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MECHANICAL, THERMAL AND FLAMMABILITY PROPERTIES OF DOLOMITE FILLED POLYPROPYLENE COMPOSITES

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ABSTRACT

In this project, the composites of polypropylene (PP)/dolomite were prepared via extrusion and injection moulding. Dolomite mineral was used as reinforcement in PP matrix. PP reinforced dolomite composites with various concentrations of dolomite (5, 10 and 15 wt%) were characterized by mechanical properties through tensile, impact and flexural test, morphological analysis by scanning electron microscope (SEM), thermal analysis by differential scanning calorimeter (DSC) and flammability analysis by the limiting oxygen index (LOI). The incorporation of dolomite into PP had improved Young's modulus while flexural modulus, tensile, flexural and impact strengths were decreased. The incorporation of dolomite up to 15 wt% increased the stiffness of the composites in tensile mode while in three point bending mode, 10 wt% dolomite was the optimum concentration. Tensile and flexural strength showed a slight reduction in the values while impact strength was continuously decreased with the addition of dolomite. SEM images showed poor interfacial adhesion between PP and dolomite, thus supported the decreased of tensile, flexural and impact strengths. The melting and crystallization temperatures (T_m and T_c) of the composites slightly increased with the addition of dolomite. LOI test showed that flammability of the composites decreased with the increasing content of dolomite.

Keywords: dolomite, polypropylene, flammability, mechanical, thermal

1.0 INTRODUCTION

Polypropylene (PP) is the most widely used polyolefin and one of the commodity plastics that has the highest growth rate [1]. PP has a good chemical, heat and fatigue resistance, semi-rigid, translucent, tough and integral hinge property. Due to its great qualities, PP has become a versatile polymer with various applications including packaging and labelling, textiles, stationery, plastic parts and reusable containers of various types, laboratory equipment, loudspeakers, polymer banknotes and automotive components. In the last 50 years, PP has become one of the most widespread commodities, employed in the industry due to its low cost and good mechanical performances. PP is normally tough and flexible and these advantages permit it to be used as a building plastic, battling with other materials such as acrylonitrile butadiene styrene (ABS).

Polymers are traditionally reinforced with inorganic fillers to improve their properties as well as to reduce cost. PP is one of the polymers that are widely used in industry due to its well-balanced physical and mechanical properties, as well as processability at a relatively low cost [2]. For the automotive application, PP has to fulfill a balance of several mechanical properties especially impart resistance and stiffness. Nevertheless, the ability of PP to be used as an engineering thermoplastic has been limited by its high flammability, the tendency to brittleness at temperatures below its glass transition temperature, and low stiffness particularly at low temperatures. Besides, the nature of PP is nonpolar because it does not contain any polar groups on its backbone, so it is not quite compatible with almost all the fillers and reinforcements used in polymer industry which are polar [3].

Due to these limitations, PP needs to be further improved so that it can be used in broader applications. Thus, PP is improving by adding a filler into it. Leong *et al*. [4] had done a research on mechanical and thermal properties of talc and Calcium Carbonate ($CaCO_3$) filled PP hybrid composites.Their main aim was to study and compare the mechanical and thermal properties of talc and $CaCO_3$/PP composites and single-filler PP composites. The results showed that a synergistic hybridization effect was successfully achieved. The nucleating ability of talc which was influenced as the main nucleating agent in the hybrid fillers had contributed to the improvement in mechanical properties of PP hybrid composites. In another study, Tuah [5] studied on the mechanical and thermal properties of PP/clay roof tiles waste (CRTW) composites. CRTW improved the hardness properties and melt density with the increases in the filler content. For the thermal properties at maximum loading of filler content, it shows the maximum temperature of weight loss rate, T_{peak} and the onset decomposition temperature, T_{onset} were higher compared to unfilled PP.

The introduction of particulate inorganic mineral fillers such as montmorillonite, silica, $CaCO_3$, hydrated alumina, talc and mica into polymers is the most common approaches to improve polymers properties. Inorganic polymer composites are created by reinforcing polymer with fillers to produce a material with properties which were improved to those of the individual base polymer. The incorporation of inorganic fillers into polymers gives a reinforcing effect to the properties of the individual polymers. Mineral fillers which are otherwise called as inorganic fillers are inert substances added into the polymer to lessen the polymer cost and/or enhance its physical properties, hardness, firmness, optical properties, flammability, thermal properties and impact strength [6]. Furthermore, the effects of filler on the mechanical and other properties of the composites depend strongly on its shape, particle size, aggregate size, surface characteristics and degree of dispersion [7].

Recently, instead of common inorganic fillers mentioned earlier, polymer composite industries had discovered and commercialized a new material which is known as dolomite. It is combination of the elements calcium (Ca), magnesium (Mg), carbon (C) and oxygen (O_2) with formula composition of $CaMg(CO_3)_2$ and has a crystal lattice consisting of alternating layers of Ca and Mg and separated by layers of carbonate (CO_3). This ideal chemical structure of dolomite was reported by Warren [8]. Dolomite mineral is commonly found in deposits of sedimentary carbonate rock called dolostone. An attempt of using it as a filler to improve the properties as well as the flame retardant properties in the polymer is a new approach than using other inorganic mineral filler.

