Chemical Resistance, Void Contents, and Morphological Properties of *Hildegardia* Fabric/Polycarbonate-Toughened Epoxy Composites

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ABSTRACT: A uniaxial natural fabric of *Hildegardia populifolia* was used as a reinforcement for a polycarbonate-toughened epoxy. The *Hildegardia* fabric was treated with a 5% sodium hydroxide solution for 1 h. The fabric was spray-coated with a 1% silane-based coupling agent. The variation of the chemical resistance and void content with different fabric contents and fiber orientations was studied. The morphology of the fractured composites was investigated with scanning electron microscopy (SEM). SEM micrographs indicate that the bonding between the *Hildegardia* fabric and the matrix was enhanced partially by the alkali treatment. The alkali treatment in the presence of the silane coupling agent gave rise to matrix skin formation on the surface of the fibers, which indicated good bonding between the reinforcement and the matrix. *Hildegardia*/polycarbonate-toughened epoxy composites were found to have reasonable chemical and water resistance. The liquid absorption increased when the fabric was treated with an alkali, when the coupling agent was used, and in the presence of water and aqueous solutions. The void content of the composites decreased with increasing fabric content. © 2007 Wiley Periodicals, Inc. J Appl Polym Sci 106: 3945–3951, 2007

Key words: composites; electron microscopy; matrix; orientation; polycarbonates; reinforcement; renewable resources

INTRODUCTION

The structure and properties of the fiber–matrix interface play a major role in controlling the mechanical and physical properties of composite materials. To use the excellent strength and stiffness properties of fibers, they should have good bonding characteristics with the matrix. Composite materials with weak interfaces have relatively low strength and stiffness but high resistance to fracture, whereas materials with strong interfaces have high strength and stiffness but are brittle. The nature of the bonding between the matrix and the fiber is dependent on the chemical properties of the fiber and the finish (coupling agent) used. In our previous articles,[1,2] we reported the effects of alkali and silane coupling agent treatments on the tensile and flexural properties of *Hildegardia*-fabric-reinforced, polycarbonate-toughened epoxy composites. We found better mechanical properties when alkali and coupling agent treatments were used.

The main reason for the interest in fiber-reinforced polymers for structural applications is the high specific strength and/or modulus of the reinforcing fibers. Glass fibers are most commonly used as reinforcements, followed by carbon and boron fibers. Carbon and boron fibers are prohibitively expensive, and their use is justified only in aerospace and defense applications. To probe whether a composite can find applications in chemical and water storage tanks, its chemical resistance properties have to be studied. Rajulu and coworkers[3–8] studied the chemical resistance of bamboo fibers coated with epoxy and styrenated polyester as well as epoxy/polycarbonate, epoxy/poly(methyl methacrylate), and epoxy/unsaturated polyester blends. They reported improvements in both the tensile strength and chemical resistance of the composites. They further reported that the water absorption by the composites decreased drastically.

The performance of composites depends on three important factors: (1) the aspect ratio of the reinforcement, (2) the hydrophobicity of the reinforcement, and (3) proper bonding between the reinforcement and the matrix.[9] Several investigators[10–22] have used scanning electron microscopy (SEM) to study
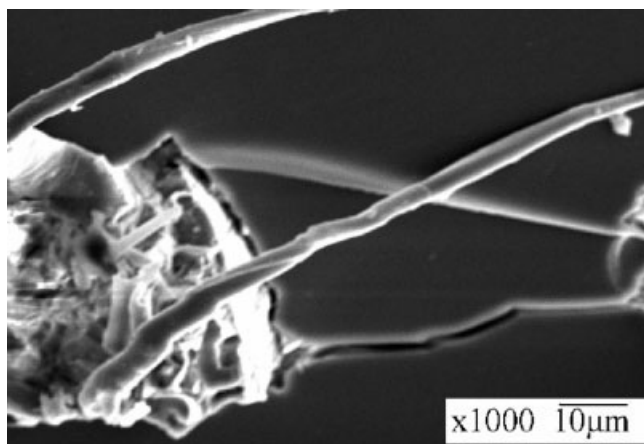
Figure 1 SEM photograph of an untreated *Hildegardia*-fabric-reinforced epoxy/polycarbonate blend without a coupling agent at a magnification of 1000×.

