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A study of the electrophoretic deposition of Bioglass® suspensions using the Taguchi experimental design approach

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Abstract

This paper presents a study of the Taguchi design method to optimise the rate of electrophoretic deposition (EPD) of Bioglass® particles from aqueous suspensions. The effect of Bioglass® concentration, pH and electric field was investigated. An orthogonal array of L_{16} type with mixed levels of the control factors was utilized. Multivariate analysis of variance (MANOVA) and regression analysis based on the partial least-square method were used to identify the significant factors affecting the deposition rate and its stability during constant-voltage EPD. It was found that the pH of the suspension significantly influences the deposition rate whereas the applied electric field has the smallest effect on the deposition rate. In addition, the interaction between the Bioglass® concentration and pH, which are key processing parameters influencing the deposition rate, was found to be significant. Although a high deposition rate was obtained at low electric field (5 V/cm) and pH 7, the instability of the suspension in particular at high Bioglass® concentrations resulted in an increase in the variability of the deposition rate. The optimal EPD condition predicted was verified by experiments and good qualitative agreement was obtained. The experimental results and statistical analyses are discussed based on the current knowledge of the EPD of ceramic materials.

Keywords: Electrophoretic deposition; Bioglass; Taguchi design; Coating

1. Introduction

Increasing needs in orthopaedics and bone reconstruction have led to production of different biomedical grade materials to be used in the treatment of bone diseases.[1–3] Although metallic implants, particularly Ti alloys, are being widely utilized, the poor adhesion of living tissue to these almost bioinert materials may cause detachment of the two surfaces which has negative consequences.[4,5] In order to improve the surface adhesion and biocompatibility of metallic implants, surface modification of prostheses used in bone replacements is being extensively investigated.[6–10] It is well established that chemical modification of implant surfaces by bioactive coatings enhances the cell-implant attachment.[11–17] Particularly, bioactive ceramics and glasses promote bioactivity by formation of an active phase of hydroxycarbonated apatite (HCA) on their surfaces, which is similar to the mineral phase of natural bone.[15,18] So far, various techniques including plasma spraying, sol–gel, enamelling, slurry dipping, electrophoretic deposition, and sputter coating have been utilized for the surface coating of metallic implants.[19–23] Among the various methods, electrophoretic deposition (EPD) is particularly attractive because it can be utilized to produce uniform coatings with controlled microstructure and properties on complex-shaped parts and porous structures at ambient temperature without the need for expensive processing equipment.[24,25] Detailed reviews on the fundamentals and applications of EPD, including the use of EPD for coating metallic implants have been published.[24–26]

Despite the simplicity of the process, many parameters influence the deposition rate and quality of the deposited layer in EPD. The characteristics and concentration of solid particles in the suspension, the electrophoretic mobility, the suspension conductivity, pH, electric field, and deposition time control the EPD

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rate.[24] Therefore, in order to obtain a controlled deposition rate leading to desirable coating microstructure, de convolution and optimization of the processing parameters is necessary. In most current EPD activities, a trial-and-error approach is followed to determine the "best" process parameters.[26] Krause et al.[23] showed that a uniform layer of Bioglass® coating on 304 stainless steel and Nitinol substrates can be achieved for a defined combination of time, concentration and voltage. Stojanovic et al.[27] has recently shown that by controlling the EPD voltage and deposition time, functionally graded bioactive glass–apatite composite coatings can be fabricated on Ti alloy substrates. The effect of processing parameters on EPD of calcium phosphate[28] apatite–mullite[29] and bioactive glass[30,31] suspensions to fabricate biomedical coatings has also been reported. In this context, the EPD of biopolymer and biopolymer/bioceramic composite coatings is also being investigated.[31] Recently, we have shown that the deposition rate of chitosan from aqueous solutions is significantly influenced by the pH of the suspension.[32] Studies of EPD of hydroxyapatite/chitosan[33,34] and Bioglass® reinforced alginate, chitosan and PEEK polymers[31,35] have also revealed the importance of an accurate control of the processing parameters. Evidently, EPD of composite suspensions is more difficult than that of single-phase suspensions because of differences in the electrophoretic mobility of the two or more phases. Therefore, fine tuning of the processing parameters is required to fabricate bioactive composite coatings with controlled microstructure.

