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A simplified mix design procedure for geopolymer cement mortars based on metakaolin and industrial waste products activated with potassium silicate

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ABSTRACT: This paper presents a mix design methodology for geopolymer mortars based on metakaolin and industrial waste products activated using potassium silicate. The work is aimed at enabling performance-based specification and compressive strength prediction to drive forward their adoption as an alternative to Portland cement-based mortars used in fibre reinforced cladding systems. Few studies currently quantify the effects of mix parameters on broad families of geopolymer materials and no standard mix design methodology exists. Resultant mortars must have high strength to create light, thin panels, have high flow to enable effective dispersal of reinforcement fibres and as low an environmental impact as possible to maximise the impact of replacement. For a standard geopolymer mix, the effect of binder composition on mechanical performance and environmental impact is initially studied using ternary contour maps for a range of material blends. Next, the effects of altering mixture parameters such as the liquid/solid, silica/alumina and activator/binder ratios are quantified for three binder compositions identified as having high performance. Finally, correlation analysis is used to identify mix variables strongly correlating with compressive strength and regression analysis of the most deterministic to create a prediction models. Geopolymer mortars have been developed with compressive strengths over 80 and 100 N/mm^2 at 7 and 28 days respectively and the methodology presented allows design of such mortars by non-experts. Model predictions of compressive strength is shown to be relatively accurate, with average errors across binder compositions ranging from 2.3-5.8%. Further research expanding the range of materials and mix compositions is ongoing to advance this innovative methodology further.

KEY WORDS: Geopolymer; Mortar; Metakaolin; Industrial waste; Potassium silicate; Liquid to solid ratio; Mix design; Compressive strength, Prediction.

1. INTRODUCTION

There is a significant need for change in the way we design, build and use energy in our buildings. The materials currently used are struggling to keep up with demands for increasingly high levels of thermal performance, fire safety and finish to be achieved with increasingly limited environmental impact, greenhouse gases and energy allowances. The EU recognise this and plan to invest around €40 billion a year through schemes like the €5.9 billion horizon 2020 project by renovating existing buildings, making construction projects more sustainable and making all new builds require no energy from the grid by 2020 [1].

Geopolymer-based materials have the potential to form the next generation of cladding panel systems with improved performance over current alternatives such as glass reinforced concrete (GRC). Geopolymers form, cure and gain strength rapidly in ambient temperatures by combining water, user friendly alkaline reagents and alumina/silicate source materials that are either commercially produced, such as metakaolin, or industrial wastes, such as slags and ashes. The result are materials with a strong, durable, solid matrix that behaves like Portland cement (PC)-based concrete [2]. In comparison to GRC, geopolymers offer increased strength (over 20 and 100 N/mm^2 at 4-hours and 28-days respectively), and improved fire protection and chemical resistance. This is coupled with up to 90% reductions in embodied carbon and the use of a 100% recycled waste binder [3-5]. In this way, the material has the potential to offer the construction industry with a novel approach to producing high performance, lightweight cladding panel systems for buildings.

However, the lack of recognised, performance based mix design methodologies for geopolymer binder systems enabling attainment of specified strength and/or workability presents a major stumbling block to its widespread adoption [6]. Previous related research focusing on mix design methods has focussed on single proportioning ratios such as silica/ alumina (S/A), activating solution to binder powder (A/B) or liquid to solid (L/S); an approach analogous to the water/cement ratio in Portland cement-based concrete [7] While other studies have used multiple parameters synergistically to create empirical formulas and neural networks to predict strength [8], existing work describing generic relationships between geopolymer strength and key mix design parameters is limited; typically focusing on specific materials such as fly ash-based systems requiring curing at high temperature and/or sodium based activating solutions. As such, these methods are of little relevance to wider groups of geopolymer binder systems and potassium silicate activation.

Against this background, the aim of this study is to produce a methodology enabling strength prediction for potassium silicate activated geopolymer mortars comprising a wide range of binder combinations and mix parameters. In this way, the intention is to drive forward the adoption of these systems as a high performance, low impact alternative to PC-based materials such as GRC in building cladding components.

