

# The Effect of Alkali Treatment and Filler Size on the Properties of Sawdust/UPR Composites Based on Recycled PET Wastes

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**ABSTRACT:** In this study, natural sawdust fillers from *acacia* were mixed with unsaturated polyester resin (UPR), which was prepared by recycling of polyethylene terephthalate (PET) waste bottles to prepare sawdust/UPR composite. PET wastes were recycled through glycolysis and depolymerized to produce a formulation for the resin. The effects of alkali treatment, filler content, and filler size on the tensile, flexural, hardness, and water absorption of the composites were investigated. The results show that the modulus of both tensile and flexural increased with increasing filler contents, but the tensile and flexural strength of composites decreased. The size of sawdust also

played a significant role in the mechanical properties, with smaller size sawdust producing higher strength and modulus. This is due to the greater surface area for filler-matrix interaction. The results also show that alkali treatment causes a better adhesion between sawdust and UPR matrix and improves the mechanical properties of the composites. Furthermore, surface treatment reduced the water absorption of composites. © 2008 Wiley Periodicals, Inc. *J Appl Polym Sci* 109: 3651–3658, 2008

**Key words:** PET recycling; composite; UPR; natural filler; alkali treatment

## INTRODUCTION

Polyethylene terephthalate (PET) is one of the versatile plastics, which is widely used in the manufacture of various kinds of packaging, especially for soft drink bottles. The overall annual world consumption of PET amounts to about 13 million tons.<sup>1</sup> With such a large consumption, the effective use of PET waste is a significant subject for environmental protection researchers. Although several methods have been proposed for recycling PET wastes, it was found that the most attractive method is chemical glycolysis into the corresponding monomers and dimers that could be reused for the production of plastics or other advanced materials.<sup>2</sup>

The concept of preparing unsaturated polyester resins (UPR) from digested products of PET is well developed. The glycolysis of PET bottles with glycols and a transesterification catalyst yields terephthalic

oligomers. The oligomers may then be reacted with maleic anhydride to form UPRs. UPR is of interest as a candidate because it is one of the most widely used thermoset resins as a matrix for polymeric composite. One of the applications of unsaturated polyester is in sheet molding compounds. The use of PET waste is both inexpensive and effective in incorporating terephthalic functionality into the backbone of polyester resin.<sup>3</sup>

Although a small number of studies have been carried out to utilize the glycolyzed product of PET into fiber-reinforced composites using glass fibers,<sup>4</sup> no report on using natural fiber or filler was found. These fillers are not only inexpensive but also able to minimize the environmental pollution. Biodegradable lignocellulosic fillers possess several advantages compared to inorganic fillers, such as lower density, greater deformability, smaller abrasiveness, and lower cost.<sup>5</sup> Many researches have been done on natural fillers using commercial polyester. It has been shown that the waxy substances are the cause of poor filler wettability and adhesion characteristics, but they can be eliminated by extraction with organic solvents.<sup>6</sup> Different kinds of surface modifications were used to improve natural fibers/fillers with thermoset resin matrix interactions by different researchers.<sup>7–9</sup> Sharifah and Ansell used alkali treatment for hemp and kenaf fibers/polyester

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composites. They reported that the flexural properties improved for treated fibers.<sup>10</sup> Investigations on flax fiber/epoxy matrix composites by Van et al. also proved that alkalisation is a simple and effective method to enhance the fiber/matrix interaction.<sup>11</sup> Thus, this study aims at using *acacia* sawdust with and without the alkali treatment in the production of UPR composites based on recycled PET. In this study, the focus is also on the effect of filler content and filler size on the mechanical properties of *acacia*/UPR composites from PET bottles.

## MATERIALS AND METHODS

### Materials

Ethylene glycol and zinc acetate were purchased from Ajax Chemicals Ltd. and Riedel-de haën, respectively. All reagents were used without further purification. Glycolized product, maleic anhydride (Aldrich Co.), hydroquinone (Merck), and styrene monomer (Fluka) were used for the synthesis of resin. Methyl ethyl ketone peroxide (MEKP; catalyst) and cobalt octoate (accelerator) from the Aldrich Co were used to cure the resin. *Acacia* fillers with density of 1.4 g/cm<sup>3</sup> were obtained from Forest Research Institute of Malaysia. The fillers were dried in an oven at 110°C for 8 h, and then sieved to a size of 250–350 and <100 µm.

### Glycolysis of PET soft drink bottles

Postconsumer PET soft drink bottles (washed, dried, ground) were used for glycolysis. Ethylene glycol was added to PET wastes in a four-necked round-bottom flask of 1000 mL capacity with the ratio of PET : EG 1 : 4 (w/w) with 0.5% zinc acetate based on weight of PET as catalyst. The reaction was carried out under reflux in nitrogen atmosphere at 190–200°C for 5 h. The contents in the reactor were allowed to cool down to room temperature.

