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EVALUATION OF NIGERIA DIRECT REDUCTION (DR) STEELMAKING SLAG MINERAL PHASE CHEMISTRY BY ELECTRON MICROPROBE ANALYSIS (EMPA) FOR INDUSTRIAL APPLICATIONS

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ABSTRACT

Fifteen prepared field samples of Delta Steel Company (DSC) direct reduction (DR) steelmaking slag were studied with an energy dispersive electron microprobe including photo microscopy. Belite, melilite, merwinite (silicates), periclase, perovskite and wustite were the mineral phases present in the slag. Slag composition elements were very highly differentially concentrated in the mineral phases. Mean phase concentration of SiO₂ in, the silicates was (30.69%), periclase (0.04%), perovskite (3.71%), wustite (0.26%); CaO in silicates (50.52%), periclase (0.99%), perovskite (42.38%), wustite (1.44%); Al₂O₃ in silicates (8.78%), perovskite (6.75%), wustite (0.49%); TiO₂ in silicates (0.05%), perovskite (30.45%), wustite (0.16%); FeO in silicates (3.53%), periclase (4.40), perovskite (11.92%), wustite (60.39%); Cr₂O₃ in periclase (0.01%), perovskite (0.54%), wustite (0.66%); MnO in silicates (0.16%), periclase (0.39%), wustite (3.17%); MgO in silicates (4.36%), periclase (94.97%), perovskite (0.41%), wustite (33.27%); Na₂O in silicates (0.05%), periclase (0.61%), wustite (0.59%); P₂O₅ in silicates (0.67%), perovskite (0.30%); V₂O₅ in wustite (0.01%). This overall high elemental concentration of the slag and varying concentrations in the different mineral phases confirming its similarity with cement clinkers, made it useful in the blast furnace iron and steel making, slag ceramics, slag cement and phosphate fertilizer industries.

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INTRODUCTION

Delta Steel Company (DSC) Ovwian-Aladja, western Niger Delta, Nigeria, used the direct-reduction (DR) process in steelmaking slag production, with direct reduced iron (DRI), some scrap iron as ingots or raw materials and lime from Mfamosing Limestone as flux. Electron microprobe analysis (EMPA) has been used since the early 1960's as one of the best techniques for the investigation of the mineral chemistry of Portland cement clinker. Early microprobe analysis was presented by Midgley, (1964) as intensity maps of individual elements in different cement minerals. Moore, (1965), produced images of element distribution by atomic number of the aluminate phase (C₃A), using the output of a detector which was sensitive to back scattered electrons. He obtained some quantitative measurements of the magnesium content by holding the electron beam stationary and recording the X-ray count for a fixed time period using olivine with a known content of MgO as a standard.

The 13% high value for C₃A found was attributed to the unintentional analysis of some periclase within the aluminates. Peterson, (1967), gave variable values for Al₂O₃, K₂O, Na₂O, and SO₃ in the alite and belite grains which had uncertain correction procedures. Regourd and Guinier, (1974) investigated and reported on the crystal chemistry of the constituents of Portland cement and; Yamaguchi and Tagaki, (1968) used EMPA to measure the Al₂O₃, FeO, MgO, Na₂O, and K₂O in alite and belite in Japanese Portland cement clinker. The standard samples used were made from lithium borate glasses with 50% by weight of the constituent added to the specific phase to calibrate the analyser. The values of the analyser were very variable, therefore, the standards and samples were analysed at the same time with the same sample holder to give reproducible results. Steelmaking slags were found to be chemically and mineralogically similar to Portland cement clinkers (Kristmann, (1977); Kubodera, *et al.*, (1979). A variety of mineral phases that have reportedly been identified in steelmaking slags included alite, aluminate, belite, melilite, merwinite (silicates), periclase, perovskite, wustite, free lime and in the presence of water, portlandite may be present (Wachsmuth & Geiseler, 1981; Monaco & Wu, 1994; Geiseler, 1996; Luxán *et al.*, 2000; Barra, *et al.*, 2001; Qian,

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2002; Shi, 2002; Juckes, 2003; Shen *et al.*, 2004; Manso *et al.*, 2006; Yildirim & Prezzi, (2011). The elemental composition of each of the phases varied with some preferential distribution of some elements into some phases in Portland cement and steelmaking slag clinkers (Midgley, 1967; Moore, 1965; Peterson, 1967; Medland, 1983; Wessey, 1988). In this investigation, the energy dispersive X-ray spectrum electron microprobe was used to determine the mineral phase chemistry of the Delta Steel Company, steelmaking slag. EMPA technique made it possible to utilize a one micron diameter beam to carry out a series of analysis across a specific crystal in a polished thin section or block. It provided the information on possible elemental substitution into the mineral structure and indicated the partitioning of element between the main phases.

