7 min.

A peak torque at 5 min, determined from the processing curves of PP/rattan powder/talc hybrid composites, is displayed in Fig. 2. An increase in torque value between 0 phr and 10 phr of talc content indicates that the addition of talc filler induced the dispersive force needed to allow wetting of the filler by the molten PP. Peak torque values of composites varying from 2 phr to 6 phr, however, showed negligible differences. The structure of talc undergoes cleavage readily, which makes talc relatively easy to disperse, as talc particles are weakly attached to each other through secondary forces (Hancock and Rothon 2003). The small filler loading range of talc fillers may also contribute to the minuscule differences in their processing properties.

Figure 2 also displays the stabilisation torque of PP/rattan powder/talc hybrid composites, where the mixture at this point is homogenised. A similar pattern as the peak torque was observed for the stabilisation torque, whereby the torque values increased with increasing talc loading, although the increment was more pronounced in the latter. The flow of the PP matrix was restricted by the addition of rattan powder, and the presence of additional filler easily added to the viscosity of the mixture.



**Fig. 2.** Peak and stabilization torque for PP/rattan powder/talc hybrid composites with increasing talc filler loading

Although the dispersive ability of talc particles may provide less resistance towards the shearing action, higher content as well as the platelet structure of talc particles could lead to an increase in the contact surface between the talc particles and the PP matrix. This in turn would increase the resistance towards the flow of the mixture.

#### **Tensile Properties**

The tensile strength of PP/rattan powder/talc composites under the influence of different talc filler loadings is displayed in Fig. 3. A steady increase was observed within the 0-phr to 4-phr range of talc content, while the hybrid composite with a talc loading of 6 phr showed a sudden decrease in tensile strength. Further addition of talc filler gradually reduced the tensile strength of the composite. Therefore, 4 phr of talc filler loading provided optimum reinforcement ability to the composite. Rattan powder, due to its larger size and poor adhesion to the PP matrix, was more likely to be discontinuous and did not support stress transferred from the matrix. With the addition of talc filler up to 4 phr, however, stress was efficiently distributed across the filler-matrix interphase; thus, the tensile strength of the composite was enhanced. This reinforcement by talc fillers can be attributed to its structure, where a brucite (Mg(OH)<sub>2</sub>) sheet is sandwiched between two siloxane (Si<sub>2</sub>O<sub>5</sub>) sheets. Therefore, the surface of the talc structure comprises oxygen atoms, lacking the polarity of hydrophilic fillers. Due to this surface nature, talc was more likely to have a better interfacial interaction with the PP matrix compared with rattan powder. This effect was further enhanced by the platelet structure of talc, whereby the high aspect ratio would allow more surface area to be in contact with the PP. Also, the hydrophobic nature of talc platelets would result in weak attachments between adjacent talc platelets by secondary forces; therefore, separation of the attached platelets could be accomplished by mechanical force (Hancock and Rothon 2003). These characteristics of talc filler allowed for good dispersion in the composite; the distribution of stress can prevent a catastrophic failure by reducing the amount of stress towards rattan fillers.



**Fig. 3.** Effects of different talc filler loadings on the tensile strength of PP/rattan powder/talc composites. Error bars represent the standard deviation of the measurements.

Addition of talc filler at 6 phr and above abruptly weakened the tensile strength of PP/rattan powder/talc composites. Higher talc filler loadings caused smaller interparticle distance between fillers during and after mixing, leading to a higher tendency for stronger filler–filler interaction as compared with interaction between filler and matrix. This caused attachments of talc platelets to form aggregates. It is possible that the mechanical shearing during mixing was unable to separate the aggregates, which eventually resulted in many stress-concentrated sites in the composite.

Figure 4 shows the tensile modulus of the PP/rattan powder/talc composites with different talc filler loadings. The modulus of the composite increased with larger additions of talc fillers, although this effect is only discernible in PP/R20/T6 and PP/R20/T10 composites. Additions of talc fillers up to 4 phr showed minor increases in the tensile modulus of the composite. According to Bakar et al. (2007), the increase of tensile modulus in composites is due to the replacement of PP by filler with higher rigidity and restriction of the mobility and deformability of the matrix by the introduction of a mechanical restraint by the filler. In this case, higher amounts of talc filler were able to impose larger limits on the chain mobility of the PP matrix. The fairly hydrophobic surface nature of talc, as previously mentioned, allowed proper wetting by the PP matrix; thus, with good adhesion of the filler onto the matrix, the rigidity of talc filler was able to be imparted on the composite. Another aspect that should be taken into consideration is the orientation of talc fillers in the composite. Fillers with high orientation increase the resistance towards plastic deformation of the matrix, and the stiffness of the composite can thereby be increased (Leong et al. 2004; Bakar et al. 2007). In this study, however, talc filler does not seem to be highly oriented (as displayed in Fig. 7a and Fig. 7b in the Morphological Study section). This may explain the low increment in the tensile modulus of PP/R20/T2 and PP/R20/T4, despite their higher values of tensile strength.



