Optimization of Cementitious Grouts for Semi-Flexible Pavement Surfaces Using Response Surface Methodology

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Abstract. In recent years, semi-flexible pavement surfaces have been identified as one of the alternatives to conventional rigid and flexible pavements due to its advantages over both the pavements. In semi-flexible pavement surfaces, the cementitious grouts are spread on the surface of a porous asphalt skeleton and allowed to penetrate through the depth. The cementitious grouts contribute to the durability (in terms of resistance to oil, water and adverse weather conditions) and the performance of semi-flexible pavement surfaces. The physical and performance properties of semi-flexible pavement depend on the voids in the porous asphalt skeleton and suitable compositions of cementitious grouts. This paper presents the design and optimization of compositions of cementitious grouts using Response Surface Methodology (RSM) technique. Similarly, statistical models are developed to predict the flow and compressive strength properties of grouts using RSM. Water-cement ratio (w/c) and Superplasticizer (SP) were chosen as two independent variables, and their effect on flow and compressive strength properties were investigated. Based on RSM analysis, the adjusted R2 was in reasonable agreement with predicted R2 because the difference was less than 0.2, and the models were found to be significant. Finally, the optimized compositions of grouts were validated by performing experimental program.

1. Introduction
Semi-flexible pavements are gaining popularity due to their advantages over conventional flexible and rigid pavements during the last few years. It comprises of porous asphalt skeleton with 20 to 30% voids over which highly flowable cementitious grout is spread and allowed to penetrate. The physical and strength properties of cement grouts mainly depend on the proper selection of various compositions of cementitious grouts as well as admixtures and other supplementary cementing materials. For example, water-cement (w/c) ratio, superplasticizer and silica fume have a significant effect on flowability and compressive strength of grouts [1-3]. In a study, it was revealed that increasing w/c ratio improves the workability while reduces the compressive strength of cement grouts and hence admixture and superplasticizer were introduced to produce high performance cementitious grouts [4-5].

Various studies have been conducted to evaluate the strength, stability and durability properties of cementitious grouts [2, 6]. However, there is a need to optimize the proper compositions of cementitious grouts using statistical tools. Therefore, Response Surface Methodology (RSM) is used in this study to optimize the compositions of cementitious grouts based on w/c ratio and dosage of superplasticizer as two independent variables (factors). RSM is a powerful statistical tool for designing the experiment with a smaller number of experiments, developing statistical models for dependent variables (Responses),
establishing a relationship between factors and responses and finally developing a model to predict the output [7-11]. RSM has been widely used in the concrete industry to optimize and model the experimental outputs [12]. In a study, a statistical model was developed using RSM for three main variables (molarity of NaOH, Na2SiO3/NaOH, and curing temperature) and their effect on fresh and hardened geopolymers were evaluated [7]. Similarly, in another study, six different factors and compressive strength as a response to develop statistical models [13]. Although statistical modelling and optimization using RSM have been widely used in concrete, however, its utilization in optimization and modelling in the field of cementitious grouts for semi-flexible pavements is in the very beginning stage. Therefore, the main objective of this study is the optimization and statistical modelling for selecting the best combination of independent variables to predict the flow and strength properties of cementitious grouts for semi-flexible pavement surfaces.

2. Experimental program

2.1. Materials

In this work, Ordinary Portland Cement (OPC) was used and was obtained from a local supplier. Master Glenium® ACE 8538, a new generation Superplasticizer (SP), was used to produce high flowable grouts at relatively low water-cement (w/c) ratios. Master Glenium® ACE 8538 is a Polycarboxylate-ether type polymer used as a superplasticizer and was supplied by BASF Sdn Bhd, Malaysia. This type of SP is compatible with all types of cement and producing high quality of concrete at low w/c ratio and desirable slump value.

