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European Polymer Journal 37 (2001) 1037–1045

EUROPEAN
POLYMER
JOURNAL

The effect of acetylation on interfacial shear strength between plant fibres and various matrices

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Received 6 January 2000; received in revised form 22 May 2000; accepted 26 June 2000

Abstract

Interfacial shear strength between acetylated fibres and various matrices was studied. The chemical modification of oil palm empty fruit bunch (EFB) and coconut fibre (coir) using non-catalysed acetic anhydride were investigated. Proof of acetylation of EFB and coir was indicated by the increase in weight percent gain, and was confirmed by Fourier transform infrared spectra. Pull-out tests were employed to study the compatibility of acetylated fibres with different commercial matrices such as "Epiglass" (epoxy), "West system" (epoxy), "Crystic" (unsaturated polyester), "Metset" (unsaturated polyester) and polystyrene. An increase in the interfacial shear strength between acetylated fibres and hydrophobic resins was noted. Modified fibres were shown to have a smoother surface compared with unmodified, which was believed to be a factor in improving the fibre–matrix adhesion. © 2001 Elsevier Science Ltd. All rights reserved.

Keywords: Acetylation; Interfacial shear strength; Plant fibres; Compatibility; Thermoset; Thermoplastic

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1. Introduction

Incorporation of fibres (man-made or natural) into a polymer is known to cause substantial changes in the mechanical properties of composites [1]. A good interfacial bond between fibre and matrix, can substantially improve the mechanical properties of the composite. Interfacial shear strength is one of the means of determining bonding between fibre and matrix.

The term "interface" has been defined as the boundary region between two phases in contact. The composition, structure or properties of the interface may be variable across the region and may also differ from the composition, structure or properties of either of the two contacting phases [2]. This interfacial region exhibits a complex interplay of physical and chemical factors, that exerts a considerable influence on and controls the properties of reinforced or filled composites. The interfacial interaction depends on the fibre aspect ratio, size of the interface, strength of the interaction, anisotropy, orientation, aggregation, etc. [3]. Extensive research has been done on the interfacial shear strength (ISS) of man-made fibre (e.g. carbon and boron fibres, polymeric fibres and different types of glass fibres) [3–7] and for the natural fibres [8–10] by using methods such as fibre pull-out tests, critical fibre length and microbond tests.

The problems encountered when attempting to combine any plant fibres or any lignocellulosic material with a thermoplastic or thermoset matrix is one of incompatibility. The reason for this is due to the abundant hydroxyl functionality of fibre cell wall polymers which ensures that the material is hydrophilic. Whilst, generally thermoset, thermoplastic or elastomer polymers almost are hydrophobic. The problem then is one of 'wetting' the fibre. There are various methods available to improve the compatibility between lignocellulosic and the matrix.

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PII: S0014-3057(00)00199-3

1. Chemical modification of the lignocellulosic (anhydrides, epoxides, isocyanates, etc).
2. Grafting of polymers onto the lignocellulosic.
3. The use of compatibilisers and coupling agents.

The mechanism of reinforcement, is dependent on the stress transfer between the matrix material and embedded fibre [11]. In particular, the fibre–matrix ISS is one of the most important parameters in controlling the toughness and the strength of a composite material [4–11]. Its value is particularly dependent on fibre surface treatment, modification of matrix and other factors affecting the properties of the fibre–matrix interface.

With single fibre composite method, the fibre is embedded in a polymer matrix and loaded in the axial direction with a force. The ISS along the fibre length can be described by the Cox equation [12].

$$\frac{\sigma_{\mathrm{fu}}}{\tau_{\mathrm{y}}} = \frac{2l_{\mathrm{c}}}{r} \tag{1}$$

where σ_{fu} is fibre tensile strength, r, radius of fibres, l_{c}, critical fibre length and τ_{y}, ISS.

This method is only applicable to systems where the resin has a significantly lower modulus than the fibre, and a constant fibre diameter is required for each individual fibre. The latter condition must be good for man-made fibre study of fibre–matrix bonding, but does not apply to natural fibres.

For small diameter fibres, a meniscus effect prevents an accurate determination of the embedded fibre length. For natural fibres, such as wood and other plant fibre materials, short length and lumen structure make it virtually impossible to obtained a uniform embedded length [13]. The ISS generally can be approximated as:

$$\tau_{\mathrm{a}} = \frac{L\sigma_{\mathrm{f}}}{2r} \tag{2}$$

where τ_{a} is apparent ISS, r, the radius of the fibre, L, the thickness of the matrix and σ_{f} the normal stress of fibres as interfacial debonding occurs.

