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Original Article

Statistical analysis and Response Surface Modelling of the compressive strength inhibition of crude oil in concrete test cubes

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ABSTRACT

The use of crude oil contaminated fine aggregates in the production of concrete significantly affect the properties of such concrete, especially the compressive strength. In the present investigation, response surface methodology (RSM) of the Design-Expert software version 11.1.0.1 was used for the statistical analysis and predictive modelling of the compressive strength of concrete cubes made from crude oil contaminated fine aggregates at 7, 14, 28, and 56 days curing periods. The fine aggregates were mixed with varying concentrations of crude oil contamination (ranging from 0% to 5% by weight of the fine aggregates, at 1% interval). Concrete test cubes were produced for compressive strength determination and prediction phase of the modelling. A steady reduction in the compressive strength of the concrete cubes was recorded as the crude oil content increases, due to the inhibitive and surface shielding influence of crude oil molecules on the fine aggregates, thereby hindering physical bond formation between the cement paste and the aggregates. Statistical analysis of the output/response was carried out; a correlation coefficient of 0.9923 was obtained. The result of the modelling has shown that the use of RSM is adequate in the prediction of the compressive strength inhibition of crude oil in concrete made from crude oil-contaminated sand.

1. Introduction

Concrete is unarguably the commonest construction material used in structural and civil works. It is a versatile construction material adaptable for different uses. Concrete is a mixture of cement, aggregates (fine and coarse), and water. The slurry of cement and water serves as a binder for the fine and coarse aggregates, by the chemical reaction of the cement and water known as hydration. Depending on the requirements demanded by the engineer, the properties of concrete can be altered to yield the expected result, by the addition of several other materials like admixture and additives.

Concrete produced in regions where crude oil is exploited or environment where there is an occurrence of oil spillage, can have significant changes in its properties, such as flexural strength, compressive strength, durability, slump, compacting factor, water absorption, linear shrinkage, surface resistivity, and fire resistance, because of crude oil contamination of its constituents [1]. This contaminated sand may be used in the production of concrete for erecting structures in the neighbourhood or may be transported for

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use at other locations. The amount of this contamination affects the strength of the concrete made from the sand. Experimentation as well as predictive modelling can be employed to ascertain the extent of such alteration in properties of the concrete [2–7]. Crude oil have been found to inhibit the compressive strength of concrete [3,8–10]. Research findings suggested that the optimum crude oil contamination for the achievement of normal compressive strength of concrete cube is as low as 0.3% [1,11]. Concrete gains strength over a long period after pouring in the formwork or mould. In quality control of concrete, the compressive strength of concrete is adjudged the most important property and it is used for its strength specification [1,12]. This characteristic strength is denoted as the normal compressive strength of concrete at 28 days [10]. Development of rapid and reliable prediction methods and models for the strength of concrete would be of great significance. Several factors have been found to influence the compressive strength of concrete including water-cement ratio, the volume of entrapped air, type of cement, curing conditions, presence of additives and admixtures, and aggregates‘ characteristics like size, shape, surface texture, grading, and chemical composition (mineralogy) [6,8,12–14].

Experimental design, optimization, statistical analysis and predictive modelling are effective tools that could reinforce experimental results [15]. They are used to conduct minimum experiments by varying input factors between their respective levels, analyzing the factors quantitatively for their statistical significance, derivation of predictive model equations, and validation & confirmation of the adequacy and suitability of the models based on the collected input-output data [15]. Objective functions for engineering applications, which are considered by designers, could be computed using predictive modelling and optimization software [16]. According to the reviewed literature, central composite design (CCD), mixture design, factorial method, response surface method (RSM), box Behnken design (BBD), Taguchi robust design, finite element modelling, artificial neural network, fuzzy logic method etc., are mostly used experimental design tools and methodologies [4,6,16–21]. Statistical analysis, modelling and optimization tools and methodology could be applied to numerous civil engineering problems such as; prediction of the settlement of shallow foundations, novel methods for designing a concrete mix, modelling of the slump of concrete, prediction of strength properties for high-performance concrete, construction duration of buildings, modelling soil behaviour, settlement of structures, slope stability, water demand, and so forth [4–6,22]. In particular, ANN was used in [6] to predict the behaviour of different constituents of concrete mixture to obtain the compressive strength of concrete. Mathematical models capable of predicting the effect of mixture matrix on the compressive strength of concrete have also been proposed [22–24]. Response surface methodology (RSM) as a statistical and experimental design tool correlates the response of a selected dependent variable to the variations in one or more independent or factor variables. It is a useful technique for statistical analysis, predictive modelling and optimization of process parameters and properties [25]. The aim is to optimize the response of the dependent variable (output) affected by varying the design settings for one or more independent variables (inputs/factors) [25].

