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Enhancing Geopolymer Performance: Optimization of Sand to Fly Ash and Water to Geopolymer Solid Ratios Using Response Surface Methodology

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Abstract. Recently developed Geopolymer is an innovative class of green and high-performance binders. Despite increasing interests in this unique material, insufficient work is available for optimizing mixture ingredients, which in turn slows the material advancements and its applications. Response Surface Methodology (RSM) has been recently utilized to optimize principal polymeric variables (concentration of sodium hydroxide, sodium silicate to sodium hydroxide ratio and curing temperature) in geopolymer. The aim of this study, is to optimize two basic geopolymer related parameters (sand to fly ash and water to geopolymer solids ratio) for compressive strength and drying shrinkage by developing the statistical models through RSM. The models are verified in a statistical manner and gives any value of geopolymer property with significance level of 95% or higher. Considering the two optimum proportions, consist of 0.64 sand/fly ash and 0.22 water/geopolymer solids. Finally, the optimized results were experimentally validated as the difference between projected and experimental results was found not greater than 5%. The statistical models developed by RSM can be used to navigate geopolymer property and RSM allows designer to obtain an optimal solution of the product that improves its yield and reliability.

Keywords: Geopolymer; RSM; Optimization; Desirability.

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1. Introduction

Significant environmental and sustainability issues in concrete construction have been caused by the large consumption of Ordinary Portland Cement (OPC). It is the second most used material by human beings on this planet after water and we know that concrete is made with OPC (Ordinary Portland Cement) as its main component and the concrete is responsible for 10% of global CO_2 emissions (Wang et al., 2017). This environmental burden prompts the call for more environmentally benign binder materials in the concrete industry impact. In this regard, geopolymer which is a novel process to use a cement-free matrix is a very promising solution (Zahid et al., 2018). Geopolymers are formed when alumina and silica-rich precursors are combined with an alkaline solution and heat cured to form a binder. A number of these are industrial byproducts which were about to go to waste, spreading environmental pollution (Yu et al., 2016). With the high alumina and silica contents present in fly ash, this byproduct of coal-fired power plants has served as a promising precursor for geopolymers causing environmental health and sustainability (Zahid et al., 2017). Fly ash-based geopolymer presents better mechanical characteristics, more durability resistance, and enhancement in micro-structure than OPC (Davidovits, 1991). The Taguchi method (Sivaraos et al., 2014), Plackett-Burman (Ekpenyong et al., 2017), and Response Surface Methodology (RSM) are performed and considered to optimize the product reliability (Mohajeri et al., 2010). RSM is one very powerful to conduct systems experimentation, statistically significant model development, and multiple responses optimization. The method of RSM has been employed for the optimization of cement-based concrete but for optimizing with geopolymer technology, its use is very rare (Eriksson et al., 1998). In the previous study, response surface methodology was applied to optimize OPC-based concrete mixes whereas its application to develop high-performance geopolymer.

Response surface methodology (RSM) teaches a practical user/knowledge-friendly approach to the capturing of the manufacturer-relevant mixture components in the cement-based concrete industry. Ghafari et al., (2014) develop empirical statistical models by using Central Composite Design Analysis Approach (CCD) for prediction of cementitious concrete. Meanwhile, Bayramov et al., (2004) optimized the fiber related parameters in the concrete in order to increase the ductility of the steel fiber reinforced concrete as much as possible. Moreover, Al-Alaily and Hassam (2016) used the RSM technique to optimize the durability, as well as the mechanical properties of concrete mixes having metakaolin. The RSM technique is extensively used for OPCC (Optimal Proportion of Concrete Components) concrete mixes but the application in Geopolymer technology is very limited in the literature. Mermerdas et al., (2007) studied optimization procedure of some mixture components of light weight geopolymer with the minimization of the curing temperature, curing time and binder content by maximizing its compressive strength. As a result, in order to design a mix with optimum performance of geopolymer, it is important to have the right dosages of these mix ingredients for each specific principal polymeric variable (NaOH molarity, sand to fly ash ratios sodium silicate to sodium hydroxide ratio and water to GP-solids ratio) (Nematollahi, 2016). In their study, Zahid et al., (2019) optimized RSM parameters for the principal polymeric variables (concentration of sodium hydroxide, sodium silicate to sodium hydroxide ratio and curing temperature) for maximizing mechanical performance of geopolymer.

