Conference paper

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Factorial Design Applied to Waste Immobilisation in Geopolymer-based Systems

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Abstract: Concentrated alkali, ground glass and air pollution control residues were mixed in various proportions and cured for up to 28 days. These blocks were tested in strength and analysed by thermal gravimetric analysis to assess the success of the geopolymerisation process. A Taguchi factorial design approach was then adopted to investigate the effects of different variables upon the strength development of these blocks.

Results

Six different mixes were prepared, as shown in Table 1. Mixes A-C were prepared with an aged (carbonated) APC residue, whilst mixes 1-3 were prepared with a fresh (uncarbonated) residue. From these mixes, and the corresponding elemental composition obtained by XRF, the data in table 2 could be obtained. The compressive strength of each mix was then determined, with the average strengths based on triplicate measurements shown in tables 3 and 4.

The results show that the compressive strengths vary greatly between the carbonated and fresh APCs, and there were far greater variations in UCS found in the aged APC, due most likely to sample heterogeneity.

Table 1: Composition of the six mixes investigated.

	APC(g)	Glass(g)	Caustic(g)	Weight total (g)	Liq/Solid
Mix 1/A	105	250	270	625	0.761
Mix 2/B	150	200	310	660	0.886
Mix 3/C	218.8 *	109.09	272.72	600.61	0.832

Table 2: Key composition data fr the mixes, as used to determine the factors.

	Si/Al	%CaO	$Ca(OH)_2$	CaO/(Al+Si)	% Cl
Mix 1/A	16.53	11.04	1.135	0.360	2.34
Mix 2/B	12.85	12.33	2.350	0.522	3.16
Mix 3/C	9.64	14.02	4.132	0.807	4.08

Table 3: Compressive strength test results for aged (carbonated) APC

	Strength Result (kN) Test Number			Strength Result (MPa) Test Number			Average Strength (MPa)	Standard Deviation
Mix	1	2	3	1	2	3		
A	19.75	13.85	12.20	7.90	5.54	4.88	6.11	1.59
B	5.00	2.49	3.25	2.00	1.00	1.30	1.43	0.51
C	8.10	4.95	5.10	3.24	1.98	2.04	2.42	0.71

Table 4: Compressive strength test results for fresh APC

	Strength Result (kN) Test Number			Strength Result (MPa) Test Number			Average Strength (MPa)	Standard Deviation
Mix	1	2	3	1	2	3		
1	9.50	10.50	9.85	3.80	4.20	3.94	3.98	0.20
2	14.50	12.85	13.00	5.80	5.14	5.20	5.38	0.36
3	6.50	5.25	5.00	2.60	2.10	2.00	2.23	0.32

Factor analysis was then performed on these data to ascertain the influence of some variables on strength development. In many areas of engineering the performance of a manufactured product is affected by a large number of parameters or factors, and factor analysis is an effective method to optimise manufacturing parameters so that improved engineering tolerances can be achieved in future designs. Factor analysis has been used in many experimental design processes a good example of which would be by Saravanan[1] although the scope of this factor analysis is much smaller its potential benefits still exist.
In this study 4 factors were selected to find their potential effect upon the quality characteristic of compressive strength:

- Si/Al ratio of original mixes (H)
- Ca $(OH)_2$ % of mixes (I)
- Fresh or aged APC used (J)
- Cl % content in original mix (K)

These factors were chosen for several reasons primarily because they are all known quantities that could have resulted in lack of solidification and strength development found in the product. The potential factors that could possibly be used in the analysis were also heavily influenced because of the nature of the samples

Table 5: Factors chosen and their levels (data calculated from table 2)

Factors	Levels		
	1	**2**	**3**
(H) Si/Al ratio	9.64	12.85	16.53
(I) $Ca(OH)_2$ %	1.135	2.350	4.132
(J) Fresh/aged APC source	Fresh	Aged	N/A
(K)Cl% content in orginal mix	2.34	3.16	4.08

Table 6: Matrix Experiment showing assigned factor levels

Expt. No.	Factor Assigned				Observation n Strength (MPa)
	Si/Al (H)	**$Ca(OH)_2$ % (I)**	**Fresh/Aged (J)**	**Cl content % (K)**	
1	3	1	1	1	3.98
2	2	2	1	2	5.38
3	1	3	1	3	2.23
4	1	1	2	1	6.11
5	2	2	2	2	1.43
6	3	3	2	3	2.42

The experimental design is shown in table 6, it consists of 6 individual experiments corresponding to 6 rows with the 4 columns of the matrix representing the four factors and their levels as indicated in table 7, the final response found is given by the end column. As an example the settings of experiment 4 is H1, I1, J2, and K1.

Due to problems with obtaining data from all of the samples the matrix in table 6 isn't the standard orthogonal array (L9)[2], which had 9 rows so all columns were mutually orthogonal. However our 6 row matrix does show a level of a balancing property with only column J not mutually orthogonal **-** for any pair of columns every combination of factor levels occur and they occur the same number of times in each column this is known as a balancing property.

Once the individual experiments set out in table 10 had been completed, statistical analysis of the results was performed to assess the influence of individual process factors on, in this case, compressive strength. By using the known average compressive test results (n) as shown in tables 3 and 4 it is possible to establish the individual factor effects for the 6 experiments identified in table 6. The overall mean value of 3.59 MPa was found from the data by equation 1.

