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WATER ABSORPTION BEHAVIOR AND EFFECT OF ACCELERATED AGEING ON FLEXURAL PROPERTIES OF COCONUT SHELL PARTICLE REINFORCED EPOXY COMPOSITES

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ABSTRACT
This work focuses on the water absorption behavior and its effect on flexural properties of coconut shell particle reinforced epoxy (CSPE) composites. CSPE composites were developed using coconut shell particles as fillers in different sizes namely 0.25, 0.5, 1 and 2 mm and epoxy resin as matrix. Samples were prepared with different filler volume fractions of 40%, 50% and 60% for each particle size. Water absorption test was conducted at room temperature by immersing the composite samples for 238 days and saturated moisture absorption and diffusion coefficient were determined. At accelerated ageing condition, composite samples were immersed in water maintained at elevated temperatures of 400C, 600C and 800C for 35, 30 and 25 days respectively. The effect of accelerated ageing on flexural properties of composites has been investigated. Results elucidated that composite with 0.25mm particles and 40% filler volume showed less diffusion coefficient. The decreasing trend in flexural strength and flexural modulus was observed in the aged samples at increased temperatures.

Keywords*: Coconut Shell Particle, Filler volume fraction, Water absorption, Accelerated ageing.*

I. INTRODUCTION
The strength of composite mainly depends on the type of reinforcing material used. In the recent years, for the replacement of conventional synthetic and ceramic materials, there has been a trend of using natural fillers as reinforcement in the composites. Natural fibers / particles are renewable, degradable, recyclable and available in plenty at cheaper cost compared to synthetic and ceramic materials. Though the natural filler reinforced composites possesses lower strength and modulus compared to synthetic and ceramic based composites, they are so sensitive to environmental agents like water. Now a days, natural filler composites are gaining variety of applications in automobile industry [1,2], marine [3], domestic such as doors, flooring, window panels, decorative, bath room fittings [4] etc, where it is exposed to atmospheric moisture. The dilapidation of the composite under humid atmosphere is a consequence, which the composite can absorb the moisture and inter penetrate between the matrix and reinforcement forming the micro pores that may lead to degradation of the composite. Hence, it is essential to examine the water absorption behavior of natural filler composites under normal and accelerated condition in order to predict the life span of the composite. Many researchers have undergone the water absorption tests on natural fiber/particle composites [5-8]. Also, it is necessary to conduct post ageing test to evaluate the strength degradation of the composite. For moisture ageing, usually the temperature is an accelerating factor. Lot of research has been taken up to forecast the shelf life of natural filled composites by accelerated moisture ageing [9-11]. The objective of this work is to evaluate the effect of different filler composition (40%, 50% and 60%) and filler size (0.25, 0.5, 1 and 2mm) on water absorption behavior and the effect of accelerated ageing on flexural properties of Coconut shell particle reinforced epoxy (CSPE) composites.

II. DEVELOPMENT OF COMPOSITE

Material Details
Dried coconut shells collected from southern part of India were cleaned and crushed into grains of distinct sizes of 0.25, 0.5, 1 and 2 mm size. Epoxy resin LY 556 and hardener HY 951 was used as matrix material in the ratio of

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10:1 respectively. 5% of melamine based on volume of the epoxy was also mixed to increase the rate of curing, bonding strength and to improve the surface finish of the developed composites. The epoxy resin, hardener, and melamine were purchased from M/s Insulation house, Bangalore, India.

Fabrication of Composites

The CSPE composite boards were fabricated in different filler volume fractions of 40%, 50% and 60% using distinct particle sizes of 0.25mm, 0.5mm, 1mm, and 2mm by open mould process. The designation of fabricated CSPE composites is shown in Table 1.

