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Response Surface Methodology Optimization of Concrete Strength Using Hydroxyapatite Nanopowder As Admixture

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Abstract: Conventional method that utilize chemical admixture in concrete is not sustainable to the environment. Nowadays, the use of natural hydroxyapatite (HAp) as admixture in concrete is getting attention. This study aims to optimize the compressive and tensile strength of concrete at various HAp dose and time by using response surface methodology (RSM). The modelling and statistical analyses was conducted via historical data design based on experimental data obtained in previous study. The polynomial regression equation was found to provide the best fit for the model, with a coefficient of regression of 0.99. The analysis of variance indicate that the models were significant. The actual and predicted values plots of compressive and tensile strength shows good correlation. In addition, the individual and synergistic effects of the HAp dose and time on the compressive and tensile strength of concrete were analysed using 3D surface plot. The results obtained indicate that 21.0 % HAp dose and 28 days are the optimum working parameters to achieve a maximum compressive and tensile strength of 48.90 and 8.10 MPa, respectively. It was observed that the model prediction values accurately matched the experimental values for tensile strength and within 1.0 % matched for compressive strength. The results of this study demonstrate that RSM is a robust tool that can used for process optimization of concrete performances.

Keywords: Admixture, concrete, HAp, response surface methodology, historical data

1. Introduction

Generally, chemical admixtures are usually added as liquids or powders in relatively small quantities and may be used to modify the properties during the plastic or hardened state of concrete [1]. Chemical admixtures can be divided into five types which are accelerating, retarding, water

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reducing/plasticizing, air entraining, and water proofers. All of these admixture has its own function [2].

Chemical admixtures should be used carefully because the addition of wrong quantities can affect the long-term performance of concrete in many ways. Hence, some of the chemical admixture can contribute to pollution. For example, Air-entraining admixtures function by incorporating air into concrete to provide resistance to damage from freeze–thaw cycles and to improve workability. These materials can include various types of inorganic salts (salts of wood resins and salts of sulphonated lignin). Fungi cides, germicides, and insecticides are also added to some concretes. Because of these chemical admixtures, the concrete could conceivably release gas that contains small quantities of formaldehydes and other chemicals into the indoor air [3].

Apart from chemical, admixture can also be produced from natural sources and waste products. Fish waste are municipal waste that is produce from the fish processing industry and wet market. It had contributed to high percentage of waste in Malaysia. According from the [4], municipal waste had the highest percentage of waste produce compare than other waste. This situation is alarming to the public, as drastic increase of uncontrolled waste will lead to inadequate landfill space for waste disposal areas [5].

Effort towards preserving the environment for future generation and reducing tilapia fish scales waste from fisheries industry has initiated the study on possibility of integrating this waste in concrete production. The synthetization of this fish scales able to produce Hydroxyapatite (HAp) nanopowder. HAp nanopowder is suitable to be used as admixture in concrete because of the chemical properties in HAp that act as binder in concrete and contribute to the its strength [6].

Response surface methodology (RSM) consists of a set of statically method can be used to develop improve, or optimize product [7]. RSM is typically uses in situation where several factors influence one or more responses. In the study of concrete using RSM, the factors are usually the effects of component materials or other external parameters that govern the concrete production, while the responses are the properties or performances of the concrete. Additionally, RSM design is important in finding a suitable mix design for concrete containing admixture that can enhance the concrete performance in both fresh and hardened states[8].

Hence, the goal of this study is to analyse the experimental data of previous research on the effects of HAp dose and time towards the compressive strength and tensile strength of concrete. For this purpose, response surface methodology (RSM) was used in optimizing the experimental data. Regression equation was yielded relating to the independent variables and responses. With the help of this method, the optimum conditions can be obtained, and experimental results could be predicted within an acceptable range of experiment errors.

