



Modeling setting time and compressive strength in sodium carbonate activated blast furnace slag mortars using statistical mixture design



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ABSTRACT

The alkali activation of industrial by-products have the potential to reduce the carbon dioxide emissions from the cement industry and to develop alternative mortars and concretes with properties similar or better than the ones based on ordinary Portland cement. The use of sodium carbonate as an activator for blast furnace slag offers potential advantages over other Alkali-Activated Materials (AAMs) in terms of environmental impact and cost. This study explores the use of statistical mixture design to determine what affects initial setting time and compressive strength in sodium carbonate activated blast furnace slag mortars. This experimental design approach allows for a more thorough analysis of the effects of the mortar components on response variables over the entire design space. In this study the amount of each component was restricted to certain values in order for the mixture to react and produce a mortar with acceptable mechanical strength and setting time. The mortars obtained were tested according to ASTM C403 and ASTM C109 standards for initial setting time and compressive strength. The statistical design and analysis was done using Design-Expert 9.0. This study finds that initial setting time and compressive strength of these materials are highly dependent on the composition of the materials and proposes a model for setting time and compressive strength prediction. It was found that the two-factor interactions with sodium carbonate are statistically significant in the compressive strength model but not in the initial setting time model. A typical sample was characterized by Scanning Electron Microscopy.

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1. Introduction

It has been estimated that cement production is responsible for about 5% of total anthropogenic carbon dioxide emissions [1]. Due to planetary concern about global climate change, interest in reducing the emissions associated with the cement industry has been growing. Alkali-Activated materials (AAMs) are a category of alternative cementitious materials that have been shown to reduce greenhouse gas emissions by up to 40% [2]. Studies have shown that AAMs can be used in building applications where Ordinary Portland Cement (OPC) would traditionally be used [3]. AAMs have exhibited better resistance to freeze-thaw cycles, higher mechanical strength and better resistance to acid attack than Portland cement based materials [4]. However, many AAMs set too fast to be

used in applications where the material must be manipulated after the initial pour. Setting times for Sodium carbonate activated slag have been shown to be as much as two days longer than other common sodium based activators such as sodium hydroxide and sodium silicate [4].

In practice, the initial setting time of the material is more important than final setting time because the initial setting time determines when the material is still workable. In addition to setting time, it is necessary to maintain a certain minimum level of compressive strength if these materials are to be used. For some common applications like pavements and basement walls the minimum compressive strength requirements range from 17 to 24 MPa [5] depending on climate. With a wide range of setting times (30 min [6] to 3 days [4]) and compressive strengths (20–100 MPa [7]) reported in the literature for sodium carbonate activated blast furnace slag, further research is needed to determine the combined effects of the ingredients on the AAM

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properties.

In the AAM literature when experimental design has been used, the Taguchi method [8,9], fractional factorial [10–12], and mixture design [7] have been used. Previous studies have also explored the effect of silicate modulus, activator concentration, curing temperature and type of activator on compressive strength [9,11]. In this study, individual mixture ingredients are used as the studied factors in order to determine statistically significant components in the mixture. Statistical Mixture design offers advantages over fractional factorial and Taguchi design methods because the amount of total material can be held constant.

The purpose of the current work is to determine the relationships between two responses (initial setting time and compressive strength) and the mixture components in sodium carbonate activated blast furnace mortars.

2. Materials and methodology

2.1. Experimental design

Traditional experimental design methods, such as the factorial design, or Taguchi design (including crossed arrays), assume that all variables in the design may be controlled and manipulated independently of one another. In a cementing material experiment, this can be achieved if the experimenter is only interested in varying one or two components involved. For example, if we were only interested in manipulating the amount of slag and the amount of water, over a few levels in the mixture, a factorial design might be a good choice because this would provide an easy and efficient way to create the design and study the relationship between these variables and a response such as the compressive strength of the resulting mix.

