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Bai Kamara, K. B., Ganjian, E. & Khorami, M.

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THE USE OF WASTE GYPSUMS, RECLAIMED ASPHALT FILLER AND GGBS AS A FULL REPLACEMENT OF CEMENT IN ROAD BASE

Kande Bure Bai Kamara CEng MICE [1], Eshmaiel Ganjian PhD [2] and Morteza Khorami PhD [3]

[1]Research Student, School of Energy, Construction and Environment, Faculty of Engineering, Environment & Computing, Built & Natural Environment Research Centre, Coventry University, Coventry, CV1 2JH

[2]Professor of Civil Engineering Materials, Built & Natural Environment Research Centre, Faculty of Engineering, Environment & Computing, Coventry University, Coventry, CV1 2JH

[3]Lecturer in Civil Engineering, School of Energy, Construction and Environment, Faculty of Engineering, Environment & Computing, Built & Natural Environment Research Centre, Coventry University, CV1 2JH

Abstract

Laboratory experiments were used to determine the suitability of raw industrial by-products obtained within the United Kingdom that are being taken to landfill sites and develop a hydraulically bound cementitious material for applications in road (base), foundation and sub-grade in pavement construction. The by-products were predominantly sourced locally. Tests were carried out to determine the mechanical stability of the by-product binders and performance determined in the strength development by time. High pressure permeability tests were performed to determine the permeability of the materials and frost susceptibility tests were conducted to determine the freeze / thaw resistance of the materials. Compressive strength tests were conducted at 7, 14, 28, 90 and 180 days of age. Strength development on the hydraulic paste was slow during the early stages of hydration for mixtures containing 40 – 60% Ground Granulated Blast Furnace Slag (GGBS). After 28 days and up to 90 days when the ultimate strength of the hydraulic paste is achieved, strength increases with the

presence of GGBS of up to 60%. Ternary mixtures with the proportions of 20% PWG (Plasterboard Waste Gypsum), 20% RAF (Reclaimed Asphalt Filler) and 60% GGBS; and 10% V-B5G (Vitamin B5 Gypsum), 30% RAF and 60% GGBS attained the highest compressive strengths of 41MPa and 40MPa respectively at 90 days. One of the dominant factors that influenced the strength was the presence of calcium sulphate - $CaSO_4$ ($CaO + SO_3$) in the PWG / V-B5G materials, calcium silicate – $CaSiO_3$ ($CaO + SiO_2$) in the GGBS and the pozzolanic activity ($SiO_2 + Fe_2O_3 + Al_2O_3$) in the RAF. The results suggest most of the mixes in the groups are suitable for use as road (base) and foundation materials.

Keywords: gypsum; reclaimed asphalt filler; GGBS; hydraulically bound mixtures; pozzolans; pavement

1 Introduction

As the world is becoming aware of climate change and, as environmental policies and public concerns over the manufacture of cement and the extraction of our natural resources intensify, there is an increasing pressure and desperate need to use alternative materials to replace cement with by-product binders for pavement. Environmental concerns over our desires for using products that are manufactured from the continuous extraction of our natural resources has necessitated a growing use of secondary waste minerals such as coal-fired power station waste (PFA), municipal incinerator ash (IBA) and cement by-products (cement kiln dust – CKD and by-pass dust – BPD). Concerns about the depletion of our natural resources and the effect that

meeting the demand for quarried materials may have on the environment has increasingly focused attention on the possibility of finding alternatives to naturally occurring materials as mentioned by (Sherwood 1995). In a study carried out by (Pouya et al. 2007) on plasterboard waste gypsum, it was stated that statistics on waste plasterboard arising are scarce. It was estimated that some 300,000 tonnes (330,693 tons) of waste plasterboard are generated each year in the UK from new construction activities (largely as offcuts).

Limbachiya et al. (2015) investigated the effect of Pulverised Fuel Ash (PFA), By-pass Dust (BPD) and Ordinary Portland Cement (OPC) in a hydraulically bound mixture. The study revealed that the effect of BPD can be noticed when analysing Mix 1 (OPC-60% / PFA-40% / BPD – 0%), Mix 7 (OPC-60% / PFA-30% / BPD-10%) and Mix 9 (OPC-60% / PFA-35% / BPD-5%) in which OPC content remains unchanged and BPD was used to replace PFA by 0%, 5% and 10%. The 28 days compressive strength results showed that a 5% BPD replacement provided very close strength (31.9 MPa) to that of 0% BPD (34 MPa) and that of 10% BPD replacement (30 MPa).

Bai Kamara et al. (2020), studied the effect of Quarry Waste Dust (QWD) and Reclaimed Asphalt Filler (RAF) in a binary and ternary semi-dry paste containing Plasterboard Gypsum (PG) and Ground Granulated Blast Furnace Slag (GGBS). The findings revealed that the presence of GGBS is highly beneficial between 40 and 60% replacement for the mixtures of PG/RAF/GGBS and PG/QWD/GGBS. Mixes with the

proportions of PG20%/RAF20%/GGBS60 and PG10%/QWD30%/GGBS60% achieved compressive strengths of 41 MPa and 38 MPa at 90 days respectively. They concluded, after 28 days and up to 90 days when the maximum strength is gained, strength increases with the presence of GGBS of up to 60%.

Previous research studies have been carried out to explore the partial replacement of cement using different industrial by-product materials in the construction industry. Unlike previous studies investigated by Tan et al. (2017), Raghavendra et al. (2016), and Limbachiya et al.(2015), the aim of this research was to fully replace cement in concrete. Sharma and Khan (2016) performed an experiment on effect of different supplementary cementitious materials on mechanical and durability properties of concrete using the following pozzolanic materials: Fly Ash, Silica Fume, Metakaolin and Ground Granulated Blast Furnace Slag. The new replacement materials will reduce the impact the extraction of naturally occurring materials have on the environment.

