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The Coupled Effect of Nano Silica and Superplasticizer on Concrete Fresh and Hardened Properties

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Abstract: *The The effect of individual and combined additions of both nano silica (NS) and polycarboxylate-ether superplasticizer (SP) admixtures on concrete mixes were studied. Twenty concrete mixes were prepared, keeping the water/cement ratio constant at w/c = 0.40, with different amounts of SP admixture, and NS. the superplasticizer was studied in the range of (0 – 0.88)% (over cement weight), nano silica additions were tested at (0 – 3.00)% range (over cement weight). Early, and late compressive strengths, as well as results of fresh concrete (slump) test of formulations were evaluated by means of design of experiments, in order to identify the primary factors and their interactions on the measured properties. The most desirable NS and SP % were determined. The results showed that regardless of the used NS percentage, the higher compressive strength results were reached at, or around SP observed saturation dosage (0.66% by weight cement). The statistical approach applied here enabled to develop relations, which could adequately describe the dependency of both the compressive strength, and slump on the composition of NS, and SP concretes. These relations were presented as contour plots, which from a practical point of view could serve as a basis for mix design.*
Keywords: *full factorial design, nano silica, superplastecizer*

I. INTRODUCTION

Recently, nanotechnology has attracted considerable scientific interest due to the new potential uses of particles in nanometer scale. Thus industries may be able to re-engineer many existing products that function at unprecedented levels. Due to these developments in nano science and technology, various forms of nano-sized amorphous silica have become available. As these materials have higher specific surface area compared to silica fume, a considerable research effort has been attracted to investigate the influence of nano silica on the properties of cement based materials.

Although numerous papers have studied the influence of nano silica on the properties of cement composites, their effects have not been adequately characterized yet, and some discrepancies and inconsistencies in compressive strength, and workability results are witnessed. [1-5].

Since the major problem in utilizing Nano-particles is that they are highly agglomerated and if used directly in a bulk composite, they often lose their high-surface area due to grain growth. Effective means of de-agglomerating and dispersing are needed to overcome the bonding forces after wetting the powder; the ultrasonic breakup of the agglomerate structures in aqueous and non-aqueous suspensions allows utilizing the full potential of nano-sized materials, besides the addition of proper chemical dispersing admixtures.

Currently, the extended use of superplasticizer (SP) improves the workability of grouts and concretes mixtures. Superplasticizers are adsorbed onto the surface in order to de-flocculate cement particles, which release trapped water from cement flocks [6]. The mechanism behind SP mortars and concretes is associated with the adsorption of molecules onto particles surfaces, causing mainly electrostatic (e.g., polynaphtalin-sulphonate) or electrostatic/steric (polycarboxylic-ether) repulsive forces [7,8]. In this sense, it is known that mix designs containing silica as cement replacement increase the strength due to a pozzolanic and filler effect [9]. However, the compaction necessary to produce a mixture as homogeneous as possible is directly related to its rheological behavior, which depends on the chemical compatibility between the materials. In fact, the presence of mineral admixtures (MAs) in conjunction with SP additions affects the workability in the fresh state of grouts and concretes [6]. This motivates the study of optimal levels of superplasticizers and water/binder (w/b) ratios that produce an adequate balance between strength and workability. There are several qualitative and quantitative methods that can be used for these purposes. In a previous work [10], the rheological behavior of plain and micro-SiO2 (SF) additions of plasticized cement grouts was studied based on the Marsh cone test (MCT). [11]

This study aims to estimate the optimal dosage levels of SP and w/b ratio for and nano-SiO2 (nS) additions. The program addresses the relationship between the fresh and hardened states in concrete incorporating nano silica, and SP in diverse dosages.

Typically, a trial and error approach is followed which consists in selecting and testing a first trial batch, evaluate the results, and then adjust the mixture proportions, based on deducted relationships between the mixture parameters [12] and existing knowledge or recommendations [13] and, finally, re-test the adjusted mixture.

This process is repeated until the required properties are achieved, which may involve carrying out a large and unpredictable number of trial batches. Besides, this optimization technique may not lead to a general solution of the problem. In contrast, statistical experimental design is a more scientific and efficient approach for establishing an optimized mixture for a given constraint, while minimizing the number of experimental data points [14]. Models established on the basis of a factorial design highlight not only significance of the experimental variables but also that of their interactions.

These models are valid for a wide range of mix proportioning and have a predictive capability for the responses of other points located within the experimental domain. This design approach was followed by other authors for various purposes, namely, to design and optimize the mixtures, to compare the responses obtained from various test methods, to analyze the effect of changes in mixture parameters (to evaluate SCC mixture robustness) and to evaluate trade-offs between key mixture parameters and constituent materials (for example, superplasticizer and viscosity agent) [14–21].

The influence of different combinations of nano silica-binder (NS/b), and superplasticizer-binder (SP/b) ratios on the workability (slump test) and mechanical properties (compressive strength) of 20 concrete mixtures of constant water-binder ratio (w/b) were investigated by means of design of experiments. The effects of studied parameters were characterized and analyzed using ANOVA and regression models, which can identify the primary factors and their interactions on the measured properties.

