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A Comparative Study of Physicochemical and Mineralogical Properties of LD Slag from Some Selected Steel Plants in India

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ABSTRACT

The scope of this study is to compile several analytical techniques in order to carry out physico-chemical and mineralogical properties of Linz-Donawitz (LD) slag. The physical properties such as bulk density, specific gravity, particle size distribution, porosity, permeability and water holding capacity etc., have been determined. The pH and electrical conductivity of the samples were very high, indicating high percentage of lime and ionic form of various salts. The specific gravity and bulk density were found high due to fine particles. Particle size distribution results showed that the LD slag samples were well graded. The EDS X-ray showed that major elemental compositions of LD slag samples were O and Ca by weight. The XRD analysis showed that the pattern of LD slag samples were very complex, with several overlapping peaks and the presence of free MgO and CaO. From the FE-SEM analysis it was observed that LD slag particles had sub angular to angular shapes and very rough surface texture with distinct crystal structures. Fourier transform infrared spectroscopy (FTIR) analysis revealed that portlandite ($Ca(OH)_2$) and periclase (MgO) are the dominant phases in LD slag. The XRF analysis showed that the major components of the LD slags are CaO, FeO and SiO_2.

Key words: Steel slag, FTIR, FE-SEM/EDS, XRD and XRF

INTRODUCTION

Life without steel is impossible in our modern way of living. The material is an essential ingredient for making our homes, cars, structures etc. For steel production, integrated steel plants are using the raw materials like iron ore, fluxes, fuel, power, air and water. Depending upon the quality of raw materials and its metallurgical properties, considerable solid wastes are generated during the steel production. These solid wastes are Linz-Donawitz (LD) slag, blast furnace slag, coke breeze, tar sludge, etc. Open dumping of these wastes creates many environmental threats. For economic and environmental advantage, reducing, recycling and reusing of waste have become necessary today due to shortage of dumping space. Due to increasing of environmental awareness the disposal of steel slag wastes without harming the environment has become a prime concern for the industry. Use of steel slag in civil engineering applications can utilize the waste and reduce the usage of native natural resources (Singh *et al*., 2013).

A better understanding of the properties of steel slag is required for its large volume utilization in civil engineering applications. The LD converter steel slags are industrial byproducts resulting

from a steel making process in oxygen converters (Linz-Donawitz process). In India, the generation of steel melting slag is over 4-4.5 Mt per annum. The amount of steel slags from different steel industries is 150-200 kg t^{-1} of steel produced (Das *et al*., 2007). Their interesting mechanical properties made it possible to use these as a natural aggregates replacement in road construction (Xue *et al*., 2006; Wu *et al*., 2007; Shen *et al*., 2009). High volume utilization of this product is beneficial because it helps to save natural resources (Motz and Geiseler, 2001) and reduces the tonnage of slag grains that are stocked every year. On other hand, other researchers found that products were only a small part of these slags can actually be used in road construction because their hydraulic reactivity is not very efficient (Shi and Qian, 2000; Reddy *et al*., 2006; Kourounis *et al*., 2007; Mahieux *et al*., 2009).

The knowledge of the physico-chemical, mineralogical and morphological properties of steel slag are essential due to their cementitious and mechanical properties. These properties play key roles in their utilization. For example, the frictional properties of steel slag are influenced by its morphology and mineralogy. Similarly, the volumetric stability of steel slag is a function of its chemistry and mineralogy. These properties are determined by the processes that generate this material. Therefore, knowledge of the different types of steelmaking and refining operations that produce steel slag as a byproduct is also required (Yildirim and Prezzi, 2011).

This research provides the physico-chemical and mineralogical properties of LD slag in order to make good quality utilization of steel byproducts in civil engineering and other applications.

MATERIALS AND METHODS

Sample collection: Three steel plants such as Bokaro Steel Plant (BSP), Rourkela Steel Plant (RSP) and Tata Steel Plant (TSP) in India were selected for study due to locational advantage. Bokaro Steel Plant is located geographically at 23°38'0.5.2"N 86°18'0.3.7"E in the district of Bokaro, Jharkhand. Rourkela Steel Plant is located in the North-Western tip of Odisha at 22°12'42.25"N and 84°52'18.7"E in Sundergarh district. The Tata Steel Plant is located at 22°47'05.1"N and 86°11'57"E at Jamshedpur in East Singhbhum district of Jharkhand. The sampling was carried for a period of four post-monsoon months (November and December, 2013, January and February, 2014). All these steel plants are situated in the Eastern part of the India. The properties of LD slag samples were evaluated by physical, chemical and mineralogical properties.

The physico-chemical and mineralogical properties of LD slag samples of BSP, RSP and TSP are studied as follows:

Study of physical properties: The pH is the logarithm to the base 10 of the reciprocal of the hydrogen ion concentration. The alkalinity/acidity of the soil is indicated by this parameter. Dry LD slag sample of 30 g was added to 75 cc of distilled water in a 100 cc beaker. The suspension was stirred thoroughly at first and then covered with a glass plate and left standing for 1 h with occasional stirring. The suspension was again stirred well just before the test. The pH values of the LD slag samples were determined by electrometric procedure. The instrument mainly used for pH measurement was a glass electrode pH meter with camel reference electrode including salt bridge.