the interface between reinforcements and matrix materials. The performance of composites also depends on their void content. Several studies on composites made from epoxy matrices and natural fibers such as jute, wood, bamboo, sisal, cotton, coir, and wheat straw have been reported in the literature.[23–29] In this work, we made *Hildegardia* fabric/polycarbonate-toughened epoxy composites and studied their chemical resistance and void contents with various fiber contents and orientations. The morphology of the fractured surfaces of the composites was studied.

EXPERIMENTAL

Materials

The epoxy resin, Araldite LY-556, and the curing agent, Hardener HY-951, were supplied by Ciba–Geigy (Mumbai, India). Araldite LY-556 is a clear liquid with a viscosity at 25°C in the range of 9000–12,000 mPa s.[30] The epoxy in the resin varies between 5.2 and 5.45 equiv/kg. Hardener HY-951 is a clear liquid with a viscosity at 25°C in the range of 5000–11,000 mPa s. Dow Chemical Co. (Mumbai, India) supplied the polycarbonate. It had a melt flow index of 15 g/min (at 300°C and 1.2 kg). Dichloromethane, used as a solvent in the preparation of the blend, was supplied by S.D. Fine Chemicals (Gujarat, India). A silicon-based silane coupling agent supplied by Aldrich Chemical Co. (Bangalore, India) was used.

The naturally occurring fabric *Hildegardia populifolia* was used as the reinforcement. This fabric was procured from Kadiri Taluk (Andhra Pradesh, India). This fabric exists under the bark of the trunk and branches of the tree. The fabric was removed from the branches of the tree. The tree belongs to

the *Sterculiaceae* family. The average length, width, and thickness of the fabric were found to be 300–700 cm, 70–100 cm, and 0.18 mm, respectively. The extracted fabric, which was naturally woven, was dried in the sun for a long time until it was completely dry. The weaving appeared to be intermingled. The dry fabric was treated with a 2% aqueous NaOH solution to remove the soluble hemicellulose and lignin. After this, the fabric was washed with water and dried in the sun. The properties of this fabric have been discussed elsewhere.[31]

Methods

For making the composites, a steel die mold with dimensions of $50 \times 15 \times 3$ mm^3 was used. The mold cavity was coated with a thin layer of an aqueous solution of poly(vinyl alcohol), which acted as a good releasing agent. A 90/10 (w/w) epoxy/polycarbonate blend was prepared by the addition of the epoxy resin to the polycarbonate dissolved in dichloromethane. The solvent was removed through degassing in a vacuum for about 1 h. To this, the hardener was added in a stoichiometric ratio. The blend was used as the matrix for composites prepared through the variation of the orientation (15, 30, 45, 60, 75, and 90°) of the fabric and the fabric content (15, 20, 25, 30, 35, and 40 w/w). The different angles of orientation were achieved by samples being cut at different angles from the molded composite. We took a piece of *Hildegardia* fabric that was approximately 150×100 mm^2 and inserted it layer by layer into the mold; at the same time, the matrix material was also added until the mold was filled with the fabric and matrix.
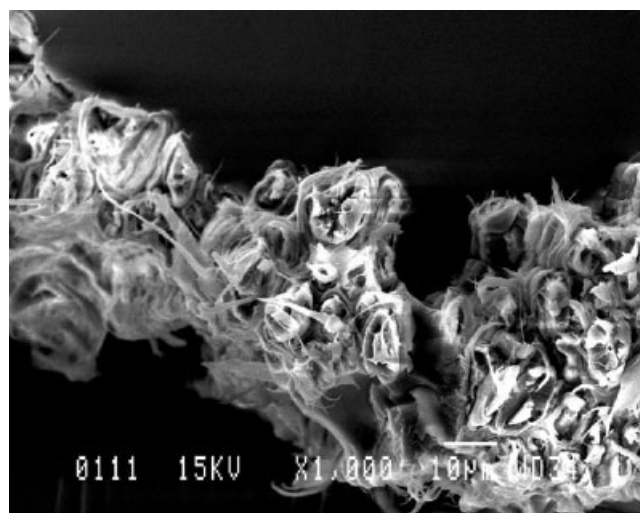

