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Investigation of Engineering and Microstructural Characteristics of Developed Concrete through fly ash & Hazardous Solid Waste

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Abstract— The use of fly ash & hazardous solid waste to concrete production is environmental friendly because it contributes to reducing the consumption of natural resources, the pollution concrete production generates and the power it consumes. This paper presents study of fly ash & hazardous solid waste (sludge) as raw material for use in concrete. The concrete specimens were prepared according M15 grade (1:2:4) of concrete. Concrete samples were made with different percentage of fly ash & Sludge additions (relative to the cement weight) and engineering and microstructural properties were investigated, i.e., compressive strength and XRD. Tests are carried out after 7, 28 and 90 cured of the specimens. The mean compressive strength required at a specific age, usually 28 days, determines the nominal water/cement ratio of the mix. Compressive strength of developed concrete for 7 days cured samples 2.2 – 6.5 MPa, 28 days cured 3.5 – 9.0 MPa, 90 days cured 4.6 – 10.4 MPa respectively. CSH, portlandite, ettringite, CH, Al2O3 etc. was clearly detected as a predominant crystalline phase in developed concrete matrix. Factors affecting unconfined compressive strength of developed concrete are: by-product (fly ash), waste (sludge), waste/binder ratio, water/cement ratio, curing days. Reuse of heavy metal containing sludge, as alternative concrete material could be a promising alternative for the management of waste sludge.

Keywords— *Heavy Metals, Compressive Strength, By-Product, XRD, crystalline structure*

I. INTRODUCTION

Construction industry has taken considerable strides forward over the last two or three decades with regard to many materials. The use of various types of waste materials as additives in the production of cement and concrete has received substantial attention during recent years. Paper present potential usages of fly ash and sludge in concrete were researched. The construction industry is also characterized by a high potential to use waste in construction materials. Thus, many research activities have been carried out with the purpose of using waste as a raw construct material. In case of the usage of these by-products and solid wastes in concrete as raw material replacement, each of them would be a raw material having economical value [1,2]. These materials are compacted and vibrated through stabilization/solidification (S/S) in various matrices and characterized by a porous structure with good mechanical properties. Therefore, they are generally considered as “concrete” materials exposed to the environment by surface contact [3,4,5].

The presence of some mineral admixtures such as natural pozzolan, fly ash or granulated blastfurnace slag in the cement can modify the kinetic of hydration, reduce the heat evolution and produce additional CSH hydrates [6,2]. Typical modern Portland cement achieves about two-thirds hydration in 28 days [7]. The main chemical that is considered in hydrated cement is colloidal calcium silicate hydrogel, known as CSH, and this gel product is formed at the cement particle surfaces [8]. CSH has important implications for the mechanisms of fixation during solidification [9] and is principally responsible for strength development [10]. Concreting processes are based on hydraulic reactions of hydraulic cements or pozzolanic reactions between ordinary portland cement (OPC) and pozzolanic materials such as fly ash, sand, aggregate and hazardous solid waste [4,5]. The development of hydration products provides an interlocking framework to physically encapsulate waste particles and is also responsible for the strength development of the monolithic solid [3,4,5,11,12].

II. MATERIALS

1. Cement: Ordinary Portland (43 grade) OPC was used. It was tested as per Indian Standard Specifications IS: 8112-1989 [13]. Chemical composition of cement is presented in Table 1.

2. Fly Ash

Class F fly ash obtained from cement industry was used in this investigation. Chemical composition of the fly ash was determined as per IS 3812:1981 [14] and the results are given in Table 2.

3. Fine Aggregate

Natural sand with a 4.75-mm maximum size was used as fine aggregate. It was tested as per Indian Standard Specifications IS: 383-1970 [15].

4. Coarse Aggregate

Coarse aggregate used in this study was 10-mm down nominal size. It was tested as per Indian Standard Specifications IS: 383-1970 [15].

5. Sludge

A grab composite sample of sludge was collected from the sludge drying bed of a commercial effluent treatment plant. The physicochemical properties of sludge are given in Table 3 [16].

III. METHODOLOGY

Preparation, Casting and Testing of Specimens:

The S/S products were prepared through mixing different ratios of sludge with OPC, sand aggregate and water (Table 4). The mixture was compacted, vibrated, and molded in a cubical mold of dimension 10x10x10 cm. The concrete specimens were prepared according M15 grade of concrete. The common method of expressing the proportions of ingredients of a concrete mix is in the terms of parts or ratios of cement, fine and coarse aggregates. For e.g., a concrete mix of proportions 1:2:4 means that cement, fine and coarse aggregate are in the ratio 1:2:4 or the mix contains one part of cement, two parts of fine aggregate and four parts of coarse aggregate [11]. The proportions are either by volume or by mass. The water/cement ratio is expressed in mass (Figure 1). The cubes were unmolded after 24h and specimens were cured for 7, 28, 90 days. Tests are carried out after 7, 28 and 90 cured of the specimens. Unconfined Compressive Strength obtained through Universal Testing Machine IS9013:1978 [17]. Microstructural analysis XRD carried out through X-Ray diffraction.

