

# Lightweight Composites Based on Rapidly Renewable Natural Resource

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Over the last few years, a number of researchers have been involved in investigating the exploitation of natural fibres as load bearing constituents in composite materials. The use of such materials in composites has increased due to their relative cheapness, their ability to recycle and their mechanical and physical properties. Composites based on natural fibers have many advantages over their synthetic counterparts owing to their low cost, low density, high specific strength and stiffness, excellent sound- absorbing and high impact energy absorption. The technical hemp as fast-growing plant has prospect in the construction field in accordance with the principles of sustainable development. A special attention is paid to the use of fibrous hemp as well as hemp hurds (nonfibrous fraction hemp stalk) in the building materials preparing in various forms, such as insulation boards, mass-spraying hempcrete for the production of lightweight composites, where it is in the role of filler.

The presence of surface impurities and the large amount of hydroxyl groups make plant fibers less attractive for reinforcement of composites. Incompatibility of interface between fibres and matrix, the tendency to form aggregates during processing and poor resistance to moisture can be eliminated by physical/chemical treatments of fibers and/or matrix or the use of coupling agents.

In this paper, some aspects of durability of hardened composites based on chemically modified of hemp hurds slices and MgO-cement as binding agents after long termed storage in deionised water by testing on some important material properties as water absorbability, thermal conductivity coefficient, apparent density and compressive strength was studied.

## 1. Introduction

The construction and building industries are two of the major and most active sector in Europe, but are also responsible for the depletion of large amounts of non-renewable resources and for 30% of eq. CO<sub>2</sub> emissions. One way to achieve a more sustainable construction industry consists in reducing the raw materials consumption by adding by-products or recycled materials. Such a strategy offers the triple advantage of creating new opportunities for these by-products while preserving natural resources and without changing the conventional construction methods (Colinart et al., 2012). The other way consists in looking for alternative materials, technologies and methods of construction which significantly reduce resource and energy consumption providing better energy efficiency without causing pollution and damaging health and eco-systems. In this respect, vegetable components could be used as a possible input to lighten concrete mixtures (Monreal et al., 2010). The use of plant particles allows producing building material with lower environmental impact especially in regard to the carbon footprint.

Since the beginning of the nineties, a new building material obtained by mixing together hemp particles (the non-fibrous fraction of the hemp stem called "shives" or "hurds") and a binder is developed. It enables to build sustainable buildings (for new buildings construction or existing buildings renovation) as filling material of a load-bearing structure (Arnaud and Gourlay, 2012).

Hemp composite (hemp concrete) is more and more recommended by the eco-builders of a sustainable development. This vegetable material has low environmental impact. In comparison with other materials in construction, the hemp concrete has a low heat conductivity, which reduces heat diffusion and thus

reduces winter heat losses and protects from summer heat waves. It has also a low mass density, which reduces its storage capacity. However, its density remains higher than classical insulation materials such as mineral wool, fibreglass. Besides, since hemp concrete is a hygroscopic material, it has the air humidity and air-conditioning loads (Tran Lea et al., 2010). In our previous papers (Kidalova et al, 2012) and also (Stevulova et al, 2012), experimental study of the parameters affecting the physical and mechanical properties of hemp composite based on conventional and alternative binders was performed. Properties of chemically treated hemp hurds as well as behaviour of composites based on modified hurds with alternative binder of MgO-cement were investigated (Terpakova et al, 2012) and the year after (Cigasova et al, 2013).

In this paper, some aspects of durability of hardened composites based on chemically modified of hemp hurds slices and MgO-cement as binding agents after long termed storage in deionised water by testing on some important material properties as water absorbability, thermal conductivity coefficient, apparent density and compressive strength was studied.

## 2. Materials and Methods

### 2.1 Materials

The technical hemp hurds slices (*Cannabis Sativa L.*) coming from the Netherlands company Hempflax were used in experiments. This hemp contains more shives material (the woody inner part of stem) than fibres. The used hemp material was polydispersive. Mean particle length of hemp slices was 24.2 mm. Chemical composition of used hemp hurds is shown in Table 1. The density of hemp material was 117.5 kg·m<sup>-3</sup>. The average moisture content of hemp material determined by weighing of hemp sample before and after drying for 24 h at 105 °C was found 10.78 wt.%.

