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AN EXPERIMENTAL INVESTIGATION ON MINE FILLING MATERIALS PERPERATION USING FLY ASH, CARBIDE SLAG, FGDG AND GBF

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ABSTRACT: Owing to the increasing depletion of nonrenewable resources and effects produced by global warming, the high levels of energy consumption and pollution generated during the production of cement clinker have attracted widespread concern. Utilization and recycling of industrial waste and by-products to prepare building materials is the green style and contributes to the resource conservation and environmental protection. Recovering solid waste represents one of the major methods for manufacturing such "green" cementitious materials. Hence in this work, an experimental investigation on mine filling materials preparation using fly ash, carbide slag, FDCG (Flue-Gas Desulphurisation) gypsum and GBF (Granulated Blast-Furnace Slag. Environmentally friendly and cheap composite green cementitious materials have been prepared from carbide slag, fly ash and flue gas desulphurisation Gypsum (FGDG) and Granulated Blast-Furnace without using cement clinker. This work investigates the effects of the raw materials on the amount of water required for reaching standard consistency, slump value, and strength of the produced materials after curing for 7 d and 28 days. Scanning Electron Microscopy (SEM) and X-ray diffraction (XRD) techniques are used to analyze the sample microstructure and hydration products as well as for the exploration of possible hydration mechanisms.

KEYWORDS: Mine Filling Materials, Recycling Industrial Waste, Flue-Gas Desulphurisation Gypsum (FGDG) and Granulated Blast-Furnace (GBF).

I. INTRODUCTION

With the rapid development of nation's industrialization progress, the consumption of natural resources increases continuously. During the process of exploiting and smelting, natural resources produce a large amount of waste residue.

If those waste residues cannot be solved reasonably, lots of lands and resources will be wasted. Once the harmful substance in the waste residue penetrates the ground with surface water, the serious harm to soil, groundwater, surface water and organisms that depend on the environment will be caused [1]. The development of industrial activities such as steel and iron manufacturing, smelt and power generation lead to the release of large quantities of flue gas desulfurisation (FGD) gypsum, blast furnace slag (BFS), silica fume (SF) and fly ash (FA), which occupies much land and poses a threat to the environment if disposed in landfills. Therefore, it is a hot topic to use and recycle these industrial wastes instead of landfilling [2].

FA, BFS and SF are by-products from coal burning thermal power stations, manufacturing of pig iron and the electrometallurgy industry, respectively. Due to their good pozzolan and water-hardening nature, they are usually utilized in the manufacturing of cement and concrete, which has a low post-performance loss. FGD gypsum is a by-product of power station where FGD equipment is installed to remove SO2 from the flue gas by adding limestone/lime. The higher content of calcium sulphate dihydrate, fewer impurities, smaller and more uniform particle size, wider range of sources, better fire resistance, lower thermal expansion coefficient, lower cost and sounder insulation properties are the

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obvious advantages of FGD gypsum. Energy conservation and emission reduction have become the modern world's current environmental protection themes. Pollution and the disposal of industrial wastes like fly ash (FA), desulfurization gypsum (FGD), and phosphogypsum have proven to be hot topics and seriously threaten subjects dealt with by scientists and technologists.

It is commonly known that cement production is one of the most energy consuming processes, equal to the disposal of industrial by-products. Ordinary Portland Cement is composed of most man-made materials used worldwide, but it not only requires a mass of calciumrich materials, but also requires high-temperature sintering for its formation, e.g. 1450°C. Consequently, the cement industry contributes around 6 % of all CO_2 anthropogenic emissions, which arose from calcite decarbonation. Over the years, much attention has been paid to the development of modified cement clinkers, giving rise to energy preservation as well as interests concerning its use as a binder for waste encapsulation. The main way to reduce the cost of cementing materials is to maximize the use of industrial waste [5].

Mining activities bring us resources, as well as geological hazards and environmental pollution, most prominently in the form of surface subsidence. Cement filling is an effective technique to avoid or reduce surface subsidence. Cementitious materials prepared from industrial waste, such fly ash (FA), are frequently used for this purpose. This filling method offers new ways of utilizing waste, and effectively promotes the sustainable development of the coal industry [6].
Recovering solid waste represents one of the major methods for manufacturing such "green" cementitious materials. The utilized waste typically includes fly ash (the fine ash collected from the soot produced during coal combustion), which exhibits a pozzolanic effect and can be activated in an alkaline environment. Another solid waste material consists of granulated slag (or water quenched blast-furnace slag), which can potentially exhibit hydraulic cementitious properties. The major component of FGD gypsum is calcium sulphate dihydrate. At present, these industrial wastes are primarily utilized for the production of cement, concrete, geopolymers, and cementitious materials for mine filling.