IV. RESULTS AND DISCUSSION

UCS is one of the most important properties of concrete and it influences hardened durability and leachability properties of concrete. The mean compressive strength required at a specific age, usually 28 days, determines the nominal water/cement ratio of the mix. According to Abraham's law the strength of fully compacted concrete is inversely proportional to the water/cement ratio [11, 18, 19].

$$fc = \frac{k_1}{k_2^{w/c}}$$

Where, w/c represents the water/cement ratio of the concrete mixture and k1 and k2 are empirical constants. The specified W/C ratio is the perfect medium between the maximum possible strength of the concrete and the necessary minimum workability requirements [9, 20].

The compressive strength of the concrete was determined according to the IS 9013:1978 at 7, 28, and 90 days [17]. Triplicate cubes were used for each testing age and the average values were calculated. Compatibility of different sludge was observed through engineering property.

1. Compressive Strength

The effect of replacement of OPC with three different percentages of fly ash on the compressive strength of concrete is given in figure 2. It is clear from table 4 that the replacement of OPC with 25%, 50% and 75% of fly ash reduces the compressive strength of concrete: at 7 days 6.17%, 38.27%, 54.3%, at 28 days 7.6%, 10.48%, 38.1% and at 90 days 11.46%, 20.38%, 25.47% respectively. A marginal compressive strength increase was observed in pure concrete than developed composition. The rate of strength gain is initially faster and get slow with age [5]. It is also reported that a significant strength was gained after 28 days of curing. Pure and cement/fly ash mixed Sample found at utmost strength. Result of developed concrete samples was found to be close to reference sample [21].

The result of addition of sludge on the compressive strength of fly ash concrete is shown in figure 2. Compressive strength of developed concrete for 7 days cured samples 2.2 – 6.5 MPa, 28 days cured 3.5 – 9.0 MPa, 90 days cured 4.6 – 10.4 MPa respectively. It is clear from figure 2 that for a particular percentage of fly ash, there was a decrease in the compressive strength of concrete. The percentage of sludge increased from 0.75 – 0.5% it does not attain the UCS. Reduction in compressive strength was between 3.7 – 13.5 MPa for 25 - 75% fly ash content. However, in this case of adequate amount of sludge was replaced with sand, was not significant decrease UCS [2,11,22]. The compressive strength of concrete depends on continuous hydration. It is customary to assume that the 28 days strength as the full strength. The increase of strength of 28 days used to get immersed with the factor of safety, but concrete became more strengthen after 28 days of curing.

2. Microstructure Analysis

The X-ray diffraction graph indicates the presence of crystalline structure in the samples. The XRD pattern for pure cement, fly ash and sludge has given in figure 3, 4, 5 respectively. The expected crystalline hydration products are clearly evident. Calcium hydroxide (CH) was found in appreciable amount at 28 days, and remained in the matrix as a good crystalline product throughout the period of experiment. Crystal structure present in the raw materials is also play a major role in development of crystalline products during hydration and curing [23].

XRD graph of developed concrete has given in figure 6 (a-j). Calcium silica hydrated gel (CSH) was clearly detected as a predominant crystalline phase in developed concrete matrix. The high peaks frequency shows the presence of CSH, portlandite, ettringite and low peaks in the graph shows the presence of CH, Al_2O_3 etc. The diffraction peaks situated between 10 - 30 two theta (2θ) corresponds the hydrogenate composition termed ettringite, CSH, portlandite etc. The amount of unhydrated C_3S decreases with increasing time, as expected. Ettringite was found to be increased substantially with increasing curing days. All the sludge mixed Samples have very similar hydration products, but in different quantity.

The X-ray diffraction spectra shows the presence of large quantities of CSH that could be indicative of a pozzolanic reaction. The CSH gel phase is thought to be highly reactive and is most probably produced by copolymerization of individual calcium and silica species [9]. Ettringite is a secondary hydration product commonly found in concrete. Crystalline structure helps in the development of unconfined compressive strength. The developed concrete have the sufficient amount of Crystalline structures and give the strength accordingly. Bothe and Brown mentioned that the hydration product ettringite was formed in high amount [10, 24] due to the formation of hydrogenate phases where most of the heavy metal trapped in concrete.

Factors Affecting Unconfined Compressive Strength of Developed Concrete

1. By-Product (Fly Ash)

Unconfined compressive strength of Samples has improved due to pozzolan present in fly ash. Strength development of cement paste is strongly affected by the volume of CSH formed during cement hydration [7, 25]. The 28 day compressive strengths were measured for the composition made from OPC/fly ash & OPC/fly ash/sludge at various ratio of water/solid [21]. The results of compressive strength measurement are given in figure 2. The replacement of fly ash 15–25 wt.% gives better strength as compare to 50 –75 wt.%. If the replacement level was higher compressive strength found to be lower. Waste/solid ratio did not significantly influence the strength development. Lower Water/Solid ratio improved the strength of fly ash mixtures [25]. Samples made from fly ash produce compressive strength close to cement paste. Effective surface area and pozzolanicity of the mineral admixture reduce hydration rate and help to produce additional crystalline structure like CSH, ettrignite etc. [25,26]. Generally, later age strengths (beyond 28 days) are expected greater for pozzolanic mixes as compared to plain cement control at given water/binder ratio [8].

