

Recycled Polypropylene (PP) - Wood Saw Dust (WSD) Composites: The Effect of Acetylation on Mechanical and Water Absorption Properties^{*1}

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ABSTRACT

Recycled polypropylene (RPP) - Wood Saw Dust (WSD) composites with and without acetylation of filler were produced at different filler loading (15%, 25%, 35% and 45% w/w) and filler size (300, 212 and 100 μm). The RPP-WSD was compounded using a Haake Rheodrive 500 twin screw compounder at 190°C at 8 MPa for 30 minutes. The mechanical properties and water absorption properties of modified and unmodified WSD-PP composites were investigated. Acetylation of WSD improved the mechanical and water absorption characteristic of composites. The decrease of filler size (300 to 100 μm) of the unmodified and acetylated WSD showed increase of tensile strength and impact properties. The composites exhibited higher tensile modulus properties as the filler loading increased (15% to 45%). However tensile strength, elongation at break and impact strength showed the opposite phenomenon. Water absorption increased as the mesh number and filler loading increased. With acetylation, lower moisture absorption was observed as compared to unmodified WSD. The failure mechanism from impact fracture of the filler-matrix interface with and without acetylation was analyzed using Scanning Electron Microscope (SEM).

Keywords : recycle polypropylene, wood saw dust, acetylation, mechanical properties, water absorption

1. INTRODUCTION

Malaysia is known as one of the developing countries which is abundantly endowed with agricultural resources like lignocellulosic fibres

and scraps from the agricultural and wood based industry especially wood saw dust. These wastes materials have become major environmental problem as there are no alternatives for reutilization of the scrap. Wood saw dust (WSD)

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is one of the by-products of lignocellulosic material. It can be defined as a substance that contains lignin, hemicellulose, cellulose and extractive. Recently, interest in the use of materials derived from natural resources as fibre reinforced/filled composites has increased significantly. For example, wood saw dust, oil palm wastes (empty fruit bunches, trunk, press fibre, frond), bamboo, baggasse, coconut coir, rice husk, pineapple leaf, and banana stems are among the natural fibres which have draw attention of many researchers [Khalil *et al.*, 2000; Hill *et al.*, 1998; Bledzki and Gassan, 1999]. The advantages of using these waste lignocellulosics based materials are they are light weight, easy processing, high performance and low cost [Hanafi *et al.*, 2003].

Wood saw dust is one of the preferred filler as they available in a great quantity in Malaysia. The total production of Malaysia round-wood rose 3.5% to 21.4 million cubic meters (cum) while plywood production rose by 8.4% to 4.4 cum in 2003 [Raymond, 2004]. It is estimated that the export earning from the timber sector was RM 16.3 billion in that year. These amounts show the large quantity of wood based materials produced annually. However, the dimensional instability of lignocellulosics material with changing moisture content is the main disadvantages of using these types of filler. This phenomenon was due to the interaction of the cell wall polymers containing hydroxyl and other polar groups that interact with moisture by hydrogen bonding [Ana *et al.*, 2004; Bolton, 1994]. The properties can be altered by modifying the chemistry of fibres and the cell wall polymers [Khalil *et al.*, 2001]. Acetylation is considered as the promising economical modification method to modify the wood saw dust properties. Acetylation can be done in a simple manner, yet capable to increase the potential of wood saw dust greatly [Youngquist and Rowell, 1998].

Plastic have desirable properties with respect to dimensional consistency, impact resistance and high strength-to-weight ratios [Edward, 1981]. Polypropylene (PP) is one of the polyolefin families that have been used as matrix as it is very compatible with most of the composite filler. PP has excellent mechanical properties compared to other thermoplastics. It has higher softening point and its mechanical properties are constant at general temperature.

However, low stiffness of PP often limits their uses. In order to increase the impact resistance of the material, the stiffness is sacrificed because these two properties are in opposition to one another. However, technological advances are making plastic more attractive by enhancing their stiffness-to-weight ratios and other mechanical, physical and thermal properties without greatly sacrificing their impact resistance [Miles and Briston, 1996]. Moreover, recycled polypropylene (RPP) is commonly used and each day plentiful of RPP is thrown away and caused the environmental problem [Welling, 1979]. There are abundant of RPP wastes available in Malaysia. In previous studies, much work has been done on lignocellulosic filled PP composites. In fact, products made from the composite has been commercialized as the products are easily processed, low in cost and has good mechanical properties [Ser van der Ven, 1990].

The objectives of the present work are to investigate the potential of the acetylation of WSD to be used as filler in RPP composites and to evaluate their mechanical and water absorption properties.

