THE INFLUENCE OF A SHORT-TERM TISSUE CULTURE MEDIUM STORAGE ON THE MECHANICAL PROPERTIES OF COMPOSITES BASED ON GLASS FIBERS AND POLYSILOXANE

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Fabric composite materials to be used as bone plates and bone substitutes were investigated. The composites were composed of E- and R-glass fibers and a matrix based on polysiloxane resin. The effect of a short-term storage in the tissue culture medium upon the composite materials elution was studied and, subsequently, their mechanical properties were tested. It was concluded that the composite materials investigated exhibited almost no change in their material properties after storing in the medium used and drying.

INTRODUCTION

Composites as substitutes for bone tissue have been tested for more than the last quarter of the 20th century [1, 2, 3, 4]. Composites reinforced with fibers made of carbon, glasses, polymers, nitrides, carbides, oxides and of matrices based on carbon or various polymers, exhibit, in comparison with metallic substitutes commonly used, in many cases more suitable mechanical properties (especially their stiffness is practically identical with that of the human bone) [4]. Besides testing the mechanical properties of the composites, "in vitro" and "in vivo" tests are applied. When implanted to the human body, these materials are exposed to the somatic liquids, the pH value of which varies from 1 to 9 [3]. Therefore, the solubility of these materials is tested in water as well as in various artificial somatic liquids. In this respect, a great attention is paid especially to dental prostheses.

In two studies [5, 6], the flexural strengths of particle composites with various reinforcements (porous silicon dioxide, quartz, barium glass) embedded in a polymer, after their exposure to deionized water at 60°C for up to 6 months, were compared. In the first place, special attention was given to the negative impact of water on the mechanical strength of the matrix.

The authors of the study [7] investigated the water sorption of unidirectional composites based on E-glass and on various polymers. They emphasized that the water sorption depends mainly on the content of micropores in the matrix.

Behr et al. [8] tested for 30 days in water at 37°C composites based on R- and S-glasses and a polymer, prepared by a procedure resulting in varying the reinforcement volume contents. They found that a higher percentage of fibers did not lead to a higher resistance of the composites.

Other authors [9] investigated unidirectional composites based on prepregs of E-glass with polymethylmethacrylate and three types of diacrylate resins used as matrices. The samples were immersed in distilled water at 37°C for 30 days. The authors measured the water sorption, the flexural strength and the Young's modulus of elasticity of the samples just prepared, of the samples after their exposure to water and after drying. The study showed that the water sorption was lower with the samples with a higher volume content percentage (V_f) of the glass fibers. The dried samples exhibited a higher value of the Young's modulus of elasticity in comparison with the original samples. The authors interpreted this fact by the washing-out of the residual monomers and other small molecules from the matrix which resulted in an increasing stiffness of the matrix. However, this effect was not observed in the case of the flexural strength: its values were higher with dried samples than with the wet ones but they did not attain the values measured with the original dry samples. According to the authors, this fact was caused by the plasticization effect of water on the matrix and on the interface between fibers and matrix. They emphasized the fact that the decrease in the flexural strength depended more on the type of the matrix than on the volume content in the fibers. The higher was the water sorption by the matrix, the higher was the decrease in the flexural strength of the materials.

In this study, fabric composites to be used as bone plates and bone substitutes [10] were investigated and the effect of their short-time exposure in the tissue culture medium upon their elution and mechanical properties were tested.

EXPERIMENTAL

The composites were prepared from a plain-woven V240 fabric (E-glass, VETROTEX, Litomysl, Czech Republic) and from a sateen-woven 21005 fabric (R-glass, VETROTEX, Saint Gobain, France). The glass composition is presented in Table 1.

The siloxane resin Lukosil M 130, polymethylsiloxane - commercial product of Lucebni zavody Kolin, Czech Republic - was used as the matrix precursor [11]. The soaked prepregs were stacked, cured at the temperature of 250 °C and the pressure of 0.5 MPa and without impregnation cut to pieces of the required size (51×8×2) mm. The surface topography of the investigated composites is shown in Figure 1a,b.

The flexural strength of the composites was determined in a three-point bending arrangement and the Young´s modulus of elasticity was determined in a four-point bending arrangement with the Inspekt 100 HT (Hagewald & Peschke, Germany) tester. The in-plane shear modulus was measured using the Erudite (London, UK) electrodynamic resonant frequency tester. The open porosity of samples was determined from density values measured by water penetration according to ASTM C-20 and the surface topography was measured by MahrSurf TS 50/4 (Mahr GmbH, Germany) non-contact measuring equipment.