2 EXPERIMENTAL METHODOLOGY

2.1 Materials

Metastar 501 metakaolin (MK) from Imerys UK was used as the primary binder due its commercial availability, consistent and highly amorphous nature and its rapid dissolution and geopolymerization at ambient temperatures [9]. While MK has a low environmental impact compared to Portland cement (PC), partially or fully replacing it with industrial waste products has been shown to significantly reduce this impact and provide reduced set times, greater strength or higher flow [7]. The industrial waste materials used in this study included GGBS from ECOCEM Ireland, silica fume (SF) from Elkem, fly ash (FA) from Kilroot power station in Northern Ireland and iron silicate fines from Aurubis Bulgaria. Iron silicate is a low impact by-product of copper production and novel in its usage as a geopolymer source material. GGBS geopolymers require a much smaller amount of activator solids and, therefore, have lower environmental impact than metakaolin systems which have a lower Si:Al ratio necessitating a greater amount to be used for full dissolution to occur [10]. SF has been shown in the literature and in this project to increase the Si:Al of the binder to help with this and provide increased strength development between 7 and 28 days, especially at around 20% binder mass. Geosil activating solution with a potassium silicate solids content of 45% by mass was sourced from Woellner and used in all mix designs. Potassium, rather than sodium, silicate activator was chosen due to its reactivity and emergence as a cost-effective solution for geopolymer production [11]. Mortar mixes were studied in this work, with the lough sand fine aggregate component sourced from Stanley Emerson & Sons Ltd. Measured chemical compositions and published embodied CO_2 values for the binder materials considered are given in Table 1 with PC for comparative purposes.

2.2 Sample preparation and testing sequence

All samples for compressive strength testing were cast in 50 mm cubes, covered with plastic for 24 hours to ensure uniform drying conditions, then stored in a sealed container until testing at 7 and 28 days in accordance with BS EN 1015-11:1999. Ambient laboratory temperatures of approximately 20^0C were provided over this casting and curing period. Rheological behaviour was determined using flow table testing in accordance with BS EN 1015-3:1999 to ensure sufficient workability and minimal void creation when casting. While this method specifies a 250 mm-wide flow table, this was identified as too small for comparing high flows created during the binder variation studies. As such, the flow exhibited by many of the mixes produced in Phase 1 could not be compared accurately.

3 PHASE I – INFLUENCE OF BINDER COMPOSITION

3.1 Mix designs

From Table 1 it is evident that significant variation exists in the major oxide contents of the various binder materials considered, suggesting potential to achieve geopolymer mixes with a wide range of performance and embodied CO_2 levels. To investigate the impact of binder composition in this regard, a base MK only geopolymer mix with L/S and paste/sand ratios of 0.51 and 0.84 respectively was initially developed as part of a preliminary research phase (see Figure 1-a). This mix design was then held constant and replicated with the principal variation being binder powder composition, enabling investigation of effects on mortar compressive strength, flow and environmental impact. Binder combinations considered included MK/GGBS/FA, MK/GGBS/SF and MK/GGBS/IS, with a wide range of unary, binary and ternary binders considered for each by considering respective binder increments of 20% in the range 0-100% by mass. By adopting this approach, it was recognised that performance levels were likely to vary considerably and potentially beyond limits of suitability. MK-based mixes, for instance, are reported to require more liquids than fly ash or slag geopolymers to ensure monomer transport, full dissolution and reorganisation [6].

3.2 Phase I results and discussion

Plotted in Figure 1 for the MK/GGBS/SF mixes are contoured ternary graphs illustrating the significant influence binder powder composition has on geopolymer mortar strength at 7 and 28 days (Figures 1(b) and (c)). Plotted at the pinnacle of the ternary plots in Figure 1, the 100% MK mix attained 7 and 28 day strengths of 41.5 and 46 N/mm^2 respectively. Relative to this, performance levels ranging from 4-69.5 N/mm^2 at 7 days and 5-106 N/mm^2 at 28 days were attained by the various alternative binder combinations considered. GGBS has been shown to be a successful replacement for MK in high strength geopolymers, producing improved strength in binary blends at all increments of addition to maximums of 85 and 105 N/mm^2 at 7 and 28 days respectively. All binders exhibiting strengths over 50 N/mm^2 comprised at least 20%GGBS, suggesting the formation of CASH gel as vital to achieving high strength [9]. Binder blend 20%MK/80%GGBS was stronger than the 100% GGBS mortar, suggesting that geopolymer gels and CASH hydration products formed simultaneously and bonded well together as the latter expanded into the pores of the former to create a homogenous microstructure. SF offered significant strength increases to both MK and GGBS mortars at 28 days, despite reducing the 7-day strength of these unary blends. From 7 to 28 days the 80%MK/SF and 80%GGBS/SF binary mixes more than doubled in strength from 19 to 45 N/mm^2 and 45 to 106 N/mm^2 respectively, producing the highest compressive strength measured in the study. As suggested previously this is likely due to an increase in the amount of Si-O-Si bonds present in the mix caused by the increasing silica to alumina ratio.