MATERIALS AND METHODS

Fifteen field spot samples were collected and taken to the laboratory for investigation. Carbon (6) coated flat surface polished thin sections and blocks of the samples of the slag were prepared. Photomicrographs of each sample were produced using reflected mode of a zeiss ultraphot microscope, followed by electron microprobe analyses (EMPA) using the Cambridge Instruments 'GEOSCAN' with Link System Model 290 2KV energy dispersive spectrometer attached. Beam conditions were, accelerating potential of 15kv, specimen current of 2.7mA and the instrument conditions were, ke-vex detector resolution of c. 150 eV, take off angle of 75°, Live time of 100 live seconds. The standard- cobalt metal block and carbon coated polished thin sections and blocks samples were used.

Data processing for analysis

A data collection processing program was stored on disk system. The signal was processed by a Harwell processor model 2010 and converted to a digital format. The digitized information was stored in a 16k Nova 2-10 computer. The software was from link using a ZAF4 program. Carbon 6 coating was to prevent the non-detection of low atomic number elements in the analysis system and to prevent the beam charging the specimen.

Analysis

The bombarding electron beam was focused on small spots of 1µm approximate diameter each for normal operating conditions to enable quantitative analysis of selected phases in the specimens which were carefully prepared. A phase being studied was always larger than the diameter of the bombarding beam and its depth of penetration for the accurate chemical analysis of the phase. This enabled the analysis free of any contamination by another phase below or adjacent to the crystal under investigation. The stoichiometry of the system phases and the partitioning of the minor elements in the individual phases were also deduced using the electron microprobe analysis results. Information from smaller phases, were obtained by plotting the EMPA analyses on ternary phase diagrams from which compositional trends were deduced (Scott *et al.*, 1986; Wessey, 1988.).

RESULTS

DSC slag mineral phases, their electron microprobe (EMPA) analyses elemental oxide and elemental concentration analyses results and element detection limits in per centage (%) are shown in Table 1.

DISCUSSION

The element oxide and element composition of the slag mineral phases were determined by EMPA. The mean chemical composition of each phase is presented in Table I which also shows elemental detection limits of the slag. Detailed discussions of the chemistry and mineralogy of steelmaking slags are presented in published works of Fletcher, 1968); Norish and Hutton (1969); Gutt *et al.*, (1974); Emery, (1977); Wessey, (1988). From Table I the preferential distributed concentration in the mineral phases was evident. Belite, merwinite and melilite were the silicate mineral phases present in the slag. The electron microprobe analyses (EMPA) result for iron for DSC slag was reported as FeO (Table I). The possible industrial applications of the mineral phases on extraction or separation from the slag are discussed below.

Belite (Dicalcium silicate phase)

Table I showed that the mean major element oxide/element per centage compositions in DSC slag belite were CaO (61.102), SiO₂ (33.036), P₂O₅ (1.320), FeO (1.218), and MgO (1.164), usual constituents of belite (Barnes *et al.*, 1972; Sarka, (1977). This was found to be similar to that of Portland cement belite reported by (Nurse, 1952; Fletcher, 1968). This oxide concentration result was an indication that the slag belite was rich in Ca, Si, P, Fe and P, known major elements necessary for slag use as a blastfurnace feed (Thom and Wood, (1973; Lee, 1974; Pugh and Fletcher, 1974; Emery, 1977). and fertilizer (Dippenaar, 2004; Torkashvand, 2011; Yildirim and Perez, 2011; Branca *et al.*, 2012). It can therefore be extracted or separated from DSC slag for use in such industries. The slag belite had the preferential distribution and concentration of P (P₂O₅ (1.320%). The concentration of Al₂O₃ in the slag was low and therefore cannot create any danger of sulphate attack due to hydration if used in the stated industrial applications. The presence of or substitution of minor elements into belite can increase its hydraulicity for use in slag cement (Welch and Gutt, (1960; Shi, 2002, Tsakiridis *et al.*, al. 2008; Waligora *et al.*, 2010).

Merwinite

The analyses of all samples in the slag showed close similarity in results with means of CaO (49.882%), SiO₂ (34.181%), MgO (9.038%), P₂O₅ (0.699), FeO (3.411%), and Mn (0.252%); and sometimes Na₂O (0.38%) which was an industrially necessary composition for merwinite. This indicated that an extracted or separated merwinite can be suitable for applications in the iron and steelmaking blastfurnace, slag cement and fertilizer industries. The detection of Fe in merwinite indicated its presence as cryptocrystalline inclusions (Scott *et al.*, 1986). There was consistent deficiency of Mg in the merwinite formulae (Mg average 0.76% per formular unit), some of which may be accounted for by Fe and probably Al substitution for Mg. The high Fe merwinites contained least Mg.