Since many factors affect the EPD process, studying the influence of all parameters on the deposition rate and product quality is time-consuming, costly and inefficient; many trials have to be carried out as the relevant parameters are inter-related. Hence efforts must be placed on developing effective, analytical methods to optimise EPD process. In order to ease the optimization process by limiting the number of experiments, employing statistical methods such as the Taguchi design of experiment (DOE) approach[36] could be very useful. The procedure is a factorial DOE method which avoids the great number of experiments required for a full factorial design study and can be used for modelling and analyzing the influence of control factors on performance output.[37] The concept of signal/noise ratio is a measure of the robustness of the design and helps the investigators to identify the optimal combination of factors to achieve the targeted mean value of the property under study, with the least variability. Recently Corni et al.[38] have used a neural network (NN) approach for the EPD of PEEK–alumina composite coatings in which a design of experiment analysis was utilised to optimise the NN mathematical model. In the present work, the Taguchi DOE method was employed for the first time to study the electrophoretic deposition of Bioglass® particles from aqueous suspensions. The aim is to elucidate the effect of the key EPD parameters on the deposition rate in order to determine which control factors are more important in their effect on deposition rate and to what extent these are effective in controlling the kinetics of EPD. It should be mentioned that aqueous suspensions were utilized not only due to environmental considerations but also because the final goal of the research is to optimise the EPD process for polysaccharides/Bioglass® composite coatings which are produced from aqueous suspensions only.[31] This paper reports experimental results on EPD of Bioglass® suspensions as a function of Bioglass® concentration, suspension pH, and EPD voltage and the interaction between these parameters is shown. The optimum combination of the control factors for achieving a high deposition rate with the least variability is presented based on the multivariate analysis of variance (MANOVA) and least-square regression methods. The predictions of the method are discussed and compared with the experimental results.

2. Experimental procedure

Bioglass® 45S5 powder with nominal composition: $45SiO_2$–$24.5Na_2O$–$24.5CaO$–$6P_2O_5$ (wt%) was kindly supplied by Dr. I. Thompson (Kings Colloge London, UK). The particle size was between 0.1 and 73 μm with a typical particle size of about 6 μm (median). For EPD, suspensions with composition of 2, 5, 8, and 10 g/l Bioglass® particles in distilled water were prepared. The suspensions were sonicated for 600 s using a VWR USC 300 sonicator (VWR International, Malaysia) and the pH was measured by a JENWAY 3510 pH meter (Essex, UK). The procedure used for the EPD was similar to that employed for chitosan polysaccharide and is described in detail in Ref.[32]; the pH of the Bioglass® suspensions was adjusted in the range 3.2-9 by acetic acid addition (≥99.7%, Sigma–Aldrich). The fresh suspensions were then transferred quickly to the EPD cell in order to minimize any possible effects of dissolution of Bioglass® in the aqueous solution. Eletcrophoretic deposition was performed at two voltage levels of 10 and 20 V for a short period of time (<600 s) at room temperature ($24 \pm 2\,^{\circ}C$). The electric voltage was applied by a Thurlby Thandar Instruments (TTi) EL561 power supply (Cambridgeshire, UK) and the current through the suspension during EPD was recorded by using a TTi 1906 Computing Multimeter. Electrophoretic deposition at constant voltage was performed for five different deposition times in order to determine the mean deposition rate and the standard deviation. Due to the low dissolution of the Bioglass® particles in water, particularly in the acidic solution, the pH of the suspension was found to change by 0.3 pH unit after the test. 316L stainless steel plates with a thickness of 0.2 mm were utilized as the electrodes. The distance between the electrodes in the EPD cell and the deposition area were 2 cm and ∼10 mm × 10 mm, respectively. The cathodes were dried overnight and the deposition weight was measured by an accurate balance with 0.1 mg resolution (Adventurer Pro AS214, Switzerland). The change in the conductivity of the suspensions versus deposition time was determined according to the procedure in Ref.[39] Scanning electron microscopy (LEO Gemini 1525 SEM) was used to study the microstructure of the Bioglass® deposits. The zeta potential of the particles in the aqueous suspension was determined as a function of solution pH with an Agilent 7020 ZetaProbe (Foster City, USA).

