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Journal of Materials Research and Technology
www.jmrt.com.br



Original Article

The effect of nano-silica and waste glass powder on mechanical, rheological, and shrinkage properties of UHPC using response surface methodology

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ARTICLE INFO

Article history:

Received 8 March 2018

Accepted 23 June 2018

Available online xxx

Keywords:

Response surface methodology

Ultra high performance concrete

Compressive strength

Flowability

Drying shrinkage

Central composite method

Waste glass powder

Nano-silica

ABSTRACT

This study provides the experimental and statistical modeling in order to increase the performance of ultra high performance concrete (UHPC) within reducing the cement consumption. Also showing the effect of waste glass powder and nano silica fume on mechanical, rheological, and shrinkage properties. For this purpose, a fraction of binder was added with the range of 0–5% of nano-silica fume and fraction of Portland cement were substituted with 0–20% of waste glass powder, the maximum particle size of 63 μm . The mechanical properties were obtained by testing on 28 days compressive strength. The rheological property found by doing the flow test. Numbers and randomization orders of experiments were designed by central composite face-centered (CFC) and modeled by response surface methodology (RSM). The validity of models was controlled by analysis of variance (ANOVA). The study showed adding nano-silica and waste glass powder and especially their interaction improved the properties of UHPC.

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1. Introduction

Ultra high performance concrete (UHPC) contains the high amount of cement. This issue places this type of concrete in “unfriendly environmentally manufacturing product” category [1]. On the other hand, the amount of water in the

mixture is very low which both together lead to early hydration and consequently, the higher amount of shrinkage, comparing to conventional concrete. Khatri et al. [2] stated that replacing 10% of silica fume with cement in high strength concrete raised the shrinkage at the early age.

Mixtures which were prepared by nano-silica binder have lower workability comparing to plain mixtures. It is believed

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<https://doi.org/10.1016/j.jmrt.2018.06.011>

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because of high water absorption of nano-silica particles, on the other hand, the addition of nano-silica reduces the initial and final setting time of binder because of the additional surface which is provided by nano silica particles and leads to acceleration of hydration [3–5].

Million tons of glass is being dumped every year all over the world. Although it is a recyclable material once it is mixed in different colors, it becomes useless for recycling [6,7].

More than 75% of glass is basically composed of silica and due to a chemical component, the glass could be a suitable material to be used as a replacement of cement in concrete [8]. When the glass is pulverized up to microparticle sizes, it performs pozzolanic reaction and leads to the formation of the high amount of C–S–H productions in cementitious mixtures [9,10].

Ultra high performance glass concrete (UHPGC) is an environmentally friendly version of ultra high performance concrete which provides economical, technical and environmental advantages due to the replacement of glass powder with a part of cement in concrete [11]. Soliman [11] modified UHPC by using glass powder as cement replacement and found that replacing glass powder up to 20% accelerated the hydration process and developed the mechanical properties of this kind of concrete.

Some researchers studied the effects of glass powder on the workability of cementitious materials and they all mentioned that replacing of glass powder with cement increases the slump due to the surface structure of glass powder particles which were crystalize and smoother than cement particles and absorbs less water comparing to cement [9,12–14].

Shilpa [15] showed that glass powder performs pozzolanic reactivity when it pulverizes and particle sizes become less than 75 μm which leads to increase compressive strength. Generally the size of glass powder particles has a great role in effects on mechanical properties of concrete, especially, compressive strength. Many researchers studied the effect of different particle sizes of glass powder on compressive strength of concrete, they showed that glass powder within the range of 30–300 μm has the better reaction with cement particles and production of C–S–H increases due to pozzolanic reactivity of glass powder which finally leads to enhance compressive strength [16–19].

Glass powder affects the durability of concrete by affecting on drying shrinkage. Mixtures containing glass powder show higher drying shrinkage comparing to mixtures without glass powder [20,21].

Response surface methodology (RSM) is a combination between mathematical and statistical techniques which is used for modeling and analyzing several parameters which gives a good interpretation by finding the individual contribution of variables, curvature, and possibly interactive effect between those to achieve the optimum responses [22–25]. Although, there are many methods which find the mix proportion.