Ions are the carrier of electricity, thus the electrical conductivity of the LD slag water system increased according to the content of soluble salt in the LD slag samples, giving rise to more ions or dissociation as it happens in case of a dilute solution.

Specific gravity is one of the important physical properties required for geotechnical and other applications. It is defined as the ratio of the weight of a given volume of solids to the weight of an equivalent volume of water at 4°C and is a dimensionless quanitity. The specific gravity of LD slag

sample was determined by density bottle method as per IS: 2720 (Part III/Sec 1), 1980. The LD slag samples (50 g) were initially passed through a 2 mm IS sieve for determining specific gravity.

Bulk density is an indicator of soil compaction and soil health. It affects infiltration, available water capacity and soil porosity. The bulk density of solid or soil depends greatly on the mineral make up of soil and the degree of compaction. Bulk density is the measurement of the weight of the solid (such as soil) per unit volume (g/cc). This volume includes the volume of solid particles along with pores among soil particles. The dry bulk density of LD slag samples were carried out usually given on an oven dry basis (110°C).

Particle size or grain size distribution is an important property of the soil or any material to be used for civil sector. Particle size distribution indicates if a material is well graded, poorly graded, fine or coarse etc. and used in classifying soils. It has their direct influence over various geotechnical and physical properties of the material such as bulk density, porosity, compactness, permeability, etc. The gradation analyses were done in accordance with IS 2720: Part IV, 1985 by conducting sieve analysis. Subsequently each LD slag samples were sieved for 30 min in a sieve shaker using different size of sieves. Each of the size fractions so obtained were separately analysed for particle size distribution.

Study of the chemical and mineralogical composition: For analysis of chemical properties, some LD slag grains were oven dried at 105°C and ground to fine powder. The LD slag chemical composition was determined by using (Minipal4 PANalytical X-ray fluorescence) and inductively coupled plasma optical emission spectrometry (PerkinElmer Optima 2100 DV ICP-OES spectrometer) after melting and acid digestion at Central Characterization Cell, Institute of Minerals and Materials Technology (IMMT), Council of Scientific and Industrial Research (CSIR), Bhubaneswar, India. Complementary carbon, hydrogen, nitrogen and sulphur analyses were done by using C-H-N-S analyser (Elementar Vario Micro Cube) and loss on ignition study was carried out at 980°C at Indian School of Mines (ISM), Dhanbad. The main mineral phases contained in the slag were identified using X-ray diffraction (XRD) and complementary analyses were made using fourier transform infrared spectroscopy (FTIR), X-ray fluorescence (XRF) and Field Emission Scanning Electron Microscopy (FE-SEM) coupled with Energy Dispersive Spectrometer (EDS).

In order to determine the mineralogical phases present in the LD slag samples, X-ray diffraction analyses were carried out. Representative samples of oven dried steel slag (with both gravel size and finer particles) were crushed until the powder passed through the No. 200 (0.075 mm or 75 μ opening) sieve size. The mineralogical composition of the LD slag samples were determined by the X-ray diffraction (XRD) method using (Xpert PRO, PANalytical X-ray diffractometer) equipped with a CuK radiation and a graphite monochromator at Central Characterization Cell, IMMT, CSIR, Bhubaneswar, India. The X-ray diffraction patterns of the steel slag samples were analyzed by comparing the peaks present in the XRD patterns with those provided in The Joint Committee for Powder Diffraction Standards (JCPDS) for identification of inorganic compounds. Only qualitative analyses were performed due to the presence of overlapping peaks in the XRD patterns and to the complexity of the crystalline phases in the slag samples tested. Fourier transform infrared spectroscopy (FTIR) is a technique which is used to obtain an infrared spectrum of absorption of a solid, liquid or gas. In infrared spectroscopy, IR radiation is passed through a sample. Some of the infrared radiation is absorbed by the sample and some of it is passed through transmission. The resulting spectrum represents the molecular absorption and transmission, creating a molecular fingerprint of the sample. The dominant phases and the changes in the LD slag sample were determined by fourier transform infrared spectroscopy. The analysis was used in the range from 4000-500 cm^{-1} by the spectrum One FTIR Spectrometer, (PerkinElmer

Spectrum GX) at Central Characterization Cell, council of scientific and industrial research, Institute of Minerals and Materials Technology, Bhubaneswar, India. Samples were characterized in their basic form without any preparation using Attenuated Total Reflectance (ATR) chamber.