3. Taguchi design of experiments

In the present work, the effect of processing parameters on the deposition rate of Bioglass® suspensions was studied

Table 1
Control factors and levels of variables used in this study.

Symbol	Control factor	Levels			
		1	2	3	4
A	Bioglass® concentration (g/l)	2	5	8	10
B	pH	3.2	4.1	7	9
C	Voltage (V)	20	10	–	–

using the Taguchi DOE method. In DOE, the most important stage is the selection of control factors. Based on the Hamaker model,[40] the deposition weight per unit substrate area is proportional to the concentration of the suspension, deposition time, electric field, and electrophoretic mobility of the particles. Therefore, the deposition rate, *i.e.* weight of deposition per unit area of substrate per unit time, should directly be related to the Bioglass® concentration and applied voltage (for a constant distance between electrodes). On the other hand, the electrophoretic mobility of particles is influenced by the zeta potential, which is pH-dependent.[24] Therefore, the Taguchi's orthogonal array was used by choosing the three control factors (concentration of Bioglass®, pH and applied voltage) that could influence the deposition rate. Table 1 shows the parameters and levels used in this study. Two four-level parameters (Bioglass® concentration and pH) and one two-level parameter (voltage) were positioned as mixed L_{16} ($4^2 2^9$) orthogonal array design (Table 2). A total of sixteen runs were conducted, using the combination of levels for each control factor (A–C). As compared to conventional full factorial experiment design, the Taguchi method can eliminate 80 EPD runs (considering that each test was repeated five times to determine the deposition rate with high accuracy), offering great advantages in terms of time and cost. The analyses were carried out using MINITAB 15 statistical software.

Table 2
The basic Taguchi L_{16} orthogonal array.

Run	Control factors and levels		
	A	B	C
1	1	1	1
2	1	2	1
3	1	3	2
4	1	4	2
5	2	1	1
6	2	2	1
7	2	3	2
8	2	4	2
9	3	1	2
10	3	2	2
11	3	3	1
12	3	4	1
13	4	1	2
14	4	2	2
15	4	3	1
16	4	4	1

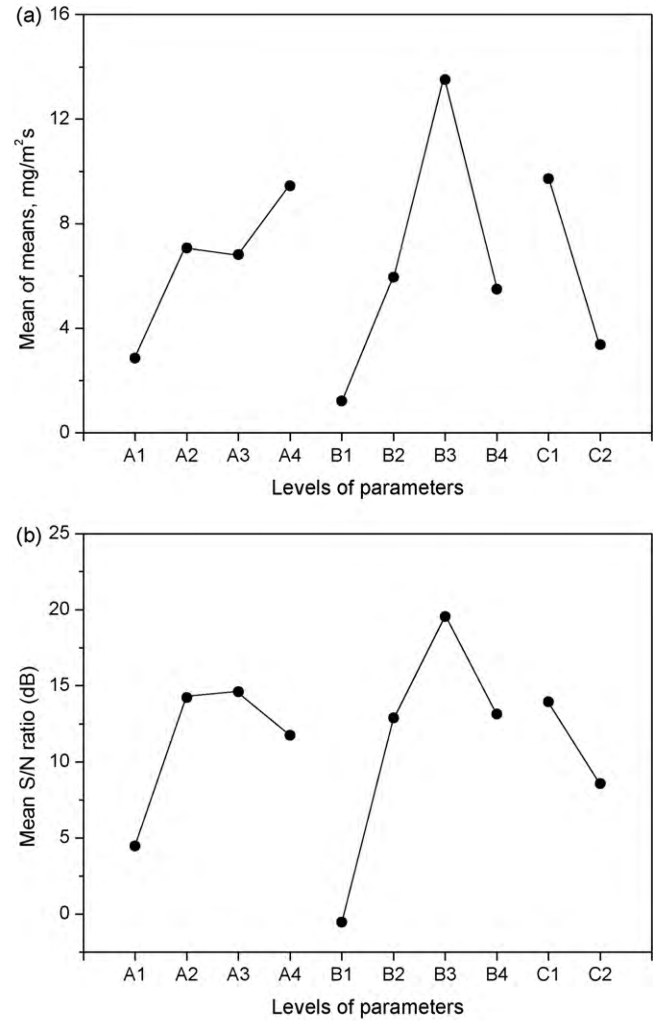

Fig. 1. Effect of control factors on Bioglass® deposition rate: (a) mean graph and (b) S/N ratio graph.

