Effects of Two Pre-Treatment Methods on Water Absorption Property of Polyester-Fibre Composites

Pauline N. Obianuko and Tessy U. Onuegbu

Abstract — The use of plant fibres as reinforcement in composite materials is finding increasing interest in the automotive and building industry, and the properties of plant fibre composites have been addressed in numerous research studies. The replacement of synthetic fibres with natural fibres in composites is based on their renewable and environmentally friendly nature.

The aim of this work is to investigate the effects of two chemical pre-treatment methods on the water absorption property of polyester-fibre composites. Fully grown kenaf (*hibiscus cannabalis*) bast fibres were manually retted from the stalk, washed, and cut into short fibre lengths of about 10 cm. One portion of the fibres was pre-treated with 5% pbw NaOH solution before immersing it in glacial acetic acid and then in acetic anhydride. The second portion was pre-treated with 5%pbw of NaOH and 0.125% of KMnO₄ in acetone. The third portion of fibres was untreated to serve as control. The ground fibre was incorporated into ortho unsaturated polyester and cast with Teflon square shaped mould. The cured samples were weighed and immersed in water at room temperature until no further gain in weight was observed. The SEM of the fibres and the composites was taken. The results showed that the acetylation pre-treatment method reduced the water absorption property of the composites by a minimum of 46.0% across all fibre volume fractions while the permanganate pre-treatment reduced it by at least 25.5%. Chemical pre-treatment of fibres can therefore reduce the hydrophilic property of natural fibres.

Index Terms — natural fibre, composites, pre-treatment.

I. INTRODUCTION

Thorough knowledge of water sorption in composites is the basis for an assessment of the water related dimensional stability of composites. Moreover, it is important with respect to the many properties of composites that are affected by water sorption: e.g. mechanical properties [1] electrical properties and thermal properties. As opposed to plant fibre composites, the mechanisms of water sorption in synthetic fibre composites (e.g. glass/epoxy) are well documented [2]. The water sorption capacity of these composites is typically low and is mainly confined to the matrix. However, in plant fibre composites, the water sorption capacity is large and is mainly confined to the fibres. Thus, the existing knowledge of composite water sorption does not necessarily apply to plant fibre composites. The cell wall of plant fibres is mainly composed of three large polymers: cellulose, hemicelluloses and lignin [3]. These polymers differ in molecular

composition and structure and therefore they display different mechanical properties as well as different water sorption properties. Because the properties of plant fibres result from the chemistry of the cell wall components, the basic properties of a fibre can be changed by modifying the basic chemistry of the cell wall polymers. Dimensional stability can be greatly improved by bulking the fibre cell wall either with simple bonded chemicals or by impregnation with water-soluble polymers [4]. For example, acetylation of the cell wall polymers using acetic anhydride produces a fibre composite with greatly improved dimensional stability and biological resistance. The same level of stabilization can also be achieved by using water-soluble phenol-formaldehyde polymers followed by curing [5]. Biological resistance of fibre-based materials can be improved by several methods. Bonding chemicals to the cell wall polymers increases resistance due to the lowering of the equilibrium moisture content point below that needed for microorganism attack and by changing the conformation and configuration requirements of the enzyme-substrate reactions. Kenaf fibres change dimensions with changing moisture content because the cell wall polymers contain hydroxyl and other oxygen-containing groups that attract moisture through hydrogen bonding. The hemicelluloses are mainly responsible for moisture sorption, but the accessible cellulose, non-crystalline cellulose, lignin, and surface of crystalline cellulose also play major roles [3]. Moisture swells the cell wall and the fibre expands until the cell wall is saturated with water. Beyond this saturation point, moisture exists as free water in the void structure and does not contribute to further expansion. This process is reversible, and the fibre shrinks as it loses moisture [6.] Moisture absorption of fibres has also been reported to depend largely on the polarity of the polymer matrix [7]. Moisture absorption is reduced where the polarity compatibility (unlike charges) of the fibre and matrix reduces the voids between the polymer and fibre. However, where the polarity of fibre and matrix are not compatible (like charges), there is poor wetting of fibre, which increases the number of void and flaws between the fibre and matrix. Under this condition water molecules can penetrate the composite easier and reach the fibres, thus causing the reduction in mechanical properties of composites. Hemicelluloses is the cell wall polymer with the largest water sorption capacity. The hydroxyl groups in the crystalline regions of cellulose are therefore limited, and confined to the amorphous regions. The cell wall polymer with the lowest sorption capacity is lignin. It has been shown for wood that the water sorption capacity can be calculated as a linear mixture of the sorption capacities of its chemical constituents [8]. Thus, water sorption can be notably reduced if the content of hemicelluloses is lowered,

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However, water sorption can as well be reduced by substitution of less polar groups for the hydroxyl groups, and this is the underlying principle in acetylation. Fibre/matrix compatibility is mainly governed by the surface polarity of the two parts. Thus, in the case of polar (i.e., hydrophilic) plant fibres and a non-polar (i.e., hydrophobic) thermoplastic matrix, the compatibility is low, and this restricts fibre/matrix bonding. It has been reported [9] that coupling with maleic anhydride treatment reduced water absorption to a great extent in hemp, banana, and sisal fibres and their composites.