The oxide compositions of the LD slag were determined by using X-ray fluorescence (XRF) analysis. X-ray spectrometry is a non destructive technique used to determine the percentage of element in a substance. The LD slag materials required a previous pretreatment to meet the conditions for homogeneity and to ensure the quality and reproducibility in measurements. This procedure was based on the crushing and grinding of materials into fine powder. Boric acid by weight, 10% (as binder) was added into the powder followed by exerting 200 KN of force for 10 sec to form a pellet which was placed in XRF (Minipal4, PANalytical X-ray fluorescence) equipment at Central Characterization Cell, IMMT, CSIR. A high intensity beam of X-ray is directed on the sample causing secondary X-ray to be emitted which contains characteristic wavelength of each element present in the sample. This characteristic radiation was analyzed by crystal detector and was processed in an electronic circuit and computer for determining the concentration of element. The Field Emission Scanning Electron Microscopy (FE-SEM) is a versatile, non-destructive technique that reveals detailed information about the morphology and the composition of natural and manufactured material. In order to investigate the microstructure and composite homogeneity of the slag LD slag samples were examined by field emission scanning electron microscope (Supra 55, Carl Zeiss, Germany) at Central Research Facility, ISM. Finer size LD slag particles were examined under the FE-SEM. The LD slag particles were first made conductive for current and then coated with an extremely thin layer (1.5-3.0 nm) of gold or gold palladium. The coated LD slag particles were examined and the FE-SEM images were captured on both photomicrographs and digital files. The accelerating voltage of the instrument was fixed at 5 kV for BSP LD slag, 3 kV for RSP LD slag and 4 kV for TSP LD slag. The LD slag samples were examined at the magnification of X18.59, X33.90 and X24.95. Energy dispersive X-ray spectroscopy analysis measurements were performed under standard conditions.

RESULTS AND DISCUSSION

The results of the physico-chemical and mineralogical properties of LD slag samples of BSP, RSP and TSP are given as follows.

Physical properties: The physical properties of the BSP, RSP and TSP-LD slag samples are given in Table 1 and the pH value of the BSP, RSP and TSP LD slag samples were observed to be 11.85, 11.86 and 11.67, respectively. It indicates that the samples were very alkaline due to the

Table 1: Physical properties of LD slag samples

Parameters	BSP-LD	RSP-LD	TSP-LD
pH	11.85	11.86	11.67
Conductivity (mS)	7.67	8.62	7.18
Colour	Dark gray	Dark gray	Dark gray
Specific gravity (G)	3.16	3.13	3.44
Bulk density (ρ) (g cm^{-1})	1.92	1.99	1.98
Porosity (φ) (%)	39	32	36
Coefficient of permeability, ($k\times10^{-4}$) (cm sec^{-1})	1.514	1.422	1.332
Water holding capacity (%)	28.26	21.05	27.05
Particle size distribution analysis			
D_{60} (mm)	0.71	0.51	0.72
D_{30} (mm)	0.23	0.21	0.22
D_{10} (mm)	0.07	0.08	0.07
Coefficient of uniformity (Cu)	10	6.25	10
Coefficient of curvature (Cc)	0.851	1	0.987

BSP-LD: Bokaro steel plant-Linz Donawitz, RSP-LD: Rourkela steel plant-Linz Donawitz, TSP-LD: Tata steel plant-Linz Donawitz

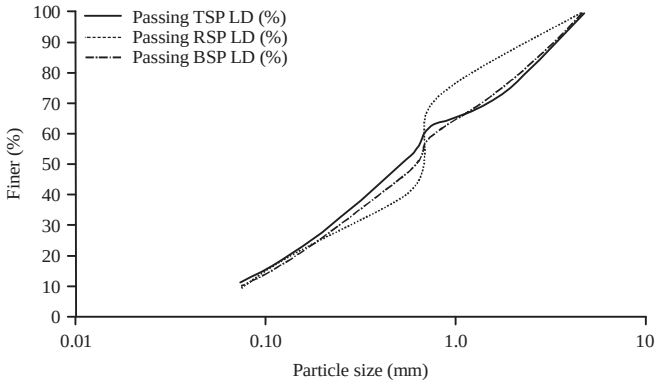

Fig. 1: Particle size distribution from sieve analysis for RSP, BSP and TSP-LD slag

presence of high percentage of lime. The pH range of RSP was higher than the other two samples which were confirmed by XRF analysis. The electrical conductivity of the LD slag sample of BSP, RSP and TSP were found as 7.67, 8.62 and 7.18 mS, respectively. It indicates that the conductivity of samples were very high due to the presence of ionic form of various salts. The electrical conductivity of RSP-LD slag sample was higher than BSP and TSP-LD slag samples. The specific gravity of LD slag sample of BSP, RSP and TSP were found to be 3.16, 3.13 and 3.44, respectively. This was higher than specific gravity of normal soil which implies that the erosion potential of LD slag is low and is more stable. The porosity of LD slag samples were found in the range of 39, 32 and 36%, respectively. The bulk density of the LD slag samples of BSP, RSP and TSP were found in the range of 1.92, 1.99 and 1.98 g/cc, respectively. This high bulk density was due to its mineral composition and degree of compactness.

