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# PHYSICO-CHEMICAL AND MINERALOGICAL ANALYSIS OF MILL SCALE OF ROURKELA STEEL PLANT THROUGH MULTI ANALYTICAL METHODS AND ITS ECO-UTILIZATION FOR THE PRODUCTION OF SPONGE IRON

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***Abstract-The Eco -management study of solid wastes generated from integrated steel industries is now becoming a herculean task due to stringent environmental protocols. Thus, it becomes indispensable to recover the iron from solid wastes generated from steel industries, which will diminish pressure on natural resources and become economically profitable. Mill Scale mainly consists of magnetite. The total generation of mill scale in RSP is about 51,565 ton / annum with an average of 141 ton/day. It comprises about 60–71% FeO and 30– 36 % Fe2O3.The mill scale has to be smelted in order to recover iron in either a blast furnace or some other furnaces. But, it cannot be directly smelted because of its fineness. Its use can be made by agglomerating it either through pelletization or trough sinter mix. The objective of this study is to carry out Physical, chemical and mineralogical analysis and the usability of mill scale to produce sponge iron by employing the reduction reaction along with low grade coal and lime fines.***

**Keywords:** Mill Scale, Magnetite, Mineralogical properties

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## I. INTRODUCTION

Steel industries generate different types of solid wastes along with mill scale. Mill scale generated from rolling mill during the process of cooling and rolling of hot steel. During this process about 6% of steel gets wasted. In the whole world more than 6 million tons of mill scale is produced annually [1,2]. Mill scale can be regarded as a resource material for steel industry as it contains substantial amount of oxides of iron. But its eco-utilization is not fully realized due to presence of 10% water and 20% oil. The source of mill scale is a significant source of water pollution as the mill scale generated from it contains iron oxide and oil which contaminates water.[3] The composition of mill scale differs depending upon the type of steel produced and the process of production employed [4,5,6]. In mill scale the content of oil varies from 0.2 to 2% and can arrive up to 30%. Since, mill scale constitutes high percentage of metal with low alkali and nonferrous metal content; therefore, its eco-utilization can become economically viable. Thus, the chemical analysis of mill scale is essential for its proper utilization, so that pollution load on the environment can be mitigated. Its Characterization helps us in the exact % of use of mill scale as pellet. In this study various techniques have been adopted for the characterization of mill scale and the reduction experiment is executed by varying the sieve size of mill scale along with low grade coal and lime f in order to make sponge iron. [7,8, 9].

## 2.0 Study Area

Presently Rourkela Steel Plant is selected for carrying out the characterization and eco-utilization study of mill scale as its utilization rate is very low and dumping rate is very high. Mill Scale is being produced from rolling mill of Rourkela Steel plant. Mill scale was collected from rolling mill and its physico-chemical characterization was studied by applying multiple techniques. After Its characterizations, its eco-utilization study was executed to get valuable products with reduction of pollutants. The schematic diagram of the study area was shown below in Figure1. The mill scale and lime collected from Rourkela steel plant was represented in Figure 2

