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EFFECT OF COMPOSITION ON MELT FLOW AND DENSITY OF POLYPROPYLENE COPOLYMER/KAOLIN GEO-FILLER COMPOSITES

This study examined the effects rheological properties of different composition kaolin and kaolin geo-filler in polypropylene composites. Polypropylene composites with varying composition of kaolin geo-filler 0 wt%, 2 wt%, 4 wt%, 6 wt%, 8 wt%, and 10 wt% was prepared and compared with polypropylene composite with raw kaolin. Kaolin is an aluminosilicate based mineral filler was used to prepare geopolymer paste by combining with alkaline activator solution. The polypropylene composite was compounded using a twin-screw extruder and the melt flow index was determined by a constant weight pressure of 2.16 kg at 230°C in 10 min. Knowing the melt flow index is necessary to predict and control the process, the study has demonstrated that the composition of kaolin filler and kaolin geo-filler affects the melt flow, melt density and surface morphology at varies composition. Composites with kaolin geo-filler have demonstrated high melt flow index process and having better distribution and flow.

Keywords: Geo-filler; kaolin; composites; polypropylene; rheological

1. Introduction

Kaolin is a common phyllosilicate mineral, belonging to a broad wide-range group known as clays and was produced millions of years ago. The structure of kaolin is made up of silicate sheets that are bonded to the aluminium oxide/hydroxide layers [1-2]. Kaolin's structure and properties are suited for usage as a filler in thermoplastic composites as well as an aluminosilicates based in geopolymer. Geopolymer are synthesized inorganic material comprises of two main constituents, namely alumino-silicate based source material and the alkaline activator solution which able to cure from ambience temperature to elevated temperature [3-5]. The most common used alkaline activators in geopolymer concrete are combinations of sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) solutions [6-9]. The development of geopolymer has attracted global attention due to its advantages of the simple production process, reduction of greenhouse gas emission, and little energy consumption.

Polypropylene (PP), one of the most widely used thermoplastics materials, has a high performance to price ratio because PP is structurally vinyl polymer in which each carbon atom is attached to a methyl group. PP also possesses varying properties according to the process conditions, copolymer components, molecular weight, and molecular weight distribution [10]. However, properties of PP such as toughness or strength, thermal stability, and barrier properties limit its application as a high-performance material and unique materials [11].

Therefore, to improve the properties of polypropylene copolymer, it can be incorporated with kaolin geo-filler. Fillers are widely used to reduce both the production cost and also to improve the properties of the thermoplastics such as its rigidity, strength, hardness, flexural modulus, dimensional stability, crystallinity, electrical and thermal conductivity [12].

The addition of inorganic fillers into thermoplastics enhance various physical properties of the materials such as mechanical strength and modulus. Generally, these properties can be affected by processing condition since the molten polymer is subjected to stress and deformation. Hence, a knowledge of melt rheological may provide useful information to optimize processing conditions, for process design and troubleshooting. Thus, this paper reviews the rheological properties of polypropylene composites with various composition kaolin and kaolin geo-filler.

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2. Experimental

Materials

Polypropylene copolymer grade TitanPro SM340 sourced from Lotte Chemical Titan Holding Sdn. Bhd. Raw kaolin purchased from Kaolin (Malaysia) Sdn. Bhd. Sodium silicate (Na_2SiO_3) type C140HP and molarity of 12 M were used to prepare the sodium silicate solution. Sodium hydroxide (NaOH) of 40 $gmol^{-1}$ molar mass sourced from HmbG Chemicals.

Methods

Kaolin was sieved and placed in an oven at a temperature of 100°C for 24 hours. Kaolin and sodium silicate/sodium hydroxide solutions were mixed at a ratio of 1. The geopolymeric paste was heated in the oven at 60°C for the curing process. Curing the fresh geopolymers at temperatures higher than the ambient is preferred to accelerate the geopolymerization reaction and reduce the setting time [13]. After 24 H, the material was crushed into a fine powder using a ring mill machine. The fine powder was sieved again using a 63 µm sieve to obtain an even distribution of particle size. Polypropylene pallets/kaolin (PPK) and polypropylene pallet/kaolin geo-filler (PPKG) were pre-mixed according to TABLE 1 and TABLE 2 before they were fed into the twin screw with a barrel temperature ranging from 180°C-200°C such in TABLE 3. The extrudate pallets were filled inside the mold between two OHP film and were hot pressed at 180°C for 10 min pre-heat, 5 min compression and 5 min cooling with 15 kg/cm^3 pressure.

