The Effect of Environmental Exposure Upon the Mechanical Properties of Coir or Oil Palm Fiber Reinforced Composites

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Received 16 July 1999; accepted 26 December 1999

ABSTRACT: Polyester matrix composites reinforced using nonwoven coir or oil palm empty fruit bunch fiber mats were manufactured. Fibers were used unmodified, chemically modified by acetylation, or treated with silane or titanate coupling agents. Composite test pieces were exposed to decay fungi in unsterile soil for up to 12 months, along with samples made of unreinforced, or glass fiber reinforced, resin. Water exposure tests were also performed. The effect of such exposure on the mass loss, tensile and flexural properties of the samples was evaluated. Mechanical properties deteriorated as a result of exposure. However, acetylation of fibers, or treatment with silane coupling agent was found to afford a significant degree of protection. © 2000 John Wiley & Sons, Inc. J Appl Polym Sci 77: 1322–1330, 2000

Key words: natural fibers; composites; decay; environmental exposure; acetylation

INTRODUCTION

Natural fiber reinforced composites may become a viable alternative to those which use glass fibers as reinforcement.[1,2] The current research interest is a result, in part, of the desire to find new sustainable replacements for man-made materials that require high-energy inputs for their manufacture.[3] Although a topical area at present, such applications of natural fibers have been investigated in the past.[4] However, this initial research interest stagnated once large quantities of glass and other synthetic fibers became available.[5] Although the use of plant fibers is of considerable interest, a major impediment to their use in composite applications is the degradation of mechanical properties which occurs as a result of exposure to moisture or degrading organisms.[6] This will consequently affect the mechanical properties of any composite formed therefrom.[7] Acetylation has been studied for a considerable length of time as a means of providing protection to wood exposed to environmental stresses. Acetylated wood exhibits enhanced performance in moist conditions and when exposed to biological attack.[8] It has also been shown that acetylated coir and oil palm empty fruit bunch fibers (efb) exhibit good performance when exposed to decay organisms.[6] The purpose of this work was to determine the effect of environmental exposure upon the properties of polyester matrix composites reinforced with acetylated coir, or efb fibers. The performance of these composites was compared with those reinforced with silane or titanate treated fibers. In addition, comparison was made with unreinforced, or glass fiber reinforced, polyester resin.

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Journal of Applied Polymer Science, Vol. 77, 1322–1330 (2000)

MATERIALS AND METHODS

Composite Manufacture and Specimen Preparation

Random nonwoven mats of coir and efb fibers were prepared by JB Plant Fibres Ltd., Holyhead, UK. A commercially available polyester (Crystic 471 PALV, Scott Bader) was used as the matrix phase. The resin was prepared by thoroughly mixing the resin plus hardener, followed by application of a vacuum to remove the trapped air. A high-density polyethylene liner was used for impregnation of the fiber mat. The liner was closed in the center with clips and the fiber mat positioned in one half of the liner. Activated resin was poured into the other half of the liner, the clips in the center removed, and a vacuum applied to suck the resin into the fiber mat. The process of resin impregnation was aided by application of a hand roller to the exterior of the liner. Once impregnation was complete, the vacuum connection was removed and the liner cut open. The resin-impregnated mat was transferred to an 8-mm–thick Perspex sheet, which was then placed in a cold press. Spacers of 6-mm thickness were positioned at the edges of the mat and the press closed until the top platen reached the spacers. The press was left closed overnight at room temperature to allow partial cure of the resin. The press was then released, the plate dismantled, and the composite sheet placed in an oven set at 80°C for 18 h in order to post-cure the resin. Specimens for tensile and flexural tests were cut from the composite sheets. A full description of the test methodologies has been given elsewhere.[9] Fibers for the reinforcement were used as received, but were subjected to solvent extraction before use. Fibers were then used without further treatment, acetylated (to 10% weight percent gain) [Fig. 1(a)], or treated with silane (γ-methacryloxypropyltrimethoxy silane; Aldrich Chemical Company, Gillingham, Dorset, UK) [Fig. 1(b)] or titanate [neopentyl(diallyl)oxy tri(dioctyl)pyro-phosphate titanate; Lica 38, Kenrich Petrochemicals Inc., Bayonne, NJ] [Fig. 1(c)] coupling agents. Full experimental details have been given in a separate article.[9] Glass fiber reinforced composites and nonreinforced resin specimens were also prepared for comparison. All composites in this study had a fiber content of 45% by weight.