2. Waste (Sludge)

Figure 2 shows the strength development of concrete containing sludge in different composition. It is observed that the rate of strength development of the solidified sludge decreased with increasing concentration of the sludge [7,8]. This reduction in strength was due to the resolubilization of metal nitrates from sludge in the highly acidic environment [27]. In addition, strength reduction of the solidified wastes could be resulted due to replacement of fly ash with a binder and the sludge was added at an increasing amount. It can be hypothesized that the lower pH solution (pH 2) compared to that of higher solution (pH 14) results in extensive resolubilization of several metal nitrates from the sludge. It was found that metals could react with cement paste and affect the hardening and strength development during the early stage of cement hydration.

3. Waste/Binder Ratio

Waste/Binder (W/B) ratio integrated with Water/Cement ratio (W/C) and Water/Solid (W/S) have represented in figure 1. W/S ratio is inversely proportional to compressive strength and directly proportional to W/B ratio. Water/Solid (W/S) ratio was increased from 0.088 – 0.12 for developed concrete. Unit weight of sludge is lower than the unit weight of fine aggregate. Therefore, this enables to produce lighter concrete as the ratio of W/B increases [7]. Although an increase in W/B replacement ratio leads to a decrease in the compression strength level. The high level of water/cement ratio (0.4 – 0.9) decreases compression strength and it also increases the level of the Air pores.

4. Water/Cement Ratio

Cement requires about 25% by weight of water for complete hydration, while the less workable concrete usually requires at least 35%. Water that remains free gradually evaporates leaving pores, which weaken the mechanical strength of concrete. Concrete with high dosage and large grain size of aggregate have good workability even though their W/C ratio is lower [8, 10]. W/C ratio Increases with increasing in W/B ratio. It was increased from 0.4 – 0.9.

5. Curing Days

The compressive strength of concrete depends on continuous hydration. Strength gain rate was faster to start with and the rate gets reduced with curing age. It is customary to assume the 28 days strength as the full strength. The increases of strength 28 days used to get immersed with the factor of safety. Strength is increases with increase in curing days from 7 to 90. Strength increase is also depends upon the grade of cement fineness of grinding. Method of curing and temperature is also affect strength of concrete [1,8].

V. CONCLUSION

In this study, it was found to be feasible to use heavy metal containing sludge and fly ash as alternative concrete materials. At low levels of sludge replacement, the heavy metals carried by the sludge would have the positive contribution to the phase formation in concrete. However, if too high of heavy metal concentration (>1.5%) was present in the concrete mix, an auspicious effect on concrete was observed. Reuse of heavy metal containing sludge, as alternative concrete material could be a promising alternative for the management of waste sludges.

The paper has demonstrated the suitability of fly ash as a supplementary cementing material and sludge for use in concrete production. The following conclusions may be drawn from this study:

- With the replacement of fly ash up to 50%, the compressive strength of the concrete was significantly intensified like pure concrete at 28 days. The addition of sludge results slightly decrease in the concrete's compressive strength. Adequate hydration increases hydrogenate phases in the concrete structure and it improves compressive strength.
- The UCS decreases with increase of sludge and Waste/Binder ratio (0.5–4.0) from 15.7 – 2.2 MPa after 7, 28 and 90 days of curing. There is an increasing trend in UCS from 7, 28, and 90 days of curing in all samples.
- It is observed that a period of 28 days may not be sufficient for the development of full marginal potential strength for the fly ash and sludge as concrete. Developed concrete gains strength beyond 28 days also.
- Hydrogenated crystalline phases have been identified through XRD analysis. The XRD graphs showed that the development of structure was delayed due the pozzolanic contribution of fly ash, which resulted hydrogenate phases. The X-ray diffraction spectra show large quantities of crystalline structure in the form of CSH gel, portlandite, ettringite, CH and some Al_2O_3 etc. XRD spectra resulting improved fixation of heavy metals in the matrix of samples. It is also observed that developed crystalline structure helps in the strength gain.
- Addition of waste has changed the morphology of the concrete significantly indicating the formation of a denser structure, which improves the concrete strength.

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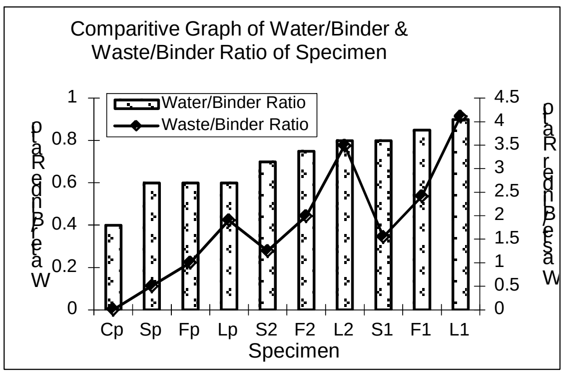

Figure 1: Comparative Graph of Waste/Binder Ratio & Water/Cement Ratio of the Specimen