2. Methodology

This study uses the Design-Expert software for mathematical modelling and statistical analysis by Response Surface Methodology (RSM) to explore the relationship between the variables (HAp dose and time) and responses (concrete compressive and tensile strength). The input parameters are substituted in actual and coded form in the corresponding equations to predict the responses. The analysis of variance (ANOVA) from the developed RSM models is the indicator of the statistically significant model. The relative change in adjusted R^2 and predicted R^2 of each response are the indicators of model acceptance. The verification and the adequacy of the chosen model is done quantitatively and graphically. Then, the optimization model searches for a combination of factor levels that simultaneously satisfy the requirements placed on each of the responses and process factors. All the experiment data sets of a total of 15 runs were design point for modelling and optimizing level of chosen variables from the experiment of [9]

To determine the relationship between the variables and the responses under consideration, the data collected must be analysed by a statical approach of using regression equations, For the regression equation, the preparation variables were coded according the Equation (1) :

$$y = \beta_0 + \sum_{n}^{k} \beta_i x_i + \sum_{n}^{k} \beta_{ii} x_i^2 + \sum_{n}^{k} \beta_{ij} x_i x_j + \epsilon \qquad \text{Eq.1}$$

where y is the response (dependent variables, β_0 is constant coefficient, β_i, β_{ii}, and β_{ij} are the regression cofficients for linear, quadratic and interaction terms, respectively, whereas x_i and x_j are the independent variables, while ϵ is the error. [10]

The operating parameters, their designated symbols, response and range of conditions are presented in Table 1.

Table 1: Ranges of the factors investigated using Historical Data Design

Operating variables	Symbol	Ranges	Low Coded	High Coded
Hap Dose	A	0% - 40%	-1	+1
Time	B	7 – 28 Day	-1	+1

3. Results and Discussion

The results for the actual values were acquired from the past research [9] and the predicted values obtained from the generated quadratic model corresponded to the value of compressive and tensile strength.

3.1 Model fitting

The regression analysis was performed on the response to determine the coefficients of the model terms. Model reduction by manual exclusion of larger insignificant terms were not performed since bulk of the model terms were significant. All factors in consideration would be include, thereby eliminating the need for experimental determination of theoretical parameters. The historical data design of the factors and response in this study is shown in Table 2.

Table 2: Historical data design of the factors and response

Factors			Response	
Run	Hap Dose (%)	Time (Day)	Compressive Strength (MPa)	Tensile Strength (MPa)
1	0	7	30.49	5.35
2	10	7	26.79	5.05
3	20	7	33.87	5.75
4	30	7	24.68	5.01
5	40	7	24.13	5.05
6	0	14	38.24	5.32
7	10	14	34.83	5.12
8	20	14	42.92	6.23
9	30	14	27.69	5.1
10	40	14	26.4	5.12
11	0	28	45.45	6.23
12	10	28	37.32	5.78
13	20	28	48.96	8.1
14	30	28	29.44	6.05
15	40	28	27.66	5.98

The collected data was analysed by statistical approach of regression equations to determine the relationship between the factors and the responses. The obtained quartic model was represented by a polynomial equation for compressive and tensile strength as shown in Equation 2 and 3, respectively.

$$\text{(Compressive) } y = 45.65 - 8.99A + 7.51B - 2.89AB - 63.09A^2 - 4.12B^2 - 18.55A^2B + 2.02A^3 + 2.02A^2B^2 + 0.0367A^3B + 51.49A^4 - 3.41A^3B^2 + 15.66A^4B \quad \text{Eq.2}$$

$$\text{(Tensile) } y = 6.63 - 0.0682A + 1.18B + 0.0410AB - 7.17A^2 + 0.2773B^2 - 3.63A^2B + 0.2707AB^2 + 5.91A^4 - 0.345A^3B^2 - 0.0868A^2B^3 + 2.99A^4B \quad \text{Eq.3}$$

3.2 Analysis of variance (ANOVA) of the model

The analysis of variance (ANOVA) was obtained for the modified regression model. The ANOVA result for the compressive strength is shown in Table 3. The p-values showed the significant of each coefficient as well the interaction effectiveness between each independent variables. The p-value < 0.0237 with the model F-value of 416.99 and around 3.83 % (a large value occurring due to noise) for the modified model, suggests that the regression model is significant. P- values less than 0.0500 indicate the model terms are significant. In this case B, A^2, A^4 are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. It can observe that (3) of the (13) model terms are significant. The significant model terms have synergistic effect on the regression model while insignificant terms have antagonistic effect. Therefore, model factor that significant positively contribute to the model equation while non-significant have negative impact on the developed model. The most influential model parameter was B because it had the least p-value.