This study is aimed at optimizing the compressive strength of crude oil contaminated concrete and generating mathematical regression models capable of predicting the inhibition effect of varying crude oil concentration and the curing conditions on the compressive strength.From the optimization solution, the minimal crude oil content for optimum compressive strength would be ascertained.

2. Materials and Methods

2.1. Materials Preparation and Production of Concrete Test Cubes

The following materials were procured locally and utilized in the production of the test cubes: fine aggregates, coarse aggregates (15-22 mm), portable water, ordinary Portland cement (OPC), and crude oil. The fine aggregates were air-dried and divided into six equal parts. One part of the fine aggregates used in casting the control test cubes was uncontaminated with crude oil, while the remaining five parts were contaminated independently with the specified quantity of crude oil in the order of 1, 2, 3, 4 & 5% by weight of the fine aggregates and used to cast the crude oil contaminated test cubes. A prescribed constituents’ mix of 1:2:4 and water-cement ratio of 0.5 was employed in the production of the concrete cubes. The casting of the concrete cubes was done in a 150x150x150 mm steel mould smeared with oil for easy removal of the concrete cubes. Seventy-two concrete test cubes were produced.

2.2. Experimental Design

The Design-Expert version 11.1.0 was used to assess the relationship of the response variable with the independent variables. The steps involved in the experimental design are: Central Composite Design (CCD) was used to generate experimental runs; carrying out the compressive strength experiments on the concrete cubes; analysing the compressive strength results and obtaining regression equation for various process factors and the response variable; analysis of variance (ANOVA); and predicting the optimum process variables and compressive strength. The CCD methodology as described in [26,27] was adopted in the experimental design. However, the process variables used in this study were; curing period (7 - 56 days), and Concentration of crude oil contaminant (0.00 – 5 %) respectively, while the Response Variable was Average compressive strength (Mpa). Table 1 provides the experimental design layout and modelling data, while Tables 2 & 3 provide the Central Composite Design with the factors coding & levels and the design summary for the response variable respectively.

Table 1. Experimental design layout showing the modelling data for the factors and the corresponding response.

Run No	**X: Curing Period (Days)**	**Y: Conc. of Crude Oil (%)**	**Average Compressive Strength (MPa)**
1	7	0	13.96
2	14	0	17.9
3	28	0	22
4	56	0	23.6
5	7	1	11.24
6	14	1	15.65
7	28	1	17.61
8	56	1	18.45
9	7	2	9.82
10	14	2	13.72
11	28	2	15.82
12	56	2	16.24
13	7	3	8.36
14	14	3	11.24
15	28	3	12.12
16	56	3	13.63
17	7	4	7.44
18	14	4	8.86
19	28	4	10.24
20	56	4	11.56
21	7	5	6.82
22	14	5	7.02
23	28	5	8
24	56	5	10.46

Table 2. Experimental range of the independent variables, with factor levels and coding.

Factor	**Name**	**Units**	**Type**	**Min**	**Max**	**Coded Low**	**Coded High**	**Mean**	**Std. Dev.**
X	Curing Period	days	Numeric	7.00	56.00	-1 ↔ 7.00	+1 ↔ 56.00	26.25	19.17
Y	Conc. of Crude Oil	%	Numeric	0.00	5.00	-1 ↔ 0.00	+1 ↔ 5.00	2.50	1.74

Table 3. Experimental design summary for the response variable.