Table 1. Designation of CSPE composite samples

COMPOSITION	SERIES NAME											
	Series A			Series B			Series C			Series D		
	A1	A2	A3	B1	B2	B3	C1	C2	C3	D1	D2	D3
Particle Size (PS) (mm)	0.25	0.25	0.25	0.5	0.5	0.5	1.0	1.0	1.0	2.0	2.0	2.0
Filler Volume Fraction (Vf)	40%	50%	60%	40%	50%	60%	40%	50%	60%	40%	50%	60%

III. TEST PROCEDURE

Water absorption Test

The samples required for water absorption test were cut from the composite board using diamond wheel saw and finished to the dimensions of 6 mm × 6 mm × 50 mm using abrasive grinder. Three identical samples were used for each of the composition to conduct the water absorption test in accordance with ASTM D-570. The samples were first dried in oven and the weights of the dry samples were measured. The samples were then immersed in water at room temperature up to 238 days. During first 12 weeks, the samples were taken out from the water at every 24 hours, the surfaces were cleaned and weighted to an accuracy of 0.0001 gm using digital electronic weighing machine. During the subsequent weeks, the sample weights were measured for every 48 hours till the saturation level was reached. The percentage moisture gain at saturation level was determined using Equation (1).

$$M(\%) = \frac{W_w - W_d}{W_d} \qquad (1)$$

Where, $W_{w\,is}$ the weight of wet sample at saturation level, in grams and W_d is the weight of dry sample in grams.

The diffusion co-efficient was calculated using Equation 2.

$$D_z = \pi \left[\frac{h}{4\,M_m}\right]^2 \left[\frac{M_2 - M_1}{\sqrt{t_2} - \sqrt{t_1}}\right]^2 \qquad (2)$$

Where, $\left[\frac{M_2 - M_1}{\sqrt{t_2} - \sqrt{t_1}}\right]^2$ is the slope of moisture gain v/s √time

The applicability of Fick's law of diffusion assumes that the water penetration into a material depends on the moisture concentration gradient ∂_c / ∂_z and the time. The moisture weight gain (G_m) as a function of time is given by Equations (3) and (4).

$$G_m = \frac{M\%}{M_m\%} = 1 - \frac{8}{\pi^2} exp\left[-\frac{D_z t}{h^2}\pi^2\right] \quad \text{for} \quad \frac{D_z t}{h^2} > 0.005 \qquad (3)$$

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$$G_m = \frac{M\%}{M_m\%} = \frac{4}{h}\left[\frac{D_z t}{h^2}\right]^{1/2} \quad \text{for} \quad \frac{D_z t}{h^2} < 0.05 \qquad (4)$$

Where, t is time (sec), z = distance inside the composite in the direction of diffusion, D_z isdiffusion coefficient in z direction (mm^2/sec), h is the thickness of sample (mm), M is moisture uptake at any time t sec, M_m is moisture uptake at fully saturated condition.

Acceerated Ageing Test

Tests were carried out to determine the flexural strength and modulus of accelerated aged CSPE samples.The samples were prepared according to ASTM D790-03 with dimensions of 200mm × 12mm × 12mm and the span length as 160 mm.ASTM D 5229 was followed to carry out the accelerated ageing test.The test samples were immersed in water at different temperatures of 40^0C, 60^0C and 80^0C for 35, 30 and 25 days respectively using simple two wire thermostat, until all the samples reaches its moisture equilibrium. The moisture equilibrium was achieved as the condition when the change in the measured sample weight and previously measured sample weight has an interval less than 0.1% [12]. Three point bending test was carried out using universal testing machine, Instron 3382 of 100 kN capacity using data acquisition software Instron's series IX^{TM}/s.The samples were tested at a crosshead speed of 3 mm/min. The flexural strength and flexural modulus were calculated using the equations (5) and (6).

