

PARAMETERS OF THE ELECTROPHORETIC DEPOSITION PROCESS AND ITS INFLUENCE ON THE MORPHOLOGY OF HYDROXYAPATITE COATINGS. REVIEW

PARAMETRY PROCESU OSADZANIA ELEKTROFORETYCZNEGO I ICH WPŁYW NA STRUKTURĘ POWŁOK HYDROKSYAPATYTOWYCH. PRZEGLĄD

ALEKSANDRA LASKA ORCID 0000-0002-4872-3959
MICHAŁ BARTMAŃSKI ORCID 0000-0003-0185-6460 RESEARCHERID: M-9589-2018 SCOPUS AUTHOR ID: 57194511871

Gdansk University of Technology,
Department of Materials Engineering and Bonding, Gdansk, Poland
*aleksandra.laska@pg.edu.pl

Metallic materials intended for bone implants should exhibit not only appropriate mechanical properties, but also high biocompatibility. The surface treatment modifications, for example acidic treatment, laser treatment, ion implantation and deposition of highly biocompatible coatings, are practiced. One of the most popular methods of surface modification is to deposit hydroxyapatite (HAp) coatings. HAp naturally occurs in human body, but can be also synthesized in laboratory conditions. Among diverse deposition techniques, electrophoretic deposition (EDP) is a cost-effective method in which charged particles, dispersed in an organic medium, after applying voltage migrate to the counter charged electrode forming a thin coating. There are several parameters that can be controlled during the process and that directly affect the morphology of the surface. The zeta potential and pH of prepared colloidal suspension are closely related to suspension stability and affect the susceptibility for agglomeration of the particles. Electrical settings, especially applied voltage, affect primarily the mass of deposition, but also the porosity of the coating, as well as its homogeneity. One of the basic parameters of EDP method is time of process. With increasing process time, the thickness of the deposited coating increases. Importantly, its mechanical properties also decrease. Moreover, the particles shape and size also affect the morphology of the deposited coating. The analysis of many variables is necessary to choose the right parameters to obtain the coating with desired morphology. In this paper, the influence of each parameter on the morphology of hydroxyapatite coatings is discussed.

Keywords: hydroxyapatite, electrophoretic deposition, biomaterials, osteosynthesis

W artykule omówiono parametry procesu osadzania elektroforetycznego (EDP) ze szczególnym uwzględnieniem wykorzystania tej metody do osadzania powłok hydroksyapatytowych (HAp) na metalicznych implantach kostnych. Bazując na obszernym przeglądzie najnowszej literatury, przedstawiono wpływ każdego z parametrów na strukturę powstającej powłoki.

Słowa kluczowe: hydroksyapatyt, osadzanie elektroforetyczne, biomateriały, osteosynteza

1. INTRODUCTION

The mechanical, chemical and biological properties of the layer of the implant determine the success of the implantation surgery and proper behavior of the implant in the human body. Generally, metallic materials intended for bone implants have relatively low biocompatibility in comparison to the materials naturally occurring in a human body and the bonds between metal implants and bone are usually weak [1]. It resulted in a variety of surface modification techniques, such as nanooxidation [2], acidic treatment [3, 4], micro- and nanopatterning [5], laser treatment [6, 7], ion implantation [8–10] and deposition of highly biocompatible coatings [11–13]. One of the most popular solution is to deposit a hydroxyapatite (HAp) coatings [14–16].

HAp naturally occurs in human body and it is a major component of bone and tooth enamel. The inorganic phase of the bone is composed by calcium phosphates (CaP), mostly in the form of HAp corresponding to 65–70 wt. % [17].

A chemical formula of hydroxyapatite is $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$. HAp has a theoretical composition of 39.6 wt. % Ca and 18.5 wt. % P. Ca/P molar ratio is equal to 1.667. The most common structure belongs to the hexagonal lattice with space group P63/m and with cell dimensions of $a = b = 0.9418 \text{ nm}$ and $c = 0.6884 \text{ nm}$ [18]. Synthetic HAp has been commonly used as a coating material for metallic implants due to its biocompatibility and ability to form strong bonds with bones. Its mechanical properties such as low strength and high brittleness restrict its applications mostly for coatings deposited on metallic substrate [19].

