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FEASIBILITY OF NICKEL EXTRACTION FROM INDIAN CHROMITE OVERBURDEN BY SOLID STATE REDUCTION AND SMELTING ROUTE

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Abstract

The present work demonstrates the extraction of nickel from low-grade chromite overburden by using solid state reduction and direct smelting route. Goethite & Quartz are present as major phases whereas chromite, hematite were identified as minor phases in the mineral. Solid state reduction of pellets were carried out inside a horizontal tube furnace at 1000°C, 1200°C, 1400°C for 30, 60, 90 and 120 minutes respectively with creating reducing atmosphere. Pellets of varying basicity (i.e. 0.5, 0.6, 0.7, 0.8 and 0.9) were used directly in the EAF for smelting studies. Highest percent of nickel (2%) having ~ 91% recovery were obtained in solid state reduction route for pellets which was reduced at 1400°C for 120 minute. Similar recovery (~90%) of nickel was obtained inside the ingot (0.67% Ni) by using pellets of 0.9 basicity through smelting route. From the present investigation, it could be concluded that the solid state reduction as well as smelting routes are feasible for the recovery of nickel from low grade chromite overburden. The production of nickel pig (low grade ferronickel) could also be feasible by smelting route.

Keywords: *Nickel pig; Solid state reduction; Smelting; Chromite over burden; Recovery*

1. Introduction

Traditionally nickel is extracted from laterite and sulphide nickel ores. Around 70% nickel occurs as a lateritic ore reserves, but 60% of the world production of nickel is coming from sulphide ores and rest comes from nickel laterite ores. Currently due to rapid depletion of sulphide ores and raising demand of stainless steel production, the world is focusing towards laterite ore treatment technology for the extraction of nickel. Demand for stainless steel increasing day by day so there is a huge amount of nickel is required and the shortage of nickel also will happen [1]. There are no reserves of nickel in India, so Indian markets purely depend upon imports for the entire annual requirement with a lot of costs. Sukinda (Orissa, India) chromite mines are generating huge amounts of overburden along with the production of chromite ore. About 5-10 million tons of overburden is generating every year in opencast chromite mining and estimated 140 million tons deposits of chromite overburden existing and storing as dumps in huge volume [2,3]. Storage of huge amount of overburden creates environmental problems like soil erosion, water pollution etc. This overburden contains nickel (0.5-1% approximately) as lateritic (limonitic type) form up to satisfactory level with other metals like iron, chromium etc. So effective utilization of this overburden is required. The primary mineral phases in the overburden ore are goethite, quartz, hematite, chromite etc. Nickel is mainly bounded with Goethite [4-6]. Due to complex mineralogy and heterogeneous mixture of ore, it is quite difficult to extract nickel.

During laterisation/weathering process Ni, Mg and Si are leached from the rocks so in this process iron with nickel is oxidized near to the surface and precipitates as goethite/hematite. Chemical and mineralogical composition of laterite/oxide ores are differ with respect to SiO_2/MgO, Fe/Ni weight ratios as well physical and chemical water contents. Based on composition and mineralogy laterites/oxide ores are classified as five zones shown in Table 1 [7, 8].

Hydrometallurgy methods are primarily using High pressure acid leaching (HPAL) method and Atmospheric acid leaching method (AL) to extract nickel from limonitic lateritic ores. By applying suitable leaching parameters studies investigated that maximum 95% nickel was recovered from pre-

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Table 1. *Nickel Laterite Ore Zones [9]*

Laterite Zone Type	Approximate Analysis (%)			
	Ni	Fe	Cr_2O_3	MgO
Ferricrete	<0.8	>50	>1	<0.5
Limonite	0.8-1.5	40-50	2-5	0.5-5
Nontronite	1.5-1.8	25-40	1	5-15
Saprolite	1.8-3	10-25	1-2	15-35
Peridotite	0.3	5	0.2-1	35-45

reduced limonitic laterite ore at mild temperature and pressure. But this hydrometallurgy methods are drawbacks of requires high acid consumption, descaling and corrosion of reactor walls, safety controls [10].

