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Comparison of selected properties of cements modified with glassy carbon and cancellous bone

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ABSTRACT

Purpose: The aim of this manuscript was to study and analyse the properties of bone cement (VertaPlex) before and after modification with glassy carbon (Alfa Aesar) and human bone (MaxGraft).

Design/methodology/approach: To achieve the assumed goal, a series of samples was made - five samples for each mixture, where: 5 bone cement samples, 5 bone cement samples mixed with 20-50 μ m glassy carbon in the ratio of 1 g carbon per 40 g of cement, and 5 samples of bone cement mixed with 20-50 μ m glassy carbon and human bone in the ratio of 1 g of carbon per 40 g of cement and 0.4 g of bone per 40 g of cement. The produced samples (4 for each mixture, 1 was the reference sample) were subjected to tests - compression test, microscopic observations with a 3D microscope, surface profile tests and hardness tests.

Findings: The study has shown that modifications with glassy carbon and bone change the mechanical properties, as well as the strength of the samples. Compression tests have shown that the material without admixtures is characterized by the highest compressive strength and the doping of the glassy carbon itself makes the material more brittle. A significant increase in hardness was also observed for samples with glassy carbon and bones after the pressing process.

Practical implications: The study was made synthetically, without taking into account the effect of the environment of body fluids and the human body temperature. This study is an introduction to further considerations where samples for which these conditions will be applied are currently being prepared.

Originality/value: For commercial use, in treatment of patients, cements modified with glassy carbon and bone glassy carbon have not been used so far. Due to the prerequisites of a positive effect of glassy carbon addition on osseointegration and biocompatibility, the study in this area has been undertaken.

Keywords: Bone cement, PMMA, Acrylic bone cement, Vertebroplasty, Kyphoplasty, Glassy carbon, Cancellous bone

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BIOMEDICAL AND DENTAL MATERIALS AND ENGINEERING

1. Introduction

The most common compression fractures of the spine are those located in the thoracic spine and the thoracolumbar section, whereas this fracture occurs in the sacrum section rather rarely. Compression fractures within the spine are characteristic and a relatively common type of spine fractures that lead to damage and collapse of the vertebral body [1-3]. As a consequence of a compression fracture, the spine is damaged, the vertebral body collapses, and thus its height is reduced by about 15-20%. Compressive fractures of the vertebral body may form as a result of mechanical injuries in which the spine is subjected to high forces, and consequently, the spinal body is compressed and damaged [3-6]. Fractures within the spine can be caused by various pathologies, including pathologies of the skeletal system, such as skeletal diseases that reduce bone tissue quality primary and secondary osteoporosis, metabolic diseases, as well as compression fracture of the spine can be a symptom of many other diseases, such as: generalized cancer (myeloma) or local (metastasis), hemangioma, hyperparathyroidism and other [2, 6-8]. Most often, the front part of the vertebral body is damaged, and the spongy substance in the body is crushed and fragmented. The main symptom of a compression fracture of the vertebral body is sudden and severe pain that occurs practically at the time of injury, it is caused by the richly innervated vertebral body. However, damage to the spinal body is dangerous, as it can fragment its posterior part, bone fragments can move to the lumen of the spinal canal, and compress and damage the nerve structures in its lumen. In this case, apart from severe and sudden pain in the place of the spine fracture, there may also be disturbances of the nervous system in the form of sensory disturbances or paresis of the lower limbs [1-8].

Compressive fractures of the spine result from a disease that reduces the quality of the bone tissue, i.e. osteoporosis; contrary to traumatic fractures, disorders of the nervous system or neurological symptoms of the spine or nerve roots are relatively rare. The most common complication of the disease that reduces bone tissue quality – osteoporosis – are compression fractures of the spine [1-8].

With the increasing ageing of the population, and the related increase in the incidence of osteoporosis, the demand for synthetic bone substitutes is increasing. In the United States alone, 700,000 new spine fractures have been reported annually [2-4, 9]. It has been reported that in 1000 people, 1.45 women and 0.73 men suffer from spine fractures per year due to osteoporosis [10]. With age, the incidence rate increases significantly, in women over 50 compression fractures of the spine have been found in 21% of women, while in women over 70, the fractures have been observed

in up to 80% of women. What is more, most osteoporotic compression fractures of the spine can occur during normal daily activities such as getting out of bed or sneezing. They can be formed during normal everyday activities, when getting out of bed or when sneezing. The consequences of vertebral compression fractures are usually chronic pains, deepened thoracic kyphosis, the so-called dowager's hump, and lowering of the vertebral body by an average of 2.1 cm, thus lowering the patient's height. Thoracic kyphosis and other deformities of human figure associated with compression fractures may lead to a deterioration of the cardiovascular function, as a consequence of a reduction in the respiratory function of the lungs and impaired intestinal passage [3,5,11].