Several researchers had reported the addition of dolomite into PP [9-11] . Ali *et al*. [9] had studied on the preparation and characterization of polyether-based PU/dolomite composite. The mechanical properties exhibit higher tensile strength and elongation when the composite is at the lower filler loading. Besides, it shows that up to 30 wt% filler loading, thermal conductivity (λ) increases. According to Saleh *et al.* [10], the addition of dolomite which was hybrid with Multi-wall Nanotubes (MWNTs) can improve the mechanical and thermal properties of PP. He studied the use of single dolomite and MWNTs-dolomite as a hybrid fillers in phenolic composites.The work has analyzed the effect of both fillers to the thermal conductivity and hardness of the phenolic compared to the pure one and the results showed that they were capable of increasing those properties up to 7% and 100% respectively. A study by Adik *et al.* [11] showed that the elongation at break decreased with filler loading and showed better and improved result after dolomite is being treated with stearic acid. The incorporation of stearic acid coated filler into PP matrix enhanced the break elongation of the composites that makes the composites more ductile. Morphological analysis using Scanning Electron Microscopy (SEM) proved better interfacial adhesion and less agglomeration of dolomite filler after treatment with stearic acid at low filler loading (5 wt%).

In 2015, Valverde *et al.* [12] studied the decomposition of dolomite under carbon dioxide (CO_2). The results showed that natural dolomite is a potentially advantageous alternative to limestone for CO_2 capture in the Calcium-Looping (CaL) technology as well as sulphur dioxide (SO_2) in situ removal in oxy-combustion fluidized bed reactors. carbonation reactivity in the solid-state diffusion controlled phase is remarkably enhanced for carbon oxide (CaO) derived from dolomite in the presence of CO_2. The use of dolomite would therefore allow a significant increase in the carbonation efficiency by prolonging the residence time of the solids in the carbonator reactor [13]. Thus, it can be concluded that dolomite owned a flame retardant properties since it releases CO_2 gas at elevated temperature [14].

Until now, the influence and effect of dolomite minerals on the mechanical, thermal and flammability properties of PP resin has not been reported in the literature. In this study, dolomite reinforced PP composites were prepared by melt intercalation method using twin-screw extruder and injection moulding. Then, the effect of dolomite (5, 10 and 15 wt%) on PP is being investigated.

2.0 EXPERIMENTAL

2.1 Materials

In this study, dolomite was obtained from the Jabatan Mineral & Geosains (JMG) at Ipoh, Perak, Malaysia. The dolomite was produced by a high vitality processing preparation that includes rehashed blending, disfigurement, comminuting, welding and re-welding of the reactant powder particles in a shut vial of a planetary ball plant. PP was supplied by Lotte Chemical Titan (M) Sdn. Bhd. at Pasir Gudang, Johor, Malaysia.

2.2 Preparation of PP/Dolomite Composites

The samples of four formulations of PP/dolomite composites according to the formulation in Table 1 were prepared by adding different amounts (5, 10, and 15 wt%) of dolomite and abbreviated as PP/D-5, PP/D-10 and PP/D-15, respectively. Sample which contained pure PP act as the reference and the control. The dolomite mineral was physically mixed with PP resin before it was extruded. Then, the mixture was oven-dried at 70 ˚C for 24 hours to remove the moisture before processing. After a complete drying, the mixture was melt blended in a co-rotating twin-screw extruder with L/D = 36 which was used in the compounding process (Sino-Alloy PSM32). Extrusion was conducted at a range of speed of 162-169 rpm and the temperature profile adopted during compounding was 200 ˚C. The strands were then air-dried and pelletized (2000g = basis of the extruder). Then, the pellets were oven-dried at 70˚C for 24 hours before they were processed in the injection moulding machine (HAITIAN, HTF58X). The temperatures profile adopted during moulding were T_0 = 200 ˚C, T_1= 210 ˚C, T_2 = 200 ˚C and T_3 = 190 ˚C. The process took about 5 minutes to be completed.

Table 1 Formulation of PP/dolomite Composites

Sample designations	PP (wt%)	Dolomite (wt%)
PP	100	-
PP/D-5	95	5
PP/D-10	90	10
PP/D-15	85	15

2.3 LOI Test

The LOI test was done according to ASTM D2863. The apparatus held a small specimen of material which is clamped vertically in a tube known as the chimney in an atmosphere where the relative concentration of oxygen and nitrogen can be changed. The test was repeated by using various concentrations percentage of oxygen and nitrogen to determine the minimum oxygen concentration needed to burn the sample in three minutes. The flow rate of oxygen and nitrogen were referred from a reference table. The dimensions of sample were 6.5 mm x 72 mm x 3 mm which were cut from injection moulded plaques.

2.4 Differential Scanning Calorimetry

Samples weighing between 5 and 10 mg were placed in an aluminum pan and sealed. The test specimens were heated from 30 to 250 ˚C at a heating rate of 10 ˚C/min and the temperature was held for one minute at 250 ˚C. After cooling process from 250 to 30 ˚C at -10 ˚C/min, specimens were held for one minute at 30 ˚C. Then, heating from 30 to 250 ˚C at 10 ˚C/min was conducted. The equipment was operated in a nitrogen environment.

2.5 Flexural Test

Flexural test of composites was carried out using Llyod universal tester (Model 5533) according to ASTM D790 at a cross-head speed of 3 mm/min at room temperature (25 ± 2 ˚C). The distance between the spans was 100 mm. The mean value of at least five specimens for each formulation was then calculated.

