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Research paper



Mechanical and Thermal Properties of Sayong Clay/HDPE Composites

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Abstract

The present study showed the effect of Sayong clay as filler on the mechanical and thermal properties of high-density polyethylene (HDPE). Different concentrations of clay (1 wt%, 3 wt% and 5 wt%) were introduced which are reinforced to HDPE composites and was prepared by melt mixing using a compounder system. The prepared composites then undergo mechanical testing and thermal study to evaluate its effectiveness after added clay. The effect of Sayong clay addition was different for each clay content and the highest tensile and flexural strength were observed for HDPE/clay composites with 5 wt% of clay at 20.98 MPa and 14.73 MPa respectively. The results showed that Sayong clay/HDPE composites with 5 wt% of clay have considerable effect upon increasing the strength properties of HDPE/clay composites. In addition, the result revealed that the accumulated clay particles that arise with addition of 3wt% of clay caused the decreased in impact strength. To study the melting point and crystallization behaviour of clay reinforced with HDPE, DSC were conducted on the samples at the temperature of 50°C to 180 °C. The result indicated that the higher the melting point of the mixture was achieved by increasing content of Sayong clay. The crystallization behaviour of HDPE is affected with the addition of Sayong clay in which make the composite easily become crystal form even at high temperature. As such, Sayong clay is can act as reinforcing filler and produced polymer composites with improved strength properties.

Keywords: *HDPE; Sayong clay; mechanical testing; thermal analysis (DSC).*

1. Introduction

High density polyethylene (HDPE) is one of the polyethylene thermoplastics produced from the cracking process of petroleum. Application of HDPE are widely used mostly in industrial application because it is known as an engineering thermo-plastic [1]. The unique feature of high-density polyethylene (HDPE) and the abundant of the raw materials also makes it one of the best plastic types to use in daily life. This is due to its light weight, good mechanical properties, almost zero dampness retention, and is a great boundary for mugginess and protection from scraping and corrosion. It is a very crystalline structure in contrast with different sorts of polyethylene [2]. However, there are some issues involved in the use of HDPE which is their ability to cope with high heat and high impact because HDPE is known to have low thermal resistance [3]. It is important for a polymer to have high capacity to withstand heat for its future application. Hence, the composition of the HDPE needs to be improved in order get the good structure that can bear high temperatures and impact compared to the pure structure of HDPE.

In order to improve the structure of HDPE, the addition of filler is important. Filler can be organic, inorganic, carbonaceous, or metallic and in solid, liquid or even in gas phase. One of the potential fillers is nanoparticle such as montmorillonite that can escalate the properties of polymer [4]. There are many types of clay such as ball clay, fire clay and porcelain clay that can contribute certain characteristics instead of having the same compositions of molecular compounds. Clay is obtained from the soil layer which has high surface area that contains trace of metal oxide and other minerals. Due to its ability to withstand high temperature, clay was chosen as filler in the polymer. It also offers a high level of polymer–clay surface relation which brings about boundary and mechanical properties that are superior to those of the base material [5]. Clay minerals that are raw are the most commonly used in the inorganic material. Clay is also considered as a low-cost material and eco-friendly and is abundant all over the world. Counter balanced clay such as Na-Mt contains mostly polar structure which makes the clay a suitable candidate with the most polymer [6]. The incorporation of polymer and clay theoretically showed good results in terms of impact and has the ability to increase the resistant of the polymer towards high temperatures [7].

A trace of clay nanoparticles has greater heat resistance and force attraction in the molecule structure because of the larger surface area of clay. This is due to the mineral content in the clay such as silicate which is a rich intercalation and ion-exchange that makes the polymer matrix compatible. It is known that the exfoliation within the clay layers creates maximum interfacial interaction and encourages the matrix reinforcement [8]. The compatible structure of the polymer matrix with clay can provide a minimal increase in

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the strength of the HDPE. The amount and the size of the clay added determines the reinforcement of the polymer matrix [9]. However, the addition of clay will not affect the HDPE crystallization rate if the clay does not reach the appropriate amount [3].