Rotary kiln-electric arc furnace (RKEF) process is most accomplished method to treatment of massive scale low grade nickel oxide laterite ores. Alternative methods are looking to RKEF process as its suffering of high energy consumption which is seen as a big drawback. Vaniukov process, lowgrade-FeNi production, ESS Tecnology, ISASMELT TSL process and NST are most recent developing methods for the replacement of RKEF process. Selective reduction at low temperatures (1000-1300^{0}C) followed by magnetic separation process are current hotspot process & eco-friendly process among the recently developing process as its very less cost effective industrially too for treating low grade laterite ore [11].

Some Chinese plants produces pig iron with changing their production methods into NPI production [12, 13].The process is similar to pig iron production but raw ore contains high amount of nickel. The use of electric arc furnace expects more amount of nickel content with less operational costs [1].

The feasibility for direct reduction process of low grade chromite overburden from Sukinda mines was studied and found >90% Fe and <30% Ni was recovered from pyro-metallurgical route processed nuggets [2]. Though various investigations of utilization of chrome overburden for iron making processes have been carried out, the processes which are economically feasible has not been established in India till today [2-6, 14, 15].

For Laterite ore smelting, slags can be expressed by the $FeO-Fe_2O_3-SiO_2$ system. The final nickel bearing product depend on the melting point of slag, which is affected by the SiO_2/MgO ratio of the ore. For an ore with low melting point of slag, with SiO_2/MgO ratio in the range of 1.8-2.2, the production of nickel matte is more preferable. High melting point slag ore, SiO_2/MgO ratio either <2 or >2.5, are suitable to produce ferronickel/nickel. Ores having SiO_2/MgO ratio between 2.3-2.5 are very corrosive to the furnace lining and require a modification to the feed chemistry prior to smelting [16, 17].

Phase equilibria and solution thermodynamic studies play a major role to extract metals from complex ores. For the production of metallic species from nickel metal oxide (laterite) and sulfide, reduction atmosphere of calcium sulfate (gypsum) and lime was used to investigate reduction aspects of complex minerals.

The main objective of this study is to make effective utilization of the low grade chromite overburden from Sukinda mines for the extraction of nickel either as a nickel sponge through solid state reduction or as a nickel pig by smelting route.

2. Experimental

2.1 Raw materials and its characterization

The low grade chromite overburden sample for the present studies was collected from Sukinda chromite mines, India. Chemical analysis of chromite overburden showed iron oxide, chromite and silica in a major quantity. The average nickel content was around 0.55 wt. %. Coal, lime and bentonite were supplied from the ferroalloys and minerals development division, R&D Tata Steel, Jamshedpur. The Chemical composition, proximate and ultimate analysis of raw materials is given in the Table 1 and Table 2 respectively.

Table 2. *Chemical analysis of raw materials*

Raw Materials (Wt. %)								
Components	Fe(t)	Ni	SiO_2	MgO	Cr_2O_3	Al_2O_3	CaO	LOI
Chromite Overburden	37.93	0.55	11.4	2.83	16.2	4.09	0.009	8.74
Lime	0.05	-	-	-	-	-	97.5	-
Bentonite	7.7	-	58.0	1.7	-	5.1	1.05	-

Table 3. *Proximate and ultimate analysis of coal*

Proximate and ultimate analysis of coal (wt. %)								
Component	Fixed Carbon	Volatile Matter	Moisture	Ash	CaO	SiO_2	P	S
(%)	80.8	1.76	4.6	7.8	1.83	1.27	0.003	0.022

X-ray diffraction (XRD-Rigaku Miniflex-600 model with Dtex ultra detector and Cu-Kα radiation λ=1.54nm, acceleration voltage = 40V, current = 15mA) technique was used for determining the different mineral phases present in the chromite overburden ore.

The samples were scanned at a rate of 2°/ minute from 20 to 80°. XRD peak of chromite overburden is shown in Figure 1. The minerals identified from XRD data are quartz, goethite and chromite as major phases and hematite, iron, silicate as minor. There are no

