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Mechanical, thermo-mechanical and morphological characterization of ABS based composites loaded with perlite mineral

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Abstract
Acrylonitrile-butadiene-styrene (ABS) copolymer was filled with perlite mineral (PER) at four different loading level of 2.5%, 5%, 10% and 15%. ABS/PER composites were produced using lab-scale micro-compounder followed by injection molding process. Mechanical, thermo-mechanical, melt-flow and morphological properties of composites were reported by tensile and impact tests, dynamic mechanical analysis (DMA), melt flow index (MFI) test and scanning electron microscopy (SEM), respectively. Mechanical characterizations revealed that tensile strength, elongation and Youngs’ modulus of ABS were improved by PER inclusions. However, impact strength of ABS reduced with increase of PER concentration. Glass transition temperature of ABS displayed increasing trend for %5 concentration of PER. MFI test implied that PER addition caused slight decreasing for MFI value of unfilled ABS. Homogeneous dispersion of PER particles into ABS matrix for their lower loading level was obtained from SEM micrographs of composites. According to findings, 5% PER containing sample exhibited the best performance and it was remarked as the most suitable candidate among fabricated ABS based composites.

1. Introduction

Acrylonitrile-butadiene-styrene (ABS) copolymer is composed of elastomeric polybutadiene segments which provides dimensional stability, toughness, and chemical resistance to the polymer. The polybutadiene is dispersed through styrene and acrylonitrile parts [1–3]. The advanced mechanical properties and easy processing characteristics are the reasons for usage of ABS as an engineering plastic. The most utilisations of ABS copolymer across a range of numerous products including toys, musical instruments, automotive components, protective helmets, telephone switchboard panels, sporting goods and medical devices [4, 5]. Due to the increase in its usage for 3D printing technology, the ABS became popular in recent years. The utilization of ABS in this technology is in filament form with various colors and dimensions. This is due to its proper rheological features [5, 6].

Minerals are widely used as additive for polymeric materials since they have advantages including low cost and easy processing [7, 8]. Perlite is a natural clay mineral which is a form of amorphous volcanic silica glass having considerable water content. This aluminosilicate has ability to expand 30 times its initial volume as it subjected to heat. Turkey has the largest perlite abundance which is more than half of the world. Greece, USA, Mexico, Hungary, Italy, Iran and Japan are the other countries have significant perlite reserves [9–11]. Perlite mineral found effective usage on several areas including insulation products, light-weight construction parts, paints, tiles, cement and gypsum plasters, mortar, pharmaceuticals, dental compounds, cleansers, soaps soil and water filtration applications [12]. Perlite is also used as low-cost additive for plastics [7].

Perlite reinforced ABS based composites has not been found in the literature. However, there have been several academic studies performed by researchers dealing with perlite filled polymer composites. Arsalani et al
prepared perlite/polyaniline composites and they observed that electrical conductivity of composites drops down with increase in particle size of perlite [13, 14]. Oktem and Tincer prepared PER containing high density polyethylene (HDPE) composites and they reported that silane coupling agent was very effective to observe improvement on mechanical, flow and thermal properties of PER/HDPE composites [15–17]. Atagur et al studied the thermal conductivity of HDPE/PER composites [18]. Sahraeian et al performed the production of low density polyethylene (LDPE) based composites melt blended with perlite. They postulated that increases in mechanical, morphological, dynamic mechanical and thermal properties were observed after surface treatment of PER particles [19–21]. Several research groups conducted works dealing with PER filled polypropylene (PP) composites. Mattausch et al examined the effects of processing conditions of PER/PP composites such as screw geometry and they found that homogeneous dispersion was achieved with high shear processing [22]. Spoerk et al investigated that smaller size of PER may led to increase of mechanical properties of perlite containing PP composites due to shrinkage reduction [23]. Mattausch H. et al blended expandable graphite/PP composites with mineral fillers including perlite and they showed that PER can be used as flame retardant additive with combination of other fillers [24]. Perlite was also used on research studies dealing with polyurethane foam composites. In these works, thermal insulation and sound absorbing performance of PER containing polyurethane foams [25, 26]. Mahkam et al examined the drug delivery behavior of PER loaded poly (methacrylic acid) composites. They demonstrated that using PER with coupling agent caused reduction on drug release rate [27]. Akkaya studied the PER filled poly (hydroxyethyl methacrylate) composites and he proved that these composites can be cost effective alternative adsorbent for the recovery radiotoxic compounds [28]. Tian and Tagaya investigated that PER addition caused increases in mechanical and thermal properties as well as photochemical stability of poly (vinyl alcohol) composites [29, 30]. Zhang X. et al exhibited that thermal conductivity of polyethylene glycol was improved with the addition of PER [31]. Rattanaplome et al showed that PER had a potential of odor adsorbing additive for natural rubber products [32].

