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Optimization of Hybrid Cement Composite with Carbon Nanotubes and Nano Silica Using Response Surface Design

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THE MOST common choice for fitting a second order model in response surface methodology is the central composite design. The central composite response surface methodology had been selected in order to investigate the performance of the cement-based design mixtures incorporating Nano silica particles (NS) in combination with multi walled carbon Nano tubes (CNTs). The proposed evaluation of the utilized hybrid NS particles - Multi walled CNTs within the cement matrix via the response surface method, showed that the combination of NS with relatively high amount of Multi walled CNTs (0.04%) had a positive effect on the hydration reaction. The presence of individual Multi walled CNTs worked as extra nucleation sites for the hydrates and enhanced the activity of the NS. While the combination of a lower amount of Multi walled CNTs in the presence of NS had a negative effect on the hydration reaction. NS agglomerations suck water between its particles and negatively affect the production of hydrates of the cement due to its re-agglomeration. The generated re-agglomeration of CNTs decreased the availability of Ca $(OH)_2$ for the NS particles to react with and produce more Calcium Silicate Hydrate (C-S-H). Finally, as for the statistical analysis, the response surface model showed that the combination of the CNTs and NS interacted effectively for enhancing the mechanical properties of the cement matrix.

Keywords: Response surface design, Carbon nano tubes, Nano silica, Compressive strength, and Scanning electron microscopy.

Introduction

Based on the importance of decreasing the number of experiments, trials, costs, time, and physical efforts, design of experiment (DOE) is an important statistical and mathematical tool for solving complex and multifactor engineering problems. It incorporates response surface methodology (RSM), factorial design, and artificial neural network (ANN) in some specific case studies [1]. However, the central composite design (CCD), D-optimal, and Box-Behnken were observed to generally be utilized as an optimization method for Fenton oxidation [2–4] because of the advantage of optimizing the multifactor problems with ideal number of trial runs.

RSM is a type of advanced new experimental design technique. The utilization of RSM could enhance the preparation idea of a new cementitious material composite, as it conquers the limitations of a one-dimensional linear model. The interaction and the influence of quadratic term are acquainted with the model, so the test precision is enhanced, which guarantees the logical idea of the investigation or study. At first, the test plan was made on the basis of a response surface orthogonal rotational combination design method, and the design parameters were the percentages of utilization of the NS particles, and the Multi walled CNTs. Then at that point, the compressive strength of specimens after 28 days of standard curing were tested, the response surface between compressive strength and design parameters was determined, the effect of the design parameters on the compressive strength was analyzed and examined, and prediction and verification of the

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compressive strength were conducted. recently, the utilization of micro scale to Nano scale particles within different cement matrices have been extensively studied; specially the use of NS particles, and multi walled CNTs in order to reach low cost cement products [5-18]. The NS particles addition to cement matrix showed a tremendous effect for the compressive strength of the cement matrices, while their effect on the flexural, and tensile strengths have not been at the same level, however, the Multi walled CNTs addition showed a significant effect on the tensile, and flexural properties on contrary to the compressive strength results. As such, and in order to study the effect of NS particles in enhancing the bonding of the Multi walled CNTs with the cement matrix, and consequently enhancing the compressive strength values for the Multi walled CNTs incorporated cement composite, hybrid NS Particles - Multi walled CNTs combinations are to be studied and statistically evaluated and optimized.

Generally, the structure of the connection between the response and the independent variables is obscure. The initial phase in RSM is to locate an appropriate estimate to the actual relationships. The most widely recognized forms are low-order polynomials (first or second-order). Graphical interpretation of interactions through utilizing three- and two-dimensional plots of regression model is highly recommended and is exceedingly prescribed and is utilized to survey the effects between the process variables and the compressive strength.