II. EXPERIMENTAL PROGRAM

2.1. Materials

Ordinary Portland Cement (OPC) conforming to ASTM C150 standard was used as received. Chemical and physical properties of used cement are given in Table 1. SiO2 nano particles with average particle size of 30 nm and 45 m2/g Blaine fineness produced from WINLAB laboratory chemicals, UK was used as received. The properties of SiO2 nano particles are shown in Table 2. Transmission electron micrographs (TEM) and powder X-ray diffraction (XRD) diagrams of SiO2 nano particles are shown in Figs. 1a,b. Crushed limestone aggregates, as well as sand free of alkali-reactive materials were used to insure producing durable Concretes; the aggregates were mixed by percentages of 65% for coarse aggregate, and 35% for fines by volume. A polycarboxylate with a polyethylene condensate de-foamed based admixture (Glenium C315 SCC) was used. Table 3 shows some of the physical and chemical properties of polycarboxylate admixture used in this study.

2.2. Samples preparation

A total of 20 mixtures were prepared as shown in table 4. Sets of 6 cubes (15*15*15 cm3) were cast to perform compression strength tests after 7, and 28 days of water curing. Cubes were consolidated in accordance to ASTM C 192 in three layers on a vibrating table, where each layer was vibrated for 10 seconds, and then the specimens were de-molded after 24 hours and cured in normal free water at room temperature until the day of testing. Five SP percentages are used in order to perform the mentioned investigation; 0%, 0.44%, 0.66%, 0.73%, and 0.88%, while four NS substitution percentages are used in parallel; 0%, 1%, 2%, and 3%. The constituents of the 20 mixtures are presented in table 4. Preparation of mixtures was performed in the following sequence: (a) Weighing components, (b) mixing the solid components inside a turn tilt mixer for 1 min, (c) adding sonicated nano silica with a portion of water and mixing for 1 min, (d) adding superplasticizer into the rest of water for helping dispersing the nano silica, and (e) finally mechanical mixing for 2.5 min.

III. RESULTS AND DISCUSSIONS

3.1. Effect of changing SP% on slump of different nano silica concrete mixes

- Saturation dosage of superplasticizer can be defined as the dosage beyond which higher contents of superplasticizer do not increase the slump value significantly.
- From figures 2 (A-D) we can conclude a saturation point of 0.66% by weight cement for all conducted mixes, either those with nano silica contents, or the plain mix.
- Increasing the superplasticizer dosage beyond the saturation point induced substantial bleeding and segregation for the control, and the 1% nano silica (NS1) mixes, while by increasing the nano silica addition above 1% (mixes NS2,and NS3), the nano silica acted as anti-bleeding and neither bleeding nor segregation occurred, this can be attributed that by increasing nano silica content , more silica particles got adsorbed on the ettringite phase of cement hydration and so more un adsorbed polymers are found free and thus resistance occurring when two neighboring polymers approach each other, this resistance increase with the increase of superplasticizer beyond the saturation point where the un adsorbed polymers increases thus increasing viscosity. As mentioned by (yamada et.al 1998) [22].
- At low superplasticizer doses, as the nano silica addition increases the slump results decreased and this can be attributed to the increase of the attractive forces that are predominant over the hydrodynamic forces exerted by the flow field and, therefore, the formation of aggregations takes place.
- While by increasing the dosage the hydrodynamic forces become higher and overcome the attractive inter-particle forces leading to breakdown of aggregations into small particles. Consequently, the liquid entrapped within aggregations is gradually released, thus increasing workability. [23]

3.2. Effect of changing SP% on compressive strength of different nano silica concrete mixes

From figures 3a-d we can conclude that regardless of the nano silica percentage used, the higher compressive strength results were reached at, or around saturation dosage (0.66%), this finding was also mentioned by [23]

The highest compressive strength result of all 20 mixes were reached using 3% NS, and 0.73% SP. The early and late compressive strengths were 540 kg/cm2, and 634 kg/cm2 respectively. The use of 3% NS, and 0.73% SP increased the late compressive strength by 135% than the non plasticized control mix. No matter the nano silica % used is the early strength results proved to be highly correlated to the late strength results with R2 values exceeding 95% for all mixes as it can be seen from figures 4 a-d, such findings were previously discussed in sections. The concrete fresh and hardened behavior cannot be predictable when SP saturation dosage is exceeded. We can finally conclude that to ensure adequate concrete fresh and hardened behavior, a percentage close to superplastesizer's saturation point should be used.

3.3. Regression model correlating NS, and SP percentage with the compressive strength and slump of concrete mixes

Based on the above mentioned results, and conclusions, the effects of studied parameters were characterized and analyzed using ANOVA and regression models, which can identify the primary factors and their interactions on the measured properties. To find out the best possible mixture under the condition of this research concept for the desired workability, and mechanical characteristics, a multi-objective optimization problem was defined and solved based on developed regression models.

Statistical design of experiments can be used for optimization of linear and non-linear systems. When non-linear effects and interactions of several different variables (factors) are anticipated, factorial designs provide the minimum number of experiments needed to investigate those effects and combine them into a property response model. the chosen k factors are set at four different levels, a 4k factorial design is created and the properties are evaluated at all combinations of those k factors and levels. A regression polynomial is then fitted to the experimental values obtained and the model is considered valid only when the differences between the experimental and the calculated values (error) are uncorrelated and randomly distributed with a zero mean value and a common variance. In what follows, the effect of NS, and SP % on the 28 days compressive strength, and slump results will be discussed statistically and the optimum percentages will be determined.

3.3.1. Evaluation of Compressive Strength Results

Based on the conducted experimental program, the following results can be drawn:

3.3.1.1. Conditional sums of squares, and analysis of variance:

The analysis of variance (ANOVA) table 5A, 5B, and 5C decomposes the total variability in the dependent variable into two components: one due to the regression, and the second due to deviations around the fitted model. 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 94% 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 25.26. Since the P value corresponding to the F-ratio is less than 0.05, the model as a whole is statistically significant.