In this study, the response surface methodology and full factorial experimental design including central composite design (CCD) method were applied to model mechanical, rheological, and drying shrinkage properties of UHPC in normal curing condition by adding nano-silica and substituting of cement with waste glass powder.

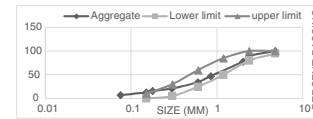


Fig. 1 – Sieve analysis of used mining sand.

The main purpose of this study is to improve the performance of UHPC in terms of mechanical, rheological, shrinkage properties and making the greener and more environmental friendly product by adding pozzolanic and waste materials.

2. Experiments

2.1. Materials

The cement used was Portland cement, 42.5N covered by European standards EN 197-1 Cement Composition. Limestone with the max particle size 5 mm was used in this study. Aggregate sieve analysis has been done based on ASTM C136 and controlled by ASTM C33 which is shown in Fig. 1.

Ordinary tap water was used for UHPC producing and curing superplasticizer was a polycarboxylic ether based, high range water reducing new generation superplasticizer admixture developed for using in UHPC which was called GLENIUM 27 and manufactured by BASF. The superplasticizer is consistent with EN 934-2. A white undensified silica fume with purity more than 95% of silicon dioxide and with the particle size of 0.1–1 μm was used as the pozzolanic material. The diameter and length of steel fiber were 0.55 mm and 13 mm respectively. The tensile strength of fiber was 1345 MPa with the young modulus of 210 GPa, the fiber was confirmed by ASTM A820 which was manufactured by Dramix, Belgium. Silicon oxide nanoparticle (SiO_2) with aerodynamic particle size (APS) of 20–30 nm, specific surface area (SSA) of 180–600 m^2/g , and bulk density of $<0.10 \text{ g/cm}^3$ with purity more than 99% was selected.

In this study, brown (amber) glass powder was prepared from waste bottles dumped in nature. After collecting the bottles, they were washed to remove paper labels, dust, or any undesired particles from the surface. Subsequently, they were broken into small pieces to be prepared for grinding with a rotary grinder machine and were pulverized up to 63 μm in particle size. Chemical components of glass powder are shown in Table 1.

Table 1 – Chemical composition of waste glass powder.

Compound	Glass powder (%)
Silicon dioxide (SiO_2)	71.09
Aluminum oxide (Al_2O_3)	3.52
Sodium oxide (Na_2O)	10.46
Iron oxide (Fe_2O_3)	1.77
Calcium oxide (CaO)	10.59
Magnesium oxide (MgO)	1.56
Potassium oxide (K_2O)	0.89
Loss on ignition (LOI)	0.60
Sulfur trioxide (SO_3)	0.03

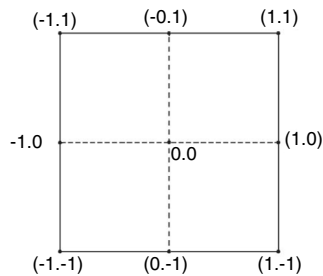


Fig. 2 – Central composite face centered design (CFC) for $n = 1$ and $\alpha = 1$.

Table 2 – Design of experiment.

Mix no.	Run	Nano-silica	Waste glass powder	CFC subdivision
1	1	-1	-1	Factorial points (2n)
2	6	-1	1	
3	8	1	-1	
4	2	1	1	
5	5	0	-1	Axial points (2n)
6	7	0	1	
7	10	-1	0	
8	3	1	0	Central points
9	4	0	0	
10	9	0	0	

2.2. Experimental design

In this study, the experiments have been designed by using design of experiment (DOE) method. DOE is a controlled method to find the relationship between few defined variables which were individually effective on the output of the process with the minimum number of experiments. Central composite face-centered design (CFC) from DOE was applied in order to screen the effects of two variables; nano-silica fume and waste glass powder on UHPC performance.

CFC is composed of full factorial in two-level design (2n), plus a number of repetitions of the nominal design which is assumed 2 times in this study, as well as, the 2n points found by changing one design variable at a time by an amount $\alpha = 1$ which is shown in Fig. 2.

The statistical software “Design-Expert version 10”, Stat-Ease, Inc., was used to design the experiments and order randomization. Design of experiment table is given in Table 2.