2.6 Tensile Test

The dumbbell shape samples were tested using a Llyod Universal Testing Machine with a load cell of 20 kN according to ASTM D638. The cross-head speed used was 5 mm/min. At least seven samples were used for each formulation.

2.7 Impact Test

Impact test was done in accordance to ASTM D256 using Izod Pendulum Toyoseiki with the maximum energy of 11 J. Samples with the dimensions 63 mm x 13 mm x 3 mm were notched (1 mm) using Atlas Automatic Sample Notcher before the test.

2.8 Scanning Electron Microscopy

The surface morphology of composites was analyzed using SEM model Tm3000 Hitachi. The tensile test fractured surface samples were cut into smaller pieces and used as the samples. The specimens were mounted on aluminum stubs before being placed in the specimen chamber. The specimens were then coated with a layer of palladium and platinum which act as a conductor. The resulting image of the reflection electron radiation was used to scan the sample surface and taken at magnifications of 2000x and 5000x a voltage of 15kV.

3.0 RESULTS & DISCUSSIONS

3.1 Limiting Oxygen Index

The results of oxygen index testing on the PP/dolomite composites are listed in Table 2. Based on the data below, the percentage of oxygen index increased as the dolomite content was added to PP. As we all know, the percentage of oxygen in the air is 21% and the percentage of oxygen needed for the composites to burn showed that the composites are still in the range of flammable composite. The highest LOI value was 20% with the addition of 15 wt% dolomite. It shows that dolomite do help to increase the flame retardant properties of PP by taking a longer period for the polymer to completely burn. Other factors that affected these results are the filler scale and the aspect ratio of the fillers. From the observations, the composites were burning lightly at the beginning and vigorously in the middle of testing. Thus, the time taken for the composites to burn was much longer at the beginning of the testing.

Table 2 Data of LOI of PP with Various Dolomite Content

Dolomite Content (wt%)	Percentage of Oxygen Index (%)
0	17
5	18
10	19
15	20

Wagenknecht *et al.* [15] claimed that all inorganic fillers included those without endothermic effects, exhibit a flame retardant effect in polymer composites. In their studies, they found out that natural clay are much more effective at the same loading and the highest degree of the LOI instead of synthetic filler. Thus, they supported that dolomite mineral is one of the inorganic fillers that enhance the flame retardant properties to the reinforcing polymer.

3.2 Differential Scanning Calorimetry

The DSC analysis was conducted to identify the melting and crystallization point (T_m and T_c), of pure copolymer PP and PP filled dolomite. In order to ease the processability of PP/dolomite composites, it is important to know the T_m as well as the T_c. Table 3 shows the data of DSC at different dolomite content while Figures 1 and 2 show the thermogram for the crystallization and melting point, respectively. Based on Figure 1, T_c of PP increased with the addition of 5 wt% dolomite and remained constant at 123˚C when dolomite was added from 5 to 15 wt%. A significant increase of T_c was found with the addition of 5 wt% dolomite compared to pure PP. Based on Figure 2, the DSC scan of the PP resins that have been moulded shows that T_m had almost the same value with an insignificant change after the addition of dolomite. The maximum peak was 162 ˚C when dolomite content at 10 wt%. However, there is a slight reduction on the melting peak at 15 wt% dolomite from 162 ˚C to 161 ˚C.

Table 3 Data of DSC of PP with Various Dolomite Content

Dolomite Content (wt%)	Melting Temperature, T_m (˚C)	Crystallization Temperature, T_c (˚C)
0	158	109
5	159	123
10	162	123
15	161	123