Studies conducted by University College London had found that the durability of clay exposed to high heat is dependent on the type of mineral content [10]. Nanoparticle clay is a well-known adhesive that can modify the properties of the HDPE [11]. In this research, Sayong clay was used as the adhesive compared to the other nanoparticle clays to elucidate its capability in increasing the properties of HDPE. Sayong clay contains high kaolinite compounds, 25.5% other than alumina 32.5%, silica 57.4%, water and oxygen [12]. These compositions are expected to enhance the properties of HDPE. Moreover, Sayong clay was used due to its abundance and environmental friendliness. In most studies where clay was used, only a small amount is needed as a compatibilizer. However, based on our knowledge, there are only a few reports done using similar systems [13].

This research explores the possibility of using Sayong clay as a significant part of the filler in polymer matrix and compares the properties of these newly developed composite with pure HDPE. Different compositions of clay (1wt%, 3wt%, and 5 wt%) were studied. Theoretically, the addition of clay may influence the change in properties of HDPE especially towards thermal behavior and mechanical properties. The objectives of this study are to investigate whether the addition of Sayong clay in HDPE can improve the thermal behaviour of this polymer when exposed to high temperatures and the mechanical properties.

2. Methodology

2.1. Materials

Sayong clay was obtained from Sayong, Kuala Kangsar, Perak and high-density polyethylene (HDPE) used in this study (homopolymer) was obtained from Lotte Chemical Titan Sdn. Bhd in Pasir Gudang. Melted plasticizer was used as a compatibilizer. The tray full of clay was placed in the oven at a range of 135°C – 140°C for 20 minutes. The clay is then crushed into small particles and sieved into 125 microns in size. The Sayong clay is then stored in a sealed plastic bag to avoid humidity.

2.2. Preparation of composites

Dried Sayong clay and high-density polyethylene (HDPE) pellets were mixed based on different weight ratios (C1: 1wt%, C2: 3wt%, and C3: 5wt% of Sayong clay). Once the ingredients of each composite formulation were weighed to a 0.01g precision, they were manually mixed. Care was taken to mix the ingredients as homogenously as possible. The compounding of the three compositions was performed with mixing conditions of 100 rpm and 180°C for 15 min in the single-screw extruder to produce Sayong clay/HDPE composites in pellet form. The mixture of Sayong clay/HDPE composites were prepared in a Techno Vation micro compound extruder (EXT30-10-01) operating at a constant speed of 16.2 Hz and a barrel temperature of 180 °C. All the materials were fed into the extruder using single feeder at a total flowrate of 1 kg/h. The other samples were also prepared under the same conditions. The samples for analysis were produced by injection moulding. The extruded materials containing Sayong clay/HDPE were melted using heater bands and the frictional action of a reciprocating screw barrel with the temperature of 200°C and screw speed of 60 rpm. The plastic is then injected through a nozzle into a mould cavity where it cools and hardens to the configuration of the cavity.

2.3. Composites analysis

2.3.1. Mechanical Testing

Tensile tests were conducted in accordance with ASTM D638-99 (ASTM, 2002) using a standard material testing system (MTS-810 load frame with 5 kN load cell) at a crosshead speed of 50 mm/min. The extensometer was used to measure strain over an 80 mm gage length. The flexural properties were measured in three-point bend tests using a standard Material Testing System (MTS-858 load frame with 2.5 kN load cell) at a crosshead speed of 2.8 mm/min in accordance with ASTM D790 (ASTM, 2002). The flexural test specimens were also obtained from the composite panels with dimensions of 130 X 12.7 X 6.4 (thickness) mm. The tensile and bending measurements were performed at room temperature conditions of 27°C. Five replicates of each polymer composites formulation were tested for tensile and flexural properties. The Izod impact tests were carried out using Toyo Seiki Digital impact tester with an impact velocity of 1 m/s. The v-notched specimens were subjected to the impact test in the temperature range of 25°C in accordance with ASTM D-256.

2.3.2. Differential Scanning Calorimetry (DSC)

The crystallization and melting characteristics of Sayong clay/HDPE were determined by using differential scanning calorimetry (DSC) in accordance with ASTM E-793. The Perkin Elmer DSC 6000 was utilized, running composites samples through a heat-cool-heat cycle at temperature 50°C to 180°C and held about 1 minute at high temperature to erase the thermomechanical history and to achieve a relaxed melt. Then the samples were cooled to 50°C at the rate of 10°C/min, and a second scan was reheating at 50°C to 180°C at the rate of 10°C/min.

