A method of producing ceramics-based composite

The invention relates to a method of producing composite based on ceramics, especially HAp, for implants.

Ceramic granules from hydroxyapatite HAp or from tricalcium phosphate TCP are used as materials for implants but due to their fragility and poor plasticity their application in surgery, especially oral and facial, is limited.

From patent description DE2238265 there is known a press having independently adjustable stages for manufacturing a multi-layered laminated product from powdered materials. The number of these stages depends on the number of powdered materials used. Each stage includes a piston whose stroke is guided by and limited by a cylindrical portion of an adjacent piston placed below the first one. The punch is hydraulically driven and its position is adjustable. The press is equipped with a feeder.

There are known methods of making the above mentioned ceramic materials more malleable by using water suspension of curdlan heated to 40°C.

From Polish patent PL 212866 there is known a method of producing bio-active composite based on calcium-phosphate bio-ceramics in the form of granules and which is mixed with curdlan in proportions according to a specific formula and then the mixture is heated in temperatures of 80-100°C.

The object of the invention a method of producing ceramics-based composite characterised by high mechanical endurance.

Unexpectedly, it turned out that reducing presence of air within composite structures to a minimum significantly improves their endurance.

According to the invention a method for producing ceramics-based calcium phosphate composite is characterised in that into a water suspension, which contains 76-175 grams of β-1.3-glucan (hereinafter referred to as curdlan) per 1 litre of water, calcium phosphate ceramics in the form of porous granules with a total weight of 300-800 g, favourably from 415 to 800 g, is added and mixed until an optimum homogeneity is achieved and then preliminary moulding takes place whereby a preform trough is thoroughly filled with the matter and then the matter is pushed into a mould where each portion is rammed separately until consistency containing less than 15% air is achieved and, after accurate ramming, it undergoes 5-120 minutes of heat treatment in temperatures between 80-100°C, and after that a finished product is removed from the mould.

Favourably, the granules have sizes from 0.1 to 0.9 mm and an open porosity from 50% to 70%. The most favourable porosity is between 60 and 70%.

Favourably, prior the heat treatment, the mould containing the rammed matter is sealed with a plug or, otherwise, the air exposure of the rammed matter is restricted by using other means.

Examples

Example I

170 g of β-1.3-glucan was mixed with 1.000 ml of water and then 350 g of calcium phosphate ceramics HAp granules with a diameter of 0,3-0,4 mm were added and the ingredients were mixed until homogeneous consistency has been achieved. From the resulting matter a sample having about 6.5 cm^3 in volume was taken and the preform trough with a capacity of 5550 mm^3 was thoroughly filled with it. The remaining excess of the matter was removed. In the next step portions of the matter having a volume of about 0,4 cm^3 were being fed successively from the trough into a mould having a diameter of 13 mm and each portion was rammed three times with a force of 15 N. The obtained semi-finished product having a volume of 4 cm^3 was heat-treated for 25 minutes at 98°C and after that cooled to room temperature and then removed from the mould. It has been concluded that the homogeneous composite thus obtained had low plasticity and during a compression test the sample sustained deformation of 18% prior its destruction.

The composite has been dried for 12 hours at 40°C. The overall volume of pores (air content) in the dried sample of the composite, as measured with a method of computed micro tomography (with threshold value for pores detection of 15 µm), amounted to 6.74% ± 1.15%. More than 90% of all pores in the sample were of the closed type.

Example II

For testing purpose 40 g of β -1.3-glucan mixed with 500 ml of water was used. 225 g of calcium-phosphate ceramics TCP granules with diameters of between 0,4-0,6 mm was added to the water suspension and then the components have been mixed until homogeneous matter was obtained.

From the obtained matter a sample having about 10 cm^3 in volume was taken and put into the preform trough with a capacity of 8.350 mm^3 which was thoroughly filled and the remaining excess of the matter was removed. In the next step portions of the matter having a volume of about 750 mm^3 each were being pushed out the trough into a mould with a diameter of 15 mm where each portion has been rammed three times with a force of 20 N. The obtained product having a volume of 7 cm^3 was heat-treated for 20 minutes at 93°C. The finished product, after having been cooled and after removing it from the mould, was examined under a microscope and no empty spaces resulting from the air remaining in the semi-finished product undergoing the moulding process have been discovered during this examination.

The overall volume of pores (air content) in the dried sample of the composite, as measured with a method of computed micro tomography (with threshold value for pores detection of 15 µm), amounted to 7.24% ± 1.11%. About 30% of those pores have diameters between 45-75 µm. No pores with a diameter more than 0.3 mm had been detected, which indicates highly homogeneous structure of the product and high efficiency of the moulding process performed by the machine hereby described.

Patent Claims

1. A method of producing composite based on calcium phosphate ceramics mixed with curdlan, characterised in that into a water suspension containing 76-175 grams of β-1,3-glucan (hereinafter referred to as curdlan) per one litre of water calcium phosphate ceramics in the form of porous granules weighing 300-800 g in total, favourably from 415 to 800 g, are added and mixed until an optimum homogeneity is achieved and then preliminary moulding takes place whereby a preform trough is thoroughly filled with the matter and after that the matter is pushed into a mould where each portion of the matter is rammed separately until consistency containing less than 15% air is achieved and, after accurate ramming, the matter together with the mould is being kept for 5-120 minutes in temperatures between 80-100°C, and after that the finished product is removed from the mould.
2. The method in accordance with claim 1, characterised in that the granules have a diameter of between 0.1 to 0.9 mm and an open porosity ratio of between 50% and 70%.
3. The method in accordance with claim 2, characterised in that, favourably, the porosity of the granules is between 60% and 70%.
4. The method in accordance with claim 1, characterised in that the mould containing the rammed matter, prior to heat treatment, is plugged or, alternatively, the air exposure of the rammed matter is restricted by using other means.

Abstract

The inventions solve the problem of finding a method for producing composite for implants.

The essence of the method consists in that a preliminary moulding is being performed by thoroughly filling a preform trough and then the matter is being pushed out of the preform trough in portions and then each portion is rammed separately until consistency containing less than 15% air is achieved.

[Fig. 1]