ISSN 1392 – 1231. CHEMINĖ TECHNOLOGIJA. 2016. Nr. 1 (67)

MgO-based cement as an inorganic binder for hemp hurds composites

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crossref http://dx.doi.org/10.5755/j01.ct.67.1.15000

Received 16 May 2016; Accepted 30 June 2016

The aim of this work is to study the suitability of the MgO-based cement as an inorganic binder instead of the traditional Portland cement into composites with an organic filler material such as hemp hurds. MgO-based cements, in contrast to Portland cement which requires high temperatures (about 1450 °C) during its production, demand less energy (the maximum temperature for the controlled calcination to obtain MgO from magnesium carbonate is 750 °C), becoming a more efficient cement from the environmental point of view.

The first part of this paper summarises the results of physico-mechanical properties such as density, thermal conductivity, water absorbability and compressive strength of hardened hemp hurds composites that make this material useful and interesting mainly for its thermal insulating properties which can be improved by hemp hurds treatment processes. The second part of this work is devoted to the characterization of the MgO–cement matrix in the 28-day hardening stage. The MgO-based cement as an alternative binder appears to be suitable for the preparation of biocomposites based on hemp hurds.

Key words: hemp hurds, composite, physico-mechanical properties, MgO–cement matrix

Introduction

Principles of the sustainable construction of buildings bring new requirements for building materials [1]. The actual strategic role in a sustainable building consists of the rational use of material and energy resources by the controlled minimization of the total production of emissions. The priority task of the sustainability of structures is the development of building materials with qualitatively better physical properties. Progressive building materials are developed on the base of environmentally friendly materials [2]. The research and development of new building materials lead to the replacement of cement by non-traditional active ingredients of natural character and the use of secondary raw materials. The environmental concerns of energy utilization in buildings to provide indoor comfort have given rise to an intense research over alternative construction materials. In this sense, the use of bio-based materials or biocomposites based on natural plant fibres is very attractive because its highly sustainable and renewable character allows reducing energy and raw materials [1]. Moreover, their environmental impact is lower than that of traditional building materials, because relatively large amounts of atmospheric CO_2 can be sequestrated through photosynthesis [3].

Among the new vegetable fibres utilization, hemp-based biomaterials stand out from the rest because of their wide availability, low requirements of fertilizers and irrigation, permanent renewal character, good humidity control and very favourable energy and ecological balances [4]. Hemp materials have attracted great attention in the recent years also due to their excellent thermal insulation, acoustic and antiseptic properties, environmental benefits and low cost [5]. The seeds, fibres and woody core (hurds) can be profitable in many manners. The woody core or hemp hurds make up 40–60 % of the mass of the hemp stalk and can serve as animal bedding or more recently as an aggregate for bio-composites [4, 6]. A hemp bio-composite is commonly formed by the hemp hurds and a mineral binder. The initial studies were carried out with Portland cement as the binder, although other compounds such as lime were later tested in order to improve the performance. Thus, hemp hurds composites can be obtained. Both materials present an excellent hygrothermal behaviour, and they can be used as insulators in walls, floor, and roof [7].

According to Collet and Pretot (2014), hemp composites induce a reduction ranging from 15 % to 45 % in energy consumption, depending on the ventilation strategy used [8]. Despite of its great hygrothermal performance, this hemp material presents low mechanical properties that restrict its use for structural purposes as a not load-bearing material, being often associated with a rigid frame [9]. The incompatibility between natural fibres and matrix, caused by heterogeneity and hydrophility resulting in a high moisture sorption sensitivity of the natural material is the key problem for a successful hemp hurds application, because it leads to a low interface strength in comparison with glass or carbon fibre composites. Many research projects have been devoted to study the enhancement of the adhesion at the fibres–matrix (polymer or mineral) interface, the use of various methods for the modification of the surface fibres and matrix.