Response	**Name**	**Units**	**Obs**	**Analysis**	**Minimum**	**Maximum**	**Mean**	**Std. Dev.**	**Ratio**	**Transform**	**Model**
R1	Average Compressive Strength	MPa	24	Polynomial	6.82	23.6	12.99	4.65	3.46	None	Cubic

2.3. Compressive Strength Determination

A destructive compressive test was carried out on the test cubes with dimension 150 X 150 X 150 mm using the electronic compression test machine. The concrete mix was placed into a mould smeared with oil for easy removal of the concrete test cube. Concrete compaction was done in three layers using a 25 mm square steel punner with 35 strokes per layer and the surface was levelled using hand trowel after the stroke. The mould was opened thereafter and the concrete cubes immersed in the curing tank at laboratory room temperature. All the compression test were performed in compliance with British Standard BS 1881: Part 125: 1986. During the test, a crushing load of 12-24N/mm^2/min were constantly applied until the specimen failed. Three samples were tested and their average was taken as representative of the compressive strength of the concrete cubes. The compressive strength, given in MPa was automatically recorded at failure and displayed by the compression machine. The compressive test was performed on the test cubes at various curing ages (7, 14, 28, & 56 days) and crude oil contamination concentrations (0, 1, 2, 3, 4, & 5%).

3. Results and Discussion

3.1. Relationship of Compressive Strength of Concrete with Crude Oil Contamination Level and Curing Period.

The experimental results for the impact of the varying concentrations of crude oil contaminant on the average compressive strength at various curing conditions are presented using the scatter plot of fig 1. It could be observed from the figure that the average compressive strength of the test cubes decreased progressively as the contaminant concentration is increased from 1 to 5%. While the increase in the curing days influenced positively the compressive strength of the contaminated concrete cubes, as a good concrete is normally expected to have its compressive strength increase with age [1]. This steady reduction in the compressive strength could be attributed to the inhibitive influence of crude oil molecules in the aggregates, as the surface areas of the fine aggregates were coated by the crude oil molecules thereby hindering physical bond formation between the cement paste and the aggregates [3,8,28]. Abousnina et al had also attributed this reduction in compressive strength of crude oil contaminated concrete to incomplete hydration at the respective curing periods [28].

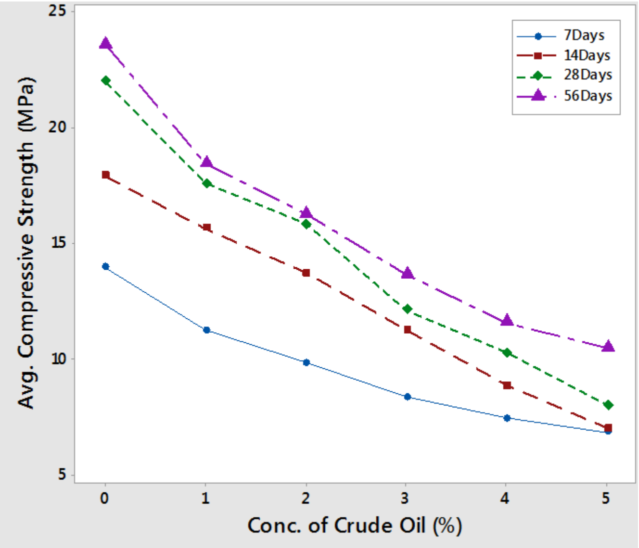

Fig 1. Scatterplot of Average Compressive Strength (MPa) at different curing period (days) against the Concentration of Crude Oil (%).

3.2. Statistical Analysis and Modeling of Compressive Strength

The analysis of variance (ANOVA) and the summary of fit statistics for the concrete cubes' compressive strength are presented in Table 4. Descriptive statistics were used to ascertain the usefulness of the models. The probability values (P-value) obtained for the response variable in the regression is less than 0.05, which is a good indication that both model terms (Concentration of crude oil contaminant and curing age) have significant impact on the test cubes' compressive strength. Significant model terms are indicated by P-values less than 0.05 [29,30]. In this case, Y, XY, X^2, Y^2, X^2Y, XY^2, X^3 are all significant model terms. On the other hand, values greater than 0.10 are used to indicate insignificant model terms. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction is recommended for improving the model. The model F-value obtained for the response variable is as well significant. The Model F-value of 199.82 implies the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise. Conversely, the lack-of-fit F-values for the model are non-significant relative to the pure error, and since the goal is for the model to fit, non-significant lack of fit is good. The interactive significance of the process variables was evaluated using ANOVA as shown in Table 4. The model adequacy was confirmed by the coefficient of determination (R^2), 0.9923 (99.23%) which is accurate. The value of the coefficient of determination reflects the percentage of the experimental results the model can explain. The R-squared value: 0.9923 (99.23%); Adjusted

R-squared: 0.9873 (98.73%) and the predicted R-Squared 0.9688 (96.88%) are also shown in Table 4. It was found that the Adjusted R-squared and the Predicted R-squared were in reasonable agreement, as their difference was below 0.2 (20%) [15,30]. The signal to noise ratio could be ascertained using Adeq Precision ratio. A ratio greater than 4 is desirable [15,25,29]. The ratio of 50.330 obtained, indicates an adequate signal. This model can be used to navigate the design space.