3. Results and discussion

3.1. Tensile testing

Fig. 1 shows that the results of tensile strength of pure HDPE and Sayong clay/HDPE. Results showed that pure HDPE has higher tensile strength as compared to the others. The tensile strength of 1 wt% of Sayong clay/HDPE and 3 wt% of Sayong clay/HDPE decreased by 5 MPa as compared to pure HDPE. It was also observed that with increasing clay content, there is a slight decrement of tensile strength, but it was increased when 5wt% of clay added.

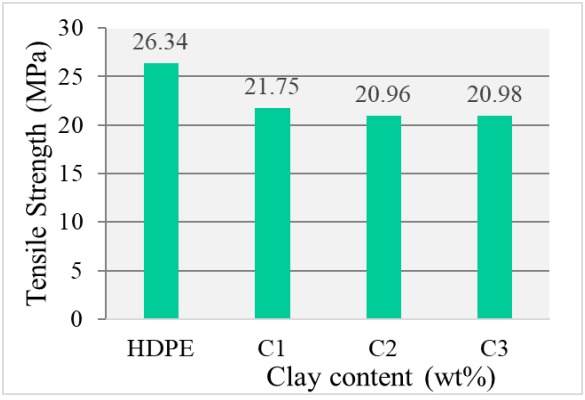

Fig. 1: Variation of tensile strength with different Sayong clay composition in Sayong clay/HDPE composite

Fig. 2 shows the tensile modulus of pure HDPE and Sayong clay/HDPE. The reduction of tensile strength and modulus with an increasing of Sayong clay were due to poor adhesion between HDPE matrix and clay in the composites. This result is similar to studies conducted by Singh et al. [14] where low tensile strength and modulus were observed with an increase of hemp fiber content. It is expected due to the poor adhesion between the HDPE

matrix and hemp fibers in the composite samples of the matrix. Besides, the lower tensile strength and modulus is probably caused by the orientation during the preparation of composites as it impossible to align the filler in a straight manner. In the present study, the low tensile strength is due to the agglomeration of clay particles which caused the poor adhesion on HDPE matrix. On the other hand, the high tensile strength of 3 wt% of Sayong clay/HDPE contributes to the good adhesion of clay on the HDPE matrix. This result is in line with the study of Krishnan et al. [15] where the surface area of interaction between the clay and polymer matrix increased after the insertion of the polymer inside the silicate layers. Based on the value of tensile strength, it appears that tensile properties increased with the use of inorganic filler such as clay at 5wt%. It is expected as stress is more efficiently transferred from the polymer matrix to the inorganic filler [16]. Thus, the tensile strength of the Sayong clay/HDPE composite falls in the range of 20.96 MPa and 26.34 MPa. The pure HDPE composite exhibits higher tensile strength (26.34 MPa) as compared to the Sayong clay/HDPE at different composition of clay (1wt%: 21.75 MPa, 3wt%: 20.96 MPa and 5wt%: 20.98 MPa). This means that 5wt% clay filler is enough to connect the polymer with the HDPE in the blends and eventually lead to improve the structure. Based on the research conducted by Asgari et al. [6], the tensile modulus and yield stress of HDPE can be enhanced by the addition of clay into HDPE. Other than enhancement in yield and tensile strength, the addition of clay also increases the elongation at break. The improvement in tensile modulus is due to strong interaction between matrix and silicate layers through the formation of hydrogen bonds.