A microbond test was developed [14] for characterising the single fibre–matrix (man-made) interface in order to eliminate any meniscus effect. The method involved the debonding of a small polymer droplet that was placed on the surface of the fibre. The test has been widely used to evaluate the ISS for both thermoset and thermoplastic resin composites [14,15] because sample preparation was simplified and the ISS is insensitive to different loading test configurations. By assuming that the shearing force applied to the fibre–matrix interface is uniform, the ISS can be calculated as:

$$\tau = \frac{F_{\mathrm{d}}}{\pi d L} \tag{3}$$

where τ is ISS, d, fibre diameter, L, microdroplet length and F_{d}, maximum debonding force.

Recently Hill and Abdul Khalil [16] and Liu et al. [17] employed this type of test using natural fibre. The method was found to be applicable to the computability between fibre and matrix. Therefore, the method was adopted in this study. The primary purpose of this work, was to determine the ISS of acetylated fibre in various matrices. The capability of the acetylation of empty fruit bunch (EFB) and coconut fibre (coir) in improving the interfacial properties in composites was studied. Acetylation was believed to impart hydrophobicity on the fibres, hence improving the interfacial properties with different matrix materials. This was evaluated using a simple fibre pull-out test.

2. Experimental

2.1. Acetylation of fibres

The selected oil palm EFB and coir fibres were extracted in a Soxhlet apparatus with a 4:1:1 mixture (by weight) of toluene, methylated spirit and acetone, for 5 h. The extracted fibres were then dried overnight in an oven at 105°C. After drying, the fibres were allowed to cool in a desiccator, over silica gel. Each sample (≈1 g) was weighed to four decimal places and placed in a round bottom reaction flask. Other round bottom reaction flasks with sufficient acetic anhydride to cover the fibres were brought to the desired reaction temperature of 100°C for 3 h to obtain optimum high weight percent gain (WPG). Temperature control was provided by a thermostatic oil bath. The heated acetic anhydride was poured into a round bottom flask (fitted with a reflux condenser) which contained the pre-weighed fibres.

After the appropriate time period, the anhydride was decanted off, and the fibres were put into acetone at ambient temperature, to quench the reaction, for a period of 1 h. Extreme care was taken not to lose any fibre during the transfer from the reaction flask into the other weighed (oven dry) flask. After 1 h, the acetone was poured out, and new fresh acetone added. The fibres were washed by refluxing in acetone for 3 h, in a round bottom flask fitted with a reflux condenser in order to removed unreacted reagents and by-products. The modified fibres were then oven-dried at 103 ± 2° C overnight, cooled in a desiccator and weighed. Weight gain (based on percentage of oven dried weight) of the chemically modified fibres was calculated as follows:

$$\mathrm{WPG}\ (\%) = [(\text{weight gain}/\text{original weight})] \times 100. \tag{4}$$

Chemical composition of EFB and coir fibres were determined using the procedure stated by the previous study [18].

2.2. Fourier transform infrared characterisation

Fourier transform infrared (FT-IR) analysis was employed to characterise the product of the reaction. The analysis was carried out on a Mattson FT-IR spectrometer Model Nicolet 750 Series II using the KBr disk method. KBr disk which consisted of 1% of powdered sample (by weight) was prepared using an evacuable die.

2.3. Interfacial shear strength determination

ISS testing was conducted accordingly to a method presented in an earlier study [16,27]. Acetylated fibres of EFB and coir of 15–18% WPG were tested. The matrices used were of commercial matrices such as "Metset" (unsaturated polyester with 33% styrene content), "Crystic" 471 PALV (unsaturated polyester with more 45% styrene content), "Epiglass" (epoxy), "West system" (epoxy) and polystyrene. The fibres were placed in a tube and held vertically in the centre. The resin was left to cure at room temperature (20°C) for overnight. Then the samples were conditioned at 65% RH, 25°C for one week, before testing.

The same testing procedure was carried out those with polystyrene, however, the preparation of the matrix followed with different procedure. Styrene was mixed with azo-iso-butyronitrile (AIBN) as catalyst, in the ratio 100:0.2 (v/w). The fibres and styrene mixture was partly cured at room temperature (20°C) for 4 h and followed by heating in an oven at 70°C for overnight to ensure a complete cure.