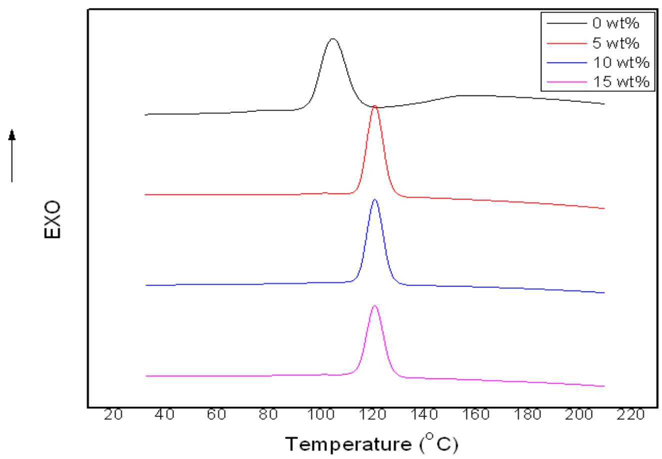

Figure 1 DSC Analysis Cooling Scans of PP and PP/Dolomite Composites

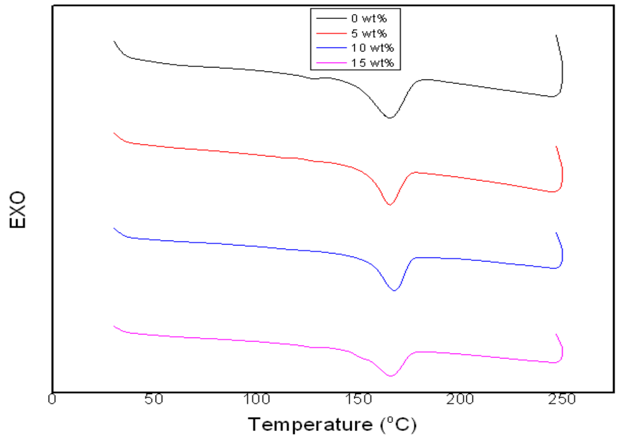

Figure 2 DSC Analysis Heating Scans of PP and PP/Dolomite Composites

3.3 Flexural Properties (Three point bending)

Figure 3 and 4 show the effect of dolomite content on the flexural modulus and strength of PP composites, respectively. Figure 3 shows the addition of 5 and 10 wt% dolomite increased the flexural modulus of PP in PP composites. The highest value was at 10 wt% dolomite, which has increased by 63.21% compared to neat PP.

The increment was due to the mobility of the PP chain was restricted by increasing the filler content, as indicated by the increase in flexural modulus. The stiffness of polymer matrices can be improved by the incorporation of rigid particulate filler as well as the interfacial contact which exist between the filler and matrix [16]. However, the flexural modulus was reduced after the addition of 15 wt% dolomite. Although it is expected that the flexural modulus to increase with increasing filler content, the agglomeration of fillers can cause the presence of voids to be present between the fillers and polymer matrix due to agglomeration of fillers at high filler content. It is believed to be the reason of reduction in flexural modulus with the addition of 15 wt% dolomite. This is consistent with the SEM image in Figure 9(c), where the presence of voids are seen and believed to be the reason for the reduction in flexural modulus with the addition of 15 wt% dolomite. However, it should be noted that such decrease in modulus was not observed in tensile mode. The plausible reason is the different mode of testing between tensile and flexural, whereby the void present is more detrimental for the polymer composite samples tested in flexural mode as compared to tensile mode.

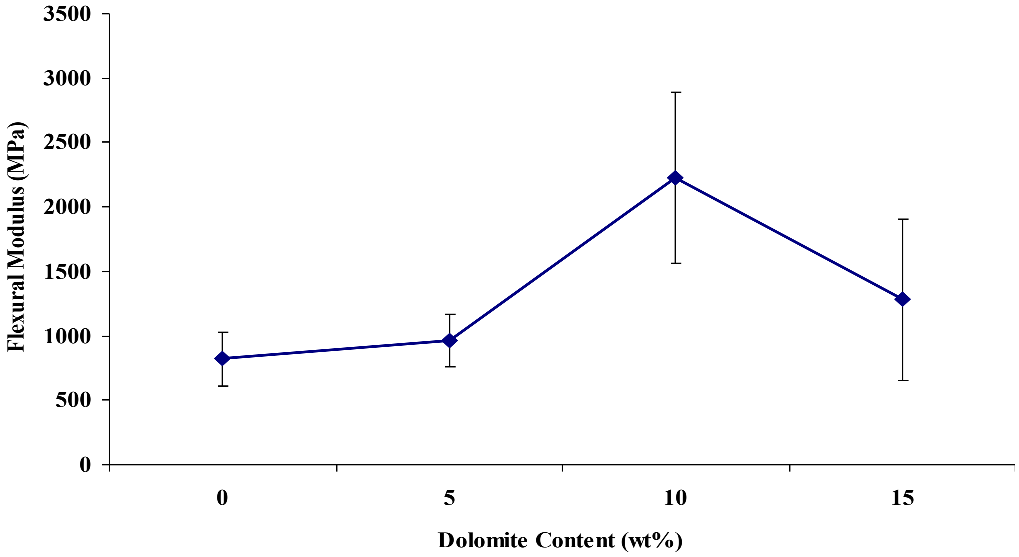

Figure 3 Flexural Modulus of PP and PP/Dolomite Composites

Figure 4 shows that the flexural strength decreased with the addition of dolomite. The range of flexural strength was from 25.10 MPa to 23.24 MPa. The flexural strength of PP was 25.10 MPa, then the value was reduced to 22.55 MPa with incorporation of 5 wt% dolomite. It is believed that the reduction in flexural strength was due to the poor adhesion of the composites as was mentioned earlier in the tensile strength properties. The absence of compatibilizer or surfactant in this study may be the reason, which resulted in poor flexural strength. This reason was supported by Zdiri *et al.* [3] where they studied the valorization of post-consumer PP by (un)modified tunision clay nanoparticles incorporation. They found out that surface treatment for clay is very important to improve the affinity between the clay particles and the matrix as well as to break down the large aggregates which will help to improve the interfacial adhesion of the filler with matrix.

