

# Characterization and Leaching Assessment of Ferronickel Slag from a Smelting Plant in Iligan City, Philippines

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**Abstract**—Iligan City ferronickel smelting plant is producing more than 500,000 metric tons (MT) of ferronickel slag annually which are deposited in the plant's premises since it has no economic value. Considering the ferronickel slag as a waste, there is a possibility that pollutant component of the slag will leach to the environment. This prompted the study on the physico-chemical and leaching characteristic of the ferronickel slag.

Using United States Environmental Protection Agency (USEPA's) Toxicity Characteristic Leaching Procedure (TCLP) and USEPA's Synthetic Precipitation Leaching Procedure (SPLP). The concentrations of the regulated heavy metals leached from the material at different pH levels were significantly lower than the maximum concentrations for their toxicity limits set forth by EPA, DAO-34 and DAO 35 respectively. With the result of leaching test, ferronickel slag can be classified as non-hazardous.

Although ferronickel slag is non-hazardous, preventive measures must be incorporated into the design of reclamation like stabilization and solidification with the use of appropriate binders and geomaterials to avoid future constraints.

**Index Terms**—Ferronickel, slag, leaching, smelting.

## I. INTRODUCTION

Iligan City ferronickel smelting plant is producing more than 500,000 metric tons (MT) of ferronickel slag annually [1]. Ferronickel slag (FNS) aggregate is a by-product of the manufacture of ferronickel used in stainless steel, and is produced by cooling the molten slag with water or air and adjusting its grading for use in concrete. It is a waste obtained from smelting of laterite ore in an electric arc furnace at a very high temperature with the presence of a reducing agent to produce the product ferronickel shots and ingots [2]. At a smelting plant in Iligan City, the slag is deposited in plant's premises since it has no economic value. Some are mainly used as backfill materials and road enhancement substitute donated to local government units and private entities. The environmental management plan of MCCI Corporation company for the ferronickel slag is a 20-hectare reclamation area of Iligan Bay adjacent to plant's docking area in Buru-un, Iligan City in a projected 10-year

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operation of the plant. Considering the ferronickel slag is considered a waste, there is a possibility that pollutant component of the slag will leach to the environment affecting the soil, ground water, surface water, marine ecosystems as well as human health. Thus this study was conducted.

Different leaching tests have been developed to determine the interaction of wastes with the surrounding environment. For instance, leaching tests can provide a good insight into the mobility of heavy metals [3]. In addition, leaching tests play a major role to assess the possibility and use of treatment within regulatory limits [4]. There are two main potential environmental liabilities when using steel slag. These are: pH that is too high (11-12 Standard Unit), and the potential of toxic trace metals to leach from the steel slag matrix. The possibility that toxic metals may become mobile in the environment when steel slag is used has been extensively researched [5,6]. However, no consensus has yet been reached because there is conflicting information on this issue. Dissolution of slag and adsorbed metal ions occurs with a condition of either low pH or high temperature [7]. With the various pH environments the properties of slag may change and the release of toxic elements may occur through the leaching process [8][9]. Thus, the leaching and toxicity tests are required to assess any environmental impacts prior to using slag for water and wastewater treatments [8].

This study will attempt to assess the physico-chemical properties as well as the leaching characteristic of ferronickel slag produced by MCCI Corporation in order to determine the mobility of metals with the use of leaching procedures if used as a fill material for land reclamation or road enhancement and identify possible environmental impacts for proper management and utilization of ferronickel slag and to incorporate preventive measures in the design and usage to reduce potential risk.

## II. MATERIAL AND METHODS

### A. Characterization of Physical and Chemical Properties of Ferronickel Slag.

#### 1) Material

The ferronickel slag used was produced by MCCI Corporation, a smelting industry from Iligan City, Philippines. Five samples of different manufacturing date with five replicates were obtained from plant's storage area. As per required by TCLP and SPLP, reagent grade chemicals used conforms to the standards set by American Chemical Society (ACS).

#### 2) pH

pH was determined using the glass electrode method as outlined by Standard Methods for the Examination of water

and wastewater (1971 & 1975) with the use of Eutech Cyberscan pH 11 pH meter. In a beaker, 100cm<sup>3</sup> of distilled water was placed and then added with 50 grams of dried ferronickel slag. pH of the supernatant was measured until measurement of pH with meter was constant.