Figure 2 SEM photograph of an untreated *Hildegardia*-fabric-reinforced epoxy/polycarbonate blend with a coupling agent at a magnification of 1000×.
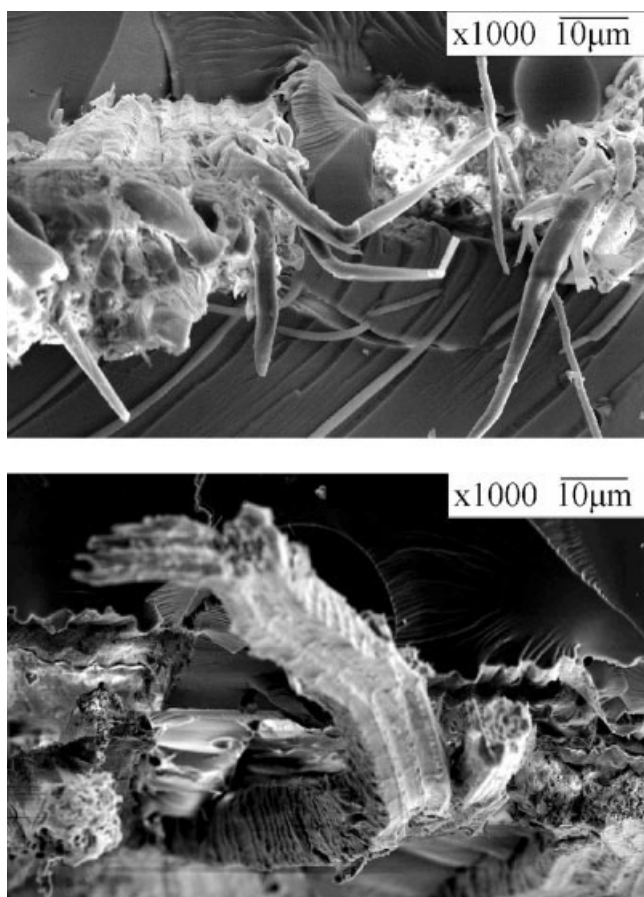
Figure 3 SEM photographs, taken at two different positions, of an alkali-treated, *Hildegardia*-fabric-reinforced epoxy/polycarbonate blend without a coupling agent at a magnification of 1000×.

We then covered the mold with a Teflon sheet and kept it in a vacuum oven overnight to remove the bubbles. The composites were cured at room temperature for 24 h, after which the sheets were removed from the mold and postcured at 100°C for 3 h. The same method was repeated with a silane coupling agent dissolved in acetone and sprayed onto the reinforcing material.

For chemical resistance, the specimens were made and tested per ASTM D 543-87.[32] The preweighed specimens were immersed in different chemicals for 24 h, removed, rinsed in distilled water, and immediately dried by being pressed between two pieces of filter paper. The weight of the samples was then determined. The weight gain or loss (%) was determined gravimetrically. In each case, 10 samples were used, and the average value is reported. In all cases, the 10 values did not differ from one another by more than 10%.

For the determination of the void contents of the composites, the ASTM D 2734-70 method[1] was employed. This method can be applied only to com-

posites in which the matrix reinforcement and any added filler burn off completely. In this case, both the matrix and reinforcement were totally combustible, so the method was employed. The densities of the resin, the reinforcement, and the composites were measured separately. Then, the theoretical composite density (T_d) was calculated with the following equation:[1]

$$T_d = 100/(R/D + r/d)$$

where R is the weight of the resin in the composite, D is the density of the resin, r is the weight of the reinforcement in the composite, and d is the density of the reinforcement. This was compared with the measured density. The void content $[V (\%)]$ was calculated with the following equation:[25]