Testing was conducted using an "Instron" universal testing machine (model 4301) at a cross-head speed of 0.5 cm s^{-1}, equipped with a PC computer. The typical load (force)–displacement curve was plotted (the force needed to totally debond the fibre from the matrix), was recorded. The average fibre diameter and embedded length were measured. The embedded length was measured with vernier calliper (an average of two readings were taken for each tube). The maximum of the interfacial shear strength was estimated using Eq. (3) [16]. At least 35 replicates were conducted for each parameter. The data was analysed by the analysis of variance (ANOVA) to determine the significance of the differences in treatment, using "MINITAB" statistical package (version 12).

3. Results and discussion

The proof of acetylation of EFB and coir fibres is indicated by the increase of WPG. The reaction scheme for acetylation is shown in Fig. 1. The chemical composition and WPG of EFB and coir are shown in Table 1. The results of WPG obtained at 3 h reaction for EFB and coir was 15% and 18%, respectively. In the study of the modification of natural fibre with acetic anhydride, it has been reported that the reactivity of the fibre cell wall polymers with acetic anhydride is in order of lignin > hemicelluloses > cellulose [18,19]. The reactivity is dependent upon the relative reactivities of the hydroxyl groups [19] in the substrate, and the rate of diffusion of the reagent into the fibre matrix [18–20].

The evidence of acetylation was confirmed by the FT-IR spectra. Figs. 2 and 3 show the FT-IR spectra of unmodified (control) and modified of EFB and coir, respectively. Upon acetylation of EFB, three major changes were observed: (1) an increase in the carbonyl (C=O) stretching region (1750 cm^{-1}), (2) an increase in

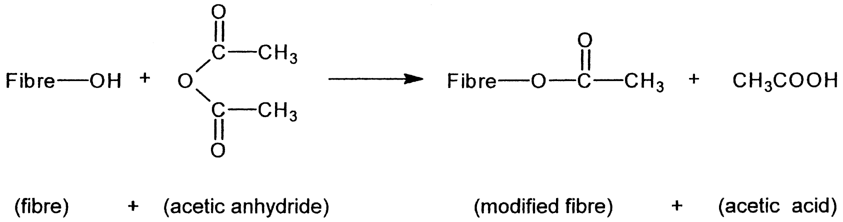

Fig. 1. Plant fibres reaction with acetic anhydride.

Table 1
Percentage chemical composition and WPG of EFB and coir in this study

Fibre	Chemical composition (%)				Weight percent gain (%)
	Cellulose	Hemicellulose	Lignin	Extractives	
EFB	53	19	22	3	15
Coir	46	21	31	4	18

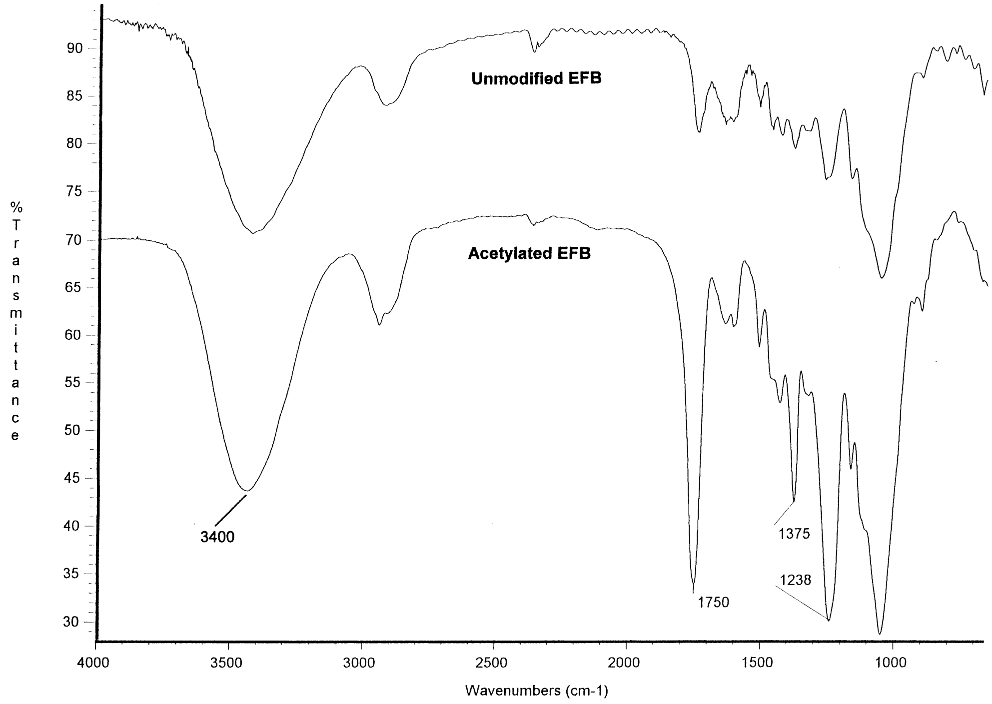

Fig. 2. FT-IR spectra of unmodified and acetylated EFB.