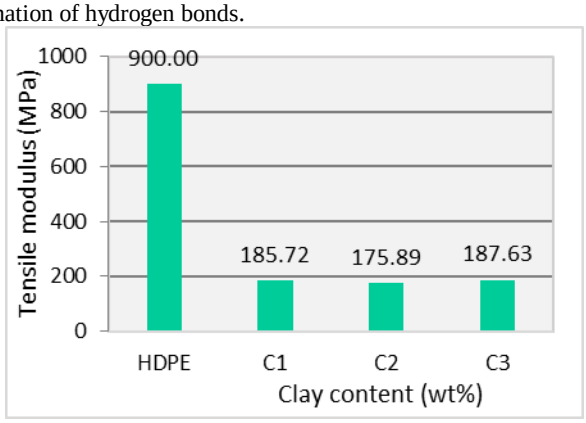

Fig. 2: Variation of tensile modulus with different Sayong clay composition in Sayong clay/HDPE composite

3.2. Flexural testing

The flexural strength of the composites was shown in Fig. 3. It shows that the flexural strength of HDPE is higher compared to 1wt%, 3wt% and 5wt% containing Sayong clay/HDPE composites. By comparing the value of flexural strength of HDPE, the flexural strength of 1wt% and 3wt% clay was decreased gradually up to 6 MPa. However, for composite with 5wt% of clay content, the value of flexural strength is higher than 3wt%.

The flexural modulus of the Sayong clay/HDPE composites are shown in Fig. 4. The flexural modulus of 1 wt% and 3 wt% are higher than the pure HDPE. The flexural modulus 3wt% is observed to experience a slight decrement when the content of clay is increased from 1 wt% to 3 wt% in a composite of HDPE and clay. By comparing the value of flexural modulus, the flexural modulus of 3wt% decreased gradually up to 74 MPa. Nevertheless, for composite C3 with 5 wt% of clay content, the value of flexural modulus is higher than C2 (3 wt%).

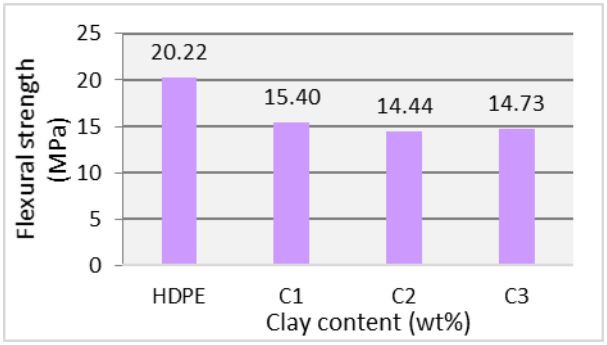

Fig. 3: Variation of flexural strength with different Sayong clay composition in Sayong clay/HDPE composite

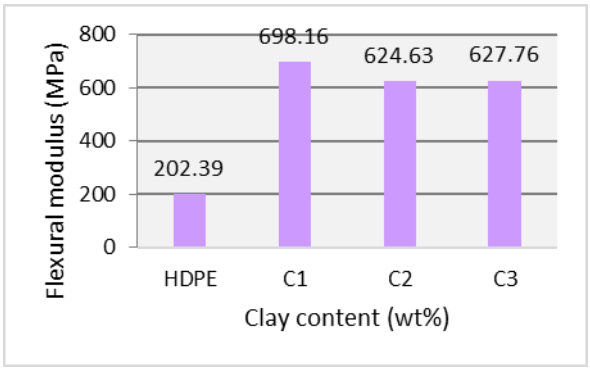

Fig. 4: Variation of flexural modulus with different Sayong clay composition in Sayong clay/HDPE composite

The flexural strength exhibits a similar trend to the tensile strength although less variations is observed for flexural strength. It can be seen that the flexural strength decreased with increasing clay content up to 3wt%. The higher flexural strength of the composite HDPE/Sayong clay at 1wt% (15.40 MPa) were observed as compared to 3wt% (14.44 MPa). However, the addition of 5 wt% of Sayong clay slightly improve the flexural strength. The poor dispersion of clay was expected such as agglomeration of clay in polymer matrix and lack of strong interfacial interaction cause some reduction in the strength of polymer/clay nanocomposites [17]. Moreover, it may be caused by the crystallinity of HDPE polymer which affects the value of tensile and flexural modulus. The increase of flexural strength and modulus indicate that nanocomposite had become more rigid and less flexible. It also recommended by Albano et al. [18] that several parameters need to be considered include of average particle size and particle size distribution of the filler. These factors will affect the mechanical properties [19] and the wettability of the filler [20]. Other than that, it is also important to note that for small particles, the interface between the filler and the binder plays a significant role in the mechanical properties of composite.

