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Finale report of the project *"Mechanical fracture quantification of role of hemp fibres on self-healing processes in selected composites (KvaRK)''*

Introduction:

Fracture parameters in quasi-brittle materials could also quantify their resistance to crack propagation, essential for assessing load capacity and ensuring the reliability and durability of structures. Quasi-brittle materials encompass a wide range of materials, including concrete, rocks, ceramics, and biomaterials. The addition of fibers, typically steel or synthetic, enhances composite resistance to crack initiation by extending the fracture process zone (FPZ). However, the high cost and energy requirements of traditional fibers have spurred interest in eco-friendly alternatives, such as agricultural wastes. Natural fibers, such as hemp, flax, sisal, and coir, have garnered significant interest in the fiber-reinforced materials domain over the past decade. This attention stems from their low production costs and minimal energy consumption during manufacturing. Moreover, natural fibers offer the advantages of full biodegradability, renewability, and global availability. Remarkably, their tensile strength and E-modulus are in the same range with those of synthetic fibers.
Mechanical fracture parameters for composites with dispersed fibers are determined through fracture tests under various loading configurations. These tests yield information on Young's modulus, fracture toughness, and specific fracture energy, crucial for understanding crack initiation and propagation resistance. Fatigue testing, crucial for structures enduring repeated loading, is commonly conducted via flexural tests. Innovations in materials science have led to the development of self-healing concrete mixtures, capable of repairing microcracks autonomously. Various methods, including autogenous healing and the use of capsules with healing agents or bacteria, show promise.
The study aimed to explore the potential of self-healing using natural fibers. These fibers, being hydrophilic, can absorb water, which is then utilized to react with calcium oxide, leading to the production of portlandite.

Project goals:

The project "Mechanical fracture quantification of role of hemp fibres on self-healing processes in selected composites (KvaRK)'' aimed to investigate the influence of distributed hemp fibers on the self-healing mechanisms within selected fine-grained composites featuring a brittle matrix. The primary objective is to quantify the impact of hemp fibers on the controlled damage of these materials through a combination of numerical analyses and advanced evaluation of experimental data derived from fatigue and mechanical fracture tests. This goal was achieved by fostering collaboration among three scientific teams with extensive expertise in fatigue experiments, fracture testing, and materials incorporating hemp fibers: the teams from the Institute of Physics of Materials, Czech Academy of Sciences, the Faculty of Civil Engineering at Brno University of Technology, and the Faculty of Civil Engineering at TU Wien. Furthermore, the specific aim of the project involved conducting a pilot study to analyze the self-healing processes within selected fine-grained cement-based composites. This analysis was focus on the transport of water, facilitated by soaked hemp fibers, to initiate secondary hydration of residual cement within the inner structure of the material. The project quantitatively assessed the role of fibers in self-healing through the evaluation of experimental and numerical data obtained from appropriate fatigue and fracture tests, where the materials will be subjected to controlled loading-induced damage and subsequent healing. In this report, we will not present all the findings from the bilateral research project, as some have already been published, and another scientific paper is currently in the process of publication, where additional results will be showcased.

Experimental methods:

Materials:

The mortar mixture design of all specimens was with the ratio of cement : sand : water equals to 1: 3 : 0.5. As cement, the Ordinary Portland Cement CEM II was used and for the sad, the natural aggregate with the particles 0.4-0.8mm was used. Primary bast hemp fibers (Cannabis sativa L) were employed for fiber reinforcement. Hemp fibers were preferred due to their extensive utilization in reinforcing cementitious materials and their longstanding cultivation tradition in Europe. Table 1 outlines their properties. The fiber length and dosage were determined through a literature review to achieve optimal values, balancing mechanical properties in the hardened state with fresh state properties such as workability and effective fiber dispersion within the matrix. To achieve a length of 10 mm, a small bundle of fibers (approximately 20–30 fibers) was manually measured with a ruler and cut using scissors.

Table 1. – Hemp fibres' properties

Fibres	Tensile strength*[MPa]	Density[kg/m^3]	Diameter*[μm]	Length[mm]	Dosage by value of the total mixture [%]
Hemp fibres	270–900	1500	8–600	10	1

*data from the literature: L. Yan, B. Kasal, L. Huang A review of recent research on the use of cellulosic fibres, their fibre fabric reinforced cementitious, geo-polymer and polymer composites in civil engineering Compos. B, 92 (2016), pp. 94-132, 10.1016/j.compositesb.2016.02.002

During the project, 30 prisms with the dimension 40 x 40 x 160 mm^3 were cast for the three-point bending test (3PBT) and compressive test and one cylinder dimensions 150 mm diameter and 300 mm height for the Brasilian Disk test (Table 2). In the finale report the

Brasilian Disk will not be discussed, since the results of this testing were not completely analyzed by the time, this reported was written.