4. Results

The results of experiments to determine the average deposition rate of Bioglass® particles under the various conditions along with the standard deviation are reported in Table 3. The signal-to-noise (S/N) ratio was added to the table considering that a high deposition rate with a low standard deviation is desirable. S/N ratio is estimated as follows:

Higher deposition rate is the better characteristic:

$$\frac{S}{N} = -10\ \log\left[\frac{1}{n}\left(\sum\frac{1}{{y_i}^2}\right)\right] \quad (1)$$

Smaller standard deviation is the better characteristic:

$$\frac{S}{N} = -10\log\left[\frac{1}{n}\left(\sum {y_i}^2\right)\right] \quad (2)$$

where y_i is the deposition rate and n the number of observations. The unit of S/N ratio is decibel (dB), as frequently used in communication engineering. Since the experimental design is orthogonal, it is possible to separate the effect of each con-

Table 3
Experimental measured values for deposition rate and standard deviation of Bioglass® particles and S/N ratio (Taguchi L_{16} orthogonal array).

Run	Control factor			Deposition rate		Standard deviation	
	A	B	C	Value (mg/m^2 s)	S/N ratio (dB)	Value (±mg/m^2 s)	S/N ratio (dB)
1	2	3.2	20	0.25	−12.04	0.15	16.48
2	2	4.1	20	6.85	16.71	0.02	33.98
3	2	7	10	2.48	7.89	0.97	0.26
4	2	9	10	1.84	5.30	0.43	7.33
5	5	3.2	20	1.15	1.20	0.20	13.98
6	5	4.1	20	12.45	21.90	1.56	−3.86
7	5	7	10	9.58	19.63	7.36	−17.34
8	5	9	10	5.08	14.12	1.42	−3.05
9	8	3.2	10	2.25	7.04	0.58	4.73
10	8	4.1	10	3.07	9.74	0.19	14.42
11	8	7	20	11.04	20.86	2.97	−9.46
12	8	9	20	10.89	20.74	2.35	−7.42
13	10	3.2	10	1.21	1.66	0.19	14.42
14	10	4.1	10	1.44	3.17	0.71	2.98
15	10	7	20	30.94	29.81	1.30	−2.28
16	10	9	20	4.18	12.42	1.10	−0.83

Table 4
S/N response (dB) table for the Bioglass® deposition rate and standard deviation.

Parameter	Symbol	Level 1	Level 2	Level 3	Level 4	Maximum–minimum
Deposition rate	A	4.46	14.22	14.60	11.74	10.13
	B	−0.54	12.88	19.55	13.14	20.08
	C	13.95	8.57	–	–	5.38
Standard deviation	A	14.51	−2.57	0.57	3.57	17.08
	B	12.40	11.88	−7.20	−0.99	19.60
	C	5.07	2.97	–	–	2.10

trol factors at different levels by averaging S/N ratios at each level. The results for the deposition rate and standard deviation are summarized in Table 4. The maximum–minimum value for the pH has the highest value; hence, pH is the most significant parameter affecting the deposition rate whereas the applied electric field has the least effect. The effect of control factors on the deposition rate is shown in Fig. 1. The analysis of the results gives the combination of factors producing maximum deposition rate with minimum variance around the desired value. It is evident that the deposition rate increases with increasing the Bioglass® concentration; hence, the optimum deposition condition at a given Bioglass® concentration should be obtained at B3 (pH 7) and C2 (electric field of 5 V/cm). Nevertheless, the analysis of results based on standard deviation (Fig. 2) reveals a low S/N ratio at pH 7, indicating that achieving a uniform and constant deposition rate over time is more difficult at pH 7 compared to the lower pH values. Fig. 3 shows the variation of zeta potential with pH for the examined suspensions.[41] The isoelectric point of Bioglass® particles is around pH 11.5 (Fig. 3). Therefore as the pH increases, the stability of the sus-

Table 5
MANOVA analysis for the effect of control factors on the deposition rate.