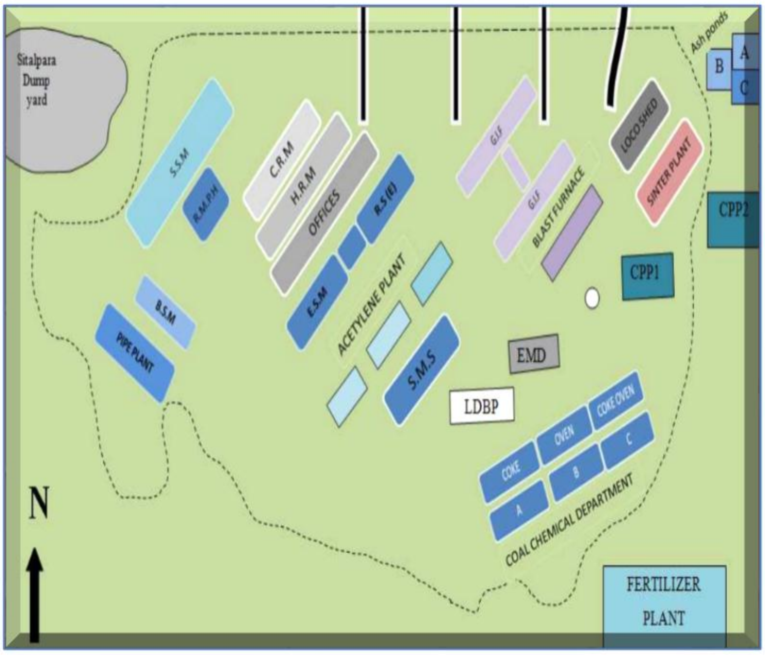


**Fig.1** Study area of Rourkela Steel Plant

**Fig.2**. Mill Scale and lime fine collected from Rourkela Steel plant Complex

## 3.0 Material and Method

Mill scale was collected from HRM & CRM of steel plant and at first its physical properties were determined by applying suitable procedure. The major physical properties were represented below in Table-1.

**Table 1** Physical Properties of Mill Scale

| **Physical property** | **Mill Scale** |
|---|---|
| Colour | Bluish black |
| Elastic property | No elastic property |
| Bulk density g $cm^{-3}$ | 2.9 |
| Specific gravity | 4.96 |
| % of moisture | 1.3 |
| % of absorption | 21.2 |
| Relative density | 1.9 |

### 3.1 Morphological Analysis of Mill Scale

The SEM image of mill scale gives the size, shape and micromorphology of the minerals, other particles and textural pattern. The SEM analysis of the collected mill scale is investigated in CIF, VSSUT, Burla.

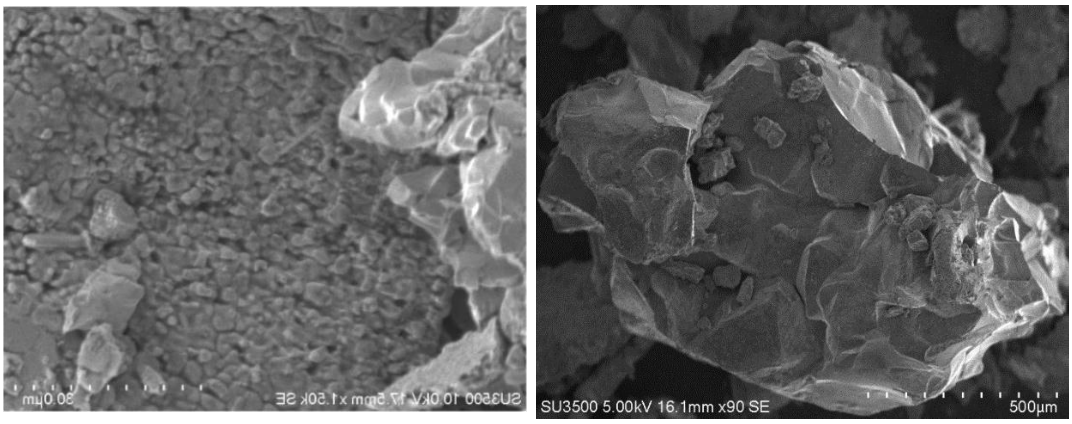


**Fig.3** SEM image of Mill Scale

From SEM Analysis it is observed that mill scale contains both elongated and globular grains. It is also noticed that elongated grain contains scales and rarely is it also having the plain surface.

### 3.2 XRD Analysis

It is an important technique used to characterize the mineralogical phase of solid crystalline materials. The mill scale was undertaken for XRD Analysis.. From XRD peaks, it is found that the chief mineralogical phases are magnetite and hematite. The XRD graph of mill scale is represented below