Table 2. – Specimens denotations and mixture design

Denotation	Mixture design cement:sand:water	Hemp fibres	Specime n type	Specimen Number	Data of casting
M	1:3:0.5	no	Cylinder	1	09.06.2022.
P	1:3:0.5	no	Prisms	30	09.06.2022.
F	1:3:0.5	yes (0.5vol%)	Prisms	30	09.06.2022.

Specimens' preparation:

For the static fracture tests standard prism specimens ($40 \times 40 \times 160$ mm^3) were cast according to the norm EN 196-1. Before molding, the specimens were vibrated on a vibration desk, in order to avoid pores, coming from the entrapped air, during mixing. After the specimens were cast they were covered with a plastic sheet for 24h, at the laboratory conditions (temperature of 20 ± 2 °C and relative humidity of 50 ± 5. Afterwards, the specimens were cured in water, till the day of testing. For each mortar group (with and without the fibres) the prisms were divided into two groups. The first group was tested at the age of 28 days and the other group of the mortars was exposed to the fracture break (during 3PBT the maximal force was reached), after what the load was stopped before the crack progressed and expanded. Then these prisms were put in water and tested again on the same tests after two years, in order to investigated if the fibre reinforced mortar could heal the crack more than non-reinforced mortar. For the Brazilian test, the cylindric specimen were cast. They mixture was filled stepwise to a third and in each layer the entrapped air removed by hand rodding (25 times). The curing and ageing of the specimens were the same as for the prism specimens.

Density Test:

Bulk density serves as a very important indicator for mortar performance. It was determined for specimens aged 28 days. Both group of mortar prisms underwent testing using six mortar prism specimens measuring $40 \times 40 \times 160$ mm³, as outlined below:

D [kg/m^3]= Mc/Vc, where Mc is the mass and Vc the volume of the test specimen. The volume of the prisms was claculated as the multiplication of the prisms' length, width and height.

3PBT Test:

The static fracture test was performed on the standard prismatic specimens with nominal dimensions 40 mm × 40 mm × 160 mm^3 and span length of 120 mm. From the 3PBT the force-displacement curves were obtained and the following values:

- fracture toughness (K_{IC})

The K_{IC} values can be calculated as follows:

$$K_{\mathrm{I}} = \sigma\sqrt{\pi a}\, Y_{\mathrm{I,3PB}}\left(\frac{a}{W}\right),$$

where a is the crack length, σ is the maximum applied stress at mid-span using measured geometry dimensions, a/W is the relative crack length, and $Y_{\mathrm{I,3PB}}$ is the geometry shape function, which is expressed as a polynomial function according to Brown and Strawley (B.L. Karihaloo, Fracture Mechanics and Structural Concrete (Concrete Design and Construction Series), Ed. Longman Scientific & Technical. United States. (1995))

$$Y_{\mathrm{I,3PB}}\left(\frac{a}{W}\right) = 1.93 - 3.07\left(\frac{a}{W}\right) + 14.53\left(\frac{a}{W}\right)^2 - 25.11\left(\frac{a}{W}\right)^3 + 25.80\left(\frac{a}{W}\right)^4,$$

- material's specific fracture energy G_{f}

The fracture energy was calculated as follows:

$$G_{\mathrm{f}} = \frac{W_{\mathrm{f}}}{A_{lig}}$$

Where Wf was the specific work of fracture, representing the area under the force-displacement diagram. The Wf was calucalted as follows:

$$W_{\mathrm{f}} = \int P(\delta)d\delta.$$

The A_{lig} is the ligament area of the specimens, where the force was applied.

- Young modulus

The Young modulus was calculated as the slope of the curve (linear part) obtained during 3PBT.

Compressive test:

The compressive tests were conducted after the specimens were tested on 3PBT. The tests were conducted on two half of the split specimens by applying the compressive force on a 40×40 mm^2 area on three identical specimens for each mortar group.

