Journal of Materials Science Research and Reviews

2(4): 574-581, 2019; Article no.JMSRR.54059

The Characterization of Fly Ash during High Temperature Oxidation of Galvanized Steel

Maame Adwoa Bentumah Animpong[1*], Robert Kwame Nkum[2], William Owusu Oduro[1], Elizabeth Von-Kiti[1], Benjamin Adjei Tuffour[3] and Andy Quarshie[1]

[1]Council for Scientific and Industrial Research, Institute of Industrial Research, P.O.Box LG 576, Legon, Accra, Ghana.
[2]Kwame Nkrumah University of Science and Technology, Kumasi, Ghana.
[3]University of Ghana, P.O.Box LG 25, Legon, Accra, Ghana.

Authors' contributions

This work was carried out in collaboration among all authors. Author MABA planned the article and wrote the first draft of the manuscript. Author RKN supervised the work. Author WOO managed the analysis of the data and also supervised. Authors EVK and AQ analyzed portions of the data for the work. Author BAT did some portions of the methodologies. All authors read and approved the final manuscripts.

Article Information

Editor(s):
(1) Dr. Oscar Jaime Restrepo Baena, Professor, Department of Materials and Minerals, School of Mines, Universidad Nacional de Colombia, Colombia.
Reviewers:
(1) Norhayati Hashim, Universiti Pendidikan Sultan Idris, Malaysia.
(2) Mandadapu S. V. K. V. Prasad, Jawaharlal Nehru Technological University, India.
Complete Peer review History: http://www.sdiarticle4.com/review-history/54059

Original Research Article

Received 15 November 2019
Accepted 17 January 2020
Published 25 January 2020

ABSTRACT

Fly Ash, a solid waste (particulate matter) generated from a steel smelting plant was analyzed and characterized. Physical and chemical investigations of the fly ash from a steel manufacturing company was conducted using X-ray diffraction (XRD), Scanning electron microscopy (SEM) and Hot stage Microscopy (HSM) to determine it properties. The fly ash sample comprised mainly of Fe, Zn, Mg, Si and Na. Zinc and Iron, the most abundant elements in the fly ash were found to be in the oxides. The amorphous nature of the ash had some distinct compounds and minerals, which had Gordaite being formed as one of the major phases as a result of the high temperature oxidation of galvanized steel auto parts. The other phases formed were Si_5O_{15} and Zinc Ferrite.

**Corresponding author: Email: mabsage@yahoo.com;*

From the analysis, it was found out that Zinc Ferrites compounds were formed from the reaction of iron oxides and zinc oxide at high temperatures. Technologies can be developed for gainful utilization of the fly ash in the manufacture of new products.

Keywords: Fly ash; X-ray diffraction; hot stage microscopy; chemical analysis.

1. INTRODUCTION

Fly ash (FA) is a solid waste (<630µm) generated in the collection of particulate material during the smelting process in steel industry. During production of steel, considerable amount of different types of solid wastes (blast furnace slag, fly ash, blast furnace flue dust, LD-Slag, coke breeze, tar sludge etc.) are generated. The composition of these waste materials varies widely depending on their source of generation, the quality of raw materials and the metallurgical operations. The main contents of fly ash derived from steel production are oxides of metals such as Fe, Cu, Zn, Mn, and Pb. Globally a million metric tons of fly ash are produced per annum during smelting processing [1].

The wastes (blast furnace slag and fly ash) produced from steel plants in Ghana are generally disposed of by dumping in landfills or used to reinforce the ground in waterlogged areas during construction, which may have environmental repercussions when not managed properly. Currently, environmental legislations and economics force steel industries to minimize generation of wastes and maximize its recycling or utilization in accordance to the Sustainable Development Goals (SDG) 12 (responsible production and consumption). Recycling or utilization of waste is necessary because it affords efficient use of storage space, waste becomes a resource for secondary production thus mitigating the effect of fast depletion of natural resources. Hence recycling and reuse of materials in industrial practice have social, environmental and economic advantages [2].