Table 1. Binder material properties

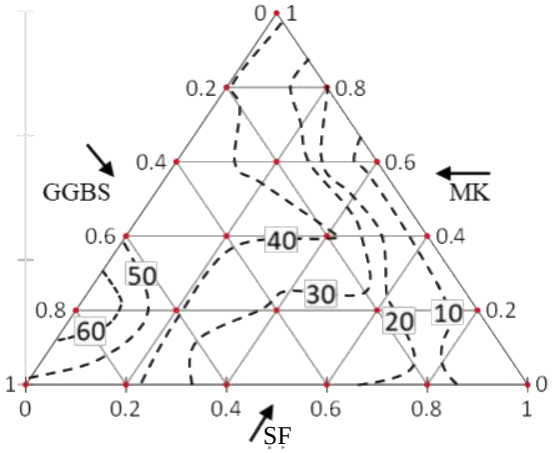
Material	Composition (% by mass)				Embodied carbon ($kgCO_2/kg$)
	SiO_2	Al_2O_3	CaO	Fe_2O_3	
OPC$^+$	20	4.6	64.6	3.8	0.73 [6]
MK	55	40	0.3	1.4	0.33[6]
GGBS	36.5	10.4	42.4	0	0.083[6]
SF	96	0.8	0.5	0.8	0.064^{++}
FA	57	24	3.9	6	0.008[6]
IS	27	3.2	1.8	46	0.057^{++}

$^+$ Included for comparative purposes only
$^{++}$ Provided by Elkem and Aurubis

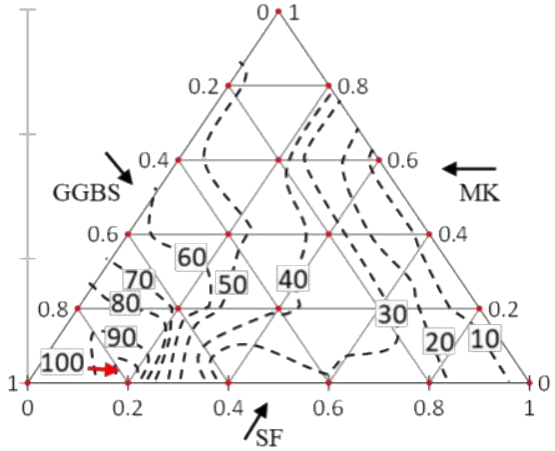
(a)

Material quantities (kg/m^3)				L/S ratio	Paste to sand ratio
Binder	Activator	Water	Sand		
540	455	135	1340	0.51	0.84

(b) 7-Day compressive strength, N/mm^2 (L/S ratio = 0.51)



(c) 28-Day compressive strength, N/mm^2 (L/S ratio = 0.51)



(d) Embodied carbon emissions (kgCO$_2$/kg)

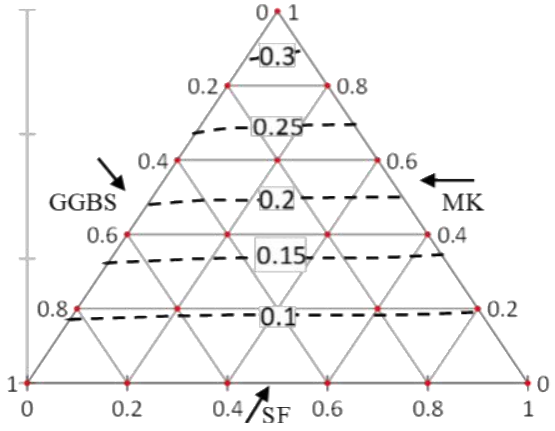


Figure 1: (a) Phase I mix design; (b) and (c) 7 and 28-day compressive strength results, and (d) embodied CO_2 contents, for unary, binary and ternary binder combinations considered