Six samples from the two series were weighed and then exposed to the tissue culture medium (TCM) D-MEM (Sevapharma a.s., Prague, Czech Republic) at 37°C and *pH* 7 for 2, 4 and 6 weeks. During this exposure, the samples were agitated for 30 minutes every day. In the time intervals mentioned above, samples of D-MEM were taken for determining the extracted silicon by the atomic absorption spectrophotometry method (ASS) with the flame atomization technique, using the Varian Spektra A 880 apparatus (Varian, Australia). The tissue culture medium D-MEM, exposed to the same physical conditions as the samples investigated, was used as a reference solution. The samples were finally taken from the medium, rinsed with distilled water, dried at 120°C up to constant weight and weighed. The tests of mechanical properties of both the original and dried samples were performed.

Table 1. Chemical composition of the glass fibers used.

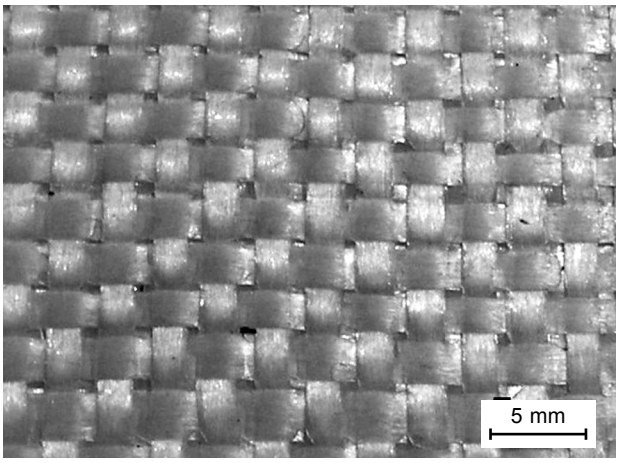
Content (wt.%)	E-glass	R-glass
SiO_2	53-57	58-60
Al_2O_3	12-15	23-25
CaO+MgO	22-26	14-17
B_2O_3	5-8	-
Na_2O+K_2O	0-0.6	-

RESULTS AND DISCUSSION

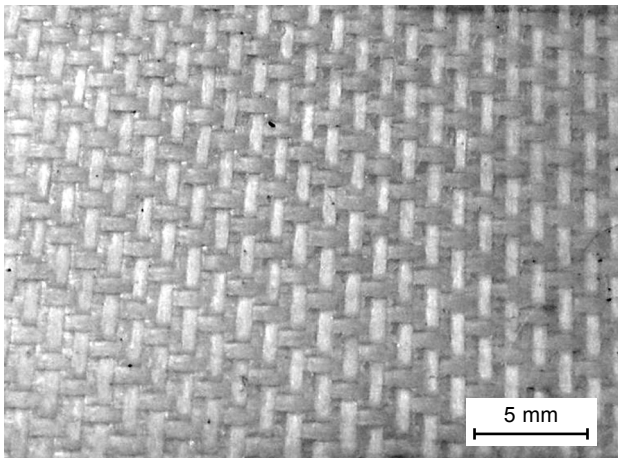
In Figure 2, the mass loss of the composites in dependence on the time of exposure to the tissue culture medium is shown. Whereas with samples based on E-glass reinforcement a practically linear increase in the mass loss with the exposure time was observed, with composites based on R-glass the mass loss stopped and remained constant after 28 days of maceration.

This difference was probably caused by the content in alkali and B_2O_3 with E-glass and by that in CaO with R-glass, see Table. 1 [9]. This was proved also by the determination of eluted Si by AAS, as shown in Figure 3. When the same matrix was used for the two series of samples, the elution depended mainly on the glass reinforcement type.

The structures of the two composites were also considered, see Figure 1 a, b. Whereas the surface of the composites based on E-glass was (due to the cloth weave) considerably open to the intrusion of the solution (see Figure 1a), the surface of the samples based on R-glass (with the sateen weave of the fabric) was more



a)



b)

Figure 1. Surface structure of the composite materials; a) E-glass+M130; b) R-glass+M130.

closed (see Figure 1b). This agreed also with the different values of open porosity with these two samples, see Table 2. The different volume content in the fibers of the composite may also have played a role, see Table 2.

With these two series, no change in the apparent density was observed, within the limits of measurement accuracy, with the time of exposure to the TCM whereas the open porosity increased very slightly, see Figures 4 and 5.