The cement industry is one of the biggest waste producer industries in the world (Modarres et al. 2015). Approximately, 7% of the global carbon dioxide emissions come from the manufacture of cement. (Siddique and Rajor 2012; Li et al. 2013; Meyer 2009; Andrew 2018; Benhelal et al. 2013).The manufacture of Portland cement itself is ecologically harmful in that the production of one tonne of cement equates to approximately one tonne of carbon dioxide being expelled into the atmosphere

(Neville 2011). The construction industry is identified by the UK Green Building Council as one of the most emission-intensive industries, accounting for approximately 50% of greenhouse gas production in the UK (Dadhich et al. 2015). There is no doubt an increasing demand for the use of industrial by-products and recycled materials due to the stress on our natural resources and environmental systems.

Waste management and the use of industrial by-products are important aspects of government policies around the world aimed at re-enforcing current trends regarding the conservation of our natural resources. Environmental regulations such as H M Government (2010) meant that companies or organisations that are cutting costs to save money through environmentally unfriendly practices are issued with penalties. H M Government (1991) requires companies or organisations to have control of the waste they produce. A breach of this regulation is a criminal offence. Recent increase in the use of environmental controls meant that some of these wastes are recovered within the manufacturing process. Most of the wastes, however, are removed and disposed of to landfill sites in accordance with H M Government (2002). The increasing shortage of land available for waste disposal, the penalties introduced by local authorities in disposing of waste, and the increased manufacturing of by-products, it is inevitable that increased use of these industrial by-products be explored in pavement and other construction applications to help prevent depletion of virgin materials which is usually accompanied by environmental degradation and lead to economic problems.

The aim of the research was to 100% replace cement with secondary waste minerals. The research objectives were: i) to identify industrial by-products that are being taken to landfill sites in the UK and to investigate their use in highways engineering particularly in road base and foundation; ii) to conduct laboratory tests to examine by-products suitability for use; iii) to conduct mechanical performance tests and obtained a novel blend; and iv) to create sustainable concrete made of the novel blend and recycled concrete as aggregates. The novelty of the study is to close the knowledge gap in the use of gypsum wastes and quarry by-products. This paper looked at two of the three ternary binders referred to as 'Groups' that were considered to have potentials (Group 2 and 2A) in the research study. Two different types of factory gypsum wastes: Plasterboard Waste Gypsum (PWG) and Vitamin B-5 Gypsum (V-B5G) were activated by two pozzolanic materials: Reclaimed Asphalt Filler (RAF) and Ground Granulated Blast Furnace Slag (GGBS) in different proportions as binary and ternary semi-dry pastes. The use of these materials in pavements help reduce the environmental impact in the extraction of primary aggregates, reduce waste being sent to landfill sites and help stimulate the use of secondary waste minerals.

2 Raw Materials

2.1 Plasterboard Waste Gypsum and Vitamin B5 Gypsum

The PWG and V-B5G satisfied the requirements of EN, BS 197-1 (2011). The PWG was sourced from a local housing development site in Nuneaton, North Warwickshire, UK. The plasterboard waste arose from off-cuts and damaged boards. The V-B5G was obtained from DSM Nutritional Products UK Ltd, Scotland. This waste gypsum was produced by the pharmaceutical industry. The industrial waste is produced from the manufacture of vitamin B5 (Pantothenic acid) supplements. The mineralogical composition of the PWG showed the mixture of calcium sulfate dihydrate ($CaSO_4$-$2H_2O$, hemihydrate ($CaSO_4$-$0.5(H_2O)$) and anhydrite ($CaSO_4$). The V-B5G showed the presence of high levels of calcium sulfate dihydrate ($CaSO_4$-$2H_2O$) and low levels of calcium carbonate ($CaCO_3$). The X-RF results for the materials are presented in Tables 1 and 2.

2.2 Reclaimed Asphalt Filler

The RAF conform with the requirements set out in ASTM C311 (2013), whereby the activity index is over 75% of the controlled mix at 28 days. The material satisfied also the requirements of EN, BS 197-1 (2011) whereby it consist predominantly of reactive silicon dioxide (SiO_2) and aluminium oxide (Al_2O_3). The RAF was obtained from Tarmac and was sourced locally from their Mancetter Quarry in Atherstone, North Warwickshire, UK. The material is produced by their Via Nova Asphalt Plant

and conveyed to the plant reclaimed duct silo. The material is a natural calcined pozzolan and is formed during the processing of asphalt using sedimentary rocks. The mineralogical composition are plagioclase, calcite, quartz, mica, chlorite and amphibole. The peaks and valleys pattern of the X-RD analysis showed high levels of plagioclase and chlorite followed by moderate levels of calcite and quartz. The X-RF results of separate batch of the materials over a two years period are presented in Table 3.

2.3 Ground Granulated Blast Furnace Slag

The GGBS conform with the requirements set out in EN, BS 197-1 (2011) for use in blended cement. At least two-thirds of the total mass of slag must consist of the sum of CaO, MgO and SiO_2. Also, the ratio of the mass of CaO plus MgO to that of SiO_2 must exceed 1.0. The major oxides present in the GGBS used in this research study were calcium (CaO), aluminate (Al_2O_3) and silicate (SiO_2). These three oxides total over 85% of the oxide composition present in the sample. The sum of CaO, MgO and SiO_2 of the total mass of slag was more than two-thirds and the ratio of the mass of CaO plus MgO to that of SiO_2 exceeds 1.0. This did not only show that the material obtained from Hanson Hiedel-berg Cement Group was compliant with EN, BS 197-1 (2011) and suitable for use as blended cement, it also shows that the material had chemical composition that conforms to the criteria for common cements. The material was obtained from Coventry University materials' inventory stock. The X-

RF results of separate batch of the materials over a three years period are presented in Tables 4.