MgO-cement as binder used in experiments consists of caustic magnesite obtained by low temperature decomposition of natural magnesite (CCM 85, SMZ a.s. Jelšava, Slovakia), silica sand (Šaštín, Slovakia) with the dominant component of SiO<sub>2</sub> (95-98 %) and sodium hydrogen carbonate (p.a). MgO has been milled in order to reduce its particle size. Dry milling was carried out in laboratory vibratory mill VM 4 for 5 min (Kidalova, 2011a).

Table 1: Selected chemical characteristics of hemp hurds

Chemical composition of hemp hurds [%]					
Toluene-ethanol extract	Holocellulose	Cellulose	Hemicellulose	Lignin	Ash
3.5	74.5	44.2	30.3	24.4	1.4

### 2.2 Chemical modification of hemp hurds

Chemical treatment of dried hemp hurds carried out in three liquid medium of ethylenediaminetetracetic acid (EDTA), calcium hydroxide and sodium hydroxide (Table 2).

Table 2: Chemicals used for modification of hemp hurds surface

Chemical	Formula	Producer	Purity
ethylene-diamine-tetracetic acid	C <sub>10</sub> H <sub>16</sub> O <sub>8</sub> N <sub>2</sub>	GAVAX s. r. o., Slovakia	p. a.
calcium hydroxide	Ca(OH) <sub>2</sub>	ROTH, Germany	≥96%, pulv.
sodium hydroxide	NaOH	CHEMAPOL, Slovakia	p. a.
acetic acid	CH <sub>3</sub> COOH		8 %

Hemp hurds surface treatment was performed as follow procedures:

- EDTA. Hemp hurds slices were immersed during 3 h in a solution of EDTA (5 g/L). In order to ensure removal of excess EDTA, fibres were then washed with water.
- Alkalization. Hemp hurds slices were soaked in 1.6 M NaOH solution during 48 h and then neutralized with 1 % acetic acid. It was then washed with water until the pH value was 7.
- Ca(OH)<sub>2</sub>. In order to saturate fibres with calcium ions, hemp hurds slices were placed in a saturated lime solution ([Ca<sup>2+</sup>] = 2.10<sup>-2</sup> M) for 48 h. Following the impregnation, hemp material was washed with water. Thus chemically treated hemp hurds were used for composites preparing.

### 2.3 Preparation of composites

Experimental mixtures prepared according to the recipe published in work (Bydzovsky, 2009) consisted of 40 vol. % of hemp hurds (unmodified as a referential material and chemically treated), 29 vol % of MgO-

cement and 31 vol % of water. For preparation of specimens, the standard steel cube forms with dimensions 100 mm x 100 mm x 100 mm were used. The specimens were cured for 2 d in the indoor climate at approximately +18 °C and then removed from the moulds. After that time, the specimens were wrapped in a household film during 28 d. Designation of prepared composites is shown in Table 3.

Table 3: Designation of the experimental mixtures

Mixture	Treatment of hemp hurds
1	Untreated - referential
2	by NaOH
3	by Ca(OH) <sub>2</sub>
4	by EDTA

#### 2.4 Storage of composites in water

Hemp composites after 28 d of hardening were used for storage experiments in deionised water. Water absorption was carried out by immersing the initial hemp composites in water bath (PE closed container) at laboratory temperature (20 °C) for 28 d of durations. After immersion the specimens were taken out from water and water from all surfaces of bodies has been removed by a clean dry cloth. The specimens were reweighed and afterwards were dried in an oven at 70 °C up to constant weight for following measurement of density, compressive strength and thermal conductivity coefficient.

#### 2.5 Testing methods

The hemp hurds (modified and unmodified) and leachates from storage of composites were characterized using infrared spectroscopy with Fourier transformation (Alpha Bruker Platinum-ATR spectrometer). A total of 24 scans were taken for each sample between 400 cm<sup>-1</sup> and 4,000 cm<sup>-1</sup>, with a resolution of 4 cm<sup>-1</sup>.

