Jurnal Crystal: Publikasi Penelitian Kimia dan Terapannya
Vol. 07, No. 02, Page. 209 – 220, 2025
e-ISSN 2685-7065

ADHESIVE BEHAVIOR OF POLYCAPROLACTONE/HYDROXYAPATITE COATINGS ON 316L STAINLESS STEEL: A DESIGN OF EXPERIMENTS APPROACH

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Article History
Received: 8 September 2025; Received in Revision: 17 September 2025; Accepted: 19 September 2025

Abstract

Enhancing the adhesive strength of bioactive coatings is crucial for improving the mechanical stability of metallic implants. This study investigates the effects of three processing parameters—sonication temperature (X_1), PCL/HA ratio (X_2), and drying time (X_3)—on the adhesive strength of poly(ε-caprolactone)/hydroxyapatite (PCL/HA) composite coatings applied to 316L stainless steel substrates. A full factorial 2^3 experimental design was employed, and the results were analyzed using analysis of variance (ANOVA) and regression modeling. The adhesive strength response ranged from 19.62 MPa to 63.27 MPa. Among the factors studied, the PCL/HA ratio had the most significant positive effect, while drying time showed a minor influence. Interaction plots and response surface analyses revealed a synergistic effect between sonication temperature and PCL/HA ratio, contributing to improved bonding at the coating-substrate interface. The optimization results yielded a predicted maximum adhesive strength of 25.76 MPa at a desirability score of 0.03, highlighting the complexity of parameter interactions. These findings underscore the importance of processing conditions in tailoring coating performance for biomedical applications.

Keywords: PCL/HA composite coatings, Adhesive strength, 316L stainless steel, Factorial design, Optimization.

Abstrak

Meningkatkan kekuatan adhesi lapisan bioaktif sangat penting untuk memperbaiki stabilitas mekanik pada implan logam. Penelitian ini menyelidiki pengaruh tiga parameter proses—suhu sonikasi (X_1), rasio PCL/HA (X_2), dan waktu pengeringan (X_3)—terhadap kekuatan adhesi lapisan komposit poli(ε-kaprolakton)/hidroksiapatit (PCL/HA) yang diaplikasikan pada substrat baja tahan karat 316L. Desain eksperimen faktorial penuh 2^3 digunakan, dan hasilnya dianalisis menggunakan analisis varians (ANOVA) serta pemodelan regresi. Respon kekuatan adhesi berkisar antara 19,62 MPa hingga 63,27 MPa. Di antara faktor-faktor yang diteliti, rasio PCL/HA memberikan pengaruh positif paling signifikan, sedangkan waktu pengeringan menunjukkan pengaruh yang relatif kecil. Plot interaksi dan analisis permukaan respon menunjukkan adanya efek sinergis antara suhu sonikasi dan rasio PCL/HA, yang berkontribusi pada peningkatan ikatan pada antarmuka lapisan-substrat. Hasil optimasi menghasilkan kekuatan adhesi maksimum prediksi sebesar 25,76 MPa dengan skor desirabilitas 0,03, yang menyoroti kompleksitas interaksi parameter. Temuan ini menegaskan pentingnya kondisi proses dalam menyesuaikan kinerja lapisan untuk aplikasi biomedis.

Keywords: Lapisan komposit PCL/HA, Kekuatan adhesi, Baja tahan karat 316L, Desain faktorial, Optimasi.

1. Introduction

316L stainless steel (SS 316L) is a prominent candidate in the field of temporary bone implant materials due to its favorable attributes, including corrosion resistance, mechanical strength, and cost-effectiveness (Haleem et al., 2024; Aroussi et al., 2019). Within the broader classification of metallic biomaterials, such as cobalt-chromium alloys and titanium-based constructs, SS 316L is

noted for its excellent mechanical properties, including high fatigue resistance, which are critical for orthopedic applications (Haleem et al., 2024; Aroussi et al., 2019). However, the biologically inert nature of SS 316L limits its long-term success due to inferior osseointegration, which can jeopardize implant durability (Fadli, 2021; Luo et al., 2018). Such limitations necessitate the exploration of bioactive coatings aimed at enhancing the material's interaction with bone tissue. Hydroxyapatite (HA) coatings emerge as a leading solution, given HA's chemical and structural resemblance to natural bone mineral (Singh et al., 2023; Ielo et al., 2022). HA fosters osteoblast adhesion and proliferation and can enhance the corrosion resistance of the underlying metallic substrate, potentially improving the implant's integration in vivo (Homa et al., 2024; Ielo et al., 2022). The structural properties of HA reveal a hexagonal lattice with parameters a = 9.432 Å and c = 6.881 Å, giving it specific characteristics beneficial for biological applications (Vasudev & Prakash, 2023). Despite the promising bioactivity attributed to HA, it has limitations; its mechanical brittleness and insufficient tensile strength can hinder its utility in weight-bearing orthopedic implants (Saputra et al., 2021; Ielo et al., 2022).