Hence in this work an experimental investigation on Mine filling materials preparation using Fly ash, carbide slag, FGDG and Granulated Blast-Furnace. The rest of the work is organized as follows: The section II describes the literature survey. The section III demonstrates the experimental investigation on Mine filling materials preparation using Fly ash, Carbide slag, FGDG and GBF. The section IV evaluates the result analysis of presented approach. Finally the work is concluded in section V.

II. LITERATURE SURVEY

Chao Ren, Keqing Li, Wen Ni, Siqi Zhang et. al., [3] presents Preparation of Mine Filling Material from Steel Slag Mud. The steel slag mud is activated through mechano-chemical activation and the mechanical activation laws are explored through X-Ray Diffraction (XRD) and Fourier-Transform Infrared Spectroscopy (FT-IR). The results shows that mechano-chemical activation can partially excite he activity of steel slag mud and that the fitness of steel slag mud is positively correlated with the mechanical performance of filling material. In addition, the low field nuclear magnetic resonance (NMR) was adopted to analyze how the fitness of steel slug mud affects the porosity distribution of filling material and disclose the relationship between fineness and the material strength.

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L. A. Krupnik, Yu. N. Shaposhnik, S. N. Shaposhnik, and G. T. Nurshaiykova et. al., [4] presents Backfilling Mixture Preparation Using Milled Granulated Blast-Furnace Slag. Backfilling mixture preparation technology using a cement–slag binder is developed for the Artem'evsky mine. It is shown that backfill with granulated blast-furnace slag reaches project strength at its fineness 80% of content milled down to –80 μm size. The authors analyze influence of milling fineness of granulated blast-furnace slag from different manufacturers on strength and rheological properties of backfill. The economic analysis of cost of binder in formation of load-bearing layer of backfill prepared using fly ash and milled granulated blast-furnace slag is performed.

Li Chao, Zhao Feng-qing, et. al., [7] presents Application of fly ash /granulated blast-furnace slag cementing material for immobilization of Pb2+. Based on activation and synergistic effect among various materials, a low-cost cementing material, FGC binder, was prepared by using fly ash, granulated blast-furnace slag(BFS), carbide slag and compound activator. The results showed that the immobilization efficiency of FGC binder for Pb2+is higher than that of OPC cement. The hydration productsand mechanism of immobilization were analyzed by using XRD. The major products of FGC binder are C-S-H, C-A-H, ettringite and zeolite-like materials. Under the experimental conditions, the Pb2+ curing efficiency of FGC binder is 1.04 ~ 1.24 times that of ordinary Portland cement.

A. Sarkar, A. K. Sahani, D. K. Singha Roy, and A. Kr Samanta et. al., [9] presents a Compressive strength of sustainable concrete combining blast furnace slag and fly ash,In this study, concretes with binary and ternary mixes, including FA as a binder and GBFS as filler constituents, were produced to investigate their effects on the mechanical characteristics of concrete with respect to various replacements and ages. In binary mix, Ordinary Portland Cement (OPC) was partially replaced (20%, 30% and 40%) with FA by weight. The test result shows that the binary mix gives up to 32.57% less strength than control mix. But the ternary mix with same FA replacement and 50% GBFS replacement improves the strength by 25.93% in 28 days and 13.93% in 90 days age

Bing Ma, Xuerun Li, Yuyi Mao, Xiaodong Shen et. al., [10] presents Synthesis and Characterization of High Belite Sulfoaluminate Cement Through Rich Alumina Fly Ash And Desulfurization Gypsum. The objective of this study was the preparation and characterization of High Belite Sulfoaluminate Cement (HBSC) from industrial residues. HBSC promises eco-friendly building materials with great mechanical performance at earlier ages than Ordinary Portland Cement (OPC). Preliminary results show the formation of main phase dicalcium silicate (C2S) and ye'elimite (C4A3$) at 1250°C, as determined by X-ray diffraction (XRD), are promising. The formation of minerals in the clinker was analyzed by differential scanning calorimetry-thermogravimetry (DSC–TG). The optimum compressive strength values of the mortars after 1-,3-,7-, and 28-days were 24.9 MPa, 33.2 MPa, 35.6MPa and 52.8 MPa which can meet the requirement of special structures.

