Optimization of hydroxyapatite coating on Ti-etched by hydrothermal method for biomedical material applications

Nguyen Viet Tung[1, 2], Cao Xuan Thang [1, 2*], Pham Hung Vuong [1, 2], Nguyen Viet Hung[1]

[1]School of Materials Science and Engineering, Hanoi University of Science and Technology, 01 Dai Co Viet, Hanoi, Viet Nam;
[2]Laboratory of Biomedical Materials, Hanoi University of Science and Technology, 01 Dai Co Viet, Hanoi, Vietnam.
[*]Corresponding author: thang.caoxuan@hust.edu.vn
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ABSTRACT

This study employed hydrothermal synthesis to fabricate hydroxyapatite (HA) coatings on surface-treated titanium substrates, combined with a second-order orthogonal regression model and statistical optimization techniques, including Response Surface Methodology (RSM) and Analysis of Variance (ANOVA), to optimize key processing parameters. The formation of a single-phase HA coating with nanoscale features on the titanium substrate was confirmed by X-ray diffraction (XRD) and FESEM-EDS analyses, together with evidence of excellent biocompatibility. The HA-coated samples were further evaluated through in vitro testing using baby hamster kidney (BHK) cells, demonstrating favorable cell proliferation and detailed cellular morphology observed by confocal laser scanning microscopy (CLSM). The RSM analysis identified the optimal processing conditions as follows: hydrothermal temperature (T_1) of 216.5 °C, hydrothermal time (T_2) of 40.2 h, and titanium substrate treatment time in an HCl/H_2SO_4 acid mixture at 60 °C (T_3) of 65.3 minutes. Under these optimized conditions, the HA coating achieved a maximum shear strength of 7.52 MPa. This HA coating on a treated titanium substrate holds strong potential in biomedical applications, particularly in military medicine for bone implants and prosthetic replacement components.

Keywords: Hydroxyapatite coating; HA coating; Hydrothermal method; DOE; Box-Wilson.

1. INTRODUCTION

Titanium and its alloys have been widely used in orthopedic and dental implants due to their excellent corrosion resistance, biocompatibility, and mechanical strength [1]. Previous studies have demonstrated that titanium surfaces etched in an HCl/H_2SO_4 acid mixture at elevated temperatures for appropriate durations develop uniform micro-roughness and improved biocompatibility [2, 3]. In particular, dual acid etching using HCl/H_2SO_4 has been reported to produce micro-roughened surfaces that enhance resistance to counter-torque and promote osseointegration [4]. Among various biomaterials, hydroxyapatite ($Ca_{10}(PO_4)_6(OH)_2$, HA) has emerged as a promising coating material because its structure and chemical composition closely resemble those of natural bone tissue. Furthermore, HA exhibits excellent corrosion resistance and biocompatibility, enabling it to function effectively as a protective and bioactive layer in orthopedic applications [5]. Various techniques have been investigated for fabricating hydroxyapatite coatings on titanium and titanium alloy substrates for biomedical applications, including plasma spraying [6, 7], hot isostatic pressing [8], thermal spraying [9], dip coating [10], electrophoretic deposition [11], sol–gel processing [12], and hydrothermal synthesis [13, 14]. Among these methods, hydrothermal synthesis has attracted particular attention because of its operational simplicity, low processing cost, and ability to precisely control coating structures from the micro- to nanoscale on titanium-based biomaterials [15]. Although hydrothermally synthesized HA coatings on titanium substrates have been extensively studied, limited research has focused on the effects of substrate modification conditions, hydrothermal temperature, and processing time on

coating durability, particularly regarding the interfacial mechanical performance and shear strength of the HA–Ti system.

Therefore, this study aims to synthesize and characterize HA coatings on titanium substrates modified with an HCl/H_2SO_4 acid mixture using a hydrothermal approach combined with Design of Experiments (DOE). The effects of hydrothermal temperature, processing time, and substrate etching time on the shear strength of the coatings were systematically investigated. To optimize the process parameters, a combined DOE and Response Surface Methodology (RSM) approach was employed.

