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# The Use of Secondary Gypsum to Make a Controlled Low Strength Material

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**Abstract:** This paper reports on the use of secondary minerals to make Controlled Low Strength Materials for trench or mine backfill. The mixes used red titanogypsum which is a by-product of titanium dioxide pigment production. A number of other secondary minerals were considered for mixing with the gypsum and steel slag from the basic oxygen process was found to be most successful. An optimised mix was developed and a site trial was carried out. The results showed a good performance but the failure of one test sample during curing showed that the material may not be suitable for other applications.

## 1. INTRODUCTION

The environmental advantages of reducing the use of cement in concrete are well known but the use of waste minerals to replace it may lead to a reduction in strength. This paper reports on the use of waste minerals to produce a controlled low-strength material (CLSM) for which the reduced strength is not a problem. The principal waste material used was waste "Red" gypsum. This material is a by-product of titanium dioxide production using the sulphate process. It contains too many impurities to be used in cement or plasterboard production.

The study includes a review of the literature on CLSM and its properties. Different materials are then discussed for mixing with the red gypsum to make it set. The most promising of these was Basic Oxygen Slag (BOS) from steel production. No materials with an existing market value were considered so Ground Granulated Blastfurnace Slag was not tested. The paper then describes laboratory trials of different mixtures. These started with numerous "cup" mixes followed by more detailed analysis of the more successful ones. Finally just one mix was selected and a subsequent site trial was carried out.

## 2. PROPERTIES OF CLSM

### 2.1. Introduction

CLSM is a self-compacting, flowable, low-strength, cementitious material used primarily as backfill, void fill, and utility bedding as an alternative to compacted fill. Many terms are currently used to describe this material, including flowable fill, unshrinkable fill, controlled density fill, flowable mortar, plastic soil-cement, soil-cement slurry, and various other names. It is produced in many countries, [1,2] normally produced at concrete batching plants.

The potential advantages of CLSM include decreased labour requirements, faster construction speed, and potentially

lower in-place cost. When considering CLSM as a construction material, two of the most important properties include flowability and compressive strength. The flowability of CLSM can directly impact on productivity and human resource requirements for placing the material. Lower flowability may require more resources. In addition, compressive strength values are required as an indicator for performance and long-term excavatability. Minimum strength requirements are necessary for performance criteria, and maximum strength requirements are necessary for long-term excavation.

Flowable fill contains many of the same materials as typical concrete and the same equipment and mixing trucks are used, but the proportions of the components are very different. Flowable fill can be used for backfilling in trenches, around pipes, and under roadways because it is self-levelling and leaves no air spaces. Flowable fill also serves as a less expensive alternative to the removal of old, underground storage tanks. The tanks can easily be filled in with flowable fill instead of being removed, saving both time and money [3].

Flowable fill is often used now because it saves so much time. Other fill materials take up to six times longer than flowable fill to place and properly compact [4]. Controlled low-strength material does not require any compaction, and in most cases does not have to be placed in lifts. Also, after only a matter of hours, CLSM can gain enough strength to support traffic loads, minimizing the time needed to close off roadways for construction [3]. Unlike most conventional fills, flowable fill can even be utilized in the winter, taking full advantage of work time. If the material does freeze and break down after being placed during colder months, it essentially becomes well-graded fill that can be left as it is [4].

CLSMs are defined by ACI 116R as materials that result in a compressive strength of 8.3 MPa or less. Most current CLSM applications require unconfined compressive strengths of 2.1 MPa or less. This lower-strength requirement is necessary to allow for future excavation of the material.

The term CLSM can be used to describe a family of mixtures for a variety of applications. For example, the upper

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limit of 8.3 MPa allows use of this material for applications where future excavation is unlikely, such as structural fill under buildings. The use of preformed foam in low-density CLSM mixtures allow these materials to be produced having unit weights lower than those of typical CLSM. Future CLSM mixtures can be developed as durable pavement bases.

CLSM should not be considered as a type of low-strength concrete, but rather a self-compacted backfill material that is used in place of compacted fill. Generally, CLSM mixtures are not designed to resist freezing and thawing, abrasive or erosive forces, or aggressive chemicals. Non-standard materials, which can be available and more economical, can also be used to produce CLSM mixtures as long as the materials have been tested and found to satisfy the intended application and project requirements (ACI 229R-99, 2003) [5].

CLSM should not be confused with compacted soil-cement, CLSM typically requires no compaction or curing to achieve the desired strength. Long-term compressive strengths for compacted soil-cement often exceed the 8.3 MPa maximum limit established for CLSM.

Long-term compressive strengths of 0.3 to 2.1 MPa are low when compared with concrete. In terms of allowable bearing pressure, however, which is a common criterion for measuring the capacity of a soil to support a load, 0.3 to 0.7 MPa strength is equivalent to a well-compacted fill.

Although conventional CLSM generally costs more per  $m^3$  than most soil or granular backfill materials, its many advantages often result in lower in-place costs. In fact, for some applications, CLSM is the only reasonable backfill method available [6]. However, more importantly, incorporating materials such as Red Gypsum in CLSM can make it a cheaper product.

The properties of CLSM cross the boundaries between concrete and soil and are most similar to soil cement. It is usually made by producers of ready-mixed concrete, using for the most part, materials used to produce conventional concrete. The mixing, transporting, and placing of CLSM is done using equipment normally used for concrete work. However, when CLSM hardens, it exhibits properties similar to that of compacted soil. Since CLSM is a composite material, the characteristics of the component materials and their proportions in the mixture control the properties of the resultant CLSM.

The critical properties of plastic or unhardened CLSM are: flowability, time of setting or hardening (final setting time), bleeding or segregation, subsidence, and pumpability. Similarly, the dominant properties of the hardened material are: strength or bearing capacity, permeability, shrinkage and excavatability. These are all important in defining the performance envelop of these materials and may assume a wide range of values.

## 2.2. Flowability

Flowability is the property that allows CLSM to completely fill a void area, self-level, and self-compact without any conventional placing or compacting equipment. This is the property that makes CLSM different from conventional

backfill and, in some applications, gives it an advantage over conventional backfill materials and methods.

However, this high degree of flowability can be a problem. The hydrostatic pressure exerted by the highly flowable CLSM will impose loads on adjacent structures. The stability under this load should be checked. If the stability of adjacent structures is a concern, multiple lifts of CLSM may be used to decrease the pressure exerted from a single high lift. After the CLSM hardens, no hydrostatic pressure is exerted and the next lift can be placed.

There are many methods of expressing flowability, perhaps the best known of which is the flow table test [7]. A mix is flowable when the spread is 510 – 620 mm in diameter. This corresponds to 110 – 210 mm in diameter for the small modified flow table used in this study. For high flowability spread of greater than 190 mm in diameter is required.

## 2.3. Hardening Time

Final setting or hardening time is the approximate period of time required for CLSM to go from a plastic state to a hardened state with enough strength to support the weight of a person. The initial setting time is the approximate time required for CLSM to go from fluid state to plastic state.