Optimizing the adhesion of fibres consists in their physical/chemical pre-treatment leading to removing impurities and amorphous components (pectin, lignin, hemicelluloses) and separation of the bundles of fibres in the fibrils [10, 11]. These low compressive strength values led to the search for alternative binders to provide an enhanced mechanical performance while complying with sustainability and recyclability aspects. Alternative binders such as lime, hydrated lime, pozzolanes, MgO–cement have been studied in the recent years in terms of reducing the environmental footprint associated with the Portland cement (PC) production and the development of sustainable building materials [12]. Magnesium oxide is formed by magnesite calcination at a lower temperature in comparison with calcite decomposition to CaO in PC production. In order to reduce the cost of the magnesium source, a by-product from the calcination of natural magnesite was used instead of pure or high-grade magnesium oxide. Taking into account all the above-mentioned facts and in order to increase the mechanical performance and sustainability criteria of hemp hurds composites, the aim of this study was to develop and characterize a new bio-based composite and environmentally friendly material composed of hemp hurds and MgO–cement. In our previous papers [13–16], MgO–cement as a mix of MgO and SiO_2 with $NaHCO_3$ addition has been used in composites based on this biomass, and the technically significant properties of hardened composites were studied.

In this paper, the results of physico-mechanical properties of lightweight composites based on unmodified and modified hemp hurds and the nonconventional binder MgO–cement in time dependence are given.

Currently, the behaviour of the MgO–cement system in a composite during the hardening process has been investigated.

Materials and methods

The technical hemp hurds originating from the Netherlands company "Hempflax" was used as a filler material of lightweight composites in the experiments. The used hemp material consisted of a large majority of core fibres (hemp hurds) over bast fibres, and it also contained fine dust particles originating from the manufacturing crushing process. The used hemp material was polydispersive (Fig. 1) with a wide mean particle length distribution (8–0.063 mm), and its density was 117.5 kg/m^3. Hemp hurds consist of cellulose (44.5 wt.%), hemicelluloses (32.78 wt.%), lignin (21.3 wt.%), extractives (3.57 wt.%) and ash (3.04 wt.%).

MgO–cement was used as a binder in the experiments, and it consists of magnesium oxide obtained by high-temperature decomposition of natural magnesite (CCM 85, SMZ Jelsava, Slovakia), silica sand (Sastin, Slovakia) and sodium hydrogen carbonate (p.a.) (Gavax, Slovakia).

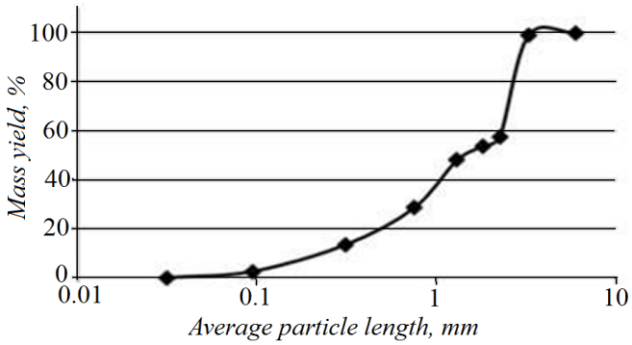

Fig. 1. Mean hemp particle length distribution

Calcined magnesium oxide contains 84.68 % MgO, 7.21 % Fe_2O_3, 5.28 % CaO, 0.65 % SiO_2, and its loss on ignition is 0.85 %. MgO has been dry-milled (laboratory vibratory mill VM 4 for 5 min) in order to reduce its particle size [17]. Its particle size distribution is shown in Fig. 2. The mean particle diameter calculated from granulometric data was 6.85 μm. The specific surface area of milled MgO (15.04 m^2/g) was estimated by a low-temperature gas adsorption using nitrogen (Quantachrome NOVA 1000e, USA).

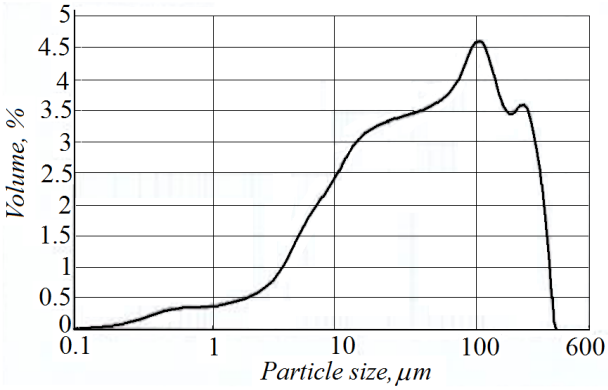

Fig. 2. MgO particle size distribution

Certified silica sand with 97.5 % of SiO_2 content and the mean particle diameter 386 μm was used in the MgO–cement.

