

# EFFECT OF ORGANOCLAY TYPES ON MECHANICAL PROPERTIES AND FLAMMABILITY OF POLYPROPYLENE/SISAL FIBER COMPOSITES

W. Chanpranon<sup>1,2</sup>, N. Suppakarn<sup>1,2</sup> and K. Jarukumjorn<sup>1,2\*</sup>

<sup>1</sup> School of Polymer Engineering, Institute of Engineering, Suranaree University of Technology, Nakhon Ratchasima, Thailand

<sup>2</sup> Center for Petroleum, Petrochemicals, and Advanced Materials, Chulalongkorn University, Bangkok, Thailand

\* Corresponding author ([kasama@sut.ac.th](mailto:kasama@sut.ac.th))

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## 1 General Introduction

Natural fiber reinforced polymer composites have received considerable attention, particularly in construction, automotive, and aerospace industries due to environmental and ecological concerns. The advantages of natural fibers are low density, acceptable specific strength, less tool wear, biodegradability, and renewability. Sisal fiber has been widely used as a reinforcing fiber in polymers due to high tensile strength and stiffness. In addition, the sisal fiber is locally planted in Thailand.

Polypropylene (PP) is a common thermoplastic used as a matrix in composites because of its low cost, low density, good mechanical properties, and ease of processing. However, natural fibers are not well compatible with PP leading to the composite with poor mechanical properties. Generally, maleic anhydride grafted polypropylene (MAPP) is used to improve the compatibility between the natural fibers and PP matrix [1,2]. Another critical drawback of PP/natural fiber composites is their high flammability. Flame retardants, e.g. ammonium polyphosphate (APP), magnesium hydroxide ( $Mg(OH)_2$ ), have been used to improve flame retardancy of the composites. However, adding the flame retardants at high content is required to obtain effective performance.

In recent years, incorporation of an organoclay as nanofiller has been considered for overcoming the drawbacks of the polymers. Adding organoclay at very low loading (less than 5 wt%) has been an interesting approach of improving mechanical properties, thermal stability, flame retardancy, and water resistance of the composites [3-6]. However, to achieve the good property enhancement,

organoclay particles should be homogeneously dispersed and exfoliated within the polymer matrix [3,4]. Several types of commercial organoclays are currently available. In general, the main difference among of them concerns type and amount of organic modifiers used in the modification which influenced the final properties of the PP/clay nanocomposites [7].

The objective of this work was to study the effect of organoclay type on mechanical, thermal, and flame retarding properties of PP/sisal fiber composites.

## 2 Material and Method

### 2.1 Materials

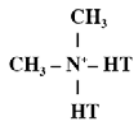
Polypropylene (PP, P700J) was supplied by SCG Chemicals Co., Ltd. Sisal fiber (SF) was purchased from the Sisal-Handicraft OTOP group, Nakhon Ratchasima, Thailand. The fiber was cut into an approximate length of 2 mm. Organoclays (organically modified montmorillonites, Cloisite<sup>®</sup> 20 A (20A) and Cloisite<sup>®</sup> 30B (30B)) were supplied from Southern Clay Products Inc., USA. The physical properties of organoclays used in this study are shown in Table 1. The chemical structures of organic modifiers of the modified montmorillonites are presented in Fig. 1. Maleic anhydride grafted polypropylene (MAPP, Fusabond<sup>®</sup> P MZ 109D, DuPont) was supplied from Chemical Innovation Co., Ltd.

Table 1. Physical properties of organoclays [8].

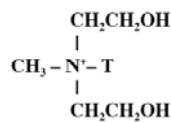
Organoclay	Organic modifier	Modifier concentration (meq/100g)
Cloisite®20A	2M2HT <sup>a</sup>	95
Cloisite®30B	MT2EtOH <sup>b</sup>	90

<sup>a</sup>2M2HT: dimethyl, dehydrogenated tallow, quaternary ammonium

<sup>b</sup>MT2EtOH: methyl, tallow, bis-2-hydroxyethyl, quaternary ammonium



(a)



(b)

Fig. 1. Chemical structures of the organic modifiers of organoclay: (a) Cloisite®20A where HT is hydrogenated tallow (~65% C18; ~30% C16; ~5% C14) and (b) Cloisite®30B where T is tallow (~65% C18; ~30% C16; ~5% C14) [8].

## 2.2 Preparation of composites

Prior the composite preparation, sisal fiber and organoclay were dried in an oven at 70°C over night. PP compounds were prepared by melt blending using an internal mixer (Haake Rheomix, 3000p). The total mixing time was 15 min. The mixing temperature was 170°C and the rotation speed was 60 rpm. Test specimens were molded by an injection molding machine (Chuan Lih Fa, CLF 80T). Composition of PP composites and their designation used are shown in Table 2.