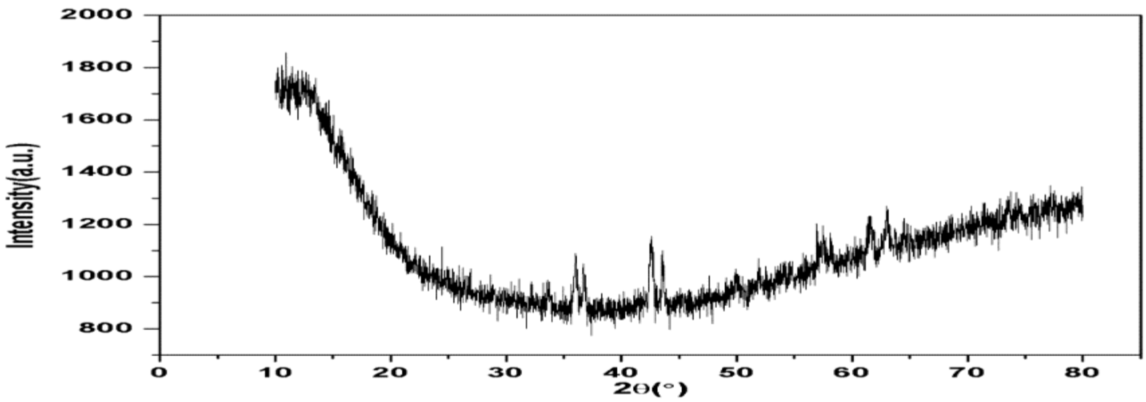


**Fig. 4** XRD peaks of Mill Scale

**Chemical analysis of the Mill Scale of Rourkela steel plant**

The chemical composition of mill scale was determined by XRF analysis and is presented in Table-2.

**Table 2** Chemical Composition of mill scale

| Elements/Compounds | % By weight |
|---|---|
| $Fe_{Total}$ | 67.4 |
| $SiO_2$ | 4.1 |
| $Al_2O_3$ | 1.84 |
| CaO | 0.7 |
| MgO | 0.25 |
| MnO | 0.65 |
| $Na_2O$ | 0.41 |
| $K_2O$ | 0.25 |
| LOI | 6.8 |
| Trace Elements | %. Wt. |
| P | 0.07 |
| Ti | 0.08 |
| Cu | 0.14 |
| Zn | 0.01 |
| Co | 0.026 |
| Ni | 0.038 |
| Cr | 0.033 |
| Pb | 0.033 |

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## 4.0 Eco-utilization Techniques

From Physicochemical analysis, it is found that the mill scale of RSP contains maximum percentage of iron and as per the environmental guidelines, it is classified as non-dangerous waste or green waste. So, it can be used to recover iron by direct reduction method. Direct reduction processes are generally of two types. The reduction processes are represented in the Table 3[10, 11]

**Table 3** Types of Reduction Processes of Iron containing material

| **Reduction Processes used for Fe rich raw material** | **Sub classification of Process** |
|---|---|
| .Gas based Reduction Process | Shaft Reduction Process<br>Fluidized Reduction Process |
| Coal Based Reduction Process | Retort Reduction Process<br>Rotary kiln Reduction Process<br>Rotary hearth Reduction Process |

In this study the reduction of iron rich material is mixed with lignite coal and fine particles of lime in order to obtain the sponge iron. To reduce oxides of iron CO (g)+$H_2$ (g) is normally used and the process is commonly known as gas-based reduction process (Shaft Process). In coal based reduction process i.e. in rotary kiln process C of coal and the CO formed are used at a particular temperature to reduce the iron rich material. The reaction involved in this process is given below. [12,13]

$Fe_pO_q(s) + b\ CO\ (g) = c\ Fe_xO_y(s) + d\ CO_2(g)$-------(1)

The required CO(g) is provided by employing the Boudouard gasification process mentioned as below.

$nC\ (s) + nCO_2\ (g) = 2nCO(g)$-------------(2)

In the coal-based reduction Process the temperature needed is within the range of $800^{-}1100^{0}C$.

**Role of lime fines in the reduction of mill scale**

The fine particles of lime predominantly contain $CaCO_3$ , whicht acts as catalyst along with powdered form of coal. This mixture scavenges the sulphur content of the coal. At first decomposition of $CaCO_3$ gives CaO and $CO_2$. Then CaO acts as catalyst to enhance process of gasification. [14,15]

$CaCO_3 + heat \rightarrow CaO + CO_2$

$CO_2 + C \rightarrow 2\ CO$

### Experimental Design:

For the reduction of mill scale, the raw materials required are

- Mill Scale from RSP
- Fine particles of lime from LDBP of RSP
- Low grade coal like lignite coal procured from Talcher, Odisha.