III. AN EXPERIMENTAL INVESTIGATION ON MINE FILLING MATERIALS PERPERATION

In this work, an experimental investigation on mine filling materials preparation using Fly ash, Carbide slag, FGDG and GBF. In this analysis, the raw materials are Fly ash,

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Carbide slag, Flue Gas Desulfurization Gypsum and Granulated Blast-Furnace. Admixtures of sodium hydroxide (≥96.0 mass%) and naphthalene superplasticiser (Cl^- content <0.4 mass%, Sulphate content are used. M-sand river sand with a silicon dioxide content exceeding 98 mass% is utilized. Tap water is used in all experiments.

Carbide Slag (CS) is the waste from the hydrolysis of calcium carbide used in the formation of acetylene gas, with CaO and $Ca(OH)_2$ as its main components. The main reaction for its formation is: CaC_2+ $2H_2O$→ C_2H_2↑+ $Ca(OH)_2$. As industry develops, the production of CS increases every year. These large quantities of CS, if untreated, produce irritating odors and atmospheric pollution. The long-term accumulation of CS consumes large areas of land, and also has a severe corrosive effect on soil. In addition, by entering the water system, CS can also clog rivers. FA is the fine ash collected from the burning of coal. It can be activated under alkaline conditions to produce a material similar to volcano ash.

Blast-furnace-slag (BFS) is formed by the cooling of molten-iron slag by water and has water-binding potential. Flue Gas Desulfurization Gypsum (FGDG) is a type of industrial auxiliary gypsum obtained by desulfurization and purification of flue gas generated after the combustion of sulfur-containing fuel (e.g., coal). Its main component is SO_2, which reacts with $CaCO_3$ and generates calcium sulfate dihydrate ($CaSO_4.2H_2O$) through forced oxidation.

Granulated blast furnace slag has been used as a raw material for cement production and as an aggregate and insulating material. A by-product of the steel-making industry, granulated blast furnace slag (GBFS) is produced by the rapid cooling of iron ore slag. This creates a hydraulic material that is naturally cementitious and high in calcium silicate hydrates (C-S-H), a compound that increases the strength and durability of concrete.

The fly ash, carbide slag, FGDG, GBF, and admixture components were mixed together to form composite cementitious materials. Prior to mixing, the materials are weighed with an accuracy of 0.01 g using an electronic balance.

The cement/sand ratio is 0.5, and the water/cement ratio is 1. Subsequently, the weighed materials are mixed using the following procedure: first, a specified amount of granular sodium hydroxide is dissolved in water, and the resulting aqueous sodium hydroxide solution is added to the bowl of the mixer with a capacity of 7L which is fixed onto its frame and raised to a set position. Afterwards, the materials are immediately mixed for 45 s at a low speed of 150 ±5 rpm. Sand is continuously added at a uniform rate during the next 45s of mixing. After the sand addition, the mixer is run for another 45 s at a high speed of 300± 10 rpm and then stopped for 100 s.

Within the first 25 s after the mixer is stopped, the mortar that had adhered to the blades and bowl wall is scraped into the centre of the bowl. After a 120 s interval, the mixing process is continued for another 60 s at a high speed. Immediately after mixing, the materials are moulded using an empty test mould (with dimensions of 40mm× 40mm× 160mm) and a bushing fixed onto a vibrating compaction table.

A scoop is used to obtain mortar from the mixer bowl, which is placed into the test mould in two layers. About 400 g of mortar is put into each groove in the first layer, which was then compacted via 70 vibrations. The second layer is added until the mould is filled, and the mortar is again

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compacted by 670 vibrations. Afterwards, the mould bushing is removed, and the test mould is transferred from the vibrating compaction table.

Any mortar exceeding the dimensions of the test mould is scraped away using a metal ruler, and the produced test block is numbered. The moulded test block is cured at a temperature of 25±2°C in the environment with a relative humidity greater than 60%. After 24 h of curing, the mould is removed and subjected to another curing procedure at 25± 2°C inside the curing chamber with a relative humidity of 96%. Each test block is cured for either 7 or 28 d, and its strength is measured after the corresponding curing period. The parameters like slump test, consistency, Compressive strength and Flexural Strength are measured.