Factors affecting the hardening or setting time of a CLSM are:

- Type and quality of cementitious material
- Permeability and degree of saturation of surrounding soil that is in contact with the CLSM
- Water content of the CLSM
- Mixture proportions of the CLSM
- Ambient and mixture temperature
- Humidity
- Depth of fill

The time of final setting can be as short as 1 hour (ACI 229R-99, 2003) [5], but generally takes 3 to 5 hours under normal conditions [3,8].

## 2.4. Strength

Unconfined compressive strength is a measure of the load-carrying ability of CLSM. A CLSM compressive strength of 0.3 to 0.7 MPa equates to an allowable bearing capacity of a well-compacted soil [5]. It may be important to determine the long-term strength of a mixture that may be excavated at a later date. As with most Portland cement and fly ash concrete and grout mixtures, CLSM can continue gaining strength with age given the proper conditions, such as a moist environment. It may be important therefore to limit long-term strength so as to facilitate later excavation. Section 2.6 provides additional information on excavatability.

## 2.5. Permeability

Permeability of most excavatable CLSM is similar to compacted granular fills. Typical values are in the range of  $10^{-6}$  to  $10^{-7}$  m/sec. Finer constituent materials and mixtures

of higher strength can achieve permeabilities as low as  $10^{-9}$  m/sec [5]. Permeability is increased as cementitious materials are reduced and aggregate contents are increased.

## 2.6. Shrinkage

Shrinkage (cracking) of CLSM is very small and has been reported to be in the range of 0.02 to 0.05% [9]. This small amount will not affect the performance of a CLSM. Where shrinkage cracks do occur, subsequent lifts of CLSM will fill these cracks. A high-strength CLSM mixture or even a concrete mixture could be placed as a top wearing surface where needed.

## 2.7. Excavatability

The ability to Excavate CLSM, as mentioned earlier, is an important consideration on many applications. In general, an unconfined compressive strength of 0.3 MPa or less can be excavated manually. Mechanical equipment, such as backhoes are used with stronger materials, typically with compressive strengths of 0.7 to 1.4 MPa [5]. The limits for excavatability are somewhat arbitrary depending upon the CLSM mixture. Mixtures using high percentages of coarse aggregate can be difficult to remove manually, even at low strengths, on the other hand mixtures using fine materials as the aggregate filler have been reported to be excavated with a backhoe up to strengths of 2.1 MPa [10]. Again, it is suggested that long-term strength test results be conducted to estimate the potential for late excavatability.

## 3. EXAMINATION OF CANDIDATE MATERIALS

### 3.1. Red Gypsum

By-product gypsum is a ubiquitous material in industrialised societies. Flue gas desulphurisation and acid neutralisation through limestone addition account for the bulk of production, although a number of other synthetic gypsum sources exist. Chemically produced gypsum is described in terms of the manufacturing process from which it originates (phospho-gypsum, pharmaco-gypsum, boro-gypsum etc.) and this study focussed on one specific material; titano-gypsum, a by-product of the pigment industry. This is a by-product from the production of titanium dioxide which is widely used as a white pigment. Current UK arisings of red gypsum are approximately 125,000 Tonnes per year.

White by-product titano-gypsum finds a ready market in cement (and particularly) plasterboard manufacture. Red gypsum, by comparison, contains a considerable quantity of ferric floc, which limits its market in construction products, owing to the potential for iron staining after wetting. Red gypsum contains around 75% gypsum and 25% ferric floc by dry mass. As supplied, the material contains and additional 30-40% moisture.

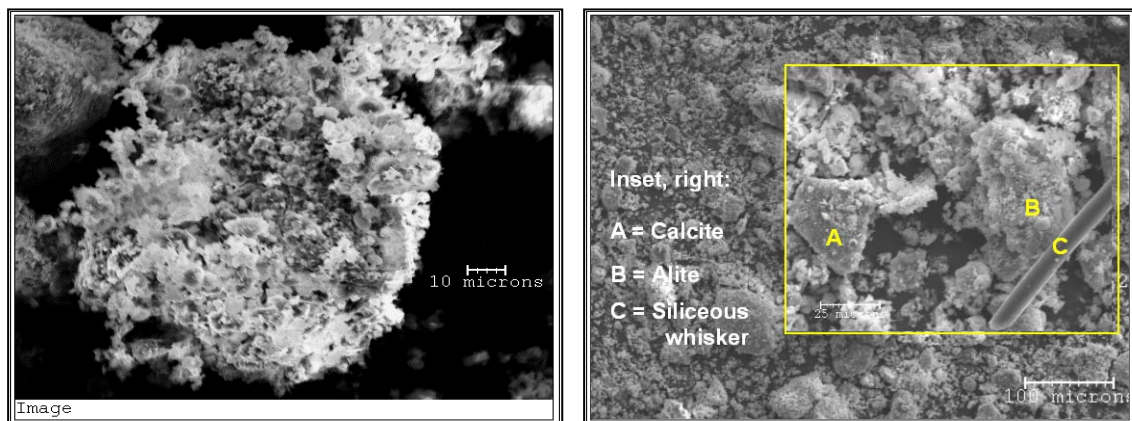
### 3.2. Cement Kiln Dust

CKD is the primary waste product from the manufacture of Portland cement and is largely calcite flour with some calcium hydroxide and cement clinker minerals. This fine-grained material comprises partially reacted feed materials used in cement manufacture, including up to 50% calcium oxide, silica, alumina and up to 10 % potassium oxide, along with cement clinker mineral grains. There are strict composition limits imposed on Portland cement and, due to its high potassium oxide content, CKD cannot be reintroduced back into the cement manufacturing process. Currently much of the CKD generated in the UK is disposed to landfill. Fig. (1) shows that the material used here ranges from small grains of a micron or two in diameter to grain clusters of a hundred microns or so. These samples are largely a mixture of partially calcined calcite with free calcium oxide, tricalcium silicate and larnite. A distorted glassy phase appears as shards of an alkaline glass (silica containing potassium ions).

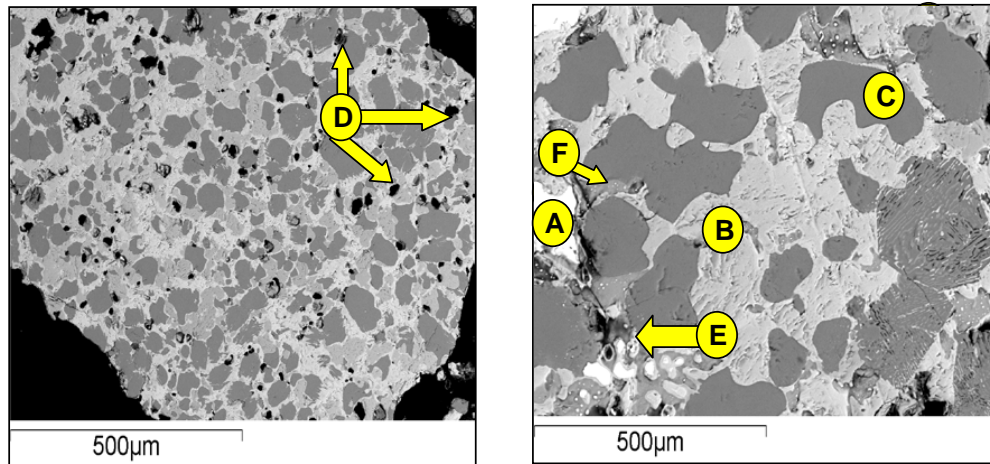
### 3.3. Steel Converter Slags

Whilst the red gypsum and CKD are simple mixtures of relatively predictable composition, the steel slag is quite different and should be described before examining the CLSM materials containing it.