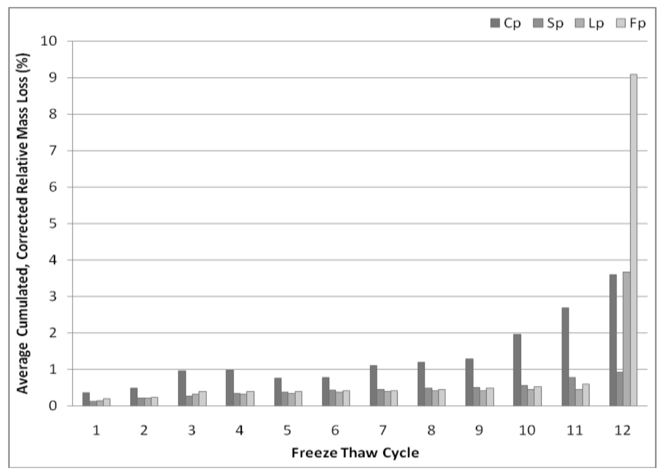

Figure 5.1 (a) : Freezing - Thawing Durability of Developed Concrete

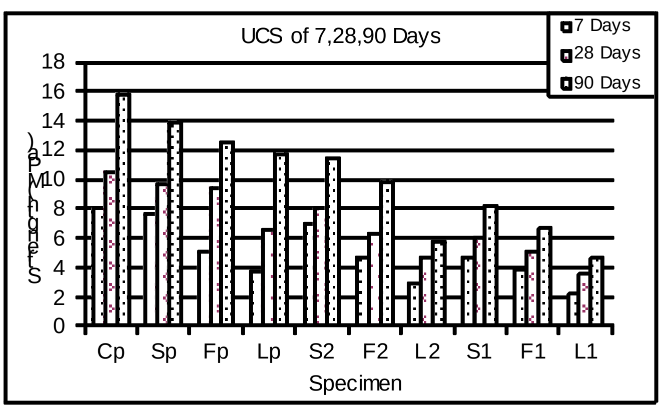

Figure 2: Comparative Graph of the Unconfined Compressive Strength at 7, 28 and 90 Days

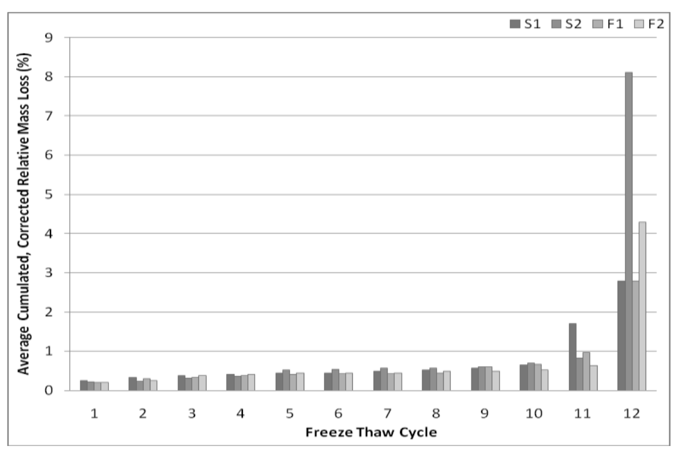

Figure 5.1 (b) : Freezing - Thawing Durability of Developed Concrete

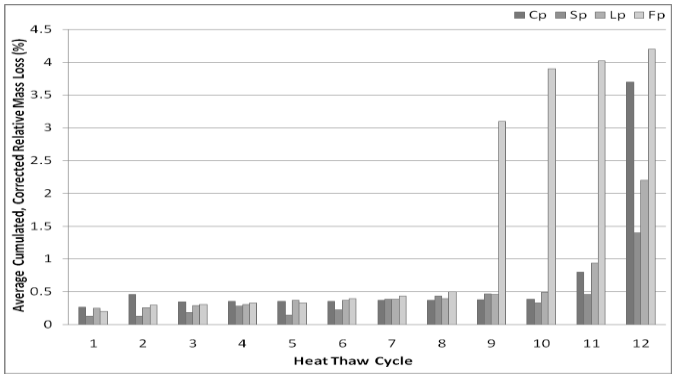

Figure 5.2 (a) : Heating - Thawing Durability of Developed Concrete

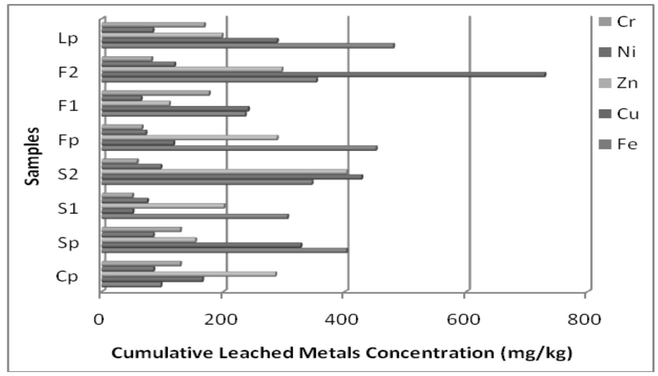

Figure 5.4 (b): Metals Leached Concentration from Developed Concrete after Heat-Thaw Cycle