Recycled steel accounts for about 100% of Ghana's steel production. It is produced by recycling scrap-based steel in an electric arc furnace (EAF). The main by-products in recycled steel production are slag, dusts (fly ash) and sludge. [3]. The high temperature processes give these by-products of unique thermal and mechanical stability which can be exploited as useful raw materials for secondary processes where mechanical strength and thermal stability is desired. Zinc ferrites can be used as pigments because of their high opacity, especially in applications requiring heat stability (above 177°C). When added to high corrosion-resistant coatings, the corrosion protection increases with an increase in the concentration of zinc ferrite [4].

Awareness of the harmful solid waste from the steel making plant has increased over the years in the world and many researchers have discussed the potential reuse treatments of these various wastes including fly ash [5].

The problem of steel FA disposal is only expected to get worse as the demand for steel/metal products grows. FA poses serious problems to the industries and is also considered a hazardous waste due to the probable leaching of potentially toxic substance into the surface water, ground water and soil. Also, the release of fine particles into the atmosphere can cause upper respiratory diseases or occasion allergies. Recycling of FA prevents air, water and soil pollution, saves energy and raw materials and reduce greenhouse gas emissions [6]. Utilization of FA can result not only in reducing the magnitude of the environmental problems, but be exploited as a raw material (conserve traditional materials) for a secondary production cycle [7].

This paper seeks to analyze the chemical, thermal and morphological characteristics of fly ash from a steel making plant in Ghana, which has its source of raw materials being scrap ferrous metals. These ferrous metals are part of municipal solid waste from automotive servicing garages and other decommissioned equipment which are frequently stored on the bare ground.

2. MATERIALS AND METHODS

The steel fly ash used in this experiment was obtained from the bag house unit of the rotary hearth furnace of a Steel Company in Ghana. 500 g of the ash sample was oven dried at 105^{o}C for 12h to remove any moisture after which it was sectioned into smaller quantities and analyzed using X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM), Hot Stage Microscopy (HSM) and physico-chemical determined.

2.1 Particle Size

The particle size distribution was determined with a FRITSCH analysette 3 PRO particle size analyzer. The particle size distribution of fly ash can vary considerably depending on how the smelting plant is operated. However, many smelting plants operate some base load generation, reducing the potential variation of the resulting fly ash [8]. The sieves were arranged with sizes 63µ, 90µ, 355µ, 500µ and 630µ with smallest mesh size at the bottom. The FA was introduced unto the 630µ and shaken for 20 mins according to ASTM C 136-01.

2.2 pH

The pH of the fly ash was determined using a Suntex pH/ meter SP-701. One part of the dry sample was weighed to 2.5 ml of water and then stirred continuously for 30mins. The leachate was then tested with the pH meter.

2.3 Analysis of Chemical Composition of Fly Ash

The chemical composition of the FA was determined using WDXRF Axios mAX spectrometer with a 4kW RH lamp and PANalytical software to determine the major constituents and minor constituents in the fly ash.

2.4 Electrical Conductivity

The Electrical conductivity was determined using Loviband SensoDtract Con 200. One part of the dry sample was weighed to 2.5 ml of water and then stirred continuously for 30 mins. The leachate was tested with the equipment.

2.5 Microscopy

A susceptibility to thermal expansion was investigated on the fly ash taken from the steel smelting plant. Adequate observation of the fly ash sample was examined under a Hot Stage Microscope (HSM) (type 3, Misura) to determine the characteristic temperatures corresponding to the changes of the shape and the cross-sectional area of the samples being heated.

They are based on observing and analyzing images of the fly ash recorded with the growth of its temperature.

2.6 X- ray Diffraction

X-ray diffraction (XRD) measurements were conducted using standard powder diffraction procedures. X-ray diffraction pattern of the fly ash was done using (D2 Phaser, 2nd Generation) at 30 kV and 10 mA. X-ray diffraction (XRD) measurements were conducted using standard powder diffraction procedures. The FA samples were smear-mounted on a glass slide and analyzed at a scan rate of 4°(2θ) min−1 using monochromatic Cu Kα radiation to determine thecrystalline phases and the various mineral and compounds present in the fly ash.

2.7 Scanning Electron Microscope (SEM)

SEM is one of the best and most widely used techniques for the chemical and physical characterization of fly ash. It uses a focused electron beam to scan the surface of the sample to generate a variety of signals [9] The morphology of the fly ash was analyzed by scanning electron microscopy (Nova Nano SEM 200; FEI EUROPE Company) equipped by EDS analyze (EDAX).