Table 4. ANOVA for Cubic model and Fit Statistics for the Average Compressive Strength

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	493.55	9	54.84	199.82	< 0.0001	significant
X-Curing Period	0.0694	1	0.0694	0.2528	0.6229	
Y-Conc. of Crude Oil	51.07	1	51.07	186.10	< 0.0001	
XY	11.36	1	11.36	41.40	< 0.0001	
X^2	7.84	1	7.84	28.55	0.0001	
Y^2	6.85	1	6.85	24.96	0.0002	
X^2Y	9.22	1	9.22	33.61	< 0.0001	
XY^2	1.31	1	1.31	4.77	0.0465	
X^3	2.92	1	2.92	10.66	0.0057	
Y^3	0.1635	1	0.1635	0.5958	0.4530	
Residual	3.84	14	0.2744			
Cor Total	497.39	23				
Std. Dev.	0.5239		**R^2**	0.9923		
Mean	12.99		**Adjusted R^2**	0.9873		
C.V. %	4.03		**Predicted R^2**	0.9688		
			Adeq Precision	50.3305		

Equation 1 gives the predictive model for the average compressive strength of the crude oil contaminated concrete cubes as a function of crude oil contaminant concentration and curing age in a coded form. When the model equation is expressed in terms of coded factors, it can be used to predict the compressive strength at a specified weight concentration of crude oil contaminant (wt%) and curing age (days). In the default setting, the high levels of the factors are coded as +1 and the low levels are coded as -1. Using the coded equation, the relative impact of the factors could be known, by comparing the factors' coefficients [15,26,29]. The maximum compressive strength obtainable at given crude oil contamination percentage and curing period could be determined using the generated predictive model.

$$\boldsymbol{Avg.\,Compressive\ Strength\ (MPa)} = 13.6774 - 0.468451x - 6.94184y - 1.32814xy - 1.6944x^2 + 1.38999y^2 + 2.25701x^2y + 0.763961xy^2 + 3.13694x^3 - 0.392433y^3 \qquad (1$$

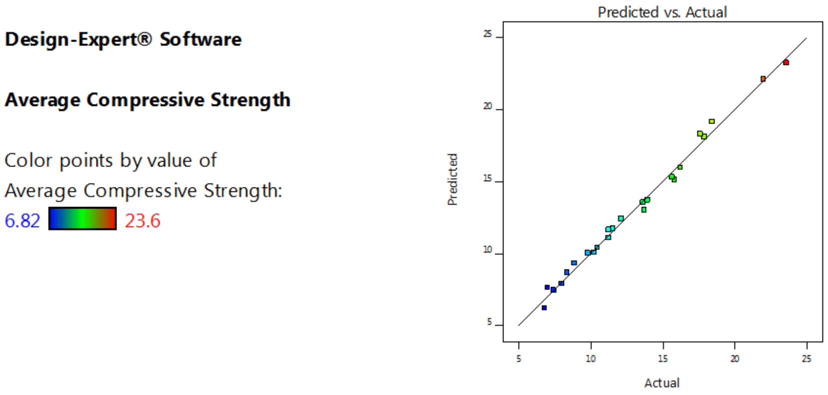

Fig 2. Plot of predicted versus actual average compressive strength.

The diagnostics and model graph of the observed (actual) response values versus the predicted response values for the two factors showed a linear graph (Fig. 2) with the data points split evenly by a 45-degree line, thus indicating that the design models are sufficient to predict the relationship of the factors-response variables [26,30]. It helps to detect observations (a value, or group of values), that are not well predicted by the model. The perturbation plot (Fig. 3) helps to compare the effects of all the factors at a particular point in the design space. The response is plotted by changing only one factor over its range while holding the other factor constant. By default, Design-Expert sets the reference point at the midpoint (coded 0) of all the factors. This can be changed to be any point (perhaps the optimal run conditions) by using the Factors Tool. A steep slope or curvature in a factor shows that the response is sensitive to that factor. A relatively flat line shows insensitivity to change in that particular factor. It could be concluded from the perturbation plot (Fig. 3) that the average compressive strength of the test cubes is highly sensitive to the crude oil contaminant content. If there are more than two factors, the perturbation plot could be used to find those factors that most affect the response. These influential factors are good choices for the axes on the contour plots.