3.3.1.2. Estimation of coefficients, test of significance and confidence intervals:

The estimated coefficients for the multiple regression model are shown in Table 5C. The P values correspond to tests of the hypotheses that the coefficients are equal to zero. Values of P less than 0.05 indicate statistically significant non zero coefficients at a 95% confidence level.

The proposed equation is

28 DAYS COMPRESSIVE STREGNTH (Kg/cm2) =

{296.88 + (300.92*SP) + (34.87*NS) -15.37*(NS-1.533)*(NS-1.533)}

3.3.1.3. Test for outliers and unusual residuals:

Fig.5a shows the Standardized residuals as a function of the predicted values. The residuals show the difference between the actual values and the predictions, and the Standardized residuals, express each deviation in terms of how many deviations it is away from the fitted line. The Standardized residuals that were calculated are based on the estimated residual standard deviation if the fitting were performed without that data value. This kind of residuals is particularly useful for detecting outliers (i.e. points that do not follow the same pattern as the others). As can be seen in Fig. 5a, the plot appears reasonably random, and none of the residuals is noticeably distinct from the others. The normal probability plot the residuals, shown in Fig. 5b, can be used to judge whether the residuals could reasonably be considered to follow a normal distribution, and may also be helpful in detecting outliers. The residuals fall fairly well along a straight line, while no outliers can be observed.

3.3.1.4. Compressive strength (actual/predicted) design charts:

Figure 6 introduces helpful design chart correlating NS, and SP percentages with actual and predicted compressive strengths respectively. The NS percentages for the predicted results were chosen to be from 0% to 6% from total binder content.it should be noted that results are constrained with the proposed experimental concrete mix. Finally based on the proposed mix constituents, and without exceeding the SP saturation dosage, the most desirable NS, and SP percentages were introduced in figure 7, as well as the corresponding predicted compressive strength value.

3.3.2. Evaluation of Slump Results:

Based on the conducted experimental program, the following results can be drawn:

3.3.2.1. Conditional sums of squares, and analysis of variance:

The analysis of variance (ANOVA) table 6A, 6B, and 6C decomposes the total variability in the dependent variable into two components: one due to the regression, and the second due to deviations around the fitted model. 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 90% 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 2.20. Since the P value corresponding to the F-ratio is less than 0.05, the model as a whole is statistically significant.

3.3.2.2. Estimation of coefficients, test of significance and confidence intervals:

The estimated coefficients for the multiple regression model are shown in Table 6C. The P values correspond to tests of the hypotheses that the coefficients are equal to zero. Values of P less than 0.05 indicate statistically significant non zero coefficients at a 95% confidence level.

The proposed equation is

Slump (cm) = 5.495 + (23.016*SP) – (1.35*NS)

3.3.2.3. Test for outliers and unusual residuals:

Fig. 8a shows the Standardized residuals as a function of the predicted values. The residuals show the difference between the actual values and the predictions, and the Standardized residuals, express each deviation in terms of how many deviations it is away from the fitted line. The Standardized residuals that were calculated are based on the estimated residual standard deviation if the fitting were performed without that data value. This kind of residuals is particularly useful for detecting outliers (i.e. points that do not follow the same pattern as the others). As can be seen in Fig.8a, the plot appears reasonably random, and none of the residuals is noticeably distinct from the others. The normal probability plot the residuals, shown in Fig 8b, can be used to judge whether the residuals could reasonably be considered to follow a normal distribution, and may also be helpful in detecting outliers. The residuals fall fairly well along a straight line, while no outliers can be observed.

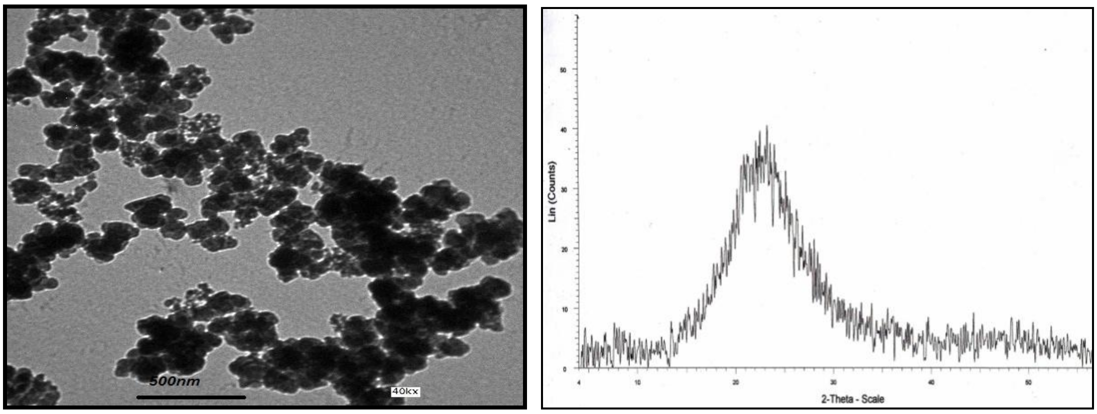
3.3.2.4. Slump (actual/predicted) design charts:

Figure 9 introduces helpful design chart correlating NS, and SP percentages with actual and predicted slump respectively. The NS percentages for the predicted results were chosen to be from 0% to 6% from total binder content. it should be noted that results are constrained with the proposed experimental concrete mix.

Finally based on the proposed mix constituents, and without exceeding the SP saturation dosage, the most desirable NS, and SP percentages were introduced in figure 10, as well as the corresponding predicted slump value.