According to the literature survey, this work is the first research study regarding the melt-flow performance of ABS based composites in order to evaluate their 3D printing applications and processability. Composites were characterized by tensile, impact and shore hardness tests, dynamic mechanical analysis (DMA), melt flow index (MFI) test and scanning electron microscopy (SEM) techniques.

### Table 1. Compounding and injection parameters of composites [33].

| Parameters        | Specification | Unit |
|-------------------|---------------|------|
| Processing temperature | 230           | °C   |
| Mixing time       | 5             | min  |
| Screw speed       | 100           | rpm  |
| Barrel temperature| 240           | °C   |
| Mold temperature  | 50            | °C   |
| Injection pressure| 8             | bar  |
| Holding time      | 3             | min  |

2. **Experimental**

#### 2.1. Materials

ABS copolymer used in this study was purchased from Lanxess, Germany under a trade name of ABS M203FC. Expanded perlite was supplied from Eti Maden, Izmir, Turkey. It has bulk density and average particle size of 300–1000 kg m$^{-3}$ and 39.6 μm, respectively.

#### 2.2. Fabrication of composites

ABS and PER were dried under vacuum at 80 °C for 12 h to remove moisture content before compounding. Composites were produced using co-rotating twin screw extruder (Micro-compounder, 15 ml, DSM Xplore, Netherlands). Filling ratios of PER in ABS matrix were 2.5, 5, 10 and 15 wt%. The test specimens in dog-bone shaped with dimensions of 7.6 × 2.0 × 80 mm$^3$ were prepared by injection molding instrument (Micro-injector, Daca Instruments, UK). The gauge length of specimens obtained by injection molding was 50 mm. The compounding and injection parameters applied for composite productions are listed in Table 1.

#### 2.3. Characterization techniques

The Lloyd LR 30 K universal tensile testing machine was used in order to investigate tensile properties of ABS and ABS/PER composites. The tests were done in accordance with ASTM D–638 standard. The load cell capacity
was 5 kN and the crosshead speed was 5 cm min\(^{-1}\) throughout the tests. In order to calculations of the tensile strength, elongation at break (\(\%\)), and tensile modulus were recorded the averages of five samples for each measurement. The unnotched izod impact strength values of ABS and ABS/P Encations based on ASTM D256 standard were investigated by Coesfeld-Material impact tester with the pendulum of 4 J. At least five specimens with standard deviations were used for the calculations of impact strengths of the samples. The temperature dependent mechanical behaviours of ABS and ABS/P Encations were examined by DMA 8000, Perkin Elmer dynamic mechanical thermal analyzer. During the tests the frequency (1 Hz) was kept constant and tests were carried out in dual cantilever bending mode. The heating ramp was 10 °C min\(^{-1}\) throughout the adjusted temperature interval (−120 °C to 140 °C). Melt flow index studies were done under specific load of 2.16 kg at the constant temperature of 230 °C using Coesfeld MeltMixer LT. MFI values for each samples were calculated as the averages of at least ten samples with standard deviations. The scanning electron microscopy with field emission via JSM-6400 Electron Microscope was utilized for the characterizations of the composites in morphological manner. Micrographs were obtained with varied magnifications from \(\times 1,000\) to \(\times 10,000\) after coating of surfaces of fractured samples obtained from impact tests. X-ray diffraction analysis (XRD) was conducted using X’Pert Pro, PAN analytical with Ni-filtered CuKα radiation (\(\lambda = 0.154 \text{ nm}\)) at 45 kV and 40 mA.

3. Results and discussion

3.1. Mechanical characterization

The tensile test data of ABS and ABS/P Encations and the corresponding curves are given in table 2 and figure 1, respectively. As shown in table 2, the addition of PER caused significant increase in tensile strength of ABS up to 5% PER concentration. However, further additions of PER led to decrease in tensile strength values of composites. The void formations nonhomogeneous dispersion of PER particles into ABS phase may be the reason for this behavior. The possible agglomerations can cause stress concentrations and they can lead mechanical failure [34, 35]. Elongation at break value of unfilled ABS increased with the inclusion of PER. The highest elongation value was observed for 2.5% PER containing composite. According to table 2, Youngs’ modulus of ABS exhibited increasing trend with the PER incorporated at higher filling ratios.