3. RESULTS AND DISCUSSION

3.1 Physico Chemical Properties of the Fly Ash

3.1.1 Physical features

Generally, the colour of FA can vary from tan to gray to black, depending on its chemical, mineral constituents and other elemental composition. A brownish colour is typically associated with high iron content. The as collected FA was very dark brown in colour. The dark hue of the fly ash could thus be as a result of high iron content or from the different chemical composition [10]. Fly ash was finely-grained upon physical examination but also consisted of traces of coarse particles of different shapes (regular and irregular) and sizes.

3.1.2 Particle size distribution

Particle size distribution is probably one of the most important characteristics which influence the activity of fly ash more than any physical properties [11].

The fly ash particle size, shape and distribution play vital roles in determining the physical properties of any composite matrix with it. Fig. 1 shows the results of the particle size analysis for fly ash particles for this study. From the graph the median of the particles lies above 400 µm. The lower and higher values of 63µm and 650µm had 0.4% and 7.8% of the fly ash particle size respectively. The highest particle size was

500µm with 86% of the FA ash retained in it. The particle size distribution of fly ash can vary considerably depending on how the smelting plant is operated. However, many smelting plants operate some base load generation at a temperature of 1600^oC- 1700^oC [5], reducing the potential variation of the resulting fly ash [8]. Generally, literature gives that, the particle size of fly ash up to 45um is more suitable as fillers for construction materials. Results from research conducted with FA showed that the addition of FA filler increases the stiffness of a plastic formulation but, like different fillers, their resistance impact is minimized [12]. The particle size distribution of the Fly ash from the recycle steel production plant is depicted in Fig. 1.

3.1.3 pH

Leaching of trace elements from fly ash in any aqueous medium is one of the important aspects to be considered in environment contamination. Recently fly ash disposal problem has been reviewed by various authors [13]. Ashes may be acidic or alkaline depending on its chemical constituents of it source. The pH was found to be 8.01 which is distinctly alkaline in nature hence the propensity to leach into water is weak.

3.1.4 Electrical conductivity

The electrical conductivity of the fly ash from the steel plant was determined to be 36200 S/m. It is generally inferred that a higher value of electrical conductivity, the lower the resistance to the flow of current in the material. The high conductivity of the FA could be due to the presence of zinc salts which is as a result of electroplating and galvanizing of iron for the production of the steel. Hence, the FA might useful for fabrications where electrical conductivity is expected to be high. This is because metals (fused in the fly ash) are dry and good conductors of electricity.

3.1.5 Chemical composition of fly ash

The chemical composition of the FA was determined using WDXRF Axios mAX spectrometer. The analysis of the fly ash as shown in Table 1 indicate the most abundant elements were Fe and Zn with relative abundance to be 21.506% and 40.442% respectively. The high levels of the Fe and Zn is as a result of the sources of materials being scrap metals for the production of steel. Also, the high level of the Na_2O can be explained from literature due the fuels used on the production of steel. Soda ash also known as trona is added to alumina during processing [14].

3.2 Morphological Characterization of Fly Ash

3.2.1 SEM/EDX

Extensive microscopic observation of the fly ash shows particles of both regular and irregular shapes. The Fig. 2a, b and c show the FA at three different magnification at 10,000x, 5,000x and 20,000x respectively. The backscattering electron images give low gray colour showing darker and lighter regions, which corresponds to areas with lighter and heavier elements from the Periodic Table [15]. Three different shapes of the FA particles were identified to be (1) cuboidal with an average size and standard deviation of 0.8 ± 0.4, (2) rod-like with an average size and standard deviation of 1.62 ± 0.57 and (3) spherical with an average size and standard deviation of 1.07 ± 0.38. Fly ash from coal has previously been identified with spherical shape particles [16]. These spherical features were attributed to the presence of certain granular deposits possibly of some iron oxide or even unburnt carbon [11]. The elements that were predominant from the EDS analysis were Pb, Zn, O, Cl, Si, S and Fe. Closer inspection at higher magnification Fig 2c shows three different crystals shapes.