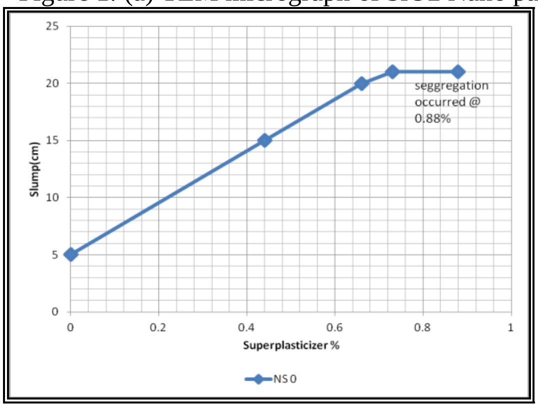
IV. Figures And Tables

4.1. Figures:

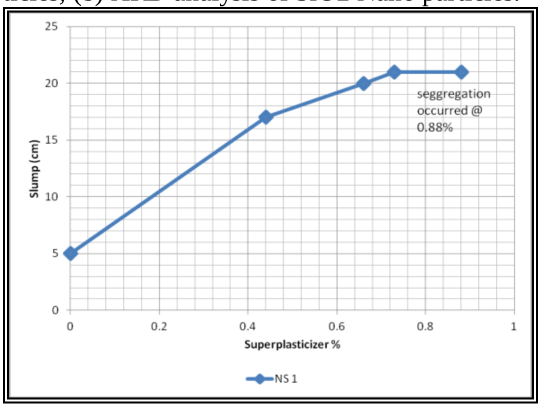


(a) (b)

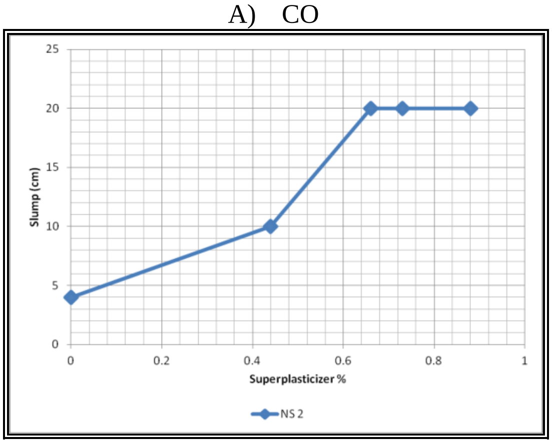
Figure 1: (a) TEM micrograph of SiO2 Nano particles, (b) XRD analysis of SiO2 Nano particles.



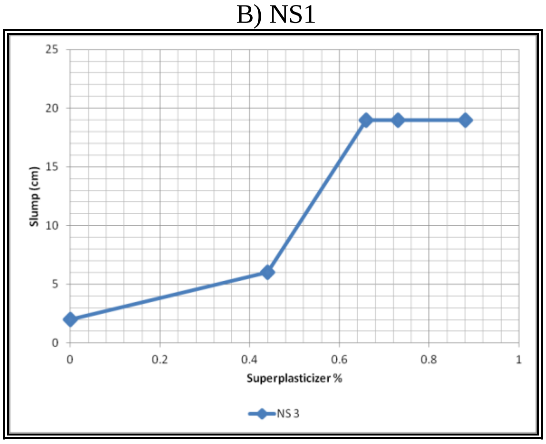
A) CO



B) NS1

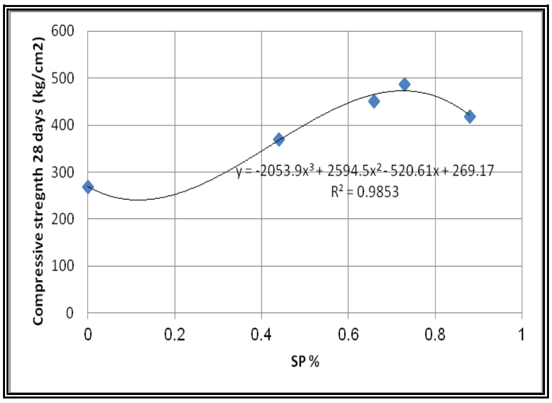


C) NS2

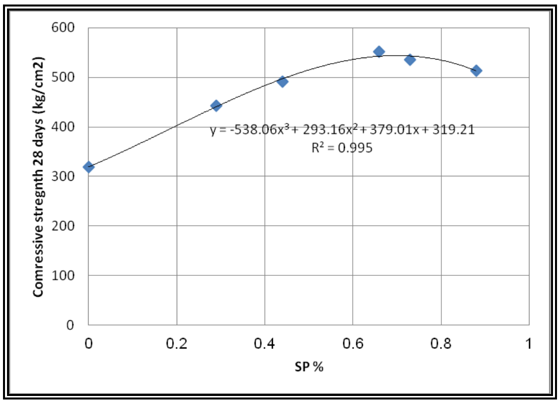


D) NS3

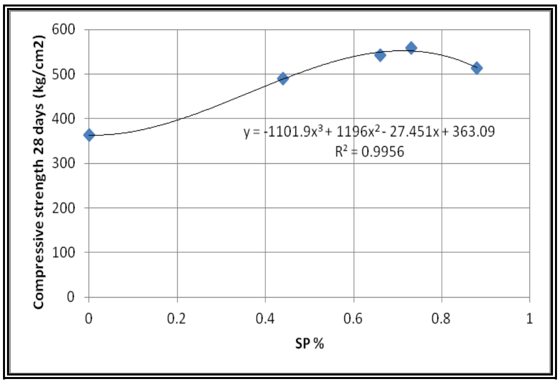
Figure 2: Effect of increasing SP on the slump of different NS concrete.