3.3. Impact testing

Table 1 shows the impact strength of HDPE for all composites compositions. Pure HDPE displayed an impact strength of 12.183kJ/m^2. However, the value decreased when clay was added. These results are supported by Valek [21] who stated that the presence of solid filler usually degrades the strength and toughness of polymeric materials. The decrease in impact strength of 3 wt% clay is due to the accumulated clay particles that arise from the composition. The increasing clay content causes an increased in clay accumulation and decrease in interparticle distance, which lead to a lower aspect ratio of clay particles and lowered the contact surface area, resulting in a weak bond between HDPE matrix and clay, subsequently lowering their impact strength. These results are supported by Bashar [22].

Table 1: Impact Strength value for all compositions

Compositions	Compositions of Clay (wt %)	Impact strength (kJ/m^2)
HDPE	0	12.183
C1	1	3.952
C2	3	3.587
C3	5	4.085

Above all, the impact strength of C3 is higher than C1 and C2 and showed better dispersion of Sayong clay in HDPE matrix than others. The quality dispersion of particles in HDPE matrix is vital as it plays an important role in improving the properties of HDPE such as impact strength [23]. The improvement in their properties usually depend on the results of toughness of the composite produced [24]. The positive results of the clay as a filler depends on the amount of clay added to the HDPE matrix. Too much clay added is not good as its limits its application [25]. Thus, a suitable amount of reinforcing clay particle present in the HDPE matrix can act as an efficient impact absorption agent in the polymer [26]. In general, the clay composition displays great improvement in mechanical properties, such as the strength compared to pure HDPE upon addition of minimal amount of micro size clay particles [27].

3.4. Differential scanning calorimetry (DSC)

The DSC test was performed to determine the melting point and crystallize temperature of Sayong clay/HDPE mixture. Fig. 5 illustrates the results plotted based on different weight percentages of Sayong clays in Sayong clay/HDPE matrix with the specific heat. Results show that all Sayong clay/HDPE samples started to melt at around 110°C and the mixture absorbed heat until it fully melts at its melting point. From the results obtained, the melting point is increasing due to an increase of composition of clay used. As expected, the HDPE has a slightly low melting point as compared to Sayong clay/HDPE. The high melting point of Sayong clay/HDPE was due to the high energy required to melt the sample. Fig. 6 represent the crystallization temperature graph of all samples. The crystallization parameters such as onset temperature (T_o) be the temperature where the sample starts to undergo crystallization process. T_e is the end peak of crystallization process and the T_p is peak temperature that the composite starts to form crystal. The data for the Fig. 6 is summarized by Table 2. The addition of Sayong clay in HDPE generally shows an increase of T_o and T_p value by increasing amount of Sayong clay from 1-5 wt%.

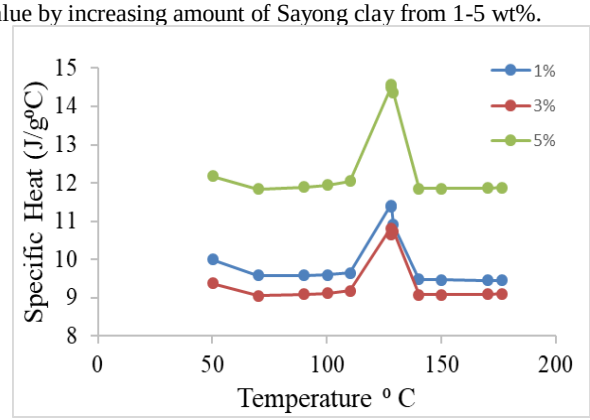

Fig. 5: Graph of specific heat and temperature with different composition of Sayong clay/HDPE