Biodegradation Test

Biodegradation tests were performed for a total of 12 months, according to BS standard EN ISO 846: 1997 (Plastics-evaluation of the action of microorganisms). The samples were completely buried in John Innes no. 2 soil, at 90% water holding capacity and a 50% moisture content. The samples were in constant contact with the soil and exposed in jars located in a controlled environment at a temperature of 29°C and a relative humidity of 97%. Two specimen batches were prepared (12 replicates), samples exposed in unsterilized soil, and samples buried in sterilized soil. All specimens were vertically buried in sterilized glass jars, which were placed in Perspex containers. The jars were suspended above a saturated solution of potassium sulfate, in order to maintain the required humidity. To ensure an adequate supply of oxygen, the lid of the container was not tightly closed, but a loop of 1-mm diameter wire was placed between the cover and the container. The pH of the aqueous soil extract (1 g of soil in 100 g of water) was between 4 and 7. Assessment was performed on sample batches at 3-, 6-, and 12-month intervals. Tensile properties were performed on an Instron model 4301, according to BS2782: part 10: method 1003: 1977. EN61: determination of tensile properties. Flexural properties were determined according to BS2782: part 3: method 335A: 1978: ISO 178-1975. Plastics: determination of flexural properties of rigid plastics. Full experimental details have been given in an accompanying article.[9] Additionally, mass loss, mass change, and sample moisture content due to exposure, were determined.

Before exposure, all samples were dried in an oven at 45°C for 5 h, transferred to a desiccator containing silica gel for 10 min, then weighed on a four-figure balance. After the required exposure

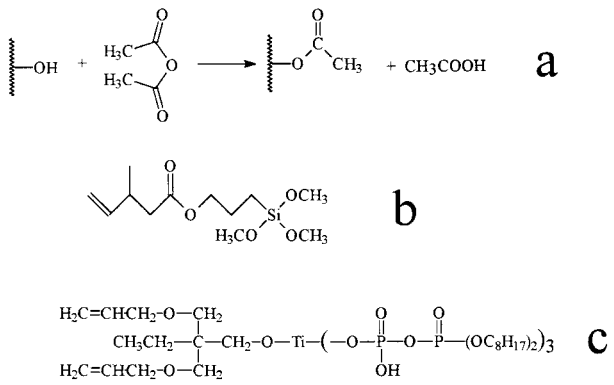

Figure 1 Reaction scheme for acetylation (a), structure of γ-methacryloxypropyltrimethoxy silane (b), and neopentyl(diallyl)oxy tri(dioctyl)pyro-phosphate titanate (c).

Table I Mass Loss and Mass Change for Samples after Exposure in Soil Burial Tests

Fiber	Exposure (Months)	Unmodified	Acetylated	Silane	Titanate	Resin	CSM
		Mass Loss (%)					
efb	3	5.78	0.69	0.00	2.81	0.02	0.07
	6	8.95	1.78	3.00	7.88	0.03	0.68
	12	17.12	3.87	6.04	14.76	1.44	2.16
coir	3	3.88	0.05	0.00	3.99		
	6	6.29	1.96	3.88	6.94		
	12	15.20	2.17	5.79	15.54		
		Mass Change (%)					
efb	3	4.45	0.56	0.00	2.32	0.00	0.03
	6	6.57	0.74	1.93	4.59	0.02	0.08
	12	11.06	1.91	3.03	11.40	0.27	0.94
coir	3	3.17	0.00	0.00	3.30		
	6	3.94	0.52	1.84	4.06		
	12	10.59	1.16	3.66	11.79		

time, samples were removed from the specimen jars using forceps, carefully cleaned of soil using a sable brush, weighed, and then transferred to an oven to dry at 45°C for 5 h. Samples were transferred to a desiccator to cool before reweighing. Mass loss percentage because of exposure was determined according to:

$$M_1\ (\%) = ((M_o - M_e)/M_o) \times 100 \qquad (1)$$

where M_o is the original sample weight, and M_e is the sample weight after soil exposure. Because mass loss may be due to a combination of leaching into the soil and due to the action of micro-organisms, mass change percentage was also calculated. This was determined according to:

$$M_c\ (\%) = (M_u/M_{ou} - M_s/M_{os}) \times 100 \qquad (2)$$

where M_u is the mass loss of the sample exposed in an unsterile environment, M_s the mass loss due to exposure in a sterile environment, M_{ou} and M_{os} are the original masses of the samples exposed in the unsterile and sterile environments, respectively. Percentage moisture content of samples was also calculated according to:

$$MC\ (\%) = ((M_{wet} - M_{dry})/M_{dry}) \times 100 \qquad (3)$$

where, M_{wet} is the weight of the sample after exposure and M_{dry} is the oven-dry weight of the same sample.