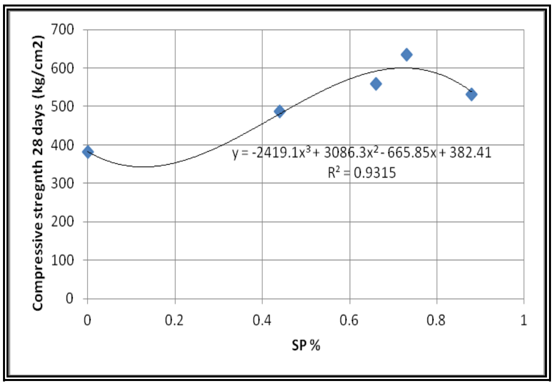
A: CO



B: NS1

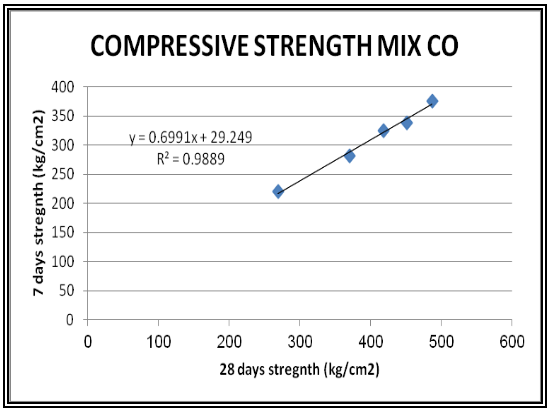


C: NS2

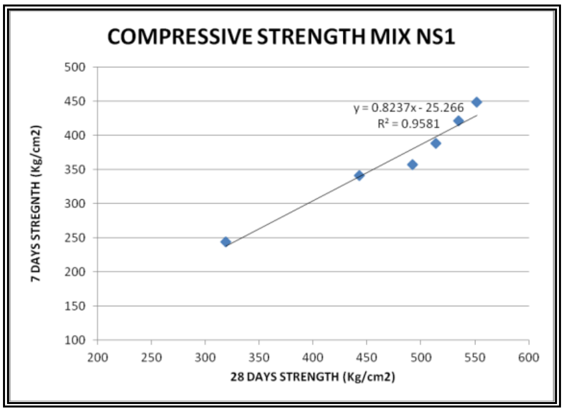


D: NS3

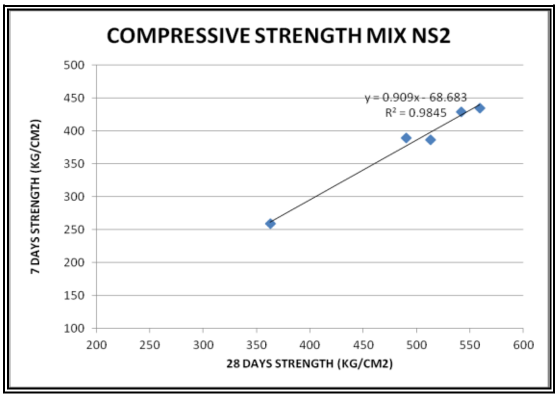
Figure 3: Effect of increasing SP on the 28 days compressive strength of different NS concrete.



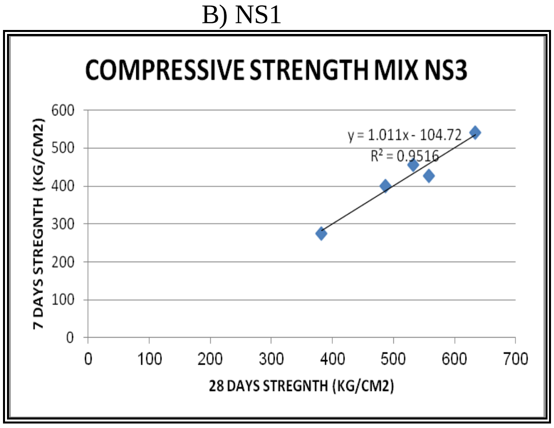
A) CO



B) NS1

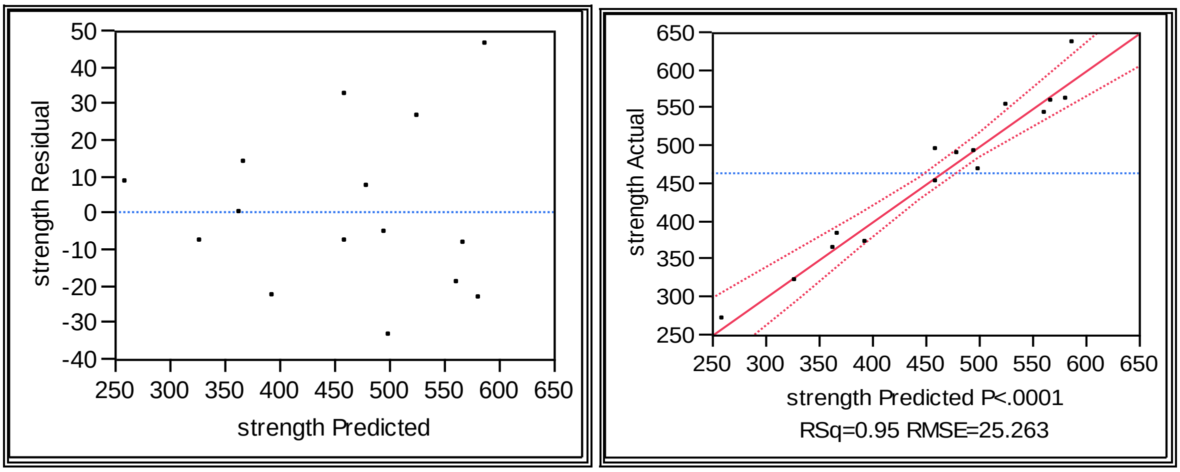


C) NS2



D) NS3

Figure 4: correlation between early and late strengths with the change of SP %.