### Preparation of UPR

UPR was prepared by reaction of the glycolysed product with maleic anhydride at a fixed molar ratio of 1 : 1. The polyesterification was carried out in a 1000 mL four-necked round bottom flask connected to a condenser and N<sub>2</sub> gas inlet. The reactants were heated from room temperature to 180°C in about 1.5 h, then held at 180°C for 3 h and finally raised to 200°C and maintained until the acid value reached 20–30 mg of KOH/g. The liquid resin was then cooled to 120°C, and hydroquinone was added in 0.45% of PET weight to prevent premature gelation. Finally, the liquid resin was dissolved in styrene monomer at 70°C to achieve a 35% w/w styrene in

the resin. If the content of styrene exceeds 45%, styrene tends to homopolymerize, forming styrene homoblocks in addition to the crosslinked polyester network.<sup>12</sup> The UPR is in a green color liquid due to pigments in the PET soft drink bottles.

### Filler treatment

A 10% solution of NaOH was added into the beaker containing dried and sieved fillers and stirred well. This was kept for 3 h at room temperature. The fillers were then washed thoroughly with distilled water to remove the excess of NaOH sticking to the fillers before drying in an oven.

### Preparation of the composite

The fillers were added to the resin and mixed with cobalt octoate and MEKP at a ratio of resin : MEKP : cobalt octoate of 100 : 2 : 1.5 with a mechanical stirrer for 15 min. The components then poured into aluminum mold castings (~ 3 mm thickness). The working surface is polished and a mold-releasing agent is applied on the surface. The composites were prepared at different filler contents of 10, 20, and 30 (v/v %) for both 250–350 and 100-µm filler size. All samples were cured at room temperature for 24 h and postcured at 60°C for 6 h and at 150°C for another 5 h. The demolded sheets were trimmed and cut for further testing.

### Analysis and testing

Fourier transform infrared (FTIR) spectroscopy (Perkin-Elmer, GX Model) has been carried out on the resin. The acid value and hydroxyl value of the prepared resin were measured according to ASTM D 1639-90 and ASTM E 222-88. The structure of UPR before and after crosslinking was identified using <sup>13</sup>C nuclear magnetic resonance (NMR) model Jeol-ECP 400 instrument.

Tensile test of the composites has been carried out using an Instron 5567 as in ASTM D 638-91 at a crosshead speed of 5 mm/min and a gripping length of 100 mm. Flexural tests were performed using the same instrument according to ASTM 790-91, under a three-point-bend test. The specimens were 125 × 12.5 × 3 mm in size. A span of 75 mm was employed, maintaining a 2.5 mm/min and a span-to-depth ratio of 16 : 1. Hardness measurements were taken in accordance with ASTM D 2240-91 using Shore Scale D, Durometer. All mechanical tests have been carried out for the average of eight samples. The experimental densities of all composites were measured using electronic densitometer (MD-200S). The theoretical density and void content were calculated according to ASTM D2734.

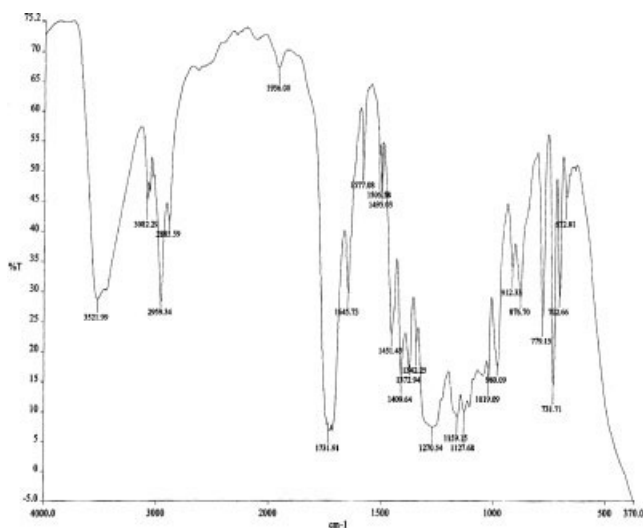


Figure 1 FTIR spectrum of prepared UPR.

Water absorption tests have been done according to ASTM D570-81. The morphological studies have been done using scanning electron microscopic (SEM) technique (Philips, XL30 model). Composites are gold coated before subjecting to SEM analysis.