2.2. Design of experiment using Response Surface Method

In the current study, commercially available Design Expert® 11.1.2 was used for design, optimization and statistical modelling. Two parameters, water-cement ratio (X1) and Superplasticizer dosage (X2) were identified as independent variables that denote the factors, while flow value (Y1) and Compressive Strength (Y2) represents the responses. The factors and their respective units and codes for the design of the experiment is shown in Table 1.

| Factors         | Units | Code | Levels |
|-----------------|-------|------|--------|
| w/c ratio       | --    | X1   | -α    |
| Superplasticizer| %     | X2   | 0      |

The range of w/c ratio (0.25 to 0.45) and Superplasticizer (0 to 2) was selected based on literature review in order to optimize w/c ratio and superplasticizer content which satisfy the minimum requirement of flow and compressive strength [2-3, 15-16]. Based on two factors, a total of 16 mix designs of cementitious grouts were chosen using Central Composite Design in the RSM technique, as shown in Table 2.

\[
Y = \beta_0 + \sum \beta_{ii}X_{ii} + \sum \beta_{i}X_i + \sum \beta_{ij}X_iX_j
\]
Table 2. Mix design of cementitious grouts in Design Expert®.

| Run | Factor 1 (w/c ratio) | Factor 2 (Superplasticizer %) | Run | Factor 1 (w/c ratio) | Factor 2 (Superplasticizer %) |
|-----|----------------------|-------------------------------|-----|----------------------|-------------------------------|
| 1   | 0.35                 | 0.5                           | 9   | 0.40                 | 1.5                           |
| 2   | 0.30                 | 2.0                           | 10  | 0.35                 | 1.0                           |
| 3   | 0.25                 | 0                             | 11  | 0.25                 | 1.0                           |
| 4   | 0.45                 | 0                             | 12  | 0.30                 | 0                             |
| 5   | 0.45                 | 2.0                           | 13  | 0.35                 | 1.0                           |
| 6   | 0.30                 | 2.0                           | 14  | 0.40                 | 0.5                           |
| 7   | 0.35                 | 1.0                           | 15  | 0.45                 | 0                             |
| 8   | 0.45                 | 2.0                           | 16  | 0.25                 | 1.5                           |

2.3. Mixing and preparation of specimens
The mechanical mixer was used to produce cementitious grouts at different w/c ratio and superplasticizer in accordance to the ASTM C305-14. A modified method of mixing the cementitious grouts was also used as adopted by Zoorob et al. [17]. In this method, the required quantity of Ordinary Portland Cement (OPC) was placed in 5 litres bowl. Initially, about 50% of the water was added, followed by remaining water containing superplasticizer. Mixing by this modified method gives improved workability by minimizing the absorption of Superplasticizer by cement binder. Flow cone test to measure flowability of the grout was conducted on fresh grouts while compression test on 50 mm cubes were performed on hardened grouts. The compressive strength test on cubes was performed at 1, 7 and 28 days curing period.

3. Results and discussion

3.1. Flow properties of fresh cement grout
The purpose of the flow test is to ensure that the proposed grout has efficient fluidity before it is poured on the surface to penetrate into the porous compacted asphalt skeleton. In this test, the flow-out time of cementitious grout is measured. The time of flow depends on the type of funnel, and the quantity of grout poured into the funnel. In the current study, the Malaysian flow cone was used in accordance with the City Hall Kuala Lumpur (CHKL) and REAM specifications [18-19].

According to the specifications, one liter of fresh cement grout was transferred to the flow cone, and the flow-out time was measured by stopwatch when the flow cone become empty. The flow-out time of one-liter grout shall be in the range of 11 to 16 seconds as per specifications. The flow-out time corresponds to the workability of cementitious grouts, less the flow time, more the workability due to lower viscosity. The interparticle lubricant is increased by adding water to cement, which causes a reduction in viscosity and hence the flow-out time of grouts [1]. The same is witnessed in this study, the flow-out time reduced with adding water and superplasticizer because the addition of water content and SP is inversely proportional to flow-out time.

The three dimensional (3-D) and contour diagrams of flow time are represented in Figure 1(a) and Figure 1(b), where w/c ratio and superplasticizer are the two independent variables (factors).