3.2.2 XRD

The results from the XRD analysis gives information of the compounds that were possibly formed during combustion. The black peaks are in reference to the red peaks using a software - Material Analysis Using Analysis (MAUD). It can also be seen from the image that the fly ash had an amorphous nature with some distinct compounds and minerals. The compounds formed are listed in Table 2. The phase with the highest fraction by both weight (72.2%) and volume (81.6%) is Gordaite. The crystallite size of the Gordaite as calculated from the XRD using the Scherrer’s equation was 425.94 nm. The chemical composition of Gordaite comprises of elements such as Zinc, Sodium and Sulphur and the chemical formula is $NaZn_4(SO_4)(OH)_6Cl.6H2O$. This mineral occurs naturally in oxidized deposits of Cu-Zn sulphide found in the mines of San-Francisco and Chile [17]. It was produced during the high temperature oxidation of galvanized steel auto parts and Cu contaminants from alloys in the scrap ferrous raw materials. The second highest phase by weight is Zinc Ferrite 18.2%. Zinc ferrites are a series of synthetic inorganic compounds of zinc and iron (ferrite) with the general formula of $Zn_xFe_{3-x}O_4$. The chemical composition is $Fe_{1.78}O_{3.71}Zn_{0.945}$. Zinc ferrite compound are formed through the reaction of iron oxide and zinc oxide at high temperatures ranging between 920°C – 960°C [18].

The compounds ZnO, Fe_2MgO_4, SiO_2 and Fe_2SiO confirms the presence of the elements found in the SEM results of the fly ash sample of which Zn and Fe were in higher percentages of 46.4% and 38.6% respectively.

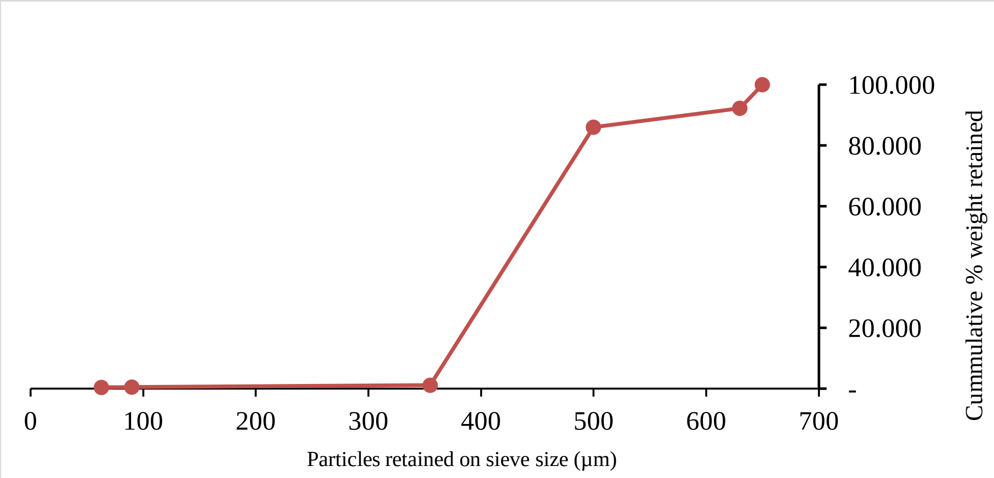

Fig. 1. Particle size distribution of fly ash indicates the median size of the fly ash of about 440 microns

Table 1. Chemical composition of the fly ash

Analyte	Compound formula	Concentration%
Na	Na_2O	10.920
Mg	MgO	2.661
Al	Al_2O_3	0.632
Si	SiO_2	4.562
P	P_2O_5	0.264
S	SO_3	3.201
K	K_2O	2.144
Ca	CaO	2.177
Ti	TiO_2	0.095
Cr	Cr_2O_3	0.554
Mn	MnO	3.171
Fe	Fe_2O_3	21.506
Ni	NiO	0.021
Cu	CuO	0.218
Zn	ZnO	40.442
Nb	Nb_2O_5	0.245
Cd	CdO	2.317
Sn	SnO_2	0.201
Ba	BaO	0.131
W	WO_3	0.184
Pb	PbO	2.077
Bi	Bi_2O_3	0.014
F	F	0.289
Cl	Cl	5.349
Br	Br	0.265
I	I	0.056