A mixture experiment is a special type of experimental design in which the components (or ingredients) of a mixture are not independent [13]. In a mixture experiment, the analysis can specify a combination of the components as a proportions of the total volume or mass of the mixture. In the case of the experiments outlined in this research, we choose to fix the total mass of the mixture, which suggests that a mixture experiment is the most efficient and appropriate design choice from a statistical aspect. A drawback to these designs is that it requires some statistical expertise in order to create the design and requires one to think about how to appropriately specify the ranges of the variables in the experiments and the constraints to abide by. In experimental design literature, the 'design space' represent the region in which trials of the experiment are feasible. For example, if we wanted to vary the amount of water between 200 and 250 g and vary the amount of slag in between 400 and 600 g, then the 'design space' for our experiment (without using a mixture experiment) would be represented by a square where the x-axis was water (200–250) and the y-axis was slag (400–600). In this square design space, water = 225 g and slag = 500 g, would represent the center of the design space.

The design space of mixture experiment with three components can be visualized using a simplex design plot (shown in Fig. 1a). The center edges up through the vertices of the triangle represent the proportions of each mixture component. For example, the middle point in Fig. 1a represents a mixture with 1/3 A, 1/3 B, and 1/3 C. Thus the total volume or mass of the mixture remains fixed at a value of 1 (meaning 100% of the mixture).

In the case of this study there were five mixture components: blast furnace slag, sand, water, sodium carbonate and amorphous SiO₂. In order to simplify the visualization of our design, we have provided a simplex design plot of the activator ingredients (water, sodium carbonate and amorphous SiO₂) in Fig. 1b. As can be seen in Fig. 1b the components of the activator have been restricted by

providing constraints. This is another benefit to using the mixture experiment approach. If there are certain regions of the design that are undesirable, the experimenter may place restrictions in the form of mathematical equation. In our experiment, we specified the weight of the mixture to be 1500 g, but also placed limits on the water to slag ration and sand to slag ration. These limits cause the design space to be a polygon instead of the traditional triangle. In this case instead of the vertices of the polygon representing mixtures of only one component, they represent an edge of the design space confined by the design constraints of the mixture. The constraints of the five component mixture design used in this study are shown in Table 1. The mass and ratio constraints that were chosen based on previous research and screening experimentation conducted by the authors and others.

$$E(Y) = \sum_{i=1}^q \beta_i x_i + \sum_{i=1}^{q-1} \sum_{j=i+1}^q \beta_{ij} x_i x_j \quad (1)$$

The experimental runs were determined by the statistical experimental design program Design-Expert 9.0. An I-optimized custom mixture experiment was used with the constraints listed in Table 1. Given these parameters Design-Expert provided an experimental design with 24 runs. The Sheffe Quadratic model is shown in equation (1), where β is the coefficient and x represents the mixture component and q is equal to five (number of ingredients). This model allows the response to be modelled by incorporating the main effects and two-factor interactions of each mixture component. The first six design runs and factor amounts are shown in Table 1. Statistical blocking was used to account for differences in day of production.

2.2. Materials

The samples utilized sodium carbonate monohydrate (ACS reagent grade, Thermo Fisher Scientific), Sand (ASTM C33-85, Manitou Concrete), ground granulated blast furnace slag (GGBFS, St. Mary's Cement, Ontario Canada), and hydrophilic amorphous silica (Aerosil 150, Evonik). The GGBFS used in all experiments had a specific density of 2.92, a Blaine fineness of 660 kg/m² and a mass composition of CaO 38.3%, SiO₂ 37%, Al₂O₃ 8%, MgO 10.5%, Na₂O 0.025% K₂O 0.043%, SO₃ 1.7%. The water used in the experiments was tap water from the Rochester, NY municipal water supply.

2.3. Sample preparation

For each of the 24 runs called for in the experimental design, a sample with the required amounts of ingredients was prepared for initial setting time and compressive strength testing. The mixing of the sample was performed in two separate parts. First the alkali activator was prepared, then 24 h later it was added to the dry ingredients.

The alkali activating solution was created by mixing water and sodium carbonate together for 30 min using a magnetic stir plate. The amorphous silica was then added to the solution and mixed for an additional 60 min. After the final mixing cycle the solution was left to mature for 24 h in an airtight container. After 24 h the dry ingredients were carefully weighed and mixed together in a Hobart mixer. Then the activating solution was added to dry ingredients and mixed in a Hobart mixer for 10 min, time that it took to form a mortar of homogeneous consistency. The mortar was then casted into one cylindrical PVC mold and two 2 inch ASTM standard cube molds. The cube samples were vibrated for 5 min before the samples were covered in plastic and cured for 24 h at 65 °C.