Impact test is a high strain rate test that determines the amount of energy absorbed by a material during fracture. This absorbed energy is a measure of the toughness of a material and used to study the temperature dependent ductile or brittle transition. Impact test results of ABS and ABS/P Encations are illustrated in figure 2. Inclusion of PER caused gradual reduction for impact strength of neat ABS. 2.5 and 5% PER loaded

![Figure 1. Stress-strain curves of ABS and composites [33].](image_url)

### Table 2. Tensile test data of ABS and composites [33]

| Samples       | Tensile strength (MPa) | Elongation at break (%) | Youngs’ modulus (GPa) |
|---------------|------------------------|-------------------------|-----------------------|
| ABS           | 35.4 ± 1.7             | 7.1 ± 0.8               | 0.92 ± 0.3            |
| ABS/2.5 PER   | 38.1 ± 1.5             | 10.2 ± 0.9              | 0.90 ± 0.1            |
| ABS/5 PER     | 41.0 ± 1.6             | 9.6 ± 0.7               | 0.92 ± 0.2            |
| ABS/10 PER    | 39.1 ± 1.8             | 9.8 ± 0.5               | 0.99 ± 0.4            |
| ABS/15 PER    | 37.9 ± 1.2             | 7.3 ± 1.0               | 1.00 ± 0.3            |
composites showed relatively higher impact strength than ABS/10 PER and ABS/15 PER samples. This finding may be explained by the formation of stress failures between PER particles and ABS matrix because of restriction for deformation ability of ABS after PER inclusion which promotes reduction for the total free energy of the composite [36–38].

3.2. DMA study
The changes of mechanical history of the material in a corresponding temperature range is examined by thermo-mechanical characterization. DMA test was utilized in order to investigate the damping properties of ABS and ABS/PET composites. Storage modulus and Tan δ curves as a function of temperature were given in figures 3 and 4, respectively. Storage modulus of ABS decreased steadily up to $-80 ^\circ C$ and then the minimum value reached at about $108 ^\circ C$ which indicate the glass transition temperature ($T_g$) of polybutadiene and poly(styrene-
co-acrylonitrile) portions of ABS, respectively [39, 40]. According to figure 3, PER additions caused decrease for storage modulus of unfilled ABS. 15% PER containing composite yield nearly identical storage modulus value with that of ABS due to restriction effect of PER particles on segmental mobility of ABS chains [41–43].

Tan δ is defined as damping factor which indicates the compatibility and interfacial adhesion between two phases of composite [44–46]. Tan δ curves of ABS and composites in figure 4 displayed that one point higher T_g value was achieved for ABS/5 PER composite compared to T_g of unfilled ABS. However, the height of Tan δ peaks which is an indicator for the bonding quality was found as nearly identical for all of the samples. ABS/5 PER composite gave slightly higher peak value ascribed to best bonding quality was obtained for that sample among composites.

3.3. Melt-flow measurements
MFI values of polymeric materials are characteristic parameters which indicates information based on the viscosity changes of polymer melt and processability of plastics. As represented in figure 5, slight decreasing for
MFI value of ABS was observed with the additions of PER. MFI reduced as the concentration of PER increased. However, these decreasing trend was obtained in the narrow range which means that PER inclusion caused no serious processing problems for ABS based composites.

3.4. Morphological investigations
In order to investigate the morphological properties of ABS/PER composites, SEM photographs of samples were examined. Perlite particles into ABS phase were indicated in figures 6 and 7. SEM photographs in figure 6 implied that PER display uniform distribution into ABS matrix for the lowest adding ratio (2.5%).
Homogeneous dispersion of PER particles for their 5% concentration can also observed from the figure 6. Dispersion homogeneity started to disappear with the 10% addition of PER. ABS/15 PER composite showed completely nonhomogeneous distribution. According to SEM photographs with higher magnifications in figure 7, large gaps between PER phase and ABS matrix indicated their poor interactions. Weak adhesion of ABS to PER surface caused agglomeration formations for higher additions of PER into ABS matrix can be seen in figure 7 which proves the previous findings in the case of mechanical performance of composites.

XRD patterns of perlite, unfilled ABS and prepared composites displayed in figure 8. The characteristic peak of PER can be seen as more intense with the increase in PER content of composites. Improvement of this PER related peak in XRD patterns composites may be attributed to the formations of chemical bonding between PER particles and ABS phase.

4. Conclusion

This work postulated the mechanical, melt-flow and morphological behavior of perlite reinforced ABS composites. Mechanical tests showed that tensile strength of ABS/PER composites increased with 2.5% and 5% PER inclusions. Further additions caused decreasing behavior for tensile strength values. Impact strength of the ABS exhibited reductions by the additions of PER. DMA study implied that glass transition temperature of ABS shifted to one point higher after the inclusion of 5 wt% of PER. According to MFI test results, PER loaded composites displayed lower MFI values relative to unfilled ABS. However, these reductions on MFI values were obtained as negligible to effect the processability of composites. According to morphological characterization with the help of SEM analysis, homogenous distributions of PER particles in ABS matrix for their lower filling ratios (2.5% and 5%) were observed. Presence of weak interfacial interactions revealed by SEM micrographs support the mechanical investigations discussed previously in which mechanical failure of the composites containing higher loading level of PER. These findings showed that the highest results were obtained for ABS/5 PER sample among composites in the case of mechanical, thermo-mechanical and morphological behaviors which indicated that the most suitable candidate for the applications of perlite containing ABS based composites.

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