Optimization of Nano Hydroxyapatite/chitosan Electrophoretic Deposition on 316L Stainless Steel Using Taguchi Design of Experiments

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Abstract

The aim of this work is to determine the optimum parameters for deposition of chitosan and mixture of chitosan and hydroxyapatite (HA) layers using electrophoretic deposition. The layers were on 316L stainless steel substrate. Taguchi approach was utilized to select the optimum parameters for both layers. The parameters used for deposition chitosan are voltage, time and temperature while the parameters used for HA and chitosan are voltage, time, concentration and temperature. Zeta potential tests were employed to measure the solutions stability. Coating layers were characterized for thickness, porosity and nanoroughness using optical microscopy (OM) and atomic force microscopy (AFM). The results from Taguchi design of experiments demonstrated that the best conditions for deposition of chitosan and HA layers are 50 V, 5 min, 3 g HA/L and 30°C. The corresponding thickness, % porosity, nanoroughness and microroughness for optimum conditions were 22 µm, 3.53, 4.48 nm and 3.85 µm respectively.

Keywords: Electrophoretic deposition; Chitosan Hydroxyapatite; Taguchi approach

1. Introduction

Electrophoretic deposition was considered as the most effective technique to produce thin and thick coats from fine suspended nanoparticles. Many advantages achieved from this technique are inexpensive equipment, simple setup, uniform and dense coatings deposition and capability for forming complex patterns and shapes [1,2]. Hydroxyapatite $[Ca_{10}(PO4)_6(OH)_2]$ was considered the most important calcium compounds that has been found in natural hard tissues as a mineral phase. Hydroxyapatite is widely used in biomedical applications because it possesses favorable properties; close similarity in chemical composition and high biocompatibility with natural bone tissue [3]. Weak or poor mechanical properties are limited hydroxyapatite employment. Therefore, to solve this problem, HA must be coated on the metallic surface [4]. In order to obtain dense and achieve high mechanical strength for coatings, EPD usually needs to be sintered at relatively high temperatures. These heating cycles may lead to significant oxidation, thermal stresses, chemical reactions between HA and the substrates and shrinkage [5]. However, these problems could be solved and eliminated effectively via fabricating of hydroxyapatite and polymer composite coatings. Bone includes organic and mineral phases; chitosan polysaccharide is in structure similar to glucosamine which is the major component of the extra cellular matrix (ECM) of bone and cartilage. Hence, significant attentions have been made to produce composites of HA/chitosan coatings [6]. Combinations of polymers and ceramic components can be applied to form organic/inorganic soft composite coatings [7].

Chitosan is an interesting polymer that has been widely used to produce a variety of coatings in combination with EPD [8]. It has been well established that chitosan is a cationic polysaccharide that has been used for biocompatible coatings and drug delivery [9,10]. Important properties of this material, such as biocompatibility, chemical resistance, mechanical strength, antimicrobial properties and thermal stability have been utilized in biotechnology [6,11]. Many previous studies have been focused on production of HA/chitosan depositions. Early reports about the electrophoretic deposition of chitosan were begun since 2002. Chitosan is positively charged and water soluble in acidic conditions at pH below 6.3. The positive charge results from the protonation of the amino groups [12].

Composite materials based on a combination of bioactive ceramics and biodegradable polymers including HA and chitosan have been taken great attention in the field of biomaterials. These composites showed physical, biological and mechanical properties as well as predictable favorable degradation behavior [13]. Composites of chitosan/HA have the best biomedical properties particularly for nanocomposite preparation [14].

Electrophoretic deposition of composite of HA/chitosan were deposited on 316L stainless steel by Mahmoodi et al and Pang and Zhitomirsky [4,6] at a constant applied voltage and different deposition times. Successful composite coatings were obtained. EPD

technique was used to deposit composites coating of hydroxyapatite/silica/chitosan on different conductive substrates [15]. Taguchi approach (DOE) was a useful statistical method which used to enable the modeling and to analyze the effects of specific control factors on the experiment output in order to avoid the high number of experiments [16, 17]. The process of EPD was found to be capable to fabricate coatings of laminates and graded composition. Taguchi approach was applied by Nameer et al [18] to study the electrophoretic deposition of composite coatings. They determined the effect of key parameters on the coating quality.

The aim of the present work is to select the optimum levels of parameters for deposition HA and chitosan layers (bioactive ceramic material) on chitosan (biopolymer) nanocomposite layers using EPD on 316L stainless steel substrate for orthopedic applications. The final target is to obtain a product that mimic and simulates human bones which consist of collagen matrix with organic and inorganic materials.

2. Experimental Procedures

316L stainless steel (Fe-16.8Cr-9.35Ni-1.87Mo-1.35Mn-0.578Si-0.043P-0.008S-0.015C (wt%) and two nanopowders of hydroxyapatite (reagent grade, powder, synthetic) and chitosan (medium molecular weight with a degree of deacetylation of about 85%, purchased from Sigma Aldrich). The stainless steel used as substrates for EPD depositions are with 20 x 10 x 2 mm. The samples were ground to produce rough surface of approximately 4.3 µm to enhance the bonding with the deposited chitosan layers. For deposition thin layers of chitosan, 0.5g/L of chitosan was dissolved in 1% acetic acid and then added to the solution of a mixture of 94% ethanol + 5% deionized water. The final solution was stirred for 24 hours to obtain the best dispersion. To deposit HA layers, different concentrations of HA (1, 2 and 3 g/L) were added to the final solution of 0.5g/L of chitosan, 1% acetic acid, 94% ethanol and 5% deionized water. The HA/ chitosan and the solution were stirred for 5 hours. The pH values for both solutions were performed at 4-4.5. The suspensions were further dispersed ultrasonically for 30 min. The electrodes were washed thoroughly with acetone and then dried. 316L stainless steel sheet was used as an anode with dimensions similar to those of substrate (cathode). The distance between the cathode and anode electrodes was fitted at 1 cm. The deposition area was performed to be 1 cm^2.

As the main aim of this study is to identify the near values of variables controlling the EPD, Taguchi design of experiments was applied for this purpose. Careful design for variables and their levels for deposition both layers of pure chitosan and the HA and chitosan were performed. These are based on the philosophy of Taguchi design of experiments and the experience of the author [293]. Tables 1 and 2 list the variables and corresponding levels for deposition of chitosan layers and HA/chitosan layers respectively. The selection criteria for deposition of chitosan layers and HA/chitosan laser are different. The optimum condition for deposition of chitosan is based on the maximum thickness. While for HA/chitosan the best layers are characterized with critical thickness, roughness and porosity. The surface layers were 4characterized by optical microscopy and atomic force microscopy.

Detailed analyses of the features of the deposited layers have been made mainly from the upper surface plan views. These were taken place without any surface metallographic preparation using optical and scanning electron microscopies. The depths of deposition thickness were determined from transverse sections. The EPD samples were cold mounted and then ground with 1200 SiC emery paper and finally polished with 1 µm diamond. Scanning electron microscopy type Tscan VEGA 3SB was used to investigate the upper surface plan views and transverse sections. The two and three dimensional configurations of the upper surface plan views were determined using Atomic Force Microscopy, type SPM AA3000, Angstrm advanced INC, USA. It has been used to evaluate the nanoroughness, morphology and the invariants of the cells. J image program was used to determine the percentage of porosity.