**Fig. 4.** Effects of different talc filler loadings on the tensile modulus of PP/rattan powder/talc composites. Error bars represent the standard deviation of the measurements.

The elongation at break of PP/rattan powder/talc composites (Fig. 5) was seen to decrease with increasing filler loading. As reflected by the tensile modulus, the amount of matrix deformation was restricted with a higher content of talc filler, leading to a brittle failure. The addition of talc fillers produced a mechanical restraint on the matrix surrounding the platelets; thus, the ductility of the composite as a whole was affected. The previously mentioned filler–filler interaction, which possibly occurred with higher amounts of talc addition, created aggregates. Along with the rattan filler in the composite, these aggregates transformed into multiple stress-concentrated sites. Therefore, the crack propagation across the composite could occur on a large scale, prohibiting the composite from dissipating stress in the form of matrix deformation.



**Fig. 5.** Effects of different talc filler loadings on elongation at break of PP/rattan powder/talc composites. Error bars represent the standard deviation of the measurements.

## **Morphological Study**

Figure 6a displays a SEM micrograph of the tensile fracture surface of rattan powder-filled PP composites at 20 phr of rattan filler loading, designated as PP/R20. Figure 6b and 6c show tensile fracture surfaces of composites with 4 phr and 10 phr talc contents, respectively, designated as PP/R20/T4 and PP/R20/T10. 4 phr of talc filler loading gave optimum results in tensile strength, while a filler loading of 10 phr showed the lowest tensile strength and elongation at tensile failure values and the highest tensile modulus among the composites in this study.

The micrograph in Fig. 6a displays a composite with some presence of rattan powder and several occurrences of voids due to pulling-out of the filler. It has been conjectured that pull-outs occur as a result of the poor adhesion of rattan powder on PP. In Fig. 6b and 6c, voids are noticeable, although rattan powders are not apparent on the fracture surface and seem to be embedded in the PP matrix despite their poor interfacial adhesion. The rougher surfaces of PP/R20/T4 and PP/R20/T10 compared with PP/R20 indicated that during fracture, crack propagation occurred at the interfaces between the dispersed talc filler and the PP. Therefore, the pull-outs corresponded to rattan powders that were located along the path of fracture, while the seemingly embedded rattans were

not disturbed by the crack propagation. This implies that with the addition of talc filler, the strength of the composite was less influenced by the presence of rattan filler. In Fig. 6c, some matrix tearing occurred at locations that may possibly be sites of stress-concentrated areas, and energy-dissipating deformations such as matrix yielding were unnoticeable. This further concludes that the nature of the fracture that occurred in PP/R20/T10 was brittle, as determined by the low tensile strength and elongation at strain failure values.





The SEM micrographs of the tensile fracture surface of PP/R20/T4 and PP/R20/T10 at a magnification of  $1000 \times$  are displayed in Figs. 7a and 7b, respectively. The talc fillers were well-distributed around the rattan powder in the PP/R20/T4 composite, which explained the enhancement of tensile strength by talc addition. Some talc fillers were exposed on the surface, though most seemed to be embedded in the matrix. Figure 7b shows a surface with denser amounts of talc that were fairly well distributed, except for the presence of an aggregate that occurred because of high filler–filler interaction. Figure 7c shows a magnified view of the talc aggregate in the

PP/R20/T10 composite, which was formed by many layers of talc and was loosely held by the matrix. Another factor that affects the tensile properties of the composite is the orientation of fillers. In a few studies, the nature of the talc filler allowed anisotropic filler orientation in the composite, as described in the Tensile Properties section. In Fig. 7a, however, the talc fillers did not seem to be highly oriented in a single direction. The circled fillers show some talc platelets that were parallel, and some that were perpendicular, to the fracture surface. As explained in the Tensile Properties section, the arrangement of these fillers was unable to provide an anisotropic enhancement of the tensile properties of the composite. Figure 7b shows a similar condition for the talc filler orientation.





#### Water Absorption

The water absorption of PP/rattan powder/talc hybrid composites with varying talc filler loadings is displayed in Fig. 8, where water is absorbed to a point of saturation. According to the results, the highest percentage of water uptake was shown by the rattan

powder-filled PP composite having no talc content. The addition of talc reduced the amount of water uptake to a large degree, and the water absorption percentage continued to decrease slightly with increasing amounts of talc filler. It is known that natural fibres have a strong affinity towards moisture due to the presence of hydroxyl groups in their lignocellulosic structure. Also, the gaps that exist between interfacial areas of rattan powder and PP matrix may store moisture. Therefore, the hygroscopic nature of rattan powder and its poor interfacial adhesion towards the PP matrix contributed to the percentage of water absorption in the composites.