Equation 1

$$m = \frac{1}{6}\sum_{i=1}^{6} n_i$$

The effect of a factor level is classed as the deviation it causes from the calculated overall mean, for example evaluating the effect of Si/Al (H) at factor level 2 is shown in equation 2.
As the Si/Al ratio was at factor level 2 for experiments 2 and 5 the average compressive strength or S/N (signal to noise ratio) is:

Equation 2

$$m_{H2} = \frac{1}{2}(n_2 + n_5)$$

Therefore m_{H2}=3.405
This assessment was performed for each of the factors under investigation for example the S/N for fresh APC as the source material would be given as shown in equation 3, three n terms are present because fresh APC was used in experiments 1, 2 and 3.

Equation 3

$$m_{J1} = \frac{1}{3}(n_1 + n_2 + n_3)$$

Therefore m_{J1}=3.32
The numerical values of n from table 6 were used to calculate the average values for all the factors at each of their levels and is shown in Figure 1. This process is known as the analysis of means (ANON) and can often help identify what is known as the main effects for the product.

The factor analysis does show some basic relationships and potentially useful information about the effects of each factor upon the mix, figure 5 shows that average MPa strengths will be lower when factor level 3 is used for factors H, I and K and when level 2 is used for factor J. Figure 1 supports the theory therefore that higher levels of Si/Al ratios, Cl content, Ca $(OH)_2$ % all contribute to a lower compressive strength as does using the aged APC. Figure 5 also shows that Ca $(OH)_2$ and Cl content have the largest most profound impact upon the mixes resulting compressive strength, when these factors are at their lowest levels increases in compressive strength are observed.

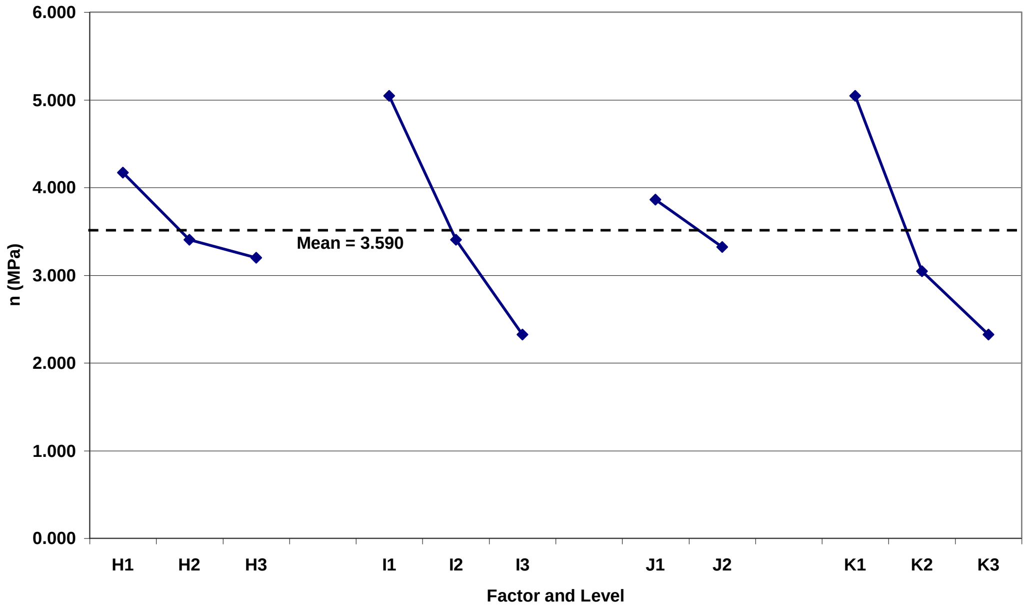

Figure 1: Plots of factor effects with mean line added

Conclusions

Factor analysis indicated potential effects of certain parameters upon each of the mixes' compressive strength. Although the results should be treated with some caution, as the error variance was high, it is possible to draw some generalised conclusions from the analysis. High levels of chloride yielded lower strengths, supporting suggestions that chloride reduces potential bonding and therefore strength between the aggregate and binder. Higher Si/Al ratios resulted in strengths, in agreement with others[3,4]. The findings also showed that the carbonated APC, as used in samples A, B and C, gave lower strengths, although factor analysis indicated less of a response than Si/Al ratios, chloride levels or Ca $(OH)_2$ content. Factor analysis also proposed that higher levels of Ca $(OH)_2$ in the mixes composition caused lower strengths, possibly due to unsoundness and incomplete mixing.

References

1 Saravanan P, Selvarajan V, Rao D.S, Joshi S.V, Sundararajan G (2000) "Influence of process variables on the quality of detonation gun sprayed alumina coatings." Surface and Coatings Technology 123:44–54

2 Taguchi G, Konishi S (1987). "Orthogonal arrays and linear graphs." Dearborn,MI: ASI Press

3 Khale D, Chaudhary R (2007). "Mechanism of geopolymerization and factors influencing its development: a review." Journal of Material Science 42:729-746.

4 Fletcher R, MacKenzie K.J.D, Nicholson C.L, Shimada S (2005). "The composition range of aluminosilicate geopolymers." Journal of the European Ceramic Society 25:1471–1477.