3. Results and discussions

3.1. Analysis of chitosan deposition

Table 1 lists the thickness of chitosan layers obtained from Taguchi design of experiments. It is important to observe that there was a wide range of thicknesses produce as different levels of variables. According to the SNs ratio theory (larger is the better) that the experiment has the highest value of SNs ratio implied the better quality (Table 1). It can be seen from Table 1 that samples 3 and 9 have the higher values of chitosan thickness and SNS ratio. The corresponding optimum levels for these experiments are 30 V, 11 min and 50 °C (experiment 3) and 90 V, 11 min and 40 ºC (experiment 9). It appears that lower deposition temperature is sufficient to obtain reliable chitosan thickness. This was preferred because the lowest temperature should be used with biopolymer and biomedical applications. Measurements of the solution stability for these optimum levels were obtained by zeta potential; it was 30.78. There is a good dispersion of particles in the solution as shown in Fig. 1.

The best homogeneous layer with good dispersion and little settling particles were obtained. Table 2 and Fig. 2 show the SNs ratio for thickness of chitosan layers for all variables at different levels.

Table 3 explains the ranking of significance of each level in various responses which derived from SNs data. It was noticed that the voltage has the highest effect on this procedure. The temperature has the second effect, while the time has the lowest effect. The best values for all levels at corresponding variables for chitosan thickness is presented in Table 4. The analysis of variance (ANOVA) shows that the chitosan thickness is highly controlled by voltage rather than the time and temperature (Table 5).

Table 1: Thickness and SNs ratio for all experiments**.**

Sample No.	Voltage	Time	Temp.	W1	W2	ΔW	Thickness (µm)	SN_S
1	30	5	30	3.1058	3.1061	0.0003	2.72	8.6914
2	30	8	40	3.0867	3.0873	0.0006	5.45	14.7279
3	30	11	50	3.0400	3.0409	0.0009	8.18	18.2551
4	60	5	40	3.1962	3.1966	0.0004	3.63	11.1981
5	60	8	50	3.0883	3.0885	0.0002	1.81	5.1536
6	60	11	30	3.0599	3.0602	0.0003	2.72	8.6914
7	90	5	50	3.4035	3.4042	0,0007	6.36	16.0691
8	90	8	30	3.1481	3.1486	0.0005	4.54	13.1411
9	90	11	40	3.0936	3.941	0.0009	8.18	18.2551

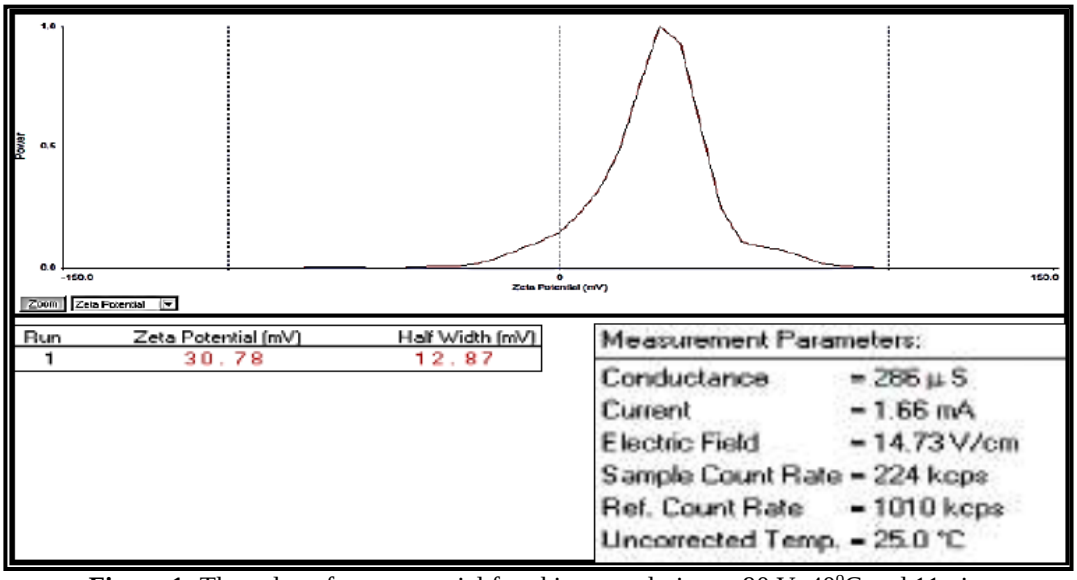


Figure 1: The value of zeta potential for chitosan solution at 90 V, 40°C and 11min.

Table 2: Signal-to-Noise ratio for each level of controlled factors for chitosan layer thickness.

	Average SN_S Ratio of levels for deferent variables		
	Level 1	**Level 2**	**Level 3**
Volt, V	13.891	8.348	15.822
time, min	11.986	11.008	15.067
Temperature, °C	10.175	14.727	13.159

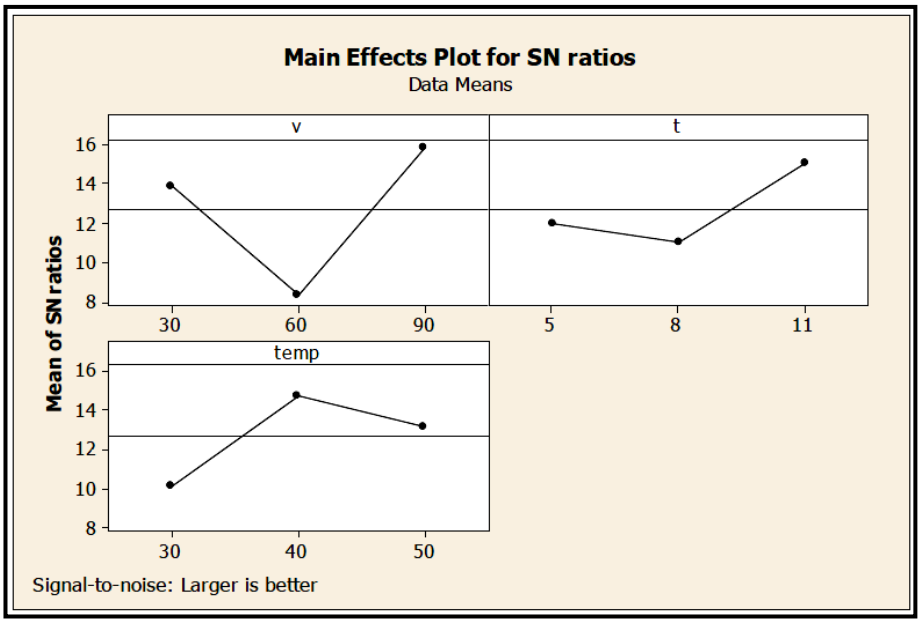


Figure 2: Mean of SNs response for chitosan thickness for different variables and levels

Table 3: Rank for variables of chitosan thickness and process performance for each level.

Level	Volt, V	time, min	Temperature, °C
1	13.891	11.986	10.175
2	8.348	11.008	14.727
3	15.822	15.067	13.159
Delta	7.474	4.060	4.552
Rank	1	3	2

Table 4: Best values and corresponding levels for all variables governed the chitosan thickness.

Variable	Value	Level
Voltage, V	90	3
Time, min	11	3
Temperature, °C	40	2

Table 5: ANOVA of thickness for chitosan layer.