### 3) Density

Density was determined using volumetric or displacement method using water as a medium. A mass of the irregular solid sample of FNS was weighed on the analytical balance (Sartorius, ED224S) reported in grams. Then water was placed in a graduated cylinder. The initial amount of water was determined by reading the bottom of the meniscus level. Then the solid was placed carefully not to lose any material into the graduated cylinder and the final level of water was read and noted. The difference of the final and initial level is the volume of the solid reported in milliliter (ml). Density was determined with the use of this formula:  $d = m / v$

### 4) Particle Size Distribution

Granulometric composition was determined by sieve analysis with rotary shaker. One kilogram of ferronickel slag were placed in ASTM and Tyler Mesh Screens 1.0 in, 0.50 in, 0.375 in, 0.25 in, no.4 (4 mesh), no. 8 (8 mesh), no. 12 (12 mesh), no. 16 (16 mesh), no. 30 (30 mesh), no. 50 (50 mesh), no. 100 (100 mesh) in a rotary shaker for 5 minutes. Particle size distribution was obtained by determining the relative percentage by weight of grains of each of the different size fraction represented in the sample [10][11]. Grain analysis was made by using Udden-Wentworth classification scale.

### 5) Mineral Composition

X-Ray Diffraction (XRD) analysis was carried out in a Maxima-X Shimadzu 7000 diffractometer with monochromator and a copper K $\alpha$  x-ray source. Microsoft Excel was used to plot and analyze the recorded intensity. The software Mineral Database from International Center for Diffraction Data (ICDD) was used to identify the peaks and determine the phases present.

### 6) Microstructure and Microanalysis

The powder morphology, elemental analysis and mapping overlays of such element in distribution on the ferronickel slag sample were obtained using scanning electron microscopy (SEM JEOL JSM-6510LA).

### 7) Chemical Composition

Chemical composition of ferronickel slag was determined using X-ray Fluorescence (XRF). The sample was pulverized (BICO pulverizer) mixed with fluxing agent then placed in the pelletizer (SPECAC) apparatus until 15 psi then analyzed using XRF SPECTRO XEPOS by Spectro Analytical Instruments GmbH.

### 8) Coefficient of acidity.

Coefficient of acidity of slag was determined by obtaining the ratio of the acidic oxide like SiO<sub>2</sub> to the basic oxide like CaO and MgO after XRF analysis

### 9) Leaching Assessment

USEPA Method 1311 – Toxicity Characteristic Leaching Procedure (TCLP) and USEPA Method 1312 Synthetic Precipitation Leaching Procedure (SPLP) were the leaching procedures used. One hundred grams (100g) of the ferronickel slag was placed in a 2.2 L high density polyethylene (HDPE) extraction vessel sealed with Teflon tape on bottle threads and 2L of the extraction solution

(TCLP fluid #1, TCLP fluid #2, SPLP #1 at pH 2.5, SPLP #2, SPLP #3) was added then mixed. The liquid/solid ratio was 20L/kg. The mixture was stirred for 18  $\pm$  2 hour period using rotary extractor at a rate of 30  $\pm$  2 rpm and then filtered with 0.7  $\mu$ m glass fiber filter. The filtrate was collected in 1L plastic bottle and preserved by adjusting to a pH of less than 2 using 1N nitric acid (HNO<sub>3</sub>) unless precipitation occurs. The extract obtained from the TCLP and SPLP test (the “TCLP or SPLP extract”) was then analyzed to determine the elemental (Ni, Fe, Cr, Co and etc) concentration using atomic absorption spectroscopy with atomic absorption spectrophotometer (PerkinElmer AAnalyst 200).

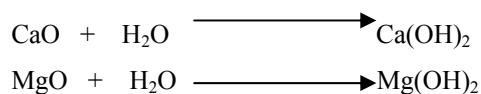
The spectrometer used a different hollow cathode lamp and different standard working solutions for each metal to be analysed. Data reported as mg/L or ppm. Sample spikes, run in triplicates and calibration check samples were performed as appropriate.

## III. RESULTS AND DISCUSSION

### A. Characterisation of Physical and Chemical Properties of Ferronickel Slag

#### 1) pH

When the ferronickel slag has contact with water, some of the components, mainly free lime (CaO) and magnesia (MgO) may dissolve partially into the solution as described by this equation.



This will increase the concentration of calcium and magnesium ions in the solution and accordingly increase pH through the ionization of calcium hydroxide and magnesium hydroxide.