Results and short discussion:

In the Table 1, the mortar prisms' dimensions are listed. It could be seen that the density of the hemp fibre reinforced mortars is slightly (1%) lower than the density of the non-fibre reinforced mortars. Generally, the reduction of the density occurs due to the air entrained and the rise in total pore volume through fiber addition to the mortar. Furthermore, hemp fibers possess a lower density (approximately 1500 kg/m3) compared to plain mortar (2054 kg/m3), thereby contributing to a slight decrease in the composite's overall density.

Table 1.- Mortar prims' measurements

Mortar specimens	Weight [g]	Length [mm]	Width [mm]	Height [mm]	Density [kg/m3]
F1	525.3	160.28	40.76	40.02	2009
F2	498.4	160.10	38.03	39.98	2047
F3	516.8	160.14	38.84	40.08	2073
F4	522.2	160.04	40.10	39.96	2036
F5	521.9	160.18	40.40	39.98	2017
F6	535.2	160.16	40.38	40.00	2069
P1	515.2	160.00	40.00	40.10	2007
P2	502.2	157.80	38.72	39.92	2059
P3	511.2	158.50	38.82	40.00	2077
P4	505.3	160.00	38.84	40.05	2030
P5	521.0	160.30	39.36	40.00	2064
P6	529.5	158.30	39.98	40.10	2086

In the Figure 1, the results of the non-fibre reinforced mortar prisms tested at the 3PBT after 28 days were shown. The first 3 specimens (P1, P2 and P3) were exposure to the bending force until the deformation of 20 mm, exceeding the maximal force followed with the almost maximal post-peak behavior. The next three specimens (P1, P2 and P3) were exposure to the same test, but after reaching the max force, the further loading was stopped. The same procedure was followed by the hemp fibre reinforced mortar prisms (Figure 2).

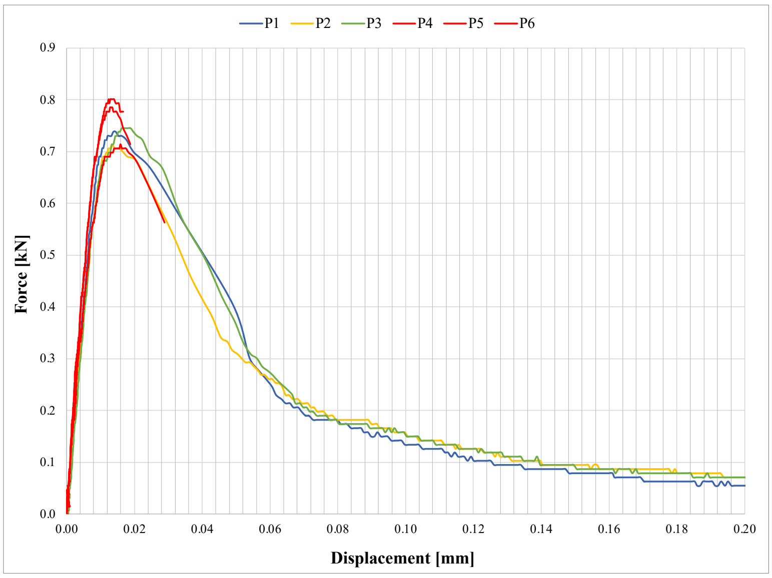

Figure 1. – Force-displacement curves after 3PBT of the non-fibre reinforced mortar prisms tested at the age of 28 days

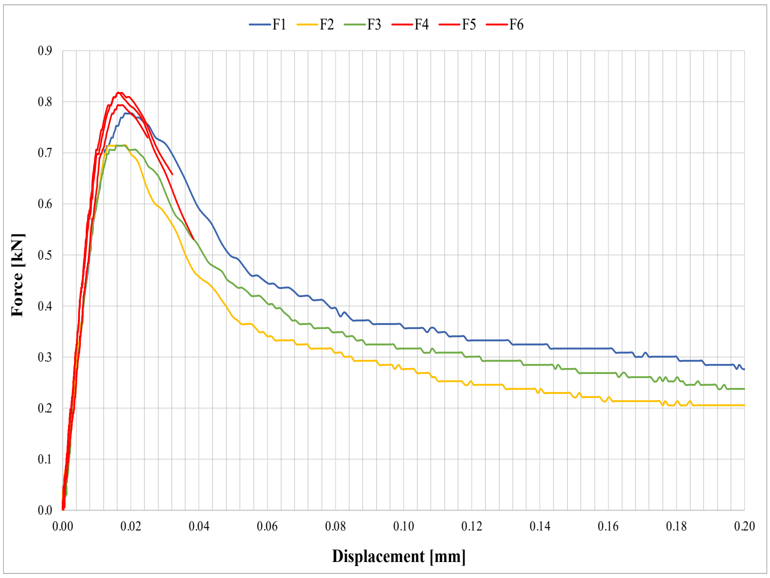

Figure 2. – Force-displacement curves after 3PBT of the hemp fibre reinforced mortar prisms tested at the age of 28 days