Also illustrated in Figure 1 is the embodied CO_2 content of each MK/GGBS/SF binder combination, with values generally reducing with corresponding reductions of MK reflecting the fact that it is commercially mined and calcined, as opposed to a by-product from other industrial activities. Of particular significance from the plots presented in Figure 1 is the fact that, for the geopolymer mortars considered, the improving levels of compressive strength corresponded with reducing levels of environmental impact (in terms of embodied CO_2). This is contrary to trends typical of conventional PC-based concrete mixes. In terms of mortar flow, results varied significantly and at almost all increments of MK replacement were in excess of 250 mm and, therefore, too high for accurate measurement and comparison. While this high range was clearly influenced by the L/S ratio of the base mix used (0.51), the ability of industrial waste materials to increase flow is a positive finding in terms of industrial-scale cladding panel production using highly-flowable, high-strength, low-impact geopolymer materials.

4 PHASE II – INFLUENCE OF MIX PARAMETERS

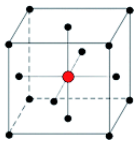
4.1 Mix designs

While Phase I clearly identified the influence of binder composition on geopolymer mortar performance, this was established for one mix design only, with other important and inter-relating key mix parameters not considered. As such, three high-performing binder powder blends were selected for further investigation in Phase II. This included: 100% MK base mix; 80%GGBS/20%MK and 80% GGBS/20% SF. The latter two blends were chosen as they achieved the highest strength at 7 and 28 days respectively from Phase I. As shown in Table 2, a face centred central composite mix design approach was used to consider three mortar component variables (binder powder, activator and free water content) across three levels (-1, 0, +1) for each selected binder blend.

In Phase 1, the L/S solid of 0.51 used provided a wide range of flow across the different binder types with pure MK geopolymers exhibiting significantly lower values than hybrid or GGBS-based blends. This is due to the fact that the MK geopolymers require higher L/S and A/B ratios than fly ash or slag geopolymers for full dissolution, monomer transport and reorganisation to take place. This trend was addressed in Phase II by lowering the binder mass and increasing water mass for the 100%MK mixes in order to maximise the potential of forming homogenous geopolymers. Ranges of binder powder, activator and free water content considered for the MK mixes were 490-590, 400-500 and 100-160 kg/m^3 respectively, while for the GGBS/MK and GGBS/SF mixes these were 550-650, 400-500 and 70-130 kg/m^3. In this way, the intention was to further investigate the influence of key relationships presented in the literature [6-9,12] as significant for geopolymers, such as S/A, L/S and A/B. For instance, via the variables and ranges considered as part of the central composite design, values of A/B and L/S ranged from 0.76-1.02 and 0.46-0.57 respectively for the 15 MK mixes. Corresponding ratio ranges for the GGBS/MK and GGBS/SF mixes were 0.62-0.91, 0.35-0.52.

Table 2. Experimental design, 7-day compressive strength results and model prediction errors

Mix	Central composite design variables			7-day compressive strength (N/mm^2)						Modeling errors (%)		
				Measured			Predicted					
	A	B	C	1$^+$	2$^+$	3$^+$	1$^+$	2$^+$	3$^+$	1$^+$	2$^+$	3$^+$
1	-1	-1	-1	42.5	75	48	42	73.5	50	1.1	1.6	4.5
2	-1	1	-1	42	69	53.5	42.5	70	53.5	0.1	1.5	0.2
3	1	1	-1	58	86	60	57.5	86.5	67.5	1	0.5	7.7
4	1	-1	-1	-	82.5	68.5	-	82.5	61.5	-	0.4	10.1
5	-1	1	1	38	67.5	38.5	34.5	66.5	42	3.4	1.2	10.4
6	1	1	1	39	79	51	39	79.5	53.5	1.6	0.4	4.9
7	-1	-1	1	33	72	34.5	33.5	73.5	36	1.3	2.4	4.7
8	1	-1	1	-	79.5	44.5	-	77	47.5	-	3.5	6.1
9	0	0	0	42	77.5	55.5	42	78.5	51.5	0.7	1.2	7.4
10	0	1	0	41.5	73.5	54.5	42.5	76	53.5	3.3	3.3	2.2
11	0	-1	0	-	71	52.5	-	76	49	-	7.1	7.4
12	-1	0	0	35	75	50.5	38	73.5	45.5	8.7	1.8	9.6
13	1	0	0	46.5	82.5	57	48	83.5	57	3.9	1.4	0.1
14	0	0	-1	51	85	52.5	49	80	57.5	4.3	5.4	9.5
15	0	0	1	39	79	47	35.5	76.5	45	8.1	3.4	3.6
									Average:	3.1	2.3	5.8