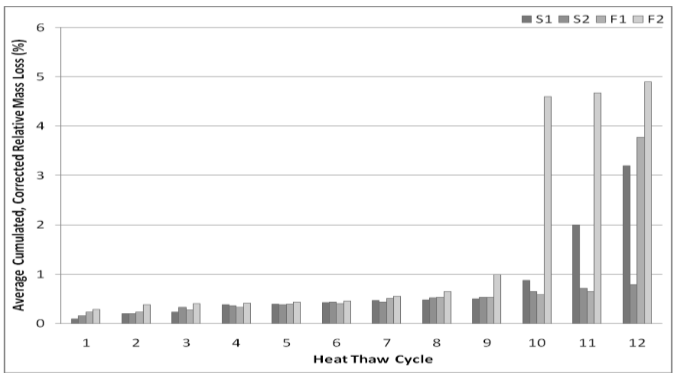

Figure 5.2 (b) : Heating - Thawing Durability of Developed Concrete

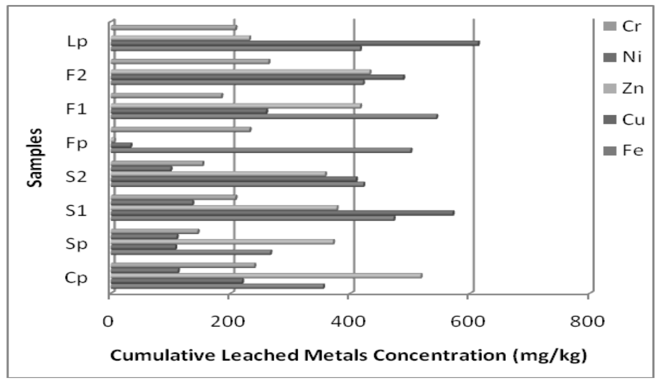

Figure 5.4 (c): Metals Leached Concentration from Developed Concrete after Freeze-Thaw Cycle

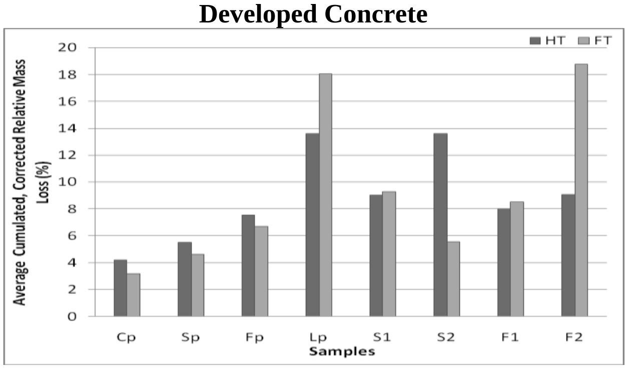

Figure 5.3: Average Cumulative Durability of Developed Concrete Samples

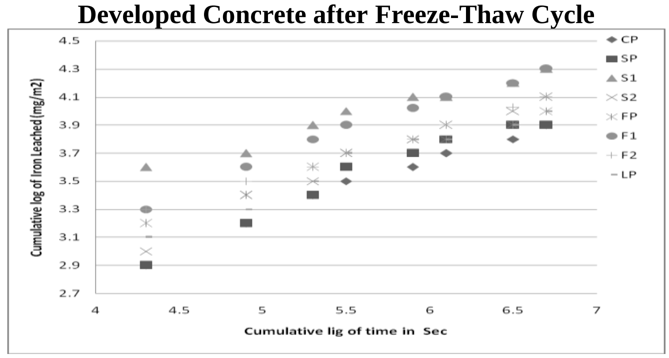

Figure 6 (a) : Cumulative Iron Leaching From Concrete Samples Over 64 Days For 28 Days Of Curing

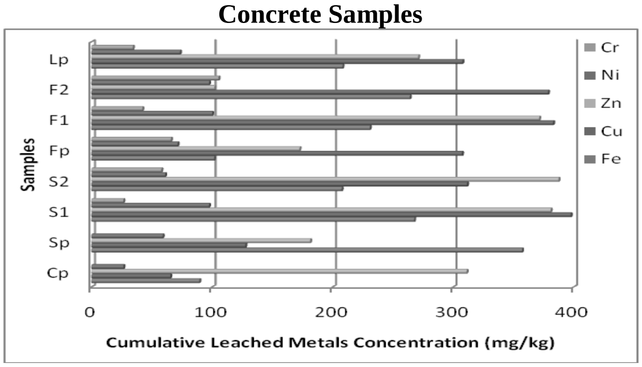

Figure 5.4 (a): Metals Leached Concentration from Developed Concrete at Ambient Temprature