As a follow-up investigation, this study aimed to utilize RSM for optimizing the influence of sand to fly ash ratio and water to GP-solids ratio on the compressive strength and drying shrinkage of geopolymer. Furthermore, an experimental program was designed to validate the optimum mix design given by RSM.

2. Experimental Work

2.1. Materials Used

This research utilizes the Fly Ash (FA) taken from coal fire power plant located in Manjung Perak, Malaysia. Spherical shape of FA particles was observed as shown in micrograph (Figure-1) which was taken using SEM. As shown in table-1, the elemental composition was evaluated using XRF testing, while BET analysis was used to find the surface area of FA. The FA has basic criterion for high calcium fly ash as per ASTM-618 (Ca0>10%; SiO_2, + Al_2O_3 + Fe_2O_3 > 70%) was confirmed from the results of XRF. Sodium based alkaline activator (NaOH+Na2SiO3) was used for the production of geopolymer; chemical features are presented in Table 2.

Table 1: Chemical composition and BET of fly ash

Oxides in fly ash (%)										
SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	SO_3	TiO_2	P_2O_5	LOI	BET (m^2/g)
35.2	10.9	22.5	20.1	2.5	2.12	1.22	1.44	1.1	2.5	0.9872

Table 2. Chemical analysis of alkaline solution (% by mass)

Na_2SiO_3 solution (%)		**8M NaOH solution (%)**	
Na_2O	14.6	NaOH	25.6
SiO_2	29.85	H_2O	74.4
H_2O	55.50		

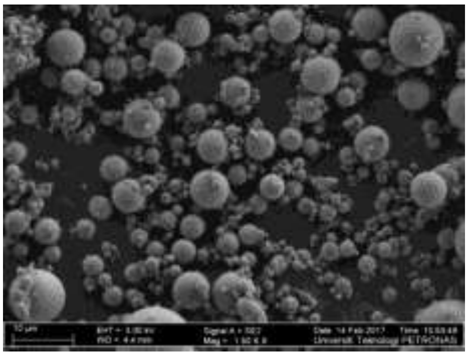


Figure-1: SEM image of Fly ash

2.2. Design of Experiments using RSM

Responses can be understood by changing one variable at a time (performing a trial-and-error) effect over them, which people have traditionally used to see the effect. However, this is significantly more time-consuming for systems having more variables. Therefore, we need a more efficient method to deal with multi-objective optimization problems. Being the powerful methodology for optimization of such a complex problem the RSM process consists of four main steps: 1) experiment designing, 2) response data collection using an experiment, 3) RSM numerical modeling, and 4) model optimization and validation (Ghafari et al., 2014).

Commercially available version of Design-Expert® software was utilized for design of experiments (DOE). The range chosen for the sand/fly ash ratio of the base mix was 0 to 0.8, and the range chosen for the water/ geopolymer solids ratio of the base mix was 0.22 to 0.26. Meanwhile, the other additional factors, including concentration of sodium hydroxide, sodium silicate to sodium hydroxide ratio and curing temperature, were fixed at 8M, 2.5, and 60 ºC, respectively (Nematollahi et al., 2016).

Table 3. Mix designs of geopolymer

	Mix design variables	
Mix Name	W/GP solids	Sand/fly ash
M1	0.26	0
M2	0.26	0.4
M3	0.26	0.8
M4	0.24	0
M5	0.24	0.4
M6	0.24	0.8
M7	0.22	0
M8	0.22	0.4
M9	0.22	0.8

2.3. Mixing and Casting of Samples

Table-3 summarizes nine geopolymer mixes for the optimization of water to GP-solids ratio, and sand to fly ash ratio. Weighed 320g of NaOH pellets and dissolved them in tap water to make one liter of an 8M NaOH solution. With the immediate exothermic reaction, in the laboratory, we allowed the solution to cool down. The mixture was uniform after 5 min with a Hobart mixer. Fly ash and fine aggregates were mixed in dry condition at low speed for 30 s followed by the slow addition of alkali solution. After that, the compounds were subjected to a 4-minute wet mixing at the same speed. The material was then poured into standard lab molds and allowed to solidify. All the samples were cured in an oven at 60°C for 24 hours. The molds were then kept in the laboratory at 23 ºC steady-state. Tests were conducted on the 28th day of casting.