To overcome the mechanical limitations of hydroxyapatite (HA) coatings, considerable research has focused on developing composite systems that incorporate polymers to improve both structural integrity and long-term performance (Ramesh et al., 2018). Among the various candidates, poly(ε-caprolactone) ($C_6H_{10}O_2$) (PCL) has garnered significant attention due to its favorable mechanical properties, excellent biocompatibility, and biodegradability (Taghizadeh et al., 2024; Ulery et al., 2011; Sowmya et al., 2021). PCL is a semi-crystalline aliphatic polyester that undergoes slow hydrolytic degradation under physiological conditions, yielding non-toxic by-products that can be readily metabolized or excreted by the body. These characteristics make PCL highly suitable for biomedical applications such as drug delivery, tissue engineering scaffolds, and implant coatings (Gunatillake & Adhikari, 2003).

However, PCL on its own lacks the osteoconductivity and cellular adhesion properties essential for orthopedic coatings (Liang et al., 2024). To address this, the combination of PCL with HA in a polymer–ceramic composite offers a promising approach—providing the mechanical flexibility and processability of the polymer while preserving the bioactivity of the ceramic component (Monia & Ridhal., 2024). In this study, PCL/HA composite coatings were synthesized and applied to 316L stainless steel (SS 316L) substrates using a dip-coating technique. This low-temperature process eliminates the need for sintering, thereby preserving the mechanical integrity of the underlying metal while enabling uniform coating deposition.

The primary aim of this work was to evaluate the effect of PCL concentration and ultrasonic processing temperature on the mechanical adhesion strength of the composite coating. A two-level full factorial design (2k) was implemented to assess the influence and interaction of processing variables on coating performance. Shear strength testing was conducted to quantify the adhesion between the composite layer and the metallic substrate, serving as a direct measure of mechanical stability. Analysis of Variance (ANOVA) was employed to identify the most statistically significant factors affecting coating adhesion. The results highlight the critical role of optimizing both the polymer–ceramic ratio and the processing conditions to achieve coatings with enhanced interfacial adhesion and mechanical robustness. These improvements position the optimized PCL/HA composite coatings as strong candidates for orthopedic applications requiring durable, load-bearing implant surfaces.

2.Methodology

Materials

Stainless steel 316L plates (30 × 20 × 3 mm) were used as substrates. The surfaces were polished with 1200 grit SiC paper, then cleaned in acetone (Merck) using an ultrasonic bath for 15 minutes, dried, and stored in a desiccator. For pretreatment, the substrates were immersed in 5 M sodium hydroxide (NaOH, Merck) at 60°C for 24 hours, dried at 80°C for 1 hour, and sintered at 600°C for 1 hour. Poly(ε-caprolactone) (PCL) pellets and hydroxyapatite (HA) powder were purchased from Sigma-Aldrich (UK) and used without further purification. Acetone (Merck) was used as the solvent for slurry preparation.

Pretreatment of SS 316L

A stainless steel 316L (SS 316L) plate with dimensions of 30 × 20 × 3 mm was employed as the metallic substrate. Prior to coating deposition, the substrates were mechanically polished using silicon carbide (SiC) abrasive paper with a grit size of 1200 to remove surface contaminants and enhance surface roughness for improved coating adhesion. Following mechanical treatment, the samples were ultrasonically cleaned in acetone for 15 minutes to eliminate residual impurities,

then dried and stored in a desiccator to prevent surface oxidation. To further investigate the influence of ultrasonic processing temperature and alkali pretreatment on the surface characteristics of SS 316L, an additional surface modification step was introduced. The polished substrates were immersed in 50 mL of 5 M NaOH aqueous solution and maintained at 60°C for 24 hours to induce chemical surface activation. After alkali treatment, the samples were dried using warm airflow at 80°C for 1 hour, followed by a sintering process at 600°C for 1 hour. This thermal treatment aimed to stabilize the modified surface morphology and enhance the interfacial adhesion properties of the subsequent coating.

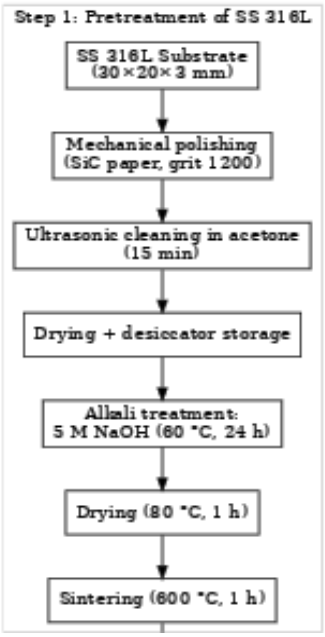

Figure 1. Flowchart of pretreatment procedure of SS 316L Substrate

Preparation of PCL/HA Slurry and Coating Deposition on SS 316L

Poly(ε-caprolactone) (PCL) pellets and hydroxyapatite (HA) powder, both obtained from Sigma-Aldrich (England), were used as received without further purification. To prepare the composite coating solution, PCL pellets (20–40 wt.%) were dissolved in acetone at room temperature under continuous magnetic stirring. Once the PCL was fully dissolved, HA powder was gradually introduced into the solution and stirred for 24 hours at room temperature to ensure homogeneous dispersion of HA particles within the PCL matrix. The coating process was conducted using a dip-coating technique at room temperature. SS 316L substrates were vertically immersed in the PCL/HA suspension and withdrawn at a constant speed to achieve uniform film deposition. Following coating, the samples were dried in an oven at 60 °C for either 10 or 14 hours to evaluate the effect of drying duration on the mechanical and electrochemical properties of the composite coatings.