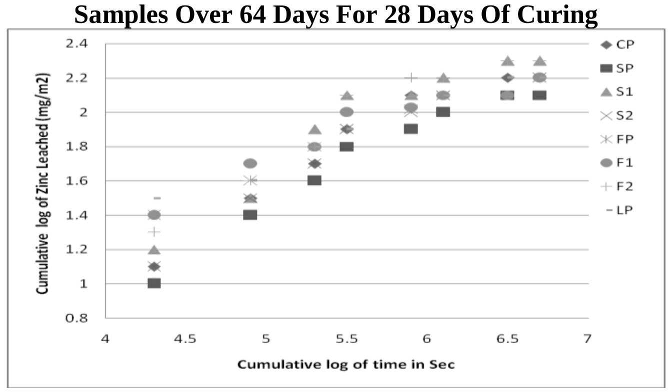

Figure 6 (b) : Cumulative Zinc Leaching From Concrete Samples Over 64 Days For 28 Days Of Curing

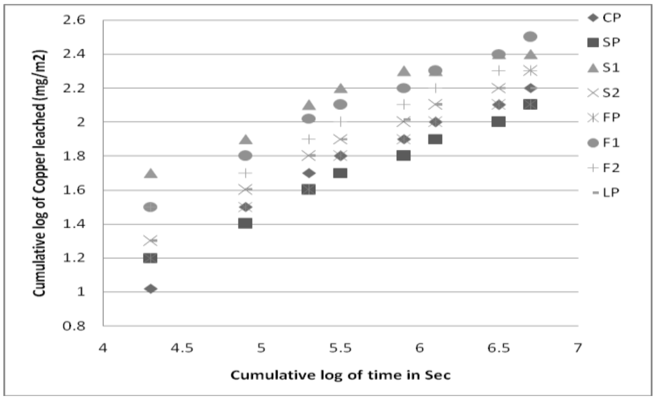

Figure 6 (c) : Cumulative Copper Leaching From Concrete Samples Over 64 Days For 28 Days Of Curing