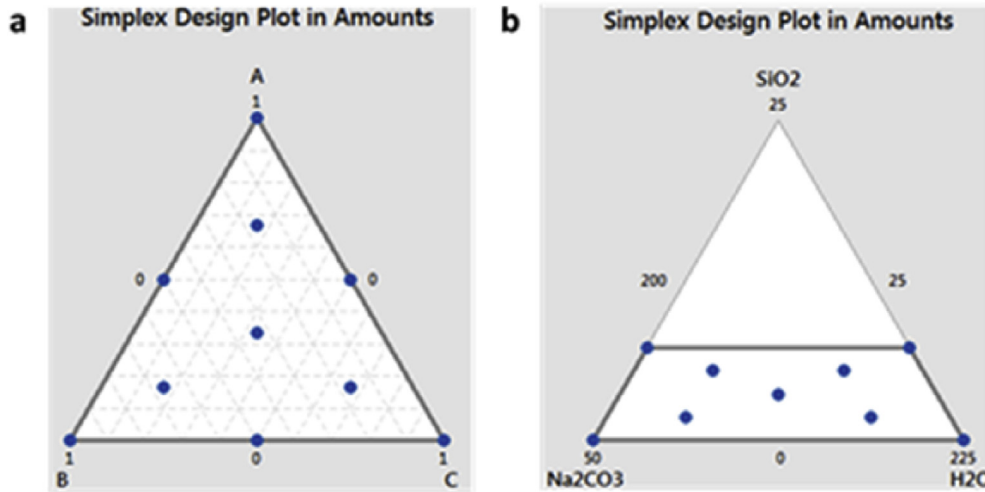


Fig. 1. a) Shows a simplex design plot of a typical three factor mixture experiment. b) Shows the design space for the activator used in this study.

Table 1
Mixture constraints.

Low limit	Constraint	High limit
0.37	Water/slag (mass ratio)	0.47
1	Sand/slag (mass ratio)	2
0	A:SiO ₂ (grams)	6
25	B:Na ₂ CO ₃ *H ₂ O (grams)	50
200	C:Water (grams)	230
631	D:Sand (grams)	780
485	E:Slag (grams)	600
A + B + C + D + E = 1500 (grams)		

2.4. Characterization of samples

The ASTM standard test method for time of setting of concrete mixtures by penetration resistance (C403) [14] was used to determine initial setting time. A Soil Test Model CT-426 was used to perform the measurement. Final setting time was not considered in this study. Analysis of the data was carried out in accordance with this standard.

Compression testing was performed on each of the samples after maturing for 28 days in the laboratory (22 °C, 73% RH). Compression testing was done based on the ASTM C109 standard [15]. In this study two cubes were tested for each experimental run. A Tinius-Olsen universal testing machine was used to determine the maximum compressive load. Scanning electron microscopy (SEM) was done with a Tescan Mira3 with a field-emission gun.

The statistical analysis of the data was carried out using MATLAB 8.3, Minitab 17.1, and Design Expert 9.0. MATLAB was used to perform the statistical model fitting required by the ASTM standard in determining initial setting time. Minitab and Design Expert were used to fit the models of initial setting time based on the mixture ingredients.

3. Results and discussion

The results for the initial setting time and compressive strength measured in each of the 24 design runs are shown in Table 2. The initial setting time results range from about 72 to 1484 min or an hour and 15 min to 1 day. This is a relatively large range. However, when considering the range of literature values (30 min to over 2 days) this design space provides important insight on what is effecting these large ranges. The compressive strength of the

samples ranged from 31 to 74 MPa. All of the values are above the minimum requirement for basic building and paving applications. In order to quantify these effects, models were fit to the data obtained using the least squares technique. While the experiment was designed to be able to model the design space using a Sheffe Quadratic model (main effects and two-factor interactions), in the initial setting time model the two-factor interactions were found to be insignificant.