Compression fractures of the spine are considered stable fractures because the ligamentous apparatus and the posterior column of the spine are not damaged. Until minimally invasive surgical techniques have been found, stable fractures were treated with conservative treatment methods, including rest, bed regimen, pharmacotherapy and orthopaedic corsets [11,12]. Unfortunately, in many cases, osteoporotic compression fractures lead to irreversible deformations of the figure and permanent pain, and conservative treatment is often not enough [13]. Nowadays, compression fractures of the spine are treated not only using conservative treatment methods but also minimally invasive surgical techniques, such as vertebroplasty and kyphoplasty [1-8]. The percutaneous vertebroplasty procedure is now commonly used to treat patients with vertebral fractures in the course of such skeletal pathology as osteoporosis, and and metastatic neoplastic some primary lesions. Percutaneous vertebroplasty is a group of treatments that are minimally invasive and give extremely quick clinical results. When anaesthesia wears off, patients feel pain relief, which is the result of increased mechanical stability of the fractured vertebral body as a result of hardening of the injected bone cement. With the minimally invasive procedure, the return to physical activity is very quick, which is especially beneficial for the elderly [14-18].

The treatment, called percutaneous vertebroplasty, was first performed in France by Deramont in 1984. The idea behind this surgical technique is to inject polymeric bone cement under pressure directly into the shaft of the fractured vertebra using a cannula. This surgical method, despite the quick relief of pain, does not restore the height of the fractured shaft. After being applied inside the broken shaft, the bone cement hardens during a polymerization process and then, after curing, it stabilizes the vertebral fracture, increasing its mechanical strength, immediately providing pain relief and thus improving the patient's comfort of life. An important aspect related to this surgical method is its performance under local anaesthesia, which is particularly important in the case of elderly patients with coronary diseases. The advantage of local anaesthesia is that constant contact with the patient can be kept, neurological complications can be detected quickly, and a quick response can be provided [19-22].

A further development of minimally invasive surgical methods is the implementation of another percutaneous technique, such as kyphoplasty, which was first performed in 1997. Kyphoplasty, unlike vertebroplasty, allows for partial restoration of the height of the fractured vertebra by inflating the balloon inside the lowered vertebral body to raise the falling areas of the vertebra to their normal position prior to cement injection. The percutaneous kyphoplasty procedure is used, in particular, when angular deformation of the spine has occurred and deep kyphosis as a result of a compression fracture has been formed. This surgical method involves inserting a high-pressure balloon directly into the fractured vertebral body, with the help of which it is possible to partially restore the height of the shaft. After removing the balloon, polymer bone cement is injected into the cavity, which is denser than in vertebroplasty and under lower pressure, as this reduces the possibility of cement leakage beyond the fractured vertebrae [11,12,23].

Minimally invasive surgical techniques, such as vertebroplasty and kyphoplasty, are now widely used to treat compression vertebral fractures using acrylic surgical cements based on a polymer matrix, consisting primarily of polymethyl methacrylate (PMMA). Surgical cements used to stabilize compression fractured vertebral bodies must meet specific biomechanical and biological requirements. Bone cement is a biomaterial that is embedded in an environment under load and must withstand complex stress patterns. Polymer cements based on PMMA have been known since 1958, and since then acrylic bone cements have been most frequently used as reconstructive material in the treatment of bone defects in orthopaedic treatment [24]. Quick treatment effects and good mechanical stability within minutes are the advantages of PMMA treatment. Thanks to these advantages, PMMA bone cements have been used in minimally invasive surgical methods such as vertebroplasty and kyphoplasty [5]. The advantages of acrylic bone cements are good handling properties, biointegrity, good biotolerance, desirable biomechanical properties and strength. On the other hand, this material has significant disadvantages, which include:

- high polymerization temperature,
- release of toxic monomers,
- cement shrinkage during polymerization,
- porosity of polymeric materials [22,25].