2.4. Experimental Equipment and Test Procedures

The raw geopolymer mix was then evaluated for its fresh workability through Mini Slump Flow (MSF) test in accordance with ASTM C1437-15. A lateral section of the truncated cone (dimension 70 x 100 x 60 mm) was put at the center of the flow table; the geopolymer paste was then upfilling the section. The paste was then driven in to the cone and lifted for the paste to come out. The paste was then allowed to spread out by subjecting the flow table to 25 mechanical jerks in 15 s. Flow was determined as a percent increase of the average base diameter of the paste over the initial base diameter. The totaling applied in the workability calculation of cement-based mortar was also based on this method (Zahid et al., 2018; Li et al., 2013). Three cubes (50 x 50 x 50mm^3) of each mix design were taken for Compressive strength test as per

ASTM C109/C109M-16a, they were subjected to the gradual load (3.0 kN/s) until their final failure. Drying shrinkage was measured according to the procedure described in ASTM C157-17. A complete set of experimental tests performed.

3. Results and Discussion

Experimental tests conducted on the slump flow, drying shrinkage, and compressive strength of the geopolymer mixes are presented in Table. The results were evaluated to examine the impacts of sand to fly ash ratio (S/FA) and water to Geopolymer solids ratio (W/GP) on these properties. Response surface diagrams, contour diagrams and statistical model was developed using Design-Expert® software. The study ends with the validation and optimization of the S/FA and W/GP ratios.

Table 4. Experimental results

Mix	Geopolymer properties		
	Compressive strength (MPa)	Slump diameter (mm)	Drying shrinkage (%)
M1	68.3	336	0.79
M2	71	315	0.52
M3	69.1	293	0.4
M4	72.2	316	0.49
M5	75.2	302	0.34
M6	75	291	0.29
M7	79.2	270	0.4
M8	82	259	0.29
M9	82.5	245	0.25

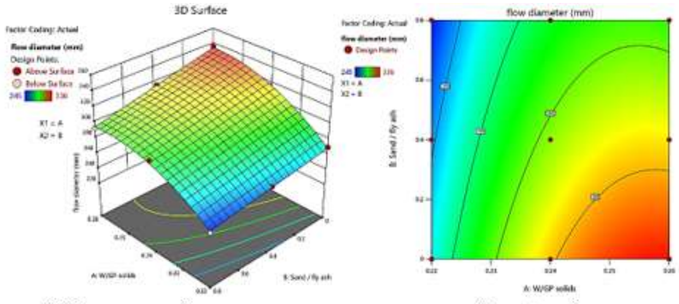
3.1. Effect of sand to fly-ash and water to GP-solids on workability of geopolymer

Workability is the ability of freshly mixed concrete to be readily mixed, placed, consolidated and finished and consists of the comfort with which concrete can be moved with minimum loss of homogeneity, segregated, and bleeding. Workability can be measured by slump flow. Figure 2 depicts a 3-D response surface graph on the slump flow and contour diagram of compressive strength bands vs the water to GP-solids (W/GP) and sand to FA ratio (S/FA) of geopolymer. A surge in W/GP solids similarly leads to a higher flow by reducing internal particle friction due to increased water curing. Increased S/FA decreased flow, probably due to a decrease in lubricating liquid. They also have a tendency to roll individually as they move with the flow due to the fact that they are spherical in shape.