2. MATERIALS and METHODS

2.1. Fiber Preparation

Wood saw dust (WSD) from Rubberwood (*Hevea Brasiliensis*) and *Acacia Mangium* species

was obtained from a local sawmill. The WSD was sieved and segregated according to 3 sizes (300, 212 and 100 μm) using Restch Test Sieve. The WSD was then dried in the oven at 105°C overnight to remove the moisture prior to compounding. The acetylation of the WSD was conducted using round bottom reaction flask filled with acetic anhydride and heated to 100°C for 30 minutes. Temperature was controlled by thermostatic oil bath. The heated acetic anhydride was poured into fibres inside the round bottom flask. The fibres were refluxed for 3 hours. After the completion, the anhydride was decanted off, while the fibres were quenched in the acetone for 1 hour at ambient temperature. After 1 hour, the acetone was poured out and new fresh acetone added. The fibres were refluxed for 3 hours in order to clean the fibres and remove unreacted reagents and its by-products. The modified fibres were then oven dried at 105°C overnight and then cooled into the ambient temperature in the dessicator over the silica gel. Recycled polypropylene (RPP) products were acquired from a local plastic product manufacturer. The RPP was crushed using Grinder 3 Phase Induction Motor model S4301 into small particle size.

2.2. Mixing Process

Four samples were prepared using different filler loading (15%, 25%, 35%, and 45%) of WSD over RPP. They were then compounded in a Haake Rheodrive 500 twin screw extruder. The compounder was first preheated to 190°C. During the compounding, the mixing process was observed to make sure that WSD mixed well with the RPP. The extruded strands were cooled before palletizing. The mixed pallets were then dried for 24 hours before proceeding to the next process. Pallets were spread in the mould that has been covered with the transparency to prevent the melting pallet sticking to

the mould. The mould was then pressed using Carver Laboratory Press model 'M'. Composites board of dimensions 21 \times 21 \times 4 mm were produced. The end product was cut using bandsaw for testing.

2.3. Mechanical Test

The unmodified and acetylated composite produced were cut for tensile and impact testing. The tensile test was conducted according to ASTM D3139, using an Instron Universal Testing Machine Model 1114. The Charpy impact tests were carried out on un-notched samples according to ASTM D256 Standard, using an Impact Pendulum Tester (Zwick) Model 5101. A minimum of 20 samples were tested in each case.

2.4. Water Absorption Test

The water absorption property of the composites was determined according to ASTM D570. Samples were immersed in distilled water at an ambient temperature of 25 \pm 3°C up to 34 days. Then, they were removed and blotted with tissue paper to remove the access water on surface and the weight was recorded. The water absorption was calculated according to the equation 1:

$$\text{Water absorption, WA(\%)} = \frac{(M_2 - M_1)}{M_1} \times 100 \quad (1)$$

Where;

M_2 = Mass of the sample after immersion (g)

M_1 = Mass of the same sample before immersion (g).

2.5. Fourier Transform Infra-red (FT-IR) Analysis

FT-IR analysis was employed to characterize the product of the acetylation on WSD and the

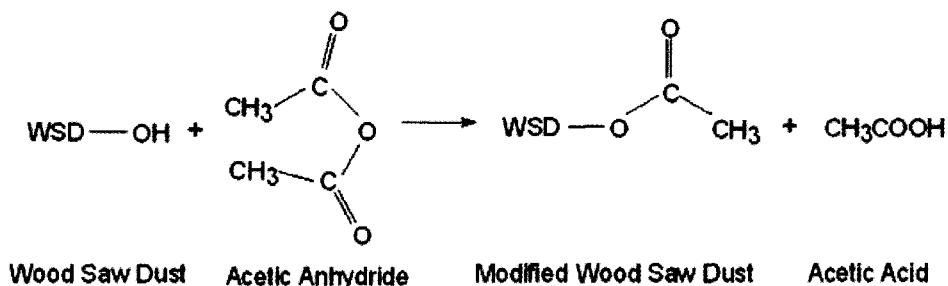


Fig. 1. Reaction of acetylation WSD with acetic anhydride.

Table 1. Main chemical composition of WSD

Main Composition	Percentage (%)
Lignin	24
Hemicellulose	65
Extractives	45
α -Cellulose	5
Ash	3

difference of IR spectra between virgin polypropylene and recycled polypropylene. The analysis was carried out using KBr disk method with Nicolet Avatar 360 FT-IR spectrometer. The samples were grounded to a powder for the analysis. The mixture of KBr disks containing 1% powdered sample by weight was mixed and transferred to a die to form a thin pallet for IR analysis.