The hydroxyapatite coatings can be obtained by applying diverse deposition techniques, such as sol-gel technique [20–22], suspension plasma spraying [23, 24], magnetron sputtering method [25, 26], pulsed laser deposition [27, 28], ion beam assisted deposition [29], dip coating [30], electrophoretic deposition [31], electrocathodic deposition [32], hydrothermal deposition followed by electrochemical seeding [33], chemical and thermal treatment [34, 35]. Among them,

the most investigated is electrophoretic deposition technique (EPD) in which charged particles, dispersed in an organic medium, after applying voltage migrates to the counter charged electrode forming a thin coating. Electrophoretic deposition relies on the coagulation of the particles to a dense mass. The coatings produced this way are relatively highly homogeneous, and the process itself does not require specialized equipment and large financial outlays [36]. To employ electrophoretic deposition successfully, a basic understanding of the colloidal stability and deposition kinetics is necessary. In this paper, the influence of EPD technique parameters on morphology of hydroxyapatite coatings is discussed.

2. TYPE OF LIQUID MEDIUM AND COLLOIDAL SUSPENSION PROPERTIES

One of the basic parameters of the electrophoretic deposition process is the type of used electrolyte and its properties such as zeta potential and pH value, which are closely related. The effectiveness of the method relies primarily on the properties of colloidal suspension. Zeta potential is related to the interaction of the charged particle with the liquid in which it is located [37]. It provides information on the charge of the particles in a liquid environment [38]. It is closely related to suspension stability, which is very important property of the suspension used during EPD process. According to the assumptions, in the liquid layer one can distinguish two parts. The first one is the region closest to the surface, called the Stern layer, in which liquid ions, charged opposite to the sign of the particle, closely adhere to. This region is considered as immobile and it may include absorbed ions. In the outer region these interactions are much weaker. This part is called the diffuse layer. Diffusion of ions is possible due to the influence of electrical forces and random thermal motion. This assumption introduces the concept of an

electric double layer that occurs around each ion. If the potential of the solution is defined as equal to zero, an electro kinetic potential, which is zeta potential, is the potential at the slipping plane [39]. A schematic representation of this phenomenon is shown in Figure 1.

The suspension that contains the particles to be deposited should provide the zeta potential that ensures a required dispersion of the particles in the solution. Low level of dispersion leads to the formation of agglomerates, which results in poor adhesion of coating and irregular morphology [40]. Zeta potential values typically range from -100 mV to +100 mV. The suspension has a high degree of stability when its zeta potential is lower than -30 mV or greater than +30 mV [41]. The zeta potential curve might be presented as a function of pH [42].

Dudek [43] deposited hydroxyapatite coatings by EPD method on NiTi alloy substrate. A colloidal suspension with 0.1% hydroxyapatite concentration in ethanol was prepared. Zeta potential, the mobility of particles and the size of particles in the function of pH value were measured. According to the conducted tests, the suspension of hydroxyapatite in ethanol reached the maximum value of zeta potential (44.0 ± 2.2) mV for pH = 5. An increase in pH value in the alkaline direction resulted in decrease in zeta potential, and the suspension was not stable. Furthermore, for a pH value of 5, the hydroxyapatite particle size was the smallest and equal to 230.9 nm. This means that the suspension was well dispersed. The particles agglomerated with the increase of pH, which is a highly unfavourable phenomenon. The highest electrophoretic mobility of particles was equal to $0.97 \mu\text{m}\cdot\text{cm}/\text{V}\cdot\text{s}$. The above tests clearly indicate the fact that for a suspension of hydroxyapatite in ethanol, the best properties are displayed with pH = 5.

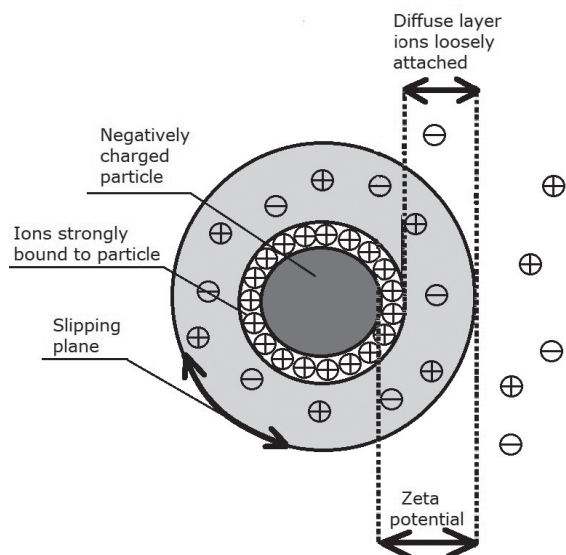
Stoch et al. [44] investigated the influence of medium type on the electrophoretic efficiency (Δm) as a function of time. For this purpose titanium plates covered with silica precoatings were used. Two types of medium were used – ethanol and isopropanol. Ethanol performed three times higher efficiency than isopropanol.