Source	DF[a]	Seq SS[b]	Adj SS[c]	Ad MS[d]	F[e]	P[f]
Bioglass® concentration (g/l)	3	89.41	89.41	29.80	0.76	0.548
pH	3	313.47	313.47	104.49	2.66	0.120
Voltage (V)	1	161.29	161.29	161.29	4.10	0.077
Error	8	314.38	314.38	39.30		
Total	15	878.55				

[a] Degree of freedom.
[b] Sequential sums of squares (measures the reduction in the residual sums of squares provided by each additional control factor in the model).
[c] Adjusted sums of squares (measures the reduction in the residual sums of squares provided by each control factor relative to a model containing all the other control factors).
[d] Adjusted mean sums of squares.
[e] F-test statistic.
[f] P value (probability value).

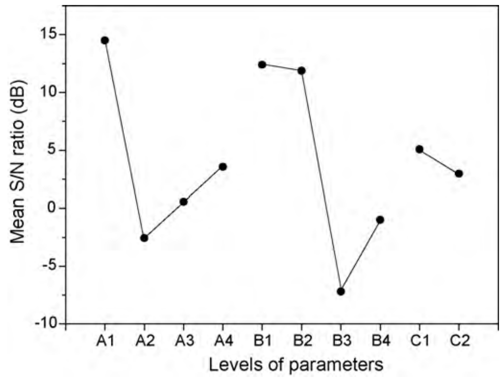

Fig. 2. S/N graph for standard deviation.

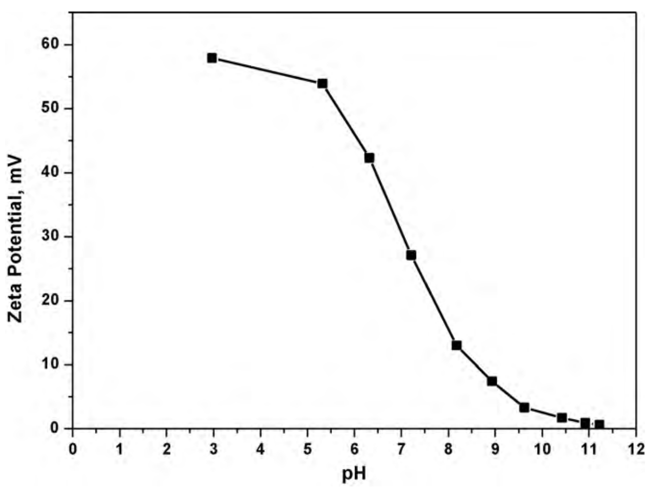

Fig. 3. Zeta potential of Bioglass® particles in aqueous suspension as a function of pH.[41]

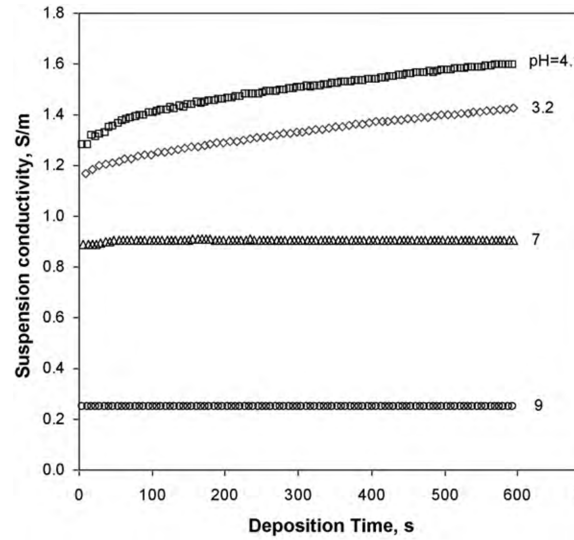

Fig. 5. Conductivity of 5 g/l Bioglass® suspensions as a function of deposition time for different pH values.

pension decreases, making it more difficult to achieve a stable deposition rate with increasing time.