Variable	DOF	Sum of squares SS	Variance MS	Contribution %
V	2	90.32	45.161	53.53
t	2	26.93	13.465	15.96
Temp.	2	32.09	16.045	19.01
Errors	2	19.37	9.687	11.48
total	8	168.72		

3.2. Effect of HA/chitosan thickness

In order to characterize the upper surface of HA/chitosan layers, the effect of parameters used in the experiments (voltages, time, % concentration of powder and temperature) on thickness, porosity and nanoroughness were studied (Table 6). It should be mentioned that all depositions have been made above the chitosan thickness layer of 8.18 µm thickness. The thicknesses mentioned in Table 6 are for the HA/chitosan composite only. The important output key is the thickness rather than the other outcome features. This is because the thickness is an important key to perform the application. In order to characterize the HA/chitosan layers, the data obtained from topography and the transverse section are necessary to describe the deposition process (Fig. 3).

Table 6. Signal-to-noise ratio of Taguchi design of (L9) for (a) porosity of HA coating layer

Experiment	Voltage, V	Time, min	HA, g/L	Temperature, °C	Average Porosity%	SNs
1	10	1	1	30	0.62	-4.1522
2	10	5	2	40	0.70	-3.0980
3	10	9	3	50	2.96	9.4258
4	50	1	2	50	3.48	10.8316
5	50	5	3	30	3.53	10.9555
6	50	9	1	40	0.71	-2.9748
7	90	1	3	40	2.35	7.4214
8	90	5	1	50	0.62	-4.1522
9	90	9	2	30	0.71	-2.9748

(b) thickness of HA layer

Experiment	Voltage, V	Tim, min	HA, g/L	Temperature, °C	Average thickness, µm	SNs

1	10	1	1	30	14.9	23.4637
2	10	5	2	40	10.0	20.0000
3	10	9	3	50	18.0	25.1055
4	50	1	2	50	14.6	23.2871
5	50	5	3	30	22.0	26.8485
6	50	9	1	40	12.0	21.5836
7	90	1	3	40	14.0	22.9226
8	90	5	1	50	12.3	21.7981
9	90	9	2	30	18.3	25.2490

(c) Nano roughness of HA layer

Variables	Voltage, V	Time, min	HA, g/L	Temperature, °C	Ra, nm	SNs
1	10	1	1	30	6.35	16.0555
2	10	5	2	40	4.50	13.0643
3	10	9	3	50	3.58	11.0777
4	50	1	2	50	3.77	11.5268
5	50	5	3	30	4.48	13.0256
6	50	9	1	40	12.90	22.2118
7	90	1	3	40	9.15	19.2284
8	90	5	1	50	4.14	12.3400
9	90	9	2	30	8.10	18.1697

(d) Micro roughness of HA layer

Experiment	Volt, V	Time, min	%HA	Temperature, °C	Ra, µm	SNs
1	10	1	1	30	3.99	12.0195
2	10	5	2	40	4.09	12.2345
3	10	9	3	50	3.73	11.4342
4	50	1	2	50	4.00	12.0412
5	50	5	3	30	3.85	11.7092
6	50	9	1	40	4.10	12.2557
7	90	1	3	40	3.55	11.0046
8	90	5	1	50	3.38	10.5783
9	90	9	2	30	3.59	11.1019

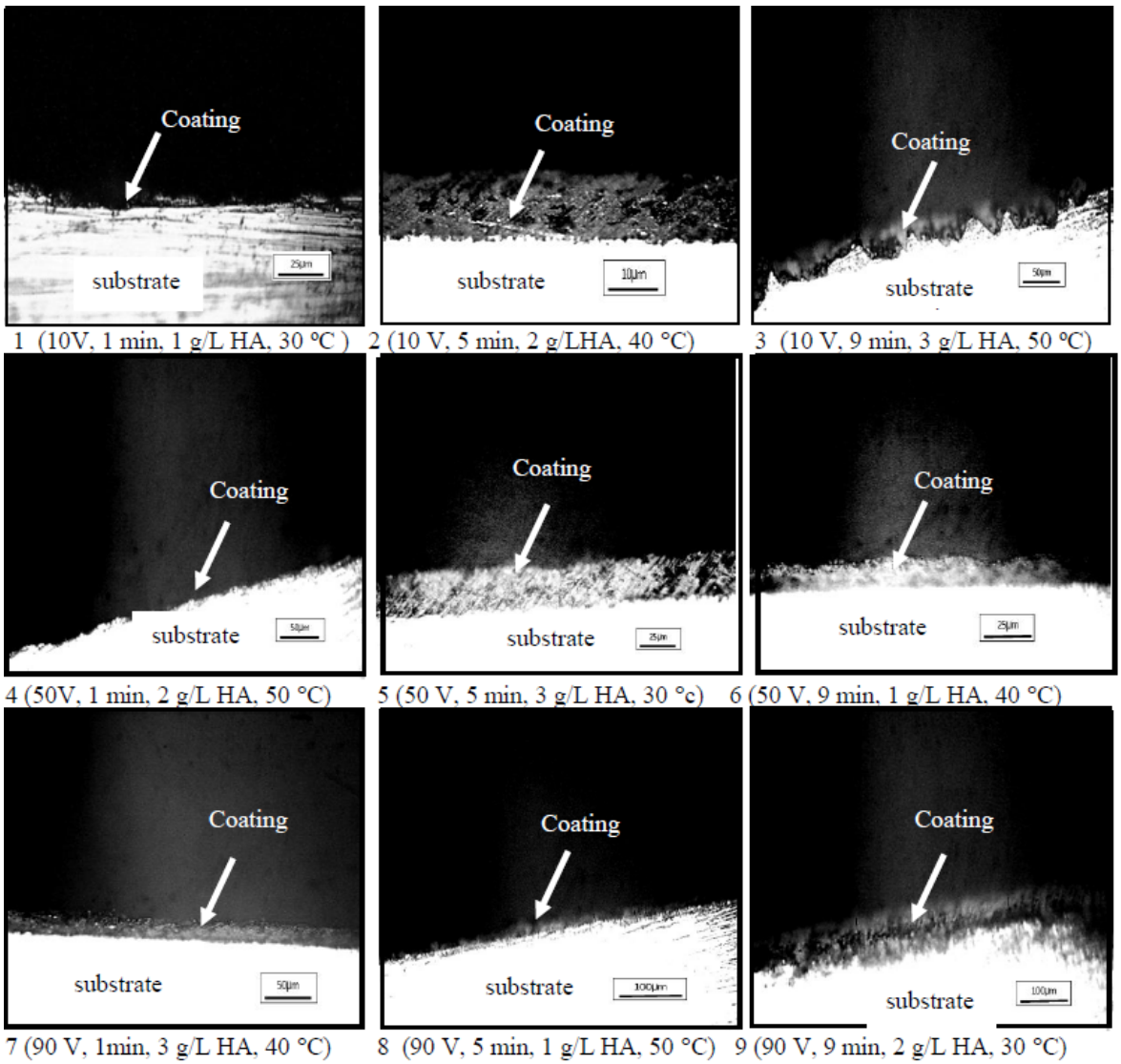


Figure 3: Optical microscopy images for samples of HA/chitosan layers.