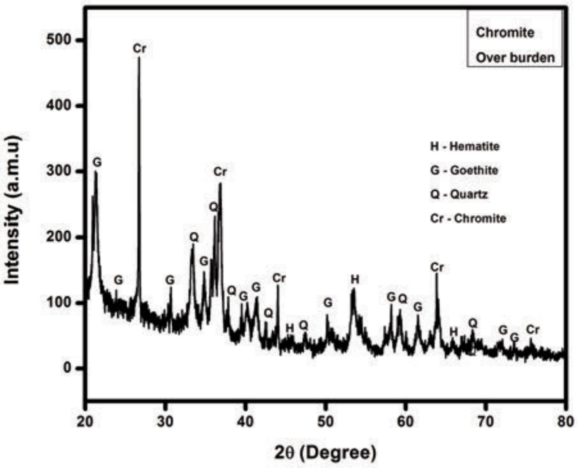


Figure 1. X-ray diffraction analysis of overburden material

direct nickel phase peaks observed in the overburden but Goethite is the major mineral phase for nickel [4, 5]. Most of the nickel in Sukinda chromite overburden was lattice bound with Goethite as it was reported by other researchers [2-6]. XRF technique was used to determine the elemental analysis of raw material. Chemical composition analysis showed that the iron was present at 37.93%, chromium oxide 16.2%, and nickel 0.55% in the overburden. Proximate analysis is used to determine the different constituents present in the coal. Carbon present in the coal was 80.8 %.

2.2. Methodology

2.2.1. Raw material preparation

Due to complex nature of the chromite overburden minerals, it is very difficult to concentrate nickel content by using conventional mineral beneficiation techniques. Therefore, grinding and screening process was used to convert the raw materials (ore, coal and lime) in to fine powder of (<74 µm) size. Green composite chromite overburden ore pellets of 12 mm diameter were prepared with the addition of coal and bentonite for the solid state reduction. For the smelting reduction studies, chromite overburden pellets were made with varying basicity (0.5, 0.6, 0.7, 0.8 and 0.9) without addition of coal. The both type of green pellets were dried in the air before oven dry at 110°C for 2hrs.The oven dried pellets have an average weight of 6.20 gram and diameter of 12mm. Process flowchart for both solid state reduction and smelting studies is shown in Figure 2.

2.2.2 Solid State Reduction

The Solid state reduction experiments were carried out in a horizontal alumina tube furnace as shown in Figure 3. Carbon monoxide (CO) gas was introduced at a rate of 0.5 liter min^{-1} throughout the experiments as a reducing gas to maintain reduction atmosphere considering reduction of nickel oxide proceeds at low temperature. Carbon was incorporated as coal to accelerate reaction rate, kinetics because at lower temperatures in solid state reduction yield of metal can be low. Nitrogen (N_2) gas was also passed after finishing of each and every test for stabilizing the inert environment inside the furnace before power off. A series of reduction experiments were performed at different temperature, i.e.1000°C,1200°C, 1400°C for exposure of 30, 60, 90 and 120 minute time respectively. The % reduction of each set is calculated by taking the ratio of weight loss due to removal of oxygen to the wt. of total oxygen present in the pellets.

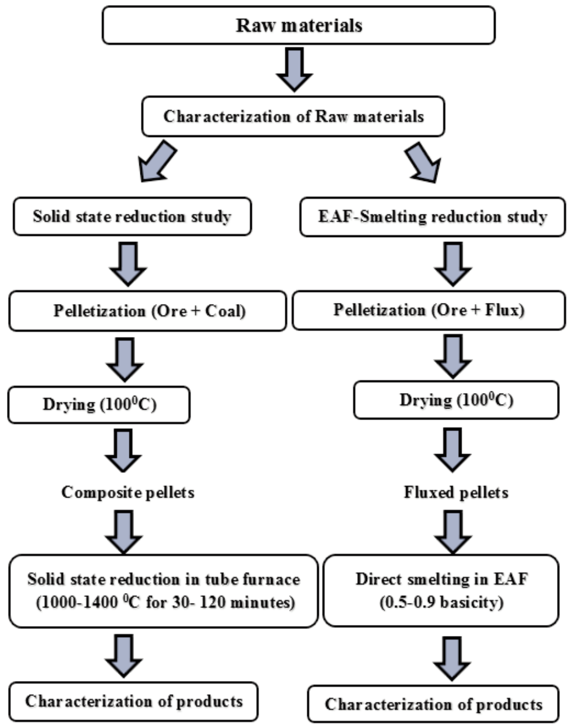


Figure 2. Process Flow sheet of Solid state reduction and EAF-smelting route

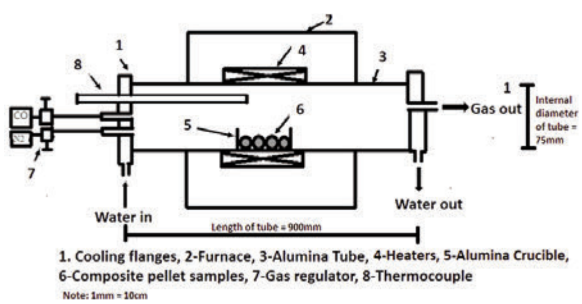


Figure 3. Schematic diagram of horizontal tube furnace for solid state reduction studies