A) B)

Figure 5: compressive strength A) Residual by Predicted Plot, B) Whole Model Actual by Predicted Plot

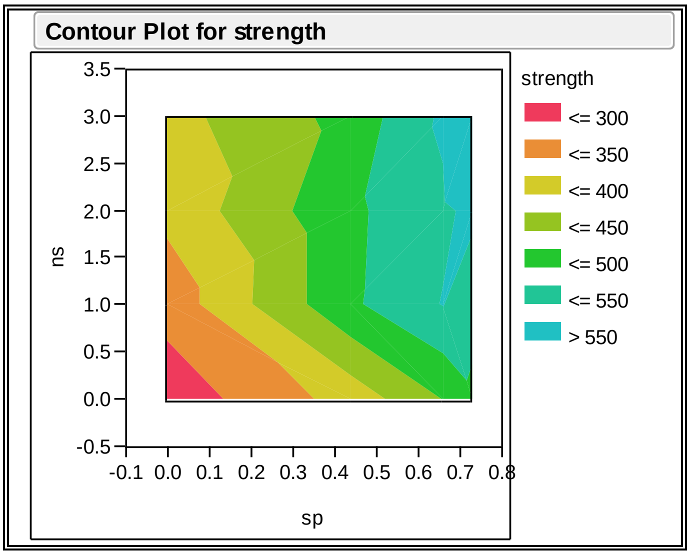


Figure 6: Contour plot correlating NS and SP with compressive strength actual results.

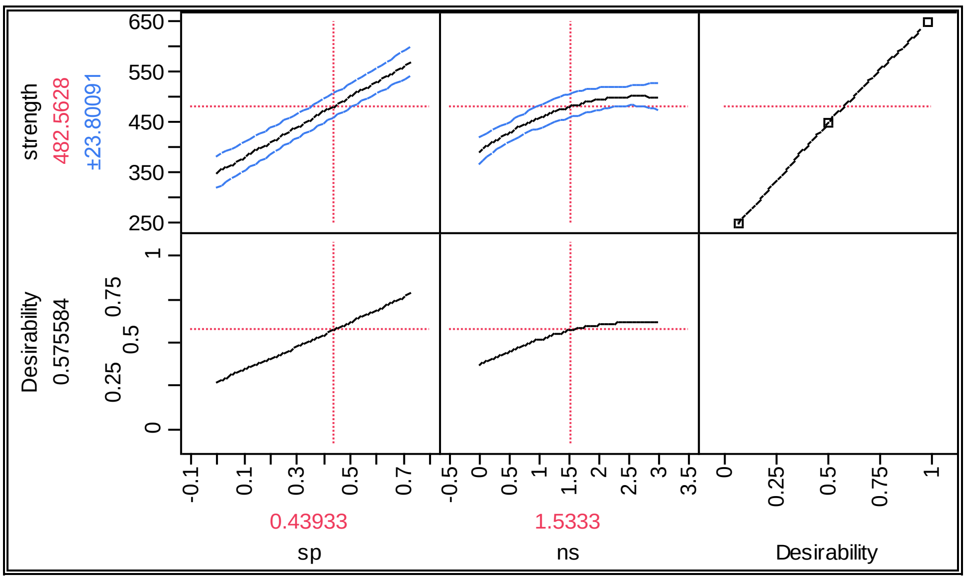


Figure 7: compressive strength most desirable NS, and SP percentages.

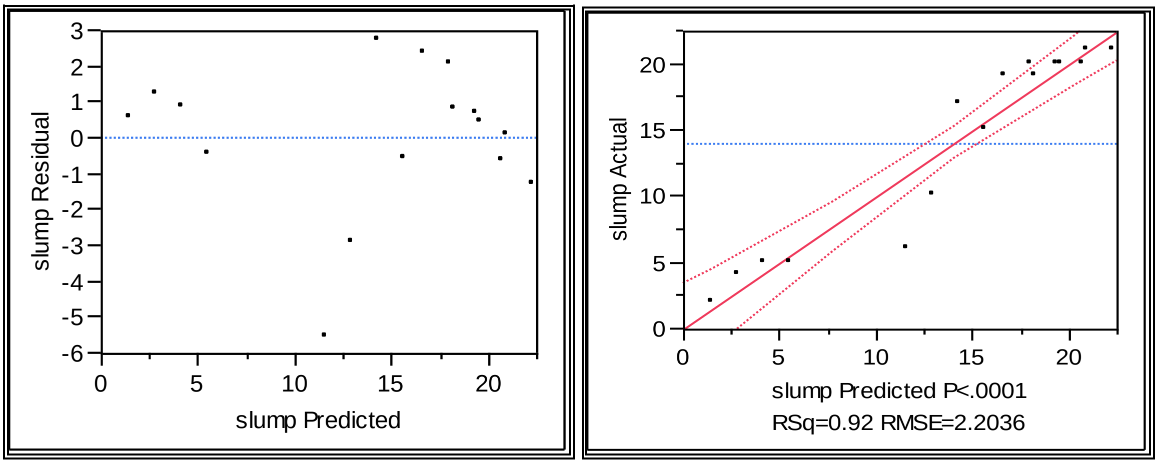


Figure 8: slump A) Residual by Predicted Plot, B) Response Whole Model Actual by Predicted Plot