Particle size distribution analyses were carried out by the dry sieve analysis. The percentage passing of sample vs. the sieve size were plotted in the graph is shown in Fig. 1. The uniformity of a sample is reflected by the grain size distribution curve. For example, a steep curve indicates a more or less uniform size whereas an S-shaped curve represents a well graded size. The uniformity coefficient ($C_u = D_{60}/D_{10}$) and coefficient of curvature ($C_c = D^2_{30}/D_{60}D_{10}$) of the LD sample of BSP, RSP and TSP were 10 and 0.851, 6.25 and 1 and 3.714 and 1.076, respectively. These values indicate that the LD slag samples were well graded. As the samples were having a good representation of grain sizes (gravel to fines) over a wide range and smooth gradation curves were obtained (Singh *et al*., 2013). The results of the permeability test of LD slag samples show that the coefficient of permeability values of LD slag samples were in the order of 10^{-4} cm sec^{-1} which were very low and equivalent to the permeability of silts. However, the permeability of the BSP-LD was higher than other two LD slag due to coarser particle size. The RSP and TSP-LD slag possess the minimum permeability values of 1.422×10^{-4} and 1.332×10^{-4} cm sec^{-1}, respectively. The results of WHC of LD slag samples were found to be 28.26, 21.05 and 27.05%, respectively. The RSP-LD slag sample, which was comparatively more porous than the other two samples, possesses highest WHC. Therefore, it is clear that water absorption is a function of porosity and the samples containing higher amounts of pores possess higher water absorption capacity (Iyer and Stanmore, 1999).

Chemical, mineralogical and morphological study: Table 2 summarizes all the mineral phases that were identified in the LD slag samples in RSP, BSP and TSP. As LD slag is cooled slowly in slag pits and there is sufficient time for formation of well defined crystals. It was observed that, the XRD patterns of the LD slag samples were very complex, with several overlapping peaks

resulting from the many minerals phases present in the crystalline samples (Fig. 2). The mineral phases identified in the LD slag samples were determined by on the basis of the intensity of the peaks, which is an indication of the quantity of the minerals present in the samples. It is important to note that the very complex mineralogical composition of LD slag, with many overlapping peaks and different solid solutions of oxides (FeO and MgO), makes the identification of the phases very difficult in slag crystals. Several other researchers have reported similar, complex XRD patterns for LD slag (Reddy *et al*., 2006; Yildirim and Prezzi, 2011; Nicolae *et al*., 2007; Tossavainen *et al*., 2007).

Figure 3, 4 and 5 shows a gravel size LD slag particle with a lime pocket seen in white. Lime pockets may not hydrate at all if they are not given access to water through the fractures extending to them. If there are fractures in the slag particles extending to these lime pockets, then hydration can progress (Juckes, 2003; Shi, 2004; Kneller *et al*., 1994).

The most abundant mineral phase present in LD slag is portlandite ($Ca(OH)_2$). The presence of this mineral is expected since LD slag contains 45.14, 44.93 and 45.67% lime (CaO), which in the presence of moisture, converts to $Ca(OH)_2$. The other major phases included merwinite ($Ca_3Mg(SiO_4)_2$) and srebrodolskite ($Ca_2Fe_2O_5$). The presence of free lime (CaO) and the probable presence of free periclase or magnesia (MgO) in the LD slag samples are an indication of the potential for volumetric instability, deleterious and expansive hydration reactions (Yildirim and Prezzi, 2011).

Table 2: Mineralogical phases of LD slag

Mineral phases	Formula
Srebrodolskite	$Ca_2Fe_2O_5$
Larnite	Ca_2SiO_4
Calcite	$CaCO_3$
Lime	CaO
Dolomite	$CaMg(CO_3)_2$
Periclase	MgO
Hematite	Fe_2O_3
Portlandite	$Ca(OH)_2$
Wustite	FeO

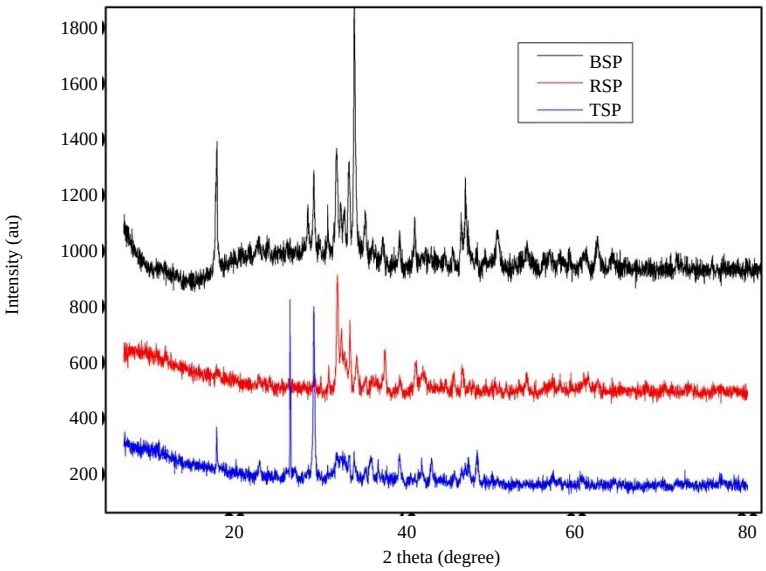

Fig. 2: XRD pattern of BSP, RSP and TSP-LD slag

Fig. 3: Gravel size LD slag (photograph taken at Rourkela Steel Plant, Odisha, India)