2.6. SEM Analysis

The fracture surface of the composites from the impact test were investigated with a Leica Cambridge SO360 Scanning Electron Microscope (SEM). The samples were mounted on SEM specimen holder using double sided electrically conducting carbon adhesive tabs to prevent surface charge on the specimens when exposed to electron beam. Then, the samples were coated with 20 nm aurum using Fison SC515 Sputter Coater for 10 minutes to form an electrically conducting layer on the samples surfaces.

3. RESULTS and DISCUSSION

3.1. The Reactivity of WSD Flow with Acetic Anhydride

The reaction scheme for acylation of wood saw dust (WSD) with acetic anhydride is shown in Fig. 1. The reaction of acetylation involves esterification of the cell wall polymer with acetic anhydride. The degree of the reactivity of WSD with acetic anhydride depends on the reactivity of hydroxyl groups in the substrate and the rate of diffusion of the reagent in the filler matrix. The order of reactivity has been found to be lignin > hemicellulose > α -cellulose. This is because lignin is comprised of the fibrous nature of the wood which mainly has abundant hydroxyl groups. The polymeric composition of WSD investigated here is shown in Table 1. The results show similar composition as the results of work done before [Bolton, 1994].

3.2. FT-IR Characterization

The proof of acetylation was confirmed by the FT-IR spectra, as shown in Fig. 2. The rate of reaction is dependent upon the relative reactivities of the hydroxyl groups in the substrate, and the rate of diffusion of the reagent into the fiber matrix. Several major bands of

Table 2. Peak assignments for several major absorption bands of unmodified and acetylated WSD and virgin and recycle PP.

Sample	Frequency (cm ⁻¹)		Assignments
	WSD (Unmodified)	WSD (Acetylated)	
	3480	3482	OH stretching
	1744	1747	C=O stretching
	1375	1380	CH deformation of CH ₂ and CH ₃
	1230	1232	C-O-C stretching

Sample	Frequency (cm ⁻¹)		Assignments
	Virgin PP	Recycle PP	
	2944	2925	CH stretch in aromatic ring and alkanes
	2872	2876	Methyl group CH
	1460	1462	Symmetrical bond of hydrocarbon CH ₂
	1368	1377	CH deformation of CH ₂ and CH ₃

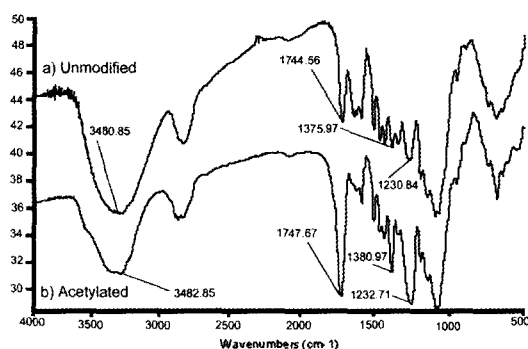


Fig. 2. FT-IR spectra of (a) Unmodified and (b) Acetylated WSD.

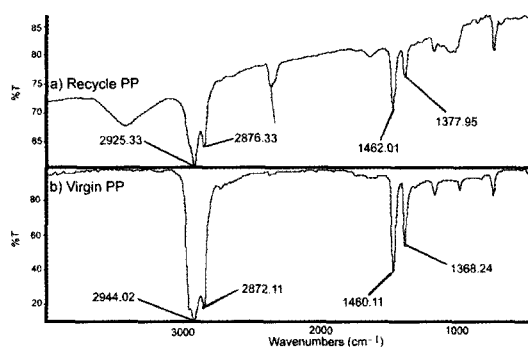


Fig. 3. FT-IR spectra of (a) Recycle polypropylene and (b) Virgin polypropylene.

unmodified and acetylated WSD recorded in Table 2. The degree of the reactivity of plant fibers towards chemical modification with acetic anhydride depends on the reactivity of hydroxyl groups. With untreated WSD, four major changes on acetylation were observed: (1) an increase in the carbonyl (C=O) stretching region (1747 cm⁻¹). The increase in intensities due to the C=O bond present in the acetyl groups; (2) an increase in the carbon-hydrogen (C-H) bond of an -O-(C=O)-CH₃ stretching region (1380 cm⁻¹); (3) an increase in the carbon-hydrogen (C-H) stretch around 1232 cm⁻¹. After acetylation, the fibre becomes more hydrophobic and the H₂O

content decreases [Silverstein *et al.*, 1991].