Fig. 8. Increase in weight after water absorption as a function of time of immersion for different talc filler loadings

Although a similar amount of rattan powder was used in all composite samples in this study, the reduced amount of water uptake in talc-filled hybrid composites suggests that the talc fillers prevented most of the absorption of moisture by the rattan powder. The moisture barrier-like property of talc fillers can be attributed to two factors: (i) the surface nature of talc fillers comprises oxygen atoms and is fairly hydrophobic, thereby preventing any permeation of water molecules into the talc fillers; and (ii) the talc fillers in the matrix imposed a tortuous path for the water molecules, which are forced to travel around the talc particles. The platelet structure and high aspect ratio of the talc fillers would add to the path length of the diffusion of the water molecules (Hancock and Rothon 2003; DeArmitt and Hancock 2003).

Given that the talc fillers were well-distributed throughout the composite for all samples, the degree of tortuosity would increase with increasing talc filler content. Also, good wettability of the PP matrix onto the talc fillers would mean that contact between the filler and polymer would not lead to the formation of air bubbles or microvoids; therefore, increasing the amount of these fillers would create a larger barrier for the water molecules.

#### **Thermogravimetric Analysis**

TGA thermograms of PP/rattan powder/talc composites with varying kaolin filler loadings are shown in Fig. 9, while Table 2 displays the detailed variation of TGA experimental data of PP/rattan powder/talc composites. The lowest and highest talc filler content, designated PP/R20/T2 and PP/R20/T4, respectively, were chosen for this study. Referring to Fig. 9, a two-step degradation process was observed for all tested samples. The first degradation step corresponds to the decomposition of rattan filler, while the second stage can be referred to as the decomposition of PP. It is determined from the initial observation of Fig. 9 that the degradation process of all samples occurred at a relatively similar temperature up to 350 °C; beyond that, further heating of samples caused a shift in the degradation temperatures for the talc-filled hybrid composites.

Figure 10 shows the derivative thermogravimetric (DTG) curves of the hybrid composites. Small degradation peaks are observed between 250 °C and 400 °C as a result of the decomposition of different constituents of rattan powder. Rattan, being a lignocellulosic material, contains cellulose and lignin constituents which have different resistance towards high temperatures. The decompositions of hemicellulose and cellulose constituents were illustrated by the two peaks mentioned, while lignin underwent a gradual decomposition taking place within the range 200 °C to 500 °C. Therefore, the residue of PP/R20 may also result from the slow degradation process of lignin content in the rattan powder (Ali *et al.* 1995; Abdullah *et al.* 2010).

Referring to Fig. 9, the hybrid composites with talc filler additions of 2 phr and 10 phr showed a smaller weight loss percentage compared with PP/R20, from 350 °C to the end of the degradation test. The shift in degradation temperature was reflected by the temperature at the maximum rate of weight loss ( $T_{max}$ ) (Table 2), which improved with increasing talc filler content. This signifies that the incorporation of talc increased the thermal stability of PP/rattan powder composite, more pronouncedly after 350 °C. Research by Zhou *et al.* (2005) found that the addition of talc in PP composites increased the decomposition temperature of the samples.



Fig. 9. TGA curves of PP/rattan powder/talc composites with varying talc filler loadings



Fig. 10. DTG curves of PP/rattan powder/talc composites with varying talc filler loadings

A rising amount of residue at the end of the degradation test with increasing talc content is shown in Table 2. Talc is known to be thermally stable up to 900 °C (Duquesne *et al.* 2008); therefore, the contents of the residue in PP/R20/T2 and PP/R20/T4 composites may constitute a char layer as a result of the decomposition of cellulose and lignin from rattan powder, as well as the talc filler content of the composites.

Sample Designation	T <sub>max</sub> (°C)	Residue (%)
PP/R20	476	3.1
PP/R20/T2	478	4.754
PP/R20/T10	481	9.571

## CONCLUSIONS

- 1. Values of mixing torque as well as peak torque show minor increases with higher amounts of talc filler loading due to easy dispersion of talc platelets. Stabilisation torque values rise with increasing talc filler loadings, attributed to flow restriction of PP by the talc fillers.
- 2. The highest tensile strength value was obtained by the addition of talc filler loading at 4 phr in rattan powder-filled polypropylene composites. Filler loadings of 6 phr and above reduced the tensile strength due to the presence of agglomeration, as confirmed by SEM.

- 3. The tensile modulus increased due to the restriction of matrix mobility with increasing talc filler loading, while elongation at tensile strain decreased as a result of increasing stress concentration sites with higher talc content.
- 4. Water uptake was significantly reduced with the addition of talc and continued to decrease gradually as the talc loading increased due to the hydrophobicity of talc fillers and the tortuous path created by the dispersion of talc in the matrix.
- 5. The addition of talc fillers increased the temperature of the maximum rate of weight loss  $(T_{\text{max}})$  as well as the percent residue, which indicated that the thermal stability of the hybrid composite was enhanced.

# ACKNOWLEDGMENTS

The authors would like to thank Universiti Sains Malaysia (USM) for supporting this study.

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Article submitted: June 10, 2013; Peer review completed: July 24, 2013; Revised version received and accepted: September 26, 2013; Published: October 25, 2013.