The different values of the mechanical properties of both composites were caused especially by the different V_f and the tensile strength of the two glasses (3.4 GPa with E-glass and 4.4 GPa with R-glass) and, in the case of the modulus of elasticity in shear G, especially by a stronger bond on the interface between the R-glass and the matrix. The values of the mechanical properties, i.e. the flexural strength R_m, the modulus of elasticity in shear G and the Young's modulus of elasticity E - see Figures 6, 7 and 8 - exhibited with the two composites practically no change after the exposure and the drying and they were comparable with the starting materials. The small deviations were close to the sensitivity limit of the applied methods of measurement. Only with the composite based on R-glass a slight decrease in the modulus of elasticity in shear G may have been stated. However, this should be confirmed by further measurements using a longer time of exposure.

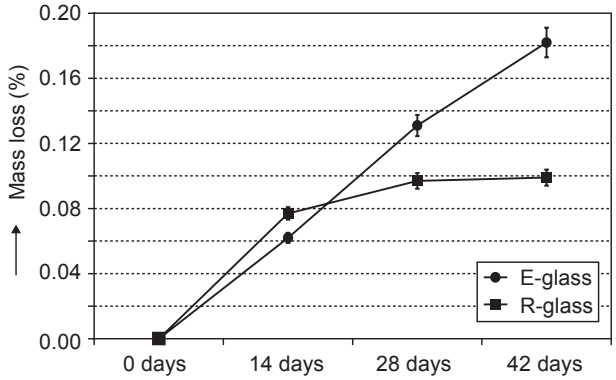


Figure 2. Mass loss of the composite materials in the course of the immersion.

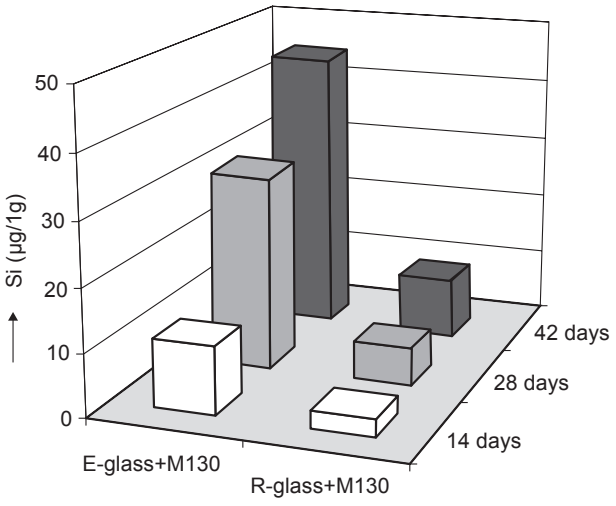


Figure 3. Quantity of the extracted Si (µg/g) in dependence on the time of immersion measured by AAS.

CONCLUSION

The results reported in this study show that the difference between the mass losses of the composites in dependence on the time of exposure to the tissue culture medium is probably caused by the content in alkali and B_2O_3 with E-glass and by that in CaO with the R-glass

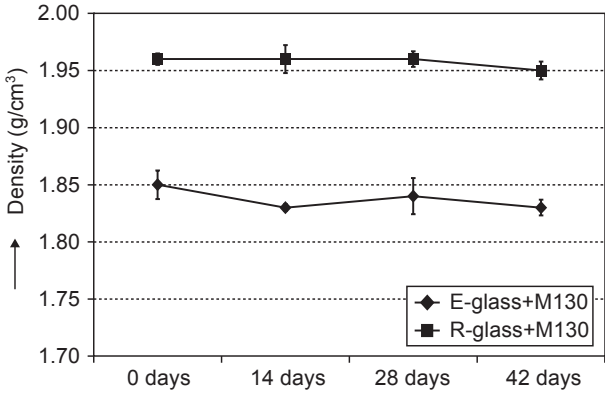


Figure 4. The influence of TCM storage on density.

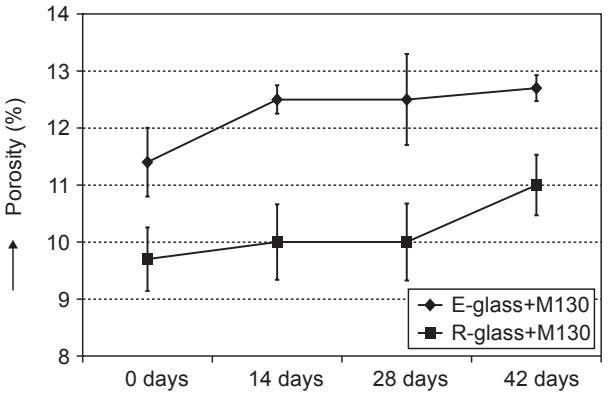


Figure 5. The influence of TCM storage on porosity.