The absorption bands of recycled PP and virgin PP shows a slight difference between one another (Fig. 3). There are decreases in intensities observed in: (1) CH stretch of aromatic ring of RPP at 2925 cm⁻¹; (2) the formation of methyl group at 2876 cm⁻¹; (3) while the stretching regions of 1462 cm⁻¹ and 1377 cm⁻¹ was formed of the symmetrical bond of hydrocarbon CH₂ and CH₃ respectively [Silverstein *et al.*, 1991; Flett, 1963].

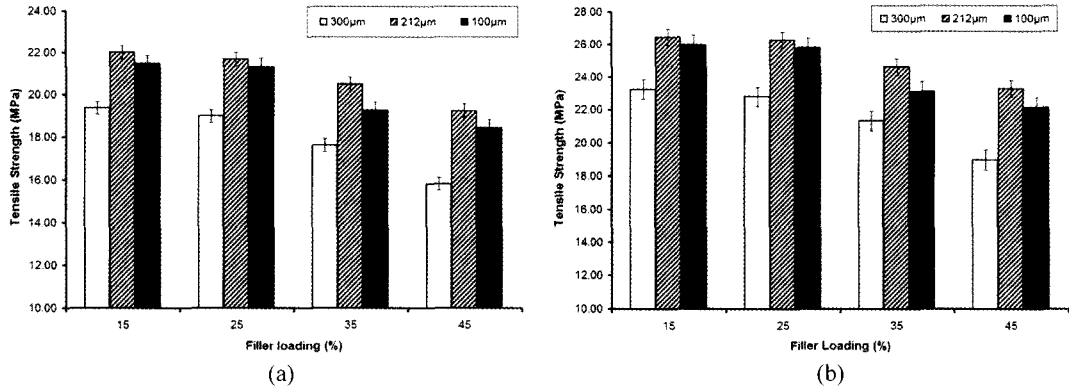


Fig. 4. (a) Effect of filler size and filler loading on tensile strength of unmodified WSD, (b) Effect of filler size and filler loading on tensile strength of acetylated WSD.

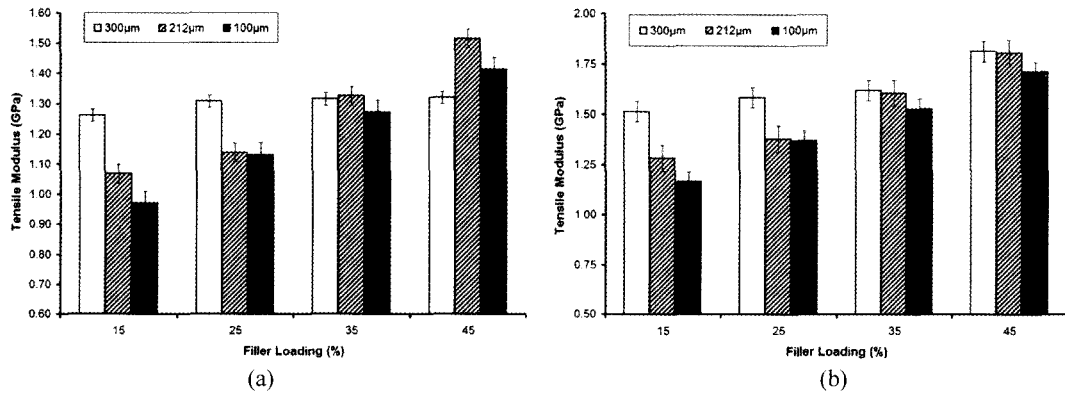


Fig. 5. (a) Effect of filler size and filler loading on tensile modulus of unmodified WSD, (b) Effect of filler size and filler loading on tensile modulus of acetylated WSD.

3.3. The Effect of Acetylation on Tensile Properties of Composites

Fig. 4(a) and (b) show the effect of filler size and filler loading on the unmodified and acetylation of WSD on tensile strength of recycled polypropylene (RPP) composites, respectively. Without modification, Fig. 4(a) shows the tensile strength increased as the filler size decreased from 300 μm to 212 μm . However, below 212 μm , the strength was slightly reduced. The strength of the composites increased by filler loading as follows: 45% of 212 μm WSD (19.23 MPa) < 35% of 212 μm WSD (20.48

MPa) < 25% of 212 μm WSD (21.69 MPa) < 15% of 212 μm WSD (22.00 MPa). With increasing filler loading 15% to 45% (interval 10%), the tensile strength slightly reduced. Higher filler loading did not improve the strength of the composites. These data significantly showed that 212 μm of WSD size exhibited highest tensile properties. The strength of composites with 300 μm displayed lowest properties among all. With acetylation (Fig. 4(b)), the trends of increasing filler size and filler loading showed the same phenomenon as with unmodified WSD. However, the values were slightly higher as acetylation improved filler-matrix bonding. The