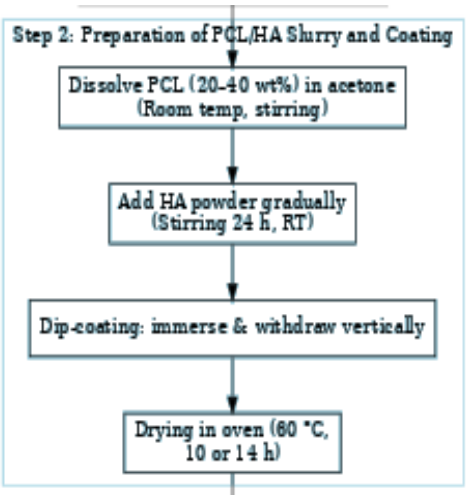

Figure 2. Flowchart of preparation procedure of PCL/HA slurry and coating

Characterization Techniques

The adhesive strength of the PCL/HA composite coating on SS 316L substrates was evaluated using a universal testing machine (Instron 5569). Prior to testing, the uncoated side of each specimen was mechanically roughened by sandblasting to ensure consistent surface texture and promote effective bonding. This surface was then bonded to an aluminum rod using a triethanolamine-based epoxy resin [$N(CH_2CH_2OH)_3$]. After the initial bonding, the samples underwent thermal curing in a drying oven at 110 °C for 24 hours. To ensure uniform stress distribution during testing, the coated surface of each specimen was similarly bonded to a flat aluminum plate using the same adhesive and curing protocol. The assembled specimens were mounted on the testing apparatus, and tensile shear tests were conducted at a constant crosshead speed of 1 mm/min. The test was continued until coating delamination or failure occurred. The maximum load recorded prior to failure was used to calculate the shear adhesive strength of the PCL/HA composite coating.

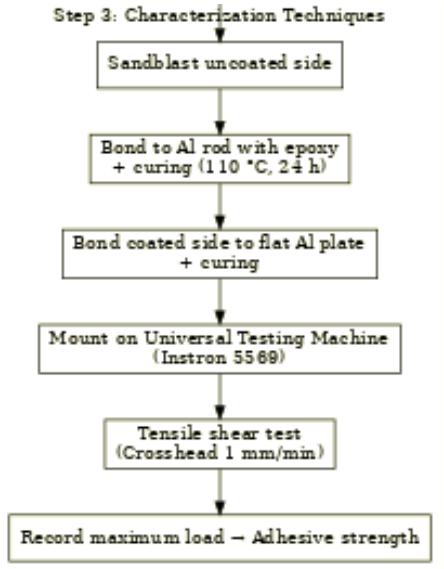

Figure 3. Flowchart of characterization procedure for adhesive strength test

Experimental Design

To systematically investigate the influence of key processing parameters on the properties of PCL/HA composite coatings, a full factorial design of experiments (2^k) was utilized. This statistical approach allowed for the evaluation of both main effects and interaction effects among the selected variables. The independent variables considered in this study were: PCL concentration (20 wt.% and 40 wt.%), ultrasonic treatment temperature (45 °C and 55 °C), and drying time (10 hours and 14 hours). These factors were tested at two levels—low and high—as summarized in Table 1. The factorial design provided a structured and efficient framework for analyzing how these parameters influence the mechanical and electrochemical performance of the coatings, particularly with respect to shear bond strength. Through systematic variation of the input variables, the study aimed to determine the optimal processing conditions to enhance coating adhesion and stability.

Table 1. Factors and their levels

Levels	Sonication Temperature (°C)	PCL/HA Ratio (%wt)	Substrate and composite drying time (hours)
High (1)	55	40	14
Low (-1)	45	20	10

In this design, 8 factorial points performance test re-conducted. Table 2 shows the experimental plan and the obtained results for this design.

Table 2. Experimental plan and results for 2^k factorial design.