2.4 Physical properties

Analysis to determine the physical properties of the raw materials used in this paper was carried out using a Malvern Mastersize 2000 equipment and the outputs created by the Malvern Laser Granulometer with an accuracy of +/-1%. Fig 1 and 2 show the particle size distributions of Groups 2 and 2A with the blends PWG/RAF/GGBS and V-B5G/RAF/GGBS respectively. The particles for the two blends range approximately between 0.3 to 1500 microns and their 50^{th} (D50) and 90^{th} (D90) percentiles are shown in Table 5. Table 5 indicates the percentage of particle size in terms of quantity. For example, the 50^{th} percentile of GGBS shows that 50% of the sample is less than 17.3 microns and 90% of the sample is less than 49.27 microns.

AccuPyc 1330 Gas Helium Pycnometer was used to determine the specific gravity values of the materials used in the research. The specific gravity values for the PWG, V-B5G, RAF and GGBS were 2.31 g/cm^3, 2.35 g/cm^3, 2.71 g/cm^3 and 2.87 g/cm^3 respectively.

Grading analysis of the RAF obtained from Tarmac is shown in Table 6. The grading analysis was determined in accordance with EN, TS 933-10 (2009) and target

specification to EN, BS 13043 (2013) *Clause 4.0: Table 5*. The results of the analysis showed that the grading complies with the specification requirements for limestone filler.

3 Research Methodology

The methodology used in this research study is represented in Fig. 3.

3.1 Waste Mineral Suitability

Laboratory experiments to determine industrial by-products suitability were carried out using X-RD and X-RF tests. Materials identified to have pozzolanic and / or cementitious properties were further explored in the research study. Materials that have neither pozzolanic nor cementitious properties can be used as coarse or fine aggregates if they are considered to have satisfactory mechanical properties that is resistant to crushing.

3.2 Mix Design

Characterisation of the mixtures for their suitability in road base construction came about in reviewing literatures and studying the chemical oxides (X-RF) present in each sample. This provided an indication of the grouping arrangements adopted in the research study and the effective use of constraints used on the statistical software package Minitab 18 for the Design of Experiments. For this research study, eleven

combinations of ternary binders referred to as 'Groups' were designed. There were thirteen mixes in each group and a total of 143 mixes derived from the eleven ternary combinations. The first set (Groups 1 to 4) referred to as sulphate activated pozzolan focused on the effects of Medium Hydrated Tailing (MHT), RAF, By-pass Dust from Cemex (BPD-C) and By-pass Dust from Hanson (BPD-H) on mixes containing PG and GGBS. The second set (Groups 5 to 8) referred to as alkali activated slag focused on the effects of MHT, RAF, BPD-C and BPD-H on mixes containing V-B5G and Steel Slag Dust (SSD). The third set (Groups 9 and 9A) concentrated on the effects of PG and V-B5G on mixes containing Quarry Waste Dust (QWD) and GGBS. Group 2A was added to compare the mechanical performance of Group 2 using the same mix design in mixes containing RAF and GGBS with two gypsums.

Approximately, over 1000 hydraulic paste cubes were tested for compressive strengths in comparing the results of the first set (Groups 1 to 4) and second set (Groups 5 to 8). The results revealed that the first set containing GGBS showed extremely slow strength gain when compared to the second set containing SSD. It was concluded from the statistical analysis that the constituents of the first set had a significant effect on the chemical compositions and reactions of the materials compared to the second set.

A statistical mixture design programme – Minitab 18, was used for the Design of Experiment (DoE) for Groups 2 and 2A. There are four types of mixture designs in Minitab: i) Simplex Centroid; ii) Simplex Lattice – 1^{st} Degree; iii) Simplex Lattice – 2^{nd} Degree; and iv) Extreme Vertices – 2^{nd} Degree. All four can be designed either

augmented or unaugmented. Augmented design is whereby design points are included in the interior of the design space, unaugmented on the other hand do not with the exception of Simplex Centroid design. The 1st Degree only allows the design to look for linear effect. The 2nd Degree on the other hand allows the design to find quadratics.

Even though Simplex Lattice – 2nd Degree and Extreme Vertices – 2nd Degree permit fitting up to a quadratic model, Extreme Vertices design is appropriate for the relationships the authors were looking for. Extreme Vertices Design (EVD) method was chosen for the experimental study because it sets the boundaries of the components in each group for the designs. The mix designs have the following vertices constraints:

$$0 \leq X_i \leq 1 \qquad [\text{Eq. } 1]$$

Where X_i represents the proportion of component i. EVD was chosen for this experimental study because of the provisions of the upper and lower bounds used in the ternary combinations, it allows precision in the design, provides better estimates, maximises variance and generate plots for all three components in the model. This allows for good coverage and the design points adequately covers the design space. The unique design space on EVD allow points to be placed not only at the centroid, midpoints and axial points, but also at the extreme vertices of the region. EVD satisfies the formulation in having constraints that ensure correct specifications. Also, it satisfies the formulation that optimizes the response for certain reaction; and more importantly it satisfies the formulation on how the different samples used in the ternary groups affect a response.