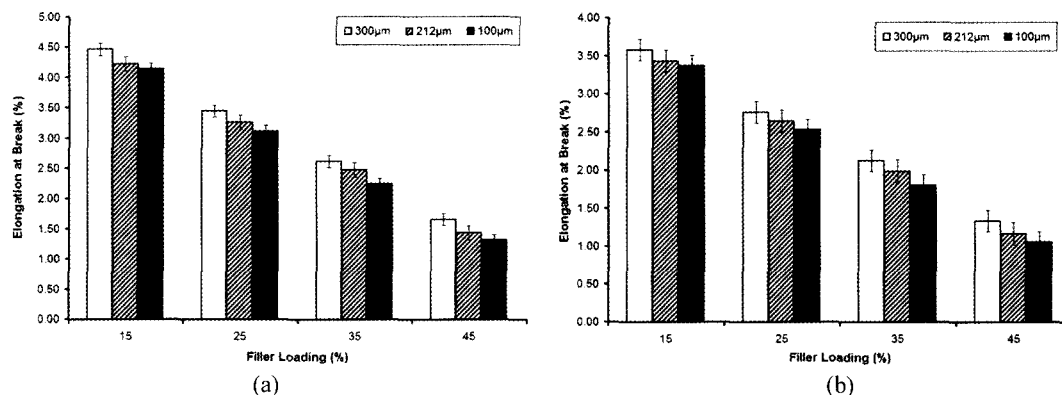


Fig. 6. (a) Effect of filler size and filler loading on elongation at break (%) of unmodified WSD, (b) Effect of filler size and filler loading on elongation at break (%) of acetylated WSD.

highest tensile strength recorded at 15% of 212 μm WSD loading composites was 26.40 MPa while composites with 45% of 300 μm of WSD exhibited the lowest tensile strength of 18.97 MPa. The tensile strength values of acetylated composites samples increased approximately 20% of the initial values for unmodified WSD.

Fig. 5(a) and (b) show the comparison of the effect of filler size and filler loading on the tensile modulus of composites with unmodified and modified WSD, respectively. With unmodified WSD, the modulus increased as filler loading increased from 15% to 45%. Composite with 45% of 212 μm WSD size displayed highest modulus, whereas the lowest value was obtained from 15% of 100 mesh WSD size. The modulus of the composites increased by filler loading as follows: 15% < 25% < 35% < 45%. Higher filler loading exhibited higher modulus compared to lower filler loading. This is expected as the incorporation of fillers has been shown by various studies to impart greater stiffness in the composites. Decreased size from 300 to 212 μm mesh increased the tensile modulus, except at low filler loading (15% and 25%). This may be attributed to the greater interaction and/or better dispersion of the smaller-size particles as compared to larger ones. How-

ever, the values dropped in every loading when 100 mesh WSD was used.

With acetylation (Fig. 5(b)), decreased filler size and increased of filler loading increased the tensile modulus, but the values were slightly higher than the tensile modulus for unmodified WSD. Also, acetylation has improved the adhesion between filler and matrix, even at high filler size loading (35% to 45%). The values of tensile modulus of 35% and 45% for 35 mesh size of WSD have increased 23% and 26%, respectively compared to the values of unmodified WSD.

Fig. 6(a) shows the elongation at break of unmodified WSD. The elongation at break decreased as the filler size decreased and filler loading increased. This was due to the less elongation of RPP plastic as filler size and loading increased. As we note, 15% of filler loading composites exhibited higher elongation for every WSD size. The lowest elongation at break was achieved at 100 μm filler size (4.16%), followed by 212 μm size (4.22%) and the highest was 300 μm filler size (4.47%). Composite with highest loading (45%) of smallest filler size (100 μm.) revealed the lowest value of elongation with 1.32%. The percentages of the elongation, however, increased with filler