It was observed that the highest thickness (22μm) was obtained at 50 V, 5 min, 3% HA and 30 °C. This layer is chracterized with low porosity and high homogeniouty. On the other hand, image J was used to estimate the percentage of porosity for each sample as shown in Fig. 4 From Table 6, it had been observed that the coating which was obtained by using the parameters of 50 V, 5 min, 3% HA and 30°C would achieved higher and suitable value of porosity (3.53%). Nanoroughness are measured by AFM observation and the microroughness was measured by using micrometer instrument and it was noticed that there is different roughnesses obtained as shown in Table 6. The higher nanoroughness was obtained with sample 6 at 50 V, 9 min, 1 %HA and 40 °C) (12.9 nm). The value with microroughness was 4.1 μm. Sample 5 at 50 V, 5 min, 3% HA and 30 °C has suitable nano and microroughness (4.48 nm and 3.85 μm respectively). Also the dense packing and fine variants for topography coating were obtained (Fig. 5). There are different particles distributions with each set of parameters. It appears that the granularity accumulations for each coating layers at different parameters (Fig. 6). Therefore, Taguchi statistical approach was used in order to select the optimum conditions.

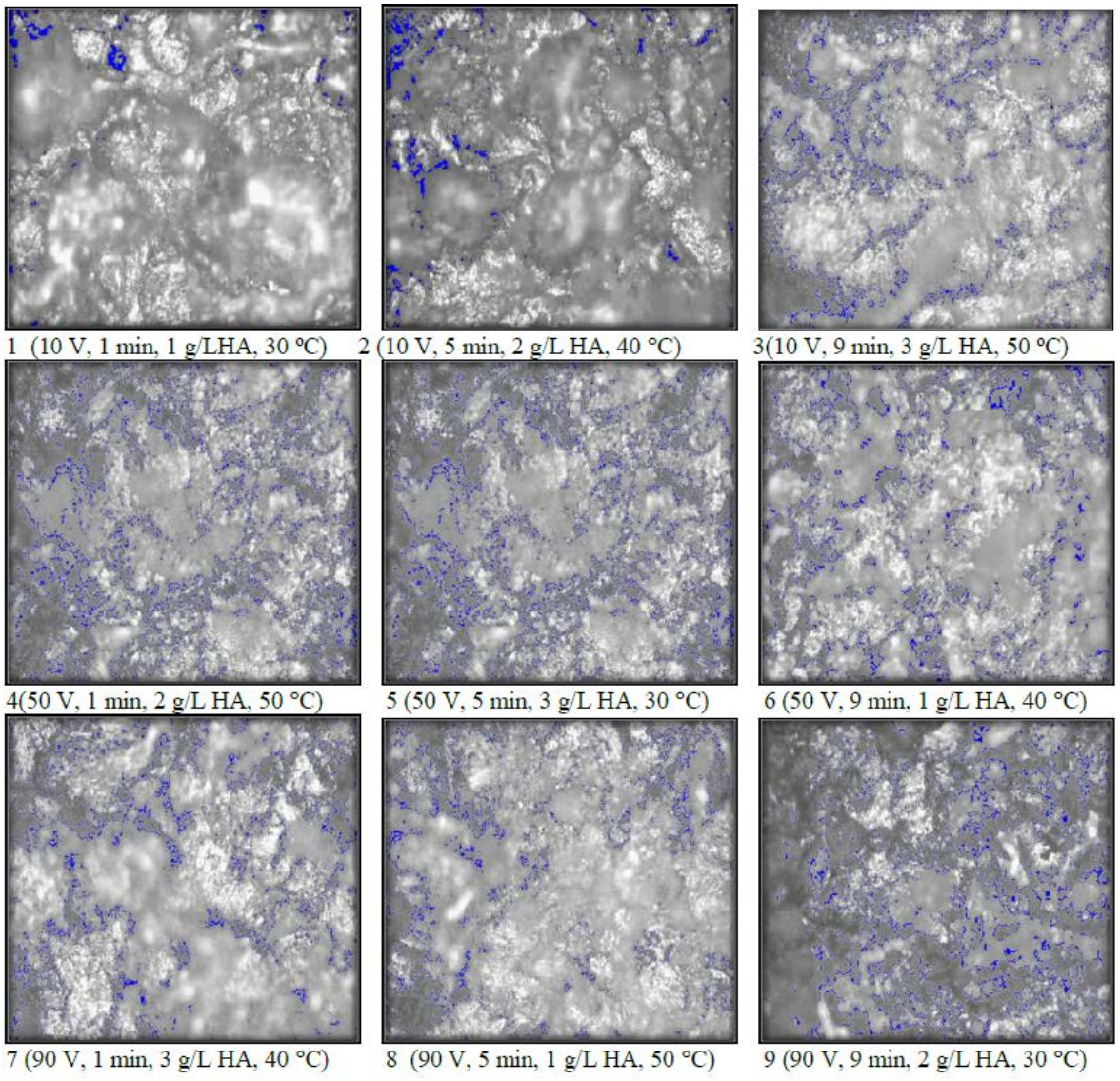
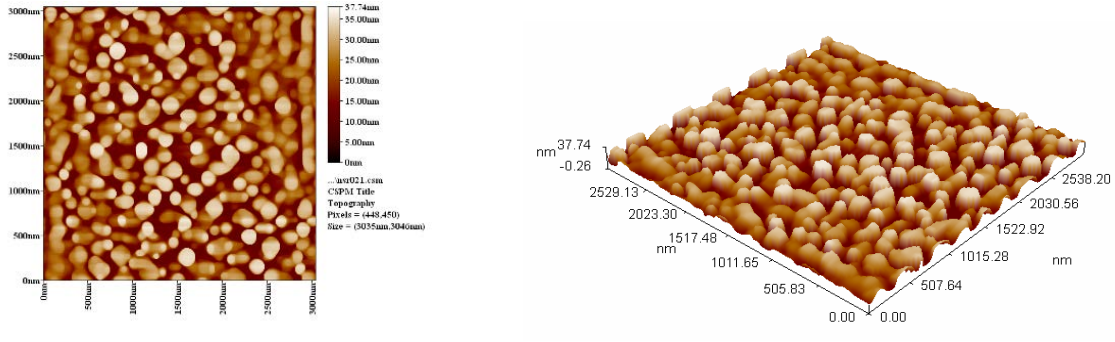
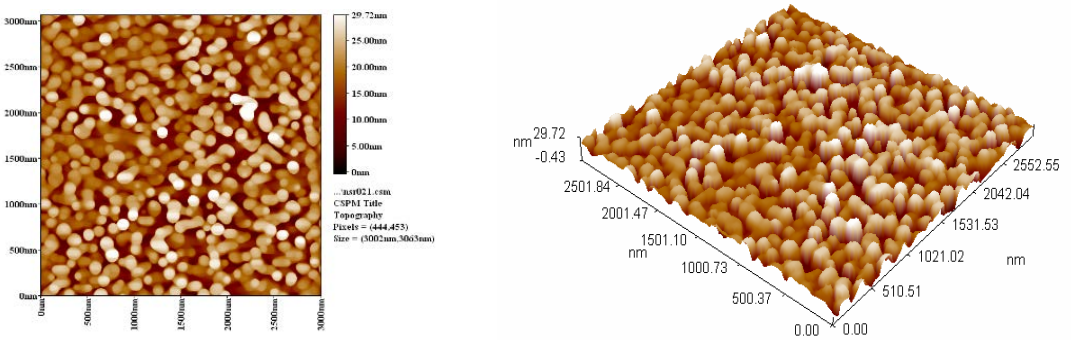


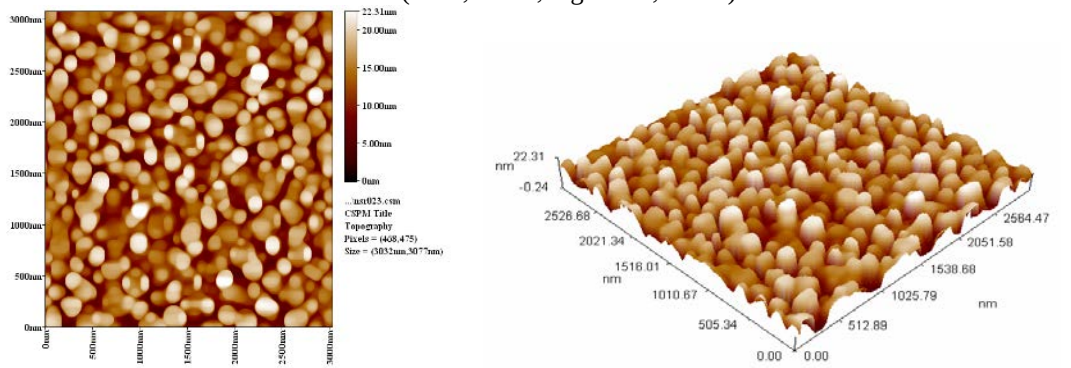
Figure 4: Optical images of porosity for HA coating (blue color refer to pores area).