Experimental Program

Materials and characterization:

Ordinary Portland cement complying with the requirements of ASTM (American Society for Testing and Materials) C150 [19] standard was used with grade 42.5N, Cement properties could be found in Table 1. Natural Siliceous sand free of alkali-reactive materials from Suez quarries, Egypt was utilized. The properties of fine aggregates are listed in Table 2. Poly-carboxylate based super-plasticizer was used, besides potable water for mixing. Multi walled CNTs with inner diameter of 12 nm synthesized by atmospheric pressure chemical vapor deposition (APCVD) system have been used. Amorphous NS particles with 5nm average particle size have been prepared by precipitation rout. All details related to Multi walled CNTs, and NS synthesizing process can be found elsewhere in our previous work [20–22]. The high-resolution transmission electron microscope (HRTEM) was utilized through this study to confirm the morphology of the Multi walled CNTs. It can be seen that the Multi walled CNTs has a tubular shape with 9 nm inner diameter while the outer diameter measure around 15nm as shown in Fig. 1. The Multi walled CNTs was also examined by X-ray diffraction (XRD) to additionally confirm its structure. The XRD pattern of Multi walled CNTs is shown in Fig. 2. The diffraction peaks at 26.2º, 43.2º can be indexed to (002), (110) planes of Multi walled CNTs [21] [23,and 24]. The HRTEM micrograph demonstrated that the obtained NS is spherical with less agglomeration. The particle size was

TABLE 1. The chemical Composition of Portland cement.

Element	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O	TiO_2	L.O.I*
Cement (%)	20.13	5.32	3.61	61.63	2.39	2.87	0.37	0.13	-	1.96

*L.O.I. = Loss of Ignition

TABLE 2. The properties of Sand and Crushed Dolomite.

Property	Sand	Specification according to ASTM C33 [25]
Specific Weight	2.89	-
Bulk Density (t/m^3)	1.67	-
Fineness Modulus	2.75	2.3: 3.1
Water absorption (%)	-	-
Crushing Value (%)	-	not more than 45%
Clay and Fine Dust Content (%)	1.95	not more than 3% for fine aggregate & 1% for coarse aggregate

* ASTM = American Society for Testing and Materials.

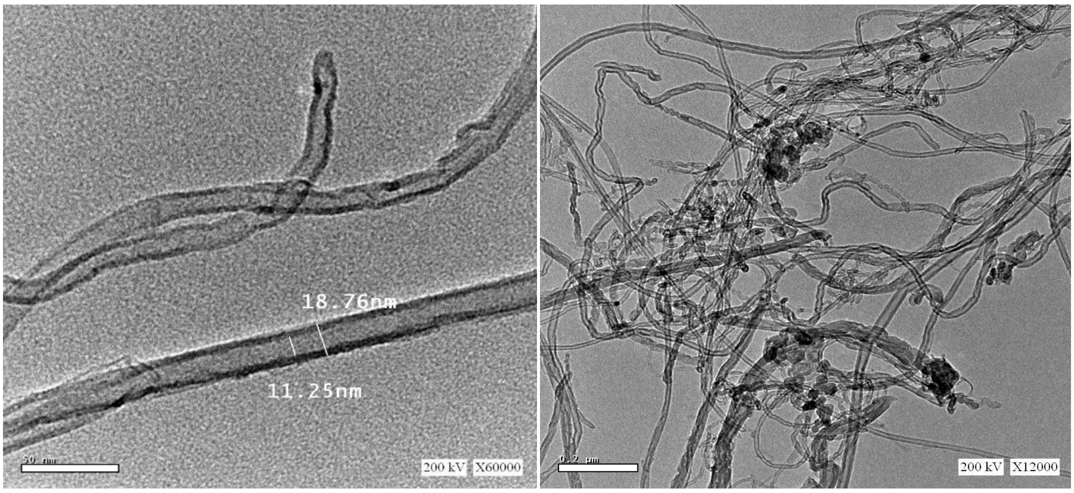


Fig. 1. HRTEM of CNTs.