Elemental analysis of the leachates was carried out using the method of XRF using SPECTRO iQ II (Ametek, Germany) with SDD silicon drift detector with resolution of 145 eV at 10,000 pulses. The primary beam was polarized by Bragg crystal and Highly Ordered Pyrolytic Graphite - HOPG target. The samples were measured during 300 s at voltage of 25 kV and 50 kV at current of 0.5 and 1.0 mA, respectively under helium atmosphere by using the standardized method of fundamental parameters for cement pellets.

Density, thermal conductivity and compressive strength were measured on dried specimens after their water storage. The density was determined in accordance with standard STN EN 12390-7. The thermal conductivity coefficient of samples as the main parameter of heat transport was measured by the commercial device ISOMET 104. The measurement is based on the analysis of the temperature response of the analyzed material to heat flow impulses. The heat flow is induced by electrical heating using a resistor heater having direct thermal contact with the surface of the sample. Compressive strength of all composites was determined using the instrument ADR ELE 2000.

### 3. Results and discussion

FTIR spectroscopy allows the measurement of variations of hemp fibre composition after chemical treatments. Spectra of hemp hurds treated by three reagents are compared with original (untreated) sample in Figure 1. Observing the spectra, we found that a partial removal of hemicellulose, lignin and removal of waxes and oil was performed. It was reported in our previous work (Terpakova, 2012). FTIR measurements of leachates from storage of composites didn't confirm the leaching of organic compounds from composites into the deionised water but showed leaching of inorganic compound. The peaks at 3323 cm<sup>-1</sup> and 1,636 cm<sup>-1</sup> are characteristic for asymmetric vibrations of the -OH group of H<sub>2</sub>O. The peak observed at 1,394 cm<sup>-1</sup> is attributed to vibration of carbonate group which has origin from NaHCO<sub>3</sub>. Elemental analysis of the leachates showed presence of Si (0.0475 %) from silica sand, Na (0.3385 %) from sodium hydrogen carbonate and Mg (0.1685 %), Al (0.0458 %), Ca (0.2016 %), Fe (0.1314 %), Mn (0.0135 %) from caustic magnesite.

The results of investigations showed that values of water content in composites are in a range of 7.27-21.9 wt. % (Table 4). Water content in specimens based on chemically modified hemp slices is higher in comparison to water value of referential composite and it is determined by nature of reagent. Hemp composite absorbs water more rapidly than, e.g. cement mortar or concrete. Sorption behaviour of hemp composites in water at room temperature is the results of several diffusion processes.