No.	Sonication Temperature (°C)	PCL/HA Rasio (%wt)	Substrate and composite drying time (hours)	Coded Variables			Response
				X_1	X_2	X_3	Adhesive strength (MPa)
1	45	20	10	-1	-1	-1	10.61
2	45	20	14	-1	-1	1	1.73
3	45	40	10	-1	1	-1	42.15
4	45	40	14	-1	1	1	15.75
5	55	20	10	1	-1	-1	11.01
6	55	20	14	1	-1	1	33.88
7	55	40	10	1	1	-1	29.59
8	55	40	14	1	1	1	61.36

3. Results and Discussion

The successful deposition of poly(ε-caprolactone)/hydroxyapatite (PCL/HA) composite coatings onto SS 316L substrates was confirmed through a combination of morphological, structural, and phase characterization techniques. Initial visual inspection revealed smooth, continuous coatings without visible defects, indicating uniform film formation across the substrate surface. This uniformity is attributed to the synergistic effects of sonication-assisted dispersion, optimized PCL/HA ratio, and controlled drying time, which together enhanced coating homogeneity and interfacial adhesion. As illustrated in Figure 4, the adhesion mechanism of the PCL/HA composite coating to the SS 316L surface involves both chemical and electrostatic interactions. The naturally occurring passive oxide layer on stainless steel introduces surface hydroxyl (–OH) groups, which facilitate hydrogen bonding with the carbonyl (C=O) groups of the PCL polymer chains (Hetemi & Pinson, 2019). Concurrently, hydroxyapatite particles are embedded within the polymer matrix and anchored to the substrate via electrostatic interactions. Specifically, calcium ions (Ca^{2+}) and phosphate groups (PO_4^{3-}) participate in ionic bonding with surface species on the oxide layer, contributing to the mechanical stability and bioactivity of the coating (Drevet et al., 2019).

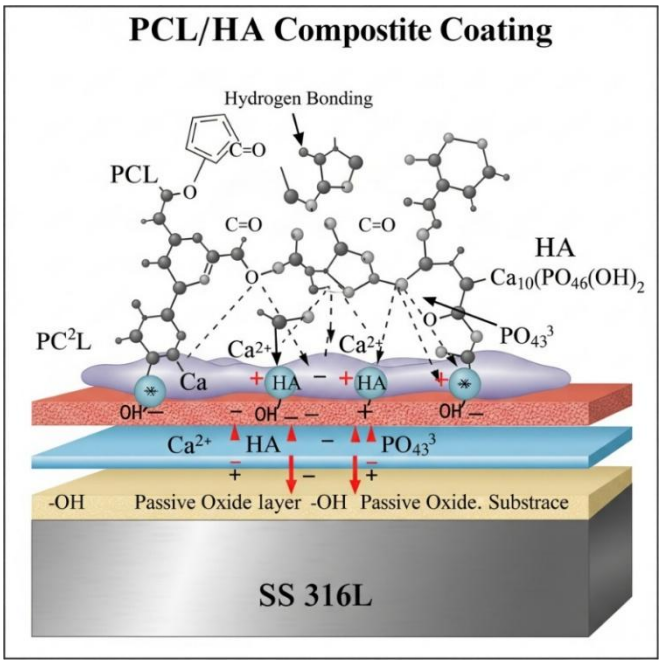

Figure 4. Schematic illustration of the interfacial interactions between the PCL/HA composite coating and the SS 316L substrate. The passive oxide layer on stainless steel promotes hydrogen bonding with PCL chains, while HA particles are immobilized via electrostatic interactions involving Ca^{2+} and phosphate (PO_4^{3-}) groups.

Ultrasonic treatment during the preparation of the PCL/HA composite coating plays a critical role in enhancing the dispersion of hydroxyapatite particles within the polymer matrix. The high-frequency cavitation effect effectively breaks up particle agglomerates, ensuring a more uniform suspension and facilitating deeper infiltration of the PCL into the microgrooves of the SS 316L substrate surface (Fadli et al., 2023). This improved dispersion and penetration strengthen the mechanical interlocking at the coating–substrate interface, thereby increasing shear resistance. In addition, controlled drying conditions contribute to effective solvent evaporation and consolidation of the polymer matrix. This promotes densification of the coating layer, minimizes porosity, and reinforces the structural integrity of the composite film (Tirumkudulu & Punati, 2022). The interfacial adhesion is further supported by multiple bonding mechanisms, including hydrogen bonding, electrostatic interactions, and physical anchoring. These synergistic interactions collectively result in a robust and adherent coating structure. Optimizing these processing parameters significantly enhances not only the surface morphology but also the mechanical stability of the coating under physiological and load-bearing conditions (Ma et al., 2022). As a result, the PCL/HA composite coatings exhibit strong potential for orthopedic applications where durable implant–tissue integration and long-term interfacial integrity are essential (Shamsi et al., 2024).

To evaluate the adequacy of the model developed for predicting adhesive strength, a regression analysis was conducted using JMP Pro 14 software (SAS Institute Inc., 2018). As illustrated in Figure 5, the regression plot compares the actual adhesive strength values against the predicted values generated by the model. The observed points closely follow the diagonal line, indicating strong agreement between experimental and predicted data. The model achieved a high coefficient of determination (R^2) of 0.97, signifying that 97% of the variation in adhesive strength can be explained by the selected process parameters. Additionally, the Root Mean Square Error (RMSE) was 9.34 MPa, suggesting minimal deviation between predicted and observed responses. These results demonstrate that the linear model provides a satisfactory fit to the experimental data. Although a formal lack of fit test could not be performed due to the absence of replicate data, the strong R^2 and low RMSE values indicate that the model is adequate for predicting adhesive strength within the studied range. Future work may include replicate runs or center points to enable a statistical lack-of-fit test and assess potential curvature or higher-order interactions.