As our previous studies also confirmed, the addition of the short hemp fibres lead to the increase of the mortars' toughness in the post-peak behavior. Fiber reinforcement in mortar is crucial for slowing down crack propagation once cracks have formed. This leads to improved energy absorption (toughness) during flexural stress. The inclusion of fibers shifts mortar behavior from quasi-brittle to a more ductile nature. How ductility rate depends on the type, geometry, and quantity of fibers used.

In the Table 2, the calculated mechanical properties from the 3PBT are listed. It was shown, that fracture toughness on average decreases 7%, but the material's specific fracture energy increases on average 3%, after the hemp fibre addition. The Young modulus decreases 16% on average, after the hemp fibres were added (Table 2, Figure 1-2).

Table 2.- The mechanical properties obtained from the 3PBT tested at the prisms age of 28 days

Year of testing: Mortar specimens	2022 K_{IC} [$MPam^{-1/2}$]	2022 E [GPa]	2022 Gc [Jm^{-2}]
F1	0.581	19.42	17.4
F2	0.525	21.92	12.6
F3	0.58	23.25	14.5
F4	0.574	22.75	14.5
F5	0.575	22.95	14.4
F6	0.64	25.67	15.9
P1	0.724	27.48	19.1
P2	0.532	25.39	11.1
P3	0.676	27.54	16.6
P4	0.62	29.58	13
P5	0.612	25.81	14.5
P6	0.564	26.05	12.2

The compressive strength from the specimens is listed in the Table 3. It could be noticed that the slight (1%) compressive strength drop happened in the case of the hemp fibre reinforced mortars, compared to their non-reinforced counterpart. This is also aligned with the obtained density results (Table 1.). Generally, the addition of fibres leads to increased porosity and decreased density, which consequently leads to a lower strength of the mortar.

Table 3.- The compressive strength obtained from the compressive test tested at the prisms age of 28 days

Mortar specimens	Fck1	Fck2	Compressive strength		Average compressive strength
	[kN]	[kN]	[Mpa]	[Mpa]	[Mpa]
F1	42.20	40.10	26.4	25.0	25.7
F2	34.90	33.40	21.8	20.9	21.4
F3	34.00	37.30	21.2	23.3	22.3
P1	40.90	40.20	25.5	25.1	25.3
P2	37.60	36.70	23.5	23.0	23.3
P3	33.30	34.50	20.8	21.6	21.2

As previously mentioned, the results after the fatigue are not presented in this report. However, it is worth mentioning that the obtained tested mechanical properties showed increase and the positive effects of the hemp fibres in healing the cracks in the cement-based mortar prisms. The results are in the detail analyze and will be published in the scientific paper (Q1 Journal).

Conclusion:

Fracture characteristics of composites containing dispersed fibers are determined by conducting fracture tests under different loading conditions. In addition to this, the compressive tests are conducted within the bilateral project. These tests provide essential data on parameters such as Young's modulus, fracture toughness, specific fracture energy and compressive strength which are critical for understanding resistance to crack initiation and propagation. Fatigue testing, important for structures subjected to repetitive loading, is typically carried out using flexural tests. Advances in materials science have resulted in the creation of self-healing concrete formulations capable of autonomously repairing microcracks. Various techniques, including autogenous healing and the incorporation of capsules containing healing agents or bacteria, hold significant promise. This research aimed to investigate the potential of utilizing natural fibers for self-healing purposes. These fibers possess hydrophilic properties, enabling them to absorb water, which then reacts with calcium oxide to produce portlandite. Throughout the project, 30 prisms measuring 40 x 40 x 160 mm^3 each were fabricated. They were designated for the three-point bending test (3PBT) and compressive test. Additionally, one cylinder, with dimensions of 150 mm in diameter and 300 mm in height, was prepared specifically for the Brazilian Disk test. The obtained results proved the mortars' self-healing with the short hemp fibres. However, the further details chemical analysis is need, in order to focus on the durability of the hemp fibres in the alkaline environment, such as cementitious materials. The significant publication resulted from the bilateral cooperation between the teams from the Institute of