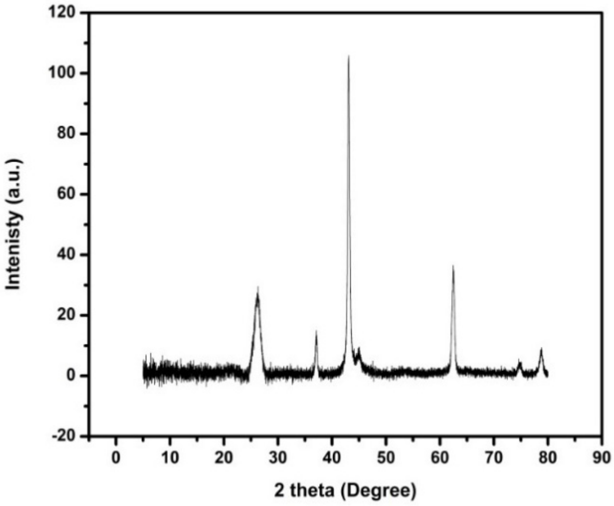


Fig. 2. XRD diffraction pattern of CNTs.

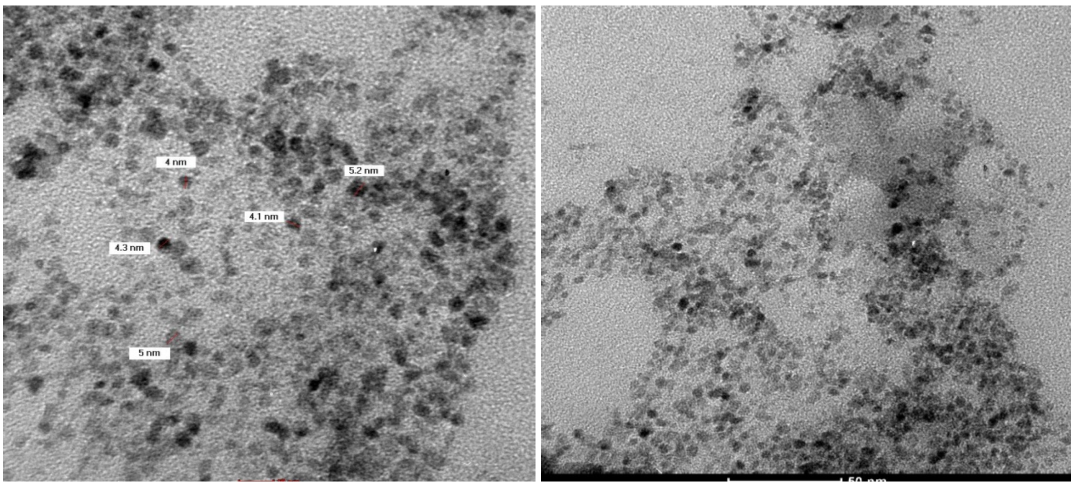


Fig. 3. HRTEM of NS particles.

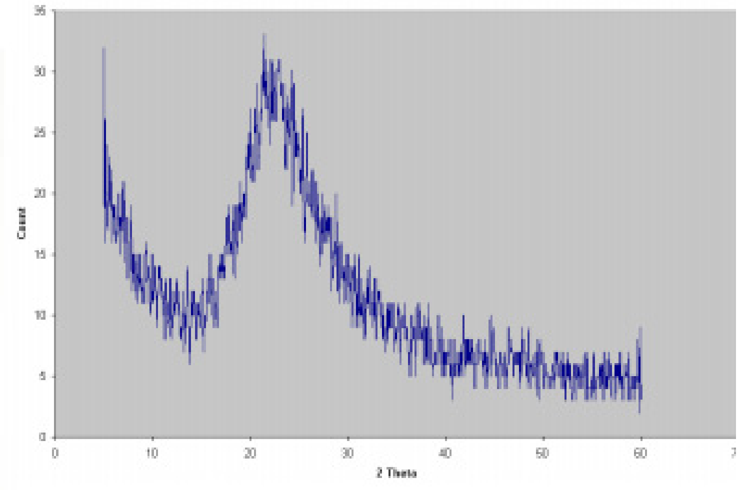


Fig. 4. XRD diffraction pattern of NS particles.

measured to be about 5nm as it could be seen in Fig. 3 the characteristic broad silica peak observed at $2\theta = 22°$ indicates the presence of amorphous silica as shown in Fig. 4.

Materials.