The regression plot reveals a strong alignment between the actual and predicted values of adhesive strength, closely following a linear trend line. This visual correlation confirms that the model effectively captures the relationship between the process parameters and the adhesive strength response. The proximity of the data points to the line of equality (Y actual = Y predicted) indicates minimal deviation and affirms the model's predictive reliability. A key metric supporting this observation is the coefficient of determination (R^2), which quantifies the proportion of variance in adhesive strength that can be explained by the independent variables: sonication temperature, PCL/HA ratio, and drying time. With an R^2 value approaching 1, the model demonstrates a high level of explanatory power, indicating that the selected variables significantly influence the adhesive strength. The high R^2 value obtained in this analysis confirms the model's adequacy in representing the experimental data and highlights its potential for guiding process optimization.

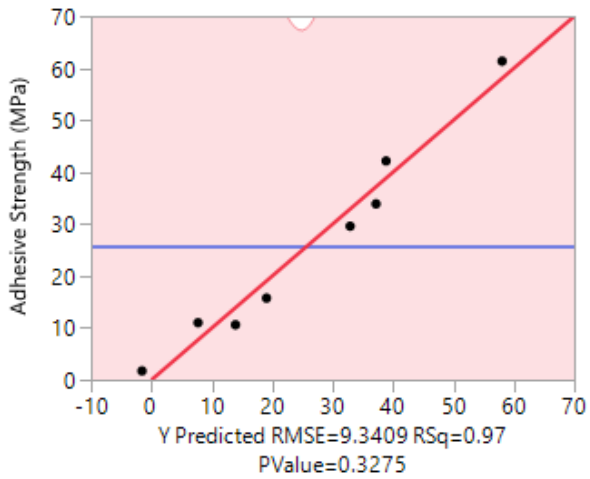

Figure 5. Regression plot of the predicted versus actual adhesive strength values.

The model shows a strong fit to the data with an R^2 of 0.97, meaning most of the variation in adhesive strength is explained by the chosen variables (sonication temperature, PCL/HA ratio, and drying time). However, the lower adjusted R^2 of 0.77 indicates that some predictors may be less important and that the model could be overfitted. The 0.20 gap between R^2 and adjusted R^2 suggests the need for caution in interpreting the results. The RMSE of 9.34 MPa shows moderate prediction errors, while the p-value of 0.3275 indicates limited statistical significance, partly due to the lack of replicate experiments. Overall, the model fits well but requires further validation or simplification, ideally through additional experiments with replicates or center points, to confirm its robustness.

Table 3. Fit Statistic summary

R^2	0.968	
R^2 Adj	0.777	
Root Mean Square Error	9.34	
Mean of Response	25.76	3682
p-Value	0.3275	

A linear model with interaction terms was developed using a full factorial 2^3 design and evaluated by ANOVA (Table 4). The overall model was not statistically significant ($p = 0.3275$), and none of the main factors or interactions showed significance ($p > 0.05$). Although the model yielded a high R^2 of 0.97, the lower adjusted R^2 of 0.77 indicates possible overfitting, likely due to the limited sample size. The lack of replicates also prevented a formal lack-of-fit test and accurate estimation of experimental error. Despite these limitations, the observed trends are consistent with expected experimental behavior. Further studies with replicate runs or center points are recommended to strengthen the statistical reliability and validate the model, in line with recommendations by Fern and Salimi (2021).

Table 4. ANOVA Summary for the Linear Interaction Model of Adhesive Strength

Source	***Sum of Squares***	***df***	***Mean Square***	***F-value***	***p-value***
Model	**2654.06**	**6**	**442.34**	**5.06**	**0.3275**
X1	537.9200	1		6.1651	0.2437
X2	1049.2781	1		12.0258	0.1787
X3	46.8512	1		0.5370	0.5974
X1*X2	0.0313	1		0.0004	0.9880
X1*X3	1010.7008	1		11.5837	0.1819
X2*X3	9.2880	1		0.1065	0.7992
Error	**87.25**	**1**	**87.25**		
Cor Total	**2741.31**	**7**			

The ANOVA results (Table 4) show that the overall regression model for adhesive strength was not statistically significant ($F = 5.06$, $p = 0.3275$), and none of the main factors (sonication temperature, PCL/HA ratio, drying time) or their interactions reached significance ($p > 0.05$). Although the model achieved a high R^2 of 0.97, the lower adjusted R^2 of 0.77 suggests possible overfitting and limited predictive reliability. The use of coded variables (−1, 0, +1) allowed relative comparison of factor effects, but additional replicates or an expanded design space are needed to improve robustness and confirm factor significance.