$$V = [(T_d - M_d)/T_d] \times 100$$
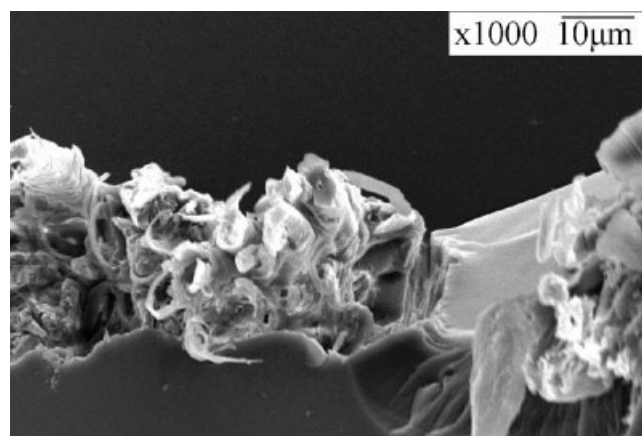
(a)
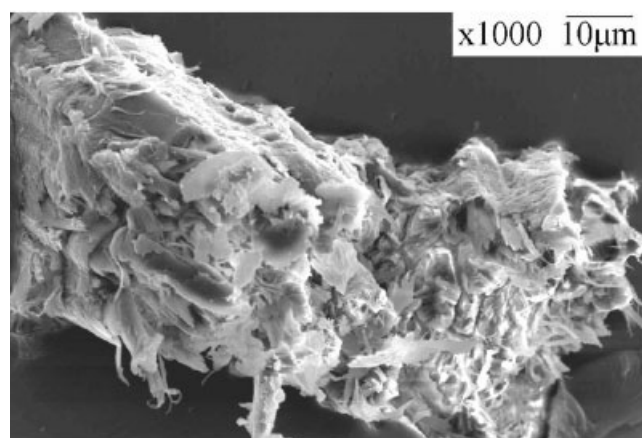
(b)

Figure 4 SEM photographs of an alkali-treated, *Hildegardia*-fabric-reinforced epoxy/polycarbonate blend with a coupling agent at a magnification of 1000×: (a) perpendicular to the fiber axis and (b) parallel to the fiber axis.

TABLE I
Weight Loss and Gain of Composites with Various Fiber Orientations upon Immersion in Different Chemicals for 24 h

	Orientation of the fabric (°)											
	15				30				45			
	Untreated		Treated		Untreated		Treated		Untreated		Treated	
Chemical	WOCA	WCA	WOCA	WCA	WOCA	WCA	WOCA	WCA	WOCA	WCA	WOCA	WCA
C_6H_6	0.9	1.0	1.7	1.9	0.9	0.3	0.6	0.8	2.3	2.1	0.1	0.07
$C_6H_5CH_3$	0.7	0.4	1.9	1.0	2.4	1.3	1.4	2.9	1.9	2.5	0.6	0.5
CCl_4	1.0	1.3	0.1	0.2	1.3	1.2	1.5	2.0	2.6	2.9	1.0	1.2
Na_2CO_3 (20%)	5.8	4.0	8.4	8.0	9.1	5.5	4.3	9.6	6.3	5.5	6.1	5.0
NH_4OH (10%)	6.0	7.3	7.8	8.1	12.2	5.7	6.1	10.2	11.4	12.7	5.4	7.6
NaOH (10%)	8.6	12.0	8.4	10.1	10.7	8.5	7.5	10.9	11.7	13.0	10.3	8.4
CH_3COOH (8%)	6.3	6.2	9.3	8.4	7.8	7.5	7.6	10.8	6.9	7.8	8.4	6.8
HCl (10%)	5.1	8.7	8.7	8.0	9.9	5.7	5.7	5.4	7.2	6.9	3.0	3.8
HNO_3 (40%)	6.3	8.6	12.4	14.3	20.7	8.4	7.4	12.9	15.3	17.8	4.3	6.0
H_2O	6.1	7.6	8.0	10.4	11.2	2.8	7.8	9.5	9.5	8.4	3.5	6.7