The chemical modification of dried hemp hurds was made by three different solutions: sodium hydroxide (NaOH) p.a. (CHEMAPOL, Czechoslovakia), calcium hydroxide ($Ca(OH)_2$) ≥ 96 % pulverised (ROTH, Germany), ethylene-diamine-tetracetic acid ($C_{10}H_{16}O_8N_2$) (EDTA) p.a. (GAVAX s.r.o., Slovakia). The chemical treatment conditions are described elsewhere [18].

The physical modification of hemp materials was carried out by their treatment in hot water (100 °C) and by the ultrasonic treatment procedure where hemp hurds slices were placed into an ultrasonic cleaner bath (TESON 10, 220 V, 50 Hz, 650 W) with deionized water as a cleaning medium. In both cases the *s* : *l* (solid to liquid phase) ratio was 1:10, and the time of treatment was 1 hour.

Experimental specimens were prepared according to the recipe [19] and consisted of 40 vol.% of hemp hurds (unmodified as a referential material and modified), 29 vol.% of MgO–cement and 31 vol.% of water. The

components of the mixture were homogenized in a dry way and then mixed with water addition. Standard steel cube forms with dimensions 100×100×100 mm were used for the preparation of samples in accordance with the STN EN 206-1/A1 standard [20]. The samples of MgO–cement-based composites were cured for 2 days in an indoor climate, and then they were removed from the forms and covered with a foil.

The curing was continued under laboratory conditions during 7, 28, 60 and 90 days.

The physico-mechanical properties (thermal conductivity coefficient, density, water absorbability, compressive strength) were measured on hardened specimens under laboratory conditions. The resulting values are the average of three measurements.

The thermal conductivity coefficient of samples as the main parameter of heat transport was measured by the commercial device ISOMET 104 (Applied Precision Ltd., Germany). Density was determined in accordance with the STN EN 12390-7 standard [21]. Water absorbability (after one hour) was specified in accordance with the STN EN 12087/A1 standard [22].

The compressive strength of all cube specimens under controlled conditions after the hardening time was determined as the maximum load per average cross-sectional area by using the instrument ADR ELE 2000 (International Limited, United Kingdom in accordance with the standard STN EN 206-1/A1) [20].

The thermal degradation of the binder was evaluated by a thermal analysis (DSC-TGA), using a STA 449F3 Jupiter (Netzsch, Germany) apparatus under the inert atmosphere.

The X-ray diffraction (XRD) analysis was performed to evaluate the mineralogical composition of the inorganic binder, using the Bruker D2 Phaser diffractometer (Bruker AXS, GmbH, Germany) in the Bragg–Brentano geometry (configuration Theta-2Theta), using the 1.54060 Å CuKα radiation, Ni Kβ filters and a scintillation detector at a voltage of 30 kV and the current of 10 mA. Scan conditions: recording time about 2 hours, the step size of 0.04° (2θ), and the step time of 3 s. The XRD patterns were processed using the Diffrac.EVA v. 2.1 software. The ICDD PDF database (ICDD PDF – 2 Release 2009) was utilized for the phase identification.

Results and discussion

The values of the thermal conductivity coefficient of all MgO–cement-based composite samples (after the hardening time of 28, 60 and 90 days), measured in the range of 0.08–0.1 W/(m·K), are comparable with other insulation building materials. The density values of these lightweight composites based on hemp hurds were determined in the range of 850–1100 kg/m^3.

The values of water content in specimens prepared with modified hemp hurds (14–8.5 %) are considerably lower in comparison with a composite with an original sample (20 %). According to [23], a high amount of water causes the swelling of the fibres and/or hurd slices. Due to the swelling of the hemp material, the micro-cracking in the matrix of the tested composites occurred. These micro-cracks can be filled with water. Another phenomenon of water sorption in a composite is connected with the additional hydration of MgO particles during the water storage of a composite.