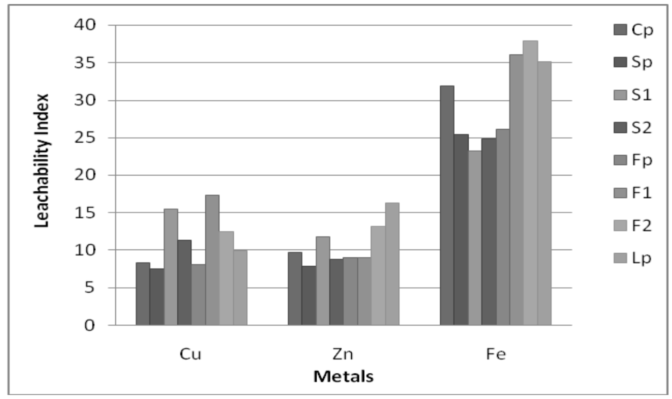

Figure 6 (d) : Leachability Indexing of Heavy Metals

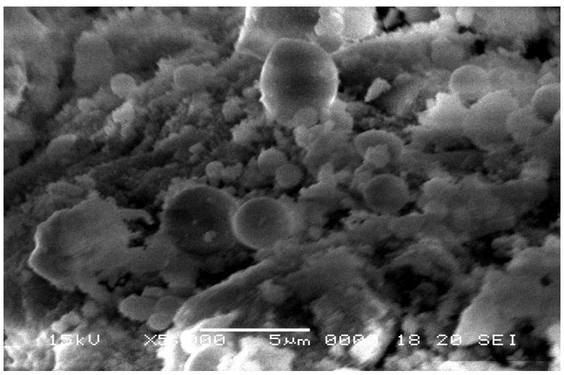

Figure 4.8: Micrograph of pure sludge

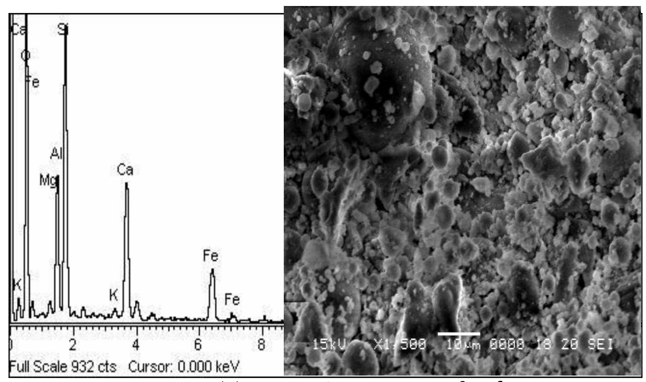

Figure 4.9 (c): EDX & Micrograph of S_1

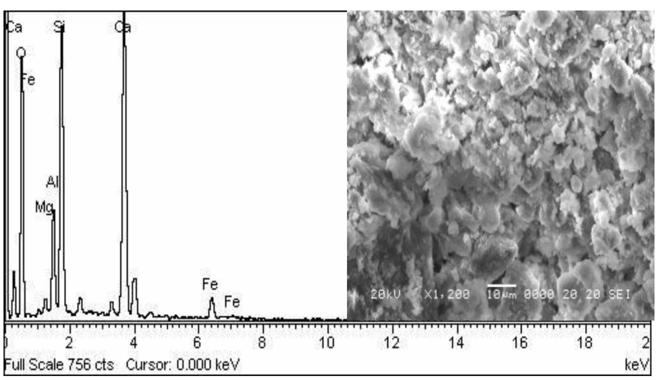

Figure 4.9 (a): EDX & Micrograph of Cp

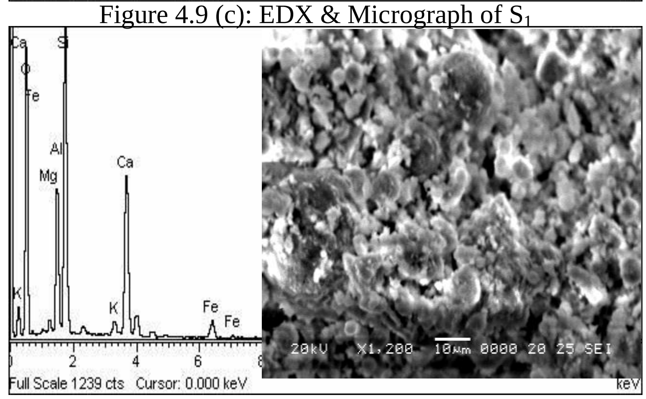

Figure 4.9 (d): EDX & Micrograph of S_2

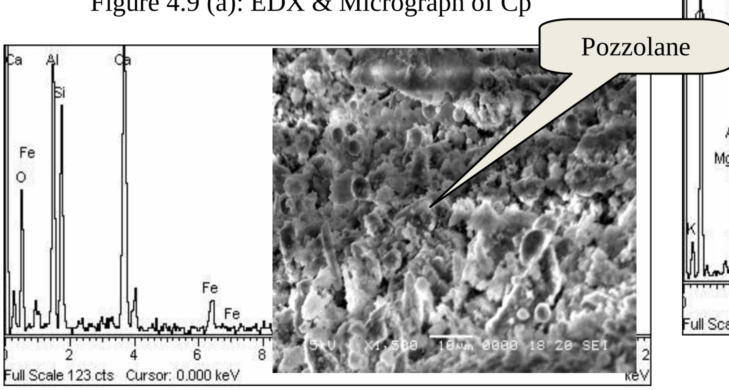

Figure 4.9 (b): EDX & Micrograph of Sp

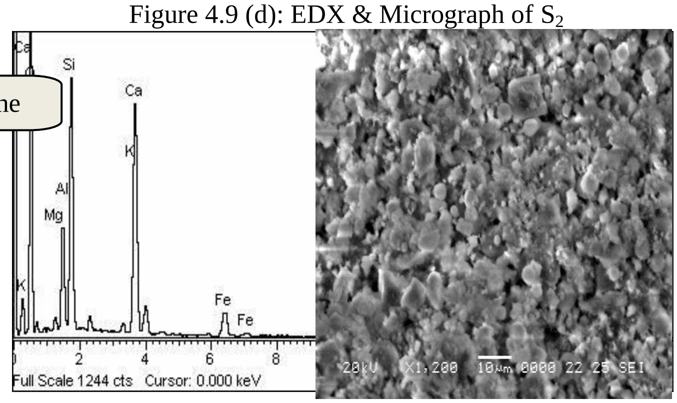

Figure 4.9 (e): EDX & Micrograph of Fp

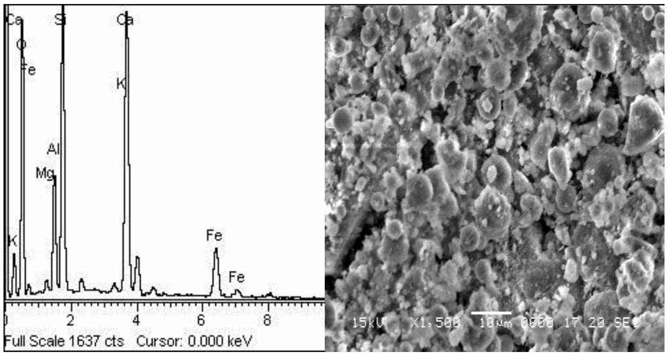

Figure 4.9 (f) : EDX & Micrograph of F_1

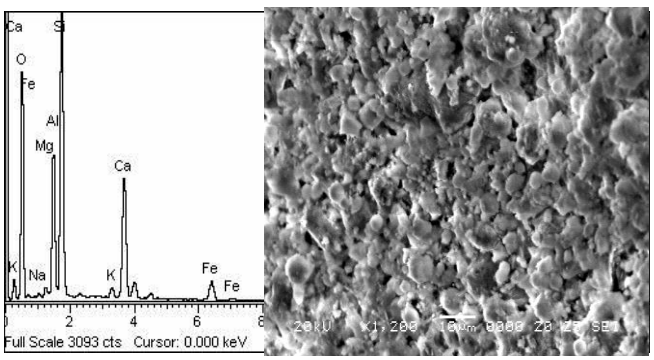

Figure 4.9 (g): EDX & Micrograph of F_2

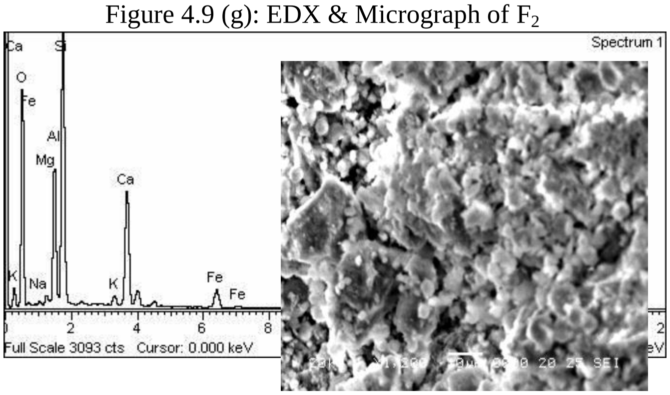

Figure 4.9 (h): EDX & Micrograph of Lp

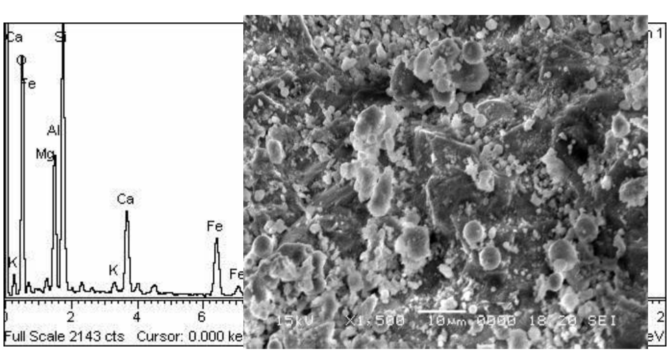

Figure 4.9 (i): EDX & Micrograph of L_1

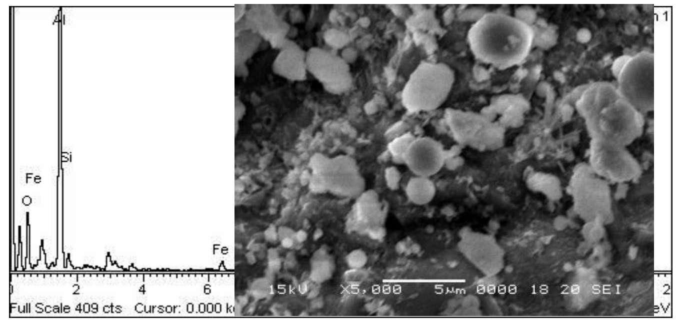

Figure 4.9 (j): EDX & Micrograph of L_2

TABLE 1: CHEMICAL COMPOSITION OF CEMENT

Oxides	Cement Concentration (% by wt)
CaO	63.4
SiO_2	16.6
Al_2O_3	5.2
Fe_2O_3	4.3
MgO	2.4
SO_3	1.9
$SiO_2 + Al_2O_3 + Fe_2O_3$	26.1

Table 2: Chemical composition of fly ash

Oxides	Fly ash Concentration (% by wt)
CaO	2.75
SiO_2	55.92
Al_2O_3	23.91
Fe_2O_3	18.27
MgO	0.60