In order to find out statistical significance of different factors on the deposition rate, multivariate analysis of variance (MANOVA) was performed, which is a procedure for testing the equality of vectors of means from multiple responses. Table 5 shows the results of MANOVA. The P values reported in the last column of the table indicate that applied voltage is highly significant ($P < 0.1$) whereas the other parameters (particularly the Bioglass® concentration and pH) seem to be not significant which might be due to their inter-relation; a matter which induces uncertain errors in the analysis. Fig. 4 shows the interaction graphs for the control factors. The results determine that the Bioglass® concentration–pH interaction is highly significant. Since the control factors are highly correlated, the relationship

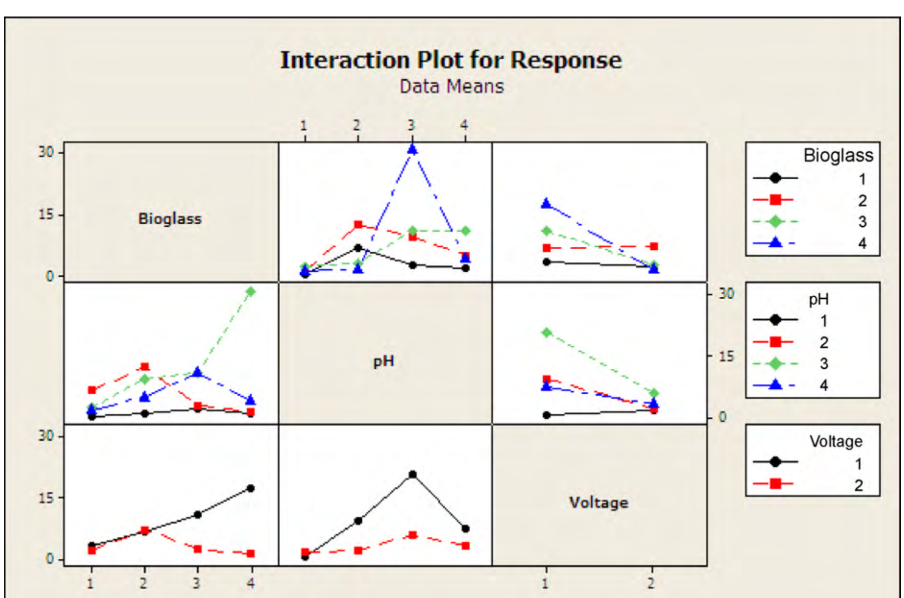

Fig. 4. Interaction graphs between the control factors for the Bioglass® deposition rate.

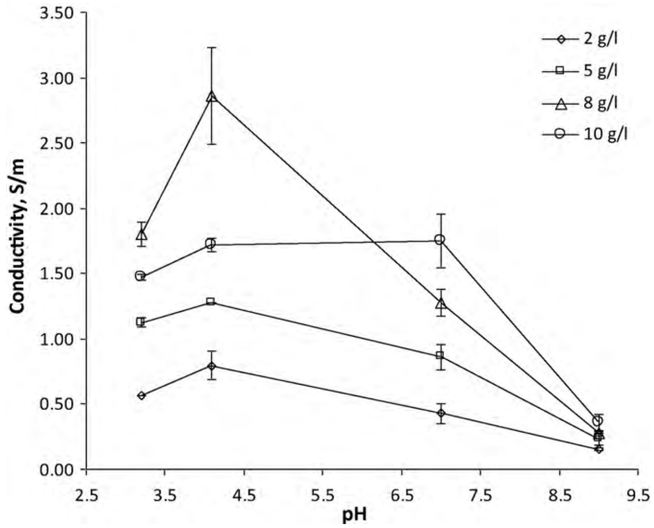

Fig. 6. Effect of pH and particles concentration on the conductivity of the Bioglass® suspensions.

between the response (deposition rate) and the control factors could be evaluated by partial least-square method. The analysis of variance (ANOVA) for the response is reported in Table 6. The low *P* value (<0.05) confirms that the factors are highly significant and inter-related. A complete study including the interaction effects among factors is an interesting avenue but is beyond the scope of the present work.