2. PROBLEM

2.1. Theoretical foundations

Hydrothermal parameters were determined through design-optimized experiments (DOE) using response surface methodology (RSM) [16-18]. A total of 17 experiments, including three central point replicates, were performed. Hydrothermal process parameters examined included hydrothermal temperature (T_1) from 220 °C to 240 °C, hydrothermal time (T_2) from 12 to 36 hours, and etching time (T_3) of the titanium substrate in a H_2SO_4/HCl solution mixture at 60 °C from 30 to 60 minutes. This study focuses on building a regression model with three factors, T_1, T_2, T_3, with coded variables X_1, X_2, X_3 and objective function values of shear strength (Y). In a system involving three significant independent variables X_1, X_2, X_3, the mathematical relationship of the response to these variables can be approximated by the second-degree polynomial equation 1:

$$Y = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_{12}X_1X_2 + b_{13}X_1X_3 + b_{23}X_2X_3 + b_{11}X_1^2 + b_{22}X_2^2 + b_{33}X_3^2 \quad (1)$$

where Y is the predicted response (Shear strength, MPa); b_0 is the model constant; b_1, b_2, b_3 are linear coefficients; b_{12}, b_{13}, b_{23} are cross-product coefficients; and b_{11}, b_{22}, b_{33} are the quadratic coefficients.

2.2. Experiment preparation

2.2.1. Materials

Commercial titanium (Merck, 99.5%) was washed with an ultrasonic vibrator in acetone and ethanol for 10 min, then rinsed again with distilled water and allowed to dry in air. These titanium substrate samples were modified by etching in a mixed acidic solution of 48% H_2SO_4 (Merck, 98%) and 37% HCl (Merck, 37%) at 60 °C for 30 - 60 minutes. Finally, the samples were rinsed with distilled water and dried in preparation for HA film coating by the hydrothermal method.

2.2.2. Experimental materials

The synthesis of HA material by the hydrothermal method was carried out using precursor materials such as $Ca(NO_3)_2.4H_2O$, $Na_2EDTA.2H_2O$, $NH_4H_2PO_4$, NH_4OH supplied by Merck, Germany. The titanium substrate was modified, and the solution was stirred and transferred to an autoclave to carry out the hydrothermal process under conditions such as temperature (180 – 240 °C), time (7 – 36 hours), [Ca]/[P] ratio = 1.67, and the above solution was adjusted to pH = 10 by adding NH_4OH. The samples after hydrothermal treatment were washed again with distilled water and dried at 60 °C for 1 h.

2.2.3 Surface characterization

Factors affecting the morphology and structure of HA crystals were measured by digital optical microscopy (VHX-7000). The field-emission scanning electron microscopy (FE-SEM, JEOL JSM-7600F) was also used to determine the surface morphology of the coating. X-ray diffraction is utilized to determine structural phase of the HA coating. The measurements were performed on an XRD, D8 Advance, Bruker, Germany using Cu-Kα radiation: λ = 1.54056 nm. The shear strength properties of the HA coating were tested according to ASTM F 1044. The proliferative capacity of baby hamster kidney (BHK cells) was observed using a confocal laser scanning microscope (FV3000, Olympus, Japan) to assess the biocompatibility of the HA coating.

3. RESULTS AND DISCUSSION

3.1. Etching process

Titanium substrate samples immersed in an HCl/H_2SO_4 solution at a predetermined ratio and treated at 60°C for varying durations exhibited significant changes in surface wettability. As shown in Figure 1, increasing the etching time resulted in a decrease in the contact angle, indicating enhanced surface wettability. The observed changes can be attributed to the chemical reactions occurring when titanium is exposed to the acidic HCl/H_2SO_4 environment. These reactions, described in Reactions (1)–(6), promote surface dissolution and restructuring, thereby altering the surface morphology and physicochemical characteristics of the titanium substrate.