Hygrothermal Aging

Samples were placed in screw top containers containing deionized water that had been sterilized by boiling. The containers were autoclaved before use and the screw top lids had a small hole bored into them into which was inserted a plug of sterilized glass wool. Specimens (12 replicates) were removed for flexural testing at 3-, 6-, and 12-month intervals.

RESULTS AND DISCUSSION

Mass Loss and Mass Change

Mass loss and mass change were determined for all of the samples, and compared with results obtained for the cast resin and glass fiber reinforced composites. Results for mass loss and mass change are presented in Table I. The magnitude of mass loss decreased in the order: unmodified fiber > titanate treated > silane treated > acetylated > glass fiber (CSM) > cast resin. Mass losses of composites reinforced with acetylated or silane treated fibers were substantially lower than those observed with samples containing un-

Table II Moisture Contents (%) of Coir and efb Reinforced Composites after Exposure in Sterile or Unsterile Soil Burial Tests

Fiber	Duration (Months)	Unmodified	Acetylated	Titanate	Silane	Glass Fiber	Cast Resin
			Unsterile Tests				
efb	3	11.3	4.7	8.1	6.5	0.8	0.3
	6	12.1	5.5	9.7	7.8	1.1	0.5
	12	12.5	6.6	10.5	8.3	1.2	0.8
coir	3	10.8	4.4	9.3	6.4		
	6	11.7	4.7	10.3	7.5		
	12	11.3	5.8	9.9	7.8		
			Sterile Tests				
efb	3	5.8	2.4	4.1	3.4	0.6	0.3
	6	6.2	2.8	6.2	3.4	0.8	0.4
	12	6.3	3.3	6.3	4.8	0.9	0.6
coir	3	4.2	2.7	5.1	3.7		
	6	5.4	2.8	5.4	3.9		
	12	5.8	3.0	5.7	4.3		

treated or titanate treated fibers. In the case of composites made from acetylated fibers, the mass losses due to exposure were of the order of 2–4% after 12 months' exposure. These values were only slightly higher than those exhibited by the CSM reinforced composites (1–2%). Because such mass losses may occur due to the combined effects of leaching and biological attack, the mass changes were also determined. In this case, the mass changes exhibited by cast resin and CSM reinforced composites were negligible, indicating that there was no significant biological attack on the matrix during these tests. Both unmodified and titanate treated fiber reinforced composites exhibited mass changes of the order of 10%, after 12 months exposure. Samples reinforced with acetylated fibers exhibited very low mass changes of less than 2%, even after 12 months' exposure, indicating that chemical modification afforded significant protection to the natural fibers. With silane treatment, the composites showed mass changes of ca. 3–4%, which were slightly higher than found with acetylated fibers; but it is apparent that such treatment was also able to provide substantial protection against decay fungi. In a study of jute reinforced polyester matrix composites exposed to fungal degradation, weight losses of 1.6–1.7% were reported after 12 weeks' exposure.[10] The results of the present study show that extended exposure times in such tests can result in substantial weight losses, if unmodified fibers are used as reinforcement.

Moisture Content

Similar trends were noted with the end moisture contents of samples after 12 months' soil burial (Table II). These mirrored the trend found with mass loss and mass change, in that unmodified fiber reinforced composites showed the highest moisture contents, and acetylated the lowest. Differences were also found in the moisture content of samples exposed to sterile and unsterile environments with all of the natural fiber reinforced composites. The differences in the moisture contents between the sterile and unsterile tests are directly attributable to the actions of decay organisms. The higher moisture content observed in unsterile tests is due to degradation by the fungi resulting in an increased porosity of the samples, combined with the ability of fungi to facilitate transport of water into the composites via the mycelial network.[11] The protection afforded by acetylated fibers was expected, since it is known that decay resistance of wood is markedly improved by acetylation.[8] However, silane treatment also appeared to provide protection. Previous studies of wood treated with propyltrimethoxy silane indicated that such treatment did not afford significant decay protection to the material.[12] It is unlikely that any reaction occurred between the wood OH groups and the silane. Furthermore, the lack of any polymerizable functionality associated with this silane would ensure that the coupling agent would be leached in moist environments.