The developed linear model with interaction terms predicts adhesive strength as a function of the selected factors. The coded regression coefficients indicate the strength and direction of each variable's effect, helping to identify key parameters and guide optimization. The full coded model is presented in Equation (1).

$$Y=25.76+8.2X_1+11.4525X_2+2.42X_3+0.0625X_1X_2+11.24X_1X_3-1.0775X_2X_3 \quad (1)$$

Regression analysis was used to model the effects of sonication temperature (X_1), PCL/HA ratio (X_2), and drying time (X_3) on adhesive strength. Residual analysis confirmed that the model adequately captured the data trend without major bias. The regression revealed both main and interaction effects: the X_1X_3 interaction showed a strong positive coefficient (+11.24), indicating a synergistic effect, while the X_2X_3 interaction (−1.0775) suggested a slight antagonistic effect. Interaction and 3D response surface plots further illustrated these relationships, helping to visualize factor interplay, identify optimal conditions, and support model validation.

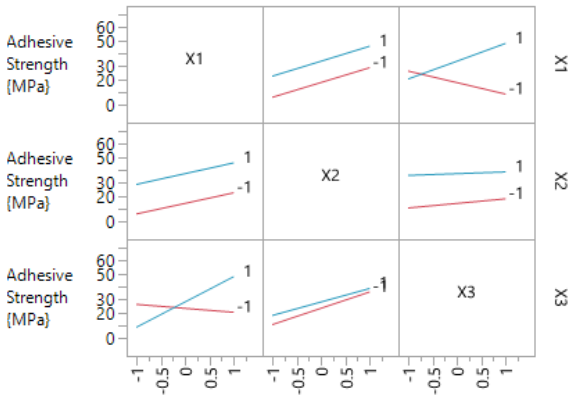

Figure 6. Interaction Plot for adhesive strength as a Function of X_1, X_2, and X_3

Figure 6 shows interaction plots for sonication temperature (X_1), PCL/HA ratio (X_2), and drying time (X_3) on adhesive strength. A strong positive interaction occurs between X_1 and X_2, where higher sonication temperatures and optimized PCL/HA ratios significantly enhance adhesion, likely due to better HA dispersion and polymer infiltration. The X_1–X_3 interaction also improves adhesion at higher levels, reflecting stronger matrix densification and moisture removal. In contrast, the X_2–X_3 interaction is weak, suggesting mainly additive rather than synergistic effects. Overall, the plots highlight that optimizing parameter combinations is more effective than adjusting single factors in achieving high adhesive strength.

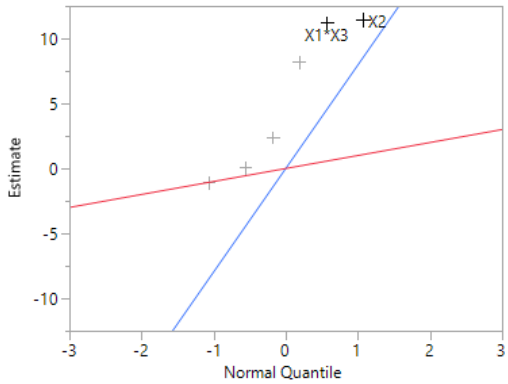

Figure 7. Normal probability plot of standardized effects for adhesive strength. Effects that deviate significantly from the reference line indicate significant contributions to the response.

Figure 7 shows the normal probability plot of standardized effects for adhesive strength. The results indicate that the PCL/HA ratio (X_2) and the interaction between sonication temperature and drying time ($X_1 \cdot X_3$) are statistically significant, lying farthest from the reference line. This highlights the critical role of PCL/HA composition and the synergistic influence of processing conditions on coating adhesion. Other effects lie close to the line, suggesting minimal contribution within the studied range.

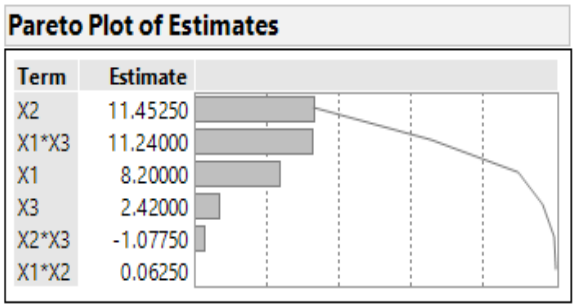

Figure 8. Pareto plot of estimates showing the relative contribution of each factor and interaction term to the adhesive strength of PCL/HA coatings.