Mixtures

A total of nine cement mortar mixtures were prepared for the required investigation, as it can be seen in Table 3, these mixtures were all prepared using ordinary Portland Cement Type I (CEMI-42.5N). The mixing procedure can be summarized as follows: the Multi walled CNTs were firstly been dispersed using a dual cavitation process (Stirred for 10 min and then ultra-sonicated for 5 minutes) with one third of the mixing water, while the NS Particles were dispersed via the application of ultra-sonication only for 5 min with another one third of the mixing water, finally the rest of the water was added into the mixer with the super-

TABLE 3: mixtures constituents.

Mixes	NS (gm)	CNT (gm)	Water (Ml) (ml)	Cement (gm)	Sand (gm)
Mix1	15	0.2	500	984.8	2000
Mix2	20	0.1	500	979.9	2000
Mix3	25	0.2	500	974.8	2000
Mix4	20	0.3	500	979.7	2000
Mix1	15	0.2	500	984.8	2000
Mix5	10	0.1	500	989.9	2000
Mix6	15	0	500	985	2000
Mix1	15	0.2	500	984.8	2000
Mix7	15	0.4	500	984.6	2000
Mix8	10	0.3	500	989.7	2000
Mix1	15	0.2	500	984.8	2000
Mix9	0	0.2	500	999.8	2000
Mix1	15	0.2	500	984.8	2000

The 5 repeated mixes are representing the central mix plasticizer. The detailed mixtures constituents are as follows:

Response Surface Statistical Analysis

The aim of the present research plan is to study, investigate, and search by the means of the RSM [26-31], for the best combination and utilization of the Multi walled CNTs and NS Particles to be used as cement additives for the strength enhancement. A central composite design [32 and 33] is set up to check the effect of two variables; NS percentage, and Multi walled CNTs percentage on the mechanical properties of the cement mortars specially the compressive strength. Total 14 runs with 5 center points were suggested by design expert to optimize the responses.

The following quadratic equation to predict the dependent variables (responses) was developed in terms of independent variables and their interactions:

$$Y = b_0 + b_1x_1 + b_2x_2 + b_{11}x_1^2 + b_{22}x_2^2 + b_{12}x_1x_2$$

Where Y is the response variable b_0 is constant, b_1, and b_2 are coefficient for linear effects, b_{11}, and b_{22} are quadratic coefficient, and b_{12} is interaction coefficient [34], respectively.

The numerical optimization will be conducted using the statistical software (JMP version 14) [35].

Results and Discussion

The mechanical properties values of the conducted research investigation and the outcome out of the performed statistical response surface analysis will be displayed.

At first, the results revealed that the central cement mortar mix is the mix with the lowest compressive strength. As the nano hybrid contents increase, the compressive strength increases as compared with the central mix.

Generally, the results confirmed the importance and significance of the proposed study, the most of all is the significance role of the NS addition in enhancing the dispersion of the Multi walled CNTs particles especially when used at relatively high contents (0.04%). The optimum mixing combinations between both nano particles to be utilized within the cement matrix has been reached. Those optimum combinations had shown very significant effect on the enhancement of the compressive strength of the final cement composite reaching a gain of about 65 percent as compared with the mix incorporating nano particles addition and having the lowest compressive strength.

The optimum compressive strength as well as any other required compressive strength could be determined in terms of the NS and Multi walled CNTs contents via the proposed design and presented here under parabolic 3-D figures and contour plots. In the following paragraphs, the detailed discussion around the statistical response surface analysis will be introduced.

The results from the conducted statistical analysis suggested the following:

The R.-squared statistic, based upon the ratio of the model sum of squares divided by the total (corrected) sum of squares, indicates that the model accounts for 81% of the variation of the mean size percentage to the origin material. The mean squared error estimates the variance of the deviations around the model to be equal to 36.9.

NS Particles and Multi walled CNTs polynomial separate terms have shown a highly significant effect on compressive strength within the variation intervals determined both for P-Values under 0.05. While their first order, and interaction terms does not have a significant effect on the compressive strength, probably due to the relatively lower surface area per unit volume of the agglomerations resulted from the Nano hybrid combination. As Nano hybrid contents increase within a certain low range an increase in compressive strength was recognized.