IV. RESULT ANALYSIS

An experimental investigation on mine filling materials preparation using Fly ash, Carbide slag, FGDG and GBF is evaluated in this section. In this analysis, XRD analysis, SEM analysis, consistency, slump testing, compressive strength and flexural strength are investigated which is as follows:

XRD Analysis: After the test blocks are cured for 7 and 28 days, the surfaces that might be possibly carbonated are removed with a knife. The samples are removed from the interior of each test block and cut into 3–6mm pieces, which are subsequently immersed in a mixture of absolute ethanol and acetone. After the hydration reaction is complete, the test blocks are dried for about 48 h at 45°C and then ground into fine powder for XRD analysis.

Scanning Electron Microscopy Analysis: After the test blocks are cured for 7 and 28 d, additional samples are taken from their interior and cut into 3–56mm pieces, which are subsequently dried to a constant weight in a vacuum drying tube with a vacuum degree of 740mm Hg at 65°C. Afterwards, the samples are metalized under vacuum, placed into a scanning electron microscope for the observation of their cross-sectional morphology, and photographed.

Consistency: An SC-145 mortar consistometer is used to determine the consistencies of the produced cementitious materials. First, each prepared cementitious material is placed inside a container. When the tip of the testing cone touched the material surface, the clamping screw is unscrewed, allowing the testing cone to fall freely. The falling depth displayed on the consistometer dial corresponded to the consistency value for the cementitious material (the results of three tests are averaged for each studied cementitious material), and the measurement accuracy is 1 mm.

Slump Testing: The cementitious material is packed inside a tube in three layers with approximately equal volumes. Each layer is treated with a tamping rod evenly inserted in the slump cylinder 25 times following the shape of a spiral. After the completion of the tamping process, the slump tube is removed, and the difference between the centre point of the specimen's top and the height of the produced slump (corresponding to the slump value) is measured by a steel ruler. The duration of the entire testing procedure is about 180s. If the studied sample exhibited collapse or shear, the results of the slump test are considered negative, and the testing procedure is repeated.

The table 1 shows the materials proportioning in investigation.

Table 1: Materials Proportions

Number	FGDG (g)	GBF (g)	CS (g)	FA (g)	S-H (g)	Water reducing admixture (g)

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1	9	24	13	95	7.8	1
2	9	28	15	95	8.2	1
3	9	30	17	95	8.5	1
4	11	26	13	95	8.0	1
5	11	28	15	95	8.3	1
6	11	32	17	95	8.5	1
7	13	28	13	95	8.2	1
8	13	30	15	95	8.4	1
9	13	32	17	95	8.6	1
10	15	24	13	95	8.4	1

The table 2 shows the experimental evaluation of presented approach.

Table 2: Performance Metrics Evaluation

Number	Consistency (mm)	Slump (mm)
1	11	210
2	11	123
3	13	131
4	11	147
5	14	168
6	14	134
7	17	195
8	15	126
9	11	181
10	9	136

From table 2 slump values, It shows that when the raw material combination is corresponds to the mass ratio of FGDG: GBF: CS of 13: 28: 13), it exhibits the greatest impact on the slump of the cementitious material. The Fig. 1 shows the slump value comparison of different cementitious materials.

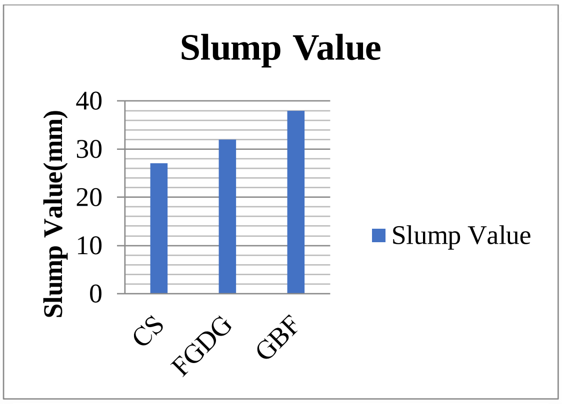

Fig. 1: Slump Value Comparison

From the Fig. 1, it is clear that the resulting slump value is affected by the following factors: GBFS > FGDG >CS, because the former is composed of the spherical vitreous bodies with a smooth and compact surface, which ensure good lubrication of the cementitious material.