II. MATERIALS AND METHODS

The kenaf bast fibres were sourced from a farm in Kaduna Polytechnic Quarters. The reagents for pre-treatment were bought from Drug and Chemical market head bridge Onitsha while the reagents for the composite casting were sourced from Polyconsult Ltd, Ikeja Lagos.

A. Experimental

The kenaf bast fibres were cut into short length fibres of about 10cm and divided into three portions. One portion of the fibres was treated by immersing it in 5% pbw of sodium hydroxide solution for 30 minutes, removed and allowed to drain thoroughly. The fibre was then immersed in glacial acetic acid for one hour. The glacial acid was decanted and the fibre was finally immersed in acetic anhydride with two drops of conc for five minutes. The second sample was soaked in 5%pbw of sodium hydroxide for 30 minutes. It was drained in a sieve before immersing in 0.125%pbw of potassium permanganate in acetone. The fibre in each case were drained and dried at temperature of 105°C until it was crisp dry. The fibre samples(pretreated and untreated) were ground and applied in ortho unsaturated polyester using methyl ethyl ketone peroxide (MEKP) as catalyst ,1% cobalt octoate as accelerator and poly vinyl alcohol (PVA) as mould releasing agent. The fibre and polyester were weighed into a beaker and mixture stirred until it was homogenous. The catalyst, then the accelerator were added using syringes and stirred. The mixture was then poured into moulds and allowed to cure for one hour.

TABLE I: THE ARRANGEMENT OF CHANNELS

TABLE I: FORMULATION OF THE FIBRE-POLYESTER COMPOSITES				
Parts by mass	(4%)	(8%)	(12%)	(16%)
Reagents	Mass (gm)			
Ground kenaf	4	8	12	16
Ortho-unsaturated polyester	96.0	92.0	88.0	84.0
Methyl Ethyl Kenote (MEKP) 2% weight of polyester	1.92	1.84	1.76	1.64
Accelerator-Colbat (1% weight of polyester)	0.96	0.92	0.88	0.84

B. Water Absorption Test of Composites

The square shaped composites were weighed and immersed in water for 5 days at room temperature. The samples were weighed after every 24hrs until no further gain in weight was detected (after the fifth day). Differences in weight were calculated and percentage weight gain determined [3].

C. Scanning Electron Micrographs of Composites (SEM)

The scanning electron micrographs of the samples were taken. Small size of about two square centimeters were cut and placed in the sample chamber where electrons are beamed on it. The plates are as shown on page 3.

III. RESULT AND DISCUSSION

The weights of the composites before and after immersion in water, and the percentage weight gain is presented in the tale below.

TABLE 2: WATER ABSORPTION OF COMPOSITES

		Initial weight	Final weight	Weight gained	% weight gained
Acctylated FC	4%	26.26	27.23	0.972	3.70
	8%	26.28	27.32	1.040	3.97
	12%	29.56	31.14	1.579	5.34
	16%	27.56	29.17	1.610	5.84
Permanganate Treated FC	4%	24.28	25.52	1.241	5.11
	8%	25.67	26.92	1.250	4.87
	12%	28.38	30.63	2.250	7.93
	16%	27.22	29.00	1.783	6.55
Untreated FC	4%	24.50	26.18	1.681	6.86
	8%	29.99	32.37	2.378	7.93
	12%	32.62	36.55	3.927	12.04
	16%	26.11	29.49	3.383	14.70
Un-reinforced	0%	17.16	17.79	0.630	3.67

From the table it is observed that the composites absorbed more water than the unreinforced polyester. This affirms that water sorption in composites is dependent on the fibres used for reinforcement and not on the polymer matrix. This also explains the observation from the results that the water absorbed increases with an increase in the fibre volume fraction. When compared with the water absorption of the untreated fibre composites, at 0.04 fibre volume fraction acetylation reduced water absorption by 46%, at 0.08 fibre volume fraction, the water absorption was reduced by 49.9%. At 0.12 fibre volume fraction, acetylation reduced water absorption by 55.8% and at 0.16 fibre volume fraction water absorption was reduced by 60%.

Permanganate pretreatment also reduced water absorption of the composites .When compared with the untreated fibre, a 0.04 fibre volume fraction it reduced water absorption by 25.5%, at 0.08 by38.6%, at .12 by34.1% and at 0.16 by 55.4%. It is easily deduced that acetylation of the kenaf bast fibres reduced the hydrophilic property of the fibres better than the permanganate pre-treatment

The scanning electron micrographs shows that the composites of the acetylated fibre are more homogenous indicating better wettability of fibre and matrix and thereby having less voids for water absorption.

Fig. 1. Water absorption of the composite.

IV. CONCLUSION

The pre-treatment of kenaf bast fibres by acetylation and permanganate methods reduced the hydrophilic property of the fibres. Acetylation is a better method of pre-treatment in comparison with permanganate pre-treatment of kenaf bast fibres.

(a)

(b)

(c)

Plate 1: a) 8% Acetylated fibre composite.
b) 8% Perm. Treated fibre composite.
c) 8% Untreated fibre composite.

(a)

(b)

(c)

Plate 2: a) 12% Acetylated fibre composite 12% Perm.
b) Treated fibre composite.
c) 12% Untreated fibre composite.

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