Table III Change in Tensile Properties during Unsterile Soil Burial Test

Fiber	Duration (Months)	Unmodified	Acetylated	Silane	Titanate	Resin	CSM
				σ (MPa)			
efb	0	35.1	37.5	36.8	34.5	25.1	94.0
	3	34.5	37.5	35.1	33.5	25.4	92.9
	6	31.3	38.0	35.6	33.9	24.6	93.2
	12	24.6	35.3	33.6	26.1	25.3	91.8
coir	0	39.8	40.5	36.6	38.4		
	3	38.2	41.3	36.7	36.3		
	6	34.7	38.8	35.9	36.1		
	12	29.0	39.0	33.1	30.0		
				E (GPa)			
efb	0	3.29	3.70	3.60	3.33	2.59	5.76
	3	3.25	3.62	3.61	3.09	2.61	5.63
	6	2.88	3.65	3.64	3.09	2.48	5.71
	12	2.32	3.44	3.60	2.55	2.60	5.46
coir	0	3.60	4.17	4.17	3.59		
	3	3.69	4.10	4.13	3.34		
	6	3.39	3.96	4.12	3.04		
	12	2.82	3.89	3.89	2.55		
				ε (%)			
efb	0	3.75	3.48	3.60	3.65	2.98	2.10
	3	3.63	3.27	3.63	3.62	2.89	2.12
	6	3.18	3.56	3.40	3.28	2.79	1.94
	12	2.48	3.30	3.49	2.91	2.92	2.02
coir	0	5.20	4.54	4.50	4.74		
	3	4.63	4.42	4.39	4.10		
	6	3.50	4.60	4.33	3.95		
	12	3.45	4.40	3.91	3.38		

Mechanical Tests

The results of tensile tests are presented for efb and coir reinforced composites in Table III. The losses in tensile strength, modulus, and elongation also follow the trends observed for mass loss and mass change. Both fiber acetylation and silane treatment were able to provide a substantial degree of protection after exposure. In the case of the fibers in this study, the matrix will provide a barrier to the penetration of the fungal hyphae, which are then forced to grow along the length of the fibers, provided the matrix remains intact. Because the samples used in this study were not edge sealed, this would not prove to be a problem with untreated fibers. Additionally, exposing such composites to a moist environment would result in swelling of the fibers, resulting in fiber-matrix debonding occurring. In this way, additional routes for fungal penetration are created. As noted in the previous section, acetylation of lignocellulosics is able to provide protection against fungal attack. This protection is due to the lower moisture content of the modified material caused by a decrease in the number of hydrophilic OH groups in the cell wall. Additionally, it has been postulated that substitution of the OH groups with acetyl moieties renders the cell wall polymers unrecognizable to the enzymes associated with the fungi.[13] The improved properties of composites reinforced with acetylated fibers are thus readily explained. The observation that silane treatment also affords significant protection also

Table IV Variation in Flexural Properties during Unsterile Soil Burial Test

Fiber	Duration (Months)	Unmodified	Acetylated	Silane	Titanate	Resin	CSM
				σ_f (MPa)			
efb	0	41.6	41.8	38.8	38.3	50.9	180.0
	3	40.8	42.2	39.6	37.5	49.7	178.5
	6	38.9	40.1	36.4	35.7	51.1	175.8
	12	27.4	38.8	36.6	33.8	51.7	175.7
coir	0	43.7	43.2	40.5	42.0		
	3	41.3	42.5	41.0	41.1		
	6	3.5	42.7	39.8	37.1		
	12	31.7	41.4	40.7	37.9		
				E_f (GPa)			
efb	0	3.85	4.57	4.46	4.04	3.23	6.11
	3	3.58	4.54	4.46	3.97	3.26	6.15
	6	2.95	4.41	4.37	3.75	3.26	6.00
	12	2.69	4.38	4.16	3.26	3.24	5.90
coir	0	4.11	4.97	4.62	4.12		
	3	4.13	4.86	4.66	4.15		
	6	4.05	4.84	4.68	3.79		
	12	3.17	4.78	4.32	3.34		