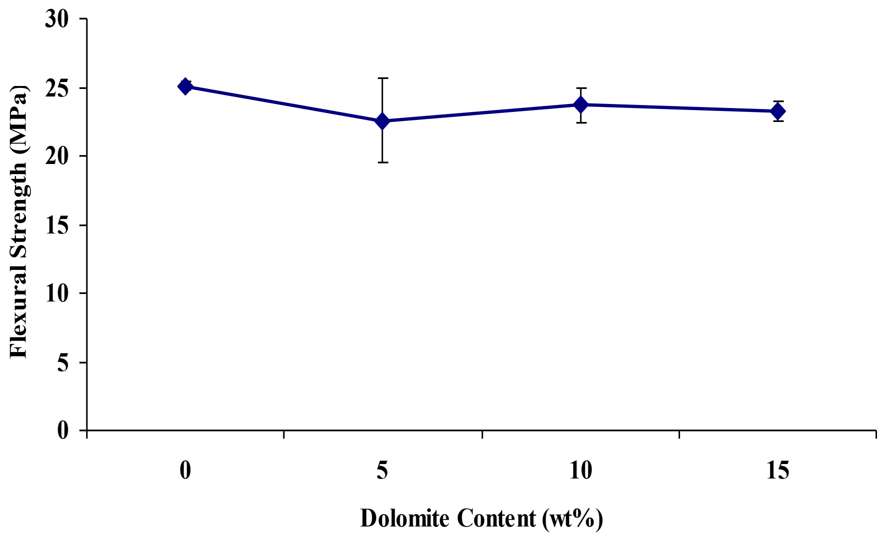

Figure 4 Flexural Strength of PP Composites

3.4 Tensile Properties

The change in tensile strength or referred as maximum stress of PP/dolomite composites were identified. The effect of dolomite content on the tensile strength of PP composites are illustrated in Figure 5. The range of tensile strength was from 19.55 MPa to 17.31 MPa. It was found that the tensile strength was slightly decreased with the initial incorporation of 5 wt% dolomite content. The tensile strength continuously reduced until the dolomite content was at its highest loading which is 15 wt%. However, the incorporation of dolomite does not much affected the tensile strength as it shows an insignificant changes with addition of 5 to 15 wt% dolomite. It is believed that the tensile strength of PP was slightly reduced after the addition of dolomite due to poor adhesion between dolomite and PP. Similar to Seki *et al.* [6] he reported that the tensile strength of polyester was decreased when the huntite content was added by 5 wt% and above into polyester matrix.

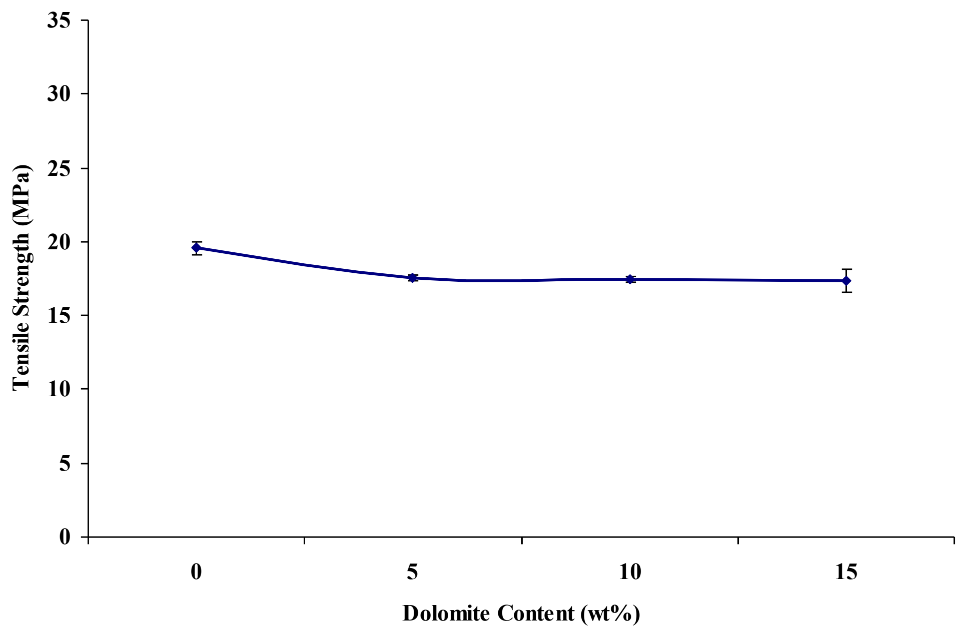

Figure 5 Tensile Strength of PP and PP/dolomite Composites

Furthermore, as shown in Figure 9(a), (b) and (c), SEM images of 5, 10 and 15 wt% dolomite contained voids proven that no adhesion between dolomite and PP occurred. Tait *et al.* [17] reported that the decrease in tensile strength indicated that either poor dispersion of the fillers, presence of agglomerates or existence of defects such as voids. Bishay *et al.* [18] also reported that the decrease of the tensile strength may be because of the fact that at higher filler contents (5 wt% and above), the interaction between fillers and polymer matrix was prevented, resulting in the lower strength of the respective composites. These are the factors, which possibly contribute to the reduction of tensile strength upon 5 wt% dolomite loadings.

The changes in Young's modulus have been identified for PP/dolomite composites as shown in Figure 6. It is found that the dolomite improved the Young's modulus of PP. Based on Figure 6, the modulus was slightly decreased from 645.24 MPa to 627.12 MPa after the incorporation of 5 wt% dolomite into PP. The percentage of reduction was 2.80%. The Young's modulus of PP continuously increased and it was the highest when dolomite content was 15 wt%. The increases in Young's modulus was expected because generally, the more mineral filler used, the greater the increase in Young's modulus due to the influence of rigid

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fillers on the stress-strain behavior of polymers under tensile loading. The improvement in Young's modulus proven that dolomite has an ability to stiffen the composites. Normally, a mineral filler produced a larger increase in modulus if it has a high aspect ratio (average equivalent diameter of the filler particle divided by its average thickness). Interestingly, even though the aspect ratio of dolomite is low (L/D = 3:1), it does help PP to improve its stiffness. This is also similar to Wiebking [19] studies that reported the precipitated $CaCO_3$ with a low aspect ratio increasing the modulus of polyvinyl chloride (PVC) compared to talc, which has a higher aspect ratio (L/D = 20:1).