2.3. Methodology

Modeling and analysis of results were done by using response surface methodology (RSM) which is a mixed of statistical and mathematical techniques useful for the displaying and analysis of different issues. Adequacy of models were estimated by using analysis of variance (ANOVA) which is showing the quality of selected models.

In this study workability, 28 days compressive strength, and drying shrinkage properties of UHPC was investigated by replacing of nano-silica (A) which is fraction of binder

Table 3 – The variables ranges.

Variables	Assigned	Levels of variables		
		-1	0	+1
Nano-silica	A ^a	0.0%	2.5%	5.0%
Waste glass powder	B ^b	0.0%	10.0%	20.0%

^a Fraction of binder material addition.

^b Fraction of Portland cement substitution.

material and substituting of waste glass powder (B) which is fraction of Portland cement. Variable levels based on proportions are given in Table 3.

2.4. Specimen preparation and test specimen

Totally 10 batches were prepared (Table 4) with 3 different levels of nano silica and glass powder. Mixing of batches was completed with a mixer as per standard ASTM C305-14. After preparation of fresh matrix, they were being molded and compacted according to ASTM C109-16. After compaction, they were kept 24 h at curing room with relative humidity of 99%. After that, all specimens were demoulded and kept under saturated limestone water up to the date of testing at normal room temperature.

2.5. Flow table test

The workability were done on the flow table according to ASTM C 1437 (ASTM, 2007). In the dynamic flow, after raising the flow cone, the flow table will be dropped 20 times and then the average diameter of the concrete spread formed is measured at two perpendicular dimensions.

2.6. Compressive strength test

The compressive strength of each specimen, 50 mm cube was measured according to ASTM C109-07. Three specimens for each mixture were tested and average values on compressive strength were calculated.

2.7. Drying shrinkage test

The drying shrinkage test was performed according to ASTM C596. Prismatic specimens $285 \times 25 \times 25 \text{ mm}^3$ dimensions were prepared and then demolded one day after. Specimens were placed in the humidity cabinet in the 80% RH with the temperature of 25°C . After three days, the initial length (L_i) of the shrinkage specimens was measured before placing them in the humidity cabinet. The length (L_x) of the shrinkage specimens was measured at the age of 7, 14, and 28 days, respectively. The length variation was calculated by the following formula (1).

$$\text{Length change : LC (\%)} = (L_i - L_x) / G \times 100\% \quad (1)$$

where G is the nominal effective length.

Table 4 – Mix proportion of the specimens of UHPC.

Mix	Crushed limestone	Silica fume	Cement	Nano-silica	Waste glass powder	Steel fiber	Super plasticizer	Water/binder
1	1.5	0.3	1.0	0.000	0.0	0.1	0.06	0.2
2	1.5	0.3	1.0	0.000	0.2	0.1	0.06	0.2
3	1.5	0.3	1.0	0.050	0.0	0.1	0.06	0.2
4	1.5	0.3	1.0	0.050	0.2	0.1	0.06	0.2
5	1.5	0.3	1.0	0.025	0.0	0.1	0.06	0.2
6	1.5	0.3	1.0	0.025	0.2	0.1	0.06	0.2
7	1.5	0.3	1.0	0.000	0.1	0.1	0.06	0.2
8	1.5	0.3	1.0	0.500	0.1	0.1	0.06	0.2
9	1.5	0.3	1.0	0.025	0.0	0.1	0.06	0.2
10	1.5	0.3	1.0	0.025	0.2	0.1	0.06	0.2

Table 5 – Average results for fresh and hardened properties of UHPC from experimental work.

Mix no.	28 days compressive strength (MPa)	Flowability (mm)	Drying shrinkage (%)
1	124	245	0.0604
2	131	248	0.0619
3	136	180	0.0441
4	149	190	0.0469
5	130	217	0.0544
6	142	220	0.0526
7	136	235	0.0599
8	147	182	0.0437
9	142	200	0.0518
10	144	205	0.0528

3. Results and discussion

Table 5 illustrates the influence of nano-silica and waste glass powder on mechanical, rheological, and shrinkage properties of UHPC.

