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EFFECT OF VOLTAGE AMOUNT USED IN ELECTROPHORETIC DEPOSITION ON THE THICKNESS OF 70S30C BIOGLASS COATING ON TITANIUM SUBSTRATES (IN-VITRO STUDY).

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ABSTRACT

INTRODUCTION Although Titanium has high mechanical properties, the lack of its biological activity led to the need for its surface modification. Titanium surface coating is one of the additive surface modifications. Adding new materials to titanium surface can bring new properties to it yet maintaining its original properties. In this study, 70S30C bioglass was used to coat the Titanium substrate by using the electrophoretic deposition technique (EPD).

OBJECTIVE Optimizing EPD parameter (voltage amount) for 70S30C bioglass nanoparticles coating on Titanium substrate. The aim of the current study is to evaluate the effect of the voltage amount on the produced coating thickness.

METHODS Twenty-four Titanium discs were prepared (10x1.5 mm), sandblasted, and ultrasonically cleaned in ethanol. Bioglass 70S30C was prepared by sol-gel technique followed by ball milling to produce nanoparticle powder. Characterization of the powder was done by transmission and scanning electron microscope. Titanium discs were divided into three groups (G1, G2, and G3) (n=8) each. They were subjected to EPD coating with 10, 30, and 50 Voltage, respectively. The coating thickness was then assessed by SEM images of the cross-section of the used specimens.

RESULTS TEM images revealed a highly agglomerated nano-powder with a size range of 8-20 nm. The coating thickness was increased by increasing the voltage amount under the same deposition time.

CONCLUSION EPD proved to be a versatile and innovative coating technique with a low cost. The resultant coat thickness is directly proportional to the voltage amount.

KEYWORDS Bioglass, dental implants, electrophoretic deposition, coating thickness, nanoparticles.

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INTRODUCTION

Titanium dental implants are becoming the gold standard treatment in replacing missing tooth/teeth. However, Titanium when exposed to any oxygen containing atmosphere, it forms an oxide layer (TiO_2) of 5nm that provides resistance to corrosion and protection of the underlying metal. However, this oxide layer hinders bone attachment to Titanium surface; therefore, osseointegration becomes relatively a long process.(1) To enhance cell–implant surface interaction, many surface modification methods have been investigated in the past to optimize surface topography and accelerate osseointegration with the host tissue. They have been classified into two main categories; subtractive and additive surface modification.(2)

The modification of Titanium surface itself without adding a new material is called subtractive surface treatment. It allows for changing Titanium surface properties such as roughness, hydrophilicity, and morphology. The main advantage of this method is that

there is no risk of detachment, since there is no material added to the surface. However, its effects on surface properties are more limited when compared to coating method (additive surface modification). Subtractive surface treatments can be done through physical methods (sandblasting, anodization or exposure to laser radiation) or chemical methods (acid etching or plasma etching).(2,3)
Coating method (additive surface modification), on the other hand, allows for deposition of a thin film that covers the original surface with a new one. The main advantage of this method is the wider range of surface properties due to presence of a different material that has different physical and chemical properties from those of the substrate. Coating deposition can be done by many techniques such as: plasma spraying, laser cladding, enamelling, sol-gel and electrophoretic deposition. (2)

Electrophoretic deposition (EPD) is a process in which colloidal particles, which are suspended in a liquid medium, migrate and deposit onto a counter charged electrode under the influence of an electric field. The reported advantages of EPD include simplicity, low cost, ability to produce coatings with variable thicknesses, and uniformly coat irregularly shaped or porous objects.(4,5)

Although several materials have been used in surface coating, bioactive glasses (BAGs) are of an increased interest. They can degrade rapidly and form a bone-like apatite layer that forms a chemical bond with the host bone.(6) Bioactivity of BAGs depends on its morphology as well as its specific surface area. The higher the specific surface area, the greater the dissolution rate, the greater the glass bioactivity will be. Obtaining a larger surface area includes an increase in the porosity and/or a decrease in the size of the material. Reducing the size into nanoparticles is of great interest not only for their large surface area, but also a higher surface energy, material shaping versatility, as well as it allows the material to be internalized by different types of cells.(7)

In the present study, we assessed the effect of the amount of voltage used in EPD coating of BAG nanoparticles on Titanium substrates on the thickness of the resultant coating. The null hypothesis is there will be no difference in the coating thickness with the different voltage amounts.

MATERIAL AND METHODS

The protocol of this study was reviewed and approved by the staff members of the Prosthodontic Department and Research Ethics Committee at the Faculty of Dentistry, Alexandria University (IRB NO: 00010556 – IORG 0008839). The study was an in-vitro (laboratory-based) research and was conducted in Alexandria University and Egypt Japan University of Science and Technology (E-JUST), Alexandria, Egypt.

Preparation of Titanium samples

A Titanium grade-5 Ti6Al4V blank *(Kera Ti 5-disc, Eisenbacher Dentalwaren, Weorth, Germany*) was cut using computer numerical control (CNC) wire cut machine *(5-axis CNC wire cut EDM, Ecowin Corp., Taiwan)* to prepare Titanium Ti6Al4V discs (n=24) with a diameter of 10mm and a thickness of 1.5mm (ISO 23317-2014).(8) The prepared discs were then subjected to airborne particle abrasion (*Korostar, Bego, Germany)* to remove the oxide layer. They were divided into 3 groups; G1, G2, and G3 each group had 8 specimens.