Despite the high effectiveness of minimally invasive vertebroplasty and kyphoplasty procedures, as every other procedure, it may also have adverse effects in the form of side effects and complications, which are mainly are related to the imperfection of the filling material [12]. The cemented vertebral body is much stiffer than untreated vertebrae, and due to mechanical stabilization, it does not undergo elastic deformation which, in turn, may lead to fractures of adjacent vertebrae, while high temperature during cement hardening in situ may lead to necrosis of surrounding tissues. Modification of bone cement to improve its performance as a filling material and stabilizing spinal compression fractures is key to improving patient health. Certain cement properties, such as the rate of polymerization and the time of cement hardening, can be influenced by lowering the ambient temperature or by cooling the components of the mixture earlier before mixing. Ideal improved bone cement should have not only good handling properties, injectability and adequate mechanical strength, but it should also be characterized by better biocompatibility, lower polymerization temperature that does not cause tissue necrosis, and a reduced modulus of elasticity after hardening, which would have a positive effect on the stress distribution, and thus it would prevent the formation of fractures of the adjacent vertebrae of the treated segments [12,26,27]. Cement modifications should also improve acrylic cement's ability to integrate with autogenous bone tissues. Currently, in various research centres, modifications of cements are made with various admixtures, such as oxide ceramics, glassy carbon [26-30], nanoparticles of metals Ag and Cu [31-35] or mineral collagen [36-42] to improve the functional properties of currently used cements. The development of regenerative and reconstructive surgery determines the need for new synthetic biomaterials. With the population's ageing, there is a need for reconstruction as part of bone surgery. Synthetic biomaterials that are based on composite structures make it possible to better imitate organic structures thanks to their continuous improvement. Therefore, there is a growing market demand for this type of materials, and the need for further development of these materials, and research on the behaviour of various materials and their modification over a long period of use [12,31,43]. Attempts to modify cement with chitosan/graphene oxide nanocomposite additive were also noted in modern literature. It has been observed that such modification of cement had a positive impact on handling properties of cement - injection, binding time, polymerisation temperature, mechanical properties, and bioactivity of cement modified in such way [44]. The next interesting modification of surgical cements on the PMMA framework that increases resistance to cracking is the PMMA-Mon-CNT composite

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[45]. The thesis [46] covers attempts to create a partially biodegradable cement composite deducting attempts made using cellulose, chitosan, polydioxanone, tricalcium phosphate. Various amounts of AgNp were added to all modifications. Test results have shown that all modifications had improved material porosity and slightly decreased both wettability and mechanical properties. Modification using polyxanone has also improved the lifetime of cells. Recent studies have also noted doping of bone cement using Multi-Walled Carbon Nanotubes (MWCNT). The studies have shown that thanks to this modification it is possible to control the osteointegration and cytocompatibility of cements [47].

The purpose of modification was to examine the influence of glassy coal and cancellous bone on selected mechanical properties, including hardness and durability to compression, as well as the impact of additives on the material's structure. The studies have indicated that the additives mentioned above have a positive influence on the development of surface profile, in particular when the cement is subjected to action of compression forces. This phenomenon is desired in case of such biomaterials as it allows to bind the material with bone in an easier way.

2. Materials and methods

In the first research stage, three types of samples were made: 1) only bone cement (VerterPlex Cement) (Tabs 1, 2) bone cement doped with glassy carbon (Alfa Aesar carbon splinter powder) with a fraction of 20-50 μ m, where 1 g of carbon was doped with 40 g of cement. Glassy carbon powder was premixed with the cement copolymer powder, and then the premix prepared this way was spread in a liquid monomer, 3) and with an admixture of glassy carbon for 40 g of cement and 0.4 g of bone for 40 g of cement. Ground cancellous bone and glassy carbon powder were premixed with the cement copolymer the premixed with the cement doped with a different structure of glassy carbon and human spongy bone (MaxGraft), 1g of carbon for 40 g of cement and 0.4 g of bone for 40 g of cement. Ground cancellous bone and glassy carbon powder were premixed with the cement copolymer powder, and then the premix prepared this way was spread in a liquid monomer.

To give the right shape, moulds (drawing) with dimensions of $10 \times 10 \times 30$ mm were made and filled with cement and mixtures of cement, coal and bone. For the cement and each mixture, two samples with the above dimensions were made. After the cement had set, the samples were pulled out and cut in half, which resulted in four samples with dimensions of $10 \times 10 \times 15$ mm (Fig. 1).

Table 1.

Chemical composition of PMMA (manufacturer's data)

Chemical composition of VertaPlex cement components					
20 g powder		9.5 ml ampoule			
Polymethyl methacrylate	14.0 g	Methyl methacrylate	9.4 ml		
Benzoyl peroxide	2.6%	N, N-dimethyl-para-toluidine	0.10 ml		
Barium sulphate	6.0 g	Hydroquinone	0.75 mg		

Table 2.