The actions and interactions of variables on UHPC performances was modeled by ANOVA. For finding the best models, linear, quadratic, and two-factor interaction model were considered in order to find the most accurate models. The quality of models were specified by coefficient of multiple determination R^2 , which displays the total deviation of the variables for each model. The probability of errors (P -value) with 95% confidence level and statistical significant test at 5% was performed for model validations.

Table 6 shows the ANOVA results for three different responses. The outputs express that the models were significant at the 5% level of confidence because the amount of P -values were less than 0.05. Additionally, the large P -values of 0.33, 0.57, and 0.30 for lack of fit (>0.05) of responses show that

Table 6 – Analysis result of regression models.

Response	R^2	Adj- R^2	Pre- R^2	F-value	Lack of fit	Model P -value
Flow table test	0.93	0.91	0.87	45.08	0.33	<0.0001
Compressive strength 28 days	0.98	0.96	0.89	58.64	0.57	<0.0002
Drying shrinkage	0.96	0.94	0.89	50.88	0.30	<0.0001

the F -value was not significant, implying significant model correlation between the variables and process responses. The models coefficient of determination R^2 has a reliable confidence with 0.93, 0.98 and 0.96 where were very close to one. The predicted R^2 of 0.87, 0.89, and 0.89 were in reasonable agreement with adjust R^2 of 0.91, 0.96, and 0.94, whereas, the differences were less than 0.2 respectively.

Fig. 3 illustrates the normal plot of the residual value of different responses, which were used to control the model satisfactoriness. Based on the models adequacy, the residuals from the least square fit were important. The created plot of studentized residual versus the normal percentage of probability were satisfied because flow test, 28 day compressive strength, and drying shrinkage residual plots agreed well with the straight lines, as shown in Fig. 3. Consequently, it could be mentioned that these models were reliable enough.

Table 7 shows the details of estimated models to reach the desired performance of UHPC. Probability factor is given for each variables which shows the importance degree of variable on response performance. Different statistically models were used to fit variables on different responses as linear, quadratic, and 2 ways interaction models were used for flow test, 28 days compressive strength, and drying shrinkage, respectively.

3.1. Flow table test

Effect of nano-silica and waste glass powder on the workability of fresh UHPC were shown in Fig. 4. The flow test range was started from 180 mm in mix no. 3 when nano-silica was in high level and no waste glass powder was used till 245 mm in the mix no. 2, when there is no added nano-silica fume and waste glass powder added, was in high level. The results show the linear and direct effect of nano-silica and waste glass powder on the workability of fresh UHPC. As response surface and counterplot show in Fig. 4, nano-silica is effectively decreasing the workability of UHPC. It is indicated that nano-silica has a huge superficial area about or more than 60 m^2 and a strong pozzolanic activity which is rising water demand. The effect of waste glass powder on the workability of UHPC is opposite of nano-silica. As results show, waste glass powder replacement by cement has slightly increased the flow of UHPC in all different level as increases the slump due to the surface structure of glass powder particles which were crystallized and smother than cement particles and absorbs less water comparing to cement [9,12–14]. According to the results,