Melt flow index (MFI) measures the weight of the polymer in grams extruded in 10 min through capillary by application of

TABLE 1

Sample composition according to weight percentage (wt%) of PP/Kaolin composites

Sample	Polypropylene (wt%)	Kaolin (wt%)
PP	100	0
PPK-2%	98	2
PPK-4%	96	4
PPK-6%	94	6
PPK-8%	92	8
PPK-10%	90	10

TABLE 2

Sample composition according to weight percentage (wt%) of PP/Kaolin-geo-filler composites

Sample	Polypropylene (wt%)	Kaolin (wt%)
PP	100	0
PPKG-2%	98	2
PPKG-4%	96	4
PPKG-6%	94	6
PPKG-8%	92	8
PPKG-10%	90	10

TABLE 3

Barrel temperature profile of the extruder

Barrel Zone	1	2	3	4	5	6	7	8
Temperature (°C)	180	180	190	190	190	200	200	200

load in accordance with ASTM D1238-73. The pre-heated temperature was set at 190°C for 6 min, under the condition of 2.16 kg load, and test temperature set at 230°C. The melt flow index (MFI) (g/min) value was calculated using the following equation:

$$MFI = \frac{600 \times W}{t} \tag{1}$$

where W is the weight of extrudates and t is interval cutting time (seconds). The melt density, ρ_m (g/cm^3) value was calculated using the following equation:

$$\rho_m = \frac{W}{A \times l} \tag{2}$$

where A is the cross-section area of the piston (cm^2), and I is the piston travel distance (cm).

The extrudate from melt flow indexer were taken for observation under Scanning Electron Microscopy (SEM) JEOL JSM-6460LA.

3. Results and discussions

Melt Flow Index and Density

The melt flow index (MFI) shows a slight variation in the values of the index of fluidity compared to that of the matrix. Fig. 1 illustrates the variations of the melt flow index (MFI) of PP/kaolin (PPK) and PP/kaolin geo-filler (PPKG) composites. According to the Fig. 1, the highest MFI value for PPK is at 8 wt% filler. Meanwhile, for PPKG, the highest MFI value is at 6 wt% filler. Beyond this percentage, the MFI value decreases.
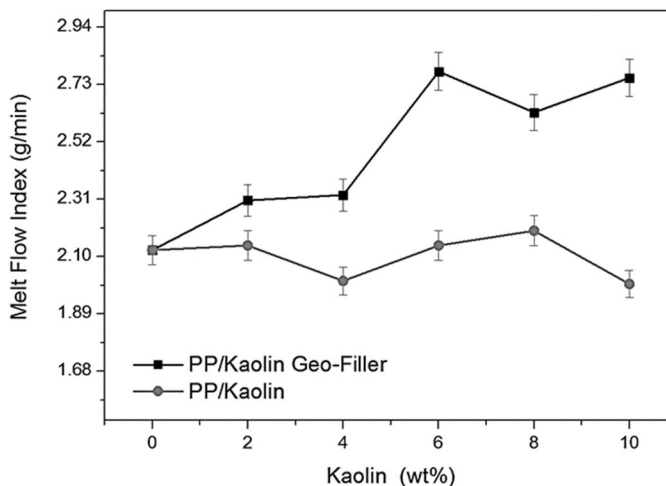

Fig. 1. PP composites melt flow index variations as a function of raw kaolin and kaolin geo-filler concentration

The decrease in MFI value was due to the formation of agglomerates arising within the matrix in consequence of poor dispersion of particles. These agglomerates can reduce the flow of polymer, increasing in viscosity and the melt index tends to decrease with higher incorporation of kaolin loading [14]. During the shear dispersion process, kaolin particles tend to agglomerate, forming a bigger particle. Thereby, when shear flow occurs, collision and friction between particles increase, resulting in flow resistance and thus decrease in mobility. The increasing viscosity resulted in limited molecular mobility and the reduction in free volume influenced by the interaction and dispersion of filler in the matrix [15].

It can also be observed that the MFI values for PPKG are higher than those of PPK. This increase was due to the addition of kaolin based geopolymer, which facilitates the flow of the polypropylene matrix chain. This behavior is highly connected by the effect of the spheroidal-like shape of kaolin geo-filler particles (Fig. 2), which can slide in the polymer melt [16]. As a consequence, the ease of flow occurred in kaolin geo-filler compared to raw kaolin which have irregular platelets that are arranged in booklets and face-to-face patterns (Fig. 2(b)).

The melt density obtained with the melt flow indexer was similar to the MFI pattern. The melt density for PPKG increases up to the 6 wt% geo-filler and decreases beyond that. Whereby for PPK, the maximum melt density obtained was at 8 wt% filler and the melt density decrease beyond that. These results are shown in Fig. 3.