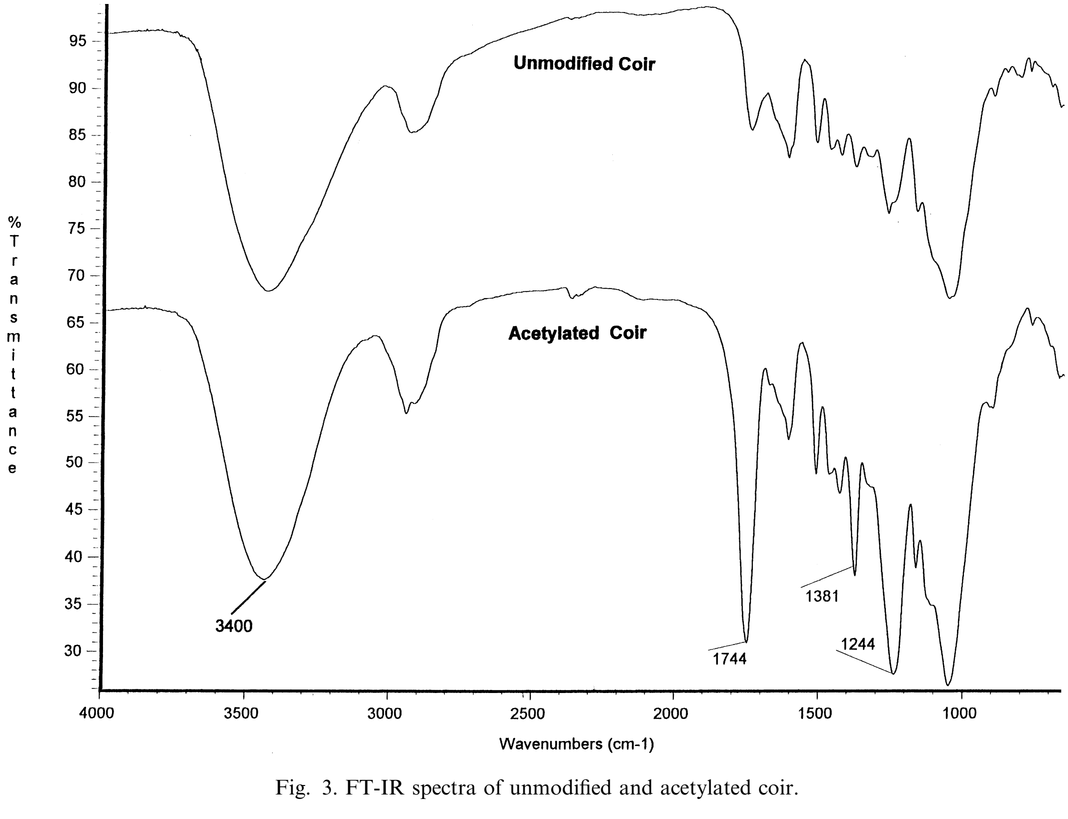

Fig. 3. FT-IR spectra of unmodified and acetylated coir.

the carbon–hydrogen (C–H) bond stretching region (1375 cm^{-1}) and (3) an increase in the carbon–hydrogen (C–H) stretch, around 1238 cm^{-1}.

The absorption at 3400 cm^{-1} (for unmodified sample) may be contributed by the hydroxyl groups and absorbed moisture through the formation of hydrogen bonding between the groups and water. It is obvious that, the same changes have occurred upon the acetylation (modification) of the sample. It can be seen that the peak at 3400 cm^{-1} has to some extent change in the degree of absorbance. The acetylation results in the increase of the hydrophobicity of the fibres due to the introduction of acetyl groups (through the substitution of the OH groups of the fibres). Thus, it can be postulated that the degree of moisture adsorption is also accordingly reduced. Hence, the changes in the absorption at 3400 cm^{-1} may be as the result of OH substitution and the decreased in the moisture absorption [19,21]. The same phenomenon was found with coir, (1) an increase in the carbonyl (C=O) stretching region (1744 cm^{-1}), (2) an increase in the carbon–hydrogen (C–H) stretching region (1381 cm^{-1}) and (3) an increase in the carbon–hydrogen (C–H) occurred around 1244 cm^{-1}. The peak assignments of the natural fibre from previous studies [22,23] are shown in Table 2.