The 3D model Surface plot shown in fig 4 is a projection of the contour plot giving shape in addition to the colour and contour. It can show the relationship between the response variable and the factors. The 3D plot shows succinctly that at higher curing age, the compressive strength was higher, while the compressive strength was lower for high contents of crude oil contaminant. Thus, crude oil contaminant content has a higher impact on the compressive strength of the concrete cubes.

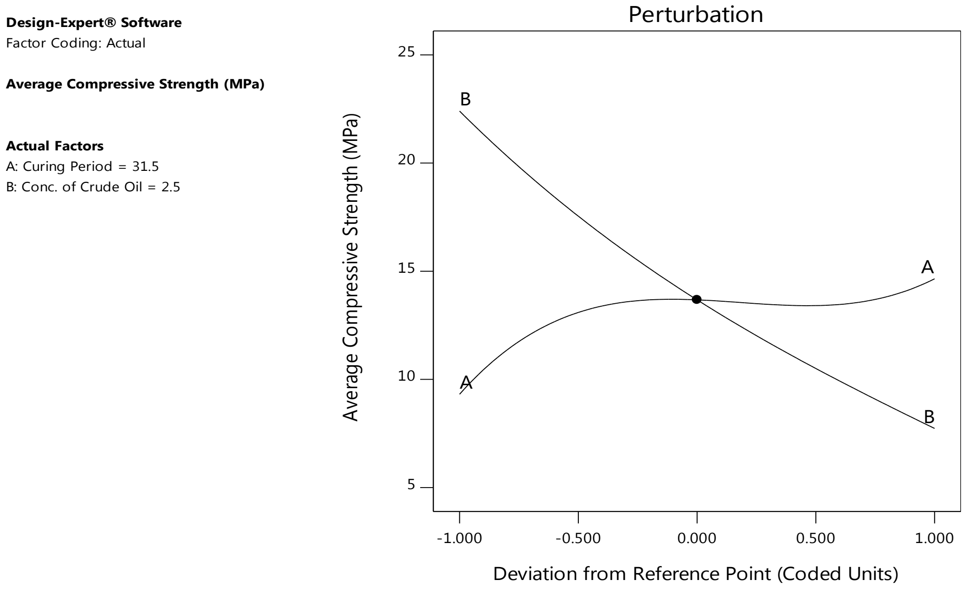

Fig 3. Perturbation plot of average compressive strength.

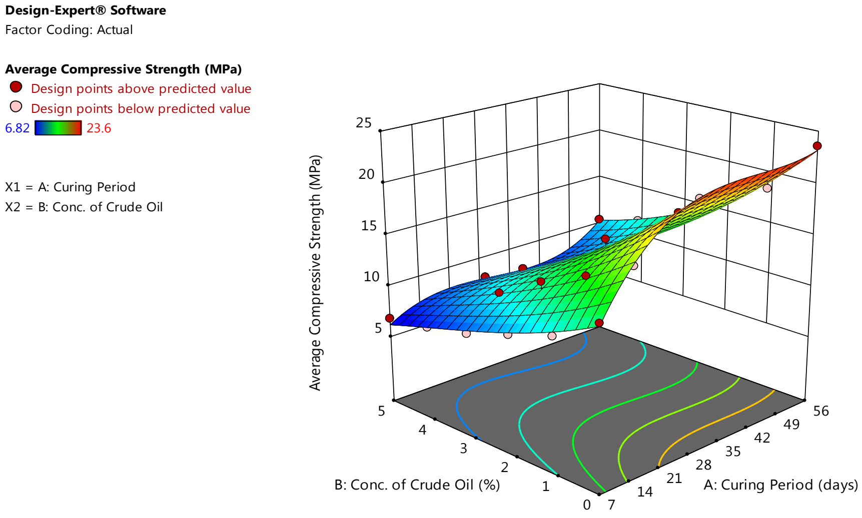

Fig 4. The 3D model surface plot.