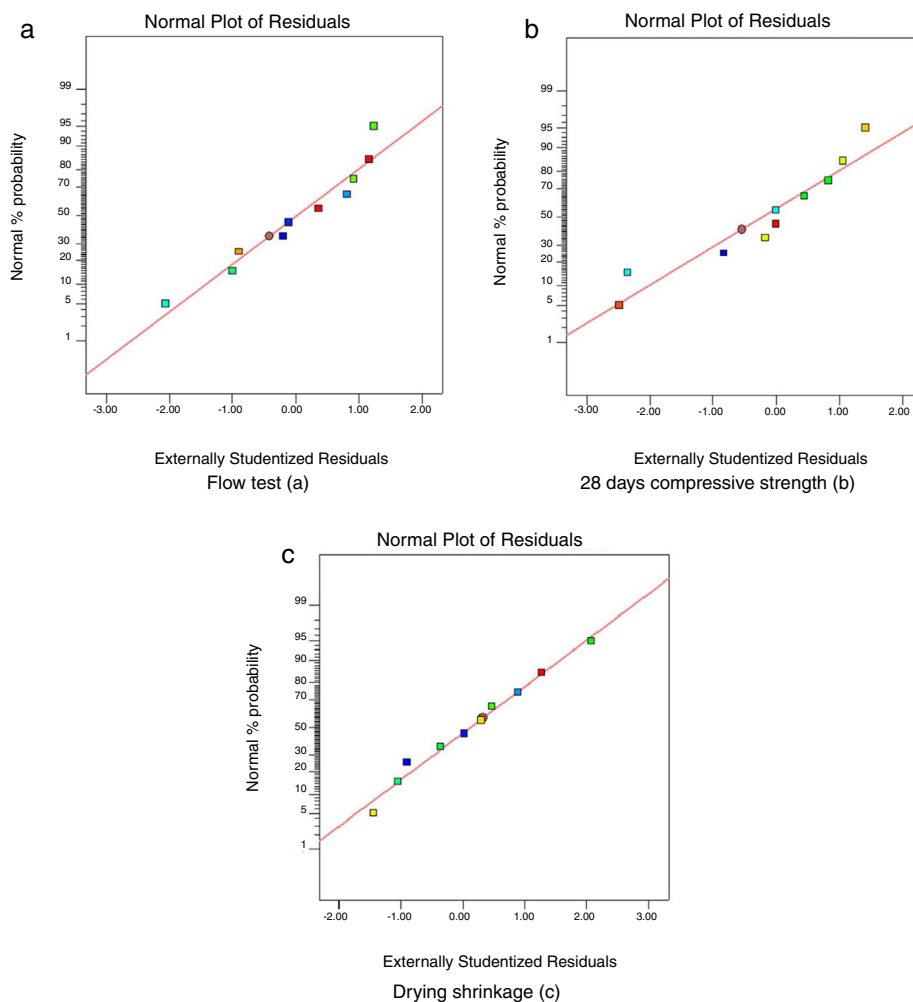


Fig. 3 – Prediction efficiency of offered models.

Table 7 – variables coefficients estimated for models.

Parameters	Flow table test		Compressive strength 28 days		Drying shrinkage	
	Estimate	Prob>f	Estimate	Prob>f	Estimate	Prob>f
Constant	21.20	–	142.25	–	5.350	–
A	–2.87	<0.0001	6.83	0.0001	0.840	<0.0001
B	0.20	0.51	5.33	0.0004	0.140	0.0884
AB	–	–	1.50	0.1134	0.042	0.6351
B ²	–	–	–6.92	0.0010	–	–

no interaction between nano-silica and waste glass powder was observed on the workability of UHPC.

3.2. Compressive strength

The effect of nano-silica and waste glass powder on 28 days compressive strength were shown by response surface and counter graph in Fig. 5. Minimum 28 days compressive strength 124Mpa was in mix no. 1 when both nano-silica and waste glass powder were in the lowest level and maximum 28 days compressive strength 149Mpa was in mix no. 4 when both nano-silica and waste glass powder were in highest level which shows the positive effect of these two variables on 28 days compressive strength. As results show, the 28 days

compressive strength will be directly increased by replacing nano-silica and parabolic increased by adding waste glass powder. The analysis shows that the positive interaction coefficient of AB which is shown the interaction nano-silica and waste glass powder additions increase the mechanical properties of UHPC. It could be verified as Vaitkevičius et al. [26] found the positive effect of waste glass powder on compressive strength of UHPC by increasing the pozzolanic activity which is due to increasing amount of amorphous. According to the regression equation of parabola shown in Table 7, it is easy to recognize that when the differential coefficient (y') of each function equals zero, the value of B represents the optimal waste glass powder amount to obtain the best mechanical properties. Hence, the optimal waste glass powder amount for