The conversion of iron into steel is dominated by two technologies, by far the largest of which is the Basic Oxygen Process in which iron is refined into steel by selective oxidation of its impurities. The Electric Arc Furnace is important in regions where electricity is relatively cheap and in some operations where scrap steel dominates the feed stock. The Basic Oxygen process differs from the Electric Arc process in that it is autogenous, or self-sufficient in energy. The primary raw materials for the Basic Oxygen Process are 70-80% liquid hot metal from the blast furnace and the balance is steel scrap. These are charged into the Basic Oxygen Fur-



**Fig. (1).** Secondary electron images of the cement kiln dust used in the CLSM formulations.



**Fig. (2).** Phase “A” is an iron-rich inclusion, “B” is a calcium-iron-manganese-magnesium oxide, phase “C” is a calcium silicate, approximating to impure wollastonite and “D” is porosity. The inclusions marked “E” are free lime and those shown as “F” are periclase (MgO).

nace vessel. Oxygen (>99.5% pure) is “blown” into the furnace at very high rates. It oxidises the carbon and silicon contained in the hot metal liberating great quantities of heat which melts the scrap. There are lesser energy contributions from the oxidation of iron, manganese, and phosphorus. The subsequent combustion of carbon monoxide as it exits the vessel, also transmits heat back to the bath.

The commercial product of the Basic Oxygen process is molten steel at 1450°C-1660°C with a specified chemical composition. From here, it may undergo secondary refining or be sent directly to the continuous caster where it is solidified into billets prior to forming processes such as rolling or forging.

In order to control the metallurgy of the steel, various additions are made to the charge in the form of fluxes, commonly limestone or fluorite ( $\text{CaF}_2$ ). The increased activity of calcium in the melt, scavenges silicon, phosphorous and sulphur from the metal as these elements preferentially partition into the flux. The term *Basic* refers to the magnesia ( $\text{MgO}$ ) refractory lining which degrades, through contact with hot, basic slags. Although steel converter slags are dominated by the oxides of Ca, Si, P, Fe and Mn, the elemental ratios,

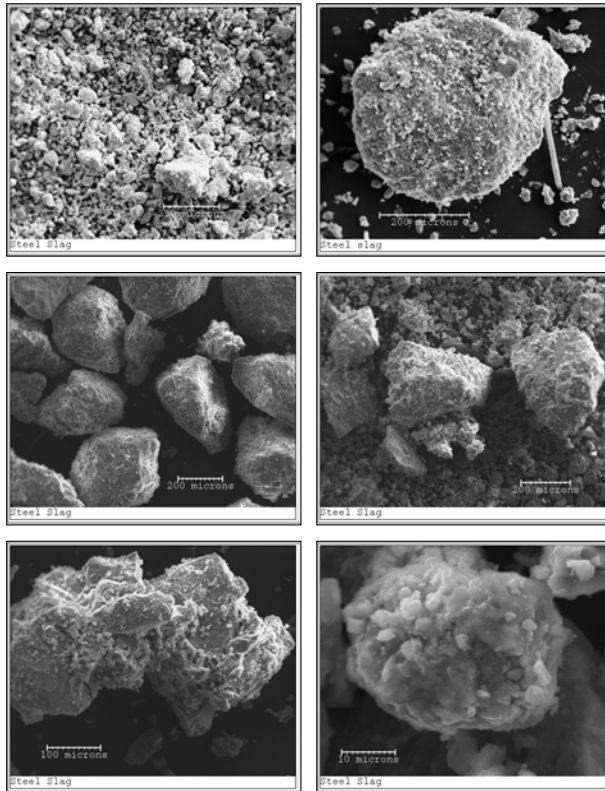
phase chemistry and minor components are extremely variable. This reflects both the chemistry of the charge (iron and gangue minerals) and the alloying components added to the vessel to produce a steel of particular composition. To add further complexity to the slag, it is tapped (removed from the vessel) before complete reaction, so the cooled slag may be far from thermodynamic equilibrium. Its phases are largely solid solutions of non-stoichiometric composition, so certain approximations are appropriate to its description. For example, consider Fig. (2), which show the range of major phases and their size distributions.

The material supplied for this study exhibits two major porosity types. The discontinuous porosity shown in Fig. (2), originated as gas bubbles, trapped as vesicles on cooling. The second type is fracture porosity Fig. (3) presumably results from cooling cracks and mechanical damage during processing. Fig. (3) illustrates the second type, which is continuous through the grains, allowing the resin used to mount the samples for microscopy to permeate the grains.

Fig. (4) shows the morphologies and range of grain sizes typical of the steel slag used in this study. Although a number of particles exceed two millimetres in diameter, the ma-



**Fig. (3).** BOS slag grain. Left: Secondary electron image showing surface morphology, right, backscattered electron image showing fracture porosity within the grain. As the efficiency of electron backscattering is a function of the average atomic number of the target, the technique reveals the internal fractures within the slag grain.

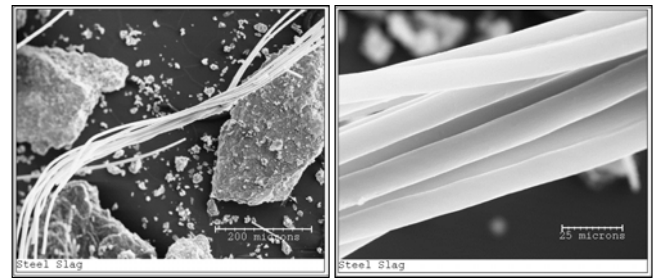


**Fig. (4).** BOS slag grains, secondary electron images. Each particle is a polycrystalline grain containing more than one distinct phase. That shown on the lower left is a polycrystalline grain, but of a single composition: free lime (CaO)

majority of the particles are angular fragments ranging from a few microns to a few hundred microns in size. The larger particles are polycrystalline, but many grains in the micron size range are monomineralic. Few well developed cleavage surfaces are seen at any scale, suggesting that the extent of crystal growth is limited.

As the slag contains occluded domains of free lime, often connected by continuous fracture porosity, the use of the slag as a coarse aggregate in concrete is limited, owing to the risk of lime hydration after setting. Ingress of pore solution will slake the free lime to form calcium hydroxide, with a corresponding increase in molar volume. This reaction limits the use of these slags as aggregates in many construction applications, as the requirement is for a large aggregate particle, some millimetres across. In the applications considered here, the steel slag is used as a fine dust, where the particle size is little larger than that of the crystalline domains in the slag. It is reasonable to expect that hydration of the free lime will largely occur during mixing.