The final step in the design of experiment approach is to conduct confirmation experiments for the optimal parameters determined. As explained above, the optimum design was determined to be A4 B3 C2. The predicted S/N ratio using the optimal level of the design parameters can be calculated as[37]:

$$\left[\frac{\mathrm{S}}{\mathrm{N}}\right]_{\mathrm{Predicted}} = \left[\frac{\mathrm{S}}{\mathrm{N}}\right]_m + \sum_{i=1}^{n}\left(\left[\frac{\mathrm{S}}{\mathrm{N}}\right]_i - \left[\frac{\mathrm{S}}{\mathrm{N}}\right]_m\right) \tag{3}$$

where $[\mathrm{S/N}]_m$ is the total mean S/N ratio, $[\mathrm{S/N}]_i$ is the mean S/N ratio at the optimal level, and n is the number of the main design parameters. Table 7 shows the comparison of the predicted deposition rate and standard deviation with the experimental results using the optimal control factors. A convincing agreement between the predicted and experimental rate was obtained.

5. Discussion

Experimental results showed that pH of the suspension has a profound effect on the deposition rate. In aqueous media, the pH of the suspension is the most important factor affecting particles zeta potential. Specific results for the present Bioglass® suspensions are shown in Fig. 3.[41] The values of zeta potential at the working pH reveal that the particles have positive charge in the suspension; hence, they move toward the cathode during EPD (cathodic deposition). By increasing the pH, the surface ionization is suppressed and the thickness of the electrical double layer decreases[42]; hence, the zeta potential decreases. In addition to the zeta potential, the conductivity of the suspensions (or ionic strength) influences the electrophoretic mobility. Fig. 5 shows the variation in the current density of 5 g/l Bioglass® suspensions