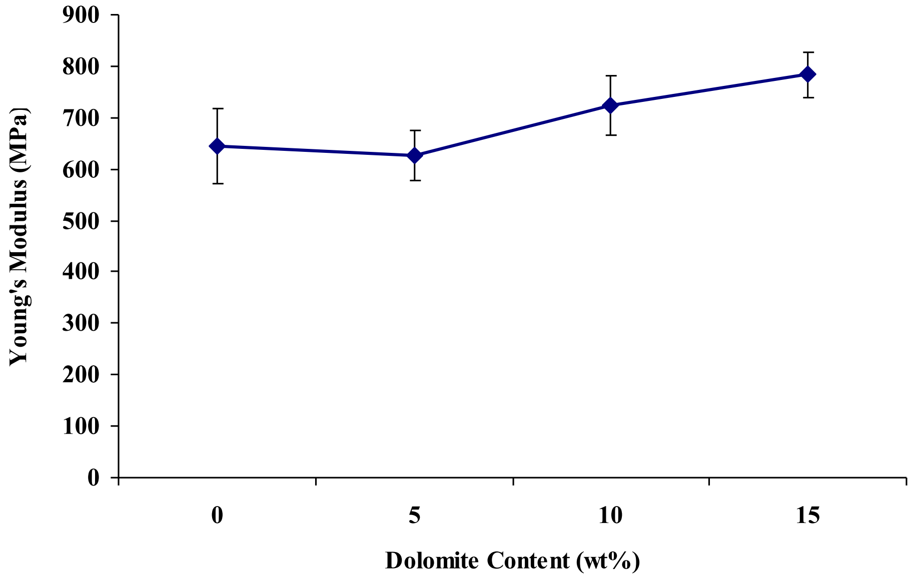

Figure 6 Young's Modulus of PP and PP/Dolomite Composites

The effect of dolomite content on percentage strain at break of PP and its composites is shown in Figure 7. It can be observed that the percentage strain at break increased as the dolomite content increased. Based on Figure 7, there is a significant change when the dolomite content increased from 0 to 10 wt%. The percentage strain at break of PP was the highest when dolomite content was 15 wt% with a 36% improvement. However, our result contradicts a previous report with regards to the effect of filler content on the percentage of strain. Seki *et al.* [6] studied on the effect of huntite mineral on mechanical, thermal and morphological properties of polyester matrix and the percentage at break of the composites was decreased as the filler content increased. However, in this study, this observation happened might be due to the presence of voids as shown in SEM images that caused the moisture to be trapped in the composites. It can be implied that the increasing of percentage strain at break was due to the moisture absorption by the samples before the characterization was done. The increased in percentage strain at break was due to the lubrication effect by the presence of moisture, which allowed the polymer chains to slip past each other. This is because hydrogen bonding between moisture and filler was formed in the composites and the dipole–dipole interaction between PP and fillers became less effective [20]. Saleh *et al.* [11] also reported that the micrometre-sized dolomite particles left vacancies to be wet by the resin in the composite system if dolomite is used as single filler. Thus, the vacancies may be also be wetted by the moisture along with the PP resins resulting in the increase of the percentage strain at break of the composites.