(Limbachiya et al. 2015) mentioned how the programme generated the vertices of constrained design space (Lower Limit < Material < Upper Limit) and then calculated the centroid point up to the specified degree using Piepel's CONAEV algorithm. Reviewing literatures (Ganjian, Essie et al. 2008) and (Singh et al. 2008) provided an indication of effective replacement levels of the materials. As the aim of this study was to compare the use of the two waste gypsums (PWG and V-B5G), the upper and lower limit boundaries used for PWG/V-B5G, RAF and GGBS were 20%-80%-60% and 0%-20%-0% by weight respectively. In other words, the constraints for both Groups 2 and 2A were the same. Due to the high volume of materials used in some of the components, the combination of binary and ternary mixes was produced in the designs.

Table 7 and [Fig. 4 - 5] show the mix proportions required in each mix and the simplex design plots.

3.3 Fabrication

The research conducted in this study was to develop a hydraulically bound cementitious material for applications in road(base), foundation and sub-grade in pavement construction. In accordance with EN, BS 13286 - 40 (2003), hydraulically bound material is a mixture that hardens by hydraulic and / or pozzolanic, sulphatic and / or carbonic reaction, which usually has a workability to suit compaction by rolling and which is generally used in bases, sub-bases and capping layers. The pastes analysed in this study were also semi-dry form and the cubes made were solely compacted to simulate real life applications in construction sites.

The mixing of the materials took approximately 6 minutes. After the materials were weighed, water was intermittently slowly added at 1 and 4 minutes of mixing. The pastes were cast in 50 mm cube moulds. The sample was pre-compacted with a square tamping rod (25 mm x 25 mm x 300 mm) in three layers of 90g per cube at 25 strokes per layer. The sample was further compacted using hydraulic machine. The hydraulic pressure was set at 90 KN for a gang of three 50 mm moulds prepared. A constant rate of loading at 5 KN/s was applied to the samples. The hydraulic compaction of 30 KN per cube pressure used in this study is in line with the requirements set out in SHW Series 800 (2020). This is whereby either a vibrating or pneumatic-tyres roller with a wheel loading of not less than 30 KN followed by at least eight passes is used to compact hydraulically bound mixtures.

The pressure of 90KN was applied to the samples for 2 minutes before it was released. The 50 mm semi-dry specimens were easy to handle and therefore casting was carried out immediately after the specimens had been hydraulically pressed without a shock or vibration. The specimens were later cured in transparent sealed propagators [Fig 6] at 20°C +/- 2°C with a relative humidity of between 95 – 98%.

4 Testing Methodology

The laboratory tests selected were tailored for the applications of the research materials. The main application for the research material is for it to be used for road base. The mechanical performance criteria for hydraulically bound mixtures or cement bound granular mixtures is compressive strength.

These research materials are exposed to the elements regard to adverse weather conditions i.e. rain, snow or possible contamination from road incidents (chemical spillage). The high-pressure permeability test was performed to determine the permeability of the by-product binder. The frost susceptibility tests on the other hand were conducted to determine the freeze thaw resistance of the materials.

4.1 Compressive Strength

Three to five samples were tested for compressive strength on the top two mixes (Group 2 Mix 1 - PWG20/RAF20/GGBS60 and Group 2A Mix 13 – V-B5G10/RAF30/GGBS60). Three replicates of the mixes were checked for consistency over a three years period. The mean and standard deviation of the samples tested are shown in Table 8. The standard errors for the 90 days compressive strength tests are shown in Figure 11. The standard deviation for the top two mixes is different. The data set for Mix 1 of Group 2 showed the most variability. Mix 13 of Group 2A on the other hand showed tighter results.

The contour plots for Group 2 and 2A at 7 and 28 days using 15% and 20% liquid/solid ratio are shown in Fig. 7, 8, 9 and 10. The liquid / solid ratios of 15 and 20% for Group 2 and 2A respectively were used because of their mechanical performance at 28 days. Mix 4, with the proportions of 5%PWG, 47.5%RAF and 47.5%GGBS attained highest compressive strength at 27.02MPa for Group 2. Group

2A on the other hand attained the highest strength at 29.45MPa with the proportions of 15%V-B5G, 37.5%RAF and 47.5%GGBS on Mix 6. The highest strength on both groups provided very close results even though their mix proportions are different. Comparison of 7 and 28 days test results revealed that all the mixes on both groups showed a significant increase in strength development as the specimen ages except for Mix 7, a binary mixture with proportions 20%(PWG/V-B5G) and 80% (RAF). It was noted on both groups there was an increase of approximately 1,500% in the average compressive strength for 7 to 28 days for the top six mixes exceeding 20MPa.

Bonferroni method (Bland and Altman 1995) -stated that a Probability Value (p-value) for any term of a mixture design should be no greater than 0.05 to ensure 95% of the associated response to the related term (s) to be true. In other words, the 95% confidence level and the p-value threshold of <0.05 helped to decide whether to reject the null hypothesis or not. Eq. 2 was used to calculate the predicted values of Groups 2 and 2A at 28 days compressive strength. Predictors with low p-values are likely to be significant to the model as they are directly related to changes in the response variables.

$$Y_{ijk} = \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{23} X_2 X_3 \qquad \text{[Eq. 2]}$$

Where Y_{ijk} represent the response for the i^{th} proportion of the component 1 (X_1), the j^{th} proportion of component 2 (X_2) and the k^{th} proportion of component 3 (X_3). Therefore, using Groups 2 and 2A with the components PWG/V-B5G, RAF and GGBS:

$Y_{PWG/V-B5G,RAF,GGBS}$ represent the response when (X_1) = PWG/V-B5G, (X_2) = RAF and (X_3) = GGBS. X_1, X_2 and X_3 are the variables and represent the response in each mix design and β_1, β_2 and β_3 are the coefficient of the terms which are constant. The equations [Eq. 3-4] that predicted the compressive strength (CS) and produced the contour plots [Fig. 9-10] for Groups 2 and 2A were:

CS = -821.884(PWG)-11.9009(RAF)-11.2269(GGBS)+1059.19

*(PWG*RAF)+1203.40(PWG*GGBS)+79.2780(RAF*GGBS)* [Eq. 3]

CS = -753.639(V-B5G)-17.2196(RAF)-251896(GGBS)+1006.09

*(V-B5G*RAF)+1175.33(V-B5G*GGBS)+117.031(RAF*GGBS)* [Eq. 4]

Equation 1 has been used to further explain the variables in Equations 3 and 4. Both Eq. 3 and Eq. 4 are made up of 3 components each, where X_i represents the proportion of the component i, i equal 1, 2 and 3. The constraints are:

$$0 \leq X_1 \leq 1$$

$$0 \leq X_2 \leq 1$$

$$0 \leq X_3 \leq 1$$

Therefore:

$$X_1 + X_2 + X_3 = 1 \qquad [\text{Eq. } 5]$$

In Eq. 3 $X_1 = 0.05$ and $X_2 = 0.475$, X_3 must equal 0.475. Similarly, in Eq. 4 $X_1 = 0.15$ and $X_2 = 0.375$, X_3 must equal 0.475. The variables in Eq. 3 are $PWG = 0.05$, $RAF = 0.475$ and $GGBS = 0.475$. Similarly, the variables for Eq. 4 are $V - B5G = 0.15$, $RAF = 0.375$ and $GGBS = 0.475$.

4.2 Refining Group 2 and 2A to Obtain Optimum Blends

The effects of different liquid / solid ratios on the long-term compressive strengths of the top four mixes in each group were evaluated. In Group 2, Mix 1 (PWG20/RAF20/GGBS60), Mix 4 (PWG5/RAF47.5/GGBS47.5), Mix 6 (PWG15/RAF37.5/GGBS47.5) and Mix 13 (PWG10//RAF30/GGBS60) at 7, 14, 28 and 90 days using liquid / solid ratios of 13, 15 and 17% were evaluated. Even though Mix 4 (PWG5/RAF47.5/GGBS47.5) at 15% liquid / solid ratio achieved the highest compressive strength of 27MPa at 28 days, at 90 days Mix 1 (PWG20/RAF20/GGBS60) achieved a strength of 41MPa with liquid / solid ratio of 17%.

The top four mixes in Group 2A have the same mix designs as those of Group 2 but with different materials. Specimens for 17, 19 and 21% liquid / solid ratios at 28 and 90 days for Group 2A were evaluated. Even though Mix 6 (V-B5G15/RAF37.5/GGBS47.5) at 20% liquid / solid ratio achieved the highest compressive strength of 29MPa at 28 days, at 90 days Mix 13 (V-B5G10/RAF30/GGBS60) achieved a strength of 40MPa with liquid / solid ratio of 19%.

4.2.1 Frost Susceptibility - Resistance to Freezing and Thawing

Laboratory tests to determine resistance to freezing and thawing of the hydraulically bound mixtures were carried out in accordance with CEN/TS 13286 (2014). The freeze-thaw cycle of the top two mixes (Mix 1 of Group 2 and Mix 13 of Group 2A) commenced at a stage which was a significant proportion of their ultimate strength i.e. 90 days of age. Longer age curing of 91 days was required for the two mixes as they were slow setting and hardening mixtures. The optimum liquid / solid ratio for Group 2 and 2A were 17 and 19% respectively.

Two sets of six specimens were used in the experiment for each mix. On completion of curing for the first set 'A' (R_A), the specimens were placed in an environmental chamber and subjected to ten freeze-thaw cycles. Each freeze-thaw cycle lasted 24 hours with a starting temperature of 20°C, to -1°C in two and half hours, maintained cool at -1°C for four hours, reduced to -17$_{1/2}$°C in three hours, maintained cool at -17$_{1/2}$°C for six hours and warm again to 20°C in eight and half hours. The relative humidity of the cabinet was set at 100%. After completion of the tenth freeze-thaw cycle, specimens 'A' was returned for 1 day curing to ensure complete thawing. The compressive strength of set 'A' and the control set 'B' (R_B) were measured and the mean value of the strength for each set compared. Fig. 12 shows the freeze thaw cycle diagram.

4.2.2 High Pressure Permeability

This experimental work was similar to the one adopted by Ganjian et al. (2004), and (2006), where a modified Hoek cell was used to measure the permeability of the hydraulic paste in preventing the flow of fluid around the sides of the sample. Figure 13 shows the layout of the test. Methods of compaction of the samples was similar to that used for the compressive strength tests on the hydraulic cube pastes to achieve better density. The liquid / solid ratios were based on the ultimate strengths. A gang of three 50mm cylindrical mould was used to produce cylindrical shaped specimens with 55mm diameter and at least 50mm in length. After the specimen had been installed in the triaxial cell and bolts on the external framework tightened, an oil pressure of up to 40 bars was applied. Flow around the specimen was prevented by maintaining the high pressure of hydraulic oil inside the flexible membrane. The pressure was maintained with the ramp pump by adjusting the weight on the balance unit. The water pressure valve was set at 30 bars, 10 atmospheres lower than the oil pressure. The permeability of the specimen was based on measuring the time it took to collect 25 ml of water passing through the Hoek cell.

5 Analysis of Experimental Results

5.1 The Effect of PWG and V-B5G in Mixes Containing RAF and GGBS

Group 2 and 2A had mixes containing PWG/RAF/GGBS and V-B5G/RAF/GGBS respectively. Selected Mixes 1, 4, 6 and 13 with the same proportions of the different materials in the two Groups (2 and 2A) have been used to evaluate the effect of PWG

and V-B5G in mixes containing RAF and GGBS at 7, 14, 28 and 90 days. The effect of PWG and V-B5G have been based on the optimum liquid / solid ratios at 90 days to achieve the strongest mechanical performance of the mixes. The optimum ratio of 17% was used for the PWG/RAF/GGBS combination and 19% for the V-B5G/RAF/GGBS. Figure 14 shows comparison of PWG and V-B5G in the selected mixes containing RAF and GGBS.

