

Study of water absorption behavior of composite material

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ABSTRACT

In this paper, water absorption studied in hemp hurts composite material. Experiment done in hemp hurds composite behavior of 30 days. Hemp fibre reinforced unsaturated polyester composites (HFRUPE) were subjected to water immersion tests in order to study the effects of water absorption on the mechanical properties. Water absorption tests were conducted by immersing specimens in a de-ionised water bath at 25 °C and 100 °C for different time durations. The tensile and flexural properties of water immersed specimens subjected to both aging conditions were evaluated and compared alongside dry composite specimens. The percentage of moisture uptake increased as the fibre volume fraction increased due to the high cellulose content. The tensile and flexural properties of HFRUPE specimens were found to decrease with increase in percentage moisture uptake. Moisture induced degradation of composite samples was significant at elevated temperature.

Introduction

The use of natural plant fibres as reinforcement in polymer composites for making low cost engineering materials has generated much interest in recent years. New environmental legislation as well as consumer pressure has forced manufacturing industries to search for new materials that can substitute for conventional non-renewable reinforcing materials such as glass fibre [1]. The advantages of natural plant fibres over traditional glass fibres are acceptable as good specific strength and modulus, economical viability, low density, reduced tool wear, enhanced energy recovery [2]. Natural plant fibre reinforced polymeric composites, also have some disadvantages such as the incompatibility between the hydrophilic natural fibres and hydrophobic thermoplastic and thermoset matrices requiring appropriate use of physical and chemical treatments to enhance the adhesion between fibre and the matrix [3]. Natural fibers are subdivided based on their origins, into four types: seed hairs (cotton, kapok), bast-fibers (flax, hemp, jute, and ramie), leaf-fibers (sisal, henequen, coir, and abaca) and wood flour (wheat husk, rice husk) [4]. Composite material based on a polymer matrix reinforced by natural fibers is called in the literature biocomposites. Polymer composites containing different fillers and/or reinforcements are frequently used for automotive application. In recent years, such composites were developed for their application into interior and exterior parts of cars in order to ensure the overall lower weight of the vehicle and increase sustainability of the automotive manufacturing process.

Hemp is also called cannabis sativa. It is an annual herbaceous plant native to Asia and widely cultivated in Europe [5]. Hemp and flax are the only commercial sources of long natural fibres grown in the UK. Plant stems are processed by various mechanical methods to extract the fibre. Fibres from hemp stems have been widely used in the production of cords and clothing, and have potential for reinforcement in polymer-matrix composites (PMCs). Recently, car manufacturers have started manufacturing non-structural components using hemp and flax fibres due to their higher specific strength and lower price compared to conventional reinforcements [5].

In order to reduce high moisture/water absorption ability of organic filler in composites, chemical [6–8] or

physical treatments of fibers surface are applied [9]. Various findings of the water absorbability of composites have been identified. Generally, chemical treatment of natural fibers reduced the overall water uptake of fibers [10] and can positively or negatively affect strength parameters of composites in dependence on plant and matrix kind, treatment conditions of fibrous filler, cellulose content, *etc.* Hemp fiber treatment with coupling agents caused decrease in water uptake of hemp/fiber/polyethylene composites, but also decreased the flexural strength after water exposure [11].

1. Material and Methods

Hemp Hurds

1.1. Material

The inorganic matrix in this study was based on Portland cement CEM I 42.5 R (classical binder component) and on so called MgO-cement consisting of the milled calcined MgO (SMZ a.s. Jelsava, Slovakia), silica sand (Sastin, Slovakia) and sodium hydrogen carbonate (p.a). Fine dispersed product of MgO was obtained by short-term dry milling (5 min) in laboratory vibratory mill VM 4

1.2. Methods

1.2.1. Chemical Modification of Hemp Hurds

The chemical modification of dried hemp hurds was made by three different solutions: sodium hydroxide (NaOH) p.a. (CHEMAPOL, Prague, Czech Republic), pulverized calcium hydroxide ($\text{Ca}(\text{OH})_2$) with purity $\geq 96\%$ (ROTH, Karlsruhe, Germany), ethylenediaminetetracetic acid (EDTA) p.a. (GAVAX s.r.o., Vranov nad Toplou, Slovakia). The specification of the chemical treatment conditions is described.

1.2.2. Determination of Hemp Hurds Components

A milled and oven-dried sample was used for the determination of chemical composition of hemp hurds. Extractives were determined in a Soxhlet apparatus (Kavalier Glass, Sázava, Czech Republic) with a mixture of ethanol and toluene (2:1) for 8 h, according to the American Society for Testing and Materials (ASTM). Total content of polysaccharides (*i.e.*, holocellulose) was determined using the method of Cellulose content was determined by the Seifert method. A mixture of acetylacetone, dioxane, and hydrochloric acid (6:2:1.5) under reflux for 30 min was used for delignification of samples. The content of hemicelluloses was determined as the difference between holocellulose and cellulose. The content of acid-insoluble (Klason) lignin was determined according to the U.S. Department of Energy, National Renewable Energy Laboratory analytical procedure. The samples were hydrolyzed in a two-stage process. In the first stage, 72% (w/w) H_2SO_4 at a temperature of 30 °C was used for 2 h, and in the second stage, the samples were refluxed after dilution to 4% (w/w) H_2SO_4 for 4 h. Total ash content (mineral substances) was determined according to the U.S. Department of Energy, National Renewable Energy Laboratory analytical procedure.