It can be seen from the analysis that the flow-out time of cement grouts reduces (from 105.8 sec the highest to 6.2 sec the lowest) with an increase in w/c ratio from 0.25 to 0.45. Similarly, a considerable reduction in flow-out time of grouts has been witnessed with an increase in the percentage of Superplasticizer. However, the increased percentage of superplasticizer beyond 1.5%, especially at high w/c ratio, causes bleeding problems in the grouts. Polycarboxylate-Ether type superplasticizer is very useful in improving the workability of cementitious grouts in reducing the flow-out time. This superplasticizer is also a very suitable admixture for precast and ready mixed concrete [1]. Superplasticizers disperse cement particles in suspension, and the particles are held far enough from each other [20]. In the current study, RSM was used for design and validating the experimental results. ANOVA equation (2) was used to predict the flowability of the cementitious grouts.
Flow = $+368.65 - 1353.23x_1 - 122.71x_2 + 210.81x_1x_2 + 1235.11x_1^2 + 13.64x_2^2$  \hspace{2em} (2)

where $x_1$ and $x_2$ are the independent variables representing w/c ratio and Superplasticizer respectively.

Figure 1. Effect of w/c ratio and superplasticizer on flow of grout (a) 3-Dimensional response surface (b) Contour diagram.

3.2. Compressive strength of hardened cement grout

Cubes of 50 x 50 x 50 mm were prepared from different cementitious grouts. A compressive strength test was performed on cubes at 1, 7 and 28 days curing. The ELE Universal Testing Machine with a capacity of 3000 kN was used at a pace rate of 0.90 kN/s. The three dimensional (3-D) and 2-D contour diagrams of compressive strength are shown in Figure 2 to Figure 4, where w/c ratio and superplasticizer are the two main factors. It can be seen from Figure 2 to Figure 4 that there is a significant reduction in compressive strength at 1, 7 and 28 days curing with the rise in w/c ratio.

Figure 2. Effect of w/c ratio and superplasticizer on compressive strength (1-day curing) of grout (a) 3-Dimensional response surface (b) Contour diagram.
The compressive strength also increased with increasing curing time. However, the strength initially increases with an increase in the dose of superplasticizer up to 1.0% and then gradually decreases, as shown in Figure 2 to Figure 4 (part-a). In the current study, RSM was used for the design and validation of the experimental results. ANOVA equations (3) to (5) were used to predict the compressive strength of cementitious grouts at 1, 7 and 28-days curing, respectively.

Compressive Strength (1-Day) = +154.59 − 724.94x_1 + 15.09x_2 − 2.86x_1x_2 + 905.85x_1^2 − 8.82x_2^2  
(3)

Compressive Strength (7-Days) = +35.52 + 131.76x_1 + 24.33x_2 − 43.30x_1x_2 − 382.29x_1^2 − 4.78x_2^2  
(4)

Compressive Strength (28-Days) = +81.11 + 20.90x_1 + 15.72x_2 − 19.08x_1x_2 − 325.60x_1^2 − 5.17x_2^2  
(5)

where x_1 and x_2 are the independent variables representing w/c ratio and Superplasticizer respectively.
3.3. Validation of models using the ANOVA approach

The effect of both w/c ratio and superplasticizer’s dosage on flow and compressive strength are significant as represented ANOVA results in Table 3 and Table 4. The adjusted $R^2$ and predicted $R^2$ are in good agreement for all responses because the difference is less than 0.2. Furthermore, the models can be accepted as all responses have adequate precision greater than 4, and these models can be satisfactorily used to predict the flow and compressive strength (1, 7 and 28 days curing) for desired factors. Similarly, the analysis of variance for all responses gives a higher value of F and lower value of p (which is < 0.005) justify the significance of models [7].