Figure 8 presents the Pareto plot of standardized estimates, illustrating the relative importance of main factors and their interactions on the adhesive strength of PCL/HA coatings. The PCL/HA ratio (X_2) emerges as the most influential parameter (11.45 MPa), underscoring its critical role in enhancing coating–substrate bonding. The interaction between sonication temperature and drying time ($X_1 \cdot X_3$) also exerts a strong positive effect (11.24 MPa), suggesting a synergistic improvement in nanoparticle dispersion and film consolidation. Sonication temperature (X_1) contributes significantly (8.2 MPa), while drying time (X_3) exerts a moderate effect (2.42 MPa). In contrast, the $X_2 \cdot X_3$ interaction shows a minor negative influence (−1.07 MPa), and $X_1 \cdot X_2$ appears negligible (0.06 MPa). Overall, the plot confirms that X_2 and $X_1 \cdot X_3$ are the dominant factors governing adhesive strength.

The three-dimensional response surface plots presented in Figures 9 illustrate the interaction effects of process parameters—sonication temperature (X_1), PCL/HA ratio (X_2), and drying time (X_3)—on the adhesive strength of PCL/HA composite coatings applied to 316L stainless steel substrates. These plots help visualize the combined influence of two variables at a time, while holding the third variable at its central level, thus allowing for interpretation of interaction effects critical to coating adhesion performance.As shown in Figure 9a, the interaction between sonication temperature (X_1) and PCL/HA ratio (X_2) has a significant influence on the adhesive strength. The surface exhibits a rising gradient from lower-left to upper-right, forming a planar and positively sloped surface. This trend indicates a synergistic effect where increasing both parameters simultaneously leads to a noticeable improvement in adhesive strength. The consistent elevation suggests that these variables contribute additively rather than interactively in a nonlinear manner. Higher sonication temperatures may enhance particle dispersion and interfacial bonding, while an increased PCL/HA ratio likely promotes better matrix continuity—both contributing to enhanced adhesion.

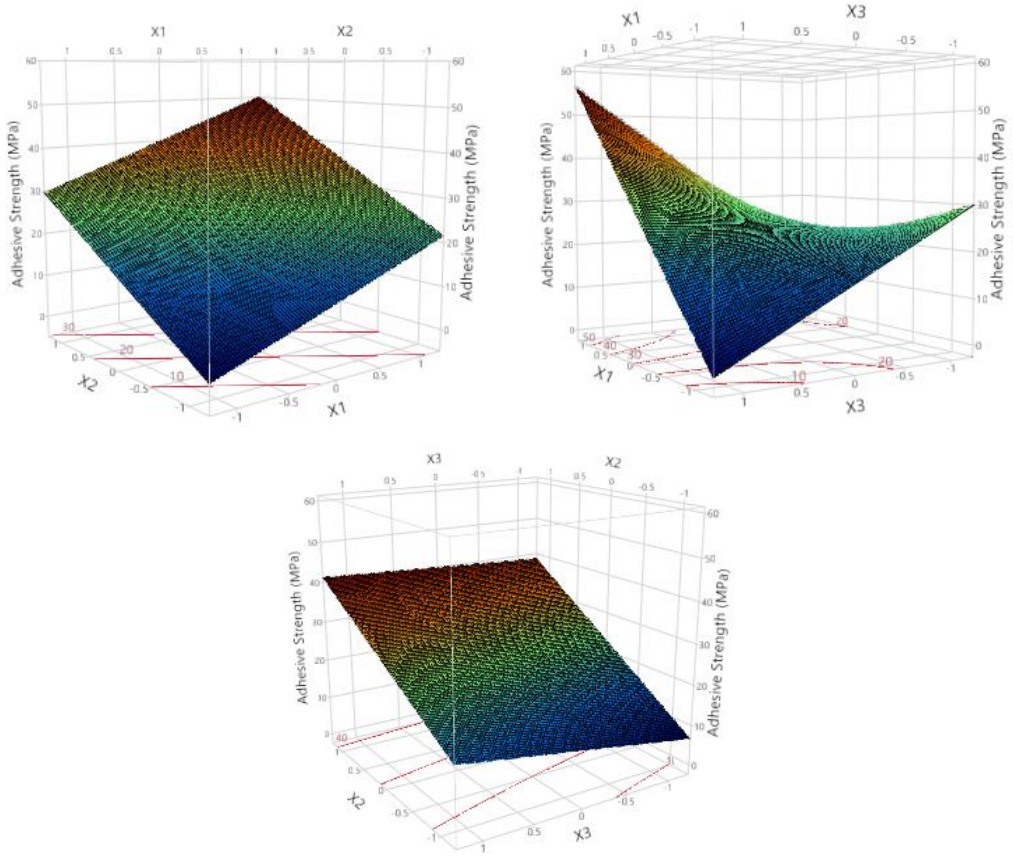

Figure 9. Three-dimensional response surface plots illustrating the interaction effects of (a) sonication temperature and PCL/HA ratio, (b) sonication temperature and drying time, and (c) PCL/HA ratio and drying time on the adhesive strength of PCL/HA-coated stainless steel.