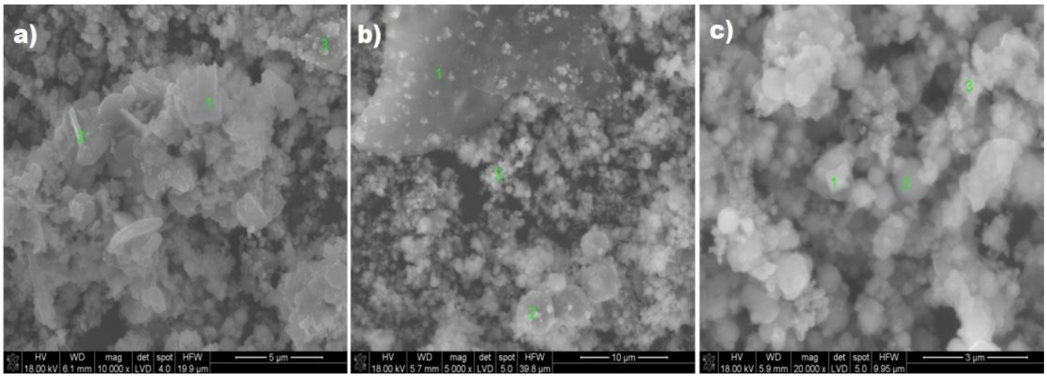

Fig. 2. A scanning electron micrograph of the fly ash magnification a) 10,000x, b) 5,000x and c) 20,000x

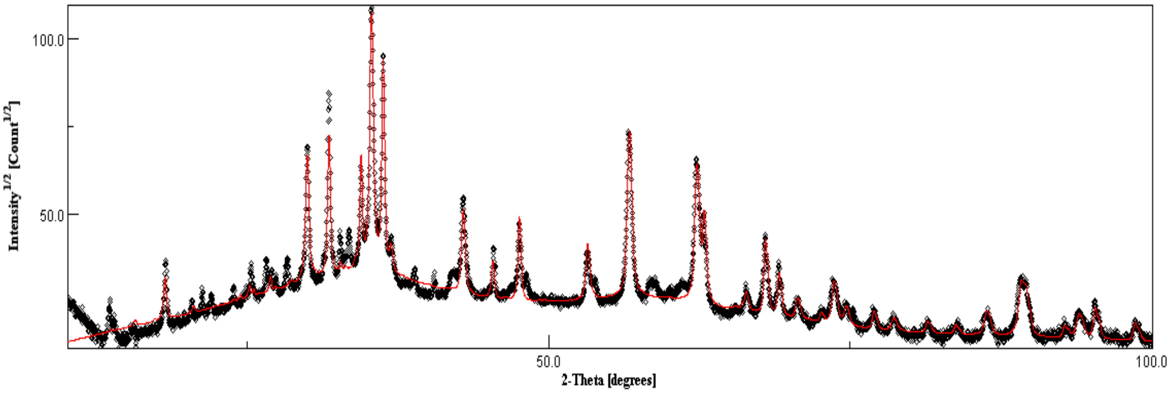

Fig. 3. X-ray diffractogram of the fly ash from an electric arc furnace in Ghana using materials analysis using diffraction (MAUD)