Samples were observed under scanning electron microscope (SEM) on the surface, before and after acetylation. Both EFB and coir unmodified samples (extracted) possessed, a large amount of silica nodule [24,25] (Fig. 4a) and at high magnification (Fig. 4b). However, after modification (Fig. 4c and d) at high magnification, it is obvious that the surface of the fibre was rather smoother as compared to the unmodified at reaction for 3 h (to achieve high or optimum WPG). During acetylation, hydroxyl groups on the fibre surface are replaced by acetyl groups. This results in a fibre that has a more hydrophobic surface.

The ISS distribution in a composite has been studied by Cox [12]. It is assumed that (a) both matrix and fibre behaved elastically (b) their constituent interface remained intact during loading and (c) no load was transmitted from the fibre ends and thus possible stress concentration effects at the fibre ends were ignored.

The effectiveness of the fibre–matrix bond is dependent on chemical compatibility and the presence of mechanical ‘keying’ between the fibre and the matrix [16]. In general, coir fibres (either unmodified or modified) exhibited higher ISS values as compared to EFB fibres. This is due to higher lignin content of coir (31%) as compared with EFB (22%). Fig. 5 illustrates the ISS results of composites of EFB in various matrices. The ISS for the composites with unmodified EFB vary from matrix to matrix. The difference in these results reflect the inherent properties of each matrix. It can be seen that the matrix which consists of thermoset system is generally higher than those of thermoplastic. This may be due to the greater wettability of the thermoset system than the thermoplastic. This is so, since the thermoset is cured in situ, from relatively lower molecular weight while mixing with the fibres, followed by polymerisation, resulting in the embeddedment of the fibre in the harden matrix. Whilst, the thermoplastic is employed in molten polymeric state, where the wettability is limited. Higher ISS values of thermoset as compared to thermoplastic may be due to lignin content and other main polymeric (hemicelluloses, cellulose) which are reactive to thermoset than thermoplastic; hence improved ‘wetting’ between fibre and matrix.

The degree of changes in the ISS upon acetylation reflects the compatibility changes at the interfacial region between the fibre and the matrix. From the results, it can be seen that the composites with polystyrene as matrix, exhibit the highest degree of improvement in the ISS, followed by both epoxies and polyesters. Thus, the results show that the introduction of acetyl groups on the fibre surface has increased the ISS of the composites with polystyrene (as compared to the unmodified), significantly higher than those of epoxies and polyesters. This results in improved wetting of the polystyrene onto the modified fibre surface, thereby increasing the work of adhesion [16]. The work of adhesion increased by an increase in the surface tensions which can be

Table 2
The peak assignments of natural fibre for FT-IR analysis

Wave number (cm^{-1})		Groups
Unmodified	Modified	
1050–1085	1085–1050	Alcoholic C–O stretch
1310	1310	CH_2 from holocellulose
1390	1390	C–H from the cellulose and hemicelluloses
1460	1460	CH_2 from cellulose
–	1752–1979	Carbonyl C=O larger peak from the bonded acetyl group
1640	1640	Carbon double bond C=O
1750	–	Very small peak showing very little C=O from holocellulose + contaminated lignin
2900–3000	2900–3000	C–H stretch in aromatic ring and alkanes
3200–3600	3200–3600	Broad, strong band of the –OH group