	Orientation of the fabric (°)											
	60				75				90			
	Untreated		Treated		Untreated		Treated		Untreated		Treated	
Chemical	WOCA	WCA	WOCA	WCA	WOCA	WCA	WOCA	WCA	WOCA	WCA	WOCA	WCA
C_6H_6	0.9	1.3	0.8	1.1	0.4	1.0	1.1	1.1	1.6	1.0	1.4	1.5
$C_6H_5CH_3$	1.2	1.7	1.0	1.2	1.8	2.1	0.4	0.6	2.2	0.7	0.9	1.0
CCl_4	0.5	0.7	0.8	0.9	0.2	0.8	1.0	0.9	0.6	1.4	1.2	1.4
Na_2CO_3 (20%)	8.2	6.0	1.4	1.0	5.3	7.8	1.4	1.2	6.7	5.8	3.4	2.8
NH_4OH (10%)	4.6	5.2	7.0	9.1	4.5	10.3	9.4	10.6	4.5	7.8	8.2	6.3
NaOH (10%)	10.2	16.3	9.4	10.3	6.6	10.8	8.9	9.9	10.6	15.7	3.4	8.4
CH_3COOH (8%)	4.8	5.2	6.3	7.1	5.0	13.5	12.3	12.8	11.9	12.1	8.4	10.4
HCl (10%)	8.9	8.5	5.7	5.0	3.2	9.3	3.9	3.2	10.9	5.5	3.6	2.9
HNO_3 (40%)	10.4	12.2	8.3	9.7	8.6	23.7	11.4	13.5	15.1	6.3	6.3	7.4
H_2O	7.8	9.8	6.1	6.8	4.7	11.7	7.3	8.7	6.2	11.2	5.7	8.3

The fabric content was 40 wt %. WOCA = without coupling agent; WCA = with coupling agent.

where M_d is the measured composite density. In each case, 10 samples were tested, and the average value is reported. Here also the individual values did not differ by more than 10%.

To observe the bonding between the reinforcement and matrix, samples that were not exposed to water or chemicals were cryogenically fractured. For SEM studies, the samples were coated with gold by a sputtering technique to ensure conductivity. The SEM studies were carried out on a JEOL (Tokyo, Japan) JSM-820 scanning electron microscope from Japan. The samples were photographed at different places on the surfaces and at magnifications of 220, 450, 800, and 1000×. Pictures obtained at a magnification of 1000× are presented in this article.

RESULTS AND DISCUSSION

Morphology

The bonding between the reinforcement and the matrix was studied with SEM, and all the SEM micro-

grams were taken on untreated, 40% *Hildegardia* fabric reinforced, polycarbonate (10%) toughened epoxy composites. The SEM photograph of a composite without a coupling agent is shown in Figure 1. This photograph reveals the pullout of the fiber from the matrix, indicating poor adhesion between the reinforcement and the matrix.

The SEM photograph of a composite with a coupling agent is presented in Figure 2. This photograph shows that the fibers were broken and not pulled out. This observation indicates an improvement in the bonding between the fibers of the *Hildegardia* fabric and the matrix when a coupling agent was used.

Composites were also made with alkali-treated fabric in the absence and presence of a silane coupling agent. Figure 3 shows two different SEM photographs of the alkali-treated *Hildegardia* fabric composite. Some fibers were pulled out of the matrix, but in some places, matrix skin formation on the fibers can be observed. These observations indicate that the bonding between the reinforcement and the matrix was partially improved, even in the absence of a cou-

TABLE II
Weight Loss and Gain of Composites with Various Fiber Contents upon Immersion in Different Chemicals for 24 h