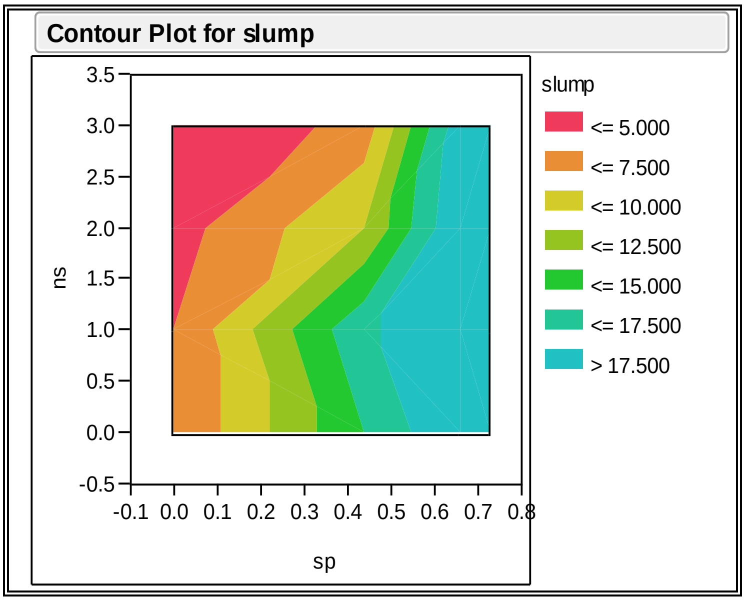


Figure 9: Contour plot correlating NS and SP with slump actual results.

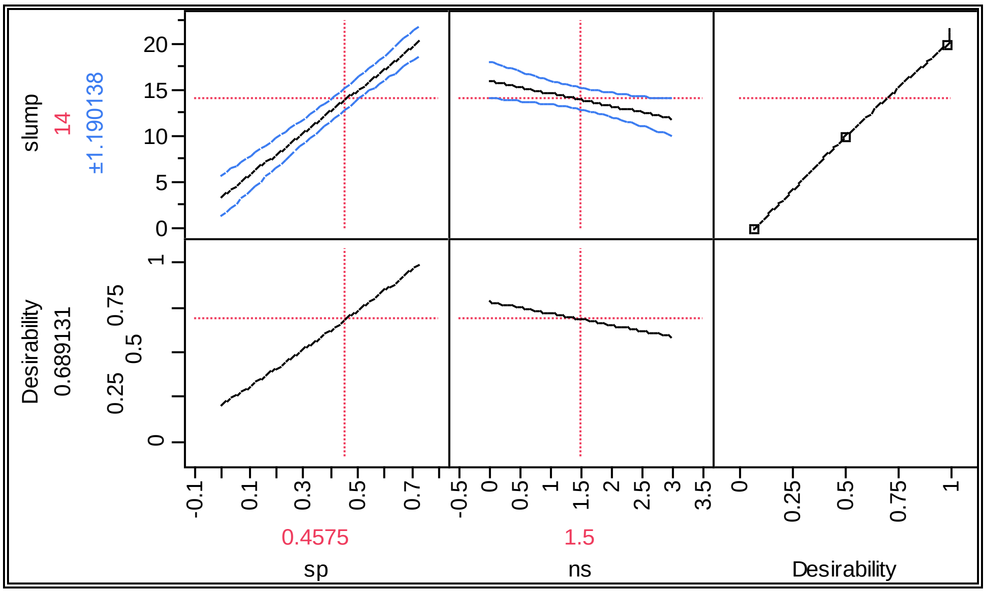


Figure 10: slump most desirable NS, and SP percentages.

The SI derived unit for **pressure** is the pascal. 1 pascal is equal to 1.01971621298E-5 kg/cm2.

4.2 Tables:

Table1. Properties of Portland cement (wt%).

Element	Sio2	Al2O3	Fe2O3	CaO	MgO	SO3	Na2O	K2O	L.O.I
Cement	20.13	5.32	3.61	61.63	2.39	2.87	0.37	0.13	1.96

Table2. Chemical composition of Nano SiO2 (wt %).

Element	SiO2	Fe2O3	Al2O3	MgO	CaO	Na2O	P2O5
NS	99.17	0.06	0.13	0.11	0.14	0.40	0.01

Table3. Physical and chemical characteristics of the polycarboxylate admixture.

Appearance	Off white opaque liquid
Specific gravity @ 20°C	1.095 ± 0.02 g/cm3
PH-value	6.5 ± 1
Alkali content (%)	Less than or equal to 2.00
Chloride content (%)	Less than or equal to 0.10

Table 4: mixtures components (kg) per 1 m^3.