Table 3: ANOVA for Compressive Strength

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	862.91	13	66.38	416.99	0.0383	Significant
A-Hap dose	16.67	1	16.67	104.73	0.0620	
B-time	114.81	1	114.81	721.25	0.0237	
AB	4.64	1	4.64	29.14	0.1166	
A^2	274.95	1	274.95	1727.27	0.0153	
B^2	17.76	1	17.76	111.57	0.0601	
A^2B	18.15	1	18.15	114.02	0.0594	
AB^2	2.66	1	2.66	16.70	0.1528	
A^3	0.6815	1	0.6815	4.28	0.2866	
A^2B^2	1.81	1	1.81	11.38	0.1835	
A^3B	0.0006	1	0.0006	0.0038	0.9608	
A^4	251.16	1	251.16	1577.78	0.0160	
A^3B^2	1.33	1	1.33	8.34	0.2123	
A^4B	16.35	1	16.35	102.70	0.0626	
Residual	0.1592	1	0.1592			
Cor Total	863.07	14				

For the tensile strength, ANOVA result is shown in Table 4. The p-values was 0.0012 and the model F-value of 116.54 implies that the model is significant relative to the noise. There was 0.1 % chance that an F- value occur due to noise. P- values less than 0.0500 indicate model terms are significant. In this case there are B, A^2, B^2, A^2B, A^4, A^4B are significant model terms. It can be observed that three (6) of the nine (11) model terms are significant. The most influential model parameter was A^4 because it had the least p-value.

Table 4 : ANOVA Tensile Strength

SOURCE	SUM OF SQUARES	DF	MEAN SQUARE	F-VALUE	P-VALUE	
MODEL	9.14	11	0.8297	116.54	0.0012	SIGNIFICANT
A-HAP DOSE	0.0087	1	0.0087	1.22	0.3505	
B-TIME	2.84	1	2.84	399.21	0.0003	
AB	0.0084	1	0.0084	1.18	0.3567	
A^2	3.80	1	3.80	533.75	0.0002	
B^2	0.0804	1	0.0804	11.29	0.0437	
A^2B	0.3478	1	0.3478	48.85	0.0060	
AB^2	0.0307	1	0.0307	4.32	0.1293	
A^4	3.31	1	3.31	464.44	0.0002	
A^3B^2	0.0525	1	0.0525	7.37	0.0728	
A^2B^3	0.0004	1	0.0004	0.0522	0.8339	
A^4B	0.5963	1	0.5963	83.75	0.0028	
RESIDUAL	0.0214	3	0.0071			
TOTAL	9.15	14				

3.3 Validation of the model

Since adequate precision measures the signal to noise ratio and a ratio value greater than 4 is desirable, the modified design model of compressive strength with adequate precision ratio of 64.5505 indicates an adequate signal. The modified model regression fitting was regulated by the coefficient of determination, R^2 which gave high value of 0.9998 for the compressive strength from the ANOVA result. The value of Adj-R^2 obtained was 0.9974. Therefore, the proximity of the R^2 and Adj-R^2 value less to 1.0 indicates a very high correlation between the experimental and the predicted values of the compressive strength. For tensile strength, the R^2 value is 0.9977 and Adj-R^2 is 0.9891. From the foregoing, the modified model presents an explicit type of the relationship between the independent factors and response.

3.4 Verification of model adequacy

The adequacy of the regression model was also ascertained between the predicted and actual values plot as shown in Figure 1 and Figure 2. The modified model fits realistically, thereby adequately expressing the experimental range studied. The actual value represents the measured result for each experimental run, while the predicted value is evaluated from the independent variables in the regression model. The predicted versus actual values for compressive strength show straight linear relationship. The regression coefficient R^2 and adjusted R^2 were 99.0 %. The predicted R^2 value is close to R^2 value and therefore experimental data for compressive strength are well in line with the predicted values from the model equation. Similarly, for tensile strength, the plot was well in line as the regression coefficient R^2 and adjusted R^2 were 99.0 % and 98.0 %, respectively.