3.1. Model of initial setting time

The ANOVA table testing for significance of regression and lack of fit is shown in Table 3. This is a statistical tests to assess whether or not any of the terms in the model contribute to explaining the response (initial setting time in this case). The small p-value associated with the linear mixture models suggests that the components in our mixture contribute to changes seen in the initial setting time. Each of the main effects (the five individual components shown in Table 1) were found to be significant at a confidence level of 95%. The two-factor interactions for initial setting time were found to be insignificant, as indicated by the lack of fit in the ANOVA output. The model with only the individual mixture components had R² = 0.8426, R²-adjusted = 0.8006 and a standard error of approximately 89 min. These values suggest that this model provides a good fit to the data. In general, R² values above 0.7 are considered fairly high.

While R² values provide some indication of how well our model is fitting the data (closer to 1 typically better, but can be inflated by over-fitting), it doesn't provide the entire picture. It is important to consider the overall quality of the model in terms of residual analysis, check for validation using additional experimental runs, and study the model from a subject matter expert perspective in terms of the meaning of these results. The residual standard error in our model is somewhat high and the predicted R² is 0.62, which is a bit low. This is probably due in part to the small sample size and in part due to the large variation in the initial setting time. We still feel confident that our model is able to explain changes to setting time and provides us with direction on the settings of our variables needed to achieve a desired initial setting time.

Verification of the model adequacy indicate that the residuals were normally distributed and with constant variance, but run number 24 was identified as an outlier using Cook's Distance analysis and thus, this observation was removed from the model.

Table 2
Experimental run order with initial setting time results. The ingredients are given by weight in grams and Initial setting time in minutes. Compressive strength is shown in MPa.

Block # (day of production)	Run order	A: SiO ₂ (grams)	B: Na ₂ CO ₃ (grams)	C: H ₂ O (grams)	D: Sand (grams)	E: Slag (grams)	Initial setting time (min)	Compressive strength (MPa)
1	1	3.13	25	213	688	569	513	45
1	2	6	50	200	703	540	446	61
1	3	0	38.9	200	776	485	202	65
1	4	0	25	230	645	600	630	43
1	5	0	36.6	226	694	542	407	60
1	6	6	50	218	740	485	634	63
2	7	3.35	50	230	638	578	318	55
2	8	2.92	36.2	215	713	531	461	51
2	9	6	37.2	222	634	600	544	51
2	10	2.92	36.2	215	713	531	300	39
2	11	0	50	200	738	511	72	58
2	12	0	25	200	734	540	239	40
3	13	0	25	217	772	485	487	31
3	14	2.37	50	200	762	485	336	70
3	15	6	25	230	694	544	792	40
3	16	3	37.5	212	719	527	623	65
3	17	3	37.5	212	719	527	628	56
3	18	0	50	217	645	587	126	66
4	19	0	50	230	730	489	229	63
4	20	6	25	200	780	489	811	53
4	21	6	50	211	669	562	312	74
4	22	0	29.8	230	664	576	390	62
4	23	0	25	200	758	516	381	62
4	24	3.25	33.5	230	743	489	1484	53

Cook's Distance is a statistical calculation involving residuals (differences between actual observations and fitted equation) that measures the effect of deleting a given observation. The main effects equation that can be used to predict initial setting time is provided in equation (2).

$$\begin{aligned} \text{Initial setting time (Minutes)} = & 55.41493 (\text{grams SiO}_2) \\ & - 11.32 (\text{grams Na}_2\text{CO}_3) \\ & + 5.16 (\text{grams H}_2\text{O}) \\ & + 0.118 (\text{grams Sand}) \\ & - 0.882 (\text{grams Slag}) \end{aligned} \quad (2)$$

Within the mixture constraints, equation (2) predicts that for a fixed water/slag and slag/sand ratios, as the amount of Na₂CO₃ increases the initial setting time decreases and, as the amount of

results based on the compressive strength data in displayed in Table 4. These results indicate that the individual mixture components as well as three two-factor interactions are important contributors in explaining changes in compressive strength. The residuals base on this model were found to be normally distributed and had constant variance. Additionally, no outliers were identified using the Cook's Distance Metric. The main effects of B (Na₂CO₃), D (slag) and E (sand) were found to be significant effects in the linear mixture portion of the model. The prediction equation for this model is given in equation (3). An R² value of 0.8534, R²-adjusted of 0.7744, a Standard error of approximately 5 MPa, and R² predicted of 0.4542 were obtained for the model. The insignificant main effects were left in the predictive equation in order to retain model hierarchy, which causes the lower R² adjusted as well as R² prediction. We feel that this model provides interesting insights to our research.

