Mechanical Properties of Acorn and Pine Cone Filled Polymer Composites

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Abstract

In this work, the composites consisted of epoxy resin reinforced with acorn and pine cone fillers/powders were investigated. Both hardness and tensile properties of composites were evaluated dependent on filler type and content. The waste fillers were added into the polymer with three different ratios (10\%, 20\% and 30\% weight percent) and manufactured with casting method. Based on the quasi-static tensile tests and hardness measurements, the pine cone based composites showed slightly better results compared to the acorn reinforced samples. In addition to that the composites independent of filler type showed higher tensile and hardness properties with increasing the particle content.

Keywords: particulate composites; natural waste fillers; tensile and hardness properties

1. Introduction

Recently, there has been a growing interest for the fabrication of natural fiber/particle reinforced polymer composites which have great offerings to various application areas such as packaging, construction, furniture, electronics and automotive industries. For strengthening the plastic matrices, the utilization of natural fillers instead of traditional reinforcement materials provides benefits such as light weight, easy processing, lower cost and lesser environmental pollution \[1-3\]. The increase for the availability of new generation bio-based composite structures leads to the decrease of petroleum based toxic products usage. Coconut fiber, kenaf fiber, oil palm fiber, pistachio shell, peanut shell, walnut shell and rice husk were combined with thermoset or thermoplastic matrices for comprising composites \[4-5\]. In the present study, two filler types with three different weight ratios were considered. Acorn and pine cone powders were combined with epoxy resin with 10\%, 20\% and 30\% weight percent.

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Tensile and hardness properties were evaluated and compared with neat epoxy. Based on the test results both of the natural fillers showed similar effects and the increase of filler content led to the increase of rigidities and hardness of composites.

2. Experimental

2.1. Materials

Thermoset based epoxy resin (Super Selva™, Turkey) was used as the matrix component in composite samples. Acorn and pine cone products (Figure 1-a and 1-b) were obtained from the Celal Bayar University Campus. Initially they were washed with deionized water and dried at 70°C in an oven for 24 hours. The grinding process was carried out via RETSCH™ pulverizer at 1000 Rpm for 5 minutes and the produced powders were given in Figure 1-c.

![Figure 1: (a) As-received pine cone products, (b) as-received acorn products, (c) powder form of acorn and pine cone waste fillers](image)

2.2. Density Measurement of Natural Waste Fillers

In this work, the densities of the acorn and pine cone fillers were calculated by considering the volume \(V\) and mass \(m\) values of the circular specimens. The fillers were compacted via a hydraulic press as shown in Figure 2. In order to know the apparent density \(\rho_a\) of various natural powders the Eq.(1) was used as shown below.
2.3. Preparation of Natural Filler Composites

Composite tensile test specimens were prepared at 10, 20 and 30% wt. of powders. A digital disperser (Ultra-Turrax T 25, IKA™, Germany) was used to mix the epoxy resin and powders for obtaining a homogenous particle distribution (Figure 3). The suspensions including acorn and pine cone particles were mixed by hand and then using the disperser at 3500 Rpm for 4 minutes. To eliminate the gas bubble formation, the prepared composite mixtures were left under vacuum. After hardener addition, the solution was gently mixed and poured into the tensile test sample silicon molds. The samples were cured at room temperature for 24 hours and subjected to post curing at 110°C for 5 hours [6].

2.4. Testing Methods

In the present study, tension and hardness tests were carried out to understand the basic mechanical responses of natural filler based composites. The quasi-static tension test of composite materials and neat epoxy were performed by considering ASTM D638M-91a standard (Figure 4). The tests were conducted in an SCHIMADZU™ mechanical testing machine at a cross-head displacement rate of 1 mm/min. The prepared composite samples are shown in Figure 5. A video extensometer connected to the test machine was used to measure the elongation of the samples. Three specimens for each weight ratio were produced and the tensile parameters such as modulus and strength values were reported as the average values of these tests.

Brinell hardness measurements of the specimens were evaluated by using a macro hardness tester (Figure 6). In the beginning of this test the composite samples were located on a flat surface. The tester was maintained in the perpendicular direction and pressed towards the sample surface. Four different points for each composite were recorded and average values of them were reported.
Figure 3: Mechanical dispenser

Figure 4: Technical illustration of tensile test specimen

Figure 5: Prepared natural waste filler composites (a) acorn (A.C.) based samples, (b) pine cone (P.C.) based samples

Figure 6: Macro hardness tester
3. Results and Discussions

The apparent density measurement results of pine cone and acorn fillers were tabulated in Table 3.1. Based on that table, it is clear that the acorn filler was denser and pine cone specimens were approximately 10% lighter than pine cone powder based samples.