requires explanation. In a study of the impregnation of wood with a methanolic solution of γ-methacryloxypropyl trimethoxy silane, it was found that swelling of the substrate occurred, indicating that the coupling agent was able to penetrate into the cell wall.[14] Furthermore, it was found that this swelling of the substrate was retained after four water soaking cycles, indicating that this silane was permanently bonded in the cell wall. It has been suggested that a reaction can occur between organosilane coupling agents and the cell wall OH groups.[15] If this does occur, then the resultant Si—O—C bond will not be hydrolytically stable, although it is possible that the long nonpolar chain attached to the Si center prevents close approach of water molecules. There is also the opportunity for polymerization of the silane to occur via the methacryloxy group, thereby preventing leaching of the coupling agent once penetration into the cell wall has occurred. The presence of the coupling agent within the cell wall would lead to masking of a proportion of the cell wall OH groups, thereby reducing the moisture content. With silane treatment, it is possible that the lower moisture content of the fibers coupled with more efficient fiber-matrix bonding is able to provide protection against biological decay. At present this is speculative and indicates the need for more research into the interaction of coupling agents with plant fibers, an area that is beginning to receive attention.[16] With the titanate treatment, the protection which does occur is not so effective as with silane. This may indicate that cell wall penetration is not occurring to the same extent, or that the coupling agent is leached more effectively. The larger molecular size of the Lica 38 compared with the silane used in this study may preclude cell wall penetration by the former.

Flexural properties of the composites also deteriorated when samples were exposed in soil burial tests (Table IV). Whereas, no changes in properties were observed when unreinforced resin samples were exposed, all of the composites exhibited deterioration to varying extents. In the case of glass fiber reinforced composites, this is attributed to fiber-matrix debonding. With natural fiber reinforced composites, degradation of the fibers will also contribute to varying extents depending on fiber treatment. Both acetylation and silane treatment of fibers again provided a significant degree of protection. Titanate treatment of fibers also improved the