At a W/GP solid of 0.26 the maximum slump flow obtainable was 336 mm for a S/FA of 0. The slump flow value was 245 mm as the lowest, and it was found in W/GP-solids equal to 0.22 and S/FA equals 0.8. Prediction of the mass of W/GP-solids and S/FA ratios to produce the desired flow values could be done by the use of the contour diagram. Response Surface Methodology (RSM) -based second-order models actually make better predictions and optimization than first-order models (Gao et al., 2016). For example, Mohammed et al., (2016) also created a second-order RSM model to predict slump flow with accurate precision, by using levels of the water/cement and the content of residues. We also proposed second-order RSM models between W/GP Solids and Slump and between S/FA and Slump Flow in this research and (Eq. 1).

$$\begin{aligned} \mathit{Slump\ flow}\ (\%) &= -2475.55 + 21641.66 * x_1 + 97.08 * x_2 - 562 * x_1 * x_2 - 4166.66 * x_1^2 \\ &\quad - 1.041 * x_2^2 \qquad (1) \end{aligned}$$

Where x_1 and x_2 represents W/GP solids and S/FA respectively



a) 3D response surface b) contour diagram

Figure 2. Slump flow versus W/GP solids and sand/fly ash

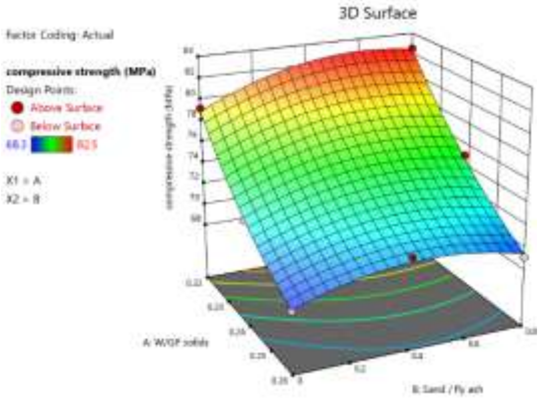
3.2. Effect of sand to fly-ash and Water to GP-solids on the compressive strength of GEOPOLYMER

Compressive strength is an important mechanical parameter of concrete, and it is the main basis for the design of concrete structures. The 3D response surfaces of the strength (Figure 3) give a clear message, the porosity decreased to the same level with almost constant W/GP (0.26-0.244), and the compaction had a positive effect on the value of the strength. Chung et al., (2017) observed similar phenomena. But W/GP reduction to 0.22 completely diminishes workability that forms macro pores inside the geopolymer, which critically lowers the compressive strength.

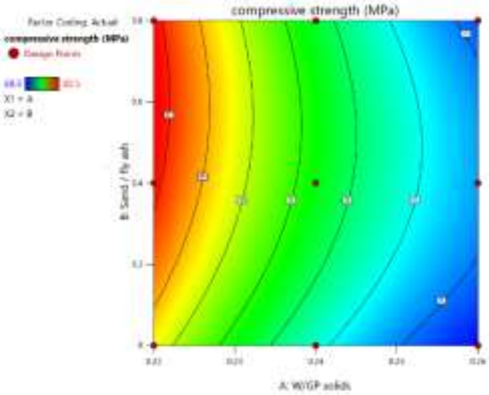
Moreover, increasing the S/FA ratio increases the compressive strength, a behavior also observed in other geopolymer literature on record (Nematollahi et al., 2016). In the second case the best compressive strength obtained was 82.5 MPa for W/GP = 0.22 and S/FA = 0.8. Güneyisi et al., (2014) optimized the effects of fly ash and metakaolin in concrete formulations by the RSM technique and found that the compressive strength could be predicted correctly by ANOVA models made by RSM. Thus, a prediction model for GEOPOLYMER compressive strength was established in the research herein, expressed as formula (2).

$$\begin{aligned}\mathit{Compressive\ strength\ (MPa)} &= +310.72 - 1722.92 * x_1 + 30.04 * x_2 - 78.12 * x_1 * x_2 + 3041.66 * x_1^2 \\ &\quad - 10.52 * x_2^2 \qquad (2)\end{aligned}$$

Where x_1 and x_2 represents W/GP solids and S/FA respectively.