List of sample dimensions and forces during compression.

Sample	Mean Dimensions, mm, height-width-depth	Mean F, N	Mean R[40%], MPa
BC	13.97 x 10.19 x 10.14	9563.88	92.17
BC + GC	15.31 x 9.90 x 10.47	8749.94	79.987
BC + GC + HB	14.28 x 9.85 x 10.23	9051.03	84.36



Fig. 1. Macroscopic photos of tested samples - made of a) cement (BC), b) cement with glassy carbon admixture (BC+GC), c) cement with glassy carbon admixture and human bone (BC+GC+HB)

To compare the selected properties of the obtained materials, tests of mechanical properties and microscopic observations were performed. Each of the samples was compressed on the device Hegewald&Peschke Inspekt20, as a result of which the samples were deformed – the samples swelled in the axis perpendicular to the axis of the force. Additionally, one sample was made for each mixture, but they were not compressed as these samples served as reference samples during the comparative tests.

Then, microscopic photos and profiles of the observed surface were taken to analyse the effect of force, and thus the level of sample deformation. These observations were made on a Keyence VXH-900 3D microscope at x100 magnification.

At a further stage of the research, the surface roughness (on swollen surfaces) was analysed using the Hommel T-1000 contact profilometer. These tests were conducted to check how the roughness parameters change in the case of deformation of these materials. This is extremely important for medical applications as surface overdevelopment can irritate or injure the surrounding tissues.

The next research stage was to measure microhardness of the obtained samples. These tests were performed on a Future Tech FM-7 semi-automatic microhardness tester.

3. Results and discussion

First, compressive strength tests were performed for the produced samples. The distance travelled by the piston from the sample surface was 6 mm, while its feed was 3 mm/min. Due to the need to enter correct data into the testing machine, all the tested samples were measured with regard to the geometry of the samples, and the results are summarized in Table 2. Moreover, the results of the forces obtained during compression are compared.

Based on the collected results, it was found that the samples made of the cement alone without any admixtures had the highest compressive strength.

For doped cements, it was observed that the addition of bone to the mixture had a positive effect on the increase in strength. To illustrate the course of changes in the pressure force, the results are summarized in a graphical form (Fig. 2).

Microscopic observations showed changes on the surface of the samples that were subjected to compression. It was observed that samples subjected to compression have characteristic fringes which are related to material deformation and cracking. Such changes were observed for each mixture, as shown in Figures 3-5. The structure is clearly spreading as a result of the acting forces.



Fig. 2. Graphical summary of the force results during the compression test for all samples



Fig. 3. Microscopic photos of samples made of bone cement, a) reference sample, b) sample after compression



Fig. 4. Microscopic photos of samples made of bone cement mixed with glassy carbon, a) reference sample, b) sample after compression



Fig. 5. Microscopic photos of samples made of bone cement mixed with glassy carbon and human bone, a) reference sample, b) sample after compression



Fig. 6. Profiles of the surface of samples made of bone cement, a) reference sample, b) sample after compression

For a more precise analysis of the effect of compression on the surface of the tested samples, an analysis of the surface profile height was performed. The obtained images confirm microscopic observations, where for the samples after compression, the fringes of Figures 6-8 and the craters of Figure 7, which are the result of material cracking, can be seen.

The photos presented above clearly show that as a result of the action of forces, and thus the swelling of the material, the samples made of a mixture of cement, glassy carbon and human bone Figures 5 and 8 proved to be the most resistant. Whereas, the samples with the lowest compressive strength (which was observed as material chipping and a crater) are samples made of a mixture of cement and glassy carbon.

The next research stage was to measure the surface development, which were then compared with each other. To obtain the results, five measurements of the roughness profile were made for each of the samples. The results are presented in the table (Tab. 3), and (one for each sample) graphs of the actual course of the surface profile were presented (Fig. 9). For reference samples, the obtained results were performed on a measuring section equal to 4.8 mm, and for samples after compression – on a measuring section of 1.5 mm. The differences in the selected lengths were related to the fact that the reference samples had a regular shape and there was no problem testing their roughness on longer sections. For samples after

compression, where the material swelled, the measuring needle was above the surface profile on longer sections, which was related to its irregular shape. Therefore, this section was shortened. To fully reflect the level of surface development measurements were made on shorter sections so that one would be a continuation of the other.