## RESULTS AND DISCUSSION

### Characterization of the polyester resin

The acid number and hydroxyl value of the resin are 26.0 and 22.2 mg KOH/g by the end group titration, which gives a number average molecular weight of 2300 g/mol. These values are similar to the values reported in literatures between 22.8 and 33.8.<sup>13</sup> The FTIR spectrum of the prepared resin in Figure 1 showed that the absorption at  $3252\text{ cm}^{-1}$  is associated with the OH group and  $2959\text{--}3082\text{ cm}^{-1}$  is for C—H stretching in polyester. The absorption at 912 is responsible for C=C vinyl in styrene, which is absent in the chemical formula of glycolysed product,<sup>14</sup> suggesting that crosslinking occurred between polyester chain and styrene as crosslinkable monomer, both at their unsaturated active sites.

The  $^{13}\text{C}$  NMR spectra for UPR before and after crosslinking are shown in Figure 2(a,b), respectively. In both spectra, carbonyl group (C=O) (at 160 ppm), O—CH<sub>2</sub>—CH<sub>2</sub>—O (at 63 ppm) and C=C (at 129 ppm) are detected. Although both  $^{13}\text{C}$  NMR spectra showed the existence of C=C, the intensity of the peak decreased after crosslinking. On the other hand, other peaks after crosslinking can be detected in the range of 126–134 ppm due to the formation of new C—C single bonds and styrene aromatic ring which are absence in Figure 2(a). This indicates that the crosslinked UPR is a mixture of uncross-linked

and crosslinked chains. The *cis*-maleated units in a polyester chain have lower reaction rates with styrene due to the steric hindrance that often remain unreacted within a crosslinked copolymer, while decreasing in the intensity of the peak related to carbons in C=C obviously reveals the reduction in the population of unsaturated carbons after crosslinking.<sup>12</sup>

### The effect of chemical modification on fillers

#### FTIR analysis

Figure 3 illustrates the FTIR spectra of treated and untreated sawdust fillers. The spectrum of untreated

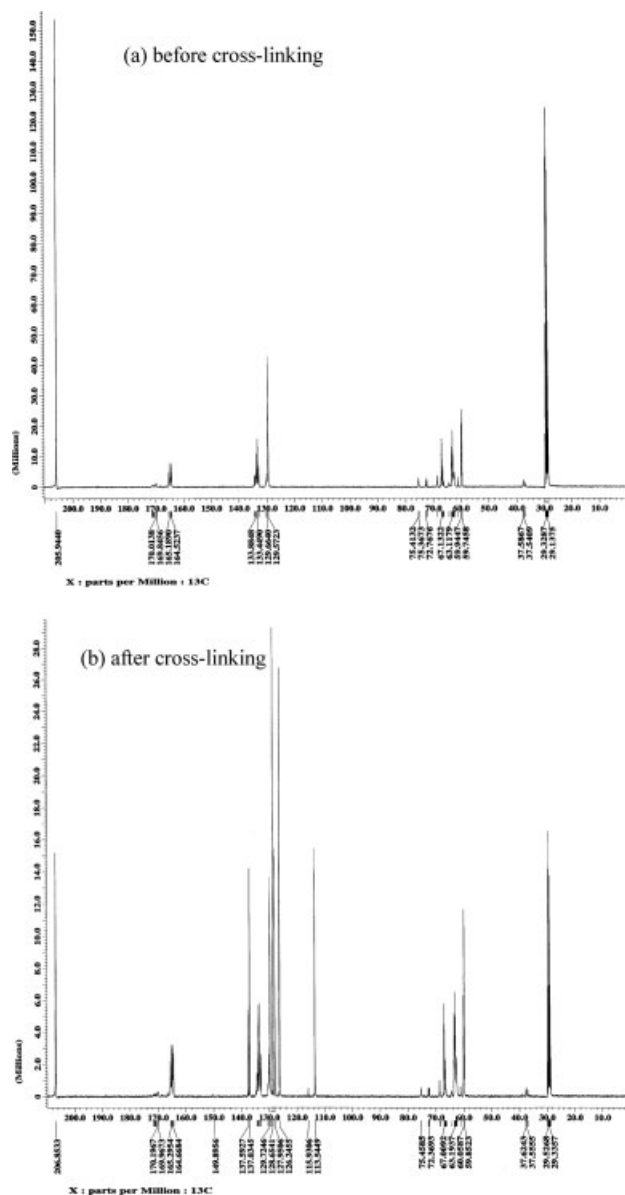
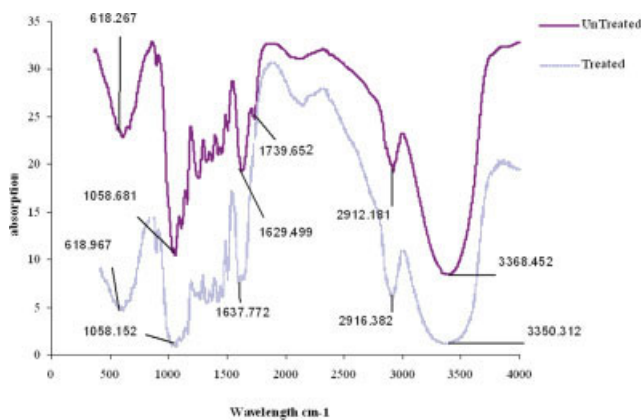


Figure 2  $^{13}\text{C}$  NMR spectra of UPR: (a) before crosslinking and (b) after crosslinking.