Table 2. Formulations of PP composites.

Designation	PP (phr)	Sisal (phr)	20A (phr)	30B (phr)	MAPP (phr)
PP/SF	100	30	-	-	-
PP/SF/20A	100	30	3	-	5
PP/SF/30B	100	30	-	3	5

## 2.3 Characterizations of materials

### 2.3.1 Structure characterization

The structure of organoclay and PP composites were determined by an X-ray diffractometer (XRD; Oxford, D5005) equipped with a Cu K $\alpha$  radiation source of wavelength 1.5406 Å operated at 40 kV and 40 mA at room temperature. Scanning rate and step size were 2.0°/min and 0.02° with 2 $\theta$  varying from 2° to 10°. XRD patterns were used to calculate the basal spacing or d-spacing from Bragg's law.

### 2.3.2 Mechanical test

Tensile properties of PP composites were measured according to ASTM D638 using a universal testing machine (UTM; Instron 5569). Impact test was performed on unnotched PP composites following ASTM D256 using an impact testing machine (Atlas, BPI). In all cases, average values from five specimens at least were taken for each sample.

### 2.3.3 Flammability test

Flammability of PP composites was characterized by a horizontal burning test according to ASTM D635 using a horizontal vertical flame chamber instrument (Atlas, HVUL). The sample was held horizontally and a flame was applied to light one end of the sample. The burning rate of the sample was calculated as follows:

$$V = 60L/t \quad (1)$$

Where  $V$  is the burning rate in millimeters per minute;  $L$  is the length the flame travels from the first reference mark (25 mm from the end) to the second reference mark, which is at 100 mm from the end; and  $t$  is the time in seconds for the flame to travel. Five measurements per sample were taken.

### 2.3.4 Thermal analysis

Thermal behaviors of PP composites were carried out on a thermogravimetric analyzer (TGA; TA Instrument, SDT2960) at a constant heating rate of 20°C/min under air flow. The temperature ranged from the room temperature to 700°C. The weight of each sample was kept within 10-15 mg.

### 3 Results and Discussion

#### 3.1 Clay structure

XRD patterns of organoclays and PP composites are shown in Fig. 2. XRD patterns of 20A and 30B showed a peak at  $2\theta = 3.32$  with a d-spacing of 2.66 nm and at  $2\theta = 4.77$  with a d-spacing of 1.85 nm, respectively. PP/SF/20A composite presented a shifted peak to a lower angle at  $2\theta = 2.74^\circ$  which corresponds to d-spacing of 3.22 nm, indicating the formation of an intercalated structure. For PP/SF/30B composite, the peak was disappeared. This implied that the silicate layers could be exfoliated and dispersed in PP matrix [9]. The observed difference between 20A and 30B may be due to the nature of organic modifiers.

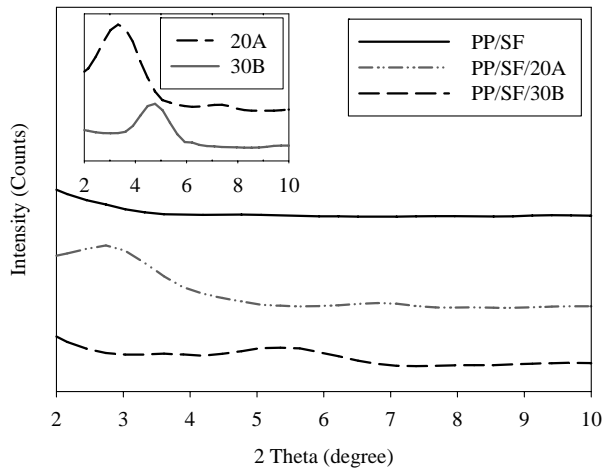


Fig. 2. XRD patterns of organoclays and PP composites.

#### 3.2 Mechanical properties

Tensile properties of PP composites are listed in Table 3. Tensile strength of PP/SF/30B composite was higher than that of PP/SF/20A composite. This was because the organic modifier of 30B had hydroxyethyl group and showed higher polarity than that of 20A. Therefore, 30B had larger interaction capacity with the polar group of MAPP leading to the delamination in individual platelets [10]. However, the addition of organoclay did not show substantial increase in Young's modulus when compared with PP/SF composite.

Impact strengths of PP composites are presented in Table 3. The addition of organoclay slightly

improved the impact strength of PP/SF composites. Lee and Kim [4] also reported that incorporating 1 phr of organoclay slightly increased impact strength of wood/PP composites. However, organoclay type insignificantly affected the impact strength of PP/SF composites.

Table 3. Mechanical properties of PP composites.