Figure 9b presents the interaction between sonication temperature (X_1) and drying time (X_3). This surface displays a non-linear, saddle-like curvature with a peak in adhesive strength observed when the sonication temperature is high and the drying time is low. Conversely, low temperature combined with prolonged drying yields reduced adhesive strength. This interaction suggests that while moderate to high sonication temperature improves nanoparticle dispersion and matrix

infiltration, extended drying time may reduce bonding efficacy due to potential microcracking or shrinkage stress during solvent evaporation. Therefore, the optimal adhesion is achieved through a precise balance between sufficient particle dispersion and controlled drying kinetics. In Figure 9c, the interaction between PCL/HA ratio (X_2) and drying time (X_3) shows a moderately curved surface, with adhesive strength decreasing as both variables increase. The surface trend indicates a negative synergistic effect, where increasing either parameter beyond a threshold leads to a reduction in adhesion performance. This could be due to a higher PCL content forming a less rigid matrix that reduces interfacial grip, and longer drying times potentially leading to brittle or poorly consolidated structures. Hence, a careful balance between polymer concentration and drying duration is essential for maximizing adhesive performance. Taken together, the response surface analysis highlights that the adhesive strength of PCL/HA coatings is strongly influenced by both individual parameters and their interactions. While sonication temperature and PCL/HA ratio exhibit synergistic enhancement, the drying time must be carefully controlled to avoid undermining the adhesion benefits gained from the other two variables.

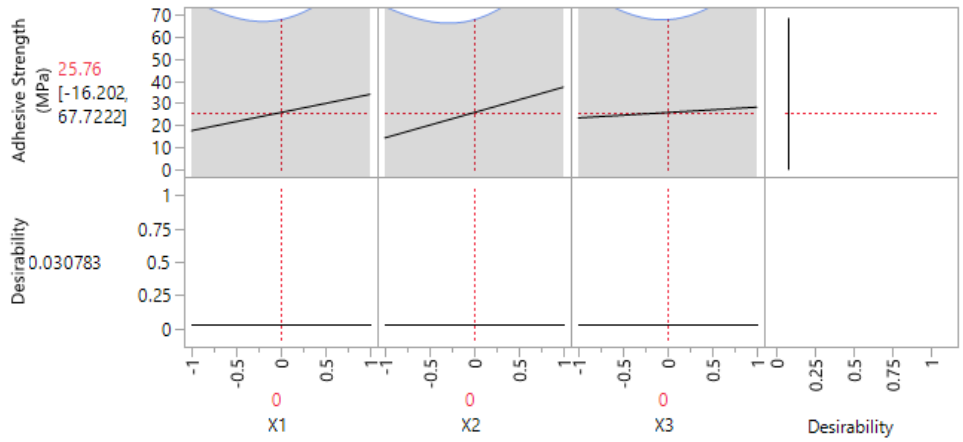

Figure 10. Desirability profiler showing the effects of sonication temperature (X_1), PCL/HA ratio (X_2), and drying time (X_3) on predicted adhesive strength and overall desirability.

Figure 10 presents the desirability profiler illustrating the effects of sonication temperature (X_1), PCL/HA ratio (X_2), and drying time (X_3) on the predicted adhesive strength of the composite coating and its associated desirability function. The upper panel shows the predicted adhesive strength (MPa) with 95% confidence intervals, while the lower panel depicts the desirability scale (0 = least desirable, 1 = most desirable). Increasing X_1 enhances adhesive strength, likely due to improved particle dispersion and polymer infiltration, whereas X_2 shows a positive trend attributed to better polymer flexibility and cohesive bonding at higher PCL content. In contrast, X_3 exerts a negative effect, suggesting that extended drying promotes brittleness, shrinkage, or microcrack formation that weakens adhesion. Despite these trends, the overall desirability score is extremely low (0.03), with an optimized adhesive strength of only 25.76 MPa. This outcome indicates that the current design space is insufficient to achieve the targeted performance, highlighting the need for parameter expansion (e.g., higher sonication temperatures, alternative drying strategies, or post-curing treatments). Consequently, while the profiler provides valuable insights into factor trends, further refinement of the process window is essential to achieve clinically relevant adhesion levels for biomedical applications.

4. Conclusion

This study successfully demonstrated the influence of three key process variables—sonication temperature, PCL/HA ratio, and drying time—on the adhesive strength of PCL/HA composite coatings applied to 316L stainless steel via the dip-coating method. Statistical modeling and response surface analysis revealed that sonication temperature and PCL/HA ratio had the most significant positive impact on the bonding performance, enhancing the interfacial adhesion between the coating and the metal substrate. In contrast, extended drying time had a detrimental effect, likely due to film shrinkage, microcracking, or reduced interfacial cohesion. Interaction plots and Pareto analysis supported the dominance of PCL/HA ratio and sonication temperature in determining adhesive strength outcomes. The optimization model predicted a maximum adhesive strength of 25.76 MPa, albeit with a low desirability score (0.03), indicating the challenge in fine-tuning all parameters to achieve optimal bonding performance. These findings highlight the importance of controlling process conditions to enhance coating adhesion, offering valuable

insights for the development of mechanically robust bioactive coatings for biomedical implant applications.

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