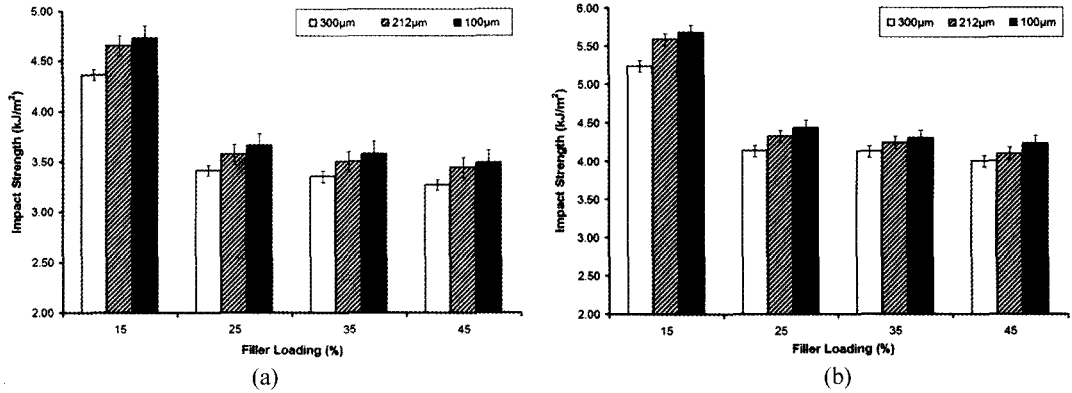


Fig. 7. (a) Effect of filler size and filler loading on impact strength of unmodified WSD, (b) Effect of filler size and filler loading on impact strength of acetylated WSD.

size increase. This was expected as bigger filler size has more ability to withstand higher tensile stress. Acetylation reduced the elongation at break as filler loading increased and filler size decreased as shown in Fig. 6(b). The acetylated WSD showed lower values of elongation at break as compared to unmodified WSD since the filler of acetylated WSD is more brittle and less elongated. The highest value was recorded at 15% of 300 µm WSD size (3.57%) while the lowest value obtained at 45% of 100 µm WSD mesh size (1.05%). The data shows a reduction of 20% elongation for acetylated WSD composites compared to unmodified WSD composites.

3.4. The Effect of Acetylation on Impact Properties

The impact strength of composites increased as the filler size decreased from 300 µm to 100 µm as shown in Fig. 7(a) for unmodified WSD. This is expected since samples with smaller-size filler have better homogeneity and relatively higher absorption energy than samples with larger-size filler during fracture process. The plot shows that composites with 100 µm size WSD have higher impact strength compared to composites having higher filler size (212 µm

and 300 µm) for each loading. However, the impact strength decreased as the filler loading increased from 15% to 45%. The highest impact strength (4.74 kJ/m²) was obtained at 15% of 100 µm WSD while the lowest value (3.26 kJ/m²) was obtained at 45% of 300 µm WSD filler composites. Composites with acetylated filler have the same trends as unmodified WSD, but higher impact strength than those with unmodified fillers, where the value increased by 20%. The increased in hydrophobicity as the result of acetylation of the filler has resulted in the increased interaction between the filler and the matrix (Fig. 7(b)).

3.5. SEM Analysis

Scanning electron microscope was used to study the impact fracture surfaces of virgin polypropylene (PP), recycled polypropylene (RPP) resin matrix, the unmodified and acetylated WSD composites. SEM micrographs are shown in Fig. 8(a) and 8(b); of PP and RPP matrix fracture morphology accordingly. It was observed that radial disruption surrounding of fracture occurred starting from the middle of the surface. The structure of RPP (in circle) was different compared to PP surface as we can see the

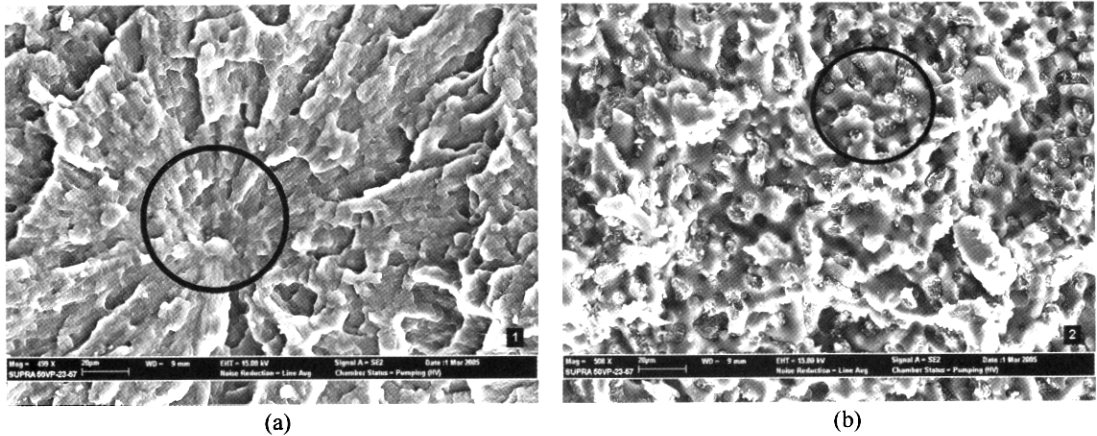


Fig. 8. (a) SEM micrograph of impact failure of PP and (b) SEM micrograph of impact failure of RPP at 500x magnification.