Preparation of bioactive glass nanoparticles

Tailored Amorphous Multi-Porous (TAMP) Bioactive glass 70S30 scaffold was prepared using modified sol-gel technique according to a technique mentioned elsewhere.(9) It was started by dissolving Polyethylene Oxide (PEO*) (Sigma-Aldrich, Egypt)* in 0.01 N Acetic Acid (CH3COOH) *(Sigma-Aldrich, Egypt)* under magnetic stirring *(Hotplate and stirrer 1000, Jenway Ltd, United Kingdom*). Then, Tetramethyl Orthosilicate (TMOS) *(Sigma-Aldrich, Egypt)* and Urea *(Loba Chemie, Mumbai, India)* were added to the mix with vigorous stirring. Afterwards, Calcium Nitrate Tetrahydrate ($Ca(NO_3)_2.4H_2O$) *(Loba Chemie, Mumbai, India)* was added. After complete dissolvement of the salt, few drops of hydrofluoric acid (HF) *(Loba Chemie, Mumbai, India)* with deionized water *(Barnstead smart 2 pure 3 thermo scientific, Hungary)* were added to the sol, followed by, pouring the solution into multi well flask *(M8812 Greiner CELLSTAR® multi-well culture plates (24 well), Sigma-Aldrich, Egypt).*

The solution was kept in 40ºC with no stirring for gelation to take place. When complete gelation occurred, aging was done at 40ºc for 24 hours, then for another day with the flask uncovered at ambient temperature. Before drying, the gel was immersed in deionized water and ammonia (NH4OH) *(El-Gomhouria Co., Egypt)* for 24 hours at 40ºC. Finally, drying, and thermal stabilization was done at 700ºC *(Muffle furnace L 5/11, Nabertherm gmbh, Germany)*. Subsequently, stored in isolated containers.

For nanoparticles preparation, the prepared BAG scaffolds were milled by using ball mill *(Planetary Ball milling machine, (Retsch – PM 400), Japan)* with ratio of BG to

balls 1:9 g and a speed of 350 rpm for 24 hours.

Coating phase

The coating was done using electrophoretic deposition (EPD) at ambient temperature. An electric cell was constructed, with two Titanium discs acting as the cell electrodes.(10) The three groups (G1, G2, and G3) were subjected to electric currents of 10, 30, and 50 voltages, respectively by using a Direct Current (DC) power supply *(Maisheng power supply, Schenzen Bost, Guangdong, China)*. The deposition time was constant in all the three groups (1 minute). Before starting electrophoretic deposition, all Titanium discs were ultrasonically cleaned *(Heating ultrasonic bath, Skymen, China)* in ethanol (99% purity) *(Sigma-Aldrich, Egypt)* for 10 minutes and left to dry under fume hood *(Purifier hepa filtered enclosure-3FT, Labconco Co., USA).*

All coated discs were then dried at room temperature for 24 hours in a desiccator *(El-Gomhoureia, Egypt*) followed by sintering in a vacuum oven *(GSL-1500X-50LVS, MTI Corp., Richmond, USA)* at 700ºc for two hours with a heating rate of 2ºC/min and allowed to cool with the same rate.(11)

Characterization of the bioglass nanoparticles

The size of the nanoparticles was measured by using transmission electron microscope (TEM) *(JEOL JEM-1400 series, USA).* After ultrasonic agitation for 15 minutes.

Assessment of the coating thickness

The coated discs were embedded in methyl methacrylate resin (*Sigma-Aldrich, USA*). After complete curing, they were sectioned by using a hard cutting micromotor (*300 CP EXAKT, Germany).* The produced cross sections were subjected to gold sputtering for 15 minutes prior to SEM analysis. The coating thickness was measured by using an image-analysis software provided by the manufacturer.(12) For each sample, 3 measurements were taken, and the average was calculated. The results were tabulated as mean ± standard deviation.

Statistical analysis

Statistical analysis was performed by using Statistical Package for Social Sciences (SPSS) (IBM SPSS Statistics version 25; IBM, Co). All data sets were assessed for normality by using Kolmogorov-Smirnov test. One-way Analysis Of Variance (ANOVA) test was used to compare between the 3 groups, followed by Tukey post hoc test.

RESULTS

The TEM image revealed high agglomerated and irregular shaped particles with size range of 8-20nm (Figure 1). Kolmogorov-Smirnov test showed that all data sets were normally distributed. The difference between the 3 groups regarding thickness is plotted in Figure 2 that showed that the coating thickness is increased with increasing the voltage amount. Table 1 shows the results of one-way ANOVA test between the 3 groups which notes to a significant difference among the study groups(P>.00001). Tukey's post hoc test revealed that the difference is between the 3 groups. However, the mean values of G1 and G2 (37.716 and 39.912, respectively) are close to some extent unlike G3 (54.524). Figure 3 shows the SEM images that revealed the uniform thicknesses in all groups with no gaps in the interface. The thickness difference between the three groups were documented as well. The porous structure of BAG layer was obvious while Titanium substrate showed a dense microstructure.