Physics of Materials, Czech Academy of Sciences, the Faculty of Civil Engineering at Brno University of Technology, and the Faculty of Civil Engineering at TU Wien. These are listed below, in the Chapter *Publications result of the collaboration.*

Publications result of the collaboration:

[1] Kumpová, I., Lisztwan, D., Poletanovic, B., Vyhlídal, M., Čairović, I., Daněk, P., Šimonová, H., Frantík, P., Merta, I., Rovnaníková, P., & Keršner, Z. (2023). Effect of slenderness ratio on compressive strength value of alkali-activated aluminosilicate composite with ceramic and fly ash precursor. In *Structural and Physical Aspects of Construction Engineering 2022 (SPACE 2022) 5th International Scientific Conference*. SPACE 2022 : 5th International Scientific Conference, High Tatras, Slovakia. AIP Publishing. https://doi.org/10.1063/5.0180791

[2] Seitl, S. (2023). On the fatigue resistance of high performance concrete. In *Structural and Physical Aspects of Construction Engineering 2022 (SPACE 2022) 5th International Scientific Conference*. SPACE 2022 : 5th International Scientific Conference, High Tatras, Slovakia. AIP Publishing. https://doi.org/10.1063/5.0182959

[3] Miarka, P., Seitl, S., Klusák, J., Malíková, L., Merta, I., & Bílek, V. (2023). High-cycle fatigue cracks in concrete investigated by μCT tomography. In J. Pokluda & P. Sandera (Eds.), *Materials Structure & Micromechanics of Fracture 43* (pp. 124–129). Elsevier BV. https://doi.org/10.1016/j.prostr.2022.12.246

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[8] Miarka, P., Seitl, S., Klusak, J., Malikova, L., Merta, I., Bilek, V., High-cycle fatigue cracks in concrete investigated by μCT tomography, Procedia Structural Integrity 43 (2023), pp. 124–129, © (2023), ISSN 2452-3216, doi: 10.1016/j.prostr.2022.12.246

[9] Miarka, P., Simonova, H., Kucharczykova, B., Seitl, S., Poletanovic, B., Merta, I., Fracture and Fatigue Resistance of Concrete with Recycled Aggregate, selected Journal: Construction and Building Materials – *paper in finalization process*

[10] Poletanovic, B., Janotka, I., Janek, M., & Merta, I. (2023). Influence of sodium hydroxide-treated hemp fibres on the mechanical properties of fly ash-based fibre reinforced mortars. In V. Bilek, F. Khestl, P. Miarka, & S. Seitl (Eds.), Proceeding of

the International Conference Non-Traditional Cement & Concrete VII (pp. 56–56). http://hdl.handle.net/20.500.12708/189673
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[13] Seitl, S., Miarka, P., Malikova, L., Merta, I., Poletanovic, B., Bilek, V., Mechanical fracture quantification of role of hemp fibres on self-healing processes in selected composites, Abstract 28th INTERNATIONAL CONFERENCE ON MATERIALS AND TECHNOLOGY, ISBN: 978-961-94088-5-8 (PDF) Copyright © Institute of Metals and Technology, Ljubljana, Slovenia

Further project collaboration:

Years of experimental research on building materials in Austria and extensive analytical expertise in the Czech Republic will lead to detailed insights into material behavior of the hemp fibre reinforced cementitious materials. The establishment of a robust connection between the Institute of Physics of Materials- Czech Academy of Sciences, the Faculty of Civil Engineering at Brno University of Technology, and the Faculty of Civil Engineering at TU Wien will form a solid foundation for future collaboration. Through this bilateral project, the accumulated experience and knowledge will lay a crucial groundwork for future partnerships within European research programs like Horizon 2020, Life+, Eurostars, and bilateral/trilateral projects such as those supported by the Austrian Science Fund (FWF). These collaborative efforts span the European Danube region, with plans for joint research proposals under the EUREKA Danube Region Programs and/or the Interreg Danube Transnational Program.

Output obtained during the project:

In addition to already mentioned scientific findings regarding the self-healing properties of hemp fiber reinforced cement-based mortars, a major advantage lies in the collaborative knowledge exchange among all three teams. This not only enhances the foundation of our cooperation but also sets the stage for future project collaborations. Figures 3. and 4. showcase photos from some meetings in Vienna (Austria) and Brno (Czech Republic) respectively.

Figure 3. – Teams' meetings in Vienna (Austria)

Figure 4. – Teams' meetings in Brno (Czech Republic)