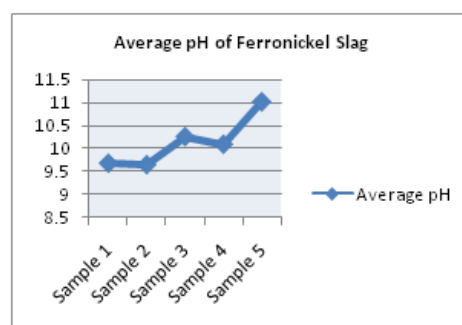


Fig. 1. Average pH of FNS

Ferronickel slag is relatively alkaline with pH range of 9.660 to 11.016 as shown in Fig. 1. With this pH, it can be used for passive treatment for acidic wastewater instead of limestone [12]. Under high pH condition, the slag surface is negatively charged and adsorbs cations, such as sodium and metal ions [7]. With these characteristic, ferronickel slag can be used as adsorbent of metals in waste and wastewater.

#### 2) Density

The density of ferronickel slag ranges from 3.13 to 3.17 as shown in Fig. 2 compared to ordinary sand (density = 2.5-2.6) is much higher which means that the self-weight of the ferronickel slag was high.

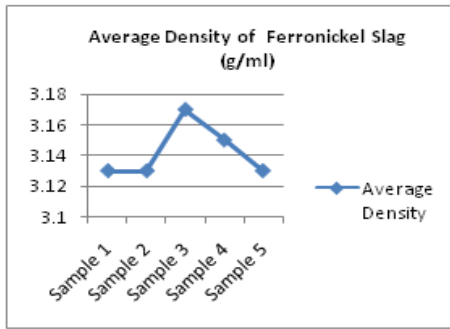


Fig. 2. Average density of FNS

3) Particle size distribution

The granulometric result of screening as shown in Table I was compared to Udden-Wentworth grain classification scale. Sample 1 has 69.67% gravel and 30.28% sand. Sample 2 has 64.15% gravel and 35.85% sand, sample 3 has 71.98% gravel and 28.02% sand, sample 4 has 40.48% gravel and 59.52% sand and sample 5 has 44.64% gravel and 55.36% sand. With these ratios, adding cement to the ferronickel slag can be used for land reclamation but the compressive, tensile and other characteristic must also be considered

TABLE I: AVERAGE PERCENTAGE PARTICLE SIZE DISTRIBUTION

Mesh No.	Sample Number				
	1	2	3	4	5
1 inch	0.17	0.48	0.00	0.00	0.00
1/2 inch	0.88	1.12	0.47	0.00	0.00
3/8 inch	2.45	2.73	0.81	0.09	0.25
1/4 inch	7.93	11.92	5.04	0.78	2.67
4 mesh	21.29	11.84	17.26	8.80	9.05
8 mesh	36.95	36.06	48.40	30.81	32.68
12 mesh	14.47	10.76	14.56	17.53	15.82
16 mesh	9.54	14.37	9.04	11.42	10.42
30 mesh	3.51	5.64	2.07	20.29	18.98
50 mesh	1.68	3.50	1.09	6.68	6.40
100 mesh	0.61	0.98	0.73	2.81	3.04
> 100 mesh	0.48	0.61	0.53	0.78	0.69
Total	99.95	100.0	100.00	100.00	100.0

4) Microstructure and microanalysis

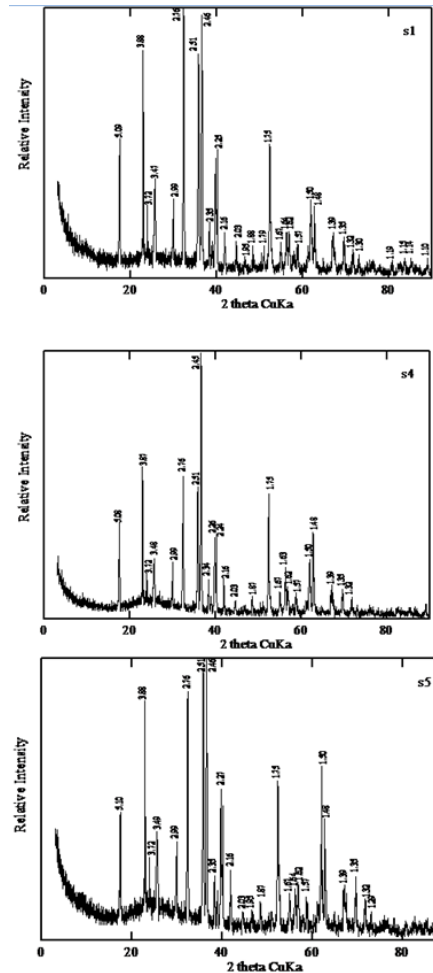
SEM-EDX was done to obtain information on trace elements found in the slag as shown in Table II. The result of main elements identified in SEM-EDX were also found in the XRF analysis.