Fig. 4: Gravel size LD slag (photograph taken at Tata Steel Plant, Jamshedpur, India)

Fig. 5: Gravel size LD slag (photograph taken at Bokaro Steel Plant, Jamshedpur, India)

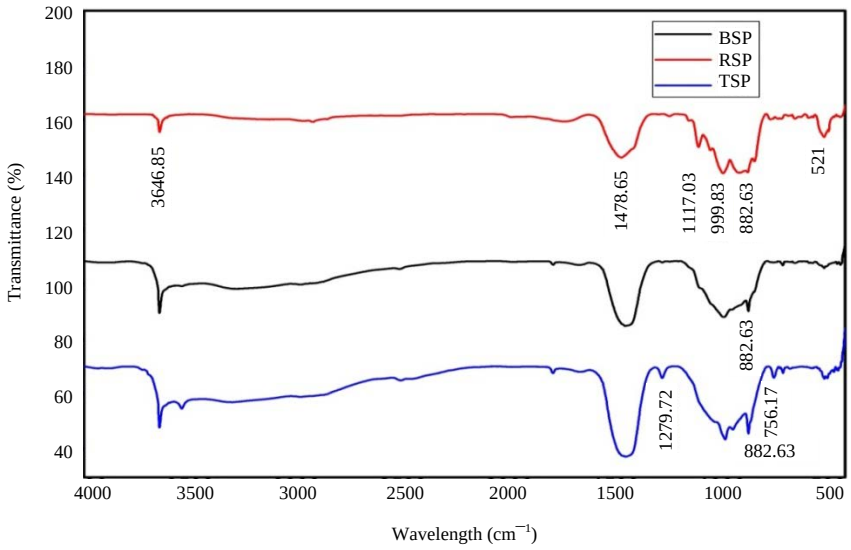

Fig. 6: FTIR spectrum of BSP, RSP and TSP-LD slag

Table 3: Chemical composition of LD slag

Slag types	FeO	SiO_2	Al_2O_3	CaO	MgO	MnO	P_2O_5	TiO_2	S	LOI	C-H-N-S
Chemical composition (weight %)											
RSP-LD	24.14	12.98	2.17	45.14	7.96	0.78	3.05	0.72	0.31	2.72	0-0.8-0-0.3
BSP-LD	23.89	14.09	3.39	44.93	8.18	0.85	1.53	0.76	0.24	2.06	0-0.6-0-0.2
TSP-LD	23.38	13.29	2.43	45.67	8.09	0.72	3.03	0.46	0.28	2.63	0-0.7-0-0.2

LOI: Loss on ignition, BSP-LD: Bakaro steel plant-Linz Donawitz, RSP-LD: Rourkela steel plant-Linz Donawitz, TSP-LD: Tata steel plant-Linz Donawitz

The IR spectra of RSP, BSP and TSP-LD slag samples are presented in Fig. 6. Absorptions correspond to the dominant phases: portlandite ($Ca(OH)_2$) and periclase (MgO) at 3646.8 and 1478 cm^{-1}, respectively. The peaks of 1117.03, 999.83, 882.63 and 756.16 cm^{-1} (although weak) corresponded to periclase (MgO). Adsorption is a surface phenomenon in which molecules of the adsorbate are attracted and held to the surface of an adsorbent until an equilibrium is reached between the adsorbed molecules and freely distributed gas or liquid. Adsorption depends on the interaction between the surface of the adsorbent and the adsorbed species. The area is a fundamentally important feature of the adsorbents in adsorption. There are different mechanisms by which metallic ions or other ions are removed from an aqueous solution. The first states that the process is based on electrostatic adsorbate-adsorbent interactions being totally dependent on the existence of surfaces functionality, especially ion exchange process. The second one states that the enhanced adsorption potentials as occurs in the narrowest microporosity may be strong enough to adsorb and retain ions (Dias *et al*., 2007).

Based on the chemical characterization of the LD slag samples by XRF analysis, it was observed that the major components of the three steel plants, BSP, RSP and TSP LD slag samples are CaO, FeO and SiO_2. It can be noticed that for the RSP-LD slag sample, the percentage of CaO, SiO_2 and FeO is 45.14, 12.98 and 24.14%, for BSP-LD, 44.93, 14.09 and 23.89% and for TSP-LD slag, 45.67, 13.29 and 23.38%, respectively. The chemical composition of the LD slag from RSP, BSP and TSP are shown in Table 3. From the previous study it was observed that, the iron oxide (FeO and Fe_2O_3) content of LD steel slag can be as high as 38% (Shen *et al*., 2009).

