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## A NOVEL CLASS OF DISPERSANTS FOR COLLOIDAL PROCESSING OF HYDROXYAPATITE

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### NOWA KLASA DYSPERGATORÓW DLA PRZETWÓRSTWA HYDROKSYAPATYTOWYCH ROZTWORÓW KOLOIDOWYCH

#### Abstract

Poly(acrylic acid) (PAA) polymer modified with poly(ethylene glycol) (PEG) was used as a dispersant in preparation of hydroxyapatite aqueous suspension. The effect of dispersant structure on suspension stabilization was investigated through the studies of physicochemical properties of the obtained dispersions. Novel dispersants and HAp suspensions were characterized using pH and viscosity measurements.

*Keywords: dispersant, suspension, hydroxyapatite*

#### Streszczenie

Poli(kwas akrylowy) (PAA) modyfikowany poli(glikolem etylenowym) (PEG) zastosowano jako dyspergator wodnej suspensji hydroksyapatytowej. Określono wpływ struktury dyspergatora na stabilność suspensji na podstawie badań właściwości fizykochemicznych otrzymanych dyspersji. Nowoczesne dyspersanty i zawiesiny HAp scharakteryzowano z zastosowaniem pomiarów pH i lepkości.

*Słowa kluczowe: dyspergator, suspensja, hydroksyapatyt*

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## 1. Introduction

Polymers are widely used in colloidal processing of ceramic powders to improve the characteristics of suspensions, such as powder concentration homogeneity, rheology and state of dispersion [1–2]. These characteristics are necessary to obtain high density green parts with a homogeneous microstructure. Polyelectrolytes are widely used as dispersants in aqueous processing of calcium phosphate family. The polymers dissociate in the media and adsorb onto the particle surface shielding particles from each other. As a result, the particles are separated and stabilized in the suspension via the electrosteric mechanism. From a number of factors, efficiency of polyelectrolyte as a dispersant is mainly dictated by its molecular structure and a suspension pH. Both parameters strongly influence the dissociation degree of polyelectrolytes, polymer chain conformation and adsorption behaviour of polymeric chains on the particle surface. Although many research groups extensively investigated aqueous ceramic processing in the past decade, there were a few profound studies on the effects of pH and dispersant structure, which compared various dispersants in a systematic way [3–5].

## 2. Experimental

### 2.1. Materials

Acrylic acid (AA), ammonium persulphate (APS), potassium hydroxide (KOH) were obtained from POCh Gliwice, Poland. Poly(ethylene glycol) (PEG) and polyethylene glycol diacrylate (PEGDA)  $M_w=256$  were acquired from Sigma Aldrich. All chemicals were of analytical grade and were used without further purification. Hydroxyapatite (HAp) applied in our research was naturally occurring, obtained in a 3-step process of pork bones – acid hydrolysis and double calcination [6]. The particle diameter of HAp was in a range of 0.05 to 0.15  $\mu\text{m}$ .

### 2.2. Dispersions preparation

Polymer matrix (SAP) was applied as a dispersant, which was received on the base of poly(acrylic acid) (PAA) modified with 10% poly(ethylene glycol) (PEG) in the field of microwave irradiation [7]. The preparation of the dispersant solution was performed as follows: SAP of 0.3 wt.% was added and HAp of various amounts of 0.1–0.5 wt.% was added, based on 100ml dispersion weight.

### 2.3. Stability of dispersion measurement

Investigation of the stability was carried out for the dispersion on the basis of the pH measurement in 10 days. The measurements were made for suspension with different HAp concentration (1, 2, 3, 4, and 5 wt. %), which contained a constant SAP amount.

## 2.4. Rheology measurement

The viscosity of HAp dispersions was measured at room temperature using the Anton Paar DV-2 P viscometer with an R2 spindle. The results of viscosity versus dispersant concentration, i.e. the deflocculation curves, suggested the optimum concentrations of each dispersant at the point where viscosity reached the minimum.

## 3. Results and discussion

When the dispersants are dissolved in water, the COOH groups dissociate, providing the  $\text{-COO}^-$  groups along the backbone and protons ( $\text{H}^+$ ) in the dispersion. Therefore, the pH of dispersion reflects the concentration of  $\text{-COO}^-$  group and HAp. For the samples containing from 2% to 5% HAp an increase of pH value in the dispersion was observed. An interpretation of this phenomenon could be connected with a low content of free calcium oxide in HAp releasing to the environment during the immersion time. The samples containing only 1% HAp had the lowest concentration of free calcium oxide and their pH was at a similar level.

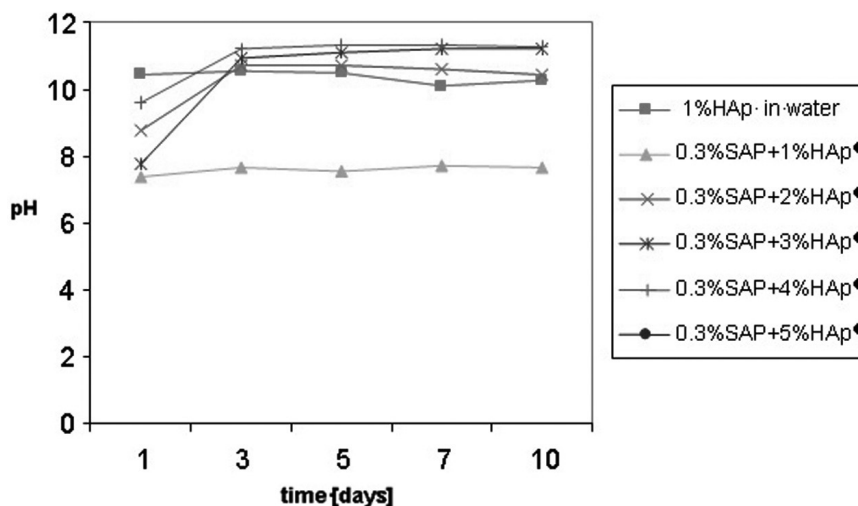


Fig. 1. Changes in dispersions pH value

Rys. 1. Zmiany pH dyspersji

Dispersion viscosity at various HAp concentrations is shown in Fig.2. The viscosity was highest in the sample containing 1% HAp because of the  $\text{-COO}^-$  groups from the polymer matrix present in a dispersion. The viscosity of dispersion decreased with an increasing pH, then it reached the lowest values and became steady for the samples with 4% and 5% HAp. In samples containing the largest amount of HAp, which have an alkaline character, the dispersion pH was decreased because of SAP acidity.

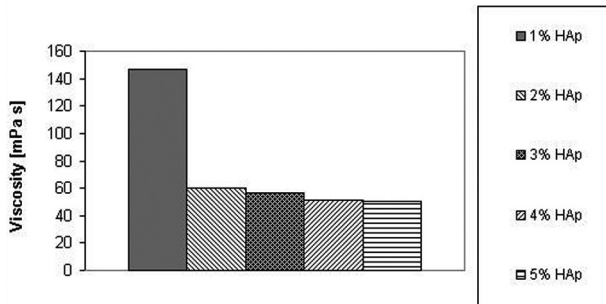


Fig. 2. Viscosity of dispersions

Rys. 2. Lepkość dyspersji

#### 4. Conclusions

HAp dispersions were prepared using SAP as dispersants. It was found that SAP enhanced the dispersion and stability of dispersion. The measurements of dispersion viscosity as a function of HAp concentration indicated that the viscosity decreased with an increasing pH and HAp content. The pH of dispersion depended on the concentration of free calcium oxide in the suspension.

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