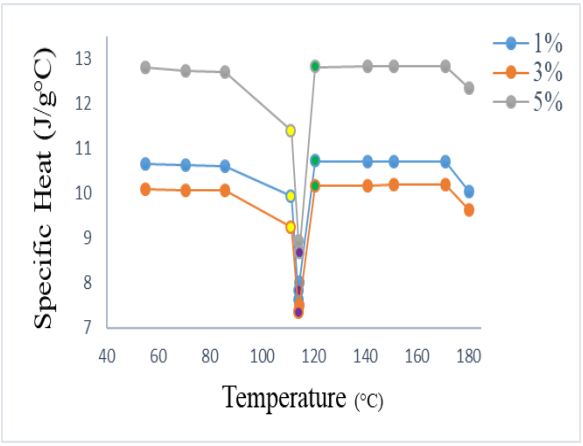

Fig. 6: Graph of crystallization temperature for 1 wt%, 3 wt% and 5 wt% of Sayong clay/HDPE

Table 2: Parameter for crystallization rate

Cooling rate (^{o}C/min)	Crystallization parameter (^{o}C)	C1	C3	C5
10	T_o	115.9	116.18	116.46
	T_p	114.1	114.43	114.63
	T_e	111.6	110.76	110.30

For the crystallization of the pure HDPE and Sayong clay/HDPE, crystallization started at almost the same temperature. The different amount of clay results in early crystallization when it reached peak temperature as compared to pure HDPE. It means that at high temperature, the Sayong clay/HDPE composite can crystallize compared to the pure HDPE. The results are almost same with the prediction that the addition of clay as filler in HDPE make the composite easily turns to crystal form even at high temperature. Other than that, the clay filler in Sayong clay/HDPE cause the size of crystal grew thicker than the HDPE composite. These results were proven by Chen & Yan [28] where higher temperatures can affect the size and thickness of the crystal. Table 3 shows the melting temperature, crystallization temperature and crystallinity level.

Table 3: Crystallinity level, X_c(%)

Composite	Melting point, T_m (^{o}C)	Crystallization peak temperature, T_c (^{o}C)	Crystallinity level, X_c (%)
HDPE	127.08	114.08	81.51
C1	127.82	114.10	19.08
C2	128.14	114.43	22.65
C3	128.29	114.63	36.51

The crystallinity level for the Sayong clay/HDPE composite had increased when the melting point of the composite increase. This is because the clay molecules in HDPE affect the structure of the composite which causing the HDPE structure to be more crystallize. Furthermore, the addition of Sayong clay as the filler in the HDPE will also affect the specific heat capacity of the composite. From Fig. 5, the Sayong clay/HDPE clay has a higher specific heat capacity compared to pure HDPE. For example, 5wt% percent of clay composite shows a higher specific heat capacity of about 8.66 J/g.°C and pure HDPE is about 1.9 J/g.°C. These results show that the Sayong clay/HDPE composite are exceptionally good at regulating the temperature. This statement was proven by Anne Marie Helmenstine [29] who stated that the higher the specific heat capacity, the higher the regulating temperature.

The crystallization rate can affect other properties of the composite such as the impact of the Sayong clay/HDPE toward high force. According to Tanniru et al. [30], clay/HDPE composites becomes more brittle at high temperature which results in a higher amount of clay produced, and lower mechanical properties toward high impact. This conclusion is based on the impact test that was conducted which is, the higher content of clay, the lower the impact of the HDPE composite.

Overall, the addition of clay in HDPE shows good result in thermal properties where it is able to crystallize at high temperature. Although the composite displays an advantage of being crystalline in a short time, the composite structure is easy to break when high impact energy is applied. However, a direct comparison of the composite properties of Sayong clay/HDPE clay is not always possible because each type of clay has its own mineral content depending on its origin.

4. Conclusion

A comprehensive study was performed on Sayong Clay/HDPE composites to obtain the mechanical properties and the thermal behaviour at different percentages of clay. Results showed that the addition of Sayong clay to the HDPE decreased the toughness of the composite. Clay contents also affect the mechanical properties of HDPE matrix by reducing the elasticity and bending force of the matrix. The thermal properties in terms of melting point and crystallization behaviour were observed. The result showed that the Sayong clay filler can easily crystallize HDPE even at high temperatures. Overall, the addition of 5wt% of clay content into HDPE matrix shows improvement in mechanical properties in terms of elasticity and bending capability. Further studies of this research can be carried out using modified clay in nano size as the fillers in HDPE matrix.

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