100% MK

	-1	0	1
A. Binder	490	540	590
B. Activator	400	450	500
C. Free water	100	130	160

80% GGBS / 20%MK
80% GGBS / 20% SF

	-1	0	1
A. Binder	550	600	650
B. Activator	400	450	500
C. Free water	70	100	130

$^+$1: 100% MK; 2: 80% GGBS/20% MK; 3: 80%GGBS/20% SF

4.2 Phase II compressive strength results

The 7-day compressive strength results achieved by the geopolymer mortar mixes considered as part of Phase II are presented in Table 2. As expected, and reflecting the mix constituent ranges introduced as part of the experimental design, broad ranges of strength were recorded for each binder combination investigated. For the MK, GGBS/MK and GGBS/SF combinations, these were 33.0-58.0, 67.5-86.0 and 34.5-68.5 N/mm^2 respectively.

Of the 15 mix compositions considered for each binder blend, mix 4 was perhaps expected to produce the greatest compressive strength as it had the lowest L/S ratio, highest mass of binder powder and the lowest amount of activating solution and free water. This was provided, of course, that sufficient activating solids existed in the mix for full dissolution to occur without leaving unreacted binder to act as microdefects. Indeed for 100%MK mix 4 (as well as for mixes 8 and 11), this proved not to be the case, with the material failing to set and gain any appreciable strength.

Alternatively, all of the GGBS/MK and GGBS/SF mixes successfully broke down the binder powder and had sufficient liquidity for monomer transport and reorganisation, allowing homogeneous hardened geopolymer mortar to form in all 15 mix iterations irrespective of the lower ratio values considered. This suggests that the amount of activator solids required for geopolymers based on these industrial waste materials is significantly lower; as this is the most expensive portion of a geopolymer mixture from both economic and environmental standpoints the benefits of partially replacing the MK with these is obvious.

4.3 Relationships between singular mixture proportioning ratios and compressive strength

As illustrated in Figure 2, work progressed to explore if clear relationships existed between the strength results obtained and the aforementioned ratios reported as being significant for geopolymer mix design (i.e. S/A, L/S and A/B). Figure 2 plots these ratios against the 7-day compressive strength measured for all 15 mixes considered for the three binder combinations under investigation.

The S/A ratio of source materials used to create geopolymers dictates molecular- and nano-scale structures formed, and theoretically there should be a direct correlation between silica content and strength due to increasing stronger Si-O-Si bonds. With that said, owing to other impacting mixture proportioning parameters optimum levels of S/A reported by researchers vary [7,12]. In this study however, the influence of S/A ratio on 7-day strength was not as significant as previously reported, with R^2 values of 0.06, 0.28 and 0.07 noted for the MK, GGBS/MK and GGBS/SF mixes respectively (Figure 2(a)).

L/S in geopolymeric materials (calculated by dividing the mass of solid materials in the binder and activator by that of the liquid portion of the activator and free water) is reported to be analogous to the water/cement (W/C) ratio in PC mix designs in terms of its impact on properties such as flow and compressive strength. In PC-based materials, compressive strength is negatively proportional to W/C. Similar, albeit varying and diminished relationships were noted for the three geopolymer binder blends considered (Figure 2(b)) reflecting the probable influence of other key mix variables not present in PC concrete. The R^2 values noted in this case ranged from 0.41-0.72, indicating a more significant correlation between L/S and strength.