Designation	Tensile strength (MPa)	Young's modulus (GPa)	Impact strength (kJ/m <sup>2</sup> )
PP/SF	30.60 ± 0.37	2.32 ± 0.04	11.39 ± 0.25
PP/SF/20A	27.00 ± 0.92	2.31 ± 0.05	12.70 ± 0.36
PP/SF/30B	31.82 ± 1.60	2.36 ± 0.03	12.79 ± 0.50

#### 3.3 Flammability

Fig. 3 shows burning rate of PP composites. The flame retardancy mechanism of organoclay involves a high performance carbonaceous silicate char, which built up on the surface during burning. The addition of 20A and 30B showed 15.5% and 37.5% reduction in burning rate of PP/SF composites, respectively. PP/SF/30B composite showed better improvement in flame retardancy than PP/SF/20A composite because PP/SF/30B composite achieved the formation of exfoliated nanostructure. Similarly, Lee et. al.[3] found that the presence of clay at a small amount decreased the burning rate of HDPE/wood fiber composites and also suggested that achieving a higher degree of exfoliation of nanoclay is the key to enhance the flame retarding properties of natural fiber/polymer composites.

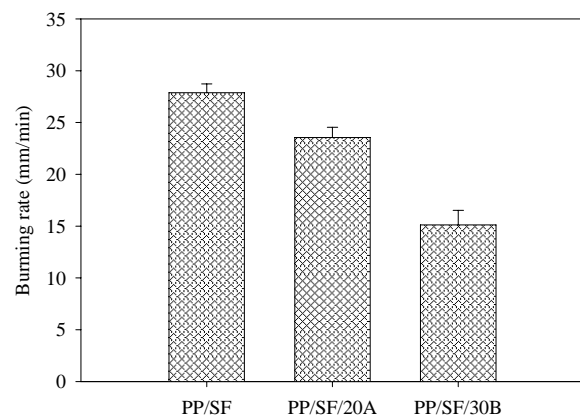


Fig. 3. Burning rate of PP composites.

### 3.4 Thermal stability

The temperature at 5% weight loss ( $T_{5\%}$ ), the temperature at 90% weight loss ( $T_{90\%}$ ), and weight residue at 600°C of PP and PP composites are shown in Table 4. PP showed  $T_{5\%}$  and  $T_{90\%}$  at 285°C and 396°C, respectively, and left no char residue. When compared with PP, PP/SF composite showed no significant change of  $T_{5\%}$ ,  $T_{90\%}$ , and left char residue about 1.8%. Incorporation of organoclay into PP/SF composite increased the  $T_{5\%}$ ,  $T_{90\%}$ , and char residue. This result indicated that thermal stability of the PP composite increased with the presence of organoclay. It may be due to some interaction between polymer matrix and organoclay which stabilized the composite. The well dispersed individual layers of intercalated/exfoliated clay sheets led to a decrease in volatile degradation products permeability [11]. In addition, char reduced the combustion rate of polymeric materials by act as a protective layer to limit the oxygen to easily reach the combustion zone [12].

Table 4. Thermal properties of PP and PP composites.

Designation	$T_{5\%}$ (°C)	$T_{90\%}$ (°C)	Char residue at 600°C (%)
PP	285	396	0.00
PP/SF	286	400	1.80
PP/SF/20A	288	414	3.44
PP/SF/30B	297	407	3.59

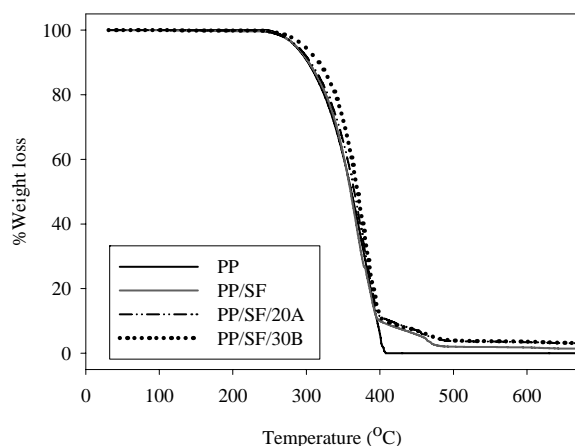


Fig. 4. TGA thermograms of PP and PP composites.

### 4 Conclusions

PP/SF/organoclay composites were prepared by melt blending. Two organoclays (Cloisite®20A and Cloisite®30B) were introduced into PP/SF composites. According to XRD analysis, PP/SF/20A composite showed intercalated structure while PP/SF/30B composite showed exfoliated structure. Adding 30B slightly improved mechanical properties of the PP/SF composite. Using a small content of organoclay improved flame retarding properties of PP/SF composites. PP/SF/30B composite with exfoliated structure showed better flame retarding properties than PP/SF/20A composite with intercalated structure. The addition of organoclay resulted in increased  $T_{5\%}$ ,  $T_{90\%}$ , and char residue.

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