2.2.3 Smelting Reduction

The smelting experiments were carried out in an electric arc furnace to know the recovery of nickel from fluxed pellets as a form of nickel pig iron (low grade ferronickel). During each heat, small pieces of mild steel scrap were charged inside the furnace for making melt pool. After achieving temperature 1550-1600°C, different types (basicity) of pellets were charged separately. After complete dissolution of pellets, casting of the melt was done in the graphite crucible. The recovery of metal was calculated based on the initial and final chemical composition of original charged raw materials and the obtained ingot material respectively.

3. Results and discussion

3.1. Solid state reduction studies

3.1.1. Reduction behavior of pellets

Reduction behavior of chromite overburden was shown in Figure 4.

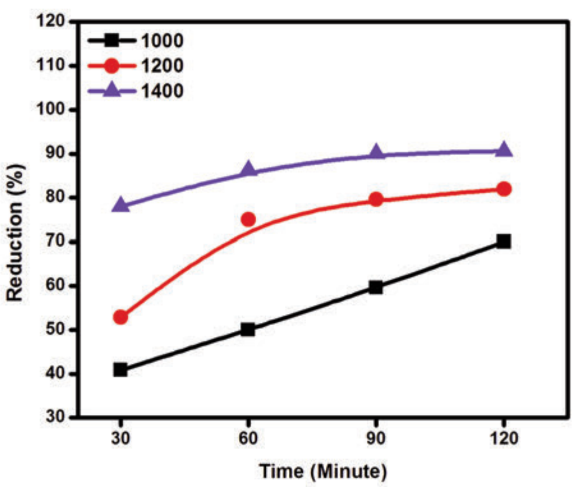


Figure 4. *Reduction behavior of chromite overburden*

Figure 4 shows that lower temperature in the initial stage percentage of reduction was not so significant compared to higher temperature. Percentage of reduction increases continuously with increasing temperature and time. At higher temperature, maximum reduction was achieved in less time compared to lower temperature. At temperature 1400°C, after 90 minutes of exposure no significant changes in reduction was observed, which indicated that the reduction reaction moved towards the completion. This may be due to the presence of nickel with goethite phase, as reduction temperature and time increases dissociation of goethite increased as well, which provided more free sites for the reduction reaction. Due to this iron ore and nickel reduces simultaneously and increased the metallic content of the overall raw material. The highest percentage reduction (91%) of nickel was observed for 120 minute time, but the major reduction (>90%) completed in 90 minutes at 1400°C. Similar type of results obtained by P.Ju et.al at temperature of 1300^0C after crushing and screening the slag. Maximum Nickel recovery of 85% was observed at 0.7 C/O ratio and 8% CaO (8%) at low temperatures from iron-nickel nuggets. This methods suggested that, suitable C/O ratio and CaO addition played crucial role for high nickel ratios and high nickel recovery [18].

3.1.2. Phases analysis of reduced pellets

XRD analysis of reduced pellet is shown in Figure 5a-c. As increasing the percentage reduction, metal value of nickel percentage also increased which was confirmed by the SEM-EDX analysis. Slag and metal separation was observed clearly at temperature of 1400°C. The highest 91% Ni, 90% Fe, and 20% Cr recovery was found at 1400°C.

The presence of nickel with chromium, and iron in oxide form, confirmed that nickel originated from the iron oxide lattice. Such a peak was not observed for unreduced pellets. Based on the gradual reaction principle, the reduction process of iron oxides included three stages above 570°C, $Fe_2O_3 \rightarrow Fe_3O_4 \rightarrow FeO \rightarrow Fe$, the reduction temperature varied from 1000°C to 1400°C (Kapure et al. 2010; Rama Murthy et al. 2010).The formation of metal became more rapid at 1200°C and 1400°C, indicating by high peak