	Weight of the fabric (%)											
	15				20				25			
	Untreated		Treated		Untreated		Treated		Untreated		Treated	
Chemical	WOCA	WCA	WOCA	WCA	WOCA	WCA	WOCA	WCA	WOCA	WCA	WOCA	WCA
C_6H_6	0.1	0.5	1.2	1.9	0.1	0.7	3.4	4.9	2.8	5.4	1.3	1.8
$C_6H_5CH_3$	0.2	1.0	5.4	7.3	0.2	1.0	4.4	2.7	1.4	1.1	1.1	1.0
CCl_4	1.2	0.9	3.6	2.9	0.2	0.1	5.4	6.2	0.2	0.1	0.1	2.1
Na_2CO_3 (20%)	3.5	3.0	8.4	7.9	3.8	3.1	13.3	12.5	7.1	9.3	10.4	9.1
NH_4OH (10%)	2.2	5.6	12.4	14.8	4.2	8.4	10.9	12.3	10.5	13.3	14.9	17.9
NaOH (10%)	7.5	6.9	10.5	11.6	4.9	6.3	10.4	12.3	18.5	10.4	17.1	18.2
CH_3COOH (8%)	4.4	4.7	10.2	10.7	4.5	4.8	5.8	6.0	10.4	10.2	16.4	15.9
HCl (10%)	5.1	5.8	15.6	16.4	4.0	4.7	9.7	9.8	9.2	10.2	13.9	13.9
HNO_3 (40%)	5.6	5.3	6.5	7.1	8.5	9.5	6.8	7.5	13.6	14.1	29.3	30.2
H_2O	4.2	4.9	11.6	11.5	4.4	4.1	5.8	6.1	10.5	10.4	15.8	15.4

	Weight of the fabric (°)											
	30				35				40			
	Untreated		Treated		Untreated		Treated		Untreated		Treated	
Chemical	WOCA	WCA	WOCA	WCA	WOCA	WCA	WOCA	WCA	WOCA	WCA	WOCA	WCA
C_6H_6	1.1	1.4	0.7	1.0	0.4	0.6	3.2	5.2	0.6	0.8	0.7	0.8
$C_6H_5CH_3$	2.2	1.1	0.2	0.1	0.3	0.2	0.1	0.1	0.9	1.0	0.9	0.8
CCl_4	0.8	1.2	0.7	0.9	0.5	0.7	1.0	1.1	0.4	0.6	1.8	2.2
Na_2CO_3 (20%)	12.1	11.3	11.8	9.6	7.6	7.1	13.1	12.7	4.9	4.4	7.0	6.8
NH_4OH (10%)	8.9	11.7	14.0	16.9	10.2	12.7	15.3	17.3	8.4	9.6	6.6	7.0
NaOH (10%)	12.6	13.2	15.6	16.1	12.0	12.7	13.8	13.9	6.3	8.0	7.3	9.6
CH_3COOH (8%)	16.5	15.9	20.2	19.6	9.3	9.6	15.1	15.7	7.3	7.9	6.3	6.9
HCl (10%)	13.9	12.4	12.8	12.9	4.0	3.8	14.6	15.4	9.0	7.1	4.6	3.8
HNO_3 (40%)	31.2	30.6	28.7	29.7	11.9	12.6	27.3	26.8	5.3	7.7	7.5	9.8
H_2O	15.0	15.8	14.8	15.4	5.6	6.5	12.4	12.5	3.1	5.1	7.2	9.4

The fabric orientation was 0°. WOCA = without coupling agent; WCA = with coupling agent.

pling agent when the fabric was treated with an alkali. Rajulu et al.[2] reported that, when *Hildegardia* fabric was treated with an alkali, the hemicellulose and some part of the lignin were removed, and as a result, the fibers became thin. The density of the fabric also increased with an alkali treatment.[2] The surface of the fibers in the fabric became rough with the removal of hemicellulose and lignin. All these factors contributed to the improvement of the bonding between the reinforcement and the matrix.

When the *Hildegardia* fabric was treated with an alkali and a coupling agent was used in the composites, an improvement in the bonding was observed (Fig. 4). Figure 4(a) shows that the fibers were broken and not pulled out. However, skin formation could not be seen as the microgram was taken in a direction perpendicular to the fiber axis. When the microgram was taken parallel to the fiber axis, matrix skin formation on the fiber surface could be seen [Fig. 4(b)]. These observations clearly indicate that the bonding between the reinforcement and the matrix was maximum when the fabric was treated with an alkali and a coupling agent was used.

