Pak. j. sci. ind. res., vol. 32, no. 1, January 1989

PREPARATION OF FERRIC CHLORIDE FROM IRON ROLLING SCALE BY OXIDATION Part I

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(Received August 9, 1988, revised January 22, 1989)

Preparation of iron (III) chloride from rolling scale in 12N hydrochloric acid and in tern oxidation with dried chlorine gas was carried out. Effects of temperature and time of oxidation were also studied. It was found that 70% of Iron rolling scale, dissolved in 12N Hydrochloric Acid at 60°, yield crystalline ferric chloride, when oxidized with dried chlorine gas.

Key words: Ferric Chloride, Iron rolling scale, Oxidation.

INTRODUCTION

The increased production of rolling scale by the Metropolitan Steel Corporation, Karachi; has resulted in consequential problem, which is not utilised by the industry and goes waste. At present the Metropolitan Steel Corporation, is not benefiicially utilising the rolling scale waste obtained from the 50,000 tons iron products handled yearly [1]. This waste is 3% of the total production of iron product mentioned. It was considered that the PCSIR can provide technical assistance in the matter by carrying out experimental processes on the metal to produce ferric chloride presently being imported. Thus the product will save foreign exchange which is presently being used for importing 41385 kg of ferric chloride yearly.

From the preliminary studies of laterite [2] and low-grade iron ore [3] by direct chlorination, anhydrous iron chloride already been obtained. Efforts would also be made to obtain ferric chloride (anhydrous) by direct chlorination of the rolling scale which will be reported in a latter stage.

Oxidation of the solution of iron in hydrochloric acid by nitric acid has been reported [4], but the economic of this process is not viable since the removal of excess nitric acid is difficult, beside the presence of HNO_3 contaminates the crystals according to the reaction;

$$3FeCl_2+3HCl+HNO_3 \longrightarrow 3FeCl_3+NO+2H_2O$$

An alternate process has been applied by which ferric chloride is readily obtained by dissolving iron rolling scale to saturation in hydrochloric acid, boiling the solution and then oxidizing it with chlorine gas [4] as indicated by the reaction:

$$Fe+2HCl \dashrightarrow FeCl_2+H_2$$

$$2FeCl_2 + Cl_2 \dashrightarrow 2FeCl_3+Cl$$

EXPERIMENTAL

The chemical analysis of iron rolling scale was undertaken by conventional method refered by Scott [5] and it was found that the scale contains 70% of metallic iron which amounts to 99% as Fe_2O_3. The chemical analys is of the scale is given in Table 1.

Preparation of ferric chloride. The iron rolling scale was dissolved in 12N hydrochloric acid so that ferrous chloride is formed. The solution is then filtered to remove undissolved matter. The ferrous chloride solution is then oxidized by chlorine gas to convert it into ferric state. This solution of ferric chloride is digested on steam bath to crystallize out ferric chloride. The ferric chloride crystals are recored by filteration. The following parameters have been studied for maximum yield of ferric chloride by the process mentioned.

Effect of temperature on the recovery of $FeCl_3$
Effect of oxidation time
Effect of scrap/HCl ratio (w/v)
Flow rate effect of chlorine gas

Effect of temperature on the recovery of $FeCl_3$. The reaction of iron rolling scale with 12N hydrochloric acid and then oxidation of ferrous chloride with chlorine gas to obtain ferric chloride was studied in cold as well as in hot condition. It was found that maximum yield of $FeCl_3$ occurs at 60-80°. The results are shown in Table 2.

Effect of oxidation time. The ferrous chloride formed as a result of dissolving iron rolling scale in HCl (12N) was further oxidized to ferric chloride by passing the chlorine gas through the saturated solution of ferrous chloride. Different time for oxidation have been studied and it was noted that oxidation for a period of 2-4 hours results in maximum recovery of $FeCl_3$ as is evident from Table 2.

Effect of scale/HCl ratio (w/v). The iron rolling scale was dissolved in 12N hydrochloric acid. Keeping various ratio of scale/HCl (w/v), the % recovery of ferric chloride was studied

Table1. Chemical analysis of iron rolling scale.

Composition	% element
Silica as SiO_2	0.25
Iron as Fe_2O_3	99.16
Calcium as CaO	0.25
Magnesium as MgO	0.18
Sodium as Na_2O	0.03
Potassium as K_2O	Nill

Table 2. Effect of Temperature on oxidation.

Wt of sample (g)	Temperature °C	% recovery of $FeCl_3$
50	R.T	25.0
"	40	42.0
"	50	48.0
"	60	70.0
"	70	70.0
"	80	70.0

Table 3. Effect of oxidation time for the recovery of $FeCl_3$.