$$\begin{aligned} \text{Compressive Strength (MPa)} = & 1.30894 (\text{grams SiO}_2) - 57.46157 (\text{grams Na}_2\text{CO}_3) - 0.80877 (\text{grams H}_2\text{O}) \\ & - 0.00150258 (\text{grams Sand}) + 0.26552 (\text{grams Slag}) + 0.052 (\text{grams Na}_2\text{CO}_3) (\text{grams H}_2\text{O}) \\ & + 0.041074 (\text{grams Na}_2\text{CO}_3) (\text{grams Sand}) + 0.036474 (\text{Na}_2\text{CO}_3) (\text{grams Slag}) \end{aligned} \quad (3)$$

SiO₂ increases so does the initial setting time. For alkali activation to occur, a high pH solution is necessary, in this case the rise in pH is produced by the dissolution of the Na₂CO₃ in water. The addition of the amorphous SiO₂ in the solution tends to reduce the pH of the solution therefore a relationship between these variables and the initial setting time exists. This relationship between Na₂CO₃ and SiO₂ as predicted by equation (2) is in agreement with previous observations made by Krivenko [16], Chang [17] and Bakharev [6].

3.2. Model of compressive strength

The ANOVA table for the test for significance of regression

This study shows that within the mixture constraints, the amount of SiO₂ did not contribute in a statistically significant way to the compressive strength. While Na₂CO₃ has the largest negative coefficient in equation (3), when the two factor interactions are accounted for it actually ends up having the largest net positive effect on the compressive strength. It is the two factor interactions between Na₂CO₃ and H₂O, Na₂CO₃ and slag and Na₂CO₃ and sand that show a statistically significant positive effect on the compressive strength. This work suggests that the relationship between Na₂CO₃ and compressive strength is in fact much more

Table 3
ANOVA table for the initial setting time results.

Source	Sum of squares	df	Mean square	F Value	p-value prob > F	Significance
Block	1.086E5	3	36213.77			
Model	6.378E5	4	1.594E5	20.07	<0.0001	significant
Linear Mixture	6.378E5	4	1.594E5	20.07	<0.0001	
Residual	1.192E5	15	7943.81			
Lack of Fit	1.062E5	13	8168.35	1.26	0.5271	not significant
Pure Error	12968.50	2	6484.25			
Cor Total	8.656E5	22				

Table 4
ANOVA table for compressive strength results.

Source	Sum of squares	df	Mean square	F Value	p-value prob > F	Significance
Block	441.31	3	147.10			
Model	1974.51	7	282.07	10.81	0.0002	significant
Linear Mixture	1725.26	4	431.31	16.53	<0.0001	
BC	202.34	1	202.34	7.75	0.0155	
BD	214.17	1	214.17	8.21	0.0133	
BE	168.43	1	168.43	6.45	0.0246	
Residual	339.30	13	26.10			
Lack of Fit	220.47	11	20.04	0.34	0.9066	not significant
Pure Error	118.83	2	59.41			
Cor Total	2755.11	23				

Table 5
Model Confirmation run ingredients.

Run	SiO ₂ (grams)	Na ₂ CO ₃ (grams)	Water (grams)	Sand (grams)	Slag (grams)
Run 1	0	34.38	200	725	540
Run 2	0	47.8	200	767	485

Table 6
Model Confirmation results.