Fig. 4 shows the crystalline and glassy region as well as the void spaces and roughness of the FNS. Mapping of elements of sample 2 for Al-Si-Ca shown by means of a leford doped as shown in Fig. 4c. Regions were dominated by silicon and least by calcium. Overlays for sample 1, O-Al-Mg is shown in Fig. 4d dominated by oxygen.

5) Mineral composition

The XRD pattern of ferronickel slag shows the presence of Forsterite (magnesium iron silicate oxide,  $2MgO.90FeO.10SiO_2$ ), aluminosilicate phases, chromium silicate phases, calcium silicate hydrate ( $Ca_2SiO_4.1/2H_2O$ ), highly crystalline phases and amorphous phases as seen on broad humps in Fig.3.

With the presence of magnesium and calcium based silicates, ferronickel slag may be capable for mineral carbonation in carbon sequestration or binding to fossil-fuel bound carbon.



as FeO, MgO, MnO entered in chemical compounds with the SiO<sub>2</sub>. [13].

The metallurgical slag in the composition of which Si and Al-oxides predominate may be used as a raw material for the production of ceramic filters, aerators and diffusers applied in the treatment of industrial gases and purification of wastewaters. [14] [15].

Result of the XRF analysis is shown in Table III. The chemical composition of ferronickel slag refers to a multicomponent silicate system. The very high content of iron oxides indicates the potential of this slag to develop magnetic phases upon appropriate processing.

TABLE II: ELEMENTAL COMPOSITION OF FERRONICKEL SLAG SAMPLES BASED ON SEM-ENERGY-DISPERSIVE X-RAY SPECTROSCOPY (EDS OR EDX).

Element	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
	Mass%	Mass%	Mass%	Mass%	Mass%
C	10.59	9.54	20.32	32.13	24.14
O	37.19	39.42	12.36	29.81	53.84
Mg	18.64	14.78	1.25	9.34	10.82
Al	1.21	3.41	31.24	2.89	1.81
Si	19.35	20.09	1.3	10.62	8.53
Ca	0.82	1.13	0.15	0.46	0.29
Cr	0.79	1.11	1.33	1.22	0.01
Mn	0	0.2		0.22	
Fe	10.86	9.07	23.53	11.7	0.04
Co	0.08	0.29		0.1	0.08
Ni	0.1	0.27		0.01	0.04
Cu	0.33	0.37	7.7	1.46	0.4
Pd		0.25			
Cd	0.02	0.07	0.82	0.04	
<b>Total</b>	<b>99.98</b>	<b>100</b>	<b>100</b>	<b>100</b>	<b>100</b>

TABLE III: X-RAY FLUORESCENCE RESULTS.

	NiO	Fe <sub>2</sub> O <sub>3</sub>	CoO	P <sub>2</sub> O <sub>5</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	CaO	Cr <sub>2</sub> O <sub>3</sub>
S1 Ave	0.120	13.59	0.031	0.001	20.28	1.117	32.43	1.397	1.109
S2 Ave	0.061	10.19	0.008	0.001	20.89	1.494	33.21	1.336	1.034
S3 Ave	0.088	12.57	0.0097	0.001	17.75	1.225	28.91	1.003	1.101
S4 Ave	0.060	12.56	0.008	0.001	13.24	2.735	29.57	1.191	1.582
S5 Ave	0.039	9.519	0.007	0.001	17.02	2.5	31.09	1.098	1.578

7) Coefficient of acidity

Coefficient of acidity ranges on the average from 1.494 to 2.049 as shown in Fig. 5. Ratio between SiO<sub>2</sub> and CaO is definition of the strength, connecting skills of the slag relations between crystal and amorphous phases [14].

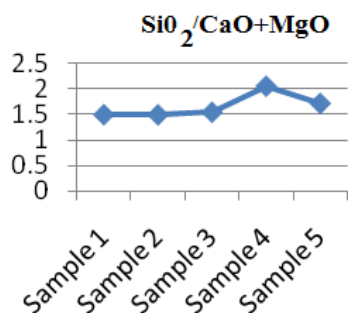


Fig. 5. Coefficient of acidity

8) Leaching assessment

The result of leaching of different metals at different leaching procedures is shown in Fig. 6.