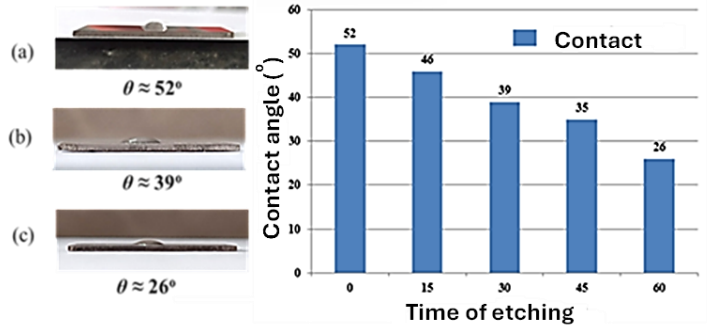


Figure 1. *Contact angle of Ti etched for different times.*

Reaction equation of titanium with H_2SO_4 acid [19]:

$$TiO_2 + 2H_2SO_4 \rightarrow Ti(SO_4)_2 + 2H_2O \quad \text{(Re.1)}$$

$$Ti + 2H_2SO_4 \rightarrow Ti(SO_4)_2 + 2H_2 \quad \text{(Re.2)}$$

$$Ti + H_2 \rightarrow TiH_2 \quad \text{(Re.3)}$$

Reaction equation of titanium with HCl acid [19]:

$$2Ti + 6HCl \rightarrow 2TiCl_3 + 3H_2 \quad \text{(Re.4)}$$

$$TiO_2 + 4HCl \rightarrow TiCl_4 + 2H_2O \quad \text{(Re.5)}$$

$$Ti + H_2 \rightarrow TiH_2 \quad \text{(Re.6)}$$

3.2. Characteristics of HA coating on Ti-etched

Figure 2a shows that acid treatment promotes the formation of the TiH_2 phase. After hydrothermal coating, the diffraction pattern is dominated by the HA phase, with only a small amount of residual TiH_2 remaining. These characteristic peaks, associated with the hexagonal HA structure (space group $P6_3/m$), are observed at $2\theta \approx 26°$ and 32.84°, in good agreement with JCPDS standard No. 09-0432 [20]. The synthesized HA exhibits the anisotropic characteristics typical of a hexagonal crystal structure, consisting of two principal crystallographic planes: the a-plane and the c-plane. Positively charged calcium ions (Ca^{2+}) are predominantly distributed along the a-plane, whereas negatively charged phosphate (PO_4^{3-}) and hydroxide (OH^-) ions are mainly oriented along the c-plane [21]. The FESEM images in Figure 2b show that it doesn't HA crystals were observed after hydrothermal treatment. In contrast, the acid-treated titanium substrate (Figure 2c) displayed a uniform and continuous HA coating. The HA rods exhibit an average diameter of approximately 1 μm and lengths ranging from 4 to 10 μm. The EDS analysis shown in Figure 2d confirms the presence of Ca, P, and O elements without detectable impurities and Ca/P molar ratio was approximately 1.67, which is consistent with stoichiometric hydroxyapatite and agrees well with the precursor composition used under the synthesis conditions.

At hydrothermal temperatures of 180 °C and 200 °C, HA phases were nearly absent and only TiH_2 phase and the titanium substrate. In contrast, when the hydrothermal temperature increased to 220 °C and 240 °C (Figure 2e), the characteristic peaks associated with the acid-etched titanium

substrate disappeared, while distinct diffraction peaks corresponding to crystalline HA were observed, confirming successful coating formation. Figure 2f presents the XRD patterns of HA coatings formed on etched titanium substrates subjected to hydrothermal treatment for durations ranging from 7 to 36 h under constant conditions of 240 °C and pH 10. Crystalline HA pure phases from 12 to 36 h, these substrate-related peaks gradually disappeared, suggesting the formation of a more continuous and uniform HA coating. Based on the XRD analysis during the synthesis process, the hydrothermal temperature range of 220 - 240 °C and treatment time range of 12–36 h were selected for the experimental design and optimization study.