The melt density values increase with an increasing amount of filler loading. Theoretically, the density increases with an increasing amount of filler incorporated into the matrix. This is due to the presence of kaolin, which has a higher density than that of polypropylene. Since there was an increasing amount of free volume, the polymer density was reduced [17]. Hence the decrease in melt density beyond 6 wt% for PPKG and 8 wt% for PPK. Besides that, the melt density for PPKG was higher compared to PPK for all filler loading. This can be due to the small particle size of the geo-filler, compared to raw kaolin, as discussed earlier. Since the particle size of the geo-filler is smaller, they are freer to slide to each other. Hence, the chain mobility of the polymer increase, the viscosity of the polymer decreases and resulting in a higher melt density value.
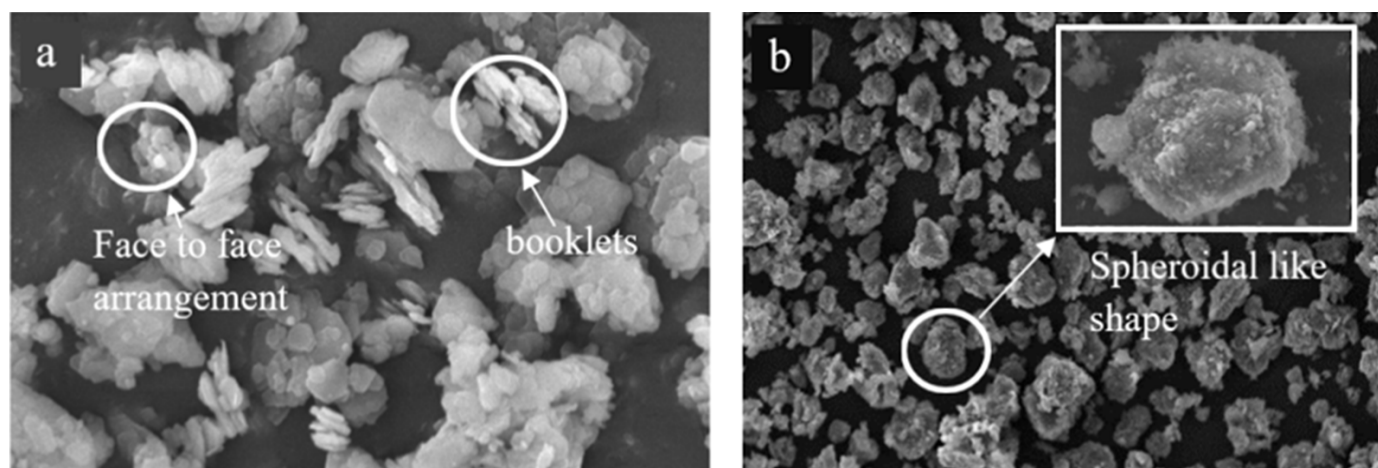

Fig. 2. SEM image of (a) raw kaolin and (b) kaolin geo-filler
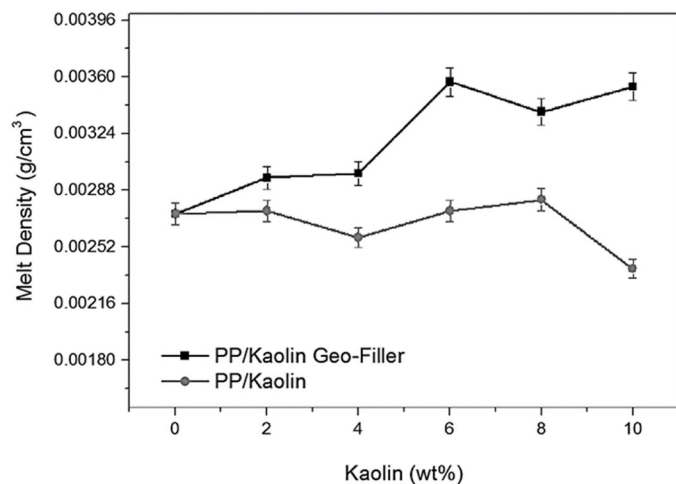

Fig. 3. PP composites melt density variations as a function of raw kaolin and kaolin geo-filler concentration