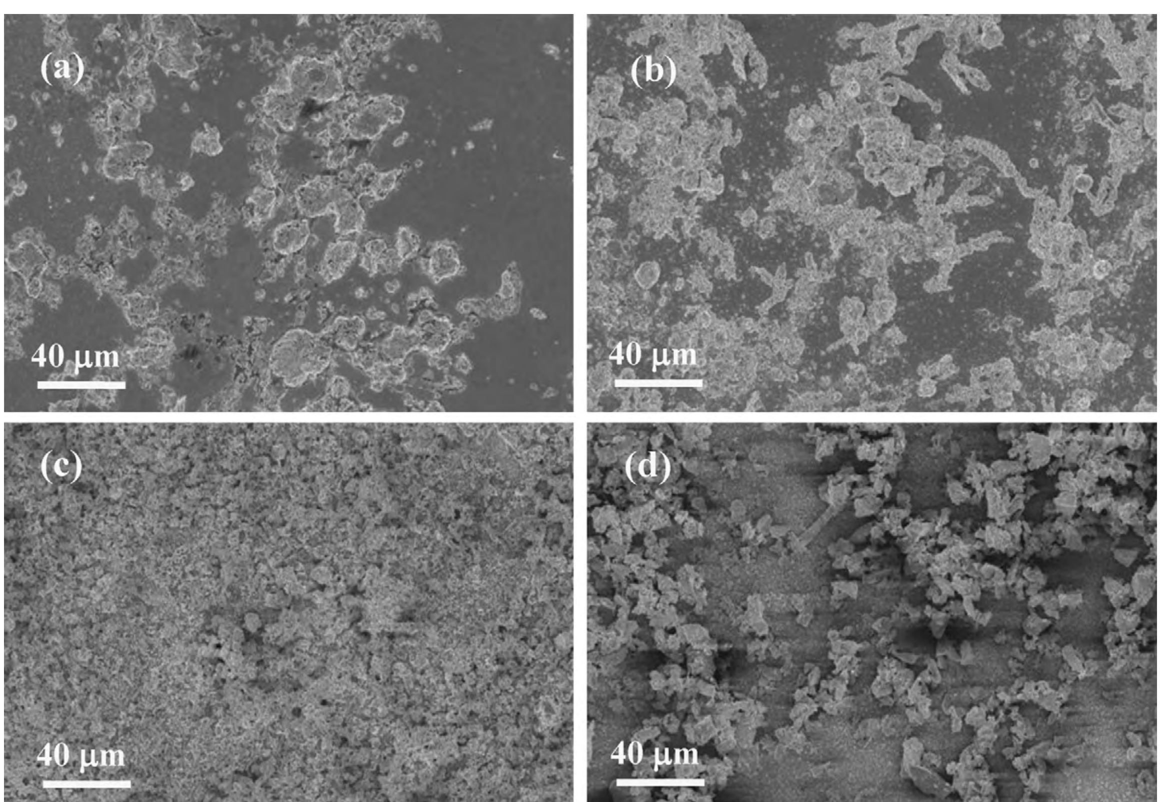

Fig. 7. SEM micrographs showing the Bioglass® electrophoretic deposits at the concentration of 10 g/l and pH values of (a) 3.2, (b) 4, (c) 7, and (d) 9.

Table 6
Regression analysis of deposition rate versus control factors.

Source	DF	SS[a]	MS[b]	*F*	*P*
Regression	1	320.691	320.691	8.05	0.013
Residual error	14	557.962	39.847		
Total	15	878.553			

[a] Sums of squares.
[b] Mean sums of squares.

Table 7
Results of the confirmation experiments for the deposition rate and standard deviation.

	Level	Deposition rate		Standard deviation	
		Value (mg/m^2 s)	S/N ratio (dB)	Value (±mg/m^2 s)	S/N ratio (dB)
Experiment	A4 B3 C2	6.62	16.42	±2.78	−8.88
Prediction	A4 B3 C2	7.37	17.36	±2.72	−8.70

versus deposition time at different pH values. Because of the partial solubility of Bioglass® in aqueous suspensions,[43] a higher current density was measured at lower pH values. The increase in the conductivity of the suspensions during EPD also reveals the progressive dissolution of the Bioglass®. Fig. 6 shows the effect of pH and Bioglass® concentration on the conductivity of the suspensions. It is confirmed that the lowest conductivity is achieved at high pH values. Therefore, it can be concluded that the interactions between the parameters of concentration and pH and the effect of it on the solubility and mobility of the particles, determine the optimum deposition yield (at pH 7). SEM images of the Bioglass® deposit surfaces prepared at different pH values are shown in Fig. 7 as an example for a 10 g/l Bioglass® suspension. The effect of pH on the quality of the deposition layer is visible at the SEM magnification selected. DOE analysis also revealed that a high deposition rate is obtained at higher voltages; however, it is suggested that EPD of Bioglass® suspensions should be performed at a low electric field, *e.g.* 5 V/cm. Although the confirmation test showed that applying a low electric field reduces the deposition rate remarkably (Table 7), a more uniform deposition rate can be obtained (least variability with a low standard deviation). This is due to the fact that at relatively high electric fields, Joule heating of the suspension during EPD could contribute to deposition instability and to changes in the local conductivity. In addition, the well-known water dissociation can occur in high electric fields and this effect will influence the process, so it should ideally be avoided or minimized by employing the lowest possible voltage.

6. Conclusions

Electrophoretic deposition of Bioglass® suspensions to fabricate bioactive coatings on metallic implants was investigated. The effect of processing parameters, including the suspension concentration, pH and electric field was studied using a Taguchi DOE approach. Optimization was performed based on the multivariable analysis of variance and least-square regression methods. It was shown that the control factors are inter-related but pH of the suspension has a critical role on the deposition rate and the stability of the EPD process. A high deposition rate was attained at pH 7. A low electric field is more suitable to obtain a stable EPD process. The prediction of the Taguchi design approach was in good qualitative agreement with the experimental results. The procedure developed in this work has broader applications in EPD and it can be used to optimise the EPD of composite suspensions containing natural polysaccharides (*e.g.* chitosan) and Bioglass® particles, which is the focus of current investigations.

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