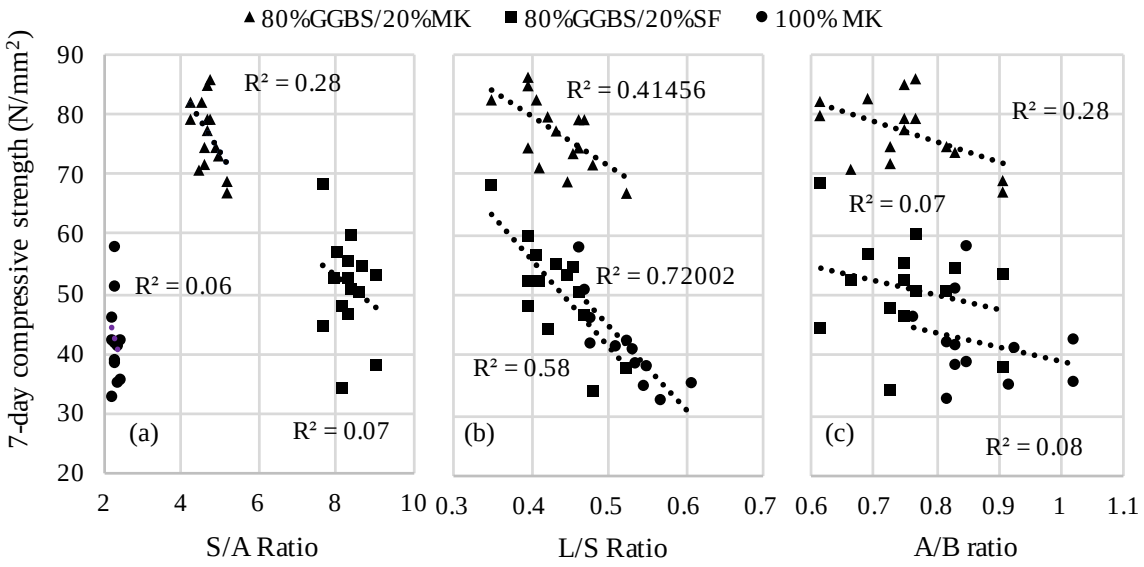


Figure 2. Geopolymer proportioning ratios vs 7-day compressive strength

Finally, A/B is reported to be one of the most important factors in the successful design of geopolymer mixes, enabling full dissolution and reorganisation of the mortar without defects from unreacted binder powder [8]. As mentioned previously, three of the 100%MK mortar mixes (4, 8 and 11) were unable to form geopolymer products owing to insufficient activator solids in the mix to break down the binder powder. With no release of silica and alumina monomers and chemically bound water, dry, sandy mortars lacking any cohesion or liquidity were formed. All MK mixes with an A/B ratio less than 0.75, or an activator solid to binder ratio of 0.34 reacted in this way. While vital to geopolymer formation, this ratio was found to be of little relevance in trying to predict strength, with low R^2 values ranging from 0.07 to 0.28 (Figure 2(c)).

In conclusion from Figure 2, it can be noted that when the all principal mix proportioning ratios are varied, none can be considered in isolation to accurately predict 7-day compressive strength. Out of the three considered, L/S emerged as the most significant, albeit with differing relationships apparent for the different binder compositions considered.

4.4 Correlation and regression analysis

As the ratios studied above in isolation were found to be poor predictors for compressive strength, work proceeded to ascertain if combinations of several mix parameters could be used synergistically with more success. Firstly, correlation analysis was carried out to determine which mix parameters were most closely linked to compressive strength. Parameters considered include the binder mass (B), activator solution mass (A), water mass (W), sand mass (S), activator solids mass (AS), total water content (TW), A/B ratio, free water to activating solution (FW/A) ratio, the free water to binder (FW/B) ratio, L/S ratio, and S/A ratio. Independent variables were then selected from this list to undergo regression analysis to produce a compressive strength predicting equation from the intercept and slope coefficients provided for geopolymers mortars at 7 days. Predicted compressive strength results for the 15 mixes for each binder blend were then compared to corresponding experimental results to quantify the success of this methodology. Comparing outputs such as: adjusted R^2; significance f; p values, allowed the most accurate prediction of strength possible from the data set. The equation used for compressive strength prediction is shown below in equation 1, with X1, X2, etc. representing the various mix parameters.

$$\textit{7-day strength} = \textit{Intercept}+(X1*\textit{slopeX1})+(X2*\textit{slopeX2})... \quad (1)$$

Table 3 shows the regression outputs and the predictions the models made for the 15 mix designs created for each of the three binder blends, and shows the average error in these as a method of analysing the success of the models. The mix parameters used for the equation relating to the MK mixes were binder mass (B), free water mass (W), FW/B and FW/A and in this way the model was capable of predicting 7-day compressive strength with an average error of 3.12%, an adjusted R^2 value of 0.91 and a statistical significance f of 1.9×10^{-4}.