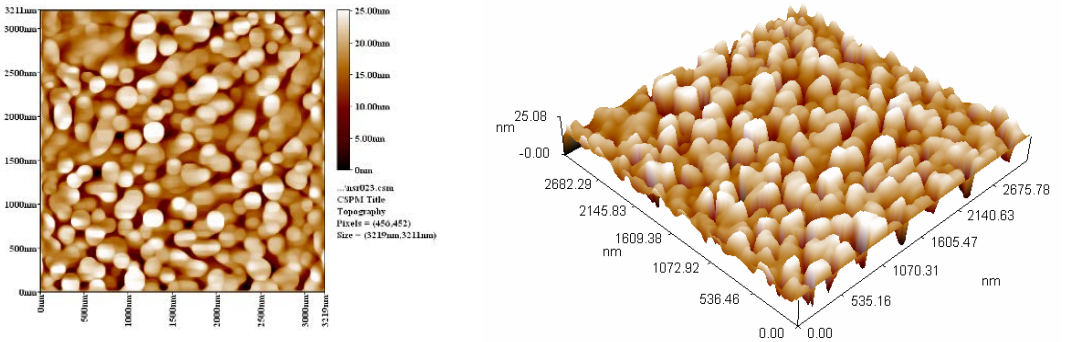
1 (10V, 1min, 1g/LHA, 30 ^{o}C)



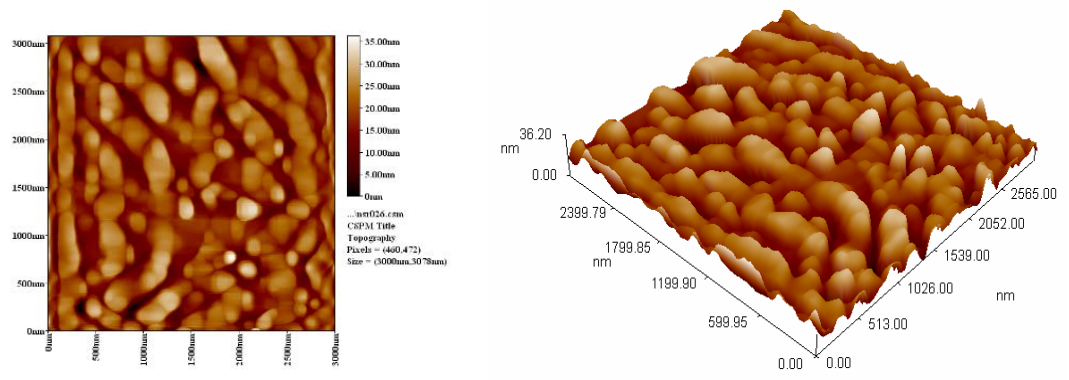
2 (10 V, 5 min, 2 g/L HA, 40 °C)



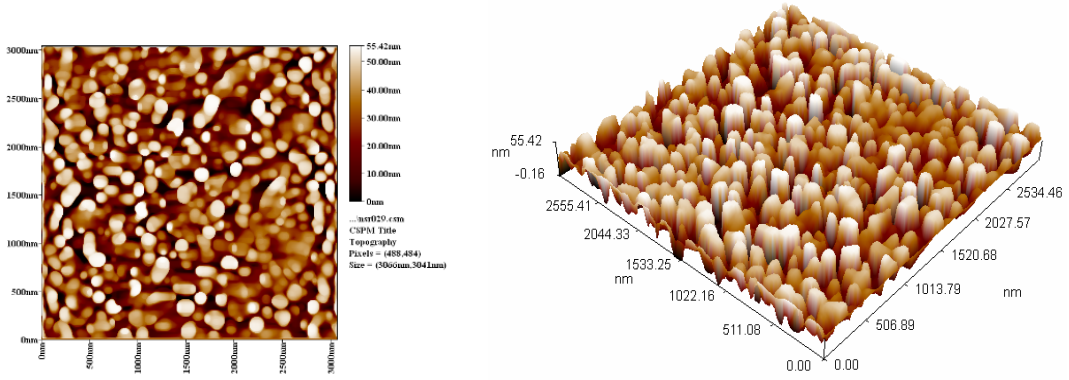
3 (10 V, 9 min, 3 g/L HA, 50 ^{o}C)



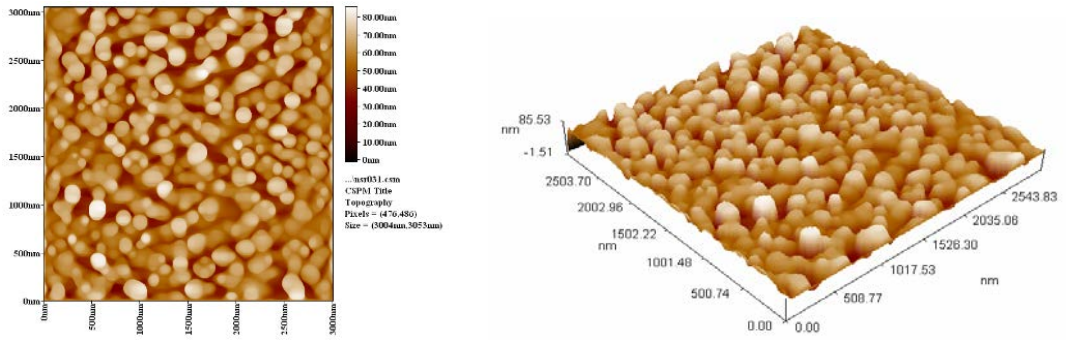
4 (50 V, 1 min, 2 g/L HA, 50 °C)



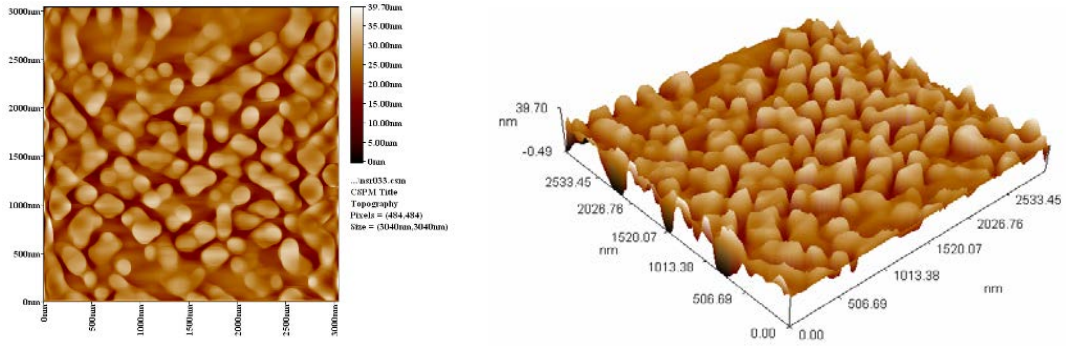
5 (50 V, 5 min, 3 g/L HA, 30 °C)