The dependence of MgO–cement-based biocomposites compressive strength on hardening time is shown in Fig. 3. The values of the compressive strength of hardened composites ranged from 0.90 to 5.75 MPa. As shown in Fig. 3, the progression in the values of this strength parameter is observed in dependence on hardening time for all composite samples. The measured values of the strength parameter of composites containing MgO–cement and untreated hemp hurds have hundred-fold higher values of compressive strength in comparison to composites based on a combination of hydrated lime and cement [24]. Biocomposites based on hot-water-treated and chemically treated hemp hurds have slightly lower values of compressive strength in each hardening time in comparison with the reference sample. No significant differences in determined strength values of MgO–cement-based composites with chemically treated hemp hurds have been observed. The cause of this phenomenon can be the nature of the used binder and the surface properties of the filler which led to a poor interaction of binder particles and hemp hurds slices [25].

The highest values of strength parameters were noted in the specimens prepared with ultrasound modified hemp hurds. It seems that this fact is related to the ultrasound process mechanism. The phenomenon known as cavitation as the most striking property of ultrasound is responsible for a sudden collapse of the cavities creating power shock waves and generating a large amount of mechanical and thermal energy in the water [26]. The local high temperature and the pressure placed in the volume of the liquid arise, and consequently the complex structure of the bundles of fibres and fibrils is broken. The effect of these conditions causes changes not in the contents of non-cellulosic constituents of hemp hurds but in the degree of cellulose polymerization from 1.302 to 585 as compared to the original hemp hurds sample as shown in [27, 28].

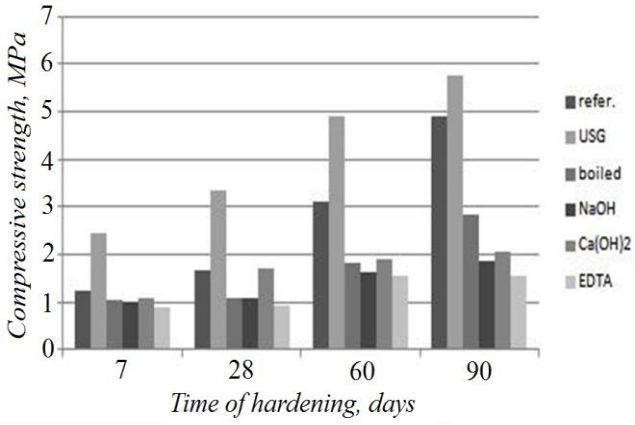

Fig. 3. Compressive strengths of MgO–cement-based biocomposites in time dependence. Hemp hurds: refer – untreated; USG – ultrasonic treatment; boiled – treatment in hot water; NaOH – modification by sodium hydroxide; $Ca(OH)_2$ – modification by calcium hydroxide; EDTA – modification by ethylene-diamine-tetracetic acid

The DSC, TG and DTG curves of the MgO–cement-based composite matrix sample measured in the nitrogen atmosphere are shown in Fig. 4.

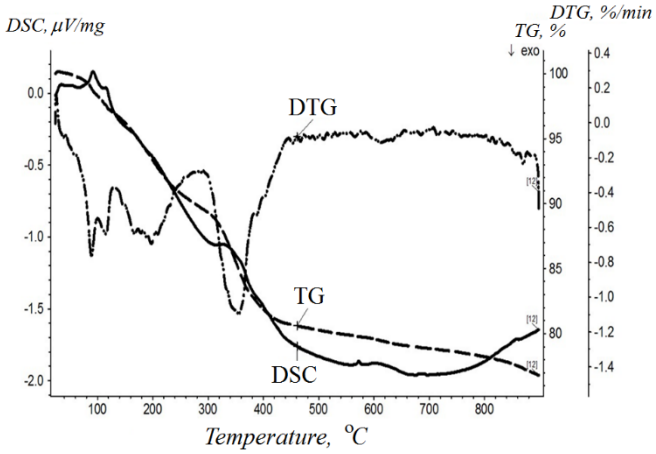

Fig. 4. TG, DTG and DSC curves of the MgO–cement matrix of the 28 days hardened hemp hurds composite