The estimated coefficients for the multiple regression model are shown in Fig. 5. The P values correspond to tests of the hypotheses that the coefficients are equal to zero. The Values of P less than 0.05 indicate statistically significant non zero coefficients at a 95% confidence level. The response surface analysis showed a significant effect for the factors (NS, and Multi walled CNTs polynomial effects).

The normal probability plot and the residuals, shown in Fig. 6, can be utilized to pass judgment on whether the residuals could reasonably be considered to follow a normal distribution, and may likewise be useful in detecting outliers. The residuals fall fairly well along a straight line, while no outliers can be watched.

The response will be represented and displayed graphically, in both; the three-dimensional space and as *contour plots* that help visualize the shape

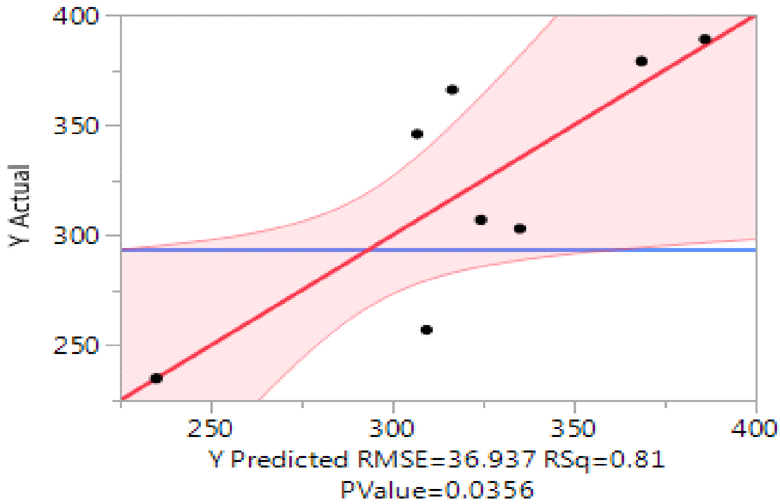


Fig. 5. Response Y - Actual by Predicted Plot - *RMSE = Root Mean Square Error.

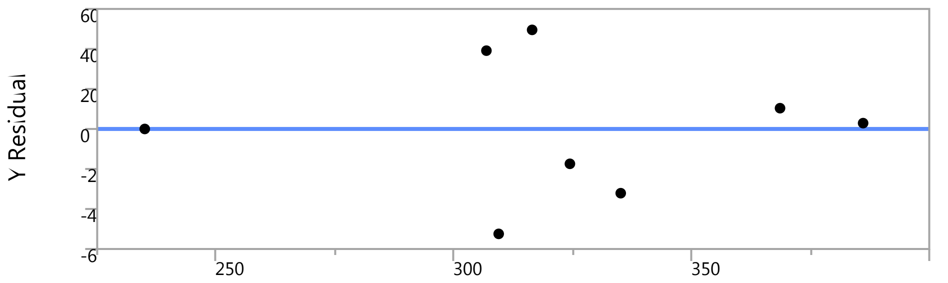


Fig. 6. Residual by Predicted Plot.

of the response surface. Fig. 8 to 10 introduce helpful design charts correlating NS, and CNT percentages with compressive strengths in 3D, and contour plot forms respectively. Generally, the compressive strength increases in a parabolic shape where the central mix acts as the base point of the paraboloid at its lowest compressive strength value. As it could be found in Fig. 5. While for the contour plots (which is the projection of the 3-D paraboloid) display both; the actual and predicted values for the response Y (compressive strength). The NS percentages for the predicted results were chosen to be from 0% to 2.5% from total binder content. It should be noted that the results are constrained with the proposed experimental cement mortar mixes.