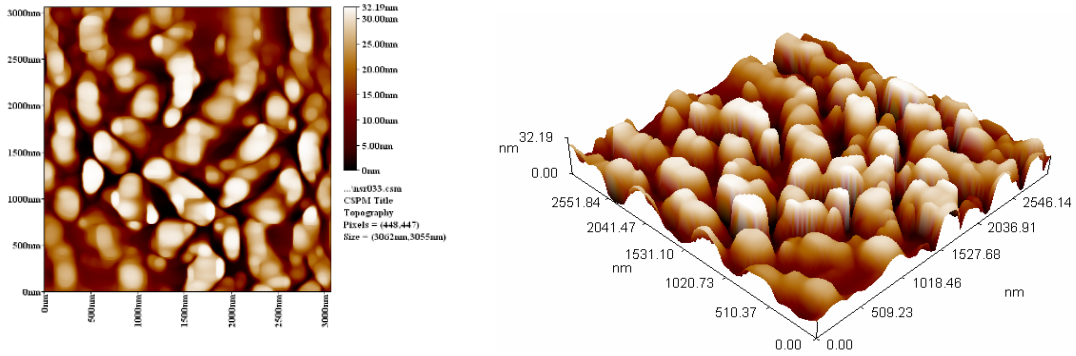
6 (50 V, 9 min, 1 g/L HA, 40 °C)



7 (90 V, 1 min, 3 g/L HA, 40 °C)

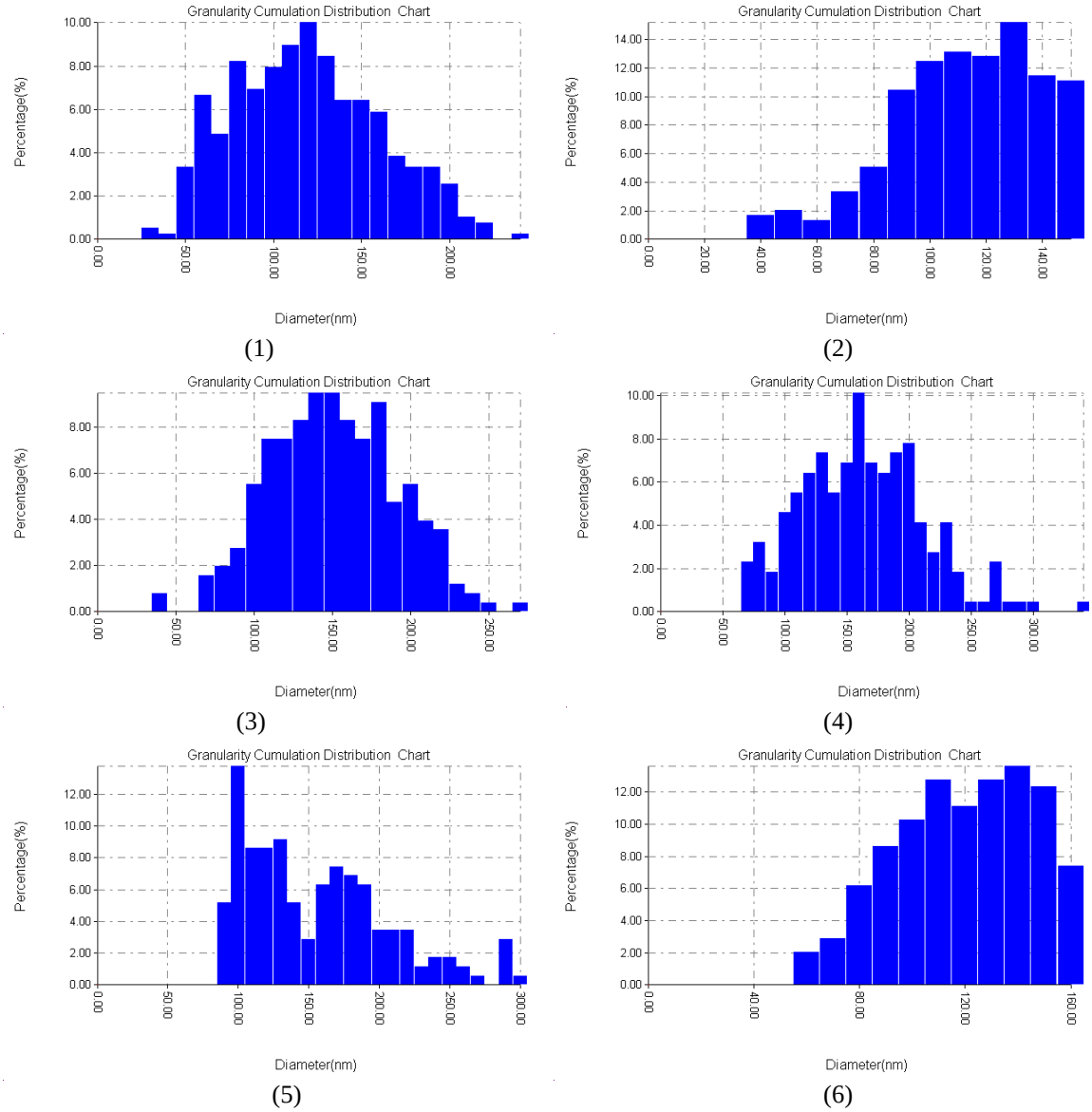


8 (90 V, 5 min, 1 g/L HA, 50 °C)



9 (90 V, 9 min, 2 g/L HA, 30 °C)

Figure 5: Two and three dimensions features of (AFM) images for HA coating samples.