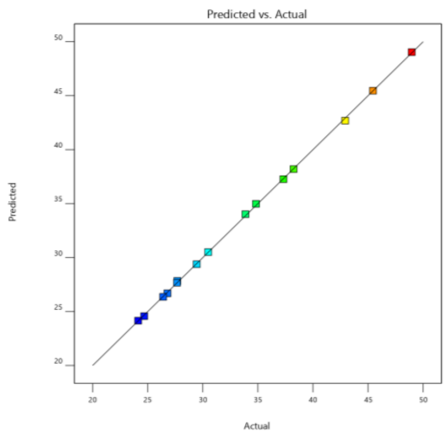

Figure 1 : Predicted Vs Actual Values For Compressive Strength

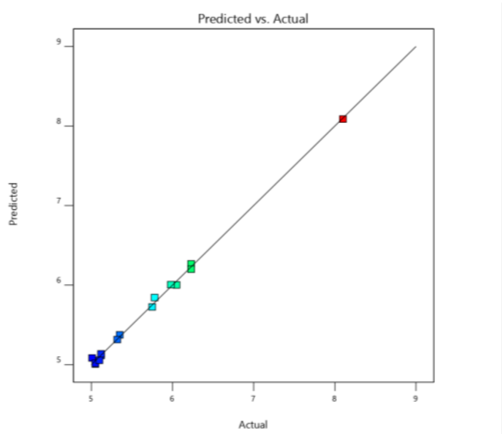

Figure 2 : Predicted Vs Actual Values For Tensile Strength

Figure 3 and 4 represents the studentized residual versus predicted values for compressive and tensile strength, respectively. This indicates that all the response values, distributed plot, the variance of the original observations are constant. This also indicates that there is no requirement of actual data transformations. No further improvement can be done to the model in making changes to the response because the data points are scattered and do not exhibit "S-shaped" curve [11]. The graph and tables thereby suggest that model can be regarded as the possible model of the historical data RSM design for compressive and tensile strength. Therefore, these shall be utilized in deriving the optimum values of the operational parameters.

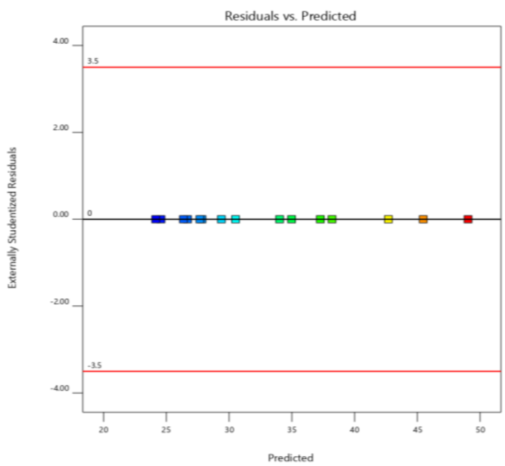

Figure 3: Externally studentized residual versus predicted compressive strength

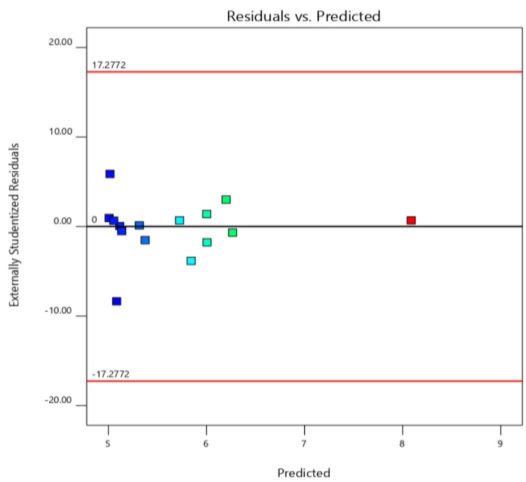

Figure 4: Externally studentized residual versus predicted tensile strength

3.5 The Effects of HAp dose and time on the compressive and tensile strength of concrete

The three dimensional (3D) plots show the behaviour of the compressive and tensile strength from the interactions of HAp dose and time. Figure 5 show the 3D plot for compressive strength. The highest compressive strength achieved was 48.90 MPa at 21.0 % HAp dose on the 28 days while the lowest compressive strength was 24.10 MPa at 40.0 % HAp dose on the 7 days. The optimum HAp dose was 21.0 %. The increased of curing time from day 7 to 28 had increase the compressive strength. Similarly, for tensile strength, the highest value of 8.10 MPa was achieved on the 28 days with 21.0 % HAp dose while the lowest tensile strength was 5.01 MPa at 30.0 % HAp dose on the 7 days as shown in Figure 6.