**Figure 3** FTIR spectra of treated and untreated *acacia* fillers. [Color figure can be viewed in the online issue, which is available at [www.interscience.wiley.com](http://www.interscience.wiley.com).]

fillers shows a strong O—H stretching absorption at  $3368\text{ cm}^{-1}$  and a prominent C—H stretching absorption at  $2912\text{ cm}^{-1}$ , unconjugated C=O stretching at  $1739\text{ cm}^{-1}$  and aromatic skeletal vibration at  $1507\text{ cm}^{-1}$ . In the finger print region between  $900$  and  $1800\text{ cm}^{-1}$ , C—H in plane deformation at  $1425\text{ cm}^{-1}$ , C—H deformation (symmetric) at  $1371\text{ cm}^{-1}$ , C—O stretch at  $1058\text{ cm}^{-1}$  and glucose ring stretch at  $889\text{ cm}^{-1}$ .<sup>15</sup> However, the FTIR spectrum of treated fillers shows the elimination of the absorption at  $1739\text{ cm}^{-1}$  which corresponds to unconjugated carbonyl groups. This could be due to the removal of the soluble lignin and hemicellulose due to alkali treatment.<sup>16</sup> Cellulose has high degree of polymerization and crystallinity and is responsible for strength in wood filler. Therefore, damaging the lignin networks and diminishing the impurities lead to increase the number of free —OH groups on the filler surface and to the —OH groups of cellulose to be more accessible for a stronger bond at the interface.<sup>17</sup>

#### Surface topography of wood fillers

The surface topography of wood particles was studied by SEM. Figure 4 exhibits the micrographs of treated and untreated sawdust fillers. Figure 4(a) indicates an uneven surface for the untreated fillers. Previous studies also revealed the presence of wax, oil, and surface impurities for untreated fillers.<sup>18</sup> It is shown in Figure 4(b) that the substances from the surface of the *acacia* filler was removed by alkali treatment and hence, may produce an improvement in the wettability property. These two micrographs also show the fibrillation of filler by treatment. It is expected that this fibrillation leads to an increase in the surface area of the filler and consequently to an

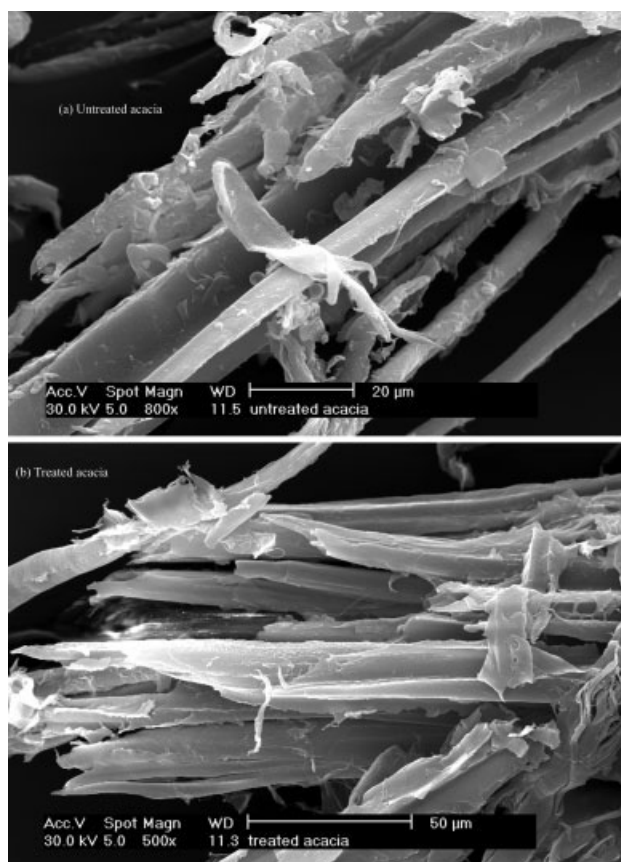
enhancement of the adhesion between the polymer matrix and the filler.<sup>19</sup>