Table 3.1: Apparent densities of waste fillers

<table>
<thead>
<tr>
<th>Apparent Density (kg/m$^3$)</th>
<th>Acorn Filler</th>
<th>Pine Cone Filler</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1289</td>
<td>1180</td>
</tr>
</tbody>
</table>

The representative stress-strain graphs of acorn and pine cone based composites are shown in Figure 7 and 8, respectively. Based on the graphs it is obvious that the neat epoxy showed ductile type behavior compared to the particulate composites. Although the addition of 10% wt. acorn filler into the epoxy significantly decreased the stress and strain values, the ductile deformation maintained. However, the composites including 20% and 30% wt. acorn particles exhibited abrupt load drops and distinctive brittle character (Figure 7). In Figure 8, similar type of trend was observed for the pine cone based composite structures. The brittle deformation was seen for all of the samples independent of particle content. In addition to that the calculated stress-strain values are higher compared to the acorn based composites. The average elastic modulus ($E$) and tensile strength ($\sigma_{\text{max}}$) parameters are given in Figure 9 and 10, respectively. Neat epoxy showed the modulus and strength values as 1.21±0.24 GPa and 25.24±3.39 MPa, respectively. Based on Figure 9, increase of filler content from 10% to 30% led to the increase of elastic modulus (rigidity) independent of filler type, as expected. The composite including 30% wt. acorn particles exhibited a significant jump in terms of modulus and possessed the highest value (1.33±0.35 GPa) among the other composites and neat epoxy. A more stable rigidity increase is observed for the pine cone based samples and the maximum value was calculated as 1.29 ±0.56 GPa.

![Stress-strain graph of acorn powder based polymer composite](image)
In Figure 10, strength parameters of composites are compared with neat epoxy. The acorn powder reinforced polymer composites displayed lower strength values compared to the pine cone based samples as seen in the same figure. The highest tensile strength was found as 21.83±5.2 MPa for 30% wt. pine cone powder filled specimens.

The increase of filler content generally leads to the extension of interfacial area. However, due to insufficient interfacial bonding between hydrophobic polymer and hydrophilic fillers, the strengthening effects of particulates become worse. Furthermore, the shape and size distributions of fillers remarkably influence the mechanical properties of composites [7]. Both of these reasons explained above are observed for the composite structures investigated in this study. The filler presence caused to the decrease of tensile parameters up to 20% wt. loaded samples. After 30% wt. particle introduction, the mentioned features started to increase prominently. Particularly, the pine cone based specimens showed better performance as compared to acorn based composites.

Hardness is also a critical parameter to evaluate the response of particulate reinforced composites. In the present work, Brinell hardness (HB) values of the samples are measured and tabulated in Table 3.2. Representative hardness measurement photos are also given in Figure 11.

Diameter of the tungsten carbide ball used in this test was 2.5 mm and the applied load during the measurement was determined as 6.25 kg for all of the samples. Based on Table 3.2, the filler addition led to the increase of hardness independent of filler type as expected. The maximum value was found as 10.65±0.54 HB for 30% wt. pine cone filled composite while minimum hardness was observed for the neat epoxy as below 2.4 HB. Among the acorn filler based specimens, the highest hardness was calculated as 8.47±0.36 HB for 30% wt. powder content which was approximately same with 20% wt. pine cone based composite. As seen from the same table, for the same particle concentrations the pine cone reinforced composites exhibited higher values compared to acorn based specimens.
Figure 9: Effects of different filler contents on the elastic modulus values of composites

Figure 10: Effects of different filler contents on the strength values of composites
Table 3.2: Brinell hardness values of neat epoxy and natural filler based composites

<table>
<thead>
<tr>
<th>Material Type</th>
<th>% wt. Filler Content</th>
<th>Brinell Hardness (HB) Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Neat Epoxy</td>
<td>0</td>
<td>&lt;2.4</td>
</tr>
<tr>
<td>Acorn based composite</td>
<td>10</td>
<td>2.52±0.19</td>
</tr>
<tr>
<td></td>
<td>20</td>
<td>4.55±0.52</td>
</tr>
<tr>
<td></td>
<td>30</td>
<td>8.47±0.36</td>
</tr>
<tr>
<td>Pine Cone based composite</td>
<td>10</td>
<td>7.50±0.27</td>
</tr>
<tr>
<td></td>
<td>20</td>
<td>8.17±0.58</td>
</tr>
<tr>
<td></td>
<td>30</td>
<td>10.65±0.54</td>
</tr>
</tbody>
</table>

Figure 11: Representative photos of natural filler based composites during hardness test (a) 20% wt. pine cone filled composite, (b) 20% wt. acorn filled composite

The microstructures of neat epoxy, 10% wt. acorn based and 10% wt. pine cone based composites are seen in Figure 12. In this figure, the darker regions with irregular shapes represented the fillers and the lighter parts corresponded to neat epoxy. Based on those pictures, it is clear that the acorn filled samples showed more porous structures and the lower mechanical properties of the samples may also be attributed to this morphological statement.

4. Conclusions

In the present study, acorn and pine cone powder fillers were mixed with epoxy resin to manufacture particulate reinforced polymer matrix. Based on the test results, the 10% and 20% wt. powder addition decreased the tensile parameters and the composites with 30% wt. filler content showed the maximum values independent of filler type. Neat epoxy exhibited quite low hardness values so the natural waste filler introduction led to the increase of this parameter. According to the hardness measurements and tension test results, the pine cone based
composites generally exhibited better performance as compared to acorn filled samples and these properties can be improved by modifying the surfaces of natural waste fillers.

![Microstructural images of (a) 10% wt. pine cone filled composite, (b) 10% wt. acorn filled composite and (c) neat epoxy](image)

**Figure 12:** Microstructural images of (a) 10% wt. pine cone filled composite, (b) 10% wt. acorn filled composite and (c) neat epoxy

**References**