The detailed description about the raw materials and their cost is given in Table-4 below.

Mill scale and lime are collected from Rourkela steel Plant and coal is purchased from the local market. As the raw materials are cheap, the production of sponge iron becomes economical and ecofriendly [16]

**Table 4** Raw materials used, their source and cost

| Sl. No. | Raw Materials | Source of raw materials | Cost of raw materials / kg |
|---|---|---|---|
| 1. | Mill Scale | From Rourkela Steel Plant | ------- |
| 2. | Lime Fines | From Rourkela Steel Plant | -------- |
| 3. | Low grade Coal | From Lingaraj Open cast mines Talcher, Odisha | □6-7/- |

Then the proximate analysis was conducted on coal and the results are represented in the Table-5.

**Table 5** Proximate analysis of coal

| Low grade coal (Lignite coal) | Parameters | % Composition |
|---|---|---|
| | **$C_{fixed}$** | 26.56 |
| | **Ash Content** | 10 |
| | **Volatile substances** | 25.24 |
| | **Other Components** | 39.20 |

**Required Ratio of Mill Scale and Coal**

The amount of fixed carbon required to reduce the oxide of iron present in mill scale is determined by the important XRD peaks of raw mill scale. Since, coal contains about 26.56% of fixed carbon; therefore the amount of coal required for reduction of mill scale is calculated. To get the optimum results 1:1 ratio of mill scale and lime powder were taken. In the present study 50 gm of mill scale, 45 gm of lignite coal and 5gm of lime powder was taken in an alumina crucible for reduction purpose. [17-19]

## 5.0 Experimental Details

**Cleaning of Raw Materials and preparation of samples:**

Mill Scale was thoroughly washed with water to eliminate oil, grease, and impurities present in it. Then mill scale, coal and lime powder both were crushed into fine particles by using crusher. Then, these are passed through the different sieves by employing sieve shaker. In the present study mill scale, coal and lime powder of particle of sieve size 50-100, 150-200 and < 350 as per ASTM specification were taken. [20,21]

**Placement of raw materials in alumina crucible:**

At first mill scale was placed in the middle of vertical column of approximately 23 mm in diameter. Then, nearby it the mixture of coal and lime powder are placed, which is depicted in the figure-3 and 4. Then the crucible was covered with silica lead and glass wool in order to prevent oxidation.

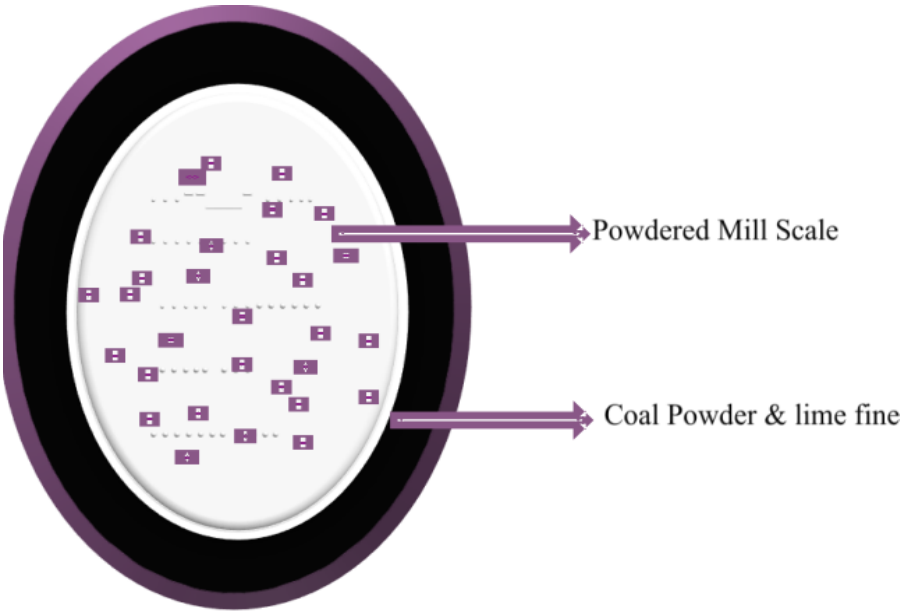


**Fig-3: Arrangement of Mill scale and coal in crucible top view**

**Reduction of sample in Muffle furnace:**

Then the samples of different sieve size are placed in laboratory muffle furnace and heated up to $920^0$C for 30, 60 and 90 minutes and then these were removed from the furnace.