$$\text{Bending strength } \sigma = \frac{3Pl}{2bh^2} \qquad (5)$$

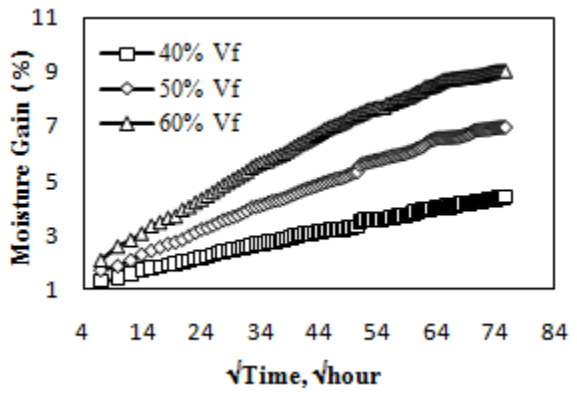
$$\text{Bending Modulus } E = \frac{mL^3}{4bh^3} \qquad (6)$$

Where, P is the maximum load (N), L is the distance between the supports (mm), m is slope of linear portion in the load v/s deflection diagram, b is the width of the sample (mm) and h is the thickness of sample (mm).

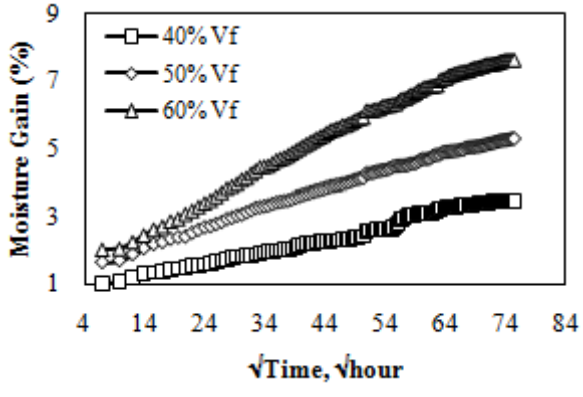
IV. RESULTS AND DISCUSSION

Water absorption of CSPE composites

CSPE composite samples were immersed water at an ambient temperature. The percentage moisture gain was calculated using equation (1). Percentage Moisture gain versus root of time curves for different filler volume fractions (40%,50% and 60%) and particle sizes (0.25, 0.5, 1 and 2mm) of CSPE composites were plotted as shown in Figures 1(a), (b), (c) and (d).



(a)



(b)

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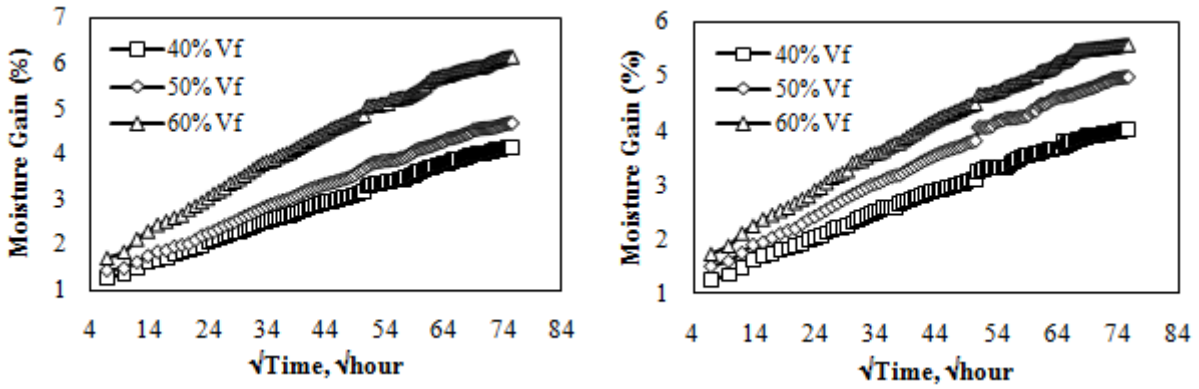

Figure 1:% Moisture Gain V/s root of time curve for CSPE Composites with (a) 0.25mm PS (b) 0.5mm PS (c) 1mm PS (d) 2 mm PS in different filler volume fraction (Vf).