Table 1. DSC slag mean mineral phase chemistry by EMPA and oxide detection limits in per centage (%)

Oxides and elements	DSC slag mineral phases						*Oxide detection limit (%)
	Belite	Merwinite	Melilite	Magnesio-Wustite	Ferro-Periclase	Perovskite	
SiO ₂	33.036	34.181	24.538	0.264	0.040	3.710	0.2
Al ₂ O ₃	0.056	0.744	25.538	0.490	0.000	6.750	0.01
FeO	1.218	3.411	5.947	60.391	4.399	11.917	0.1
Cr ₂ O ₃	0.002	0.000	0.000	0.656	0.014	0.537	0.01
MnO	0.114	0.252	0.113	3.169	0.385	0.000	0.01
MgO	1.164	9.038	2.876	33.274	94.973	0.413	0.4
CaO	61.102	49.882	40.577	1.442	0.992	42.377	0.1
Na ₂ O	0.049	0.094	0.000	0.592	0.671	0.000	0.04
P ₂ O ₅	1.320	0.669	0.021	0.000	0.000	0.297	0.05
SO ₃	0.000	0.000	0.000	0.000	0.000	0.000	0.2
TiO ₂	0.011	0.029	0.103	0.156	0.000	30.450	0.1
Si	0.962	1.937	1.113	0.002	0.000	0.091	
Ti	-	-	-	0.001	0.000	0.561	
Al	0.002	0.049	1.468	0.006	0.000	0.195	
Fe ₂	0.030	0.162	0.261	0.482	0.025	0.244	
Cr	-	-	-	0.005	0.000	0.010	
Mn	0.003	0.012	0.004	0.025	0.002	-	
Mg	0.050	0.758	0.202	0.449	0.961	0.015	
Ca	1.908	3.037	2.103	0.015	0.007	1.113	
Na	0.003	-	-	0.011	0.009	-	
P	0.032	0.034	-	-	-	0.006	

*Oxide detection limits in electron microprobe analysis (EMPA) (Dunham and Wilkinson, 1976)

This arguably suggested the non-stoichiometry of merwinites with lattice vacancies occurring in the positions normally occupied by Mg atoms.

Melilite

The EMPA analyses results of the slag melilite showed some variations. The mean result however showed that it was gehlinitic (2CaOAl₂O₃, SiO₂) rich, consisting of CaO (40.577%), Al₂O₃ (25.538%) and SiO₂ (24.538%). Comparatively, the concentrations of FeO (5.947%), MgO (2.876%), were very low and the amounts of TiO₂ (0.103%); MnO (0.113%) and P₂O₅ (0.021%) present were very minor. Na and K were not detected and if present the concentrations were, may be, below detection levels (Dunham *et al.*, (1976). From the analyses results the melilite composition can be extracted or separated for applications in the blast furnace, slag cement, slag ceramic and fertilizer industries.

Wustite (Magnesio-Wustite)

Wustite was the very high iron bearing phase (FeO, 60.391%) in the slag, had concentrations of MgO (32.274%), Mg, (0.449%); and Cr₂O₃ (0.656%), Cr (0.005%) considered high, and Mn, a minor constituent was preferentially distributed into the phase (Table1). It also contained some minor amounts of Si, Al, Ti, Ca, and Na as SiO₂ (0.264%); Si (0.002%); Al₂O₃ (0.490%); Al (0.006%), TiO₂ (0.156%); Ti (0.001%); CaO (1.442%); Ca (0.015%) and Na₂O (0.592%); Na (0.011%) respectively. The high content of Mg in DSC slag wustite resulted in naming it as Magnesio-Wustite (Mg-Fe). The high Fe content combined with the considerable content of Mg indicated that, wustite can be extracted or separated for use as blastfurnace feed. The wustite can also be suitable for iron fertilizer (Wang and Cai, 2006; Abbaspour, *et al.*, 2005).

Periclase (Ferro-Periclase (Fe-MgO))

The periclase in the slag had the highest concentration of Mg as MgO (94.973%); Mg (0.961%) (refer to Table 1). There was also a high concentration of Fe as FeO (4.399%); Fe₂ (0.025%), giving it the name ferro-periclase.

Some Fe may have substituted for Mg, just as Na and Ca may have substituted for Mg arising from non-stoichiometry of the periclase. The high MgO content can be used as an additive in Portland cement (Altun, and Yilmaz, (2002).

Perovskite

The perovskite of the slag had the preferential distribution concentration of all the detectable Ti as TiO₂ (30.450%); Ti (0.561%) and had a comparatively very low concentration of Cr as Cr₂O₃ (0.537%); Cr (0.10%).