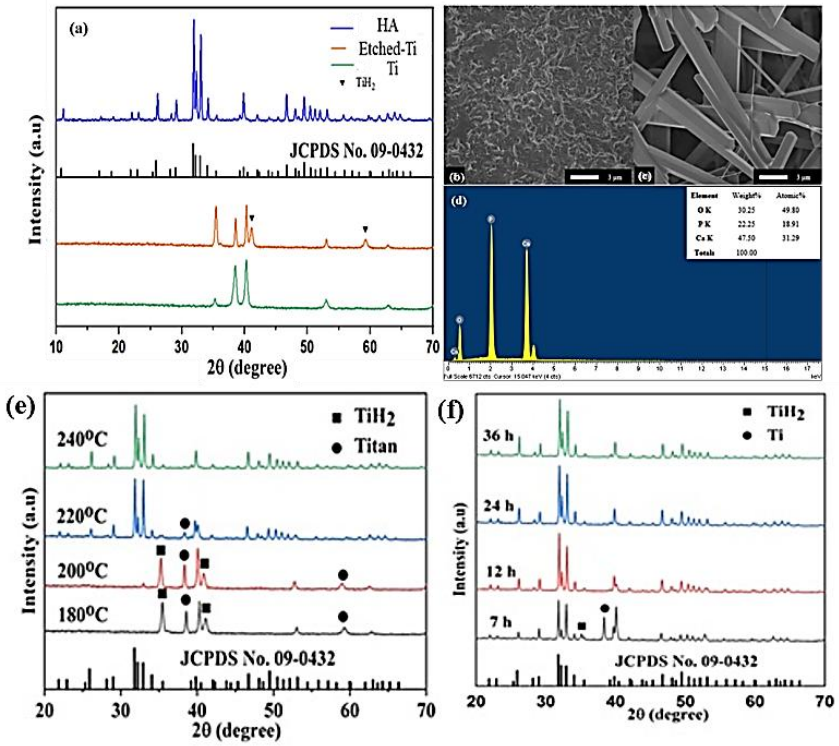


Figure 2. *X-ray of Ti, Ti-etched and HA on Ti-etched (a); FESEM-EDS of HA coating on Ti and Ti-etched prepared by hydrothermal method at 240 °C, 24 h (b,c,d); X-Ray pattern of HA coating on Ti-etched: at different temperatures with 24 h (e); at 240 °C with different times (f).*

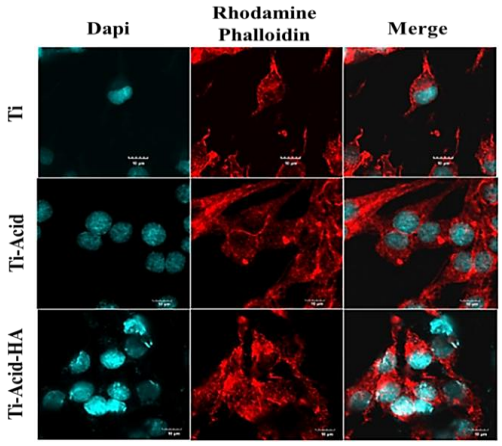


Figure 3. *Confocal laser scanning microscopy (CLSM) images of BHK cell attachment on Ti, Ti-etched, HA coating on Ti-etched after 48 h of incubation.*