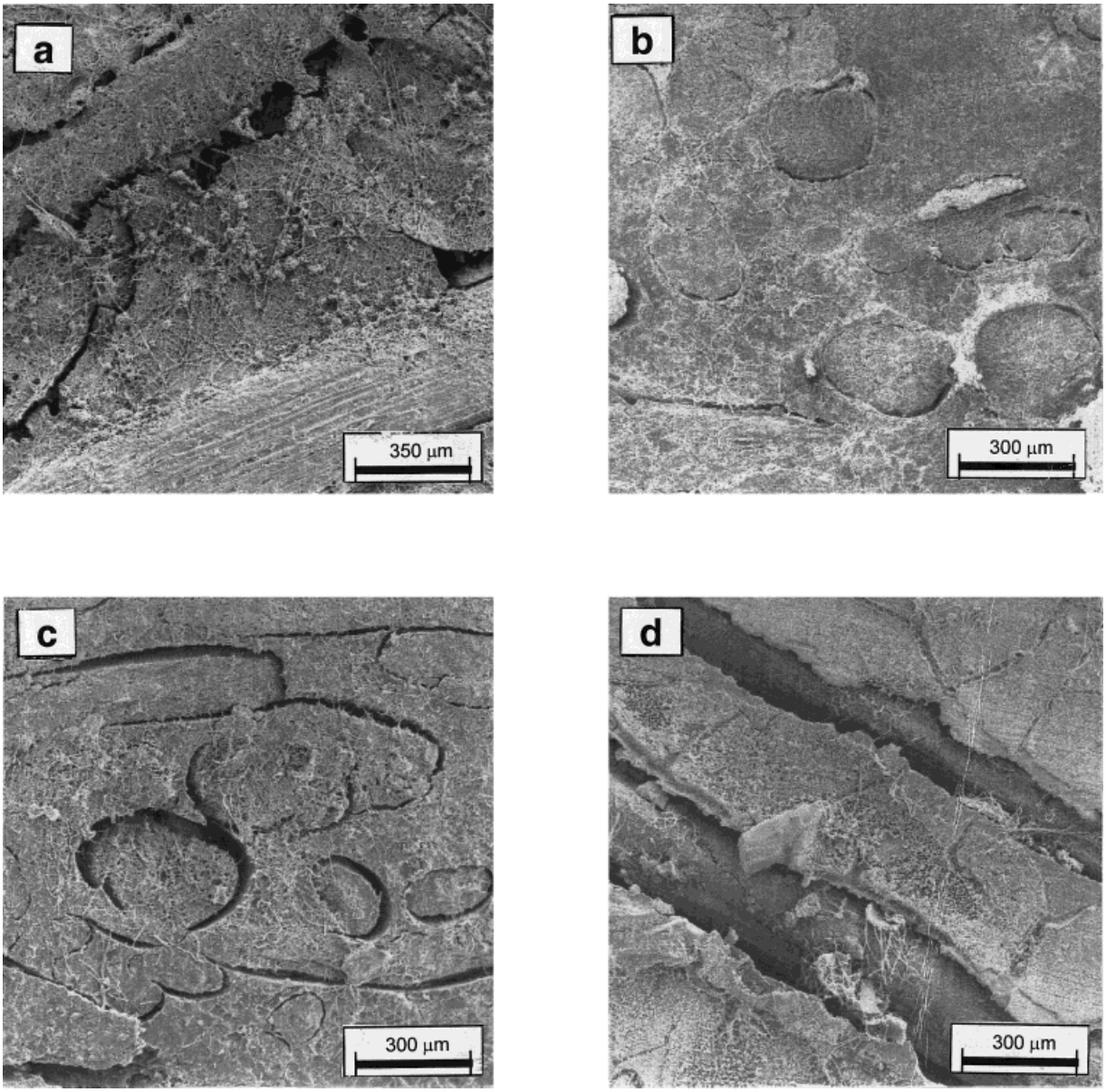

Figure 2 SEM micrographs showing efb composites after 12 months' unsterile soil exposure: surface of unmodified fiber composite (a), surface of acetylated fiber composite (b), surface of silane-treated fiber composite (c), surface of titanate-treated fiber composite (d).

performance of the composites in these tests, although to a lesser extent than that observed with silane treatment. In flexural tests, the composite is exposed to fiber-matrix shear forces to a greater extent than with tensile tests. The influence of fiber properties is thus of less significance. The difference in behavior of titanate treated samples between tensile and flexural tests may also be an indication that little protection is afforded to the fibers when using such treatment.

SEM Studies

Figure 2 shows SEM micrographs of efb reinforced composites exposed in unsterile soil tests for 12 months. In Figure 2(a), the surface of a composite reinforced with unmodified fibers is shown. There is clear evidence of cracking of the matrix and the presence of extensive mycelial networks. This contrasts with Figure 2(b), which shows a composite reinforced with acetylated fi-

Table V Variation in Flexural Properties during Water Soaking at 20°C

Fiber	Duration (Months)	Unmodified	Acetylated	Silane	Titanate	Resin	CSM
		σ_f (MPa)					
efb	0	41.6	41.8	38.8	38.3	50.9	180.1
	3	38.7	44.2	39.0	37.7	49.8	180.0
	6	37.2	40.6	37.9	36.9	49.7	175.0
	12	35.3	39.5	36.7	35.5	49.5	174.1
coir	0	43.7	43.2	40.5	42.0		
	3	41.9	46.3	42.4	39.6		
	6	39.4	41.9	38.4	39.6		
	12	38.4	40.8	38.1	34.8		
		E_f (GPa)					
efb	0	3.85	4.57	4.46	4.04	3.23	6.11
	3	3.49	4.67	4.52	3.73	3.22	6.02
	6	3.19	4.41	4.29	3.65	3.08	5.77
	12	2.92	4.31	4.04	3.29	3.02	5.74
coir	0	4.11	4.97	4.62	4.12		
	3	3.86	5.22	4.42	3.87		
	6	3.57	4.87	4.32	3.71		
	12	3.35	4.58	4.12	3.22		

bers. In this case, there is limited degradation of the matrix and limited evidence of mycelial networks. The composite reinforced with silane treated fibers [Fig. 2(c)] shows more extensive matrix degradation than in the previous case, with the presence of some mycelial networks. The composite reinforced with titanate treated fibers shows extensive degradation [Fig. 2(d)]. The surface is severely cracked, with the fibers clearly visible. Mycelial networks are also in evidence, some of which can be seen associated with the fibers.