1.2.3. Size Exclusion Chromatography

Molecular weight distribution analysis of the cellulose samples was performed by size exclusion chromatography (SEC) after their conversion into tricarbonyls. Cellulose tricarbonyls were dissolved in tetrahydrofuran and filtered through a Puradisc 25 NYL filter (Whatman International, Maidstone, UK) with a pore size of 0.45 μm . SEC was performed at 35 $^{\circ}\text{C}$ with tetrahydrofuran at a flow rate of 1 $\text{mL}\cdot\text{min}^{-1}$ on two PLgel (porous polystyrene/divinylbenzene matrix with particle size of 10 μm and internal diameter \times length of 7.5 \times 300 mm) MIXED-B columns (Agilent Technologies, Santa Clara, CA, USA) preceded by a PLgel (10 μm , 7.5 \times 50 mm), Guard-column (Agilent Technologies) as described by [34]. Data acquisitions were carried out with ChemStation software (Agilent Technologies, Santa Clara, CA, USA) and calculations were performed with the Clarity GPC module (DataApex, Prague, Czech Republic). Numerical outputs obtained for M_n (number-average molecular weight) and M_w (weight-average molecular weight) were recalculated to underivatized cellulose by multiplication with the coefficient $k = 162/519$. Polydispersity index (PDI) of cellulose was calculated as the ratio M_w/M_n . Degree of polymerization (DP) values were calculated by dividing the molecular weight by the monomer equivalent weight of anhydroglucose ($\text{DP} = M/162$).

1.2.4. Water Absorption Tests

Two kinds of absorption tests on dried cube specimens of hemp hurds composites after 28 days of hardening in deionized water bath (PE closed container) at laboratory temperature (23 $^{\circ}\text{C}$) were performed. Short-term water absorption test (after one hour immersion) was made according to Slovak standard (STN EN 12087/1). Long-term water absorption was conducted by immersing the specimens for different time durations (up to 180 days) to study their durability. After immersion for given time, the specimens were taken out from the water and water from all surfaces of bodies was removed by a clean dry cloth. All specimens were weighted again after their storage. Content of absorbed water in composites after immersion time t (M_t) was calculated by the weight difference between the samples immersed in water and dry composite samples. The water absorption kinetic model [12] was used to describe the sorption curves for composites based on natural fiber.

$$\frac{M_t}{M_{\infty}} = kt^n$$

where M_t , M_{∞} , are water content at time t and at equilibrium; k and n are constants giving some information about mechanism of diffusion taking place inside composites. Coefficients n and k were calculated from experimental data using classical method of the mathematical statistics, as regression analysis, correlation analysis and testing of hypotheses. First, the coefficient n was determined as a slope of sample regression line created by log of experimental set of data and verified by its coefficient of correlation. Using method of testing of hypotheses was proved that this corresponding coefficient of correlation between both sets, experimental and computed data are statistically significant. Second, the coefficient k was determined using the assumptions about the existence of a saturation point during process of absorption in time. This fact enables then to find the asymptotic line of the corresponding process of absorption also using the above-mentioned method of regression analysis.

2. Results and Discussion

Studying the water absorption behavior of the biocomposites is very important because of poor water

resistance of fibrous biomaterial. In building application, mainly for outdoor use of biocomposites, water absorbability is one of the most important parameters impacting their mechanical properties and dimensional stability. The study of water sorption and thickness swelling of natural fibers plastic composites [10,37,38] confirmed that this behavior of composites with a given fiber content depends on a wide array of factors, including fiber/matrix interface quality, chemical composition and length of fibers, their distribution in composite, permeability nature and porosity of fibers and density. Recently, a limited focus on the analysis of the water sorption properties of fibers and inorganic matrix has been addressed. To contribute to a better understanding of the water sorption process in composites with the same volume fraction of hemp hurds, short- and long-term water storage study of 28 days hardened specimens took place at room temperature.

2.1. Short-Term Water Absorption Behavior of Composites

The research of short-term water sorption behavior of composites was directed to study the influence of selected constituent parameters such as the mean particle length of hemp hurds and matrix materials on the water uptake of hemp hurds composites. The mean particle length of Hungarian and Dutch hemp hurds on water uptake of composites with these organic fillers and MgO-cement during their short-term storage in water was studied. Figure 1 shows water absorbability of hemp hurds reinforced composites as a function of mean particle length of filler component. Composites based on Hungarian hemp hurds samples with longer mean particle length acquired higher values of water content (11.9–25.8 wt.%) while in Dutch plant composites lower water absorbability values were observed (6.3–14.3 wt.%). Water content values in immersed composites based on Hungarian hemp hurds increase with increasing mean particle length but composites with Dutch filler behave differently. In this case, water absorbability of composites first increases and then decreases with increasing mean particle length of hemp hurds. The hydrophilic behavior of plant fibers is mainly due to two factors: their composition and their specific structure. As is evident from Table 2, this different behavior of composites based on the Dutch hemp hurds most likely cannot be caused by the chemical composition of filler. The high level of water absorption in hemp hurds is determined by their particular structure. The hemp hurds layered structure forming by fibrils, microfibrils and fibers is porous and has a high exchange surface. At the scale of cellulose microfibrils, transport of water molecules could take place in the amorphous region where hydrophilic polymers are present (hemicellulose and lignin). The effect of extremely complicated microstructure of hemp hurds, its heterogeneity of the properties due to fiber separation procedure has to be taken into account at water sorption behavior of these composites. The differences in porosity of hemp hurds slices in composite could be considered as a further possible reason for explanation of short-term water sorption behavior of samples 4–6. However, currently, the porosity content in hemp hurds is unknown.

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