a) 3D response surface



b) contour diagram

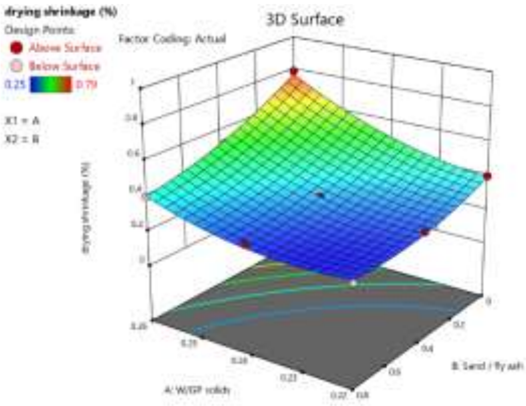
Figure 3. Compressive strength versus W/GP solids and sand/fly ash

3.3. Effect of sand to fly-ash and Water to GP-solids on drying shrinkage of geopolymer

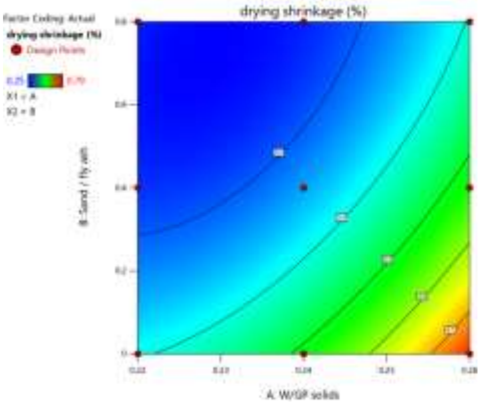
The decrease in mortar length due to the water loss during the drying process is Drying Shrinkage (Wallah et al., 2006), is a substantive basis of concern for GEOPOLYMER mortar (Minoru and Keitetsu, 2006). The 28-day drying shrinkage of geopolymer as function of long-term W/GP solids and S/FA is shown in Figure 4. On the other hand, as regards the drying shrinkage, the latter increases as the solids content of W/GP is higher and decreases as the amount of sand is higher. Equation 3 presents ANOVA prediction model for drying shrinkage.

$$\begin{aligned}DS = +8.09 - 72.58 * x_1 + 1 * x_2 - 7.5 * x_1 * x_2 + 170.83 * x_1^2 \\ + 0.33\, x_2^2 \qquad (3)\end{aligned}$$

Where x_1 and x_2 represent W/GP solids and S/FA respectively.



a) 3D response surface



b) contour diagram

Figure 4. Drying shrinkage versus W/GP solids and sand/fly ash

3.4. Validation of ANOVA Models

The statistical validation of the model supports the suitability of all the ANOVA models for the future purpose. Table 5 presents the acceptance criteria, where the direct indicators are the magnitude of the predicted R² and adjusted R² changes (no more than 0.2), the precision is greater than 4, and the response standard deviation is low. Table 6 presents the analysis of variance (ANOVA) for the statistical equations. Models would be significant if there was a high F-value and the p-value were less 0.05; so, 95% confidence. This is pretty precise that all model is significant and they can be appropriately estimating response values from any given factor combination. In the interest of brevity, we only present the predicted vs actual plot, Normal probability plot, perturbation plot for the drying shrinkage of geopolymer. The predicted vs. actual plot (Figure 5) shows that predicted values closely follow the 45-degree line, only slightly above this line. The perturbation plot (Figure 6) is provided to further apprehend the effect of independent variable on drying shrinkage at particular point. The steep slope and curve of this plot reach to the conclusion that the drying shrinkage is influenced by W/GP solid and S/FA.