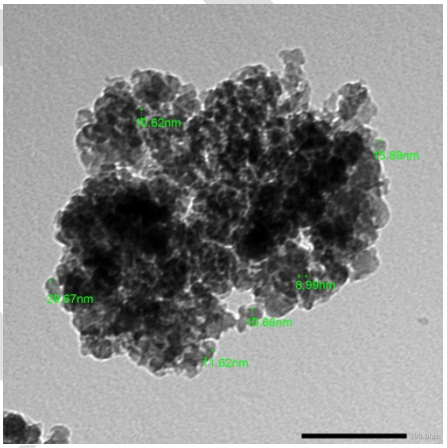

Figure 1: TEM image of the prepared BAG nano-powder.

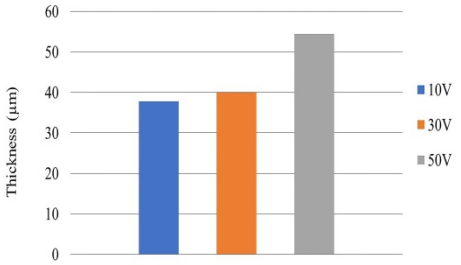

Figure 2: The mean thickness of coatings with different voltage amount.

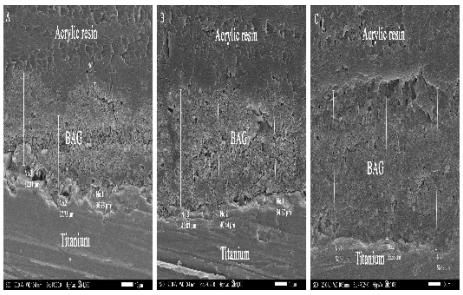

Figure 3: SEM images of cross sections of coatings done by applying 10V (A), 30V (B), and 50V (C).

Statistical parameter	10V	30V	50V
Mean±SD	37.716±0.592	39.912±0.351	54.524±0.9423
Median	37.88	40.03	54.23
Range	36.676-38.316	39.266-40.32	53.416-55.953
P-value	<.00001		
f-ratio value	1471.172		

Table 1: Statistical data of the studied groups.

DISCUSSION

The main advantage of EPD coating technique is the production of a homogenous coating thickness even in complex geometrical shapes such as dental implants. However, different parameters can affect the resultant coating characteristics such as deposition time, electric potential,(13) the distance between the electrodes,(14) the conductibility of the substrates, the sintering temperature, as well as the suspension medium.(11) In the present study we studied the effect of electric potential (expressed in voltage) on the coating thickness with fixing all the other parameters so that the results can be related only to the voltage amount. The results did not support the null hypothesis as the voltage amount had a significant effect on the resultant coating thickness.

In our results, G1 and G2 were close in readings, yet statistically significant. While G3 had a much higher reading. Stojanovic et al.(10) explained such findings as, at low voltages, the electric field is not high enough to enable mobility of the glass particles, only small ones can move leading to a thin coating layer. However, when the voltage is increased, the electric field becomes stronger which can move the larger glass particles resulting in a relatively thick coating layer.

To the authors' knowledge, this study reports the first attempt to coat sol-gel 70S30C BAG on Titanium substrates by using electrophoretic deposition. Mehdipour et al.(15) studied the influence of electric potential on the microstructure of BAG (SiO_2–P_2O_5–CaO–MgO) coating on stainless steel substrates. They observed that the higher the voltage, the coarse the coating particles are. Which was also explained by the relation of the voltage to electric field strength.

The current study does not state that the thicker coatings have the best behavior. However, an optimal coating thickness that concur with the bonding strength and bioactivity of the coating should be produced. The main purpose of bioactive coatings is their bioactivity which gives a picture of how the material will react in the human body. Borrajo et al.(16) concluded that for stimulating a complete in-vitro bioactive process, a critical glass coating thickness of 30µm is needed.

For a strong adhesion between the glass coating and the underlying substrate, thinner coatings are more preferrable. The development of structural defects and microcracks in thicker coatings weakens the interfacial bond.(17) In the same context, Baino(12) concluded that a silica based glass coating thickness below 250 µm was considered convenient for the biomedical purposes. However, Monsalve et al.(18) found that decreasing the BAG coating thickness below 80 µm enhanced the adhesive strength of the coating to the substrate. The wide range between the two studies may be due to the different substrate material and/or different BAG formula.

CONCLUSIONS

The current study proved that EPD is a convenient technique for coating of Titanium substrates with controlled coating characteristics. The electric potential used in EPD has a primary role in the produced coating thickness. A direct proportional relation is found between the electric potential and the coating thickness.

RECOMMENDATIONS

Authors recommend further characterization of different thicknesses regarding biological and mechanical properties to decide the optimal thickness for sol-gel BAG coating on Titanium substrates.

CONFLICT OF INTEREST:

The authors declare that they have no conflicts of interest.

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