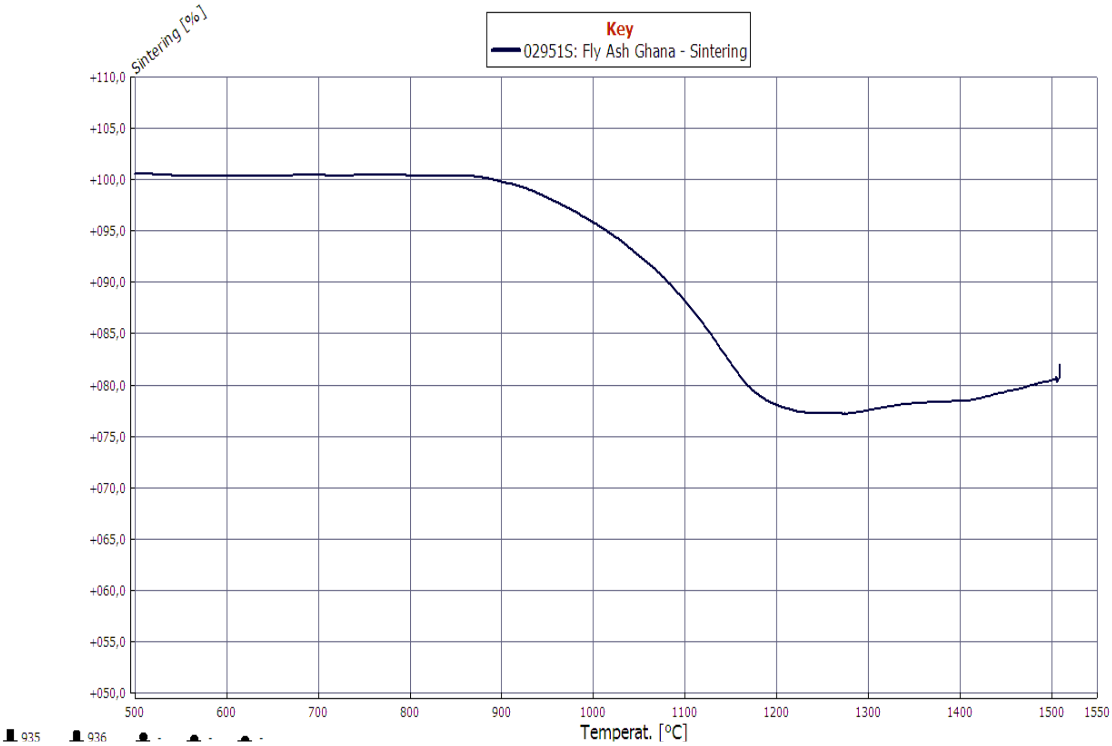

Fig. 4. HSM of the fly ash

Table 2. Mineralogical composition of the FA from XRD

Phase	Gordaite	Si_5O_{15}	Zinc ferrite	Fe_3Si	Disodium oxide	Antimony
Wt Fraction	0.722	0.001	0.182	0.005	0.002	$9.61x10^{-4}$

3.2.3 Hot Stage Microscopy (HSM) analysis of fly ash

HSM has found its applicability to studying various raw materials and products as well as to solving some technological problems such as finding the characteristic temperature corresponding to the changes of the shape in the ceramic, glass and energy industries as well as in metallurgy and foundry engineering [19]. It affords a combination of microscopy and thermal analysis to enable the study of the material as a function of temperature and time. Fig. 4 shows the result of the Hot Stage Microscopy of the FA. The sintering process of the steel FA was investigated in order to have information with regards to the melting point and other transformations which occur during heating. When conducting investigations of raw materials, their susceptibility to expansion on heating is often expressed as the changes of the coefficient of thermal expansion (S). The coefficient is calculated on the basis of the cross-section area changes of the solid body that is an equivalent of the sample volume at the given temperature [20]. The plot of the graph is characterized by a long plateau. The contraction around 890^o-900^oC (Fig. 4) may have been as a result of sintering or collapsing of a lattice structure. Expansion may also be as a result of formation of new lattice structure from the formation of new crystals during the heating process. The melting process starts at the end of the plateau with a gradual drop in the sintering percentage. After the shrinkage stage, the curve starts to show a gradual expansion around 1200^oC. There is a short plateau after which the expansion increases due to the formation of Zincite and MagnesioFerrite in the Fly Ash which has melting points of 1977^oC and 1750^oC respectively.

4. CONCLUSIONS

The FA from typical scrap ferrous metal smelting using a rotary hearth furnace has high carbon content, alkaline pH and high electrical conductivity. The FA particles gives shapes which are predominantly spherical, some rod-like and cuboidal with irregular particle size. The average particle size comprised mainly of Zn, Fe, Si, Na and Mg with other elements in the minority. The particle size was less than 630um and XRD confirmed the presence that Zinc Ferrites compounds were formed from the reaction of iron oxides and zinc oxide at high temperatures. The HSM showed the FA sintering at 890^oC-900^oC showing a possible formation of new lattice or expansion of the ash. Technologies can be developed for gainful utilization of the fly ash in the manufacture of mixed fly ash clay bricks which is found to be cheaper, high in structural and aesthetics qualities in the construction industry and also in the extraction of Zn from the FA. When mixed with clay or other materials, as a reinforcement agent, to form bricks, the Gordaite could improve the aesthetics (lustre) and the Zinc Ferrite as well increase the density of the material by reducing the porosity and hence increase the tensile strength of the materials.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