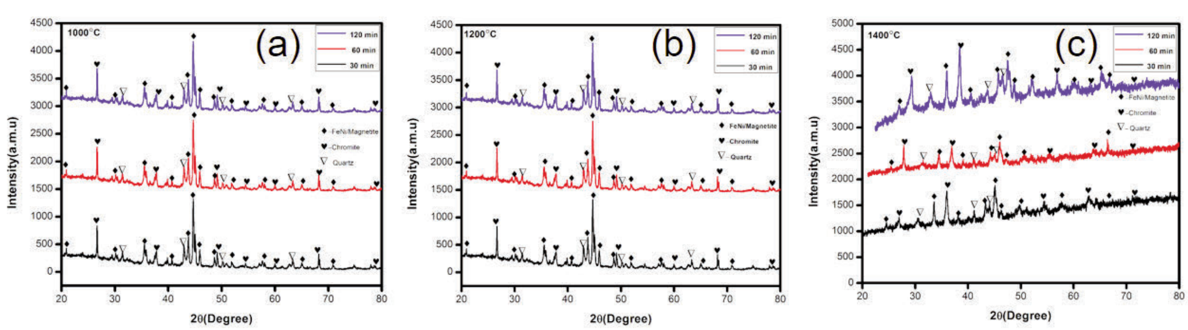


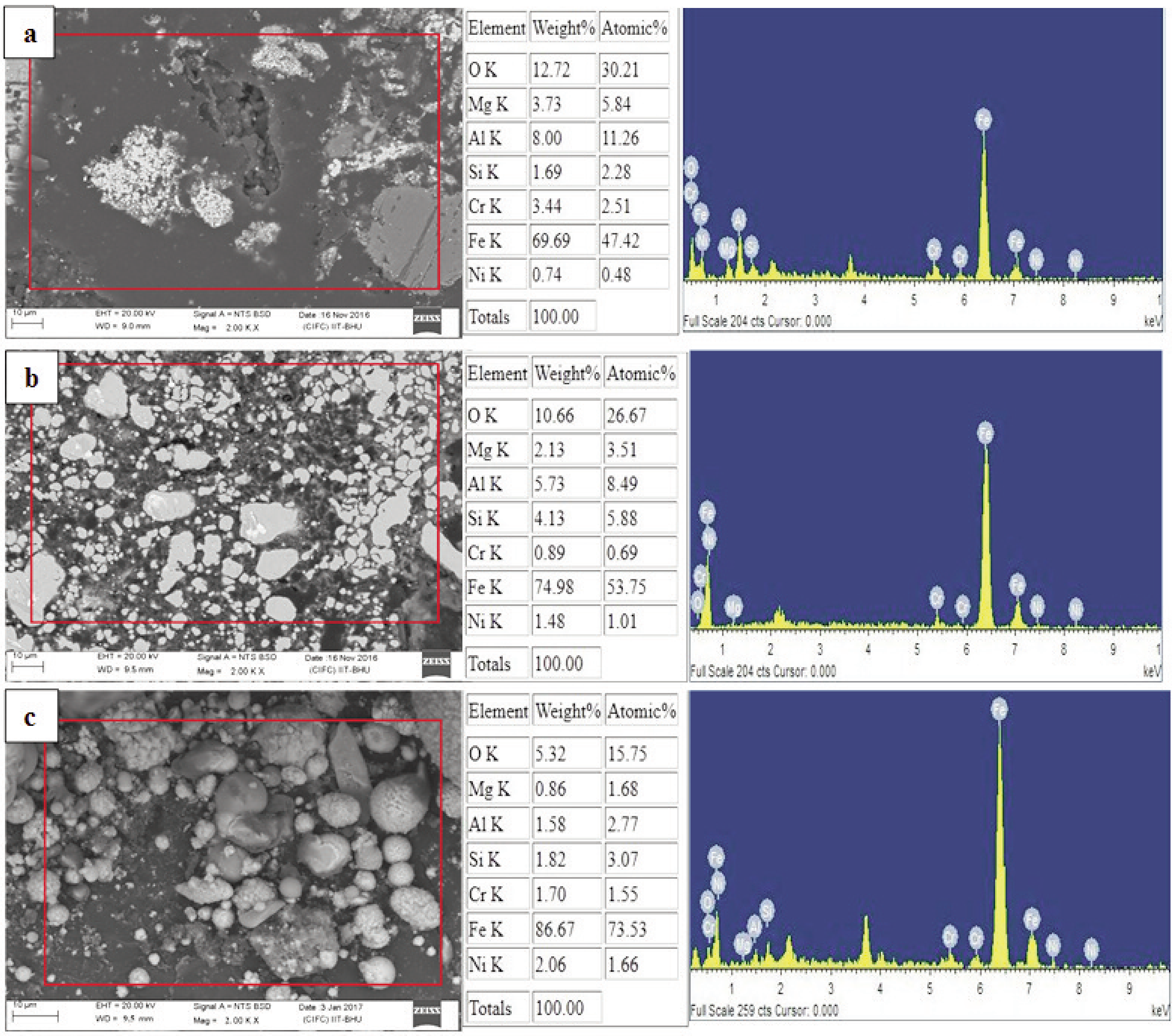
Figure 5. *X-ray diffraction analysis of reduced chromite overburden (a) at 1000°C, (b) at 1200°C and (c) at 1400°C*