It is reasonable to assume that the grain size of basic oxygen slag is of critical importance to its suitability and long-term stability in cementitious materials. If the size of the grains is little bigger than the size of the phase domains it contains, hydration of calcium oxide to its hydroxide is likely to be rapid. As the particle size of the slag increases, so too does the time taken for pore solutions to migrate through the fracture network in order to hydrate the free lime. If this hydration step can be achieved before setting of



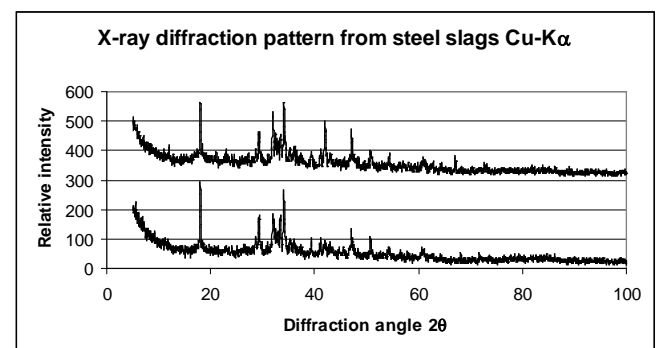
**Fig. (5).** Siliceous whiskers present in the BOS slag. Secondary electron images

the mix, any volume changes will be accommodated whilst the material is in its plastic state. For brittle materials such as concrete, which have little strength in tension, an expansive reaction may be catastrophic. By comparison, controlled low strength materials are likely to be more compliant than conventional concrete and through careful selection of the particle size of (in this case the slag) components, such concerns may be overcome at the design stage.

One feature of the basic oxygen slag was surprising; the occasional presence of siliceous whiskers. These are presumably drawn out when the hot, mostly solid slag is moved after tapping. These whiskers, shown in Fig. (5), are approximately 20µm in diameter and are largely silica with some sodium and potassium and a little calcium in their composition.

Considering the composition of these steel slags, examination by x-ray diffraction reveals little about their phase chemistry. Fig. (6) shows the highly disordered structure of the slag.

Fig. (6) shows (for two samples of BOS slag) that the x-ray diffractogram is very complex, with few strong reflections. Moreover, although the major peaks may be assigned to impure larnite, if this is the case, the lattice is considerably strained. The only other major phase which can be identified by x-ray diffraction is portlandite, suggesting that most of the calcium oxide has hydrated to calcium hydroxide during storage. This is not surprising, as finely divided free lime is highly hygroscopic and will readily hydrate if left open to the air. The complexity of the diffractogram and the disor-



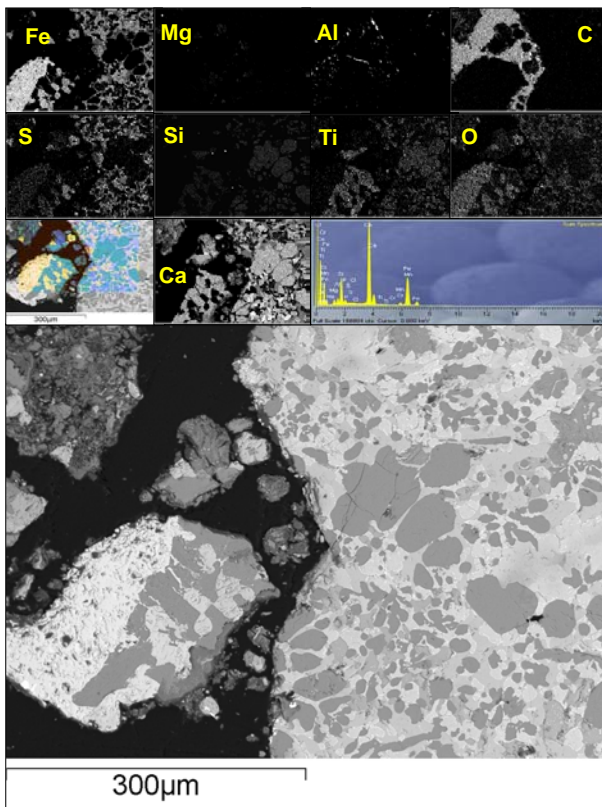
**Fig. (6).** X-ray diffraction pattern of the BOS slag. Principle reflections correspond to larnite,  $\text{Ca}_2\text{SiO}_4$  and Portlandite,  $\text{Ca}(\text{OH})_2$  suggesting that the slag has been in contact with water.

dered and non-stoichiometric nature of the slag, precludes further use of this technique for identification of anything other than qualitative indication of major phases.

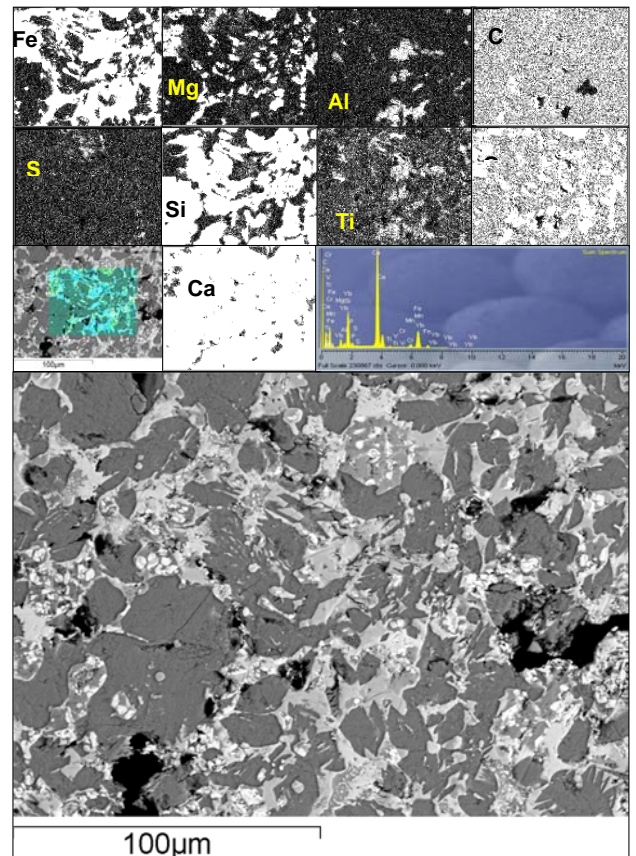
In light of the reactive nature of the free lime in these slags, a simple experiment was conducted to estimate the quantity of free lime available. Ten grams of powdered slag (sieved  $< 100\mu\text{m}$ ) was dispersed in 100ml water and shaken every ten minutes for an hour in a sealed glass flask. The liquid was filtered and titrated against standard nitric acid to pH 7, using bromo-thymol blue as an indicator. Three replicates suggested that the mass fraction of calcium oxide in the slag was: 0.8% , 1.3% and 0.7% giving an average of 0.9% free CaO by mass. Although this is not a rigorous determination (it assumes all the soluble alkali is  $\text{Ca}(\text{OH})_2$  ) it serves as a first approximation and shows the alkaline nature of the pore solution. The solution pH was measured at  $12.0 \pm 0.2$  whereas the equilibrium pH of  $\text{Ca}(\text{OH})_2$  is 12.45 at  $25^\circ\text{C}$ .

Figs. (7) and (8) show the phase distribution in the basic oxygen slags using backscattered electron images of polished surfaces. Each sample was examined by energy dispersive x-ray spectroscopy and, by image analysis to estimate the volume fraction assigned to each phase. The latter technique allows a measurement of internal porosity in the slag to be made, which is compared to that in the CLSM samples described later.