Optimization analysis for the performance characteristics of cement mortars can be performed for a combination of factor levels that at the same time fulfill the desired and ideal prerequisites for every response. The simultaneous optimization for the response has a low and high value assigned to the objective or goal. The goal or objective field for the response is one of five categories: none, maximum, minimum, target, or within a specified range. Every goal or objective is assigned a weight on a scale ranging from one to five (one being of least importance and five being of the most importance). The included parameter in the optimization analysis can be within their design range, or as a maximum/minimum of a target goal. The objective is then added into a desirability function, the desirable ranges are from zero to one for the given response of the numerical optimization, and by using statistical software (JMP*)* in the current study); the highest desirability function was determined. The goal searching starts at a randomly selected point and proceeds up the steepest slope to a maximum value. There may be two or more maximum points due to the curvature of the response surfaces and their combination into the desirability equation. The value of the goal that equals to one within the experimental domain represents the optimum condition while the zero value may represent that one or more responses fall outside the desirable limits.

Finally based on the proposed mix constituents, the most desirable NS Particles, and Multi walled CNTs percentages were introduced in Fig. 7, as well as the corresponding predicted compressive strength value.

As shown in Fig. 7, the optimum values of NS Particles and Multi walled CNTs with highest desirability of 0.9 with a corresponding compressive strength value of 386 kg/cm^2. The elliptical nature of the contour plots indicates that the interaction between the corresponding variables is significant.

Microstructure Analysis Micrographs

From the SEM and TEM micrographs the following can be observed:

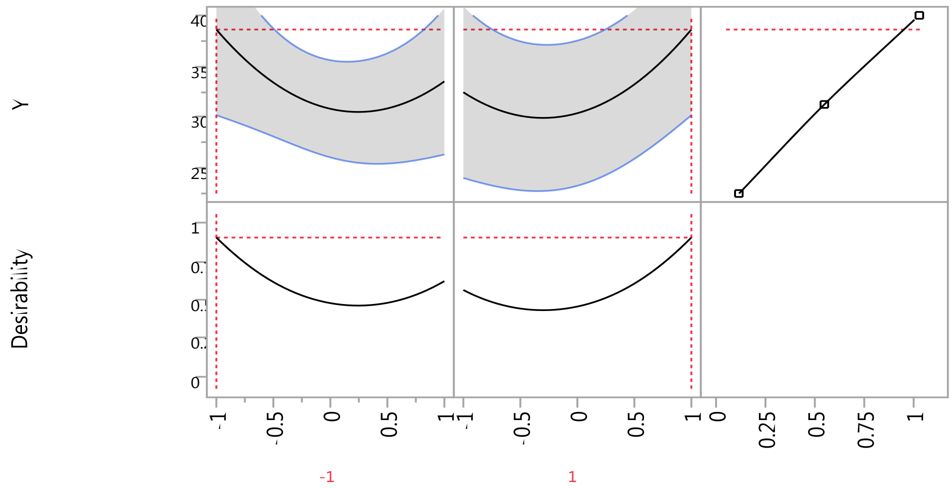


Fig.7. Prediction Profiler.