Chemical resistance

The resistance of the composites to water and certain chemicals was studied. For this purpose, composite samples with different fiber orientations (15, 30, 45, 60, 75, and 90°) and fabric contents (15, 20, 25, 30,

TABLE III
Void Contents of Natural Fabric (*H. populifolia*) Reinforced, Polycarbonate-Toughened Epoxy Composites With and Without a Coupling Agent for Various Fabric Contents

	Void content (%)			
Weight of	Untreated fabric		Treated fabric	
the fabric (%)	WOCA	WCA	WOCA	WCA
15	3.8	3.8	3.8	3.9
20	3.4	3.7	3.4	3.7
25	3.0	3.2	3.0	2.9
30	2.9	2.8	2.9	2.8
35	2.3	2.0	2.0	2.0
40	1.6	1.6	1.6	1.6

The fabric orientation was 0°. WOCA = without coupling agent; WCA = with coupling agent.

TABLE IV
Void Contents of Natural Fabric (*H. populifolia*) Reinforced, Polycarbonate-Toughened Epoxy Composites With and Without a Coupling Agent for Various Fiber Orientation Angles

Orientation of the fiber (°)	Void content (%)			
	Untreated fabric		Treated fabric	
	WOCA	WCA	WOCA	WCA
0	1.6	1.6	1.6	1.6
15	1.6	1.6	1.6	1.7
30	1.6	1.6	1.6	1.6
45	1.5	1.6	1.7	1.6
60	1.6	1.6	1.6	1.6
75	1.6	1.7	1.6	1.6
90	1.6	1.6	1.6	1.7

The fabric content was 40 wt %. WOCA = without coupling agent; WCA = with coupling agent.

35, and 40 w/w) were selected. Three acids (HCl, HNO_3, and CH_3COOH), two alkalis (NaOH and NH_4OH), a salt (Na_2CO_3), and three commonly used solvents (C_6H_6, $C_6H_5CH_3$, and CCl_4) were used. The water absorption by these composites was also studied.

Table I presents the weight gain/loss (%) of the composites (in which the fiber content was varied) after immersion in different chemicals. These values are presented for the untreated and alkali-treated fabrics in both the absence and presence of the silane coupling agent. In all these cases, a weight increase was observed. This clearly indicates that the composites did not lose weight, and therefore it does not seem as if any erosion occurred. Generally, the weight increase was larger for water or aqueous solutions, and this was to be expected as a result of the hydrophilicity of the fiber. This conclusion is supported by the weight increase in the presence of these liquids with increasing fiber content, although the void content also seems to play a role (see the following discussion). Generally, the weight gain (%) was more when the fabric was treated with an alkali and when a coupling agent was used, although there are enough exceptions not to make this a general rule. In these cases, the —OH groups in the cellulose probably were more exposed, and this increased the hydrophilicity of the system. It seems as if fiber orientation had little effect on the liquid uptake for most of the investigated liquids (Table II). This was to be expected because all the samples were prepared in the same way, and the orientation was changed only by the cutting of the samples at different angles.

Void content

The void contents (%) of composite samples with different fiber contents (wt %) and different orientations

are presented in Tables III and IV. The void content was minimum at a 40% fiber content. For a good composite, the void content must be less than 3%.[1] Generally, a lower void content indicates improved bonding between the fabric and matrix. This is possible only when there is a strong interaction between the components. In our case, the lower void content at higher fiber contents caused lower liquid absorption by the composites (especially for water and aqueous solutions), despite the presence of more cellulose —OH groups due to the higher fiber content. Although the reason for the lower void content at higher fiber contents could not be conclusively established, lower liquid absorption could be expected because of less space into which the liquid could move.

CONCLUSIONS

SEM micrographs indicated that the bonding between the *Hildegardia* fabric and the matrix was enhanced partially by an alkali treatment. An alkali treatment in the presence of a silane coupling agent gave rise to matrix skin formation on the surface of the fibers, which indicated good bonding between the reinforcement and the matrix. *Hildegardia*/polycarbonate-toughened epoxy composites were found to have reasonable chemical and water resistance. The liquid absorption increased when the fabric was treated with an alkali, when a coupling agent was used, and in the presence of water and aqueous solutions. The void content of the composites decreased with increasing fabric content.

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