Javidi at al. [45] produced hydroxyapatite coatings on 316L stainless steel substrate using deionized water and isopropanol as a medium for suspensions. The zeta potential of the suspensions was equal to -16.5 mV and 22.6 mV, respectively. Isopropanol based suspension performed higher stability.

3. ELECTRICAL SETTINGS

The electrophoresis deposition occurs only when an electric field is applied to the electrolyte suspension. Deposition takes place in an electrochemical cell in which one electrode is the substrate to be coated [46]. The force with which the field affects the electric charge of the ion depends in direct proportion to the electric field strength, which is proportional to the voltage applied to the electrodes. The relationship between the applied voltage U , the electric field strength I and the resulting electrical resistance R of the electrolyte is described by Ohm's law.

Fig. 1. Zeta potential of a particle with negative surface charge
Rys. 1. Potencjał zeta cząstki z ujemnie naładowaną powierzchnią



The result of the current passing through the electrolyte is the work done by moving the charge in the electric field and the loss of power on the circuit resistances. When an electric field is applied across a conducting medium there is an inevitable heat, called Joule heating [47]. During the process, the electrolyte resistance changes – it usually decreases as the electrolyte temperature increases, which is caused by Joule's heat. However, the electrolyte resistance also increases as due to decrease in the number of ions and changes in the distribution of electric charge under conditions of discontinuous electrophoresis. Such a system requires stabilization of temperature [48]. The analysis of many variables is necessary to choose the right electrolyte composition and stabilization of electrical parameters.

Abdeltawab et al. [49] deposited hydroxyapatite coatings on a titanium substrate by EPD method. A suspension of 1 g of HAp dispersed in 100 ml of dimethylformamide was prepared. After magnetically stirring for 15 min, suspensions were dispersed ultrasonically in an ultrasonic bath for 30 min at 50 kHz. The electrophoretic deposition experiments were performed at various voltages ranging from 20 to 140 V. The weight per unit area of the deposit and the deposition thickness were investigated. Generally, the thickness of coating increases with increasing applied voltage and the weight of coating per unit area can be approximated as a linear function of applied voltage. Neither cracks nor agglomerates of powder grains were observed on the coatings created by applying 20 V and 30 V voltages. The tendency to agglomerate particles is closely related to their size [50]. The structure of these coatings was uniform. Numerous cracks and agglomerates were observed on coatings produced at higher voltages. The quality of the deposit can suffer when greater electric field is applied, although the coating can be deposited more quickly. More uniform films can be deposited at moderate applied fields, whereas the film quality deteriorates if relatively high electric fields are applied. The formation of the coating on the electrode is a kinetic phenomenon, so the accumulation rate of the particles influences their packing behaviour in coating. With higher electric field, turbulences in the suspension may be caused and the coating may be disturbed by flows in the surrounding medium. In addition, at high voltage values, the movement of particles can be so fast that they will not be able to create a coating of the expected consistency, and their attachment process will be chaotic.

Another approach is a high-voltage electrophoretic deposition. Mondragon-Cortez et al. [36] used an electric field in a range from 200 to 800 V at a time of 0.5 to 3 s. During the voltage application of 200, 400 and 800 V, the current density was 55, 101 and 210 $\mu\text{A}/\text{cm}^2$, respectively. The deposit weight per unit area increased with an increase of applied voltage. Scanning electron microscopy observations showed that the increase of applied voltage resulted in the increase of the particles size deposited. These observations proved that the smallest particles reached the highest electrophoretic velocity.

In the research conducted by Javidi et al. [45] the hydroxyapatite coatings on stainless steel substrates were deposited at the voltage of 30, 60 and 90 V. The coatings weight and their thickness were investigated and their value increased with the increase of the voltage. The changes in roughness of the deposited coatings were also assessed. The roughness of coatings deposited at the voltage of 30 and 60 V was in the range of 0.5 μm and the roughness of the coating deposited at 90 V was four times higher. In general, the higher voltage is applied during EPD process, the more hydrogen evolution on the cathode can be observed, which causes in higher porosity of the coating [51]. Moreover, applying higher voltage might yield the deposition of the coarse particles [52]. Thereby a porous structure with high surface roughness will be formed [53].