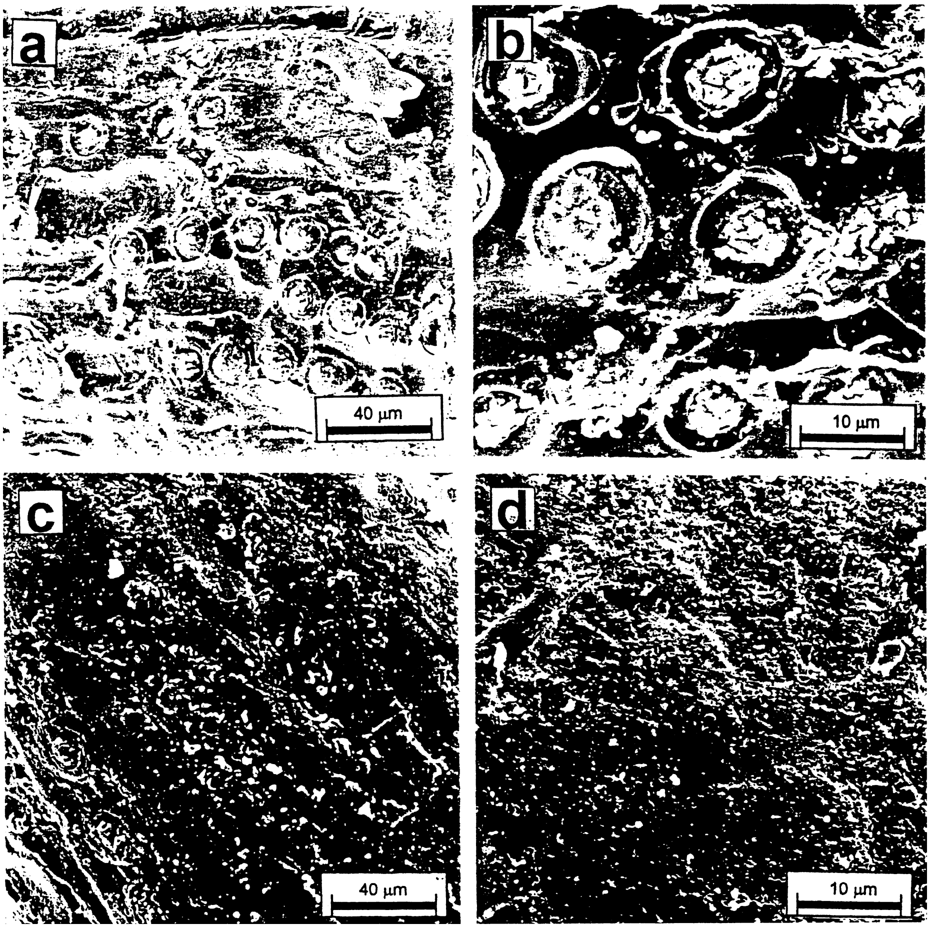

Fig. 4. SEM micrographs on surface of EFB fibres: (a) showing silica bodies of unmodified (800×), (b) silica nodule of unmodified fibre (3000×), (c) silica of an acetylated (800×) and (d) surface of an acetylated (3000×).

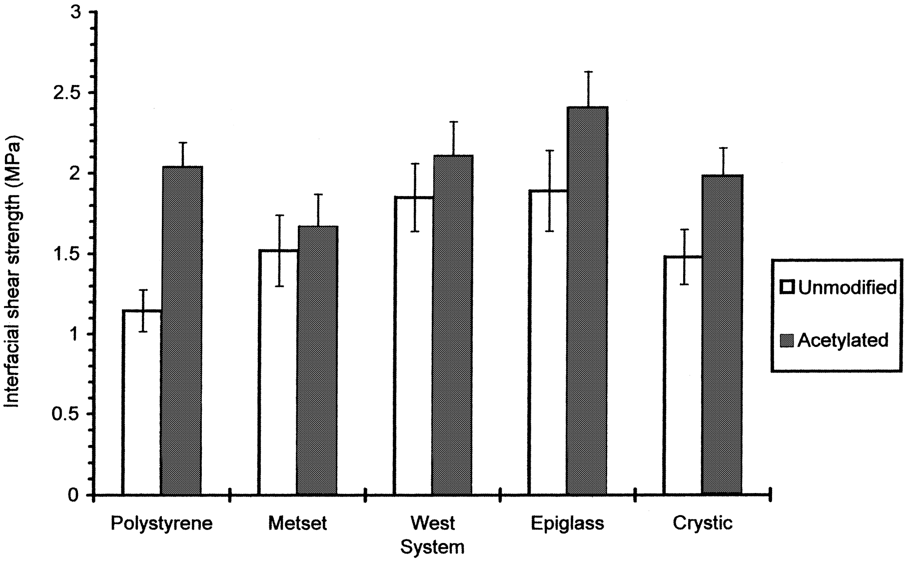

Fig. 5. ISS and standard deviation of EFB with various matrices.

explained in detail using the Young–Dupre [26] equation as follows:

$$\gamma_{S\text{-}V} = \gamma_{S\text{-}L} + \gamma_{L\text{-}V}^{\cos\theta} \qquad (5)$$

where $\gamma_{S\text{-}V}$ is surface energy between the solid and the vapour, $\gamma_{S\text{-}L}$, the surface energy between the solid and the drop, $\gamma_{L\text{-}V}$, the surface energy between the liquid and the vapour. This shows that the modification impart more efficient stress transmission from the thermoplastic matrix to the fibres than from the thermoset system. A rather same trend is shown for the ISS values with coir (Fig. 6).