MIX	CEMENT	AGGREGATE		WATER	S.P.	N.S.
		COARSE	FINE			
CO	480	1109	597	195	0	0
CO44	480	1109	597	195	2.11	0
CO66	480	1109	597	195	3.16	0
CO73	480	1109	597	195	3.50	0
CO88	480	1109	597	195	4.22	0
S1	475.2	1109	597	195	0	4.8
S144	475.2	1109	597	195	2.11	4.8
S166	475.2	1109	597	195	3.16	4.8
S173	475.2	1109	597	195	3.50	4.8
S188	475.2	1109	597	195	4.22	4.8
S2	470.4	1109	597	195	0	9.6
S244	470.4	1109	597	195	2.11	9.6
S266	470.4	1109	597	195	3.16	9.6
S273	470.4	1109	597	195	3.50	9.6
S288	470.4	1109	597	195	4.22	9.6
S3	465.6	1109	597	195	0	14.4
S344	465.6	1109	597	195	2.11	14.4
S366	465.6	1109	597	195	3.16	14.4
S373	465.6	1109	597	195	3.50	14.4
S388	465.6	1109	597	195	4.22	14.4

Where: S.P: "super plasticizer", N.S: "nano silica

Table 5: Compressive strength statistical results:

5.A. summary of Fit

Response	Value
RSquare	0.952413
RSquare Adj	0.939435
Root Mean Square Error	25.26265
Mean of Response	462.3333
Observations (or Sum Wgts)	15

5.B. Analysis of Variance

F Ratio	Mean Square	Sum of Squares	DF	Source
73.3860	46835.0	140505.12	3	Model
Prob > F	638.2	7020.22	11	Error
<.0001		147525.33	14	C. Total

5.C. Parameter Estimates

Prob>|t|	t Ratio	Std Error	Estimate	Term
<.0001	17.33	17.13187	296.88647	Intercept
<.0001	13.12	22.93379	300.92451	sp
<.0001	6.09	5.723765	34.873244	ns
0.0388	-2.35	6.556148	-15.37876	(ns-1.53333)*(ns-1.53333)

Table 6: Slump statistical results

6.A. Summary of Fit

Response	Value
RSquare	0.919892
RSquare Adj	0.907568
Root Mean Square Error	2.203584
Mean of Response	14
Observations (or Sum Wgts)	16

6.B. Analysis of Variance

F Ratio	Mean Square	Sum of Squares	DF	Source
74.6404	362.437	724.87486	2	Model
Prob > F	4.856	63.12514	13	Error
<.0001		788.00000	15	C. Total

6.C. Parameter Estimates

Prob>|t|	t Ratio	Std Error	Estimate	Term
0.0009	4.30	1.277445	5.4949307	Intercept
<.0001	11.91	1.933042	23.016545	Sp
0.0169	-2.74	0.492736	-1.35	Ns

V. SUMMARY AND CONCLUSIONS

The influence of different combinations of nano silica-binder (NS/B), and superplasticizer-binder (SP/B) ratios on the workability (slump test) and mechanical properties (compressive strength) of 20 concrete mixtures of constant water-binder ratio (W/B) were investigated by means of design of experiments. The effects of studied parameters were characterized and analyzed using ANOVA and regression models, which can identify the primary factors and their interactions on the measured properties. From the results obtained, the following conclusions can be drawn:

- A SP saturation point of 0.66% by weight cement was recognized for all conducted mixes, either that with nano silica contents, or the control mix.
- Increasing the superplasticizer dosage higher than the saturation point induced substantial bleeding and segregation for the control, and the 1% nano silica (NS1) mixes, while by increasing the nano silica addition above 1% (mixes NS2,and NS3), the nano silica thought to be acted as anti-bleeding and neither bleeding nor segregation occurred.
- At low dosages of superplasticizer, as the nano silica addition increases the slump results decreased and this can be attributed to the increase of the attractive forces that are predominant over the hydrodynamic forces exerted by the flow field and, therefore, the formation of aggregations takes place.
- While by increasing the dosage the hydrodynamic forces become higher and overcome the attractive inter-particle forces leading to breakdown of aggregations into small particles. Consequently, the liquid entrapped within aggregations is gradually released, thus increasing workability.
- Regardless of the nano silica percentage used, the higher compressive strength results were reached at, or around SP saturation dosage (0.66% by weight cement).
- The highest compressive strength results of all 20 mixes were reached using 3% of NS and 0.73% of SP by weight cement. The early and late compressive strengths were 540 kg/cm2, and 634 kg/cm2 respectively. The use of 3% NS, and 0.73% SP increased the late compressive strength by 135% than the non plasticized control mix.
- No matter the nano silica percentage used is the early strength results proved to be highly correlated to the late strength results with R2 values exceeding 95% for all mixes.
- The concrete fresh and hardened behavior cannot be predictable when SP saturation dosage is exceeded. We can conclude that to ensure adequate concrete fresh and hardened behavior, a percentage close to superplastesizer's saturation point should be used.
- The proposed regression models, correlating the NS, and SP % with both the compressive strength, and slump results proved to be highly significant.
- 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 compressive strength model accounts for 94% of the variation of the mean size percentage to the origin material.
- The mean squared error estimates the variance of the deviations around the compressive strength model to be equal to 25.26. Since the P value corresponding to the F-ratio is less than 0.05, the model as a whole is statistically significant.
- 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 slump model accounts for 90% of the variation of the mean size percentage to the origin material.
- The mean squared error estimates the variance of the deviations around the compressive strength model to be equal to 2.2. Since the P value corresponding to the F-ratio is less than 0.05, the model as a whole is statistically significant.
- The relationships calculated using statistical analysis of experimental results can be used as guidelines in the design of superplastecized concrete mixtures incorporating nano silica particles.
- The results obtained show that, for all properties investigated, the error of the estimate calculated using the relevant model is low when compared with the corresponding experimental value, which validates the calculated models.
- The statistical approach applied here enabled to develop relations, which could adequately describe the dependency of both the compressive strength, and slump on the composition of NS, and SP concretes. These relations were presented as contour plots, which from a practical point of view could serve as a basis for mix design.

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