Six distinct phase domains can be distinguished: The darkest region is porosity, largely in-filled with mounting resin whilst the brightest are free iron or steel inclusions. A Ca-Fe-Mg-Mn phase is continuous and contains an impure



**Fig. (7).** X-ray maps and backscattered electron micrograph of polished BOS slag showing element associations in the non-stoichiometric phases



**Fig. (8).** X-ray maps and backscattered electron micrograph of polished BOS slag showing element associations in the non-stoichiometric phases

calcium silicate. In addition, impure free lime ( $\text{CaO}$ ) and impure periclase ( $\text{MgO}$ ) can be seen.

#### 4. INITIAL MIXING TRIALS

Numerous “cup” mixes were initially made in disposable plastic cups and tested [11, 12]. The cup mixes are shown in Fig. (9) and were made with different proportions of the materials and left to cure for a period of time and consequently tested for disintegration under water. After hardening time, samples were submerged in water in order to determine their hydraulic integrity and hand crushed to determine their relative strength and their pH values (determined when fresh). From these preliminary tests, the pastes to be tested further for the trench fill were chosen. The optimum paste proportions were determined next.

The candidate mixes for the trench fill trial were chosen from the results of optimum pastes and are given in Table 1. The small modified flow table used for flowability test used a  $75 \times 70$  mm open-ended cylinder.

#### 5. CANDIDATE TRIAL MIXES – MICROSTRUCTURE AND PHASE CHEMISTRY

Six formulations from the trial mixes were selected for microstructural examination and these are described below. The specimens selected were short cylinders of hardened CLSM, approximately 50mm diameter and between 15 and 25mm in height. They were broken and photographed after

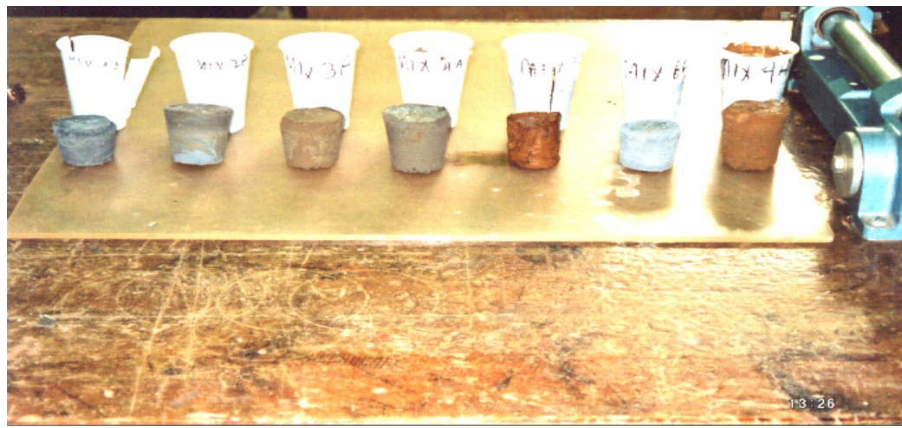


Fig. (9). Cup Mix samples.

approximately three months curing time. Fig. (10) shows the materials considered before mounting in resin. Note that there is considerable inhomogeneity in these samples, where domains of high iron content (slag dominated) contrast with the paler gypsum domains. These are agglomerations of slag particles, rather than huge pieces of slag and as such, should not present a problem in terms of occluded free lime.

The materials were dried in a desiccator for five days, prior to breaking for the photographs in Fig. (10). The samples were then divided, one portion being dry-ground by hand in an agate mortar and pestle to <50µm prior to x-ray diffraction. These samples were dry-mounted with adhesive tape and examined using Cu-Kα radiation with a nickel filter. The scan speed was 2° 2θ per minute and the spectra interpreted using proprietary Philips software referring to the JCPDS library.

The remaining samples were divided for electron microscopy: two pieces of each mixture were mounted for secondary electron imaging of the fracture surfaces and two were

vacuum-impregnated with epoxy resin prior to polishing and examination by backscattered electron and secondary x-ray methods. The compositions are shown in Table 2 along with estimates of the bulk porosity determined by image analysis.

It should be reported at this stage that the large difference in hardness between the slag and gypsum components makes these samples very difficult to polish. After resin impregnation, the samples were dry-ground on a rotary table and then polished using diamond paste dispersed in silicone oil. Fig. (11) compares the secondary electron image (left) and back-scattered electron image (right) with the topographic image (centre). This degree of polishing was considered the optimal compromise between a flat, polished surface and one degraded by “plucking” of the gypsum during finer polishing of the slag.

The use of image analysis allows a rapid estimate of the area fraction of each phase to be made. As the contrast in backscattered imaging is proportional to the electron density

Table 1. Properties of Candidate Mixes for the Trench fill Trial Site

R.G. (% by mass)	Other Materials (% by mass)	L/S	Modified Flow Table Spread (mm)	Initial & Final Setting Times (hr)	Compressive Strength (MPa)					Permeability (m/S)	pH
					1 day	3 days	7 days	28 days	90 days		
40	SSD(60)	0.19	160	1.75, 6.5	0.04	0.4	0.5	1.6	2.7	7.67×10 <sup>-9</sup>	11.3
40	SSD(60)	0.25	220	2, 9	-	-	0.6	0.65	0.75	1.86×10 <sup>-8</sup>	11.3
60	SSD(40)	0.29	190	17, 26	0.05	0.12	0.17	0.5	2.4	3.78×10 <sup>-9</sup>	11.3
30	SSD(70)	0.21	210	1.5, 6.5	0.14	0.19	0.25	0.85	2.4	7.38×10 <sup>-9</sup>	11.2
30	SSD(60) & CKD(10)	0.19	190	1, 4.5	0.17	0.31	0.78	1.15	2.8	8.21×10 <sup>-9</sup>	13.1
40	ROSA(60)	0.36	190	5, 15	0.04	0.1	0.2	0.44	0.52	-	11.3
30	ROSA(70)	0.37	210	7, 22	0.07	0.18	0.33	0.68	0.68	-	11.5
60	CKD(40)	0.41	190	74, 98	0.01	0.01	0.01	0.11	0.25	-	13
40	CKD(60)	0.45	190	3.3, 21	0.06	0.06	0.06	1.02	1.07	2.03×10 <sup>-9</sup>	13.5

Notes: SSD = Steel Slag Dust,  
 ROSA = Run Of Station Ash,  
 CKD = Cement Kiln Dust,  
 RG = Red Gypsum .