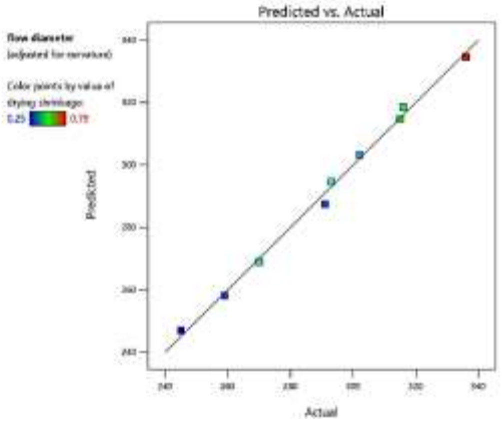


Figure 5. Predicted vs. Actual for Drying shrinkage

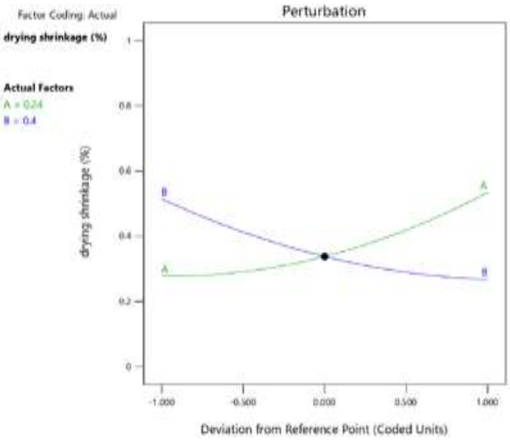


Figure 6. Perturbation curves for Drying shrinkage

Table 5. Model validation for responses

Response	Standard deviation	Mean	R^2	Predicted R^2	Adjusted R^2	Adequate precision
Slump flow	2.36	136.56	0.9988	0.9859	0.9969	63.873
Compressive strength	0.43	73.27	0.9937	0.9231	0.9831	31.105
Drying shrinkage	0.61	8.26	0.9863	0.8427	0.9634	19.166

3.5. Optimization

It is hard to get ideal values for all individual response honestly in one area. As such, multi-objective optimization method is accepted to be a good solution to cope the requirements of all responses simultaneously. Following development and validation of the ANOVA models for all responses, a multi-objective optimizations method was employed to optimize the responses. We used the multi-objective optimization procedure of response surface methodology to identify the best compromise that maximizes the overall set of responses simultaneously. Table 7 provides the definitions of factors and responses of the multi-objective optimization problem. In all, the study sought to maximize responses for compressive strength, while minimizing drying shrinkage. For satisfying the basic requirement for self-compacting mortar, the slump flow was kept between 240-260%, thus eliminate the need for manual compaction (Zahid et al., 2020; Ferdosian and Camoes, 2017; Khotbehsara et al., 2015). Seventy different solutions were obtained in total fulfilled the given criteria Nevertheless, a preferred solution which had desirability value of one was taken up. The ramps of the optimal geopolymer mix and equivalent optimal responses are illustrated in Figure 7. Validation study was carried out to analyze the change in results which can be derived out of various experiments after selecting the best solution.