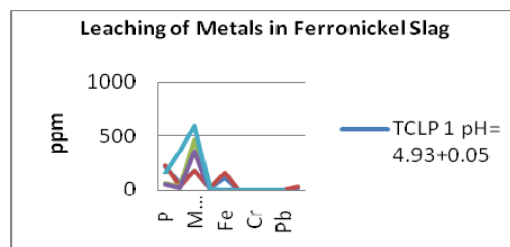


Fig. 6. Leaching of metals at different leaching procedures

When compared to EPA and Dept. of Environment and Natural Resources Administrative Order DAO-34 and DAO-35, the concentration does not exceed the standard limit set forth by agencies as shown in Table IV. Cadmium concentration in leaching was not obtained due to unavailability of lamp.

TABLE IV: COMPARISON OF LEACHING RESULTS OO REGULATORY STANDARD

Heavy Metals	Cr (VI)	Pb	Cd	Ni	Co	Mn	Fe
EPA	5.00	5.00	1.00	NS	NS	NS	NS
DAO-34 (Class SD)	NS	NS	NS	NS	NS	NS	NS
DAO-35 (Class SD)	1.00	NS	0.50	NS	NS	NS	NS
TCLP 1 pH= 4.93±0.05	nil	0.66	NO	15.03	0.49	20.67	109.68
TCLP 2 pH = 2.88±0.05	nil	0.76	NO	20.28	0.54	27.67	162.36
SPLP 1 pH = 2.5±0.05	nil	0.85	NO	2.12	0.49	NO	1.31
SPLP 2 pH = 5.0±0.05	nil	1.07	NO	2.33	0.63	NO	1.4
SPLP 3 pH = 5.6 to 7.0	nil	1.46	NO	5.08	1.1	NO	2.16

NS – no standard, not yet consider at the present time  
NO – not obtained

B. Impact on Physico-Chemical Environment

1) Air

Based on the granulometric analysis, the percentage of less than 100 mesh ferronickel slag is about 0.62 percent on the overall average. Dust will fly to the hi-way from the storage area only if there is equipment movement in the slag storage and if the wind is strong enough to fly the minute particles of the slag. When used as backfill, FNS will link with the particles in the soil.

2) Water

If the leachate is considered as effluent, the metals present in the slag have not exceeded the criteria set in DAO 35. Hence, surface and marine pollution is unlikely to happen. According to [16] land reclamation in coastal areas may have a significant effect on local ground water systems. Results of reclamation are water tables rise and salt water-fresh water interface moves seaward. An unintended advantage of reclamation is an increase in fresh ground water resources because the reclaimed land can be an additional aquifer and rain recharge takes place over a larger area.

Land reclamation will modify the physical environment and its hydrodynamics, as well as to an alteration in the character of the wetland habitat in the estuary environments. There is a high certainty that sedimentary and morphodynamic processes in the estuary changed as a result, including changes in water and sediment circulation, transport patterns and changes in the tidal prism, with associated alterations in ecology, hydrology and relative sea-level [17].

3) Impact on biological environment

From the result of baseline assessment of the planned area for reclamation [1], two species of seagrass was found. There were also four algae associated with seagrass bed.



Coral assessment using manta tow survey and line-intercept method showed that 10 coral species are found. Reef fishes using Fish Visual Census was made and found that 18 species of fishes are present. Habitat degradation may result during reclamation.

#### 4) Environmental management plan

The study have shown that with the result of TCLP analysis, stabilization and solidification of slag in the presence of selected additives is recommended in the design of reclamation as preventive measures to account for the unpredictable environmental impact that may occur. Stabilization is a pre-landfill waste treatment process, which has been used for different types of industrial wastes, but is particularly suited to those containing heavy metals. The continuing need to develop economical and improved waste management techniques has increased the potential importance of solidification technology throughout the world, in a process defined as the best demonstrated available technology (BDAT) [18].

The stabilization or mixing the slag with binders, placing alternate layers consisting the spent slag and appropriate geo-materials that serve as environmental buffer and hydraulic barriers may be incorporated. Research and development for ferronickel slag's sustainable use is highly recommended. Since the planned reclamation area is adjacent to the sea where the face of the reclamation site is subject to tidal inundation, methods to stop inundation such as a bund wall may be needed. Bund walls should be engineered to an appropriate design.

#### IV. CONCLUSION

It is apparent that the leaching of metals in the ferronickel slag is dependent on the extraction fluid and pH, therefore, the leaching and mobility of metals occurring on the environment will also depend for its exposure and usage.

Results of the study show that the ferronickel slag can be classified as non-hazardous since the no metals exceeded the standards set by USEPA. The leachability data help clarify issues regarding potential environmental risks posed by ferronickel slag to the public specially the stakeholders.

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