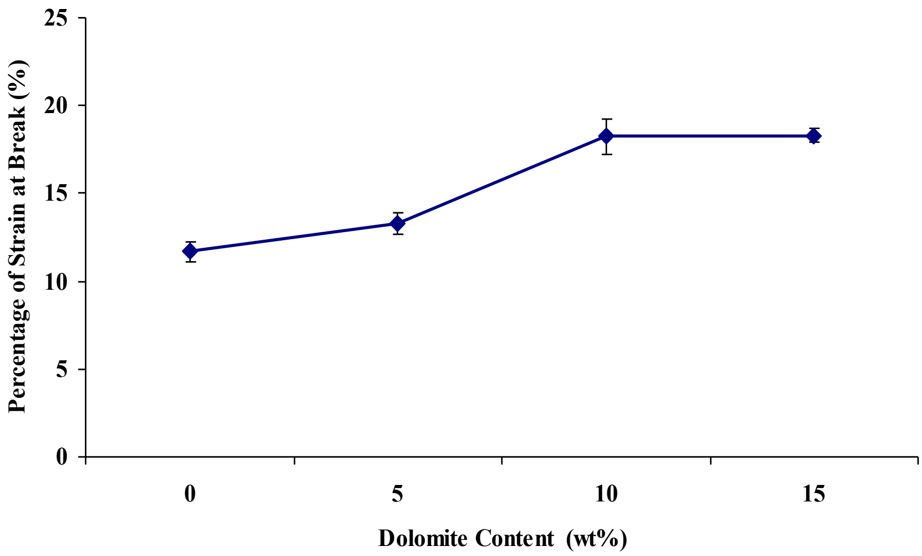

Figure 7 Percentage Strain at Break of PP and PP/Dolomite Composites

3.5 Impact Strength

The variation of impact strength with dolomite content is shown in Figure 8. It is evident that the impact strength of the composites continuously decreased as the dolomite content increased. The impact strength of pure PP was 103.87 J/m and reduced to 65.83 J/m with the addition of 5 wt% dolomite content indicated a significant change of 36.62%. The strength was continuously decreased until the dolomite content of 15 wt% with the lowest value which is 41.35 J/m. Thus, the total of failure was 60.19%. Karmarkar *et al.* [21] had reported the same trend. They suggested that sites for crack initiation were provided by the presence of dolomite-fillers in the PP matrix. Besides that, due to the stress concentration at the notch tip, the notched impact behavior was controlled to a greater extent by factors affecting the propagation of fracture that was initiated. It is also believed that the addition of dolomite that caused poor interfacial adhesion, thus the energy cannot be transferred effectively.

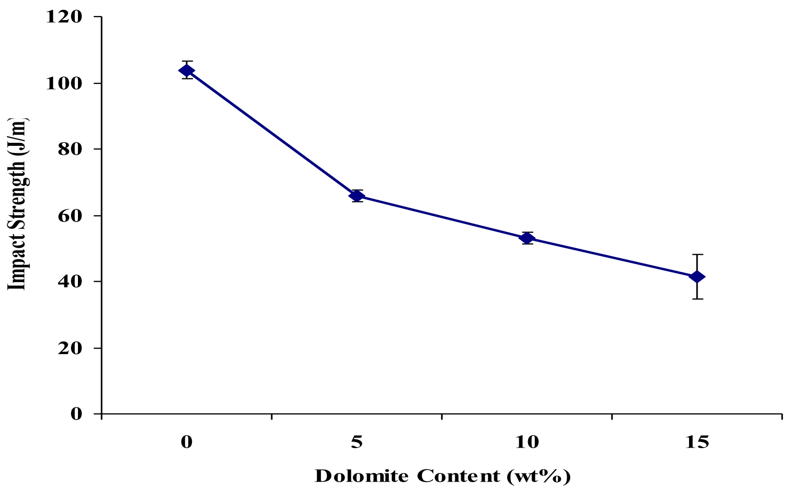

Figure 8 Impact Strength of PP and PP/Dolomite Composites

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3.6 Scanning Electron Microscope

Figure 9 illustrates the morphology of tensile fractured PP composites. It can be seen that voids were everywhere which showed the interfacial adhesion of PP/dolomite composites are poor. This is strongly caused by the absent of surfactant and surface treatment of dolomite. Surface treatment for filler is very important to improve the affinity between the filler particles and the matrix as well as to break down the large aggregates [22, 23]. This can be related to the role of surface treatment in reducing particle–particle attraction and increasing interlayer spacing. According to Razafimahefa *et al.* [24] the surfactant can open up the gallery distance of filler. Moreover, it makes the filler more hydrophobic and increases its compatibility with the matrix. Larger spaces between filler layers facilitate the penetration of polymer into filler particle.

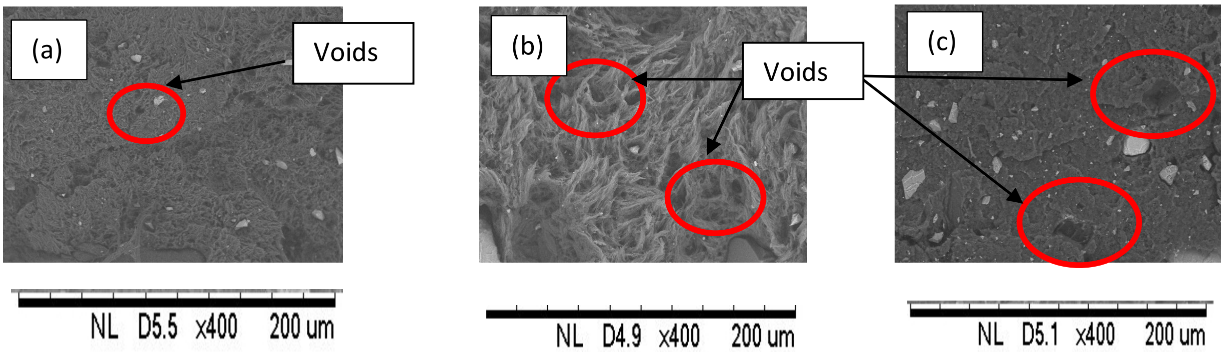

Figure 9 SEM Micrograph of PP Composites (a) PP/D-5 (b) PP/D-10 (c) PP/D-15

3.7 Optimum Composition of PP/Dolomite Composite

Figure 10 and 11 show the tensile strength and flexural modulus versus impact strength of the PP/dolomite composites. Based on the results collected, after the addition of dolomite, the tensile properties of pure PP was not much affected by the dolomite. Even though the tensile strength was slightly reduced after dolomite was added into PP, the flexural modulus experienced a continuous increment until dolomite content of 10 wt%. LOI analysis revealed that dolomite does help to decrease the flammability of PP by increasing the oxygen percentage from 17% to 20%.

