Synthesis of composites filled with modified hydroxyapatite and study of their mechanical properties

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Abstract – The purpose of this work was to increase the compatibility of composite components by modifying the surface of hydroxyapatite particles. Oleic acid and peroxide copolymer VEP-MA were used as modifiers. It was shown that modified composites have higher impact viscosity and tensile strengths.

Keywords – modification, hydroxyapatite, composite.

Dispersed hydroxyapatite (HAP) has a very high biocompatibility and capable of interacting with bone tissues, but has a low mechanical strength. Therefore, we cannot use it as a bone substitute. The good solution to this problem is use polymer composite materials based on HAP. However, this is complicated because of the significant difference in surface energy, and hence the poor compatibility of the components. One of the ways to improve the compatibility of composite components is modify the surface of the filler particles. To solve this problem, we used next modifier: oleic acid (OA) and peroxide copolymer VEP-MA to modify the surface of hydroxyapatite.

Synthesis of hydroxyapatite passed through the reaction:

\[10\text{CaCl}_2 + 6 (\text{NH}_4)_3\text{PO}_4 + 2\text{NH}_4\text{OH} = \text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2 + 20\text{NH}_4\text{Cl}\]

Modification of mineral filler and VEP-MA was carried out in a solution of organic solvent (ethyl acetate), within 2 hours. Upon completion of the modification, the filler was filtered and dried at room temperature to constant weight.

The polyacrylamide-based composite was prepared as follows: an aqueous solution of acrylamide was prepared, dispersed HAP (modified with oleic acid) added to the solution, polymerized acrylamide at 60 °C in the presence of HAP and initiator of potassium peroxide sulfate, the resulting composite was dried to constant mass.

The polyethylene composite was prepared as follows: a mechanical mixture of crushed polyethylene and disperse hydroxyapatite was made, the mixture was filled into a mold and sintered at 180 °C for 4 hours and then remained pressed to a constant temperature. To characterize the composites obtained, studies were carried out on thermomechanical properties, shock viscosity and tensile strength.

Fig.1. Thermomechanical curves of composites based on polyacrylamide filled with HAP (degree of filling = 40% by mass) and HAP (degree of filling = 40% by mass) modified with oleic acid (degree of modification = 2% by mass), T1 = 70°C, T2 = 85°C.
Fig. 2. Thermomechanical curves of composite polyethylene filled with HAP ((degree of filling = 40% by mass): a) HAP which modified oleic acid (degree of modification = 2% by mass) T = 97 °C; b) VEP-MA (degree of modification = 2% by mass) T₁ = 92°C, T₂ = 96°C

Fig. 3. Microphotographs of the fracture surface of a HAP-filled polyethylene composite (degree of filling = 20% by mass) which modified with oleic acid (degree of modification = 2% by mass).

Fig. 4. Microphotographs of the fracture surface of a HAP-filled polyethylene composite (degree of filling = 20% by mass) which modified VEP-MA (degree of modification = 2% by mass)

**Conclusion**

As a result of the conducted studies it was shown that the modification of hydroxyapatite with oleic acid (degree of modification 1% and 2% by mass) and peroxide modifier (degree of modification = 2% by mass) increases the mechanical properties of the composites obtained. Composites based on polyacrylamide filled with HAP (modified with oleic acid) have an increase of 30% impact strength and an increase in temperature of 20 °C. Polyethylene samples filled modified HAP (OA and VEP-MA) showed no significant changes in thermo-mechanical properties. At the same time, they demonstrated a significant increase (> 65%) of the tensile strength for composites which modified by 1% OA by mass, (>50%) for composites which modified by 2% VEP-MA by mass and (> 30%) for composites which modified by 2% OA by mass.