Table 3. Regression model outputs and parameters

Binder	Mix parameters used by model						Model outputs		
	X1	X2	X3	X4	X5	X6	Adj. R^2	Sig, f	Average error %
100%MK	B	W	FW/A	FW/B	--	--	0.91	$1.9\text{x}10^{-4}$	3.1
80%GGBS/20%MK	B	FW/A	FW/B	TW	S/A	L/S	0.70	$9.5\text{x}10^{-3}$	2.3
80%GGBS/20%SF	B	FW/A	--	--	--	--	0.77	$5.8\text{x}10^{-5}$	5.9

As stated previously, the equation described in Equation 1 and Table 3 is only valid for mixes with an activator to binder ratio high enough to ensure geopolymerisation occurs, and should not be used at lower ratios. For the GGBS/MK mixes, independent variables used were binder mass, total water mass, FW/A, FW/B, S/A and L/S ratios, leading to an average modelling error of 2.34% and adjusted R^2 and significance, f, values of 0.7 and 9.5×10^{-3} respectively. For the GGBS/SF mixes, the singular modelling variable FW/A ratio produced the most accurate strength predictions (average error = 5.8%; adjusted R^2 = 0.77; significance, f = 5.8×10^{-5}).

Prediction of strength using multiple mix parameters for regression analysis has been relatively successful and shows promise for future development to improve the accuracy and significance of the model in the future by expanding the range of compositions analysed to provide increased data describing the relationships present.

5 CONCLUSIONS

The goal of the work reported in this paper was to develop performance-based mix design methodologies capable of reliably producing potassium silicate-activated geopolymer mortars – based on MK and a range of industrial by-products – with specified levels of strength, flow and/or embodied carbon content. In this way, the broader aim of the work is to drive forward the adoption of geopolymers as a lower impact replacement for conventional PC-based building components such as those manufactured using GRC.

For a given geopolymer mix design (i.e. constant binder, water and activator contents), the influence of binder composition on the resulting reactions and corresponding values of strength gain were found to be significant. High performance geopolymer mortars were developed, exhibiting high flow and 7 and 28-day strengths of up to 87 and 106 N/mm^2 respectively; the latter using a binder system comprising 100% by-product materials. Indeed, many of the highest performing mortars investigated had embodied CO_2 binder levels around 30% lower than corresponding PC-based mixes. This is deemed to be a major benefit of geopolymers, where a broad range of structural performance levels can be attained using various combinations of, ideally, locally available, low impact binder materials. Further improvements to performance are possible for geopolymer mixes via further adjustments to mixture proportioning parameters, such as mass of activating solids, as these are the costly component and levels are unnecessarily high in binders without MK. While the CO_2 savings reported in this paper are modest compared to some published in the literature, if geopolymer systems were used to replace all PC-based materials, the theoretical reduction in total global carbon emission would be approximately 2.1% [13,14].

This study found that the use of single proportioning ratios was insufficient for accurate strength prediction and that a wide range of mix parameters have bearing on performance. Of the single ratios studied, L/S ratio appeared to show the greatest correlation with strength, albeit that mixes with low L/S values did not consistently provide the greatest strength in the mix designs studied. Those with the lowest L/S ratios often also had the lowest A/B ratio, causing samples to be unable break down the binder powder sufficiently to form a homogenous geopolymer without unreacted materials acting as a microdefect. In MK based mortars, A/B ratios below 0.75 produced dry, sandy mortars with no cohesion due to the lack of activating solids present causing incomplete dissolution.

For the various sets of MK-, GGBS/MK- and GGBS/SF-based geopolymers mixes studied, a suite of regression models was developed to predict compressive strength at 7 days. With an average prediction error across the binder combinations considered all below 5.8%, the methods developed were relatively successful and indicate potential for future improvements Future research will attempt to improve predictions by widening the range of mix parameters and compositions studied to increase the data sets from which they are made, and seek to develop a single model suitable for accurately predicting the performance of any geopolymer binder type.

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