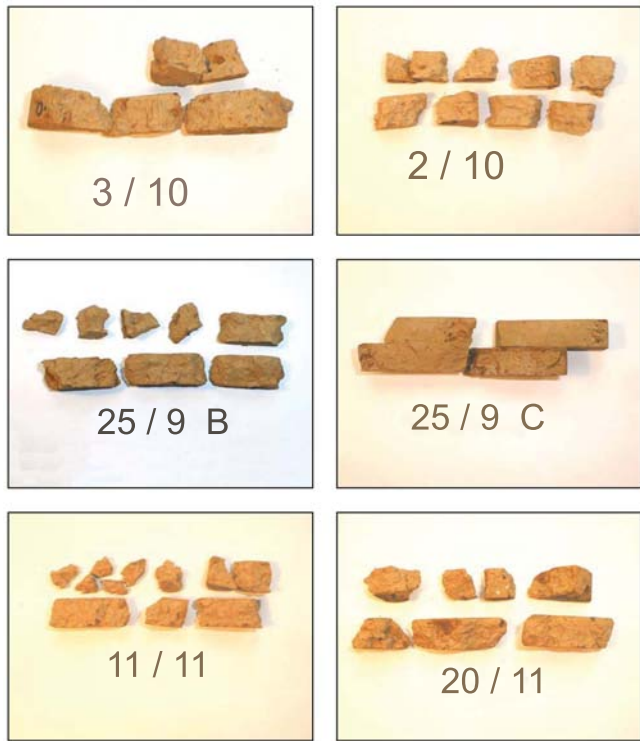


Fig. (10). Specimens of CLSM materials prior to microstructural analysis. Each field of view is 250 mm wide

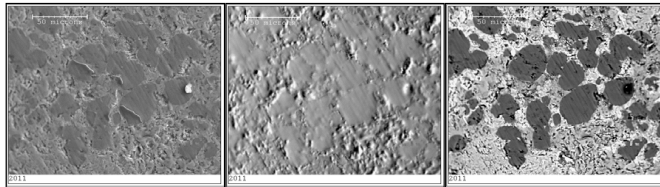


Fig. (11). Differential hardness of CLSM phases results in materials which are difficult to polish. The central micrograph shows the topography of the surface and reveals that at the stage of polishing shown here, plucking of the relatively soft gypsum has begun. Although continued polishing will remove scratches in the BOF slag, the gypsum domains will become significantly degraded

of the target, segmentation of the grey-scale of the image divides the target phases into regions of similar atomic num-

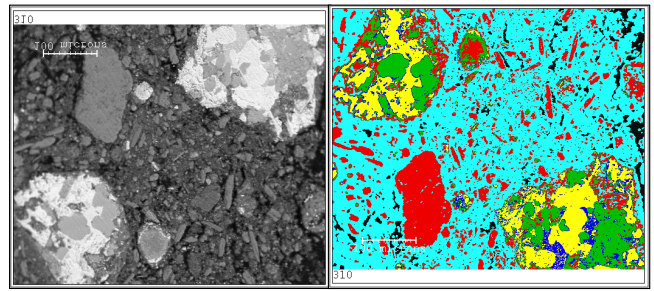


Fig. (12). Grey-scale segmentation allows false colours to be assigned to regions of equal brightness. As each region is of similar electron density, determination of the area fraction of each may be made automatically. The images are of sample 3/10 which comprises 30% RG, 60% BOS slag and 10% CKD (by mass) with a water:solids ratio of 0.19 giving a measured porosity of 4.1% (expressed as an area fraction) In this image, the gypsum is shown as pale blue and the slag phases by “warmer” colours”

ber. Assigning a false colour to each region of similar brightness illustrates the value of the technique as shown in Fig. (12). For clarity, the darkest region (porosity) is shown as black.

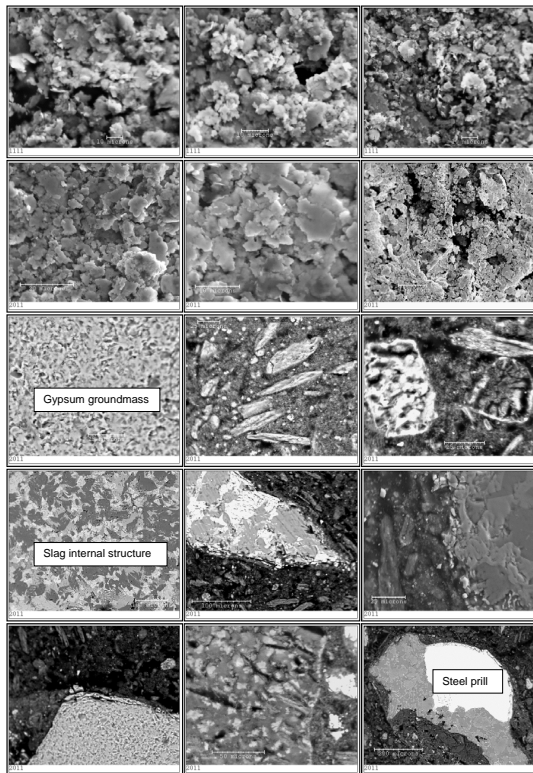
The trial mixes show a number of common features. The bulk of the image area is made up of a groundmass of gypsum (partially converted to bassanite under the vacuum of the microscope) which appears as interlocking crystals around 3 – 30µm in length. Some areas show well-defined crystal faces, yet most show foliate intergrowths suggesting the material has partially dissolved and re-precipitated on setting. The fracture surfaces illustrate the foliate nature of the gypsum crystals, whilst polished sections show the massive gypsum containing both slag grains and free porosity.

Fig. (13) shows typical features on both fractured and polished surfaces of trial mixes containing 40% by mass red gypsum and 60% BOS slag. The important features are a low degree of crack development in the pastes and relatively little connective porosity. The paste-aggregate bond seems continuous with no evidence of reaction between the two phases.

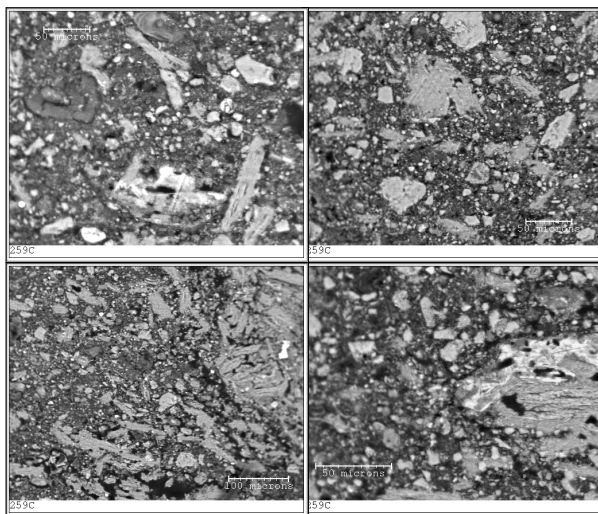
Fig. (14) shows a similar solid composition at higher water:solids ratio. The porosity has obviously increased, but more importantly, it is connective, forming large pores and disrupting the paste-aggregate bond as shown in the lower right image.