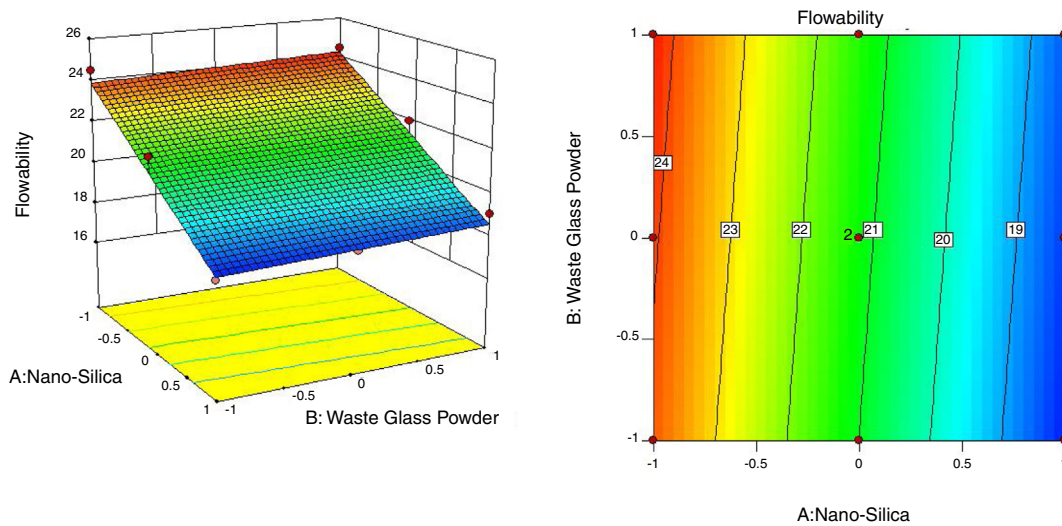


Fig. 4 – Contour plot and response surface of flowability.

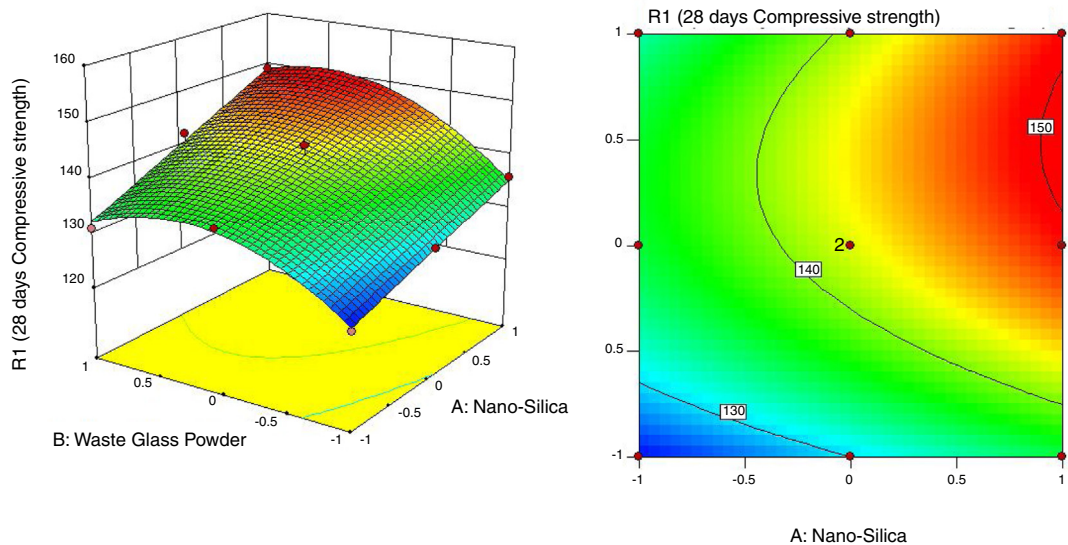


Fig. 5 – Contour plot and response surface of 28 days compressive strength.

the 28 days compressive strength will be related to nano-silica fume replacement in different levels.

3.3. Drying shrinkage

Effect of nano-silica and waste glass powder additions was studied on drying shrinkage of UHPC. It was absorbed that adding nano-silica and waste glass powder were increasing the amount of drying shrinkage as shown in Fig. 6. The reason can be explained by hydration shrinkage. Cement generally shrinks through hydration reaction. Silicon powder react with $\text{Ca}(\text{OH})_2$ produced by cement during hydration which causes to the development of shrinkage. As Q-Wald reported the chemical shrinkage was increased by increasing the pozzolanic activities [27–30]. Varghese et al. reported as the existence of drying shrinkage of concrete was due to exclusion of gel

water, the higher drying shrinkage of nano-silica of high performance concrete compared to that of reference concrete can be recognized to the presence of higher amounts of finer C-S-H holding higher amounts of gel water, which was released during drying shrinkage [31]. As Omran mentioned adding glass powder will increase the drying shrinkage by increasing the pozzolanic activities [26]. The results illustrates the positive interaction coefficient of AB which was shown the interaction nano-silica and waste glass powder addition increase the drying shrinkage of UHPC.