	Initial setting time			Compressive strength		
	Experimental (min)	Predicted (min)	Within 95% PI	Experimental (MPa)	Predicted (MPa)	Within 95% PI
Run 1	210	251.8	yes	61.6	56.8	yes
Run 2	167	153.2	yes	64.3	66.2	yes

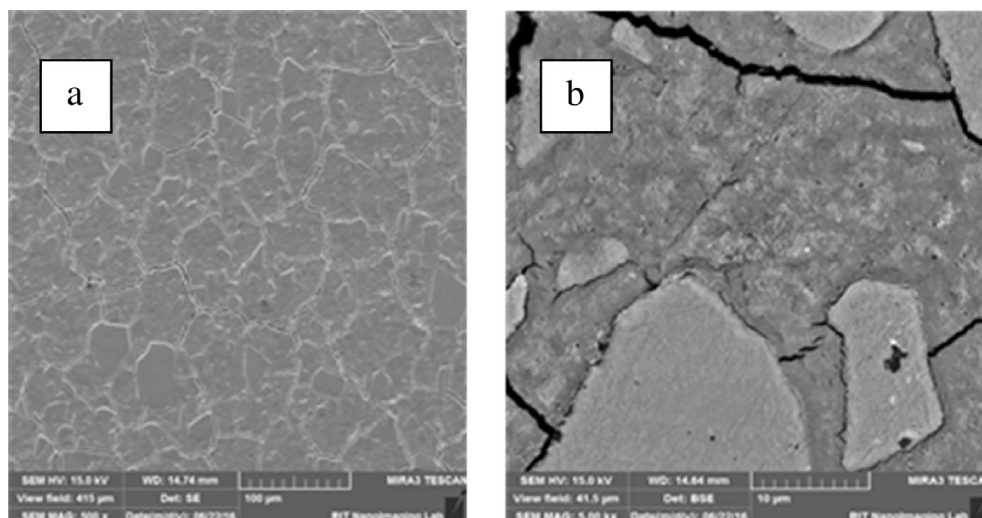


Fig. 2. SEM images from sample obtained in run 21 a) Shows an overview of the sample at 500X b) provides a higher magnification a specific area at 5000X.

nanced than a simple linear relationship.

4. Model confirmation

In addition to the replicated runs performed in the original design of experiments (runs 10 and 17 in Table 2) two model confirmation runs were performed five weeks after the initial experiment design using the same experimental techniques. The model confirmation runs were performed to test the accuracy of the mathematical models. The ingredients used in each of the confirmation runs are listed in Table 5 and the experimental and predicted values initial setting time and compressive strength are presented in Table 6. It can be seen that each of the confirmation runs falls within the 95% prediction interval of the model. This confirms the validity of the model showing that it is possible to predict, within the initial constraints, the initial setting time and compressive strength of the sodium carbonate activated blast furnace slag samples based on the ingredients used in each sample.

5. Scanning electron microscopy

The SEM imaging was done on paste samples instead of mortar samples. The mixture chosen for this phase of the study was run 21 in Table 2. This mixture was chosen due to the high compressive strength of the sample. A small piece of the inner portion of the sample was embedded in epoxy resin and polished with diamond embedded polishing film. The sample was then sputter coated with palladium and gold and then placed into the SEM chamber. Imaging was done using the backscattered electron detector. The so-called BSE image is sensitive to the atomic number of the elements in the sample.

The SEM images for this paste are shown in Fig. 2. At low magnification the sample shows unreacted particles of slag surrounded by an amorphous surface and an irregular array of microcracks. At the higher magnification no particular formations were observed either, but areas with different brightness in the region contiguous to the slag particles were observed. The brighter areas in these images indicate regions of higher atomic number, and is indicative of the presence of different phases. The identification of these phases is the subject of ongoing research.

6. Conclusions

This research has shown it is possible to produce sodium carbonate activated blast furnace slag mortars that meet initial setting time and compressive strength required by ASTM standards. Mixtures with specific properties can be designed using the prediction equations stated above. Statistical mixture design provides a valuable tool to AAM researchers in predicting properties of a material over an area of interest. The use of the mixture model allows the

properties of the mixture to be predicted from the components while the amount of the mixture is held constant.

Sodium carbonate is a significant factor in the initial setting time. As the amount of sodium carbonate in the samples increased the initial setting time decreased. In the compressive strength model a more intricate relationship between sodium carbonate and the response was observed. It was so shown that the amount of amorphous SiO₂ and the range of water used did not have a significant effect on the compressive strength of the samples.

Further study of the causes of the two-factor interactions in the compressive strength analysis and the identification of the different phases detected by backscattered electron are areas of ongoing research.

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