4. DEPOSITION TIME

The basic parameter of the electrophoretic deposition process is a deposition time. Generally, deposited coating thickness increases with increasing process time, but, importantly, its mechanical properties also decrease simultaneously. The time of the electrophoretic deposition process, in addition to electrical parameters, is the basic variable that affects the morphology and mechanical properties of the produced structures. Abdeltawab et al. [49] investigated the relationship between the thickness of the deposited coating and the time of the process. To the suspension of 1 g HAp in 100 ml dimethylformamide the voltage of 60 V was applied. The deposition times ranged from 1 to 7 minutes. It can be clearly stated that the thickness and the mass of the deposited coating increase only at the initial time of the deposition and the deposition rate is a linear function of the deposition time. But during the longer period of time, the deposition rate decreases and attains a plateau at very high deposition time. In a constant voltage EPD, this is expected while the potential difference between the electrodes is constant, the electric field decreases with the deposition time because of the formation of an insulating layer of HAp on the electrode surface. This finding also agrees with Zhitomirsky's research [54]. During the initial period of EPD, there is approximately a linear relationship between deposition mass and the time of the process. However, this information is not enough to choose the optimal duration of the process. It is necessary to carry out microscopic observations that allow determining the morphology and homogeneity of the resulting structure. These results show that with a longer process time, a less homogeneous coating containing more agglomerates and small cracks is obtained. In the research of Drevet et al. [55] the effect of electrophoretic hydroxyapatite deposition time on the morphology of the obtained coatings was examined. For this purpose, a suspension of 2 g nanohydroxyapatite in 25 ml ethanol was prepared. The powder grain size did not exceed 200 nm. The suspension was sonicated at 50 kHz for 40 min. The substrate was a disk of Ti-6Al-4V alloy with roughness of 2 μm , defatted in acetone and washed in distilled water. The electrophoretic deposition

process was carried out at a voltage of 10 V and the distance between the electrodes 20 mm. Processing times were set at 5 min, 10 min, 15 min and 20 min. When the deposition was carried out at the time of 5 min, the substrate remains almost bare. At the time of 10 min, the most homogenous surface was obtained. After 15 and 20 min, there cracks were present. The observations might be attributed to an excessive agglomeration of the hydroxyapatite particles on the substrate surface that progressively increases electrical resistance of the surface [56]. Javidi at al. [45] deposited a hydroxyapatite coating at the voltage of 90 V at a time in a range from 1 to 5 min. The deposition weight and the thickness of the coating as a function of time were investigated. The results showed that at the constant applied voltage, the deposition weight increased with time but the slope of the curve decreased with increasing deposition time. It can be seen that for the prepared sample, after 5 min of the deposition, there is a tendency to reach the yield and saturation. Stoch at al. [44] deposited hydroxyapatite coatings on pure titanium substrates. For this purpose, two suspensions were prepared – with ethanol and isopropanol as a medium. In both cases the deposition weight as a function of time of the process was investigated. At the duration of time from 0 to 60 s, the relationship between the deposition weight and the time was observed as a linear function. Mondragon-Cortez et al. [36] used high-voltage electrophoretic deposition to receive hydroxyapatite coatings on 316L stainless steel substrate. Voltages of 200, 400 and 800 V were applied to the suspension of hydroxyapatite in ethanol. The process time ranged from 0.5 to 3 s and was measured every 0.5 s. All of the samples prepared at different voltage showed exponentially increase of the deposition mass in a function of time. The non-linear behavior of the forming coating at short times in the EPD at higher voltages can be caused by the progressive increment of bigger particles on the deposits. The particle size formed the received coating when the time of deposition was equal to 0.5 s, and it was in the range from 0.35 to 0.57 μm . After 2 s, the particle size interval was 1.10–1.35 μm . The longest process, which lasted 3 s, resulted in the coating consisting particles of the size from 1.20–1.70 μm . It means that the smallest particles reached the highest electrophoretic velocity and they were the first ones in being deposited.

5. PARTICLES SIZE AND SHAPE

The size of the particles to be deposited by the electrophoretic process is crucial, particularly since the particles must be fine enough to remain in the suspension during the process. The size and shape of the powder grains used to carry out the electrophoresis process directly affect the mobility of particles in the electrolyte, but also the thickness of the deposited coating and the zeta potential of the suspension.