Chemical modification of the fibres, creates more trans-active surface molecules that would readily form bonds with matrix. A similar result has been reported in the studies of the interaction between cellulosic fibres and polystyrene, where the acetylation of fibre improved the ISS [17]. With a polystyrene matrix, a significant difference ($p \leqslant 0.05$) in the ISS was noted as a result of acetylation of the fibres. Results of an ANOVA of ISS between unmodified and acetylated fibres in a polystyrene matrix are shown in Table 3.

"Metset" (SW products 'Buehler Krautkramer') is a unsaturated polyester resin which contains 33% styrene. Acetylation slightly improved the ISS for both EFB and coir fibres the values was significantly different ($p \leqslant 0.05$), compared to unmodified, (Table 4). "Crystic" 471PALV is a ployester resin manufactured by Scott Bader Ltd. (UK) which contains more than 45% of styrene. Acetylation has significantly increased ($p \leqslant 0.05$) the ISS, compared to unmodified, for both EFB and coir (Table 5). In this study, unmodified coir had an ISS of 1.68 MPa whereas 2.34 MPa for acetylated, whereas the ISS for EFB was 1.15 MPa for unmodified and 2.05 MPa for acetylated. As compared to "Metset", the ISS values was higher and this is presumably due to the differences in styrene content.

Epoxy 105 is a product of "West System", containing epoxy resin (molecular weight < 700) and benzyl alcohol. Acetylation of coir increased the ISS from 1.82 MPa (unmodified) to 2.46 MPa. However, the statistical

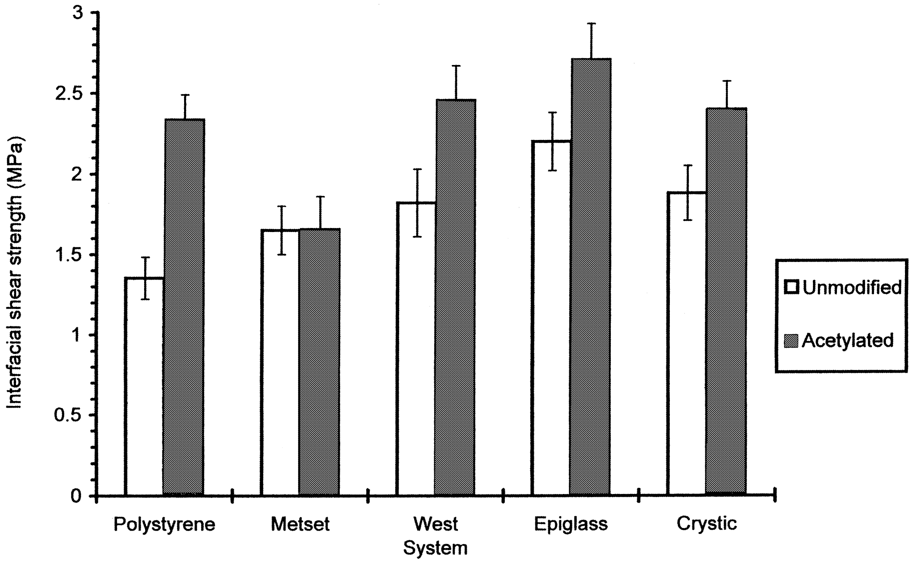

Fig. 6. ISS and standard deviation of coir with various matrices.

Table 3
Results of analysis of variance (unstacked) of polystyrene resin, correlations between unmodified coir or EFB with acetylated fibres

Source	Unmodified EFB vs. acetylated EFB					Unmodified coir vs. acetylated coir				
	DF	SS	MS	*F*	*p*	DF	SS	MS	*F*	*p*
ISS	1	21.98	21.98	29.7	0.001	1	6.66	6.57	12.4	0.001
Error	68	50.28	0.74			68	35.98	0.53		
Total	69	72.24				69	42.55			

DF: degrees of freedom; SS: sums of squares; MS: means of squares; *F*: the ANOVA test statistic (which is MS treatments/MS residuals); *p*: probability of the correlation's treatments, were significant at 95% ($p \leqslant 0.05$); ISS: interfacial shear strength; Error: the difference within unmodified and modified (EFB or Coir).