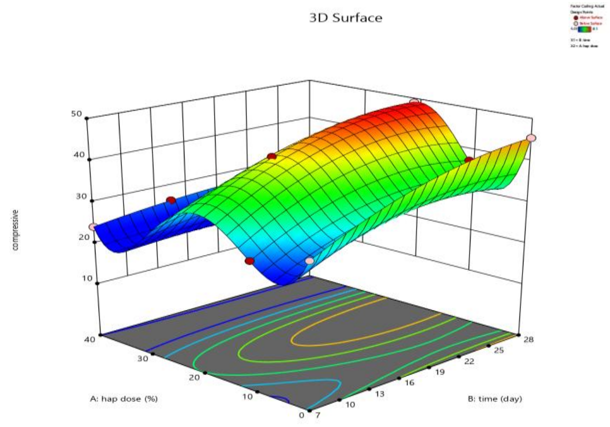

Figure 5: 3D surface plot for compressive strength

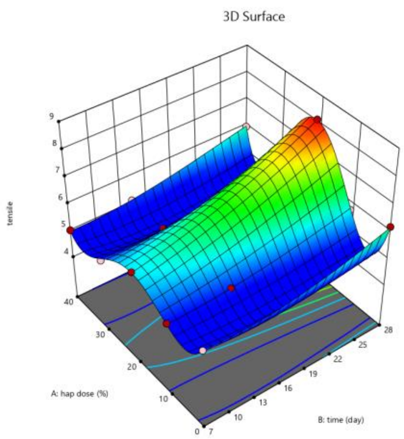

Figure 6: 3D surface plot for compressive strength

3.6 Optimization Process

The model optimization capable of predicting maximum value for the compressive and tensile strength with respect to the HAp dose and time. The optimum conditions for the compressive strength was 21.0 % HAp dose and 28 days. Under these conditions, the predicted strength value was 48.96 MPa and close to the experimental value of 48.42 MPa. The error obtained between model predicted and the experimental value is less than 1.0 % which indicates that the model is accurate for the compressive. For the tensile strength, the optimum conditions were 21.0 % HAp dose and 28 days. Under these conditions, the predicted tensile strength of 8.1 MPa was similar and accurate to the experimental value of 8.10 MPa. The optimum conditions in detailed are shown in Table 4.

Table 4: Optimization conditions for compressive and tensile strength

Optimum Conditions	HAp Dose (%)	Time (Day)	Experimental (%)	Predicted (%)
Compressive Strength	21.3	28	48.42	48.96
Tensile Strength	21.3	28	8.1	8.07

4. Conclusion

The historical data design of the Response surface method (RSM) by using the Design-Expert software was successfully employed in this study. The mathematical and statistical model on the effects of HAp dose and time toward concrete compressive and tensile strength was obtain. This study revealed that the HAp dose and time individually affect the concrete strength significantly compared the combined effect. Second order quadratic model equation was establish for predicting the response of the correlate parameters. The coefficients of the developed model were calculated for each response. High acceptability of the postulated model was proven from the high magnitude of the correlation coefficient between the actual and model predicted values. Initial percentage of HAp dose was found as the most significant factor among the process variables. The predicted compressive strength of 48.90 MPa at the optimum condition of 21.0 % HAp dose and 28 days was close the experimental value of 48.40 MPa. On the other hand, the predicted tensile strength was accurately similar to the experimental value of 8.10 MPa at the optimum of 21.0 % HAp Dose and 28 days. This study show that RSM is a reliable optimization technique that can be used to design the experiments and determine the performance of concrete.

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