4. Conclusion

This paper studied the individual contribution of nano-silica and waste glass powder as well as interactive effect between

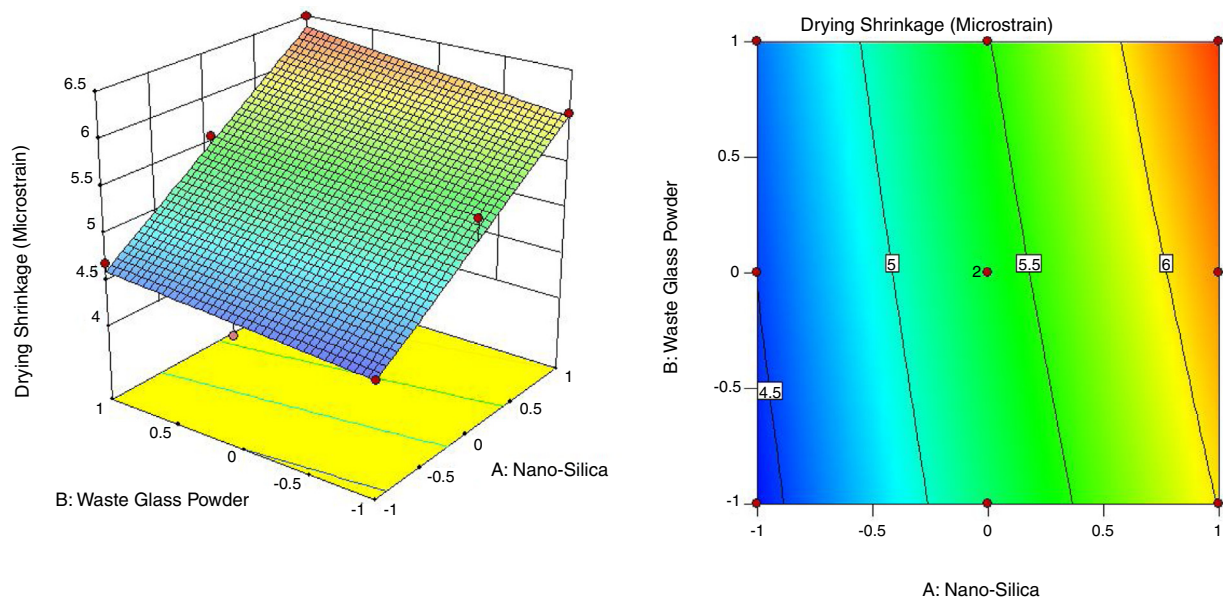


Fig. 6 – Contour plot and response surface of drying shrinkage.

those by using response surface methodology. From the results illustrated in this study the following conclusions were drawn:

- The three fitted models; 28 days compressive strength, flow test, and drying shrinkage were developed using a response surface methodology for UHPC by adding the fraction of binder with range of 0–5% of nano-silica fume and substituting the fraction of Portland cement (PC) with range of 0–20% of waste glass powder in order to increase the UHPC performance and produce the green UHPC by replacing the cement with waste material. Although the models were based on a given set of materials, it can be applied to other materials.
- Out of several models, quadratic model was fitted with waste glass powder addition on compressive strength of UHPC which represent there was optimal waste glass powder.
- Nano-silica addition increased the 28 days compressive strength and drying shrinkage of UHPC, while the flowability of fresh UHPC was reduced due to nano-silica addition.
- Adding waste glass powder increased 28 days compressive strength, drying shrinkage, and the flowability of UHPC.
- The interactive effect between nano-silica and waste glass powder was observed on 28 days compressive strength and drying shrinkage.

Conflicts of interest

The authors declare no conflicts of interest.

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