## Composite testing

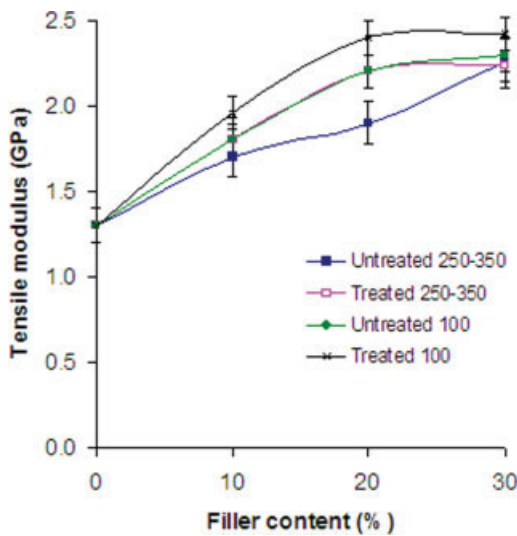
### Mechanical test

Figure 5 shows the effect of filler content, size and surface treatment on the tensile modulus of the composites respectively. The increase of the modulus is evidently due to the reduction of the volume fraction of the matrix which indicates the ability of sawdust fillers to impart greater stiffness to the matrix. It is also shown that composites with alkali-treated fillers show higher modulus than untreated fillers. This implies that alkali treatment improves the surface adhesive characteristics by removing natural and artificial impurities such as wax and pectin, thereby producing rough surface topography. The alkali treatment also helps to improve the dispersion of filler in the matrix, resulted in reducing the agglomeration of the filler.<sup>20</sup>

Figure 6 shows that the tensile strength decreases with filler content. Unlike fibers that have a uniform cross section and relatively high aspect ratio, irregular

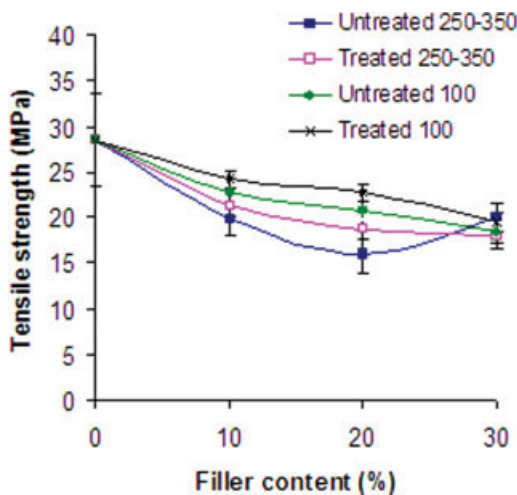


**Figure 4** SEM micrographs of (a) untreated *acacia* ( $\times 500$ ) and (b) treated *acacia* ( $\times 500$ ).



**Figure 5** Tensile modulus of UPR composites with treated and untreated *acacia* fillers. [Color figure can be viewed in the online issue, which is available at [www.interscience.wiley.com](http://www.interscience.wiley.com).]

shaped fillers such as sawdust has poor capability to support stress transmitted from the matrix. Thus, the strength enhancement in the filled composite is, in general, much lower than that of fiber reinforced composites. The increase of filler content also produced more fibers ends. This means that there is considerable stress concentration taking place where microcracks are formed and fibers debond from the matrix.<sup>21</sup> On the other hand, the poor interfacial bonding causes partially separated microspaces between filler and polymer matrix, which obstructs

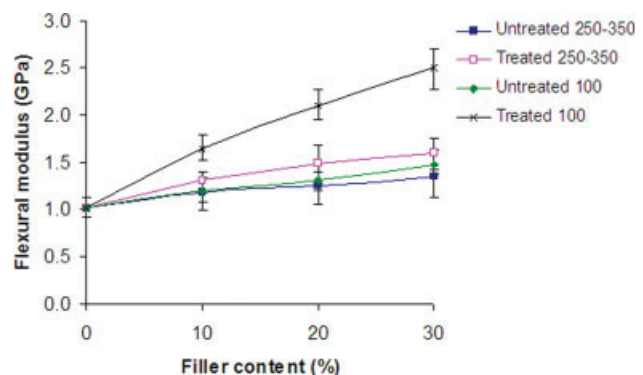


**Figure 6** Tensile strength of UPR composites with treated and untreated *acacia* fillers. [Color figure can be viewed in the online issue, which is available at [www.interscience.wiley.com](http://www.interscience.wiley.com).]

stress propagation at filler–matrix interface.<sup>22</sup> The opposite behavior for the large particle size at 30% filler content compared to 20% filler was anticipated because of the irregular-shape filler, which leads to inconsistency tensile strength and modulus at higher filler content. Nevertheless, if the composite behavior is compared based on “neat *acacia*” content, difference in the tensile strength is not significant as shown in Figure 6. Agglomeration of the filler particles and dewetting of the polymer at interphase aggravates the situation by creating stress concentration points, which account for the weakness in the composite. This indicates that composites with smaller filler size have higher strength than those with bigger size. This result agrees quite well with other studies.<sup>23,24</sup> Figure 6 also shows that the tensile strength of alkali-treated sawdust is higher than the untreated sawdust for similar filler loading. The result indicates that alkali treatment improves the interfacial bonding by increasing the additional sites of mechanical interlocking, hence promoting more resin–filler interpenetration at the interface.