REFERENCES

1. Li-Yin Hsiao, Wen-Lin Wang. High-purity ZnO nanoparticles from fly ash in copper smelting industry and its application as additive agent in Alkaline Electrolyte; 2011.
2. Rajeev Singh AK, Gorai and Segaran RG. Characterisation of LD slag of Bokaro steel plant and its feasibility study of manufacturing commercial 'fly ash–LD slag' bricks. International Journal of Environmental Technology and Management; 2013.
3. Kumar DA. Utilisation of Wastes from Integrated Steel Plant with Special Reference to India. International Journal of Engineering Research & Technology (IJERT). 2014;3(1):47-52.
4. Ayana YMA, El-Sawy SM, Salah SH. Zinc-ferrite pigment for corrosion protection. Anti-Corrosion Methods Mater. 1997;44: 381-388.
5. Sushan Sarker, Debabrata Mazumder. Solid Waste management in Steel Industry - Challenges and Opportunities. International Journal of Nuclear Energy Science and Technology. 2015;984-987.
6. Kate RO' Brien, Julien Ménaché, Liza M. O'Moore. Impact of fly ash content and fly

ash transportation distance on embodied greenhouse gas emissions and water consumption in concrete. The International Journal of Life Cycle Assessment. 2009; 621-629.
7. Piyush kant Pandey, Rajkumar Agrawal. Utilization of mixed pond ash in integrated steel plant for manufacturing superior quality bricks, Bull. Mater. Sci. 2002;25(5): 443-447.
8. Sear LKA. Properties and use of coal fly ash: A valuable Industrial By-Product, London: Thomas Telford Publishing; 2001.
9. Vassilev SV, Vassilev CG. Characterization of composition of fly ashes from coal-fired power stations: A critical overview. Energy Fuels. 2005;1084-1098.
10. Kassim TA, Williams KJ. Environmental impact assessment of recycled wastes on surface and ground waters in Concepts; Methodology and Chemical analysis, Heidelberg, Springer Verlag Berlin. 2005; 95.
11. Mohapatra R, kanungo SB. Physico-chemical characteristics of fly ash samples from thermal power plants of Orissa. Indian Journal of Engineering and Materials Sciences. 1997;271-281.
12. Chatterjee AK. Indian fly ashes, their characteristics and potential for mechano-chemical activation for enhanced usability in Second International Conference on Sustainable Construction Materials and Technologies, Ancona, Italy; 2010.
13. Prasad B, Banerjee NN, Dhar BB. Environmental assessment of coal ash disposal. Journal of Scientific and Industrial Research. 1996;772-780.
14. Huda L. The effect of material productivity on scrap reduction on aluminum reduction pot process. Materials Science and Engineering. 2018;309.
15. Echlin P. Handbook of sample preparation for scanning electron microscopy and X-Ray microanalysis, Cambridge: Springer; 2009.
16. Dan T. Babor, Diana Pliana, Loredana Judele. Environmental Impact of Concrete. Bulletin of the Polytechnic Institute of Jassy, Constructions. Architecture Section LV (LIX); 2009.
17. Nasdala L, Witzke T, Ullrich B, Brett R. Gordaite: A second occurrence in the Juan de Fuca Ridge and new data. Am. Mineral. 1998;83:1111-1116.
18. Grant R. Zinc production, encyclopedia of materials: Science and technology (Second edition). 2001;9891-9894.
19. Garcia-Valles M, Hafes HS, Cruz-Matı´as I, Verge´s E, Aly MH. Calculation of viscosity-temperature curves for glass obtained from four wastewater treatment plants in Egypt. J Therm Anal Calorim. 2013;107-114.
20. Panna W, Wyszomirski P, Kohut P. Application of hot-stage microscopy to evaluating sample morphology changes on heating. J Therm Anal Calorim; 2016.

Peer-review history:
The peer review history for this paper can be accessed here:
http://www.sdiarticle4.com/review-history/54059