The effects of stearic acid treated dolomite (Sa-Dol) on the mechanical, morphological and thermal properties of polypropylene (PP) composite were studied. Prior treatment, raw dolomite was ground in planetary mill using different grinding time to obtain the submicron size (under 10 µm). Particle size and specific surface area of dolomite was analyzed and validated using particle size analyzer (Malvern Mastersizer) and Brunauer-Emmett-Teller (BET) method respectively. Dolomite with five filler loadings (5, 10, 15, 20 & 25%) were mixed with PP thermoplastic and were compounded using hot melt blending method. Fourier Transform Infrared (FTIR) spectroscopy confirms the successful of filler treatment using stearic acid according to the peak at 2917 cm⁻¹ attributed to the alkyl group that represented the addition of C-H bond. Tensile properties indicated that tensile strength of PP/Dol decreased with the increasing of filler loading for both systems. The elongation at break decreased with filler loading and showed better and improved result after treatment 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.%).

Keywords: Dolomite; Polypropylene; Stearic Acid Treated Filler; Submicron Particles
Surface treatment of dolomite

10g of dolomite was added into 100mL of distilled water and the suspension was stirred for 30 min at the temperature 50°C. 0.1g of stearic acid was dissolved into 10mL of 2-Propanol and the solution was poured into dolomite suspension, continuously stirred for 3 h at maintained temperature. The modified dolomite was centrifuged and dried in an oven at 80°C for 24 h to remove the excessive solution. The success of modification was confirmed by Fourier Transform Infrared (FTIR) analysis.

Preparation of the composites

The composites were prepared with different composition, consisting of filler loading between 5 wt% to 25 wt%. Polypropylene/dolomite were mixed using Brabender Plastograph® EC Plus fitted with mixer W 30 EHT at 180°C with rotors speed 60 rpm and the processing duration is 10 min. Polypropylene was first added into mixing chamber until constant torque is obtained then the dolomite powder was added into the chamber.

Compression moulding

One millimeter thickness dumbbell shape samples were prepared by molding process using electrically heated hydraulic press model GT 7014 A. Hot press procedures involved pre-heating at 180°C for 8 min, followed by compressing for 2 min at the same temperature.

Fourier Transform Infrared (FTIR) analysis

The spectrums of untreated and treated dolomite were collected in the 400 cm⁻¹ to 4000 cm⁻¹ region, 32 scans using Perkin Elmer Spectrum 100.
and sputter coated with a thin layer of palladium to avoid electrostatic charging during examination.

**Thermogravimetric Analysis (TGA)**

Thermo gravimetric analysis (TGA) of the samples of treated and untreated dolomite filled composites were carried out in a Q-250 thermal analyzer in nitrogen atmosphere at a heating rate of 10°C/min as per ASTM standard. This analysis was performed using Model Pyris Diamond TG/DTA.

**Results and discussions**

**Particle size analysis**

According to table 1, six different grinding time 0.5h, 1h, 2h, 5h, 10h and 20h have been used to study the effect of grinding time on the production of submicron particles of dolomite. The result shown that the particle sizes do not vary much from each other, so the optimum grinding time was chose at 2 h, due to small particle size obtained after grinding (8.183 µm) and also less energy is consumed in the grinding process for the production of the ground dolomite. Planetary mill had been chosen due to its capability in producing particle size less than 10µm as well as other filler requirements [12].

**Specific surface area analysis**

The particle size analysis result was supported by specific surface area using Brunauer Emmett Teller machine. Table 2 above shows that after 2 h grinding, the particle size reduced to submicron size (8.183µm) and there was an increment in specific surface area before and after grinding. 2.211 m²/g to 2.328m²/g respectively.

**Fourier Transform Infrared Spectroscopy (FTIR)**

Figure 1 shows the FTIR spectra of dolomite before and after treatment with stearic acid in a range of wavenumber 650 cm⁻¹ and 4000 cm⁻¹. The main difference of the spectra peak can be observed at peak 2917cm⁻¹ which was assigned to the C-H stretching vibration mode as per discussed by M. Fuji et. al [13]. The peak was not observed for the raw dolomite spectra; hence it was predicted to be the C-H stretching mode in the modifier. The molecular formula of stearic acid, CH₃(CH₂)₁₇COOH confirms that there is more C-H bonding in the modifier thus it showed that the modification process was successful.