Table 2. Basic properties of the composite materials before immersion.

	V_f (%)	Porosity (%)	Density (g/cm^3)	R_m (MPa)	E (GPa)	G (GPa)
E-glass+M130	51.00	11.40	1.85	152.88	23.18	2.59
R-glass+M130	65.00	9.70	1.96	341.89	50.87	5.76

and also by the more closed surface of R-glass fabric used. The values of the mechanical properties exhibited by the two composites show practically no changes after the exposure and the drying and they are comparable with those of the starting materials. In the course of the further research, the mechanical properties of wet samples will be studied in order to assess whether, after drying, recuperated samples will exhibit in a renewed increase in the values of their mechanical parameters.

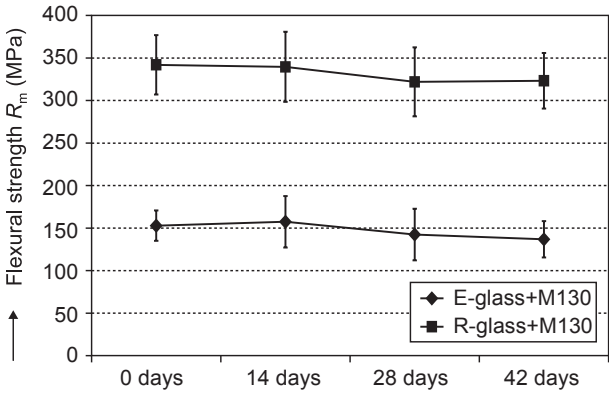


Figure 6. The influence of TCM storage on flexural strength.

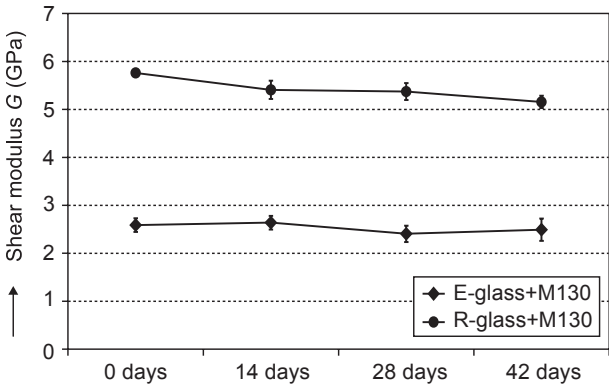


Figure 7. The influence of TCM storage on shear modulus of elasticity.

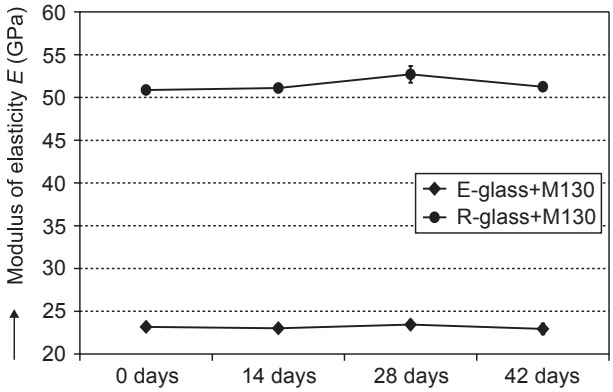


Figure 8. The influence of TCM storage on the Young´s modulus.

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VLIV KRÁTKODOBÉHO MÁČENÍ V SIMULOVANÉM TĚLNÍM PROSTŘEDÍ NA MECHANICKÉ VLASTNOSTI KOMPOZITŮ NA BÁZI SKELNÝCH VLÁKEN A POLYSILOXANU

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Byly studovány vláknové kompozitní materiály, které jsou určené pro aplikace v kostní chirurgii ve formě kostních dlah a náhrad. Kompozity byly připraveny z tkanin na bázi E- a R-skla a polysiloxanové matrice. Na kompozitech byl studován vliv loužení během krátkodobého máčení v simulovaném tělním roztoku a současně byly měřeny jejich mechanické vlastnosti. Bylo zjištěno, že studované materiály po máčení a jejich následném vysušení nevykazují téměř žádnou změnu mechanických vlastností.