Analysis of the results in general showed that there was not a great effect of PWG and V-B5G in mixes containing RAF and GGBS. The selected mixes on the two groups performed similarly in their short and long-term strength development. For the medium-term (14 - 28 days) strength, all the mixes performed similarly except Mix 6 with the average strength difference of approximately 6MPa. At 7 days, the mixes on Group 2A had high compressive strength results when compared to Group 2, except for Mix 4 which was marginal. The 90 days strength on the other hand showed that Mixes 4, 6 and 13 had similar strength development except for Mix 1 where the strength difference was approximately 8MPa.

5.2 Strength Development of Hydraulic Pastes

Compressive strength results of selected mixes on Group 2 and 2A for 7, 14, 28, 90 and 180 days have been presented in Table 9. Laboratory experiments or investigations were not carried out on the specimens in this research study to evaluate hydration of the chosen groups discussed in this paper. It was assumed that hydration in the raw

materials determined during the XRD experiments remained unchanged, therefore this information was used as a baseline to evaluate the hydration of the specimens derived from dry hydraulic cement pastes. Looking at the trends of this research study's findings, one can hypothesise that the short and long-term strength development of the hydraulic paste containing GGBS are in agreements with reports carried out by Roy (1982), Hogan and Meusel (1981). Their report found that the strength development was slow during the early stages of hydration for concrete containing 40 – 60% GGBS as cement replacement. In a separate study carried out by Khatib and Hibbert (2005), they reported similar findings that the presence of GGBS is highly beneficial between 40 and 60% replacement. They acknowledged a reduced strength during the first 28 days. After 28 days and up to 90 days, they concluded the strength increases with the presence of GGBS of up to 60%.

Generally, industrial by-products including those used in this research study are not conventional materials and therefore not expected to behave exactly like traditional materials. Industrial by-products of this sort are highly acceptable for their ultimate strength (80- 90%) to be achieved at 90 days of curing. Fig. 15 shows the percentage of strength development of the top two mixes in both groups at ultimate strength (Group 2 Mix 1 PWG20/RAF20/GGBS60 and Group 2A Mix 13 V-B5G10/RAF30/GGBS60).

Given the novelty of this research study and the introduction of new industrial by-products to fully replace cement, a direct comparison of the strength gained as the specimen ages on the preferred mixtures cannot be made to similar materials as most research concentrate on partial cement replacement. However, a review carried out by

Suresh and Nagaraju (2015) concluded that a 50% by weight of GGBS concrete will typically achieve approximately 45 to 55% of its 28 days strength at seven days, with a gain of about 20% from 28 to 90 days. Unlike Suresh and Nagaraju (2015) findings, the top two mixes in this research had 60% by weight of GGBS. Approximately, 50 to 65% of strength was gained at 28 days and over 90% of strength achieved at 90 days. Strength gained for Mix 1 of Group 2 was about 40% and that of Mix 13 of Group 2A was about 25% from 28 to 90 days.

Latif et al. (2014) carried out a laboratory study on development of compressive strength for concrete with different curing durations. It was concluded from their findings that the age of concrete plays an important role for compressive strength. As the age increases, so is the compressive strength of concrete also increases. The rate of increase of average compressive strength for cubes due to curing from 7 days to 14 days, 14 days to 21 days, 21 days to 28 days, 28 days to 60 days and 60 days to 90 days was in ranges of 7%, 18%, 31%, 46%, 62% and 62% respectively.

5.3 Resistance to Freezing and Thawing Results

After the specimens were examined having gone through the tenth freeze-thaw cycle, they showed no damage of scaling and cracking. The lack of scaling and cracking may have been the presence of smaller particle size in the components. It was assumed that the smaller particle size present improved the packing. This improved

packing may have provided better matrix, served as fillers and increased compaction of the components to eliminate air voids.

Review of research work by Mangulkar and Jamkar (2013) showed particle packing models are based on the concept that voids between larger particles would be filled by smaller particles thereby reducing the volume of voids or increasing the packing density. In other words, one of the major effects of particle packing theory is its minimization of voids or maximization of the packing density of the components.

Sarkkinen et al. (2019) applied the same experimental standard CEN/TS 13286 (2014) used in this research study to compare alkali activated composite and ordinary portland cement as stabilization agents. Their results indicate that there was no damage in the specimens due to the freeze-thaw cycles because the compressive strength and density increased after the 10 cycles. The increase in strength and density was likely caused by an increase in binder products formed due to extra moisture and high temperatures during the warmer cycle period.

The results of the two mixes (Table 10) showed a loss in strength below the strength for the control specimens. The two mixes had freeze-thaw retained strength ratios of over 90%. Both mixes had retained ratios of approximately 93%. There was a weight reduction after the freeze – thaw exercise. Hamoush et al. (2011) concluded in their research work on freezing and thawing durability of very high strength concrete that a good freeze-thaw resistance should be a durability factor greater than 85% to avoid freeze/thaw damage.