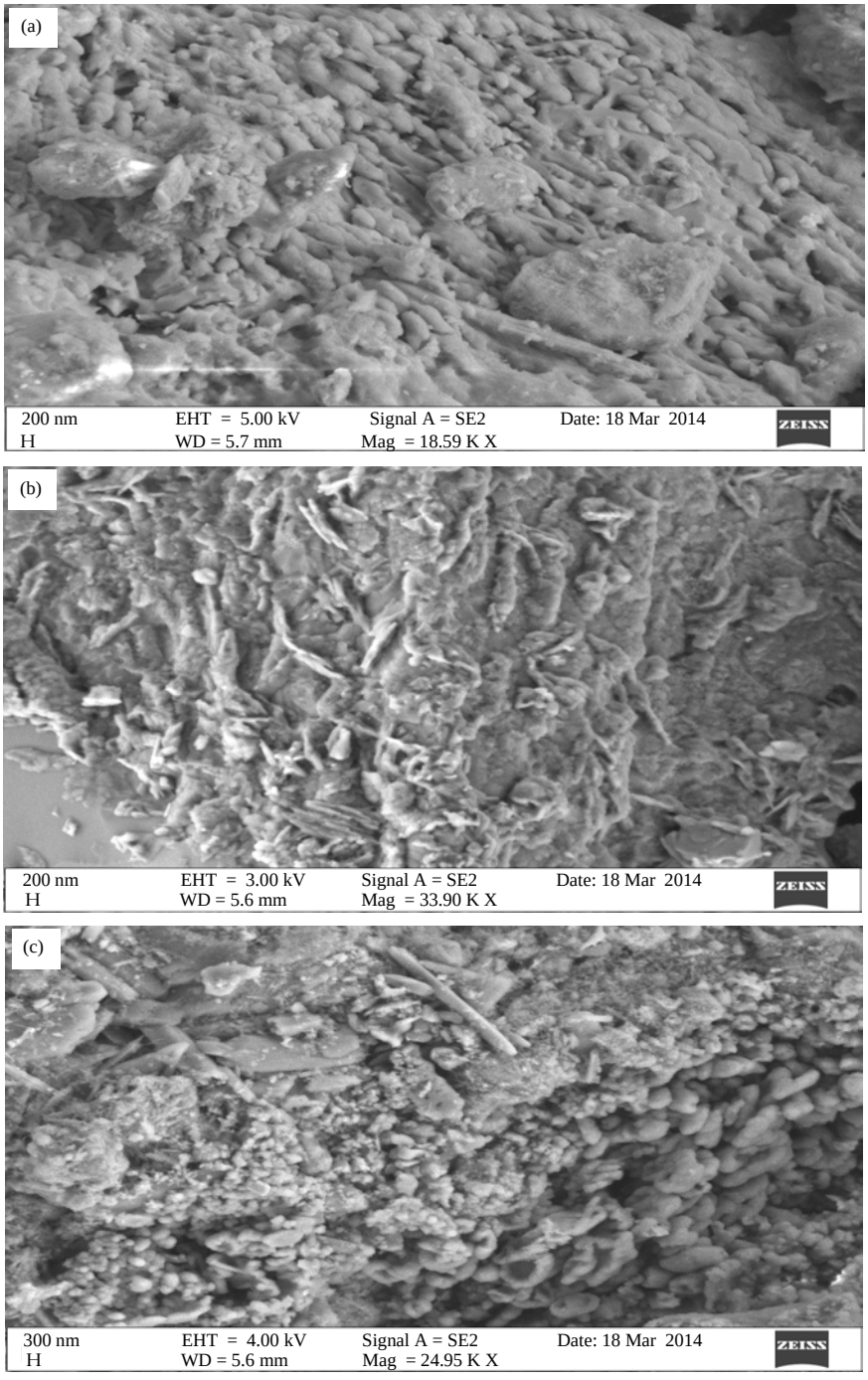

Fig. 7(a-c): (a) FE-SEM micrographs of BSP LD slag sample, (b) FE-SEM micrographs of RSP LD slag sample and (c) FE-SEM micrographs of TSP LD slag sample

The FE-SEM study of the LD slag samples shown in Fig. 7 reveals that textures are rough, cubical, bulky particle and subrounded to angular in external appearance. Heterogeneous porous structures were also observed on the surface of a few particles. Internally, each particle was vesicular in nature with many non interconnected cells. The cellular structure was formed by the

gases entrapped in the hot slag at the time of cooling and solidification. Since these cells did not form connecting passages, the term cellular or vesicle was more applicable to steel slag than that of the term 'Porous' and likely to appear as well (Singh *et al*., 2013).