Table 3. Validation of ANOVA models for responses.

| Response                  | Flow (sec) | Compressive Strength (MPa) |
|---------------------------|------------|----------------------------|
|                           |            | 1 Day | 7 Days | 28 Days |
| Standard deviation        | 5.93       | 1.40  | 2.10   | 4.02    |
| Mean                      | 24.41      | 16.51 | 37.70  | 46.83   |
| $R^2$                     | 0.9734     | 0.9841| 0.9840 | 0.9641  |
| Adjusted $R^2$            | 0.9601     | 0.9762| 0.9759 | 0.9461  |
| Predicted $R^2$           | 0.9452     | 0.9539| 0.9525 | 0.8854  |
| Co-efficient of Variance (%) | 24.30   | 8.48  | 5.58   | 8.59    |
| Adequate Precision        | 30.1330    | 36.6326| 33.0495| 20.3586 |

Table 4. Analysis of variance (ANOVA) results for each response.

| Response                  | Flow (sec) | Compressive Strength (MPa) |
|---------------------------|------------|----------------------------|
|                           |            | 1 Day | 7 Days | 28 Days |
| Sum of Squares            | 12885.02   | 1213.06| 27.18  | 4343.89 |
| Mean Square               | 2577       | 242.61 | 543.75 | 868.78  |
| F-Value                   | 73.22      | 123.85 | 122.72 | 53.65   |
| p-Value                   | <0.0001    | <0.0001| <0.0001| <0.0001 |
| Remarks                   | Significant| Significant| Significant| Significant |

3.4. Optimization and validation of results

RSM is also a significant statistical tool for the optimization of independent variables (factors) based on the responses. However, it is not practical to get optimized factors based on all individual responses. Hence, the multi-objective consideration of all responses in parallel is the key solution to get optimized factors. In the current study using RSM, the graphical representation of an optimized combination of grouts with corresponding optimum responses is presented in Figure 5.

Figure 5. RSM based graphical representation of the optimized combination of grouts.
In this study, the water-cement ratio and superplasticizer were kept “in range,” while the Flow and Compressive strengths were selected as “Maximized” for the optimization process in RSM.

After the accomplishment of the optimized factors, experiments were performed using the optimal mix such as 0.30 w/c ratio and 1.45% superplasticizer to validate the predicted value of responses (i.e. flow and compressive strengths).

Table 5. Experimental vs predicted results.

| Factors          | Response                  | Predicted Results | Experimental Results | % difference |
|------------------|---------------------------|-------------------|----------------------|--------------|
| w/c ratio = 0.306| Flow (sec)                | 15                | 15.4                 | 2.67         |
| Superplasticizer | 1 Day Compressive Strength (MPa)| 19.94           | 20.8                 | 4.31         |
|                  | 7 Days Compressive Strength (MPa)| 49.15           | 51.49                | 4.76         |
|                  | 28 Days Compressive Strength (MPa)| 60.64           | 57.91                | 4.50         |

Table 5 represents the comparison between predicted results from RSM for the optimized grouts and the experimental results. All the results are found to be in close agreement to the predicted values with less than 5% variation, as shown in Table 5.

4. Conclusions
Response Surface Methodology was used in this study for the design of experiments and for the establishment of the optimum compositions of cementitious grouts to achieve the target responses. Following conclusions are drawn from this study:

- The flow of grout is highly influenced by the dosage of superplasticizer. The flow-out time is decreased significantly with increasing superplasticizer from 0.5 to 2%. Similarly, increasing w/c ratio from 0.25 to 0.45 also plays an important role in the flow characteristics of cementitious grouts. However, increasing superplasticizer from optimum dosage causes bleeding and segregation in grouts.
- Increasing w/c ratio from 0.25 to 0.45 causes a significant reduction in compressive strength at all curing ages. However, gradual change in compressive strength was witnessed with an increasing dosage of superplasticizer.
- The correlation between factors (independent variables) and responses (dependent variables) have been efficiently observed with 3-Dimensional diagrams. The desired responses can also be predicted from the modelled equations.
- The ANOVA models in RSM of flow and compressive strengths for cementitious grouts have been developed and experimentally validated.
- Using the RSM optimization technique for the design of experiments reduces the experimental work and hence the time to produce various trials of mixes.

5. References
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