The % moisture gain v/s root of time curves for CSPE Composites shown in Figures 1(a), (b), (c) and (d) were plotted by taking the average values of three identical samples. The % moisture absorption depends on the type of reinforcement (Filler) and the surrounding temperature. From Figure 1, it is observed that gradual increase in the moisture content with the increase in filler volume fraction and immersion time. Such behavior may be due to, when CSPE composites are exposed to moisture, the cellulosic and hemi cellulosic molecules that belongs to hydroxyl group has affinity towards water molecules forming hydrogen bonding [13]. This in turn leads to moisture build up in the cell wall resulting in particle swelling. The particle swelling may cause dispersion of moisture through small gaps between polymer chains and capillary transmission of moisture through voids and flaws at the interface between particles (filler) and the resin [14, 15]. The slope of moisture gain v/s root of time for each curve was determined and Diffusion co-efficient of CSPE composites with distinct particle sizes and different filler volume fractions was calculated by using the equation (2) and presented in Figure 2.

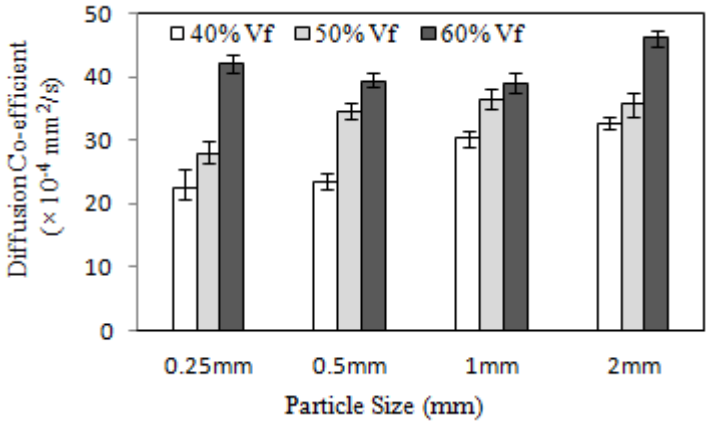

Figure 2:Diffusion Co-efficient of CSPE composite samples

The diffusion co-efficient of CSPE composites increase with the increase in filler volume fractions. The increase in filler volume makes improper bonding between reinforcement and resin material which creates a gap and leads to increased moisture uptake. The maximum value of diffusion co-efficient (46.35×10^{-4} mm^2/s) is observed in sample D3 which is 51% higher compared to sample A1 which has the lower value of diffusion coefficient (22.48×10^{-4} mm^2/s). The diffusivity behavior of composites can generally be classified into Fickian, Non-Fickian and Intermediate to Fickian and Non-Fickian. The behavior depends on factors like the crystal lattice structure of filler material, the interfacial bonding, morphology of filler material and its dimensions [16]. The moisture absorption of CSPE composites of different series (A1 – D3) immersed in water at ambient temperature showed the Fickian

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behavior, as all type of samples revealed an increasing tendency of moisture gain in the beginning. After the prolonged time, the moisture gain reached the saturation level. Such observations are elucidated in the early articles (14, 17).

Flexural behavior of aged CSPE composites

In this test, CSPE composite samples of different series (A1 – D3) were aged atvarying temperatures of 40^0C, 60^0C and 80^0C. Flexural tests were carried out on these samples. The average values of flexural strength and flexural modulus were determined for the composite samples aged at these temperatures and compared with the results of unaged samples in Tables (2) and (3) respectively.

Table 2.Comparison of Flexural strength of unaged CSPE samples with aged CSPE samples

Sl.No.	FILLER VOLUME FRACTION	PARTICLE SIZE (mm)	FLEXURAL STRENGTH (MPa)			
			Unaged Samples	Samples aged at 40^0C	Samples aged at 60^0C	Samples aged at 80^0C
1	40%	0.25	51.57	47.70	42.51	33.60
2		0.5	39.90	36.33	30.88	29.06
3		1	27.06	26.97	26.19	25.34
4		2	28.18	26.91	25.09	25.04
5	50%	0.25	37.17	36.36	33.12	29.97
6		0.5	30.10	28.96	27.32	27.09
7		1	26.75	23.52	23.02	22.13
8		2	24.42	21.63	20.68	20.13
9	60%	0.25	35.68	33.60	30.69	25.88
10		0.5	29.80	25.42	24.19	23.65
11		1	24.31	23.33	22.96	20.43
12		2	18.21	17.18	16.12	15.73