Table 2. Sample Numbers Used in Electron Microscopy and X-Ray Diffraction Experiment. Note the Porosity Estimates are from Image Analysis of the Backscattered Image Grey Scale

Sample Number	Red Gypsum % by Mass	Steel Slag % by Mass	CKD % by Mass	Liquid: Solid Ratio	Permeability (m/s)	Porosity % by Area
11/11	40	60	0	0.19	$7.67 \times 10^{-9}$	2.30
20/11	40	60	0	0.25	$1.86 \times 10^{-8}$	3.41
25/9B	60	40	0	0.29	$3.78 \times 10^{-9}$	2.75
2/10	30	70	0	0.21	$7.38 \times 10^{-9}$	3.88
3/10	30	60	10	0.19	$8.21 \times 10^{-9}$	4.07
25/9C	40	60	0	0.45	$2.03 \times 10^{-9}$	8.81

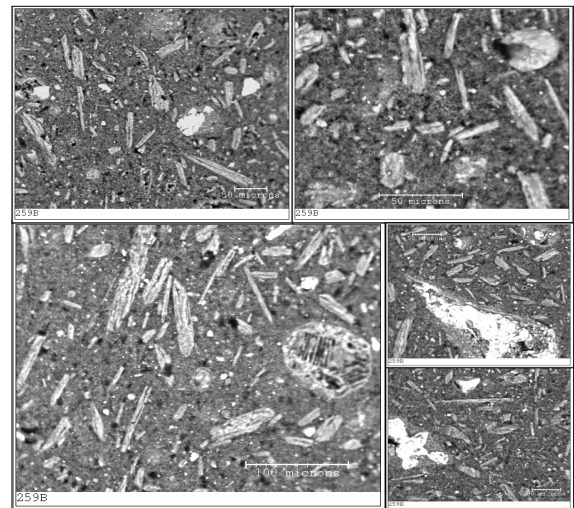


**Fig. (13).** CLSM micrographs of trial mixes containing 40% by mass red gypsum and 60% BOS slag. The upper two rows (secondary electron images) show typical fracture surfaces illustrating the foliate gypsum crystals and major pores. The lower images show backscattered images of polished sections at a range of scale. Note both iron floc inclusions from the gypsum compared with steel inclusions in the slag. The paste-aggregate bond is continuous and shows no signs of reaction between the two components.



**Fig. (14).** CLSM micrographs of trial mixes containing 40% by mass red gypsum and 60% BOS slag with a relatively high solids:liquid ratio of 0.45. backscattered electron image of polished section. Note the development of macro porosity in the lower micrographs, forming continuous regions of high porosity.

A change of solid phase composition is shown in section in Fig. (15), where the ratio has change to 60% red gypsum



**Fig. (15).** CLSM micrographs of trial mixes containing 60% by mass red gypsum and 40% BOS slag with a solids:liquid ratio of 0.19. Backscattered electron image of polished section. Note isolated nature of both the steel slag particles and the macroscopic pores.

and 40% basic oxygen slag at a water : solids ratio of 0.19. The groundmass contains discrete porosity domains and the steel slag grains are evenly distributed through it, with few points of contact between them. As with other compositions, the paste-aggregate bond seems to be intact with no evidence of reaction between the slag and gypsum or the pore solution.

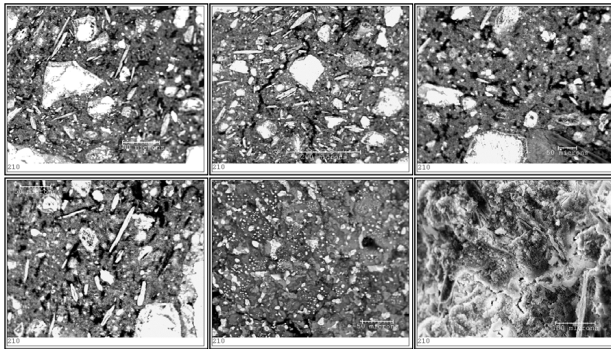
At the other compositional extreme, a trial mix was prepared with a very high steel slag content of 70% slag to 30% red gypsum by mass. This material, although not of unusually high permeability when wet, suffered from extensive drying cracking as shown in Fig. (16). The cracks are located in the gypsum-bound groundmass, but extend between grains of steel slag. The lower right (secondary electron) image shows a fracture surface intersected by several such drying cracks.

One trial mix was prepared containing 10 % cement kiln dust, 30% red gypsum and 60% steel slag by mass and this is shown in Fig. (17). The upper right image shows that the gypsum has formed a foliated crystalline mass binding both the steel slag and CKD. Uniquely, this sample shows orientated porosity (shown at the top left on a fracture surface and bottom centre in polished section). This is typical of plastic deformation and it is speculated that, as this material was the most rapid setting that the same mechanism has operated in this sample.

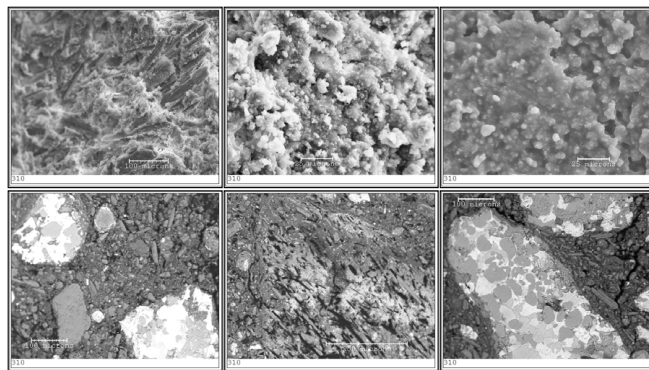
## 6. SELECTION OF MIX DESIGN FOR SITE TRIAL

Of the materials in Table 2, one was discounted as unsuitable for use as CLSM trench fill. Sample 2/10 (70 % steel slag and 30% red gypsum by mass) with the highest steel slag loading, exhibits extensive drying cracking, despite maintaining a permeability in the wet state similar to the other candidate mixes.

Of the remainder, samples 25/9C and 20/11 (40% red gypsum and 60% BOS slag), were also disregarded as the



**Fig. (16).** CLSM micrographs of trial mixes containing 30% by mass red gypsum and 70% BOS slag with a solids:liquid ratio of 0.21. Backscattered electron image of polished section except bottom right, which is a secondary electron image of a fracture plan. Note the extensive drying cracks present in this formulation.



**Fig. (17).** CLSM micrographs of trial mix containing 30% by mass red gypsum and 60% BOS slag and 10% cement kiln dust with a solids:liquid ratio of 0.19. Secondary electron images of fracture surfaces above and polished sections seen by backscattered imaging below. Note the orientated porosity features thought to be due to plastic deformation of this relatively rapid setting formulation.

high water to solids ratio (0.45 and 0.25 respectively) created sufficiently high porosity to prevent a continuous bond between the gypsum binder and the steel slag aggregate. This is also the case for the sample 25/9B (60% red gypsum and 40% BOS slag) which has 0.29 water to solids ratio.

This left two possible formulations whose microstructures show no features, which may raise concern about the materials’ future performance which are shown in Table 3.

Of these two, the first was selected for use in the field trials owing to its physical properties as explained in section 3 and having less components in the mix than the other. It is expected that any of the above formulations would be suitable for use in CLSM applications.

**7. SITE TRIAL**

**7.1. Mix Quantities**

The total quantities of materials used in the site trial are shown in Table (4): The calculated yield was 7.4 m<sup>3</sup>

**7.2. Mixing and Placing Procedure**

Mixing was carried out in a truckmixer which was filled by using a conveyor belt. Both the slag and the gypsum were supplied in sling bags containing approximately one tonne each. These were discharged onto a conveyor. (see Fig. 19).