**Separation of materials after cooling:**

Then the samples were cooled and get separated by the help of a strong magnet.

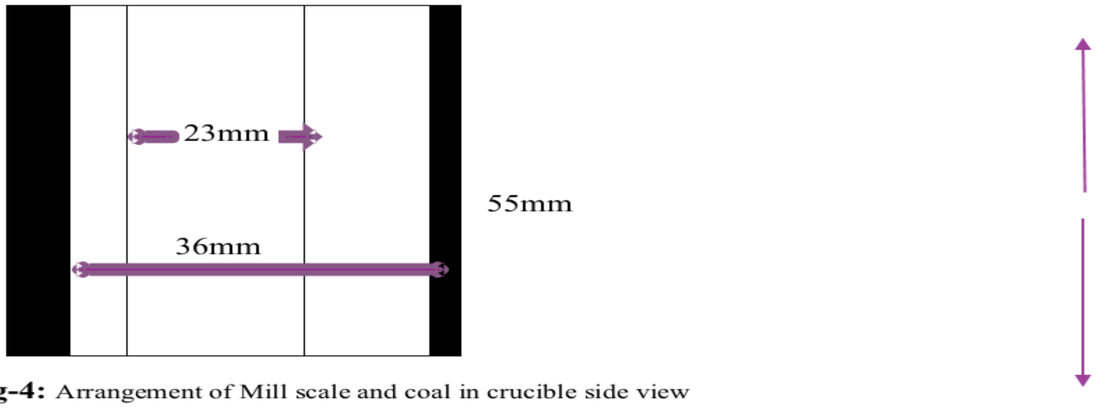


**Fig-4:** Arrangement of Mill scale and coal in crucible side view

**Fig-4:** Arrangement of Mill scale and coal in crucible side view

## 6.0 Result and Discussion

From chemical analysis prior to reduction of mill scale, the iron content of mill scale is found to be 67.4% by weight. The percentage of iron in the mill scale of different mesh size is determined at different time interval up to temperature $920^0$C. The result of reduction is represented in the Table-6. From the reduction result it was evident that as reduction time increases for all mill scale samples of different size and with decrease in sieve size of the sample the iron percentage decreases.

**Table 6** Chemical analysis of mill scale after reduction at different range of temperature and time

| Sl. No | Particle of different sieve Size (ASTM size) | Time in Minutes | Wt. % of Fe |
|---|---|---|---|
| 1.<br>2.<br>3. | 50-100 | 30<br>60<br>90 | 77.83<br>78.73<br>81.76 |
| 4.<br>5.<br>6. | 150-200 | 30<br>60<br>90 | 71.98<br>74.56<br>80.97 |
| 7.<br>8.<br>9. | < 350 | 30<br>60<br>90 | 74.92<br>76.72<br>79.13 |

## 7.0 Conclusion

Experimental results of chemical analysis indicate that as the heating time increases in muffle furnace, there is increase in the percentage of iron in case of all the samples of different particle size. The result obtained in case of mill scale with particle size 50-100 ASTM sieve size heated for 90 minutes is encouraging and shows excellent result. This is because the permeability of mill scale sample with 50-100 sieve size is more than that of other samples with other range of sieve size. In this case C and CO can easily enter into the scale column and increases the rate of reduction. Thus, the mill scale sample with 50-100 ASTM sieve size undergoing reduction with furnace temperature of about 920$^0$C yields about 81.76%.. Hence, the reduction of mill scale by using low grade coal and lime powder is not only an inexpensive method, but also it is considered as an ecofriendly and sustainable process as it reduces the pressure on the mining of iron ores.

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