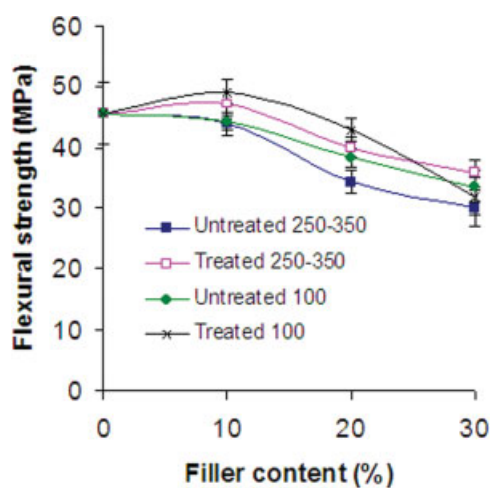
Figure 7 shows the effect of filler content, size and treatment on flexural modulus. It is well known that the modulus of a filled system depends on the properties of components, filler, and matrix; thus, the modulus of the sawdust was higher than the one of the matrix. Hence, the incorporation of sawdust fillers into the UPR matrix enhances the stiffness of the composites.<sup>25</sup> The results show that the alkali treatment can also improve the flexural modulus as well as tensile modulus for both systems.

Figure 8 shows the effect of filler content, size and treatment on flexural strength. It is obvious that the trend of the flexural strength is almost similar to those of tensile strength. It is anticipated that the reduction of the strength is likely to have been caused by a possible increase in void content with



**Figure 7** Flexural modulus of UPR composites with treated and untreated *acacia* fillers. [Color figure can be viewed in the online issue, which is available at [www.interscience.wiley.com](http://www.interscience.wiley.com).]



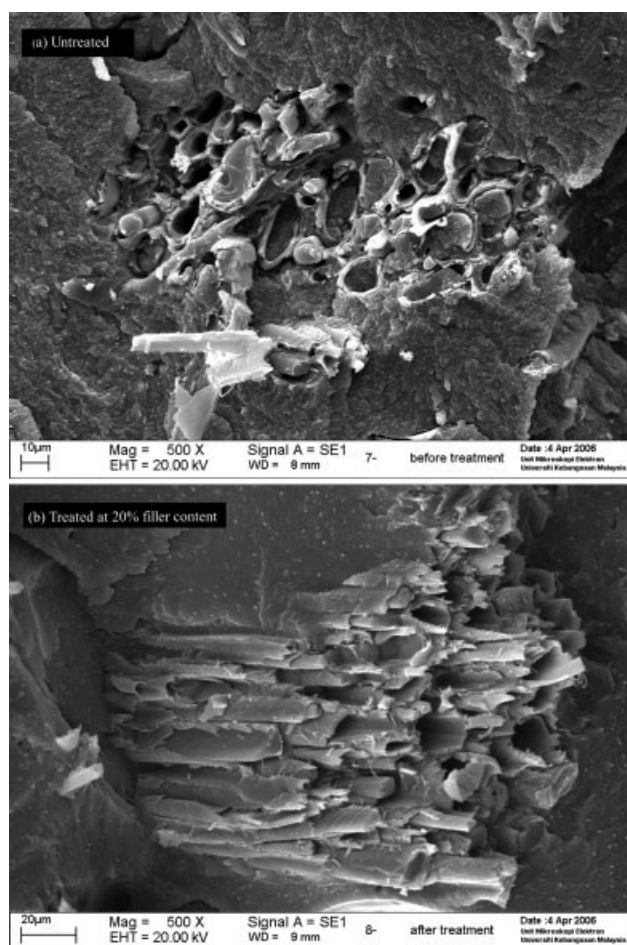


**Figure 8** Flexural strength of UPR composites with treated and untreated *acacia* fillers. [Color figure can be viewed in the online issue, which is available at [www.interscience.wiley.com](http://www.interscience.wiley.com).]