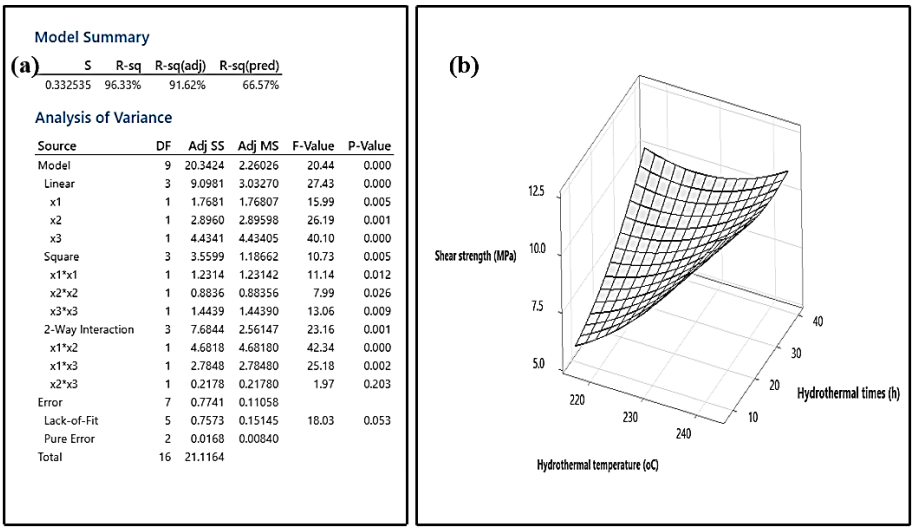
Figure 3 shows confocal laser scanning microscopy (CLSM) images of BHK cell adhesion on both bare titanium substrates and HA-coated titanium surfaces. The coatings were fabricated on titanium substrates treated with an HCl/H_2SO_4 acid mixture at 60 °C for 30 min, followed by hydrothermal synthesis at 240 °C for incubation periods of 48 h. The results indicate that BHK cells exhibited normal fibroblast-like morphology on both surfaces. However, a significantly higher cell density was observed on the HA-coated samples compared with the untreated titanium substrate, demonstrating the enhanced biocompatibility of the HA coating.

3.3. Optimization process

Based on preliminary experiments, key input parameters were selected for the study, including hydrothermal temperature (T_1: 220-240 °C), hydrothermal time (T_2: 12–36 h), and substrate etching time (T_3: 30–60 min). The response variable, Y: Shear strength of HA coating on titanium substrate (MPa). The relationship between real and coded variables: $X_1 = (T_1 - 230)/10$; $X_2 = (T_2 - 24)/12$; $X_3 = (T_3 – 45)/15$. Number of experiments conducted $N = 2^k + n^* + n_0 = 2^k + 2k + n_0 = 17$. With $k = 3$; $2^k = 8$; $2k = 6$; $n^* = 2k = 2.3 = 6$; choose the number of experiments at center 3 (Table 1). The value of the star point is calculated according to the formula: $\alpha = 1.353$ [22]. The experimental domain is constructed through a uniformly rotating second-order orthogonal mixed experimental design (CCD – Box Wilson).

Table 1. *Experimental design conditions according to a Box-Wilson experimental design with three independent variables.*

Run		**Real variable**			**Coded variable**				**Responses**
		T_1 (°C)	T_2 (h)	T_3 (min)	X_0	X_1	X_2	X_3	Y (MPa)
2k	1	220	12	30	+1	+1	+1	+1	2.61
	2	240	12	30	+1	+1	+1	-1	5.06
	3	220	36	30	+1	+1	-1	+1	5.13
	4	240	36	30	+1	+1	-1	-1	3.94
	5	220	12	60	+1	-1	+1	+1	2.71
	6	240	12	60	+1	-1	+1	-1	2.22
	7	220	36	60	+1	-1	-1	+1	5.31
	8	240	36	60	+1	-1	-1	-1	2.34
2k	9	216.5	24	45	+1	-1.353	0	0	4.01
	10	243.5	24	45	+1	+1.353	0	0	2.28
	11	230	7.8	45	+1	0	-1.353	0	2.4
	12	230	40.2	45	+1	0	+1.353	0	3.65
	13	230	24	24.7	+1	0	0	-1.353	4.33
	14	230	24	65.3	+1	0	0	+1.353	2.09
n_0	15	230	24	45	+1	0	0	0	2.5
	16	230	24	45	+1	0	0	0	2.44
	17	230	24	45	+1	0	0	0	2.32