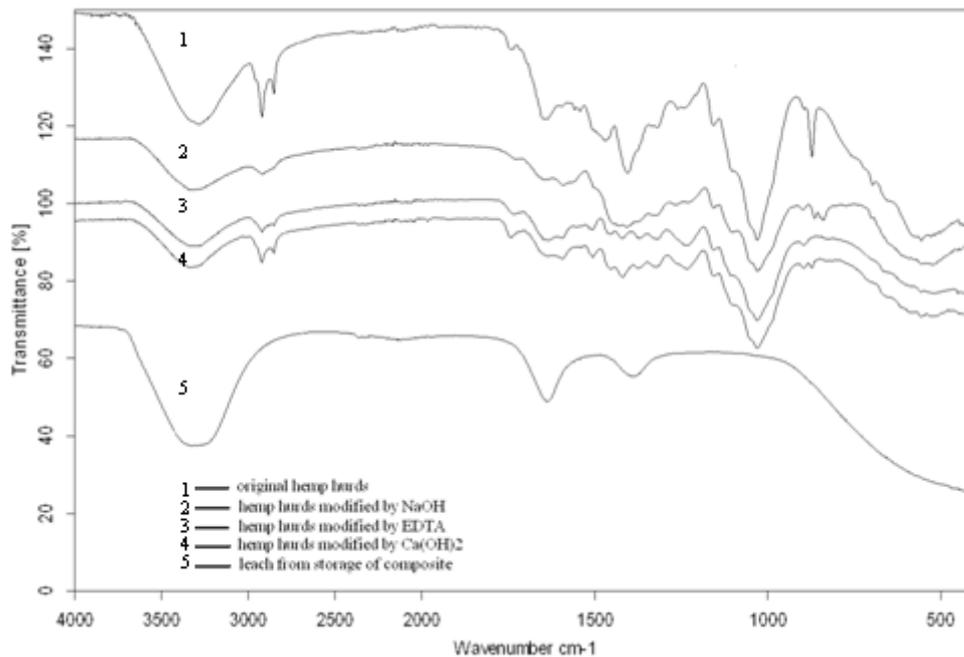


Figure 1: Comparison of FTIR spectra of the original hemp hurds (1) and chemically modified hemp hurds by NaOH, EDTA, Ca(OH)<sub>2</sub> (2-4) and leachate (5)

First water molecules diffuse into the micro gaps inside inorganic matrix. Then molecules are capillary transported of into the gaps and flaws at the interfaces between hemp fibre and/or shives and the matrix during immersion of composites in water. Further water molecules penetrate into hemp structure, mainly into shives spaces as well as into capillaries and spaces between bundles of fibres and fibrils, which presence can be not eliminated. The cellulose structure can be destroyed by penetration of water molecules into the cellulose network of the fibres. The initial absorption stage results in poor wetting and water impregnation of hemp material. According to papers (Karmakar et al., 1999) and also (Ayensu, 2000), high amount of water causes swelling of the fibres and/or shives. Due to the swelling of the hemp material, microcracking in the matrix of tested composites occurs. These microcracks can be filled with water. However, it is not only water absorption important but also the rate at which the sorption as well as desorption of water molecules takes place (Bruijn et al, 2009). Another phenomenon of water sorption is connected with the additional hydration of MgO-particles during water storage of composite. On the other hand there is dissolution of alkaline component in the matrix (NaHCO<sub>3</sub>), leading to increased pH values of solution up to 11.

Table 4: Values of water absorption, thermal conductivity coefficient, density and compressive strength of composites after 28 d of duration in water

Mixture	Water content (%)	Thermal conductivity coefficient (W/m.K)	Density (kg/m <sup>3</sup> )	Compressive strength (MPa)
1	7.27	0.068	1100	1.01
2	14.1	0.073	780	1.15
3	21.9	0.089	720	1.45
4	10.6	0.123	790	1.91

Values of thermal conductivity coefficient of composites were measured in the range of 0.068 - 0.123 W•m<sup>-1</sup>•K<sup>-1</sup>. Referential composite has the lowest value of thermal conductivity coefficient. From the composites based on modified hemp has the highest value of thermal conductivity coefficient sample with hurds modified with EDTA. The density values of samples was found to be in the range of 720 – 1,100 kg•m<sup>-3</sup>. The found values of thermal conductivity coefficient and density of hemp composites are comparable to other building materials such as aerated autoclaved concrete (for thermal conductivity coefficient: 0.04-0.17 W•m<sup>-1</sup>•K<sup>-1</sup>; for density: 800 – 1,200 kg•m<sup>-3</sup>).