The electron image of LD slag sample in EDS X-ray micro analysis is shown in Fig. 8 and its corresponding graph showing the elemental peaks is shown in Fig. 9. The EDS X-ray micro analysis of LD slag sample showed that the elemental compositions of the sample are C, O, Mg, Na, Al, Si,

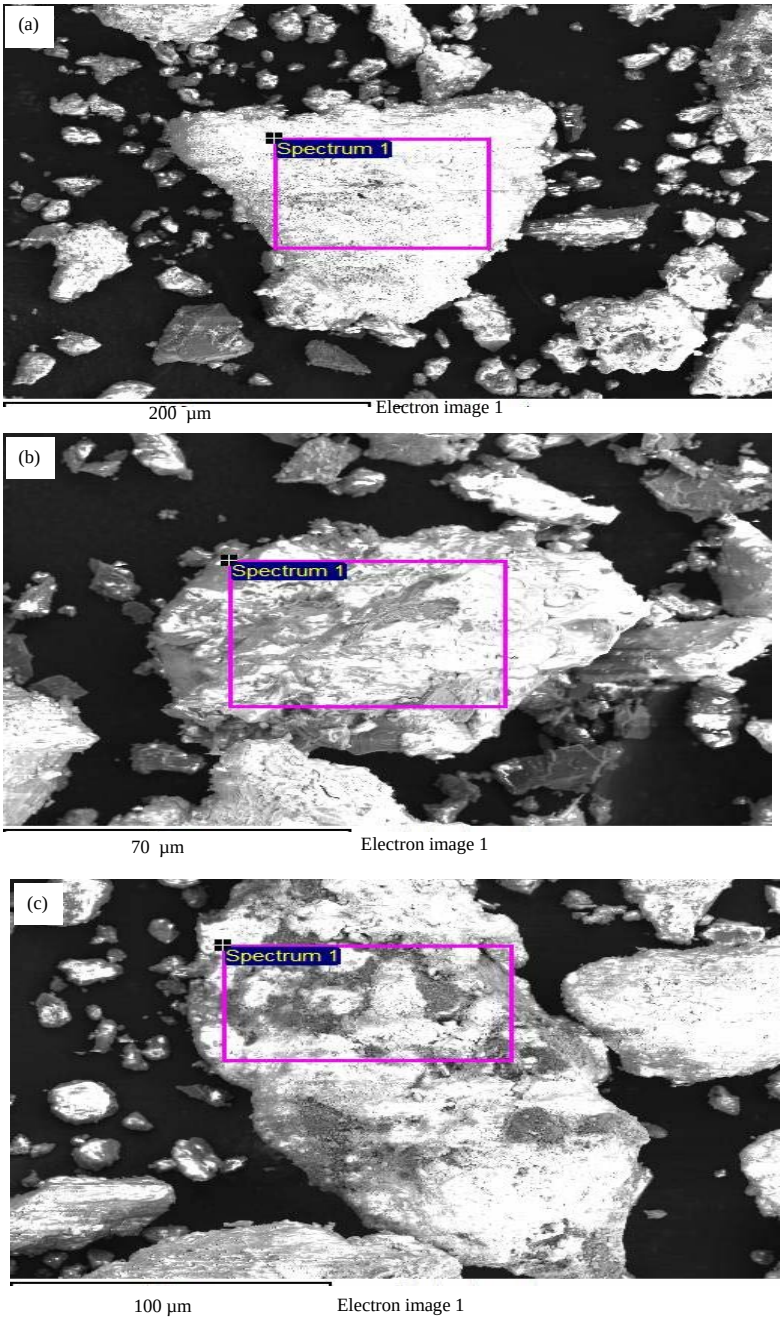

Fig. 8(a-c): (a) EDS image of RSP LD slag sample at 200 µm, (b) EDS image of RSP LD slag sample at 70 µm and (c) EDS image of RSP LD slag sample at 100 µm