5.4 High Pressure Permeability Results

Claisse, et al. (2003) stated that the surface skin of concrete is the first line of defence against the ingress of aggressive agents such as chlorides, sulphates and carbon dioxide. For this reason, there is an increasing awareness of its importance for durability of concrete. Results of the flow test at 90 days showed that Mix 1 of Group 2 had lower coefficient of permeability of 2.40E-09 compared to Mix 13 of Group 2A (1.336E-08). However, if the permeability of the materials used in this research study were to be compared to high strength concrete, the permeability of the research materials would have been higher. When the results of 28 and 90 days test were compared, the trends revealed similar outcomes. There was a decrease in the flow of liquid as the specimens age. The results of the 28 and 90 days tests are shown in Fig. 16. The high permeability for the two mixes may have been the absence of aggregates in the constituents. Ganjian, et al. 2006) stated the properties of an ideal barrier system should have a low permeability of not less than 10^{-9} m/s in the UK.

5.5——Factors Influencing Compressive Strengths

There are two key factors that influenced the strength of the optimum mixes at 90 days on Group 2 Mix 1 (PWG20/RAF20/GGBS60) and Group 2A Mix 13 (V-B5G10/RAF30/GGBS60): i) the constituent of the mix itself regards to the proportions of the different materials present; and ii) the percentage of the liquid / solid ratios used in the mixture

It can be said that the dominant factor of calcium sulphate - $CaSO_4$ ($CaO + SO_3$) in the PWG / V-B5G materials, calcium silicate – $CaSiO_3$ ($CaO + SiO_2$) in the GGBS and the pozzolanic activity ($SiO_2 + Fe_2O_3 + Al_2O_3$) present in the RAF heavily influenced the performance of the cement paste at both early and late age strengths.

Claisse and Ganjian (2006) distinguished the three main compounds of calcium sulphate and are summarised as follows: i) anhydrite ($CaSO_4$) – compound with no water molecules; ii) hemihydrate ($CaSO_4 – 0.5H_20$) – compound with partial water; and iii) dihydrate ($CaSO_4 – 2H_20$) – compound fully hydrated with water and correctly described as gypsum.

To determine the chemical oxides or chemical compositions of a given sample and to show the suitability of materials to be combined to give the required possible hydration reaction, X-ray fluorescence (X-RF) was carried out. The peaks and valleys pattern obtained from the X-ray diffraction (XRD) analysis of the PWG and V-B5G showed the presence of high levels of gypsum ($CaSO_4 – 2H_20$) and a moderate level of basanite ($CaSO_4 – 0.5H_20$) also referred to hemihydrate only for the PWG. One of the two main industrial uses of these compound mentioned by Claisse and Ganjian (2006) is in cement. Cement producers use a blend of gypsum and anhydrite as a set controller.

The PWG, V-B5G, RAF and GGBS materials used in this research study met the requirements set out in (EN, BS 2011).

The hydration process is another key element that determines the short and long-term performance of the paste mixtures in terms of strength development. It was very noticeable at 28 days strength using 13, 15 and 17% liquid / solid ratios that 100% hydration was achieved with Mix 4 (PWG5/RAF47.5/GGBS47.5) of Group 2 at 15%. Unlike Group 2A, 100% hydration was achieved at 19% with Mix 13 (V-B5G10/RAF30/GGBS60) using 15, 17, 19 and 21% ratios. There was sharp decrease in strength at 13% compared to a moderate decrease at 17% for Mix 4 of Group 2 and a moderate decrease at 15 and 21% for Mix 13 of Group 2A. There is no doubt that the 15 and 19% water contents for both mixes achieved full compaction and optimum mechanical performance at 28 days. Fig. 17 illustrates the highest compressive strength on Groups 2 and 2A at 28 days with different water contents.

It can also be said that the low strength results with mixtures of 13 and 15% ratio for Mixes 4 and 13 respectively was the effect on compaction and not enough water available to carry on during curing time. The not so good results for the 17 and 21% ratio for Mixes 4 and 13 respectively may have been as the results of less workability in excess water, that evaporates and leaves pores thereby reducing the strength. It clearly shows that the mixtures of 17 and 21% ratio had more than required water that was needed.

Another factor that may have influenced the mechanical performance of Groups 2 and 2A was the physical properties of the materials in the components. Analysis to determine the particle size distribution of the materials used in the group with the highest compressive strength of approximately 30 MPa contain more particles in the

range 54 – 212 microns. Ganjian, et al. (2007) report that pozzolanic materials with a finer particle size results in faster hydration and reduced setting time of the binder. This is due to the higher surface area and electric charges induced on the surface of the particles during the grinding process.

5.6 Applications of Research Materials

Mixes with strengths of between 8MPa to 32MPa can be used in foundation and road(base) in pavement engineering. Mixes with low strengths can be used to protect either existing or new Statutory Undertakers Services apparatus as they could be easily excavated with less energy and reinstated. Other applications include the stabilisation of earthworks and as a binder to replace cement in concrete.

6 Conclusions

Based on the findings of the investigation, the following conclusions are dawn:

- It is feasible to fully replace cement with industrial by-products. The selected mixes of PWG, V-B5G, RAF and GGBS have potentials to be used not only in highways engineering, but in wider applications in the construction industry;
- the mixes with the proportions of 20%PWG, 20%RAF and 60%GGBS in Group 2; and 10%V-B5G, 30%RAF and 60%GGBS in Group 2A were identified as the novel mixes, achieving compressive strengths of 41 and 40MPa respectively at 90 days curing;

- refining the preferred mixes to establish the correct liquid / solid ratios was one of the key factors in determining the mixes' optimum performance;
- mixes incorporating V-B5G require more water to achieve similar workability;
- when the mixture is used as base, it can be used either on its own as 'Hydraulic Bound Mixture – HBM' or with sand and gravel as 'Cement Bound Granular Mixture - CBGM.' The HBM mixture design is favourable in terms of the carbon footprint to CBGM; and
- the integration of economic regard to the use of no cement, environmental in respect to the use of virgin materials, and social about reducing noise and air pollution due to the close proximity of the by-product source of production and centre of use, were the three elements of sustainable development fully covered

Data Availability

Some or all data, models, or code that support the findings of this study are available from the corresponding author upon reasonable request.