Table 4
Results of analysis of variance (unstacked) of Metset (unsaturated polyester) correlations between unmodified coir or EFB with acetylated fibres

Source	Unmodified EFB vs. acetylated EFB					Unmodified coir vs. acetylated coir				
	DF	SS	MS	*F*	*p*	DF	SS	MS	*F*	*p*
ISS	1	15.55	15.55	35.0	0.000	1	6.57	6.56	12.41	0.001
Error	68	30.21	0.44			68	35.98	0.053		
Total	69	45.76				69	42.55			

Table 5
Results of analysis of variance (unstacked) of Crystic (unsaturated polyester resin) correlations between unmodified coir or EFB with acetylated fibres

Source	Unmodified EFB vs. acetylated EFB					Unmodified EFB vs. acetylated coir				
	DF	SS	MS	*F*	*p*	DF	SS	MS	*F*	*p*
ISS	1	3.31	3.31	15.7	0.000	1	4.46	4.46	31.3	0.000
Error	68	14.29	0.21			68	9.70	0.14		
Total	69	17.6				69	14.17			

Table 6
Results of analysis of variance (unstacked) of West system (epoxy) correlations between unmodified coir or EFB with acetylated fibres

Source	Unmodified EFB vs. acetylated EFB					Unmodified coir vs. acetylated coir				
	DF	SS	MS	*F*	*p*	DF	SS	MS	*F*	*p*
ISS	1	2.51	2.51	10.9	0.000	1	0.84	0.84	4.0	0.050
Error	68	8.56	0.13			68	14.30	0.21		
Total	69	11.07				69	15.13			

Table 7
Results of analysis of variance (unstacked) of Epiglass (epoxy) correlations between unmodified coir or EFB with acetylated fibres

Source	Unmodified vs. acetylated EFB					Unmodified vs. acetylated coir				
	DF	SS	MS	*F*	*p*	DF	SS	MS	*F*	*p*
ISS	1	11.12	11.12	33.2	0.000	1	4.75	4.75	16.7	0.000
Error	68	22.74	0.33			68	19.35	0.28		
Total	69	33.85				69	24.09			

analysis confirmed that there was a significant difference between unmodified and acetylated ($p \leqslant 0.05$) (Table 6). EFB also showed increased ISS values with acetylated fibre compared to unmodified, that was from 1.85 to 2.15 MPa. From ANOVA, the values were significantly different at $p \leqslant 0.05$, (Table 6). “Epiglass” (HT 9000) which is an epoxy resin (molecular weight < 700), yielded the highest ISS. Of all the commercial systems, this material gave the highest ISS with modified and unmodified fibres. For example, acetylation of coir increased the ISS from 2.25 MPa (unmodified) to 2.75 MPa. Acetylation significantly (at $p \leqslant 0.05$) increased the ISS with both EFB and coir, (Table 7). It may be suggested here that the differences of “Epiglass” and “West system” of epoxy products may be related to a combination of change in surface morphology and chemistry. The smoother fibre surface, following acetylation, may allow for better resin wetting [16]. This would be less compatible with a resin which has a high benzyl alcohol content. Surface modification improve fibre–matrix compatibility and bonding characteristics by creating compatible surface energies and formation of chemical, physical and mechanical bonds.

4. Conclusions

The pull-out techniques was conducted to evaluate the interfacial shear strength of EFB and coir fibres in different matrices. Improvements in the ISS were observed upon acetylation of the fibres. Whilst, those with thermoset systems show the highest magnitude in the ISS, the greater improvement upon acetylation was shown with polystyrene. The increased of hydrophobicity of EFB and coir fibres due to acetylation, increased the ISS values. Acetylation of EFB and coir fibres showed increased ISS as compared to unmodified fibre and the values are significantly higher than unmodified. The hydrophobicity of acetylated fibres we observed from SEM and FT-IR studies. Acetylation change the fibre surface morphology. Unmodified fibres presented rough surfaces, having a large number of silica bodies present. Acetylation resulted in the surfaces becoming much smoother. Since the fibres become hydrophobic, it is inevitable that the compatibility with matrix materials will change. With hydrophobic resin, the ISS increased as a result of fibre modification as would be expected.

Acknowledgements

The authors wish to express their thanks to Sabutek Sdn. Bhd. Malaysia and Biocomposites Centre, UK for supplying fibres. Thanks are also due to Dr. Callum Hill, University of Wales, Bangor, UK and Dr. Jamie Hague, Biocomposite, UK for the use of facilities and for valuable discussion.

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