Fig. 7. Profiles of the surface of samples made of bone cement mixed with glassy carbon, a) reference sample, b) sample after compression



Fig. 8. Profiles of the surface of samples made of bone cement mixed with glassy carbon and human bone, a) reference sample, b) sample after compression

List of roughness parameters obtained during profilometric tests for all samples obtained						
	R_a value, μm					
Measurement	BC before compression	BC after compression	BC+GC before compression	BC+GC after compression	BC+GC+HB before compression	BC+GC+HB after compression
1	1.73	2.33	1.6	5.17	1.98	5.21
2	1.79	2.67	1.93	5.26	1.83	5.32
3	1.29	2.73	1.72	5.03	1.77	4.93
4	1.65	2.49	1.85	4.98	1.81	5.17
5	1.44	2.21	1.91	5.08	1.67	5.29
Mean	1.58	2.49	1.80	5.10	1.81	5.18
Standard deviation	0.21	0.22	0.14	0.11	0.11	0.15

Where R_a – arithmetic mean deviation of the profile from the mean line

Table 3.



Fig. 9. List of examples of real roughness profiles for samples: a) BC before compression, b) BC after compression, c) BC+GC before compression, d) BC+GC after compression, e) BC+GC+HB before compression, f) BC+GC+HB after compression

	HV 0.1					
Measurement	BC	BC	BC+GC	BC+GC	BC+GC+HB	BC+GC+HB
	before	after	before	after	before	after
	compression	compression	compression	compression	compression	compression
1	18.0	21.3	16.6	22.4	15.0	22.5
2	17.4	22.1	16.5	22.4	14.9	22.4
3	17.1	21.2	16.0	22.5	14.8	22.6
4	17.7	21.3	16.2	22.2	15.8	22.5
5	17.6	21.5	16.2	22.3	15.7	22.8
Mean	17.56	21.48	16.30	22.36	15.24	22.56
Standard deviation	0.34	0.36	0.24	0.11	0.47	0.15

Summary of microhardness results for all samples

Based on the collected results, it was found that the samples with the admixture of glassy carbon and human bone were characterized by the highest surface development before and after compression. This state may be related to the appearance of carbon particles and bones on the surface, which combined with cement could form "granules", which could affect the overall surface development. Then, microhardness tests were performed. The tests were performed on a FutureTech FM-7 semi-automatic microhardness tester. Measurements were made by the Vickers method with a load of 980 mN, the load was applied to the sample for 11 seconds. Hardness tests were performed on the surface that was subjected to pressing. This measurement is justified by the fact that at the time of compression, cement transfers the loads axially in relation to the pressure, expanding to the sides. For each sample,

Table 4.

5 measurements were performed, then the arithmetic means and standard deviations were calculated to verify the correctness of the measurements. The results are presented in the table (Tab. 4).

Based on the compiled table, it was found that the doping of carbon and bone reduces the hardness before the compression test, compared to the samples made of cement alone. However, after compression, the hardness of samples with admixtures increases. This may be related to the denser packing and hardening of the material, which in turn increases its hardness. For samples with an admixture of carbon and bone, after the compression test, the hardness increased by as much as 50%.

4. Conclusions

As part of this manuscript, a comparison of selected properties of the produced samples based on bone cement was conducted. During the research, it was found that the doping of glassy carbon and glassy carbon with bone to cement significantly influences the development of the surface profile, especially when the cement is subject to compressive forces. For this type of materials, it is a desirable phenomenon because if the surface is developed to a greater extent, connection with the bone is easier. However, if the material is too rough, there may be pathological changes in the surrounding tissues due to injuries.

A significant increase in hardness was also observed for samples with glassy carbon and bones after the pressing process. The results indicate an almost 50% increase in this parameter. This property may be particularly useful when transferring very large, temporary loads, e.g. as a result of a patient falling.

Unfortunately, the doping of the glassy carbon itself makes the material more brittle, and as a result of compression there is cracking, as a result of which "craters" appear. This is an undesirable phenomenon for medical use.

Compression tests have shown that the material without admixtures is characterized by the highest compressive strength, but it is not much higher than the compressive strength of cement doped with glassy carbon and bone (a difference of 5.6%).

Probably all problems related to cracking, as well as a decrease in compressive strength, may result from cement slippage on glassy carbon particles, which weakens the finished sample. It should be remembered that these tests were performed synthetically without an attempt to simulate body fluids, and with loads increasing over the course of 6 minutes, which do not occur in real conditions. These

studies are preliminary studies for this type of material. Further publications on this type of modification will take into account the above-mentioned factors.

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