(a)

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
0.332535	96.33%	91.62%	66.57%

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	9	20.3424	2.26026	20.44	0.000
Linear	3	9.0981	3.03270	27.43	0.000
x1	1	1.7681	1.76807	15.99	0.005
x2	1	2.8960	2.89598	26.19	0.001
x3	1	4.4341	4.43405	40.10	0.000
Square	3	3.5599	1.18662	10.73	0.005
x1*x1	1	1.2314	1.23142	11.14	0.012
x2*x2	1	0.8836	0.88356	7.99	0.026
x3*x3	1	1.4439	1.44390	13.06	0.009
2-Way Interaction	3	7.6844	2.56147	23.16	0.001
x1*x2	1	4.6818	4.68180	42.34	0.000
x1*x3	1	2.7848	2.78480	25.18	0.002
x2*x3	1	0.2178	0.21780	1.97	0.203
Error	7	0.7741	0.11058		
Lack-of-Fit	5	0.7573	0.15145	18.03	0.053
Pure Error	2	0.0168	0.00840		
Total	16	21.1164			

Figure 4. *Results for the RSM quadratic model to predict the shear strength (Y) of HA on Ti-etched by the hydrothermal method: (a) Data of the analysis of variance (ANOVA); (b) Response surfaces.*

A quadratic regression model for predicting the shear strength (Y) of HA coatings was developed using Response Surface Methodology (RSM) (Equation 2). Figure 4a shows data of the Analysis of Variance (ANOVA) confirmed the reliability and accuracy of the model, yielding an R^2 value of 96.33%, which indicates excellent agreement between the predicted and experimental results. This high coefficient of determination demonstrates the suitability of the model for predicting process performance. Further statistical analysis revealed that the regression model is highly significant, with an F-value of 20.44 and a corresponding p-value < 0.0001, confirming that the selected processing parameters have a significant influence on the shear strength of the HA coating. Individually, the factors X_1 (hydrothermal temperature), X_2 (hydrothermal processing time), and X_3 (substrate etching time) were identified as dominant variables ($p < 0.05$). These results indicate that the mechanical strength of hydrothermally synthesized HA coatings is strongly dependent on these processing conditions. Regarding interaction effects, the terms X_1X_2 (temperature × hydrothermal time) and X_1X_3 (temperature × etching time) were also statistically significant ($p < 0.05$), suggesting strong interdependence among these variables in determining coating shear strength. In contrast, the interaction term X_2X_3 (hydrothermal time × etching time) exhibited a weaker effect ($p = 0.203$) and was considered statistically insignificant. The quadratic regression equations used to predict the shear strength of HA coatings on titanium substrates are presented below for coded variables (Equation 2) and real variables (Equation 3):

$$Y = 2.394 - 0.389X_1 + 0.498X_2 - 0.617X_3 - 0.765X_1X_2 - 0.59X_1X_3 + 0.429X_1^2 + 0.363X_2^2 + 0.464X_3^2 \quad (2)$$

$$Y = 169.1 - 1.684T_1 + 1.387T_2 + 0.678T_3 - 0.0064T_1T_2 - 0.0039T_1T_3 + 0.0043T_1^2 + 0.0023T_2^2 + 0.0021T_3^2 \quad (3)$$

The hydrothermal synthesis process for fabricating HA coatings on titanium substrates modified with an acid mixture was optimized by identifying the values of the input variables X_1, X_2, and X_3 within the experimental domain of $-1.353 \leq X_1, X_2, X_3 \leq 1.353$ to achieve maximum shear strength (Y). Response Surface Methodology (RSM) was employed for this optimization process. $X = (X_1^{opt}, X_2^{opt}, X_3^{opt}) \in D(X)$ where $D(X) = \{-1.353 \leq X_1, X_2, X_3 \leq 1.353\}$