Wt of sample (g)	Temperature °C	Time of oxidation (min)	% recovery of $FeCl_3$
50	60	15	20.0
"	"	30	25.0
"	"	45	40.0
"	"	60	50.0
"	"	90	60.0
"	"	120	70.0
"	"	180	70.0
"	"	240	70.0

Table 4. Effect of scrap/HCl (w/v) ratio for the recovery of $FeCl_3$

Scrap/HCl	% recovery of $FeCl_3$
1:1	35.0
1:5	48.0
1:10	58.0
1:20	70.0
1:30	70.0
1:40	70.0
1:50	70.0
1:60	70.0

and it was observed that maximum recovery of ferric chloride occurs at a ratio 1:20 to 1:60 (scrap/HCl). The results are given in Table 4.

Flow rate effect of chlorine gas. The number of bubbles/min of chlorine gas was noted by flowmeter and it was found that maximum yield of $FeCl_3$ occurs by passing the chlorine gas at a rate of 120 to 200 bubbles/min as shown in Table 5.

Composition of isolated ferric chloride ($FeCl_3$ $6H_2O$). The chemical composition of ferric chloride obtained was determined. Various batches of the product were analysed [6]. The results are shown in Table 6.

DISCUSSION AND CONCLUSION

The study carried out on the oxidation of iron rolling scale with chlorine gas indicates that iron scrap can easily be oxidized at 60°, without any catalyst. From the results it is found that iron chloride ($FeCl_3$) can be achieved by dissolving iron scrap in hydrochloric acid (12N), heating the solution at 60° so that ferrous chloride is formed, which is further oxidized to ferric chloride by passing dried chlorine gas through the saturated solution of ferrous chloride. The product ($FeCl_3$) is not influenced by the impurities like Ca, Mg and Na because these impurities are present in the product in such amount that these are within the permissible limits as referred by "Analar" [5].

Table 5. Flow rate effect of chlorine gas

Wt of sample (g)	Temperature °C	Time (hrs)	Flow rate bubbles/min	% recovery of $FeCl_3$
50	60	2	30	28.0
"	"	"	45	38.0
"	"	"	60	58.0
"	"	"	75	65.0
"	"	"	120	70.0
"	"	"	180	70.0
"	"	"	200	70.0

Table 6. Percent composition of end product ($FeCl_3.6H_2O$)

	1	2	3	4	5	6
Insoluble matter	0.005	0.005	0.004	0.003	0.004	0.004
Combined water (H_2O)	38.98	38.98	38.80	39.15	39.25	39.30
Ferric chloride ($FeCl_3$)	58.87	58.85	58.90	59.10	59.15	59.20
Ferrous iron (Fe)	0.02	0.018	0.018	0.016	0.016	0.016
Free chlorine (Cl)	0.0009	0.0009	0.001	0.001	0.001	0.001
Calcium as CaO	0.03	0.035	0.04	0.045	0.045	0.04
Magnesium as MgO	0.01	0.01	0.01	0.015	0.015	0.01
Sodium as Na_2O	0.02	0.02	0.024	0.024	0.025	0.025
$FeCl_36H_2O$	97.85	97.83	97.70	98.25	98.40	98.50

Average composition of $FeCl_3.6H_2O$..........98.08%

The observations in present investigations provide an explaination for reported work where no parameters were considered i.e. in ferrous chloride phase [4]. However, the yield of this compound ($FeCl_3$) is postulated on the basis of different effects e.g. temperature, acid concentration, flow rate of gas and gas bubbling period.

Under the stated conditions, 70% yield of $FeCl_3$ can be achieved. Moreover at 60° temperature, the solution with 1:20 scrap/HCl (w/v) ratio, two hours oxidation and 120 bubbles per minutes of gas can attribute increase in yield. The green colour of ferrous chloride which changes to red-brown shows that oxidation towards the composition of ferric chloride has been occured.

This research in fact not only describes the process for preparation of ferric chloride from iron rolling scale on laboratory level but also provide an opportunity to chemical industries of locally available product.

Ferric chloride which is an imported chemical, is mainly used in medicine and sugar industry. It is also employed as a catalyst in Friedel-Crafts synthesis, the preparation of alkyl chlorides from olefins and the hydrogenation and chlorination of aromatic compounds. Moreover, in solution it is extensively used as a coagolent in treatment of water and sewage and to a limited extent as an etching agent in Lethography.

Iron rolling scale which is presently not being beneficially utilized can be used for production of ferric chloride by applying simple process as discussed at a considerably low cost.

Presently, the imported figure shows that ca 42000 kg of this product is the main requirement of the nasant industries. The total foreign exchange expenditure on this product are about one million rupees. However, the production of locally produced ferric chloride will save foreign exchange being spent on its import. The production is recommended to be made at the steel mills itself.

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