The pour was placed in a trench which was approximately 5 meters long by 1 meter wide by 2 meters deep. The surrounding soil appeared to be clay. The sides of the trench were not supported. The mix filled the trench to within 0.5 meters from the top.

The detailed procedure was as follows:

- 1500 litres of water put in mixer
- 6 tonnes of BOS slag added
- 5.3 tonnes of red gypsum added
- 2 tonnes of slag added
- 500 litres of water added
- visual check on workability. Observed to be inadequate.
- 400 litres of water added
- mixed at full speed for 5 minutes
- moved to excavated trench
- approx 0.5 m<sup>3</sup> discharged into trench

**Table 3.**

Sample Number	Red Gypsum % by Mass	Steel Slag % by Mass	C.K.D. % by Mass	Liquid: Solid Ratio
11/11	40	60	0	0.19
3/10	30	60	10	0.19

**Table 4.**

Material	Mass (Tonnes)
Red Gypsum	5.3
BOS weathered slag	8
Water	2.4



**Fig. (18).** BOS Slag discharge to conveyor.

- first sample taken.
- mixed at full speed for 5 minutes.
- approx. 3.5 m<sup>3</sup> discharged in trench
- second sample taken
- approx. 3.5 m<sup>3</sup> discharged in trench
- third sample taken
- Final discharge to trench.

### 7.3. Site Observations

#### *Observations from Samples Taken*

The flow of the first sample was measured to be 545 mm (very close to 560mm target). Some unmixed lumps of red gypsum were observed. These were extracted using a 5mm sieve. The quantity was observed to decrease from the first to the second sample but increased again for the third sample. Three 50mm and one 100mm cubes were taken from each sample. Initial set was 1.5 hours from discharge and final set approximately 7 hours.

#### *Observations from Discharge*

The material was observed to flow freely in the trench (see Fig. 19). A small number of agglomerated lumps up to 150mm in size were observed. The material was self-compacting and air could be seen escaping (venting to surface) indicating that vibration was not necessary.

The trench into which the mix was poured had partly filled up with rain water since it was pumped out earlier in the morning. The first 0.5m<sup>3</sup> discharge into the trench initially appeared to mix with the water in the trench, but very rapidly “clear” water separated out on top of the mix. The mix did not appear to be adversely affected by being poured into standing water. It is suggested that this degree of cohesion is superior to the performance of foamed concrete where some grout loss might be observed in these circumstances.

The surface of the pour remained reasonably flat along the trench even though it was only poured from one end. It was difficult to see the exact slope due to the free water on the surface but the final slope was minimal (see Fig. 19).



**Fig. (19).** CLSM Discharge to trench.

### 7.4. Laboratory Testing of Recovered Samples

The compressive strength results for the 50 mm cubes were 0.15 MPa at one day of age and 0.7 MPa at 7 days. The results are the average of 3 cubes which were taken from top, middle and bottom of the truck mixer load.

The 28 days result of the average of 3 cubes (50 mm) which were taken from top, middle and bottom of the truck mixer load was 1.8 MPa cured in moist environment but the 100 mm cubes which were cured under water after demoulding disintegrated. The insides of the cubes were green in colour indicating that the iron had not oxidised. The small cubes tested at 1 day were also submerged under water but did not fail. The failure of the 100mm cube was not considered to prevent the use of this material for trench or mine backfill because expansive cracking would be constrained in these applications.

### 7.5. Inspection of Completed Pour

The pour was inspected two weeks after pouring. Conditions were dry but rain on previous days resulted in standing water to a depth of at least 100mm over the surface of the pour (Fig. 20).

The presence of the water indicated that there were no major cracks in the pour. The photograph in Fig. (20) was taken from the end of the trench where the material was discharged. From the depth of water where one of the authors is standing it is clear that the slope on the upper surface of the finished pour was minimal (approximately 50mm). It was very apparent that the mix had successfully filled the trench



Fig. (20). "inspecting" the CLSM pour.

and, if surfaced, would have formed a good reinstatement to a road. The following properties have been demonstrated:

- Excellent flow
- Self consolidation
- Set properties to equal a good compacted soil (which would normally be used for backfill).

## 8. CONCLUSIONS

This paper presents a review of the properties of CLSM and a detailed analysis of the RG, CKD and BOS that were used to make trial samples of it. Electron microscope and X-ray images of the constituent materials and the CLSM are presented. These images are discussed and it is concluded that the constituent materials are unsuitable for use in structural concrete but well suited for use in CLSM. An optimum CLSM mixture was developed and the following observations made from it:

- Waste gypsum may be used with other secondary materials to make Controlled Low Strength Materials.
- The best material to use with the gypsum is Basic Oxygen Slag. No other components are required in the mix except water.

- The mix showed excellent properties for placing and was particularly resistant to segregation in the presence of water.
- A site trial has shown that the mix is suitable for the application but the disintegration of a cube during curing shows that it may not be suitable for other applications.

## ACKNOWLEDGEMENT

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## ABBREVIATIONS

CLSM = Controlled Low Strength Material

BOS Basic = Oxygen Slag

RG = Red Gypsum

CKD = Cement Kiln Dust

## REFERENCES

- [1] A. Katz and K. Kolver, "Utilization of industrial by-products for the production of CLSM", *Waste Management*, vol. 24(5), pp. 501-512, 2004.
- [2] R. A. Taha, A. S. Alnuaimia and K. S. Al-Jabri, "Evaluation of controlled low strength materials containing industrial by-products", *Building and Environment*, vol. 24(9), pp. 3366-3372, 2007.
- [3] A. Smith, "Controlled low-strength material", *Aberdeen's Concrete Construction*, vol. 36(5), pp. 389-398, 1991.
- [4] R. W. Sullivan, "Boston Harbor Tunnel Project Utilizes CLSM", *Concrete International*, vol. 19(5), pp. 40-43, 1997.
- [5] ACI Committee 229, Manual of concrete practice, controlled Low-Strength Materials, Part 2, pp. 15, 2003.
- [6] W. S. Adaska, "Controlled low-strength Materials", SP-150, American Concrete Institute, Farmington Hills: Mich., pp. 113, 1994.
- [7] BS EN 12350-5, "British standard, testing fresh concrete: Part 5", Flow table test 2000.
- [8] R. J. Hoopes, "Engineering properties of Air- Modified Controlled Low Strength Material", The design and application of CLSM (flowable fill), ASTM STP 1331, A.K. Howard and J. L. Hitch, Eds., ASTM, 1997.
- [9] T. R. Naik, B. W. Ramme and H. J. Kolbeck, "Filling abandoned underground facilities with CLSM fly ash slurry", *Concrete International*, vol. 12(7), pp. 19-25, 1990.
- [10] R. L. Larsen, "Sound uses of CLSM in the environment", *Concrete International*, vol.12(7), pp. 26-29, 1990.
- [11] E. Nouaiser, *Use of Waste Materials and By-Products for Civil Engineering Works*, MSc Thesis: Coventry University, Sept 2003.
- [12] H. J. D. Faria, *Development of Controlled Low Strength Materials Using Mineral Wastes*, MSc Thesis: Coventry University, Sept. 2003.

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