Figure 4b presents the multi-objective function optimization results. The optimal values for the coded variables were determined as $X_1^{opt} = -1.353$, $X_2^{opt} = 1.353$, and $X_3^{opt} = 1.353$, corresponding

to a hydrothermal temperature (T_1) of 216.5 °C, hydrothermal time (T_2) of 40.2 h, and etching time (T_3) of 65.3 minutes. These conditions yielded a maximum shear strength of Y = 7.52 MPa. The shear strength increased significantly with increasing hydrothermal temperature and prolonged hydrothermal treatment time, indicating a synergistic effect of these parameters on the mechanical performance of the HA coating.

4. CONCLUSIONS

This study investigated the effects of hydrothermal processing parameters on the physical and mechanical properties, particularly the shear strength of HA coatings deposited on surface-treated titanium substrates. The results demonstrated that the maximum shear strength of the HA coating reached 7.52 MPa under the optimal processing conditions of a hydrothermal temperature of 216.5 °C, hydrothermal time of 40.2 h, and substrate etching time of 65.3 min. A Box–Wilson experimental design was employed to develop the following second-order regression equation: $Y = 169.1 - 1.684T_1 + 1.387T_2 + 0.678T_3 - 0.0064T_1T_2 - 0.0039T_1T_3 + 0.0043T_1^2 + 0.0023T_2^2 + 0.0021T_3^2$. This regression model describes the relationship between the hydrothermal processing parameters and the mechanical performance of the HA coating. Using this model, the shear strength of the coating can be predicted with high accuracy under different processing conditions, with deviations between theoretical predictions and experimental results of less than 5%. In addition, the HA coatings synthesized under the optimized conditions exhibited high crystallinity, phase purity, and excellent biocompatibility, as confirmed by X-ray diffraction (XRD), FESEM, and confocal laser scanning microscopy (CLSM) analyses.

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TÓM TẮT

Tối ưu hóa quá trình chế tạo màng phủ hydroxyapatite trên đế Ti đã được ngâm tẩm bằng phương pháp thủy nhiệt ứng dụng cho vật liệu y sinh

Nghiên cứu này sử dụng phương pháp tổng hợp thủy nhiệt để chế tạo lớp phủ HA trên chất nền titan đã qua xử lý bề mặt và kết hợp với mô hình hồi quy trực giao bậc hai cùng với các kỹ thuật tối ưu hóa thống kê, bao gồm phương pháp bề mặt đáp ứng (RSM) và phân tích phương sai (ANOVA) để các định các thông số công nghệ chính. Sự hình thành lớp phủ HA đơn pha trên nền titan với kích thước nano đã được chứng minh thông qua phân tích X-Ray, FESEM-EDS, và đặc biệt là khả năng tương thích sinh học cao. Vật liệu lớp phủ HA tiếp tục được thử nghiệm in vitro trên tế bào thận chuột (BHK) với khả năng tăng sinh tốt và quan sát chi tiết thông qua phân tích laser hội tụ confocal (CLMS). Kết quả RSM đã xác định các điều kiện tối ưu như sau: nhiệt độ thủy nhiệt (T_1) là 216.5 °C, thời gian thủy nhiệt (T_2) là 40,2 giờ và thời gian xử lý chất nền titan trong hỗn hợp axit HCl/H_2SO_4 ở 60 °C (T_3) là 65,3 phút. Trong điều kiện tối ưu này, lớp phủ HA thể hiện độ bền xé tối đa là 7,52 MPa. Lớp phủ HA trên nền đế titan đã được xử lý này hứa hẹn tiềm năng trong các ứng dụng y sinh đặc biệt các ứng dụng trong y học quân sự về các loại vật liệu cấy ghép xương hoặc các chi tiết thay thế giả.

Từ khoá: Màng phủ hydroxyapatite; Màng phủ HA; Phương pháp thủy nhiệt; DOE; Box-Wilson .