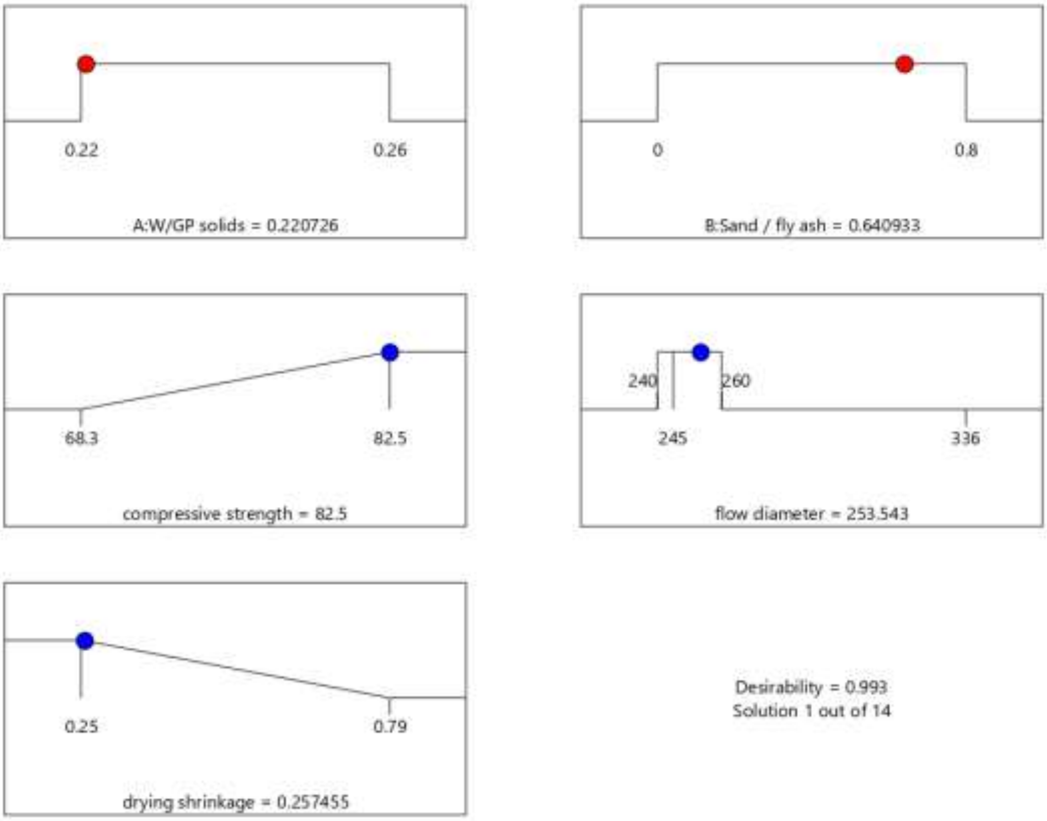


Figure 7. Ramps for multi-objective optimization of Geopolymer

3.5.1. Experimental Validation Study

In order to ascertain the accuracy of the RSM models and the reproducibility of the optimized conditions, an experiment on an optimized program was developed (Table 8). The procedures done in mixing, casting, curing and testing of the samples were the same as what had been described before in Sections 2.3 -2.4. The results obtained were close to the responses predicted, with a maximum difference lower than 5% (Table 9).

Table 6. ANOVA results for full regression model of each response

Response	Sum of squares	Mean square	F-value	p-value prob > F	Remarks
Slump flow	6894.78	1378.96	137.39	0.0001	Significant
Compressive strength	225.81	45.16	213.27	0.0005	Significant
Drying shrinkage	0.2195	0.0439	60.48	0.0033	Significant

Table 7. Definitions of factors and responses in the multi-objective optimization problem

Name of factors and response	goal	Lower limit	Upper limit
W/GP solids	In range	0.22	0.26
S/FA	In range	0	0.8
Slump flow	In range	245	336
Compressive strength	Maximize	68.3	82.5
Drying shrinkage	Minimize	0.25	0.79

Table 8. Optimized solutions with desirability

No	Factors (variables)		Responses (EGC properties)			
	W/GP solids	S/FA	Slump flow (%)	Compressive strength (MPa)	Drying shrinkage (%)	Desirability
1	0.22	0.64	253.54	82.5	0.2574	0.993

Table 9. Experimental validation of the optimized Mixture

Response	Slump flow (%)	Compressive strength (MPa)	Drying shrinkage (%)
Predicted	253.54	82.5	0.2574
Experimental	241	86.6	0.245
Error (%)	4.94	4.97	4.817

4. Conclusions

The current work aims to identify appropriate water/Geopolymer (W/GP) solids mix dosages and sand/fly ash (S/FA) ratio in the GEOPOLYMER mix by performing experiments and analysis using the Response Surface Methodology (RSM). Key Findings and Conclusions from the experimentations are:

- Based on 3D response surface diagrams, the effects of W/GP solids and S/FA on slump flow, compressive strength, and drying shrinkage have also been effectively verified.
- The values of the indicated zones can precisely be assumed and as a result contour diagrams provide the predictions of the factor ranges to be used to achieve the desired geopolymer properties. In addition, ANOVA equations were derived, statistically validated and successfully used to precisely predict responses based on the values of target factors.
- Optimum levels of S/FA and W/GP solids in geopolymer as established from RSM based on multi-objective optimization are 0.64 and 0.22 respectively.
- The use of the RSM optimization technique reduce the design time, increase the performance of the target processes and products and improve reliability and robustness of the product as well as the process

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