SO_3	0.21
$SiO_2 + Al_2O_3 + Fe_2O_3$	98.1

Table 3: Physico-Chemical Characteristics Of Sludge

I. PARAMETERS	Unit	Values
pH		7.4
Conductivity	(μS/cm)	5.5
Moisture content	(%)	12
Specific gravity	(g/cm3)	1.5
Porosity	(%)	24.25
Water holding capacity	(%)	32

II. DRY DENSITY	(g/cm3)	1.1
III. BULK DENSITY	(g/cm3)	0.97
IV. LOSS ON IGNITION	(%)	36
Cl	(mg/kg)	2490.0 ± 50
Na	(mg/kg)	342 ± 10
K	(mg/kg)	456 ± 10
SO_4	(mg/kg)	2150 ± 150
Iron (Fe)	(mg/kg)	36221.22
Zinc (Zn)	(mg/kg)	10615.25
Copper (Cu)	(mg/kg)	9615.26
Chromium (Cr)	(mg/kg)	5315.71
Nickel (Ni)	(mg/kg)	2766.3
Manganese (Mn)	(mg/kg)	1210.58
Lead (Pb)	(mg/kg)	73.50

Table 4: Different Combinations of Concrete Sample

<table>
<tr><th>Specimen Code</th><th>Cement : Fly ash (Ratio) 1:</th><th>Sand : Sludge (Ratio) 2:</th><th>Aggregate (Ratio) 4</th></tr>
<tr><td>Cp</td><td>1:0</td><td rowspan="2">2:0</td><td rowspan="10">4</td></tr>
<tr><td>Sp</td><td rowspan="3">0.75:0.25</td></tr>
<tr><td>S_1</td><td>1.25:0.75</td></tr>
<tr><td>S_2</td><td>1.5:0.5</td></tr>
<tr><td>Fp</td><td rowspan="3">0.5:0.5</td><td>2:0</td></tr>
<tr><td>F_1</td><td>1.25:0.75</td></tr>
<tr><td>F_2</td><td>1.5:0.5</td></tr>
<tr><td>Lp</td><td rowspan="3">0.25:0.75</td><td>2:0</td></tr>
<tr><td>L_1</td><td>1.25:0.75</td></tr>
<tr><td>L_2</td><td>1.5:0.5</td></tr>
</table>