**Tensile test**

Figure 2(a) showed the decreased of tensile strength with the increasing of filler loading for both composites due to poor interfacial adhesion between dolomite particles and polypropylene matrix. This will initiate micro-cracks that resulting in low tensile strength. The continuous decrease of tensile strength might cause by the inhomogeneous dispersion of filler particles due to the strong tendency of fine particles to agglomerate. Jilken [14] in the previous study explained that this might be due to insufficient amount of stearic acid that was used in the modification process. Figure 2(b) showed that the elongation at break decreased with the increasing of filler loading due to the brittleness of the composites. Dolomite forces PP matrix to deform more than the overall deformation of the composite due to the fact that deformation of filler is much less than of the filler matrix [15]. The elongation at break of treated dolomite was enhanced compared to the composite without treatment, this might due to the existence of stearic acid that only acted as plasticizer or as a lubricant in the composites. Fillers could slide over one another during stretching which resulted in extra extension and elongation. In figure 2(c) it can be observed that the Young's modulus increased with the increasing of filler loading. However no improvement can be seen after treatment of dolomite with stearic acid due to the insufficient amount of stearic acid as discussed in tensile strength result.

**Morphology of fractured surface**

Figure 3 shows the fracture surface morphology of (a) PP/Dol composites before treatment at (i) 5 wt.% , (ii) 25 wt.%, and (b) after treatment at (i) 5 wt.% , (ii) 25 wt.%. The detachment of dolomite particles from the PP matrix can be clearly observed in figure 3a(i) and a(ii). At higher filler loading, agglomerations and voids takes place in the matrix. This is resulted from the poor interfacial adhesion between filler and PP matrix. As been explain by Fu [16] the brittleness of composites will be increased with the increased of particulate filler loading if no or very little interfacial adhesion observed in the system. It contributed to the micro-cavitation which will lead to crack propagation. Figure 3b(i) shows a better and homogeneous dispersion of dolomite in the polypropylene matrix compared to figure 3 b(ii), where the agglomeration and inhomogenous dispersion of dolomite particles is very obvious. This might due to the insufficient amount of stearic acid used to treat the dolomite thus a very obvious agglomerated particles is observed in the fracture surface. This agglomerated result decreased in tensile strength and Young's modulus for treated dolomite at higher filler loading (25 wt.%), compared to lower filler loading (5 wt.%), and also when compared to the composite before treatment with stearic acid.

**Thermogravimetric analysis (TGA)**

Figure 4 shows the TGA curve as a function of both PP/ Dol and PP/Sa-Dol composites (5 & 25 wt.%). It can be observed from the graph that the weight loss percentage drop significantly at temperature around 470°C and 700°C-720°C. This is due to the decomposition of calcium magnesium carbonate (MgO) and calcium carbonate (CaCO₃) respectively. A better thermal stability is achieved for stearic acid treated dolomite for both composites than the composite without treatment, due to the reinforcing role of treated filler in the composites [17].

![Fig. 3. The fracture surface of PP/Dol composites (a) before treatment (i) 5 wt.%, (ii) 25 wt.% and (b) after treatment (i) 5 wt.%, (ii) 25 wt.%](image-url)
The reaction of dolomite decomposition is shown in equation below:

First peak: $\text{CaMg(CO}_3\text{)}_2 \rightarrow \text{CO}_3 + \text{MgO} + \text{CO}_2 \quad (1)$

Second peak: $\text{CaCO}_3 \leftrightarrow \text{CaO} + \text{CO}_2 \quad (2)$

From the TGA graph, the untreated dolomite composites (PP/Dol) is decomposed at 700°C with 93% of weight loss (5 wt.% filler loading) and 96% weight loss (25 wt.% filler loading), on the other hand the weight loss for PP/Sa-Dol after 720°C is around 74% (5 wt.% filler loading) and 76% (25 wt.% filler loading). This might be accounted for by a higher interaction of treated dolomite to the PP matrix [18, 19].

Conclusions

The grinding process for dolomite is proved to reduce the particle size of raw dolomite. This result is supported by particle size analysis and specific surface area analysis, Malvern Mastersizer and BET machine respectively. FTIR by particle size analysis and specific surface area analysis, the particle size of raw dolomite. This result is supported by the higher interaction of treated dolomite to the PP matrix [18, 19].

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References