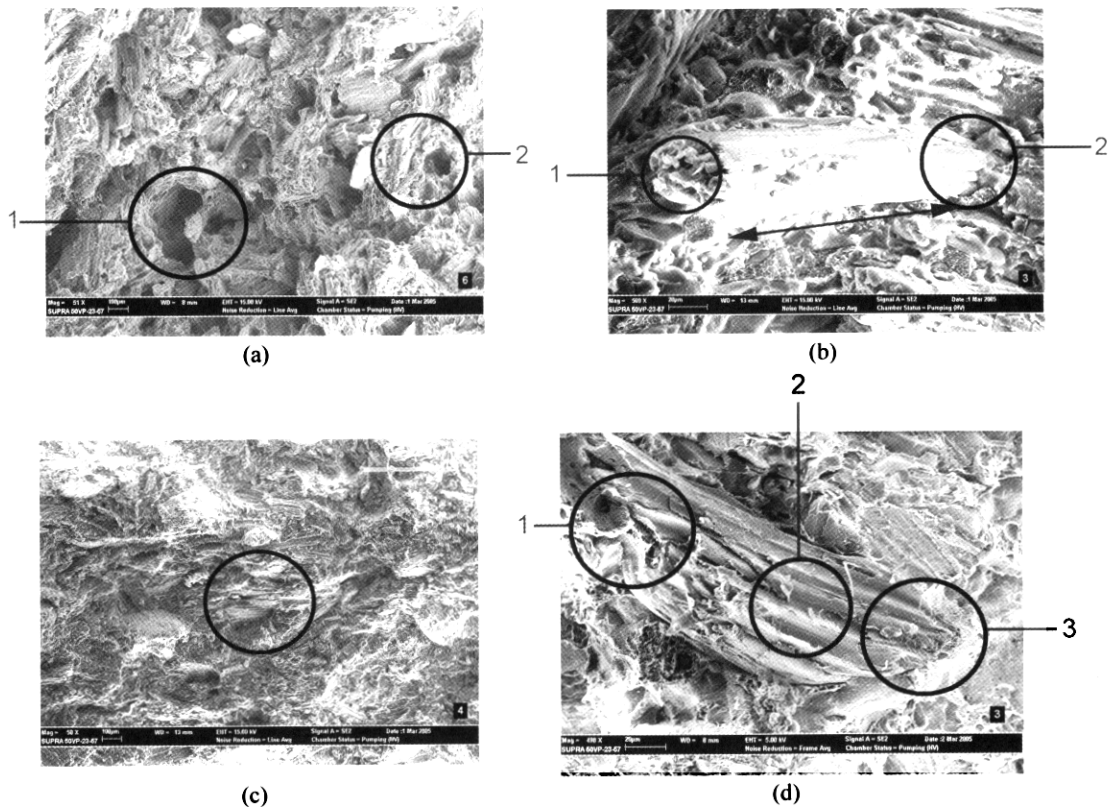


Fig. 9. SEM micrograph of impact fracture of unmodified WSD, (a) Showing voids and pull-out fibre sites formation (50x); (b) Showing long fibre pull-out and opening matrix-filler interphase crack (500x); and acetylated WSD, (c) Showing improved adhesion between filler-matrix interphase (50x); (d) Showing fibre damage and defibrillation due to good bonding (500x).

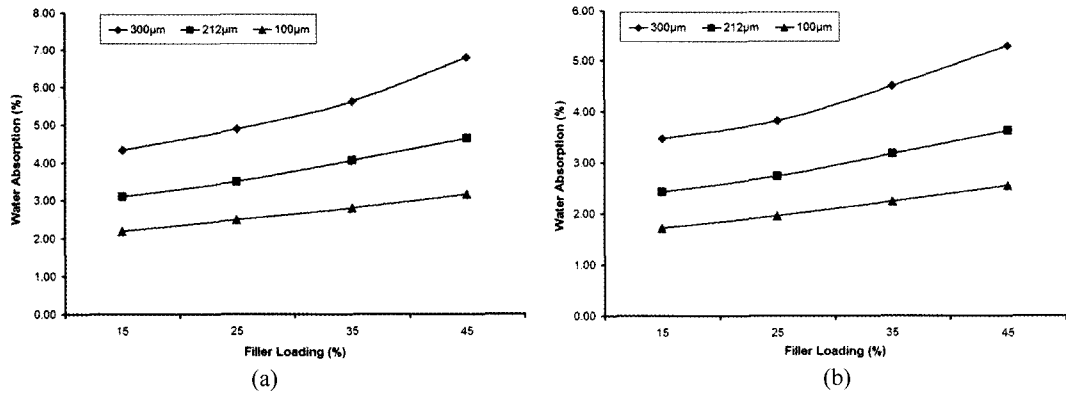


Fig. 10. (a) Effect of filler size and filler loading on water absorption of unmodified WSD, (b) Effect of filler size and filler loading on water absorption of acetylated WSD.