Table 3: Comparison of Flexural Modulus of of unaged CSPE samples with aged CSPE samples

SL. NO.	FILLER VOLUME FRACTION	PARTICLE SIZE (mm)	FLEXURAL MODULUS (MPa)			
			Unaged Samples	Samples aged at 40^0C	Samples aged at 60^0C	Samples aged at 80^0C
1	40%	0.25	4765.10	4518.3	3761.17	3310.33
2		0.5	4569.00	4369.75	3509.97	2747.42
3		1	4179.50	4109.02	2964.97	2403.58
4		2	4129.70	3931.14	2816.46	2119.44
5	50%	0.25	4526.90	4381.2	3844.33	2952.90
6		0.5	4419.00	4117.76	3689.71	2668.16
7		1	3761.00	3663.02	2734.37	2458.77
8		2	3649.92	3222.98	2622.22	2529.67
9	60%	0.25	3908.91	3512.61	3408.80	2868.41
10		0.5	3891.20	3427.35	2959.45	2671.34
11		1	3679.11	3294.88	2890.34	2422.23
12		2	2833.04	2661.93	2428.53	2202.70

Tables 2 and 3 illustrates the trend of flexural properties of CSPE samples when aged at elevated temperatures compared to unaged CSPE samples of distinct filler volume fractions and particle sizes. Gradual decrease in the flexural properties are noticed when the samples are aged at elevated temperatures of 40^0C, 60^0C and 80^0C. The maximum values of flexural strength and flexural modulus was found to be 51.57 MPa and 4765.1 MPa respectively in unaged CSPE sample A1. When this sample A1 is subjected to accelerated ageing at varying temperatures of 40^0C, 60^0C and 80^0C, the flexural strength was decreased by 7.5 %, 17.60 % and 34.8%

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respectively and flexural modulus values were reduced by 5.17%, 21.06, and 29.84% respectively when compared to unaged CSPE sample A1. Also, with the increase in filler volume fraction and particle size, the declination in the flexural property was noticed. The lowest values of flexural strength (15.73MPa) and flexural modulus (2202.70MPa) was seen in aged CSPE composite with 60% Vf and 2mm PS. The decrease in flexural properties with increase in filler volume fraction, particle size and moisture content in the composite may be due to formation of hydrogen bonding between the water molecules and cellulose and hemicellulose molecules of coconut shell particles. Coconut shell particles are hydrophilic in nature containing hydroxyl groups in its structure forming number of hydrogen bonds between the molecules of the cellulose and polymer. With this high hydroxyl group percentage, coconut shell particles tend to show lower moisture resistance which leads to dimensional changes and color difference of composites. Also, poor interfacial adhesion between filler and matrix creating the debonding caused a decrease in the flexural properties CSPE composites [14].

V. CONCLUSION

The objective of this work was to experimentally study the water absorption behavior and accelerated ageing and its effect on flexural properties of CSPE composites. The Diffusion co-efficient of CSPE composites is found to increase with the increase in filler volume fractions. Water absorption of CSPE composites showed the Fickian behavior. CSPE samples with 40% filler volume fraction and 0.25mm particle size (sample A1) showed less percent moisture gain compared to other CSPE samples. Gradual decrease in the flexural properties of CSPE composites are observed when the samples are aged at elevated temperatures of 40^0C, 60^0C and 80^0C. When CSP sample A1 is subjected to accelerated ageing at varying temperatures of 40^0C, 60^0C and 80^0C, the flexural strength was decreased by 7.5 %, 17.60 % and 34.8% respectively and flexural modulus values were reduced by 5.17%, 21.06, and 29.84% respectively when compared to unaged CSPE sample A1. Hence, CSPE composite with 40% filler volume fraction and 0.25mm particle size can be promising material for several applications in packaging industries, in automobile sectors as an alternative to wood products , interiors of aircrafts, etc,.

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