Glass

The glass of the slag showed a range of compositions similar to that of melilite anywhere glass was detected. This indicated that the growth of larger analyzable silicate crystals was by free fluid movement, rather than by diffusion- controlled mechanism (Scott *et al.*, 1986, Wessey, 1988).

Summary of element oxide concentrations in DSC slag mineral phases

Belite, merwinite and melilite were the identified silicate mineral phases in the slag which competed for element oxide/element distribution. The overall mean oxide (%) concentrations in these minerals were SiO₂ in silicates (30.69%), perovskite (3.71%), wustite (0.26%) and periclase (0.04%); CaO (lime) in silicate (50.52%), perovskite (42.39%) wustite (1.44%), and periclase (0.99%); Al₂O₃ in silicates (8.78%), perovskite (6.75%), and wustite (0.49%); TiO₂ in perovskite (30.45%) wustite (0.16%), and silicates (0.05%); FeO in wustite (60.39%), perovskite (11.92%), periclase (4.40%), and silicates (3.53%); MnO in silicates (0.16%), wustite (3.17%) and in periclase (0.39%); MgO in periclase (94.97%), wustite (33.27%), silicates (4.36%), and perovskite (0.41%); Cr₂O₃ in wustite (0.66%), wustite (0.59%), perovskite (0.54%), periclase (0.01%), and silicates (0.00); Na₂O in periclase (0.61%), silicates (0.05%); P₂O₅ in silicates (0.67%), and perovskite (0.30%); and V₂O₅ detected only in wustite was (0.01%). Some element oxides/elements present may have been in lower than detection level concentrations (Table1).

Preferential element oxide/element distribution into the mineral phases

The results of EMPA study of DSC slag mineral phases (Table 1) evidently showed the preferential element oxide/element distribution as follows:

- SiO₂/Si into the silicate (30.69%)
- CaO/Ca into silicates (50.52%) and perovskite (42.38%)
- TiO₂/Ti into perovskite (30.45%)
- FeO/Fe into wustite (60.39%) and perovskite (11.92%)
- MnO/Mn into wustites (3.17%)
- MgO/Mg into periclase (94.97%) and wustite (33.27%)
- Cr₂O₃/Cr into wustite (0.66%)
- Na₂O/Na into periclase (0.61%)
- P₂O₅/P into silicates (0.67%) and perovskite (0.30%)
- V₂O₅/V into wustite (0.01%).

Material application

EMPA results of test slag indicated that the slag was very rich in major steelmaking elements from belite, melilite and merwinite silicates, wustites, periclase and perovskite in terms of Si, Ca, Fe, Mg, and Ti. These elements in the slag are required for the blast furnace iron and steelmaking process (Thom, and Wood, (1973), Pugh and Fletcher, 1974 (Ray *et al.*, 1974); Joseph and Haddad, 1975; slag ceramics (Qian, 2002), slag cement (Spellman, 1980, Shi, 2004; Tsakiridis, *et al.*, 2008; Reddy *et al.*, 2006, Shi, 2002) and agriculture (Negim, 2010, Torkashvand, 2011; Branca *et al.*, 2012). CaO can be a fluxing and mineral forming agent in reaction with unwanted elements from the iron making ingots in the blastfurnace. The silicates can be major sources, of Si, Ca and Fe necessary for use in iron and steelmaking blastfurnace as feed and in the agricultural industry as fertilizer. Belite in the slag had hydration properties as shown by the presence of beta-belite and therefore suitable for slag cement manufacture (Barnes *et al.*, 1972; Atwell, 1974; Wessey, 1988). Ca can be a liming agent in agriculture. Perovskite contained a good proportion of Ca and can be combined with the silicates for use as fertilizer. Wustite can be a major source of Fe for use as blastfurnace feed and as fertilizer combined with perovskite. Wustite, had some MnO/Mn, suitable for use in the iron and steelmaking blastfurnace to minimize Mn addition costs. Wustite can also be a source of V, but in very low contents which may not cause any deleterious effects when used as fertilizer. Periclase and wustite were the major sources of MgO/Mg suitable for use as fertilizer in the agriculture and slag ceramics industries. The silicates and perovskite were sources of agriculture important phosphorus (P), possibly for direct application or used to produce phosphate fertilizer. These mineral phases in the slag can be separated using standard mineral separation methods for use in different industries.

Conclusion

Nigerian Direct reduction steelmaking slag had a rich and valuable mineral phase chemistry as obtained from electron microprobe analyses. The mineral phases can be separated using standard mineral separation methods and used individually or in combination in the iron and steelmaking blastfurnace, slag ceramics, slag cement and the agriculture industries.

The use of slag as a result of its mineral phase chemistry can conserve natural resources and rid the environment of physical pollutants.

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