3.3. Numerical Optimization Solution

Using RSM enables desired response optimization by setting target numerical optimization criteria for both the factor and response variables. The crude oil contaminant concentration was set to a minimum since the experimentally obtained compressive strength revealed the inverse relation. The curing age of the concrete cubes was set to the maximum since the compressive strength increases with age [1], while the response was set to the maximum since the goal is to maximize the test cubes' compressive strength. The numerical optimization solution of the factor and response variables with the degree of desirability are presented in Table 5. The optimization solution that best meets the specified criteria for the optimal setting was selected and presented in Table 5. The optimization report shows that at minimum crude oil contamination of 0.044% and maximum curing age of 56days, a maximum average compressive strength of 23.008Mpa with near-unity desirability (0.985) would be obtained. The bar graph shown in figure 5 is a graphical view for each optimal solution in terms of the desirability levels for both the factor and the response variables. Optimal factor settings are shown with a red bar, while optimal response prediction values are displayed in blue.

Table 5. Numerical optimization report.

Curing Period (days)	**Conc. of Crude Oil (%)**	**Average Compressive Strength (Mpa)**	**Desirability**
56.000	0.044	23.008	0.985

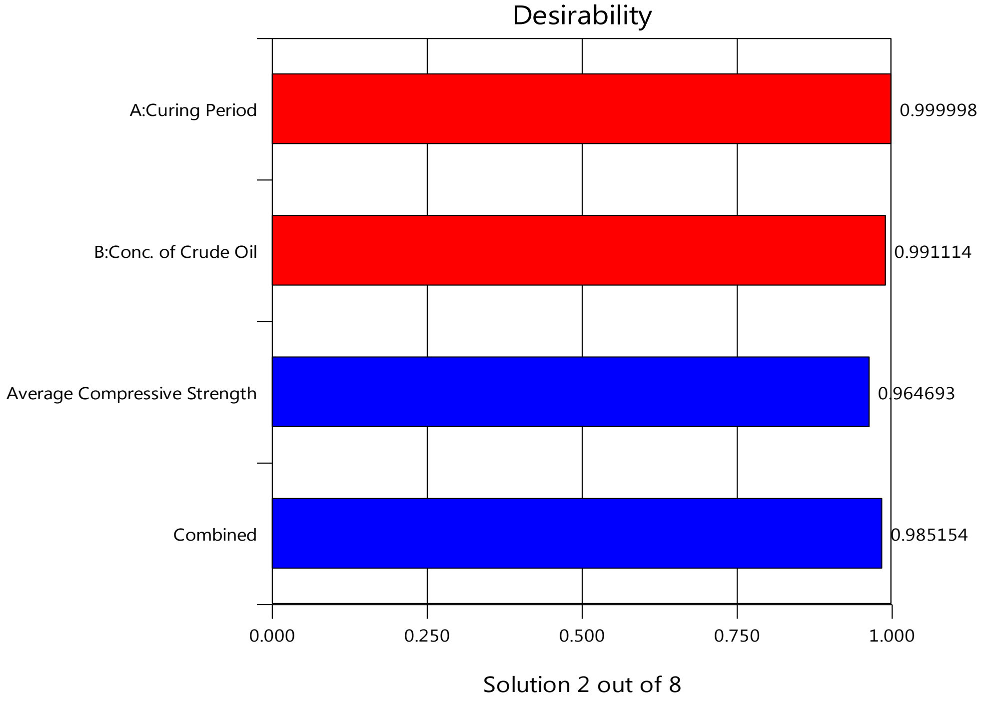

Fig 5. Numerical optimization bar graph.

5. Conclusion

The foregoing study investigated the mechanical property (compressive strength) of concrete containing fine sand with different degrees of crude oil contamination (0, 1, 2, 3, 4 and 5%) and curing conditions (7, 14, 28 and 56days). Concrete cubes produced using fine sand with 1% to 5% crude oil contamination exhibited significantly lower compressive strength than the uncontaminated samples, due to surface coating effect of the crude oil molecules on the fine aggregates which decreased bond formation with the cement paste. Moreover, a simplified empirical equation was developed for reliable prediction of the average compressive strength of concrete containing oil-contaminated sand. Statistical analysis and comparison between the experimental results and the predicted values gave a 96.88% prediction accuracy, confirming the adequacy of the proposed model. Optimal levels of crude oil contamination, curing period, compressive strength and their desirability were given at 0.044%, 56days, 23.008Mpa and 0.985 respectively by the numerical optimization solution. The practical import of this optimization result is that the minimal content of crude oil in fine aggregates for optimal strength could be determined. The derived equation could also be employed to estimate the crude oil content for desired strength and curing period.

Conflict of Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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