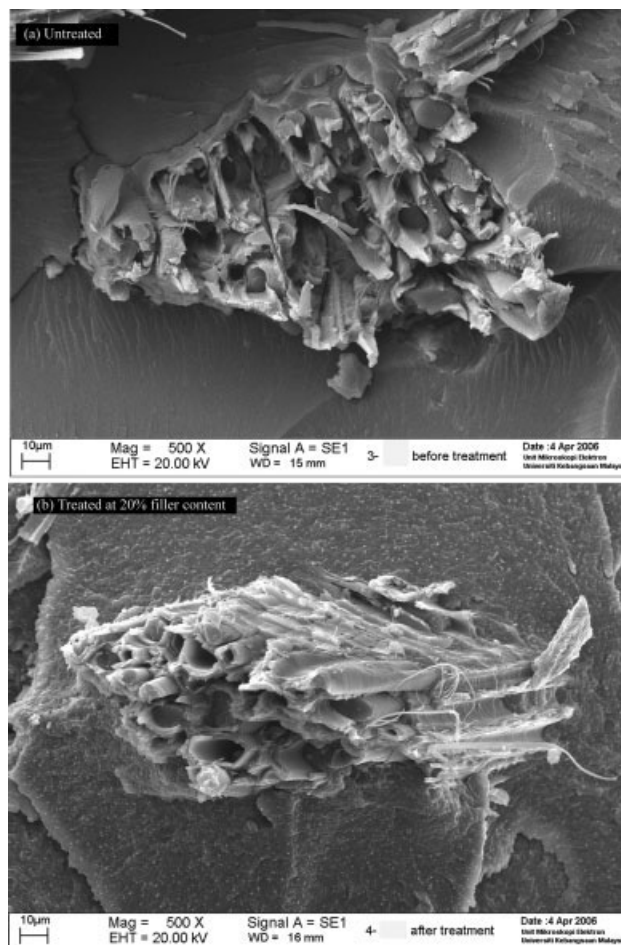
increasing filler volume fraction<sup>26</sup> (see void content measurements in Density Measurements and Void Content section). On the other hand, alkali treatment leads to increase in the effective surface area available for contact with the matrix and thus improve in the mechanical properties.<sup>27</sup>

#### SEM analyses

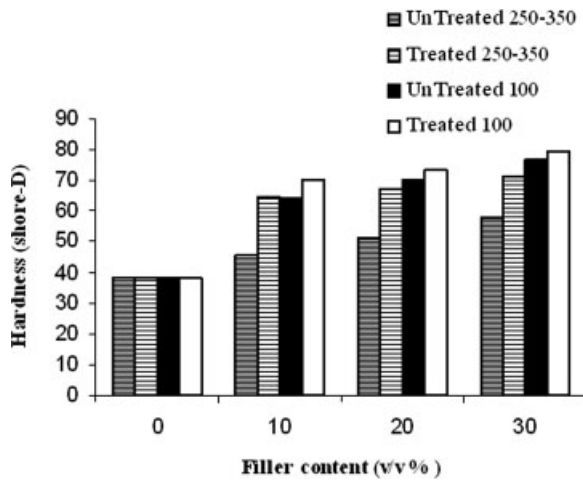
Figures 9 and 10 demonstrated the SEM micrographs of tensile fractured surfaces for *acacia*/UPR composites with filler size of < 100 and 250–350  $\mu\text{m}$  before and after treatment respectively. It can be seen in Figure 9(a) that the fillers with smaller size are more tightly embedded in UPR matrix than the bigger size. It is clearly seen that smaller size fillers possesses a higher surface area in contact with the matrix. This facilitates the effective stress transfer between the filler and UPR matrix with no crack propagation occurs at the filler–matrix interface. However, the interfacial region for composites with



**Figure 9** SEM micrographs of fracture surfaces of the *acacia*/UPR composites for smaller size: (a) untreated and (b) treated at 20% filler content.

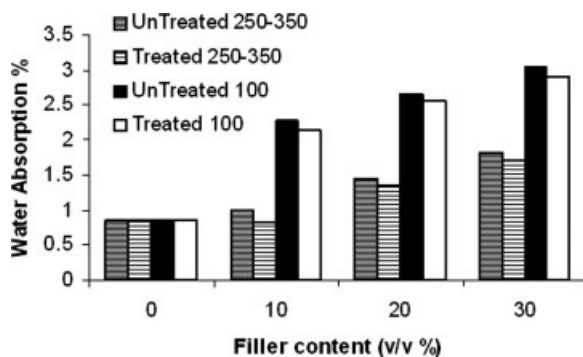


**Figure 10** SEM micrographs of fracture surfaces of the *acacia*/UPR composites for bigger size: (a) untreated and (b) treated at 20% filler content.