intensity corresponding to iron nickel intermetallic because at higher temperature more dissociation of oxides took place which provided more and more free sites for the further reaction. It can be observed from the XRD peaks, that the maximum number of metallic phases was formed at 1400°C and primary phases at all temperatures were chromite, quartz and magnetite. At 1000°C, goethite was transformed into magnetite and iron-nickel intermetallic. With increasing the reduction time, formation of metallic phases also increased.

3.1.3 SEM and Elemental analysis of reduced chromite overburden pellets

Morphology and distribution of different elements in reduced chromite overburden pellets were determined by using SEM with EDX and their patterns are shown in Figure 6.

Considering the large quantity of the matrix in the reduced ore sample, by reconciling with the XRD results and SEM images, it was concluded that the matrix was a mixture of fine grain magnetite (transformed from goethite after reduction) with some silicate, and chromite minerals. After reduction, some goethite and limonite composite particles were transformed to a porous magnetite particle interspersed with remaining Fe silicate. It was expected that at least some of the ferronickel alloy would be formed at the surface of the Ni bearing particles and the surface of the magnetite particle in the reduced ore [19, 20]. EDX Analysis was used to confirm the presence of nickel, iron and chromium. Enrichment and increment of nickel concentration compared to raw chromite overburden was observed by the analysis of EDX. Content of nickel also increased with increasing temperature due to more reduction of nickel with iron oxide present in goethite with respect to other oxide of silicon and chromium during reduction process.



(a)

Element	Weight%	Atomic%
O K	12.72	30.21
Mg K	3.73	5.84
Al K	8.00	11.26
Si K	1.69	2.28
Cr K	3.44	2.51
Fe K	69.69	47.42
Ni K	0.74	0.48
Totals	100.00	

(b)

Element	Weight%	Atomic%
O K	10.66	26.67
Mg K	2.13	3.51
Al K	5.73	8.49
Si K	4.13	5.88
Cr K	0.89	0.69
Fe K	74.98	53.75
Ni K	1.48	1.01
Totals	100.00	

(c)

Element	Weight%	Atomic%
O K	5.32	15.75
Mg K	0.86	1.68
Al K	1.58	2.77
Si K	1.82	3.07
Cr K	1.70	1.55
Fe K	86.67	73.53
Ni K	2.06	1.66
Totals	100.00	

Figure 6. *SEM-EDAX analysis of reduced chromite overburden at 120 min (a) at 1000°C, (b) at 1200°C and (c) at 1400°C*

3.2. Smelting Reduction of chromite overburden fluxed pellets

3.2.1. Chemical analysis of smelted products (Ingot)

After being dried, the samples were smelted at 1500°C-1600°C to obtain nickel pig. After being smelted, metal and slag were found to be two different phases that can be distinguished. The separation of these phases can be done mechanically by using a hammer. Chemical analysis (SEM-EDS) of metal sample and slag samples after smelting reduction for different basicity pellets is shown in Table 4 and Table 5 respectively.