The table 3 shows the compressive strength and flexural strength for 7 and 28 days respectively.

Table 3: Experimental Evaluation

Number	Compressive strength (MPa) for 7 days	Flexural Strength for 7 days	Compressive strength (MPa) for 28 days	Flexural Strength for 28 days
1	1.82	0.67	3.23	1.34
2	1.93	0.64	3.56	1.06
3	2.24	0.96	3.76	1.15
4	1.34	0.54	3.1	0.99
5	2.34	0.82	2.96	1.23
6	1.42	0.74	3.43	1.24
7	2.64	0.95	3.89	1.54
8	1.45	0.97	3.21	1.34
9	2.13	0.56	4.08	1.05
10	1.67	0.45	3.72	1.23

The compressive strengths of the cementitious material aged for 7 and 28 d are equal to 2.64 and 4.08 MPa.

The highest material strength is achieved at a mass ratio of GBF to FGDG and carbide slag of 13: 30: 24). As indicated by the range analysis results presented in Table 3, at a raw material combination corresponding to the mass ratio of FGDG: GBF:CS equal to 9: 32 : 16), it exhibits the greatest impact on the 7 d strength of the resulting material. When the compositions of the raw materials are corresponding to the mass ratios of FGDG: GBF:CS equal to 13 : 28 :15 and 9: 28: 17, resp.) they exhibit the greatest impact on the 28 d compressive strength and flexural strength of these materials. The Fig 2 shows the graphical representation of Complex strength and flexural strength for 7 days.

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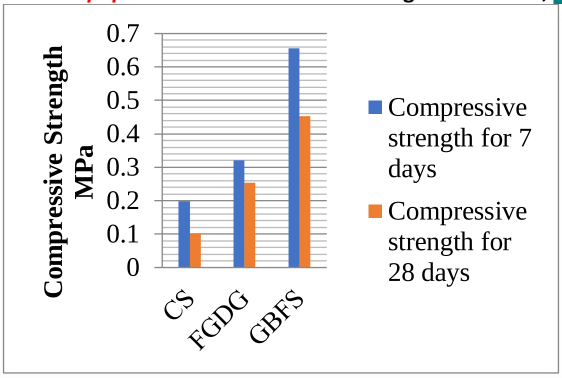

Fig. 3: Compressive Strength Comparison

that, 7 d strength of the cementitious materials as follows: GBF> FGDG > CS. after 28 d of aging, their effects on the compressive strengths can be described as GBF >FGDG >CS, respectively. The Fig.3 shows the flexural strength comparative graph.

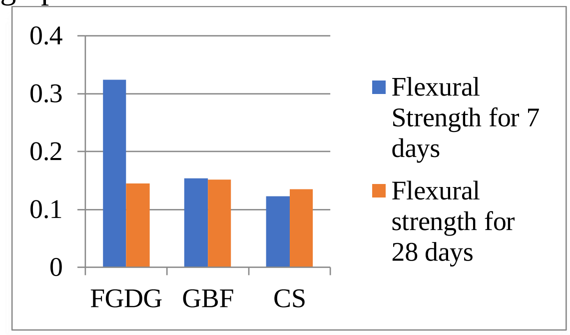

Fig. 4: Comparative graph for flexural strength

The results presented in Fig. 3 reveal that, 7 d flexural strength of the cementitious materials as follows: GBF> FGDG > CS. after 28 d of aging, their effects on the flexural strengths can be described as GBF >FGDG >CS. Among the different cemntious materials, GBF has shown better results in terms of slump value, complex strength and flexural strength.

V. CONCLUSION

An experimental investigation on mine filling materials preparation using Fly ash, Carbide slag, FGDG and GBF is presented in this work. In this analysis, the activity of fly ash and other industrial waste slag is stimulated by the presence of carbide slag in the filling cementitious materials prepared without adding any cement clinker. This work investigates the effects of the raw materials on the amount of water required for reaching consistency, slump value, and strength of the produced materials after curing for 7 d and 28 days. From the result it is found that, among the utilized raw materials, the addition of GBF had the most significant effect on the consistency and slump value, compressive strengths and flexural strength. The manufacturing of fly ash-carbide slag-GBF-FGDG cementitious materials utilizes substantial amounts of industrial waste (including fly ash and carbide slag), which can potentially produce significant social and economic benefits.

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