The items that will be made available from the corresponding author upon reasonable request are as follows:

i. Input data on Minitab 18;
ii. Output data on Minitab 18;
iii. Laboratory compressive strength results at 7, 14, 28, 90 and 180 days of the different mixes (Group 2 and Group 2A) discussed in this manuscript;

iv. Laboratory high pressure permeability results of Group 2 – Mix 1 and Group 2A – Mix 13 at 28 and 90 days tests;

v. Laboratory freeze thaw results of Group 2 – Mix 1 and Group 2A – Mix 13;

vi. Input and output data in using the Malvern Mastersize 2000 to determine the particle size distributions of PWG. V-B5G, RAF and GGBS;

vii. Evidence of test results carried out by other institutions (Leicester University and Warwick University) to determine the phases or component combinations of the mineralogical properties of the samples (materials) used in the experimental study;

viii. Evidence of test results carried out by other institutions (Leicester University and Warwick University) to determine the chemical composition or chemical oxides of the materials used in the experimental study;

ix. Evidence of email correspondence with suppliers from which samples were obtained; and

x. Data for laboratory test results for compressive strength, high pressure permeability tests and freeze thaw tests will be submitted on request in excel format. Submissions of all other test results will be in the format generated by the packaging software used to undertake the experiments.

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TABLES

Table 1: Chemical oxide composition of PWG

Composition (%)	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	Na_2O	K_2O	P_2O_5	SO_3
PWG	0.96	0.03	0.31	0.27	0.13	40.00	0.02	0.09	0.02	52.00

Table 2: Chemical oxide composition of V-B5G

Composition (%)	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	Na_2O	SO_3	LOI
V-B5G (2017)	0.21	<0.007	<0.022	0.08	0.04	47.85	0.35	50.34	1.91
V-B5G (2018)	0.20	-	0.06	0.04	0.04	41.00	0.30	54.00	-

Table 3: Chemical oxide composition of RAF

Composition (%)	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	Na_2O	K_2O	P_2O_5	SO_3	MnO
RAF (2017)	39.68	1.44	14.91	10.12	6.60	9.88	3.67	0.90	0.56	2.59	-
RAF (2018)	35.00	1.60	12.00	12.00	6.40	12.00	1.90	1.20	0.47	1.30	0.42

Table 4: Chemical oxide composition of GGBS

Composition (%)	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	Na_2O	K_2O	SO_3	MnO
GGBS (2017)	33.23	1.09	13.14	0.52	8.92	39.76	0.21	0.45	1.12	0.33
GGBS (2018)	34.20	0.52	12.62	0.69	6.77	41.12	0.07	0.48	1.75	0.31
GGBS (2019)	34.76	0.51	11.44	0.64	7.23	40.80	0.11	0.46	1.70	0.30

Table 5: 50th and 90th percentiles of materials

	PWG	V-B5G	RAF	GGBS
D50	202.54	92.77	18.18	17.3
D90	564.93	419.55	51.42	49.27

Table 6. Grading analysis of RAF

BS Test Sieve Nominal Aperture Size	Cumulative Percentage Passing	Specification Limits
2.0 mm	100	100
125 microns	100	85 - 100
63 microns	96	70 - 100

Table 7. Mix design proportions of Group 2 and Group 2A

Components	**Mix Design**												
(% wt)	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**
PWG/V-B5G	20	10	10	5	20	15	20	0	15	0	0	5	10
RAF	20	80	55	47.5	50	37.5	80	80	67.5	40	60	67.5	30
GGBS	60	10	35	47.5	30	47.5	0	20	17.5	60	40	27.5	60

Table 8. Mix 1 and Mix 13 statistical analysis

	90 Day Compressive Strength (MPa)			Mean	Standard Deviation	Density Kg/m^3
	Test 1	Test 2	Test 3			
Group 2 Mix 1	41.24	37.52	38.99	39.25	1.87	1920
Group 2A Mix 13	39.50	38.48	38.67	38.88	0.54	2135

Table 9. Strength development of selected mixes on Groups 2 and 2A

Group	Mix Design Reference	Strength at Days (MPa)					L/S Ratio
		7	14	28	90	180	
2	Mix 1 - PWG20/RAF20/GGBS60	2	13	21	41	41	0.17
	Mix 4 - PWG5/RAF47.50/GGBS47.50	2	9	19	30	38	0.17
	Mix 6 - PWG15/RAF37.50/GGBS47.50	2	9	18	35	36	0.17
	Mix 13 - PWG10/RAF30/GGBS60	2	18	25	37	39	0.17
2A	Mix 1 - V-B5G20/RAF20/GGBS60	3	12	26	33	35	0.19
	Mix 4 - V-B5G5/RAF47.50/GGBS47.50	1	19	21	31	40	0.19
	Mix 6 - V-B5G15/RAF37.50/GGBS47.50	2	12	24	33	35	0.19
	Mix 13 - V-B5G10/RAF30/GGBS60	2	13	28	40	41	0.19

Table 10. Resistance to freezing and thawing on Mix 1(Group 2) and Mix 13(Group 2A)

Mix	SET 'A'		SET 'B'		Durability Factor R_A/R_B (%)
	91d (MPa)	Density Kg/m3	91d (MPa)	Density Kg/m3	
Mix 1 (PWG20/RAF20/GGBS60)	36.43	1895	38.99	1930	93.43
Mix 13 (V-B5G10/RAF30/GGBS60)	36.13	2002	38.67	2014	93.44

FIGURE CAPTIONS LIST