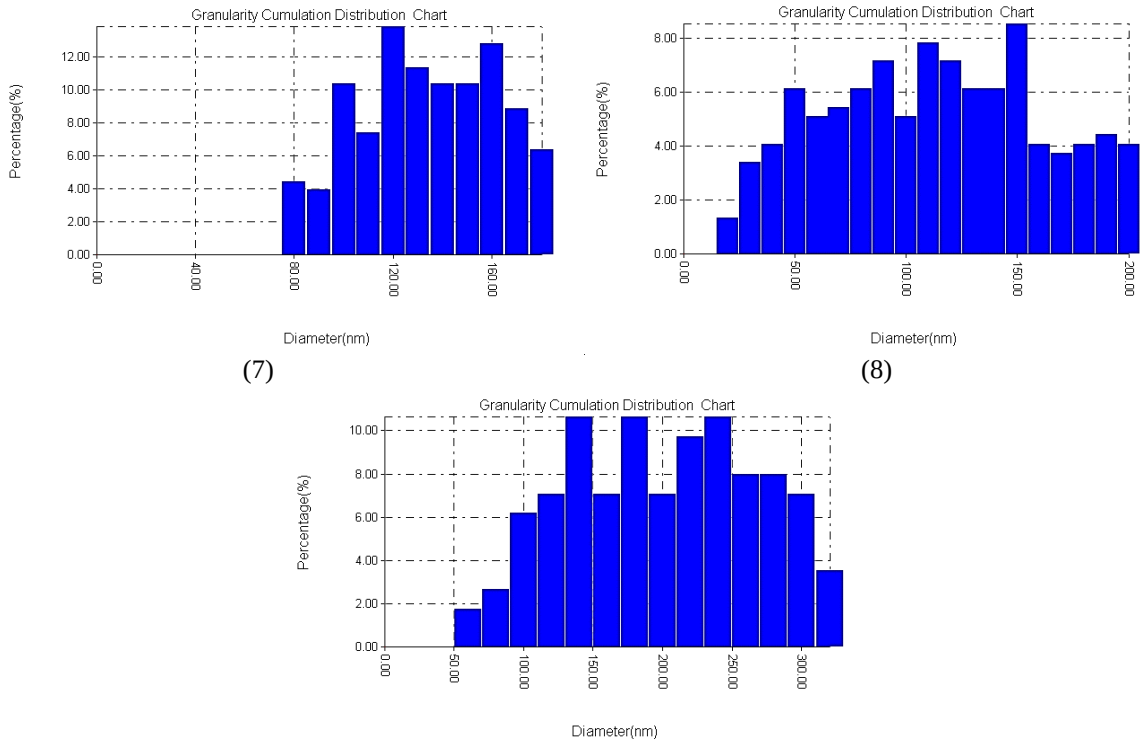


Figure 6: Charts of granularity accumulation for coating particles of HA coating layer for different samples.

From SNs ratio for the four outputs data (porosity, thickness, nanoroughness and microroughness), the higher SNs ratio for porosity was with sample number 5 at (50 V, 5 min, 3 g/L HA, 30 ^{o}C) is 10.9555. Table 7 and Fig. 7 show the SNs ratio for thickness, porosity, nanorughness and microroughness of HA/chitosan layers for all variables at different levels. Data presents in Table 7 were used to determine the values of delta and the rank for each variable (Table 8). Table 8 shows that the delta is highly different for each performance. The higher delta was observed for porosity while the lower delta was found for microroughness. Table 8 also shows that different best values for all levels were produced for the features evaluated. The analysis of variance (ANOVA) demonstrates that each feature is determined highly by different variables; %porosity determines by %HA, thickness determines by temperature, nanoroughness determines by temperature and microroughness determine by voltage (Table 9).

Table 7: Signal-to-Noise ratio for each level of controlled factors for HA layer for:

(a) porosity for HA layer

	Average SN_S Ratio of levels for deferent variables for porosity		
	Level 1	**Level 2**	**Level 3**
Volt, V	0.72521	6.27075	0.09812
time, min	4.70026	1.23510	1.15872
HA, g/L	-3.75972	1.58624	9.26756
Temperature, ^{o}C	1.27616	0.44950	5.36842

(b) thickness for HA layer

	Average SN_S Ratio of levels for deferent variables		
	Level 1	**Level 2**	**Level 3**
Volt, V	22.80	23.91	23.32
time, min	23.22	22.88	23.98
HA, g/L	22.22	22.85	24.96
Temperature, ^{o}C	25.19	21.50	23.40

(c) nanoroughness for HA layer

	Average SN_S Ratio of levels for deferent variables		
	Level 1	**Level 2**	**Level 3**
Volt, V	13.40	15.59	16.58
time, min	15.60	17.81	17.15
HA, g/L	16.87	14.25	14.44
Temperature, ^{o}C	15.75	18.17	11.65

(d) micro roughness for HA layer

	Average SN_S Ratio of levels for deferent variables		
	Level 1	**Level 2**	**Level 3**
Volt, V	11.90	12.00	10.89
time, min	11.69	11.51	11.60
HA, g/L	11.62	11.79	11.38
Temperature, ^{o}C	11.61	11.83	11.35

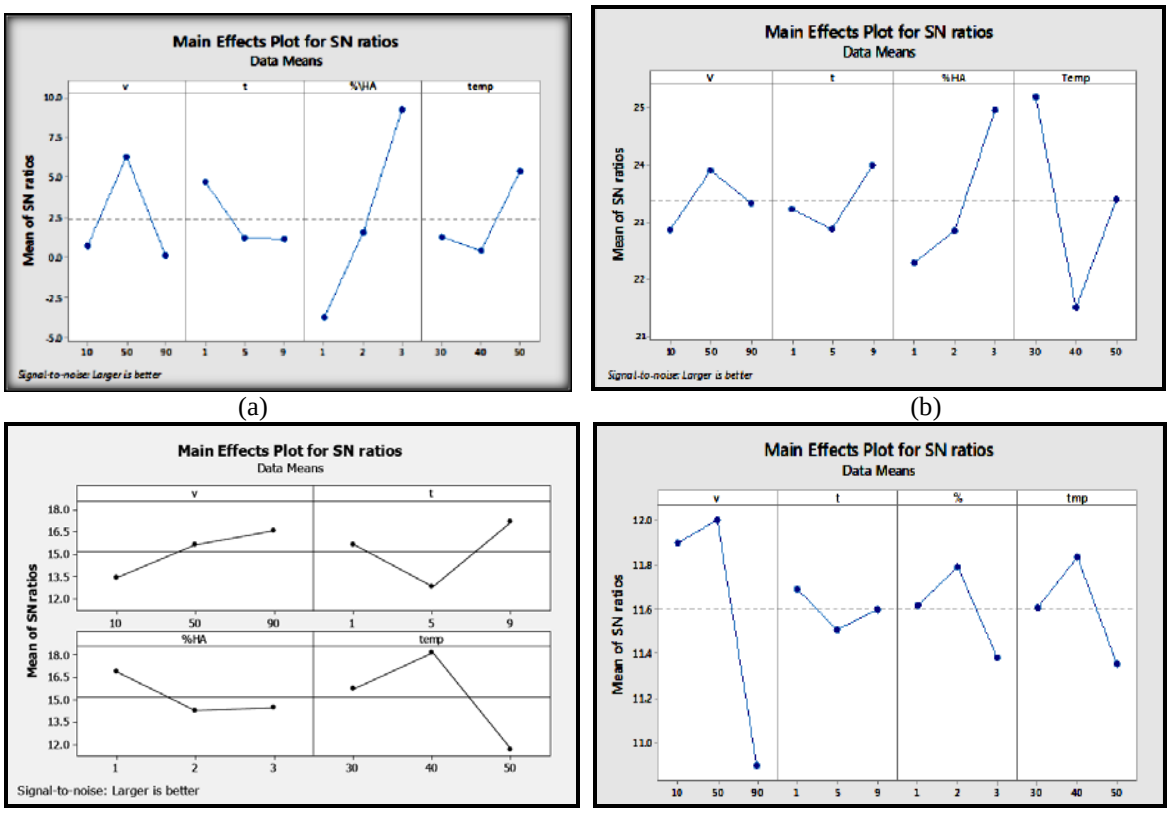


Figure 7: SNs ratio (a) %porosity, (b) thickness, (c)nanoroughness and (d) microroughness.