Farnoush at al. [50] used submicrometer (mHAp) and nano-sized hydroxyapatite (nHAp) to deposit coatings on Ti-6Al-4V substrates. The size of mHAp particles was approximately equal to 0.3 μm and nHAp – 65 nm. Two suspensions were prepared by dispersing 1 g of HAp powders into 50 ml of

ethanol. Three different values of the voltage were applied as 20, 40 and 60 V, and the deposition time was equal to 60, 180 and 300 s. The electrophoretic deposition was conducted with each of the time and voltage value for both suspensions. The weight of the coating deposited with the settled process time, voltage and the size of the particles was measured. As it was predicted, the weight of deposition increases with the increase of the deposition time and the increase of the applied voltage. Moreover, the mass of the coating is higher for the process conducted in the suspension with submicrometer particles of HAp than with nanoparticles. The distribution of particle size in mHAp and nHAp suspensions was investigated. Submicrometer particles of hydroxyapatite show a greater tendency to form agglomerates, which is a highly unfavorable phenomenon. It should be noted that there is a slight difference in tendency to agglomerate nano-sized particles. By analyzing images from a scanning electron microscope, it was possible to assess the surface morphology of both samples. It was found that the coatings deposited from submicropowder show higher porosity and structure heterogeneity. Coating prepared with nanopowder shows higher density and homogeneity. The roughness of both surfaces was tested. The average roughness height for the nHAp sample is 92.4 nm, while for the mHAp sample 903 nm. Reducing the size of the powder used in the deposition process reduces the roughness and the thickness of the coating.

Kwok at al. [57] investigated the influence of the shape of the particles on the morphology of the deposited coatings. Spherical, flake-shaped and needle-shaped particles were used. The diameter of the spherical particle was about 175 nm, the size of the flake-shaped particle was estimated at about 425 nm, and the dimensions of the needle-shaped particle were 50 \times 200 nm. The suspensions of 2.5 wt. % of each powder in ethanol were prepared. The EPD process was conducted at a constant cell voltage of 200 V for 30 min. The coating deposited from flake-shaped HAp particles was less uniform with some interconnected micro-pores with size of 0.4 μm due to the larger particle size of the powder. In contrast, spherical particles and needle-shaped particles were deposited creating coatings, which had only a few pores due to the smaller size of those particles. The hardness of all coatings was measured and it was equal to 737, 649 and 683 HV, for spherical, flake and needle-shaped particles, respectively. The coating obtained from the spherical particles showed the highest adhesion to the substrate with a value of 10.7 MPa. For the coating deposited from flake-shaped powder this value was equal to 6.8 MPa, and from needle-shaped – 8.5 MPa.

The above studies show that the use of nanometric powders allows obtaining the coating with the highest homogeneity, minimizing the risk of cracks and unwanted pores. The coating obtained by using nanopowders has a lower roughness. The most preferred powder particle shape is spherical. The coating deposited from such a powder shows the highest density and homogeneity, as well as the highest hardness and adhesion to the substrate.

6. SUMMARY

Electrophoretic deposition is a low-cost and effective method to deposit hydroxyapatite coatings, but it also requires many parameters to be controlled. One of the basic parameters of the electrophoretic deposition process is the type of used electrolyte and its properties such as zeta potential and pH value, which are closely related. The suspension has a high degree of stability when its zeta potential is lower than -30 mV or higher than +30 mV. The zeta potential curve is presented as a function of pH. Literature review proved that ethanol is better medium for suspension than isopropanol and water. For a suspension of 0.1% hydroxyapatite in ethanol, the highest stability and mobility of particles are displayed with pH = 5. The electrophoresis deposition occurs only when an electric field is applied to the electrolyte suspension. The coatings weight and their thickness increase with the increase of the voltage. The higher voltage is applied during EPD process, the more hydrogen evolution on the cathode can be observed, which causes higher porosity of the coating. The basic parameter of the electrophoretic deposition process is a coating time. Generally, deposited coating thickness increases with increasing process time, but, importantly, its mechanical properties also decrease simultaneously. The choice of the time of deposition depends also on the applied voltage. The size of particles to be deposited by the electrophoretic process is crucial, particularly since the particles must be fine enough to remain in the suspension during the process. Coatings deposited from hydroxyapatite nanopowder perform higher homogeneity comparing to coatings deposited from submicrometer powder. Moreover, reduced size of the powder results in reduced roughness and thickness of the coatings. The spherical shape of hydroxyapatite particles used during the process affects in higher homogeneity, density and hardness of the deposited coating as well as higher adhesion to the substrate, comparing to needle-shaped and flake-shaped powders. Most of the current researches focus on the investigations of the influence of one of these parameters on the morphology of hydroxyapatite coatings. Future research should be performed to investigate the relationship between each parameter and selecting appropriate sets of all parameters for electrophoretic deposition of hydroxyapatite coatings.

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