**Figure 11** Hardness measurements of UPR composites with treated and untreated *acacia* fillers.

bigger filler size [Fig. 10(a)] shows that there is some part of the fillers, which are not completely filled with the matrix. These phenomena agree quite well with the mechanical properties of the composites. Figure 9(a) depicts fracture surface of untreated *acacia* composites with filler pullout and many holes that left after pulling out the filler from the matrix. Figure 9(b) shows pullout fillers along with the existence of cracks at the broken filler end sites, suggesting failure occurred at the filler due to the strong adhesion between filler and matrix. The same phenomena can be observed in Figure 10(b) for the composites with bigger particle sizes. Micrographs of treated composites also reveal a thick layer of the matrix skin formation on the fillers. This confirms the composite capability to support stress transferred from the polymer matrix is enhanced.<sup>28</sup> The number of free OH groups on the fiber surface for the alkali-treated wood fillers have also increased, which regard to the hydrophilic nature of the resin leads to a stronger bond at the interface.



**Figure 12** Water absorption (%) of UPR composites with treated and untreated *acacia* fillers.

## Hardness

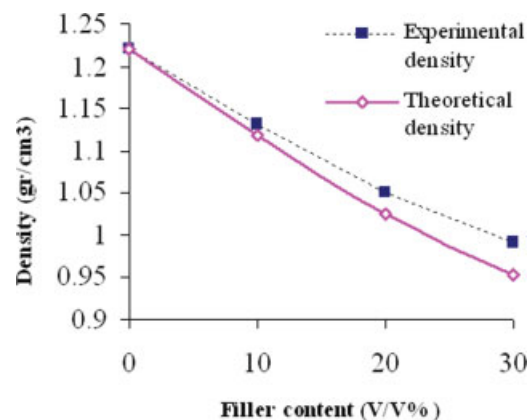
Hardness measurement for bigger and smaller filler size on untreated and treated sawdust/UPR composites is shown in Figure 11. It can be seen that composites with smaller fillers have higher hardness than those with bigger fillers. Generally, adding fillers increase the hardness of composite, but in case of composites with smaller fillers, higher hardness is expected according to greater surface area than the bigger size fillers. However, alkali-treated composites showed superior hardness than untreated ones, because of the generation of more sites for mechanical interlocking.

## Water absorption

The percentage of water absorption for treated and untreated composites is shown in Figure 12. It can be seen that increasing filler content led to higher water absorption. However, the water uptake decreased with surface modification of *acacia* fillers. In case of untreated *acacia*/UPR composite, the poor wettability and adhesion between untreated fillers towards UPR resin are attributed to the hydrophilic nature of *acacia* filler. This hydrophilicity is responsible for the higher percentage water uptake in untreated *acacia* composites. In case of surface modified composites, the filler get masked with the UPR in the laminate with the stronger adhesion resulting in greater hydrophobicity and lesser water absorption.<sup>29</sup> The results also indicate that the composites with smaller the size fillers have higher water uptake due to the larger surface area per volume.

## Density measurements and void content

Figure 13 shows the measured density of sawdust/UPR composites as a function of filler content. The



**Figure 13** Theoretical and experimental density of sawdust/UPR composites. [Color figure can be viewed in the online issue, which is available at [www.interscience.wiley.com](http://www.interscience.wiley.com).]

**TABLE I**  
**Void Content Measurements**

Filler content (v/v %)	Void content (vol %)
0	–
10	1.24
20	2.09
30	3.73

straight line shows the theoretical density of the composites when  $\rho_{\text{matrix}} = 1.22 \text{ g/cm}^3$ ,  $\rho_{\text{filler}} = 1.48 \text{ g/cm}^3$ . Theoretical density is measured according to the following equation:

$$T = \frac{100}{\left(\frac{R}{D} + \frac{r}{d}\right)}$$

where  $T$  = theoretical density,  $R$  = weight% of resin in composite,  $D$  = density of resin,  $r$  = weight % of filler in composite,  $d$  = density of filler. As the filler content increases, the density of the composites decreases. The experimental data fall very close to the theoretical ones indicates that the amount of voids are negligible, and that the existence of voids had no significant effect on the composite density.<sup>30</sup> Table I shows the trend of void content by filler content. Generally, a lower void content indicates a strong interaction between the filler and matrix. Thus, the increase in the void content of the composites by filler content as shown in Table I can be responsible factor for the drop in the strengths as discussed in previous section.

## CONCLUSIONS

The objective of the investigation was to realize a process that allows the use of UPR based on recycled PET and sawdust wastes in value added products. The results show that the tensile and flexural modulus increased with filler content but the tensile and flexural strength reduced with the smaller size has higher mechanical properties. The presence of alkali treatment improved the adhesion between the filler and polyester matrix as observed by SEM and consequently enhanced the mechanical properties and reduced the water absorption of the composites. The void content of composites also increased with filler content, which has a reverse effect on the strengths. The result shows the advantage of using recycled PET in fiber-reinforced poly-

mer applications, and its use helps to reduce the cost of these products.

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