Table 8: Rank of controlled factors for:

(a) Porosity for HA layer

Level	Voltage, V	Time, min	HA, g/L	Temperature, °C
1	0.72521	4.70026	-3.75972	1.27616
2	6.27075	1.23510	1.58624	0.44950
3	0.09812	1.15872	9.26756	5.36842
Delta	6.17263	3.54154	13.02728	4.91892
Rank	2	4	1	3

(b) Thickness for HA layer

Level	Volt, V	Time, min	%HA	Temperature, °C
1	22.80	23.22	22.22	25.19
2	23.91	22.88	22.85	21.50
3	23.32	23.98	24.96	23.40
Delta	1.11	1.10	2.74	3.63
Rank	3	4	2	1

(c) Nano roughness for HA layer

Level	Volt, V	Time, min	HA, g/L	Temperature, °C
1	13.40	15.60	16.87	15.75
2	15.59	12.81	14.25	18.17
3	16.58	17.15	14.44	11.65
Delta	3.18	4.34	2.62	6.52
Rank	3	2	4	1

(d) Micro roughness for HA layer

Level	**Volt, V**	**Time, min**	**%HA**	**Temperature, °C**
1	11.90	11.69	11.62	11.61
2	12.00	11.51	11.79	11.83
3	10.89	11.60	11.38	11.35
Delta	1.11	0.18	0.41	0.48
Rank	1	4	3	2

Table 9: ANOVA for:

(a) Porosity of HA layer

Variable	**DOF**	**Sum of squares SS**	**Variance MS**	**Contribution %**
V	2	69.25	34.62	17.63
t	2	24.56	12.28	6.25
%HA	2	257.29	128.65	65.51
Temp.	2	41.63	20.81	10.6
Errors	0			
total	8	392.72		

(b) Thickness of HA layer

Variable	**DOF**	**Sum of squares SS**	**Variance MS**	**Contribution%**
V	2	1.846	0.9230	5.17
t	2	1.946	0.9732	5.42
%HA	2	12.339	6.1696	34.4
Temp.	2	19.736	9.8682	55.02
Errors	0	-		
total	8	35.868		100

(c) Nano roughness of HA layer

Variable	DOF	Sum of squares SS	Variance MS	Contribution%
V	2	15.89	7.944	12.9
t	2	29.07	14.534	23.65
%HA	2	12.76	6.379	10.38
Temp.	2	65.18	32.592	53.03
Errors	0	-		
total	8	122.90		

(d) Micro roughness of HA layer

Variable	DOF	Sum of squares SS	Variance MS	Contribution%
V	2	2.2391	1.11956	77.53
t	2	2.04916	0.02454	1.69
%HA	2	0.25381	0.12691	8.76
Temp.	2	0.34679	17339	17.339
Errors	0			
total	8	2.88889		

It was found that the best combination of high thickness, high porosity and high microroughness could be found in sample 5 (50 V, 5 min, 3g/L HA and 30 ºC). This is due to good stability of solution which confirmed by positive zeta potential value (49.71 mv) as shown in Fig. 8. The stable zeta potential was indicated due to high surface positive charges and stability for the suspension. Additionally, good close pores were obtained because the porosity was needed as an important parameter in bone replacement.

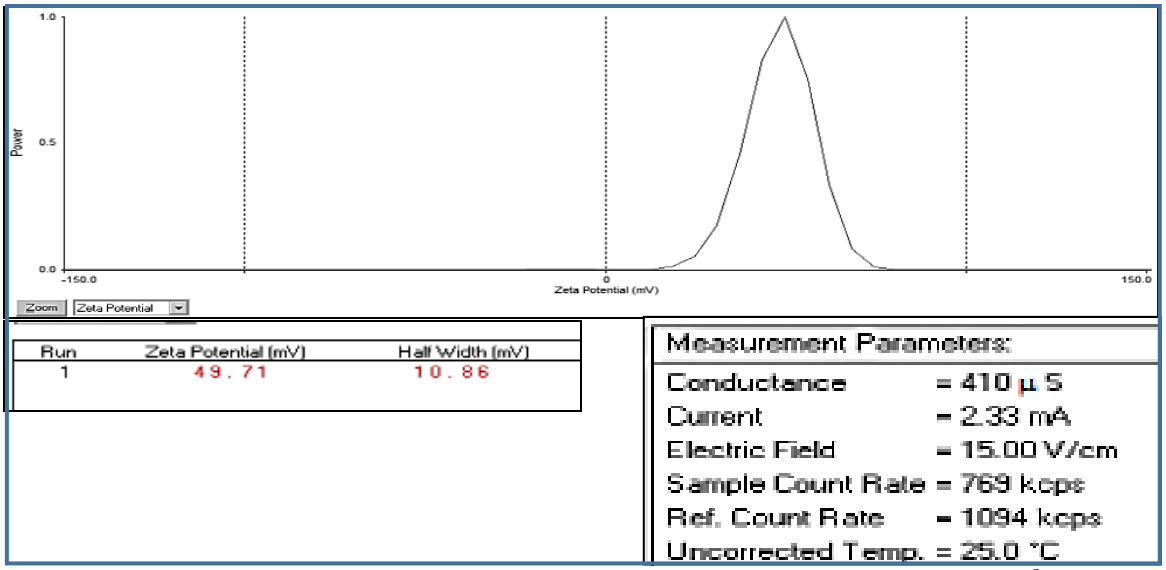


Figure 8: The zeta potential value for solution of HA/chitosan at 50 V, 6 min and 30 °C.

4. Conclusions

1- The optimum conditions for deposit thin chitosan layer can be achieved at 90 V, 11 min and 40ºC.
2- It is possible to produce uniform and dense chitosan with 8 µm thickness.
3- The optimum condition to deposit HA/chitosan layer are 50 V, 5 min, 30 °C and 3 g/L HA with 22 µm thickness .
4- The optimum conditions for deposition chitosan and HA/chitosan were obtained due to the good stability of solution at zeta potential of 30.78mV and 49.71 mV respectively.

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الترسيب الكهربي الامثل للنانوهيدروكسي اباتايت/كايتزون لسبيكة الفولاذ المقاوم للصدأ 316L باستخدام برنامج تاكوجي لتصميم التجارب

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الخلاصة

يهدف البحث الى ايجاد الظروف المثلى لترسيب طبقة طلاء الكايتوزون والهيدروكسي ابتايت على السبيكة الاساس الفولاذ المقاوم للصدا بطريقة الترسيب الكهربائي الكاثودي . حيث تم ترسيب طبقة رقيقة من الكايتوزون فضلا عن استخدامه كمادة رابطة بين دقائق طبقات الهيدروكسيباتايت. استخدم الايثانول مع نسبة 5% ماء مقطركمحلول لعملية الترسيب مع 1% من حامض الخليك لاذابة الكايتوزون. تم استخدام برنامج تاكوجي لغرض اختيار الظروف المثلى لترسب طبقة الكايتوزن وتضمنت المتغيرات: فولتية، زمن الترسيب ، ودرجة حرارة كما استخدمت نفس المتغيرات لترسيب الهيدروكسي ابتايت مع اضافة متغير اخر وهو تركيز المسحوق. تم استخدام المجهر الضوئي (optical microscopy) ومجهرالقوى الذرية (AFM) لدراسة خصائص طبقات الطلاء من سمك ومسامية وخشونة. وقد اظهرت النتائج ان افضل الظروف لترسيب الكايتووزن هي(90 فولت، 11 دقيقة و40 مº) وكان سمك الطبقة هو8 مايكرون واقضل الظروف للهيدروكسيابتايت هي (50 فولت ، 5 دقيقة ، 3 % هيدروكسيابتايت و30 مº) وكان سمك الطبقة هو 22 مايكرون والمسامية هي 3.53% والخشونة النانوية هي 4.48 نانومتر والخشونة المايكروية هي 3.85.