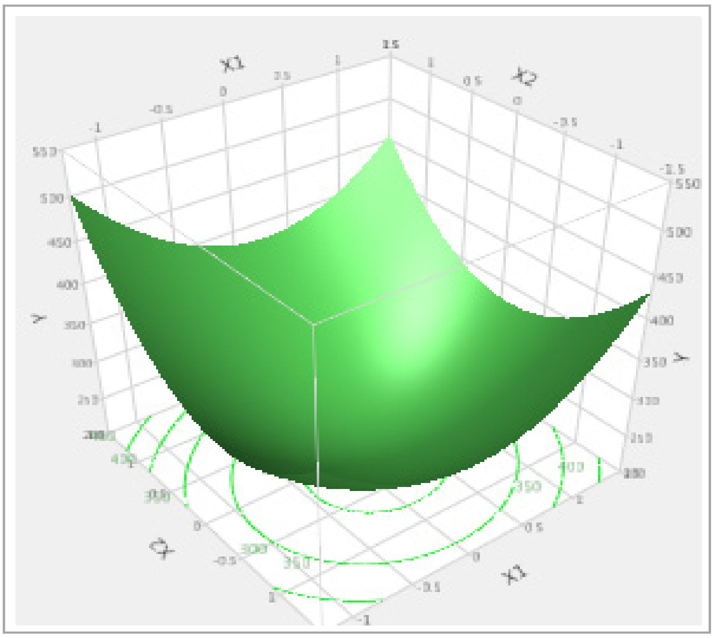
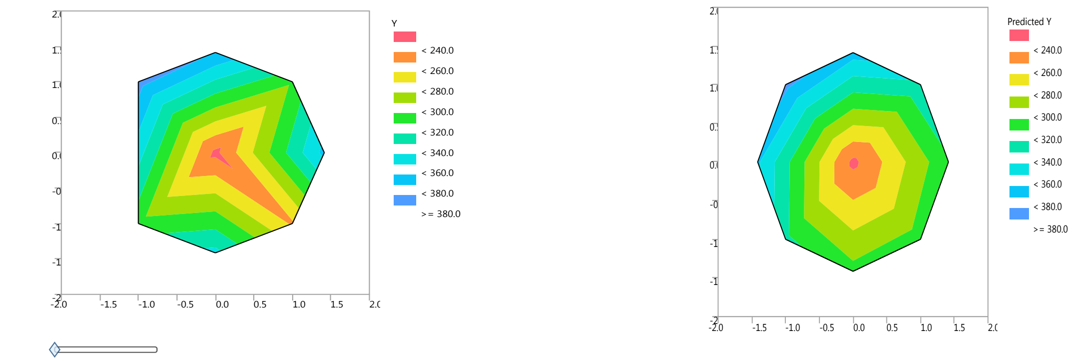


Fig. 8. 3D plot for the results.



(a) (b)

Fig. 9. (a) Contour Plot for response Y (compressive strength) actual values, and (b) predicted values.

Well dispersion of NS Particles and Multi walled CNTs were observed for the combination between 1% NS and 0.03% multi walled CNT, as shown in Fig. 10. The matrix was observed to be denser and contained fewer voids as compared to plain cement matrix.

High agglomerates were observed for the combination close to the central point proportions (1.5% NS and 0.02% multi walled CNT), as shown in Fig. 10. The performance of the NS Particles with the Multi walled CNTs during the mixing period as it can be seen from the TEM Fig. 11 is highly different when a mono dispersed multi walled CNT particle found the Nano silica got attached to the Nano tube in a manner that helps the bonding between the Nano tubes and the cement matrix, while when agglomerated the Nano silica particles got trapped between the multi walled CNT particles and a huge agglomerated particle found which act as a voided area within the matrix and consequently the compressive strength decreased.

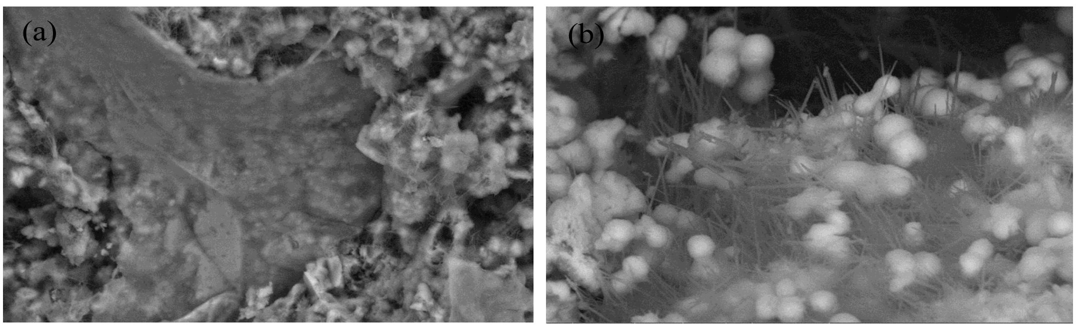


Fig. 10. (a) micrograph of well dispersed CNTs, and b) micrograph of agglomerated CNTs and NS particles.