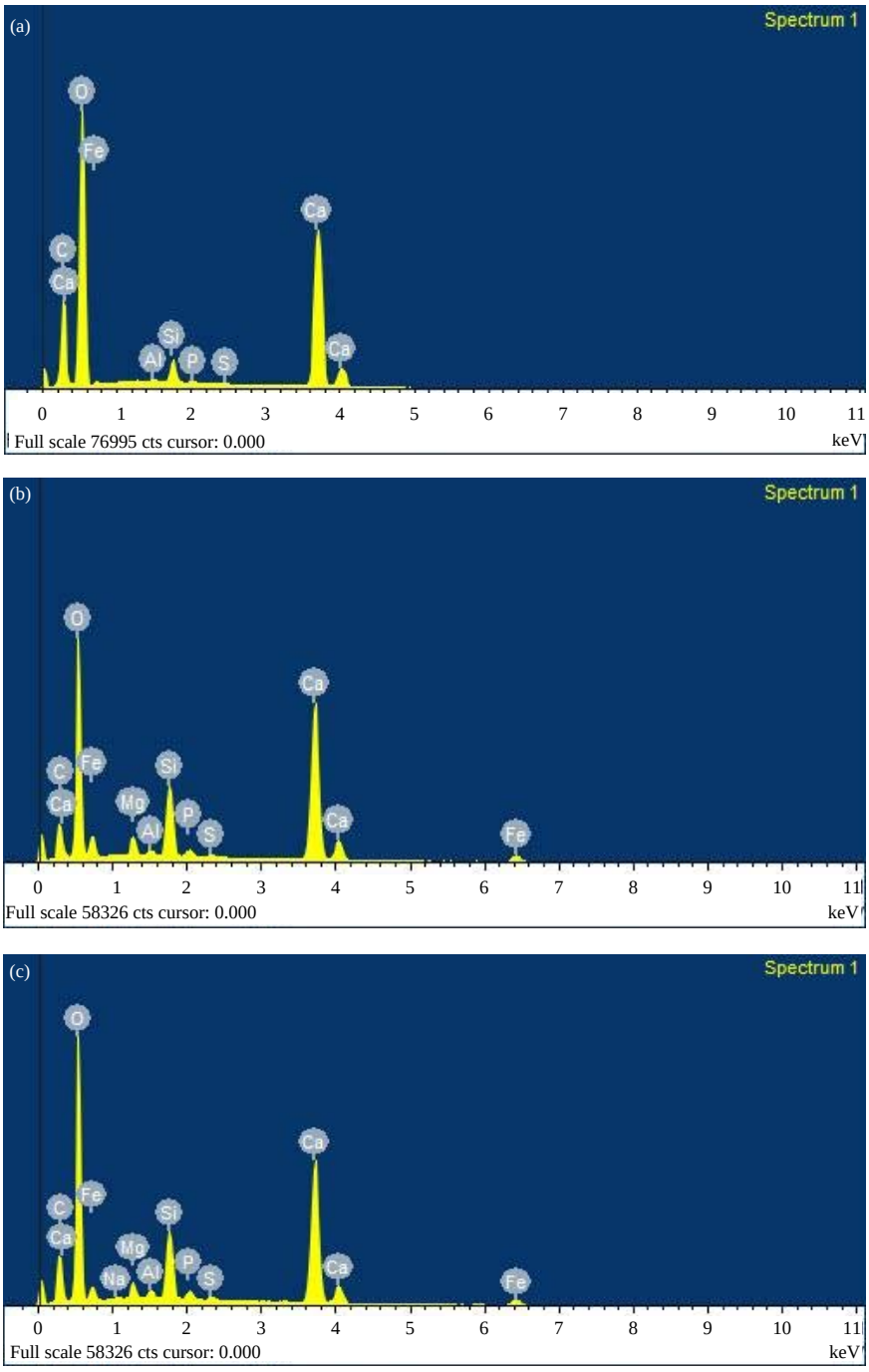

Fig. 9(a-c): (a) EDS analysis of RSP LD slag sample, (b) EDS analysis of BSP LD slag sample and (c) EDS analysis of TSP LD slag sample, Y-axis represents the presence of element in LD slag

P, S, Ca, Fe and Ti. Among the above elements, O and Ca share the major percentage by weight in the LD slag sample. Elemental composition of LD slag sample analyzed by EDS are shown in Table 4.

Table 4: Elemental composition of LD slag samples based on Fe-Sem-energy-dispersive x-ray spectroscopy

Elements	RSP-LD (Weight %)	BSP-LD (Weight%)	TSP-LD (Weight%)
CK	4.49	9.09	6.11
OK	37.05	48.45	42.63
Na K	-	-	0.13
Mg K	1.11	-	0.94
Al K	0.29	0.16	0.47
Si K	5.10	1.71	4.92
PK	0.71	0.17	0.87
SK	0.26	0.12	0.31
Ca K	37.29	38.41	33.83
Fe L	13.70	1.89	9.80
Total	100.00	100.00	100.00

BSP-LD: Bakaro steel plant-Linz Donawitz, RSP-LD: Rourkela steel plant-Linz Donawitz, TSP-LD: Tata steel plant-Linz Donawitz

CONCLUSION

The physical, chemical and mineralogical properties of LD slag samples generated from three steel plants in India were investigated and the following conclusions were reached. From the physical properties, LD slag samples showed that the pH and electrical conductivity of the samples were very high indicating high percentage of lime and presence of ionic form of various salts, respectively. The specific gravity and bulk density of LD slag samples found to be high due to presence minerals composition and can be expected to yield a higher density and stable product. The permeability was found to be similar to that of silt. The WHC of the LD slag samples were found to be high due to the porous nature. The uniformity coefficient (C_u) and coefficient of curvature (C_c) values in particle size analysis indicate that LD slag samples were well graded samples. The XRD study of LD slag sample showed that the main mineral phases were portlandite, srebrodolskite and merwinite. The XRF analysis revealed that the major components of the LD slag samples were CaO, FeO and SiO_2. Since these compounds expand when hydrated, the volumetric instability of the LD slag needs to be assessed for their use in civil engineering applications. The FTIR study demonstrated that, calcium silicates are the major compounds in the LD slags. The LD slag with such composition could be considered as an effective adsorbent. This can be related due to its high content of CaO, MgO and SiO_2 as surface functional groups which important for the LD slag adsorption ability. The FE-SEM study of LD slag sample results showed that the samples were rough textured in surface and cubical and subrounded to angular shapes in external appearance. Internally, the particles were vesicular in nature and non interconnected cells. The cellular structures were formed by the gases entrapped in the hot slag at tile of cooling and solidification. The EDS X-ray analysis of LD samples showed that the elemental compositions of the samples are C, O, Mg, Na, Al, Si, P, S, Ca, Fe and Ti. Along with the above elements, O and Ca share the major percentage by weight in the LD slag samples.

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