The DSC curve shows several small endothermic peaks below 600 °C and a wide exothermic peak above 600 °C. Five weight loss steps on the DTG curve were found. In the temperature range from 24 to 131 °C with the top at 89 and 113 °C, a 3.05 % weight loss is attributed to the desorption of physically adsorbed water and/or crystalline bound water in the $NaHCO_3$ structure. An about 2.79 wt.% weight loss should be achieved if the release of water molecules from $NaHCO_3$ ($Na_2CO_3.H_2O$) is complete. The following decomposition stage from 130 to 291 °C, corresponding to a 7.37 % weight loss, is likely due to the slow release of the carbon dioxide molecules from $NaHCO_3$ which is accelerated at a temperature above 250 °C. The calculated weight loss due to CO_2 release is 6.84 %, but concurrently in this temperature range the dehydroxylation of $Mg(OH)_2$ starts in MgO and water. According to data published in [29], the decomposition of a crystalline form – brucite – with the hexagonal structure forming periclase with the cubic structure occurs in the temperature range from 200 °C to 500 °C.

The thermo-gravimetric analysis of mechanico-chemically synthesized magnesium silicate showed that $Mg(OH)_2$ loses its structural water at 400 °C [30]. Based on data published in [30], the decomposition of the synthesized magnesium silicate hydrate (M-S-H) occurs throughout the temperature band 20–1000 °C being most intensive at 200–500 °C. The weight loss observed (8.8 %) in the temperature range between 291 °C and 450 °C with the maximum peak at 355 °C could indicate a continuous thermal decomposition of brucite and/or hydrous magnesium silicate gels formed during the hardening process of the composite. However, the measured weight loss due to the dehydroxylation of $Mg(OH)_2$ is lower in comparison with the calculated value (9.69 %). The further weight loss above 600 °C can be connected with the release of structurally bonded hydroxyl groups in the M-S-H gel and the decarbonation of carbonated phases.

The results indicate that the conversion degree of $Mg(OH)_2$ into the M-S-H gel in the composite system during 28 days is low because of the low reactivity of the starting materials – best commercially available natural raw materials SiO_2 and MgO. Magnesium oxide, calcined at a relatively high temperature of magnesite (750 °C) containing CaO impurities, has the potential to significantly increase the *pH* through its dissolution. However, such a M-S-H gel can be formed from the precipitated $Mg(OH)_2$ under a low *pH* value (around 10.5) [31]. MgO–cement due to the $NaHCO_3$ component has a higher *pH* (around 12.6). Based on published data about the formation of M-S-H gels in concrete formulations [32] and cement-clay interactions [33], magnesium silicate hydrate is a poorly crystalline (disordered) phase. As shown in Fig. 5, the presence of only starting crystalline components such as periclase, quartz and $NaHCO_3$ transformed to Na_2CO_3 ($Na_2CO_3{\cdot}H_2O$) on XRD patterns was identified. The XRD broad peaks representing a poorly crystalline structure of the pure M-S-H gel are in the 32–37° 2θ and 58–62° 2θ regions [32, 34]. In Fig. 5, there is no evidence that the formation of disordered M-S-H occurs.