Hygrothermal Aging

Table V shows the changes in flexural properties occurring as a result of soaking in deionized water at 20°C. A decrease was noted with all of the samples studied, which was proportional to time of exposure and largest with composites reinforced with unmodified natural fibers. After 3 months' exposure, composites made from acetylated fibers exhibited a small increase in flexural strength (6% efb, 7% coir) and modulus (2% efb, 5% coir). It has been reported that the tensile strength of sisal reinforced polyethylene matrix composites increased after 1 days' exposure to water at 80°C. This was explained as due to an annealing process occurring.[17] It is possible that a similar mechanism is responsible for the results observed with these experiments. After 12 months' exposure to water, all samples exhibited losses in both flexural strength and modulus. With efb reinforced samples, the losses were greatest with unmodified fibers and decreased thereafter in the order: titanate treated > silane treated > acetylated. Strength losses after this exposure period were comparable for acetylated and silane treated fiber reinforced composites (5.5 and 5.4%, respectively). Strength losses of 7.1 and 15.1% were recorded for titanate treated and untreated efb fiber reinforced composites. Losses in modulus were 5.7, 9.5, and 18.5 % for acetylated, silane, and titanate treated, respectively. Similar results were recorded with coir reinforced composites. However, in this case titanate treatment of the fibers resulted in the composites exhibiting higher strength (17.1%) and modulus losses (21.7%) than those observed with untreated fibers (strength loss 12.1% and modulus loss 18.6%).

With untreated cellulosic fibers, water is readily absorbed due to the hydrophilic nature of the material. As a consequence, the fiber cell wall

swells, which may also be accompanied by rotation of the fiber.[18] This in turn results in shear stresses occurring at the fiber-matrix interface, with associated debonding leading to a strength decrease of the composite. It is known that the strength of wood decreases when it is exposed to a moist environment.[19] However, acetylation of plant fibers results in a slight increase in strength, due to a reduction of the equilibrium moisture content of the modified fibers.[9] Acetylation of the fibers is accompanied by cell wall swelling, due to the volume that the chemically bonded reagent occupies in the cell wall.[20] When such a fiber is exposed to moisture, there will thus be little additional swelling. Acetylation will also reduce the rate and extent of water uptake and improve the compatibility between the fiber and matrix, due to the increased hydrophobicity of the modified lignocellulosic.[21] Coupling agent treatment would be expected to improve the interfacial bonding between the fiber and the matrix, but the extent to which the fiber is swollen or rendered hydrophobic will depend on the penetration of the coupling agent into the fiber cell wall.[22] This combination of dimensional stability and increased hydrophobicity of the treated fiber will result in lower strength losses, when a composite containing these fibers is exposed to moisture. Such protection arises because of a greater retention of the dry strength properties of the fiber and a reduced propensity for fiber-matrix debonding to occur.

CONCLUSIONS

Acetylation of coir or oil palm empty fruit bunch fibers for use as reinforcement in polyester matrix composites has been shown to result in good retention of mechanical properties during soil or water exposure tests. Water sorption is substantially reduced and structural integrity maintained, in contrast to composites in which no fiber treatment is used. Silane treatment of fibers was also found to afford good protection to composites formed therefrom. Titanate treatment was not found to be as effective as silane in this respect. In view of the dramatic decrease in mechanical properties observed when untreated fiber reinforced composites are exposed to environmental stresses, some form of fiber treatment would appear to be essential. Since acetylation provided only a marginal benefit over silane treatment as a means of protection, it is recommended that the latter method is used as a lower cost option.

The authors thank Mr. Andrew Davies for performing the SEM work; the Universiti Sains Malaysia, Penang Malaysia for the award of a PhD fellowship to HPSAK; Sabutek Sdn. Bhd. Malaysia for supplying EFB fibers; Mr. Gary Newman of JB Plant Fibres Ltd. for manufacture of nonwoven fiber mats; and Dr. Jamie Hague of the BioComposites Centre Bangor for helpful discussions and supply of coir fiber. We also thank Kenrich Petrochemicals Inc. for the gift of the titanate coupling agent.

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