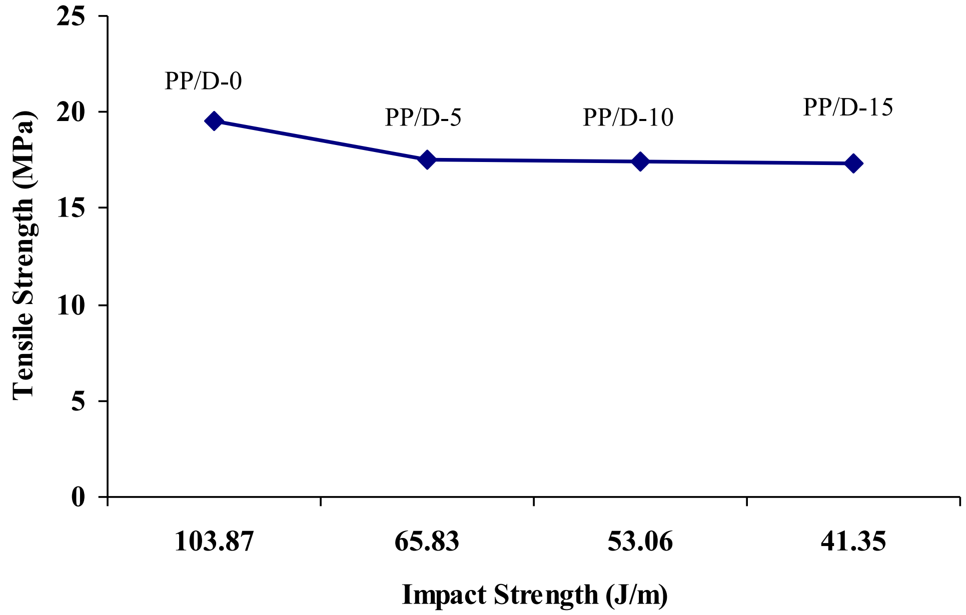

Figure 10 Tensile Strength Versus Impact Strength of PP/Dolomite Composites

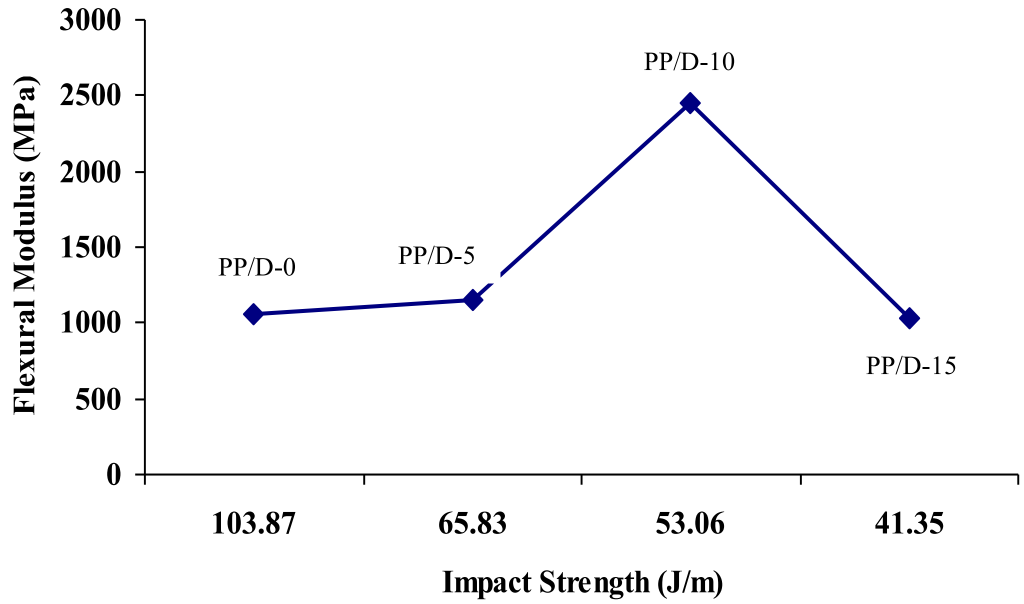

Figure 11 Flexural Modulus Versus Impact Strength of PP/Dolomite Composites

It was concluded based on flexural properties that PP/dolomite composite with 10 wt% dolomite was the optimum composition in this study. The mechanical properties of this formulation showed increased in modulus but slightly decreased in strength. Therefore, it is vital to find the suitable compatibilizer or surfactant to improve the adhesion between dolomite and PP. The flammability also decreased which makes the composite can be considered for automotive application.

4.0 CONCLUSIONS

PP/dolomite composites were prepared and it was found that tensile and flexural strengths were decreased with the incorporation of 5 wt% dolomite and remain unchanged with further addition of dolomite. Young's modulus was increased with increasing dolomite however flexural modulus was optimum at 10 wt% dolomite. Impact strength was decreased with increasing dolomite content. T_m was increased to 10 wt% of dolomite content and experienced a reduction after the addition of 15 wt% dolomite. While T_c was increased when 5 wt% dolomite was added to pure PP and remained constant with 10 wt% and 15 wt% dolomite content. As for the flammability properties, the increment of the percentage of oxygen index was directly proportional to the dolomite content. It can be concluded that dolomite do helps to enhance the flammability properties of pure PP. However, the composites are still in the range of flammable composite according to the percentage of oxygen index in the air. Based on the mechanical properties, it was concluded that the optimum formulation of PP/dolomite composite was with 10 wt% of dolomite content.

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