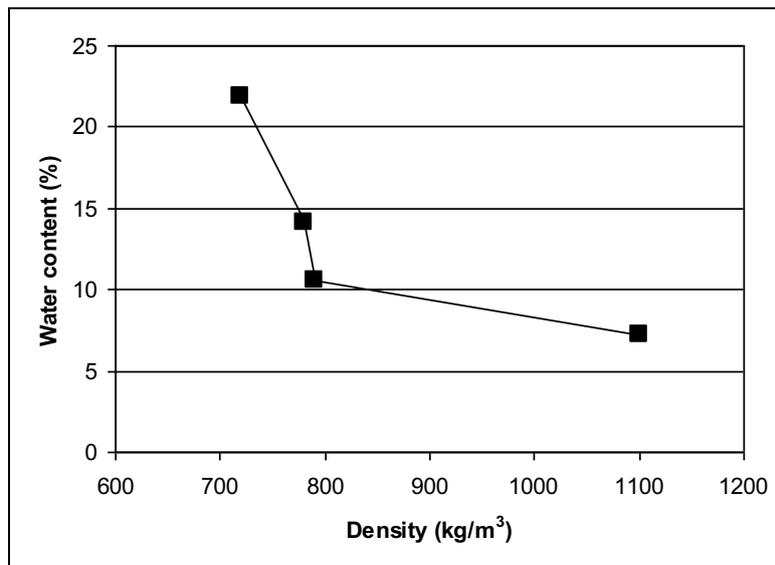


Figure 2: Dependence of water content in composites on density of immersed composites

As it can be seen in Figure 2, values of water content of samples are decreasing with increasing values of density prepared lightweight composites. This is probably due to more dense structure of composites which contain fewer pores. As regards compressive strength of composites after water storage, these values ranged from 1.01 to 1.91 MPa. In the case of referential composite, it has lower value of compressive strength in comparison with composites prepared with chemically modified hemp hurds (Table 4). The highest value of compressive strength was measured for composite with hemp modified by EDTA. This finding is accordance with data published in (Tröedec, et al., 2009). Compressive strength of immersed composite samples increases with decreasing values of density as shown in Figure 3. This behaviour of water immersed composites is different from that during hardening process, where the denser is the specimen, the higher the compressive strength is.

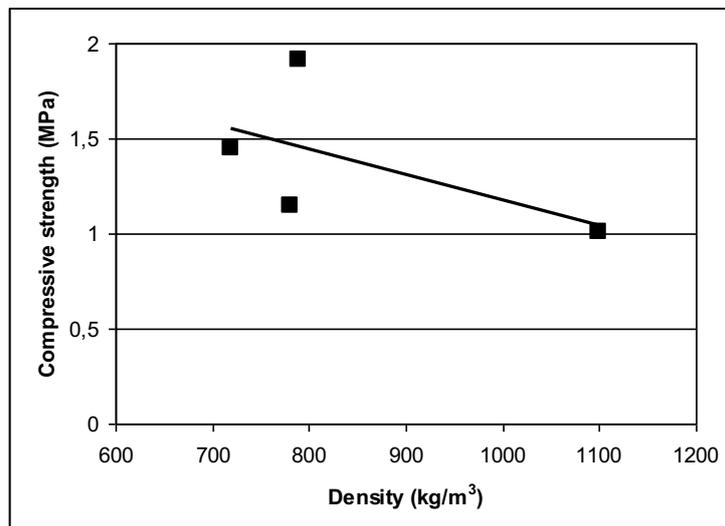


Figure 3: Dependence of compressive strength on density of immersed composites

#### 4. Conclusions

In this paper, some aspects of durability of hardened composites based on chemically modified of hemp hurds slices in three solution and referential composites with MgO-cement as binding agents after storage

in deionised water was studied. Some important material properties as water absorbability, thermal conductivity coefficient, apparent density and compressive strength of water immersed composites were tested. FTIR spectra showed a partial removal of hemicellulose, lignin and removal of waxes and oil was performed. On the other hand, spectra don't confirm the leaching of residual contents of organic compounds such as hemicellulose and lignin from composites into the deionised water whereas carbonates leaching were confirmed. By XRF analysis, the leaching of Na, Ca, Mg, Mn, Fe, Al, Si from composites was detected. The determined values of thermal conductivity coefficient and density of hemp composites are comparable to other building materials. In case of composites based on chemically treated hemp hurds the higher values of compressive strength were observed in comparison with referential composite (original hemp hurds). The highest value of compressive strength has composite prepared with treated hemp hurds with EDTA.

Processes occurring in composite materials based on natural cellulosic material and alternative binder are complicated. Their observation will be executed in our further research.

### Acknowledgements

The authors are grateful to the Slovak Grant Agency for financial support of the project VEGA1/0231/12.

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