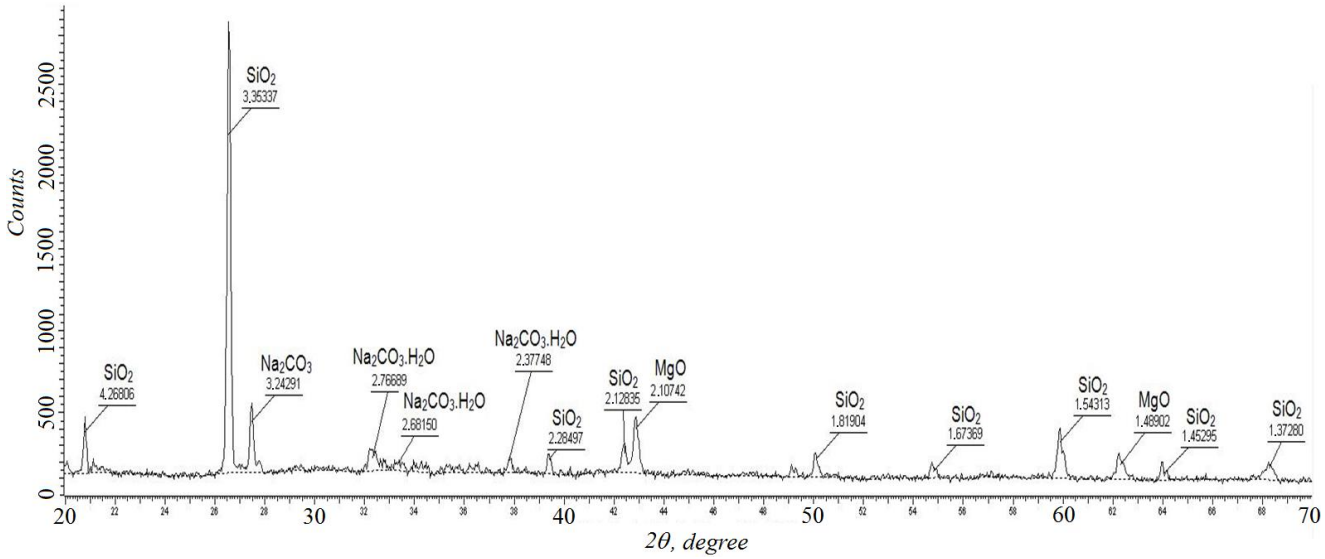

Fig. 5. XRD patterns and *d*-spacing values of the MgO–cement matrix

Based on these preliminary results of the MgO–cement matrix behaviour study, it is very difficult to explain the processes occurring during hardening. The understanding of the hydration behaviour of MgO–cement in the MgO-SiO_2-H_2O system in composite requires an extensive investigation of the M-S-H formation as a potential cementing phase in a hemp hurds composite by using the most reactive precursors.

Conclusions

In this paper, the physical and mechanical properties of the MgO–cement-based composites with hemp hurds as the reinforcing filler and MgO–cement behaviour in the matrix have been investigated. Based on the results described above, the following conclusions can be drawn:

- the physical and mechanical properties of the composites are influenced by the treatment process of hemp hurds in dependence on changes in the structure of hemp hurds constituents;
- an increase in the compressive strength values of all composites with increasing the hardening time was recorded. The highest values of the strength parameter were reached in case of ultrasonic modified hemp hurds due to the morphological structure changes brought about by this physical procedure on the fibrillary level, leading to an about 30 % decrease in the degree of cellulose polymerization as compared to the original hemp hurds sample;
- the MgO–cement as an alternative binder appears to be suitable for the preparation of biocomposites based on hemp hurds. However, there is still a need to study the hydration behaviour of a binder with a higher activity and the properties of the binder/matrix interface in detail to develop durable biocomposites.

Acknowledgements

The authors are grateful to the Slovak Scientific Grant Agency for the financial support of the projects VEGA 1/0277/15.

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NEORGANINIS MgO CEMENTO RIŠIKLIS KANAPIŲ SPALIŲ KOMPOZITUI

S a n t r a u k a

Darbo tikslas – ištirti neorganinio MgO cemento rišiklio tinkamumą vietoj tradicinio portlandcemenčio kompozituose su organiniais priedais (kanapių spaliais).

Aplinkosaugos požiūriu MgO cementams gaminti sunaudojama mažiau energijos (magnio karbonato valdomas skilimas į MgO vyksta ne didesnėje nei 750 °C temperatūroje) nei portlandcemenčiui gaminti (apie 1450 °C). Pirmojoje straipsnio dalyje pateiktos hidratuotų kanapių spalių kompozitų fizikinės ir mechaninės savybės: tankis, terminis laidumas, vandens įmirkis ir stripris gniuždant. Pažymėtina, kad minėtos savybės, ypač terminės izoliacinės, gali būti pagerintos naudojant skirtingus kanapių spalių apdorojimo procesus.

Antrojoje straipsnio dalyje apibūdinta MgO cemento matrica po 28 kietinimo dienų. MgO cementas, kaip alternatyvi rišamoji medžiaga, yra tinkamas biokompozitams su kanapių spaliais gaminti.

Reikšminiai žodžiai: kanapių spaliai, kompozitas, fizikinės ir mechaninės savybės, MgO cemento matrica.