Table 4. *Chemical composition results of EAF-Melted chromite overburden metal samples at different basicity*

Sample	Chemical analysis, Wt. (%)								
	Fe(T)	Ni	Cr	Si	Mn	P	S	C	Al
0.5 Basicity	87.5	0.36	6.38	0.24	0.53	0.12	0.08	4.05	0.02
0.7 Basicity	90.1	0.40	4.01	0.21	0.22	0.06	0.08	2.12	0.02
0.8 Basicity	91.0	0.46	2.67	0.20	0.15	0.06	0.09	5.07	0.03
0.9 Basicity	91.3	0.68	2.50	0.19	0.20	0.05	0.10	5.28	0.04

Table 5. *Chemical composition of EAF-melted slag samples at different basicity*

Sample	Chemical analysis, Wt. (%)									
	Fe(T)	FeO	CaO	SiO_2	MgO	MnO	Al_2O_3	Cr_2O_3	P	Ni
0.5 Basicity	7.70	8.26	8.27	29.18	34.33	2.31	11.41	3.67	-	-
0.7 Basicity	9.24	10.84	8.25	23.84	24.50	2.70	10.71	17.10	-	-
0.8 Basicity	13.01	14.45	7.82	17.32	27.64	2.37	8.76	17.78	0.13	0.38
0.9 Basicity	16.32	14.71	6.4	13.98	29.31	2.07	6.66	17.88	0.05	0.03

The SEM-EDX analysis, shows the increasing trends of nickel and iron with the basicity increase. Maximum values of nickel and iron was observed at 0.9 Basicity. With the increase of basicity, recovery of iron and nickel increased but composition of chromium and its recovery was poor. Phosphorous, sulphur and silicon content was very low so this product could be directly used for making alloy steels, stainless steels and as a coolant in stainless steel making.

The presence of Ni, Fe and Cr was observed from the elemental analysis of EAF-melted chromite overburden metal samples. The highest value of nickel was 0.68% at 0.9Basicity. 86% Fe, 90% Ni and 30%Cr recovery was observed at 0.9 Basicity during the smelting reduction of chromite over burden.

4. Conclusions

The following conclusions could be drawn from the present work:

1. Goethite was the main mineral phase for nickel enriched in fine fraction. The bulk chemical analysis of the over burden also confirmed the presence of nickel.
2. Porous nickel pig after solid state reduction shows the highest percentage reduction (91%) of nickel was observed for 120 minute time, but the major part of reduction (>90%) completed in 90 minutes at 1400°C.
3. The feasibility of direct reduction process (smelting) for low grade chromite overburden fluxed pellets was also observed under electric arc furnace by varying basicity for suitable production of nickel pig. Analysis of smelted products (metal ingot) showed the highest recoveries of elements, i.e. 86% Fe, 90% Ni and, 30%Cr at 0.9 basicity.
4. These results indicated that Sukinda chromite overburden could be utilized for production of nickel pig for future demands in stainless steel making especially in India where shortage of nickel is fulfilling by importing it from other countries.

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IZVODLJIVOST EKSTRAKCIJE NIKLA IZ HROMITNE OTKRIVKE U INDIJI POSTUPKOM REDUKCIJE U ČVRSTOM STANJU I METODOM TOPLJENJA

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Abstrakt

U ovom radu je predstvljena ekstrakcija nikla iz otkrivke koja sadrži niski procenat hromita postupkom redukcije u čvrstom stanju i metode direktnog topljenja. Getit i kvarc su predstavljali najzastupljenije faze, dok su hromit i hematit identifikovani kao manje zastupljene faze u mineralu. Redukcija peleta hromita je izvedena u horizontalnoj cevnoj peći na 1000°C, 1200°C i 1400°C tokom 30, 60, 90 i 120 minuta u redukcionoj atmosferi. Peleti različite baznosti (0,5,0,6,0,7,0,8 i 0,9) su upotrebljeni za ispitivanje prilikom topljenja u elektrolučnoj peći. Najveći procenat nikla (2%), gde je dobijeno ~ 91% istog, postignut je metodom redukcije iz čvrstih peleta koji je podvrgnut postupku 120 minuta na temperaturi od 1400°C. Sličan procenat dobijenog nikla (~90%) je dobijen u ingotu (0,67% Ni) kada su korišćeni peleti čija je baznost bila 0,9 prilikom postupka topljenja. Tokom istraživanja se došlo do zaključka da se postupci redukcije u čvrstom stanju i metoda topljenja izvodljive za dobijanje nikla iz otkrivke koja sadrži niski procenat hromita. Proizvodnja niskoprocentnog feronikla je takođe moguća metodom topljenja.

Ključne reči: *Niskoprocentni feronikl; Redukcija u čvrstom stanju; Topljenje; Hromitna otkrivka; Dobijanje.*