addition of pigments and other things in the RPP. This was due to the recycled process and thermal history of RPP itself. However, the amounts of these materials are too small and their addition in the RPP do not affected the properties of the matrix and the composites. As for the composites, there are lots of voids and air bubble formation (circle 1) and pull out fibre sites (circle 2) observed in unmodified WSD composites at 50 magnifications (Fig. 9(a)).

The presence of voids and porosity has apparently affected the properties of composites. Fig. 9(b) shows the fracture surface of unmodified WSD composite at high magnification (500x). Circle 1 shows that the fracture fibre surface at the ends was uniform across the tip. The long fibre pull-out path observed (arrow line) indicates the opening of matrix cracks that allowed WSD to be pulled out from the surrounding matrix. The crack and voids at the RPP matrix and filler interphase (circle 2) shows poor adhesion between matrix and filler.

Fig. 9(c) shows the micrograph of the fracture surface of acetylated WSD composites at 50x mag. The acetylation seems to improve the bonding between the WSD and RPP matrix. The matrix phase exhibits better filler dispersion with acetylated WSD. There is also less inci-

dence of filler pull-out and the fibre pull-out length is shorter compared to unmodified WSD. It can be seen that the fibre even breaks at the fracture surface (circle 1). It also can be observed that the fibre damaged and defibrillation (circle 1 and 2) of the WSD is due to good bonding of filler and matrix (Fig. 9(d)). The filler-matrix interphase also improved as there was good adhesion and the evidence of fibre failure mode occurred (circle 3), which is rarely seen in unmodified WSD. It can be concluded that the contact between matrix and fibre phases has improved.

3.6. The Effect of Acetylation on Water Absorption Properties

The water absorption behaviour of the composites depends on the ability of the filler to absorb water due to the presence of hydroxyl group. The results in Fig. 10(a) clearly showed that as filler loading increased from 15% to 45% and filler size decreased from 100 µm to 300 µm, the water absorption of unmodified WSD increased. The degree of water uptake of the composites followed in the order: 15% < 25% < 35% < 45%. The water absorption reduced as filler size reduced. As we can see, com-

posites with highest filler loading (45%) and filler size (300 μm) displayed highest rates of water absorption with 6.78% and the lowest value was taken from the lowest loading of WSD (15%) of smallest filler size (100 μm) with 2.21%. This is due to more hydroxyl groups available, thus the composites tend to absorb more water. The absorption, however, decreased when composites are filled with acetylated WSD. The lowest value obtained was 1.72% while the highest rates observed was 5.28%. These data was taken from the same composites composition and loading as unmodified WSD composites, but significantly shows lowest properties. Fig. 10(b) shows that acetylated composites exhibited lower water absorption (less 20%) than unmodified WSD. Acetylation alters the specific configuration of WSD components due to substitution of hydroxyl groups. The blocking of hydroxyl groups also changes the hydrophilic nature of WSD.

4. CONCLUSIONS

The conclusions from this study are summerized as follow:

1) In general, tensile strength of the composites increased with filler loading in the order: 45% < 35% < 25% < 15% WSD loading. The tensile strength also increased as filler size decreased (300 μm to 212 μm WSD) but the values slightly decreased at 100 μm WSD. Composites with 212 μm size of WSD showed highest tensile strength properties at each filler loading (15%, 25%, 35% and 45%).

2) Impact properties improved as filler size decreased from 300 μm to 100 μm and deteriorated as filler loading increased from 15% to 45%.

3) As filler loading increased from 15% to 45% of WSD in recycled polypropylene composites, tensile strength and impact properties

decreased, but tensile modulus increased.

4) The rate of water uptake of the composites followed in order: 15% < 25% < 35% < 45%. The water absorption decreased as filler size decreased from 300 μm to 100 μm .

5) Acetylation of WSD improved the tensile, impact properties and water absorption, since acetylation resulted in hydrophobic fillers and improved filler-matrix bonding.

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