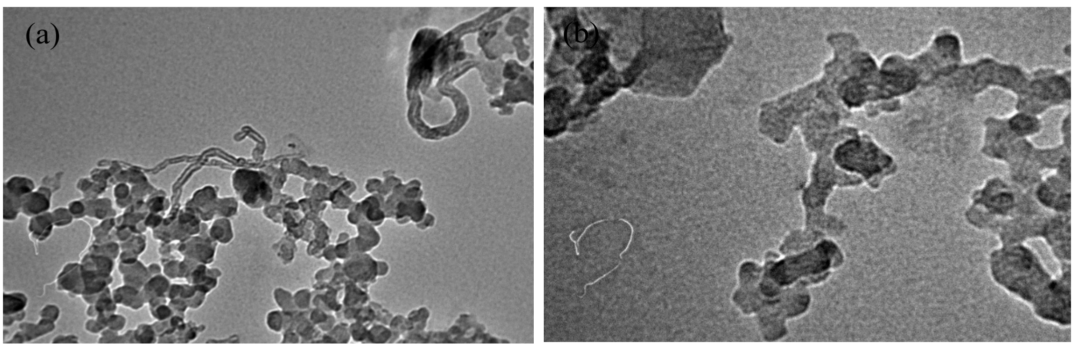


Fig. 11. (a) agglomerated hybrid, and(b)versus well attached NS particles to CNTs

Conclusion

The combination of NS with high amount of CNT had a positive effect on the hydration reaction, the presence of individual Multi walled CNTs worked as extra nucleation sites for the hydrates and enhanced the activity of the NS particles. While the combination of low amount of Multi walled CNTs and the same amount of NS Percentage had negative effect on the hydration reaction compared to the mixes with higher hybrid content. NS Particles sucks water between its particles and affects negatively the production of hydrates of the cement due to its re-agglomeration. The generated re-agglomeration of Multi walled CNTs and decreased the availability of $Ca(OH)_2$ for the NS Particles to react with and produce more C-S-H. Finally, as for the statistical analysis, the response surface model showed to represent the Muti walled CNTs – NS Particles coupled interaction effectively, where the compressive strength increases in a parabolic shape where the central mix acts as the base point of the paraboloid at its lowest compressive strength value.

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تعظيم الاستفادة من مركب الأسمنت الهجين مع أنابيب الكربون النانومترية ونانو السيليكا باستخدام طريقة تصميم سطح الاستجابة

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الخيار الأكثر شيوعًا لايجاد نموذج من الدرجة الثانية في طريقة سطح الاستجابة هو التصميم المركب المركزي. منهجية سطح الاستجابة المركب المركزي تم اختيارها من أجل التحقيق في أداء مونة الاسمنت لتصميم القائمة على الأسمنت البورتلاندي الذي يتم فية دمج جزيئات نانو السيليكا (NS) مع أنابيب نانو كربونية متعددة الجدران (الأنابيب النانوية الكربونية) بنسب مختلفة كهجين. التقييم المقترح لجزيئات نانو السيليكا NS الهجينة المستخدمة مع وحدات الكربون CNT متعددة الجدران داخل مصفوفة الأسمنت سيتم عبر طريقة استجابة السطح ، أظهرت النتائج أن الجمع بين نانو السيليكا NS مع كمية عالية نسبيا من CNTs متعدد الجدران (0.04 ٪) كان لها تأثير إيجابي على المقاومة. كذلك وجود CNTs متعدد الجدران ادى الى تعزيز نشاط نانو السيليكا NS عند استخدام هذه النسب. في حين أن الجمع بين كمية أقل من CNTs متعدد الجدران في وجود نانو السيليكا NS كان له تأثير سلبي على المقاومة. جزيئات النانو سيليكا المتكتلة تمتص الماء بين جزيئاتها وتؤثر سلبا على إنتاج هيدرات الأسمنت بسبب التكتل. وأخيرا ، بالنسبة للتحليل الإحصائي ، سطح الاستجابة أظهر من خلال النموذج أن مزيج من CNTs و نانو السيليكا NS تفاعلت بشكل فعال لتعزيز الخواص الميكانيكية لمونة الأسمنت و أعطت نموذج احصائي من الدرجة الثانية يمكن الاعتماد عليه في توقع مقاومة الخلطات الهجينة من CNTs و نانو السيليكا.