Extrudate Surface Morphology

The extrudate surface morphology of PP, PP/kaolin (PPK) and PP/kaolin geo-filler (PPKG) composites from the melt flow indexer was further studied and illustrated in Fig. 4 and Fig. 5. From Fig. 4, PP exhibits the usual polyolefins flow behavior with smoother extrudate surface. As the kaolin and kaolin geo-filler incorporated in PP composites, the extrudate shows rougher in surface with the presence of porous such in Fig. 5(a), (b), (c), and (d). This situation can be explained by the poor interaction between the matrix and filler, especially at 6 wt% of kaolin and kaolin geo-filler which reveals the presence of porous surface, resulting in a phase separation. Due to the hydrophilic nature of kaolin, whereas polypropylene exhibit hydrophobic character, there are challenges of incompatibility [18]. The polarity difference between polypropylene and kaolin has a negative influence on the performance and quality of polypropylene-based products.
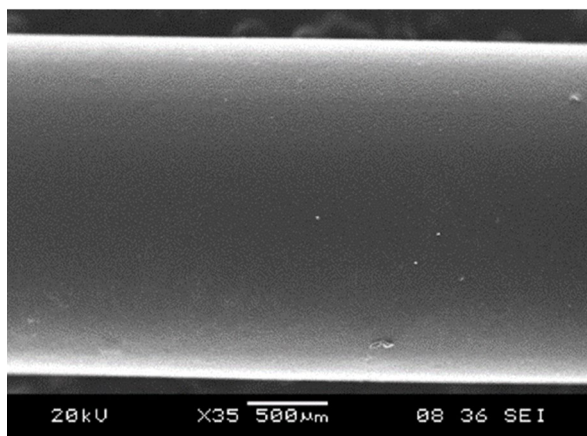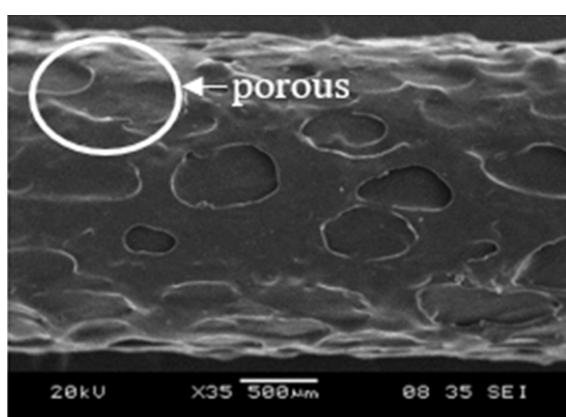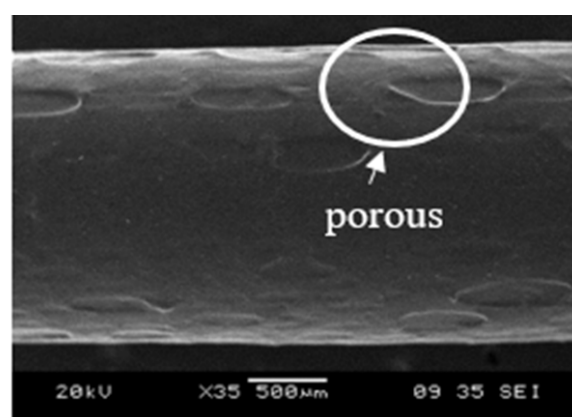
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Fig. 4. Extrudate surface morphology of PP

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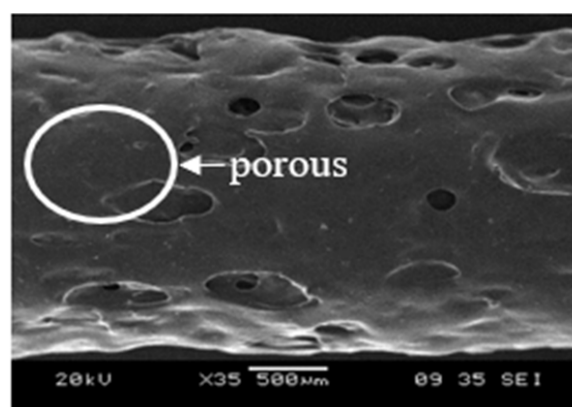
a)

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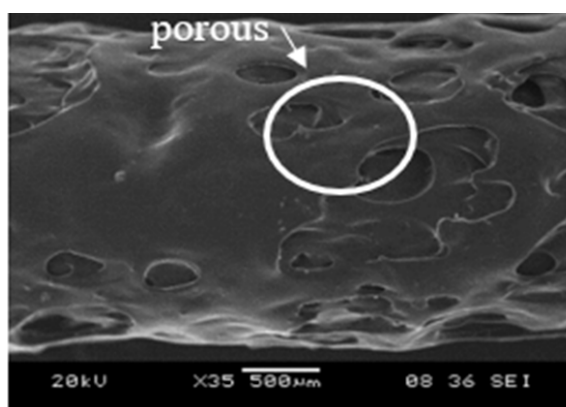
c)

d)

Fig. 5. Extrudate surface morphology of (a) PPK-6% (b) PPK-8% (c) PPKG-6% (d) PPKG-8%

4. Conclusions

This study is useful in determining density and melt flow index for different filler and various filler composition in polypropylene copolymer. From the observation for MFI under condition of 2.16 kg load, and test temperature 230°C, polypropylene with kaolin geo-filler has indicated high melt flow index due to spheroidal-like shape of kaolin geo-filler particles as compared to origin plate shape particles. The useful information is beneficial to further studies on processing polypropylene/kaolin geo-filler through extrusion.

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