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RECOVERY OF FERROUS SULFATE HEPTAHYDRATE FROM SPENT PICKLE LIQUOR OF THE STEEL INDUSTRY

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ABSTRACT:

Ferrous sulfate heptahydrate was crystallized from steel pickling waste liquors by addition of 1-propanol. Variables studied were, ferrous sulfate concentration, sulfuric acid concentration, volumetric ratio of 1-propanol to waste pickle liquor and temperature. It is concluded that the recommended conditions for crystallization of ferrous sulfate heptahydrate are, high ferrous sulfate concentration, low sulfuric acid concentration, volumetric ratio of 1-propanol to waste pickle liquor of 2:1 and temperature of 20 $^{\circ}$ C.

Key Words: Pickling, waste pickle liquors, ferrous sulfate heptahydrate

INTRODUCTION:

Steel pickling waste liquors are one of the main environmental problems of steelmaking industry. As a result, spent pickle liquor is considered a water pollutant, and a great efforts have been expended to control the waste stream from pickling processes.

Pickling of steel ⁽¹⁻¹⁰⁾ is a chemical process carried out to remove iron oxides from steel surface. Sulfuric acid has traditionally been the pickling medium for descaling steel. Acid pickling is considered a preliminary process carried out for most steel products prior to coating processes such as electroplating, phosphating, painting, enameling etc. Acid pickling consists in treating the steel work-piece with a dilute sulfuric acid for a few minutes sufficient to dissolve the surface oxides. The ferrous sulfate formed by the pickling action builds up continuously in the pickle liquor as fresh acid additions are made to the pickle tank. The pickling action is eventually stifled by the inhibiting action of the ferrous sulfate. At this point the spent acid bath must be

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replaced. The recovery of steel pickling liquor is one of the main environmental aspects that the steelmaking industry must resolve.

Although much work has been done on the treatment of steel pickling waste liquors, little has been done on the recovery of ferrous sulfate heptahydrate⁽¹¹⁾ Treatment of waste pickle liquors with acetone⁽¹²⁻¹⁶⁾, ethyl alcohol⁽¹⁷⁾, and iso-propyl alcohol⁽¹⁸⁾ were studied to crystallize ferrous sulfate heptahydrate. The object of the present work is to crystallize ferrous sulfate heptahydrate by addition of 1-propanol.

EXPERIMENTAL:

The liquors were prepared by dissolving the required amounts of sulfuric acid and ferrous sulfate hepatahydrate in distilled water. Concentrations studied were 27, 54, 81, 108, 135 and 162g/l ferrous sulfate in presence of 10, 20, 40, 60 and 80 g/l of sulfuric acid. To a certain volume of the liquor, 1-propanol was added slowly with stirring during addition. The time of addition was about two minutes, the crystallized ferrous sulfate heptahydrate crystals were then filtered. The factors studied which may affect the yield of crystallized ferrous sulfate heptahydrate during crystallization were the concentration of the liquors, 1-propanol to liquor volumetric ratio and temperature. The variations in the volumetric ratio of 1-propanol to liquor affect the yield of crystallized product ferrous sulfate. The volumetric ratios studied were 1:1, 2:1 and 3:1. Crystallization was carried out at 20 and 30 °C at different concentrations and different volumetric ratios.

After filtration, ferrous sulfate was analyzed to determine its yield and to determine the water content of the crystals. Ferrous and sulfate ions were determined according to standard methods⁽¹⁹⁾. It was found that water content is seven moles of H_2O per mole of FeSO₄ and thus the ferrous sulfate produced was heptahydrate in all experiments. The results of the experiments are shown in figures (1-14).

RESULTS AND DISCUSSION:

Effect of sulfuric acid concentration:-

The results shown in figures (1-14) indicate that increasing the acid concentration decreases the yield of crystallized ferrous sulfate heptahydrate. It can be concluded that to obtain maximum recovery of ferrous sulfate heptahydrate, liquors of minimum acid concentration must be used. It is known that waste pickle liquor is discarded at

about 40 g/l H_2SO_4 , so it is practically to crystallize ferrous sulfate at this acid concentration. The decrease of recovery with increasing acid concentration may be attributed to the increase of solubility of ferrous sulfate heptahydrate in the system $C_3H_7OH-H_2O-FeSO_4-H_2SO_4$.

Effect of ferrous sulfate concentration:-

Throughout a set of experiments, it is clear that the concentration of ferrous sulfate of the liquor has a moderate effect on the crystallization of ferrous sulfate heptahydrate. The results shown in figures (1-14) indicate that liquors with low ferrous sulfate concentrations give low yields of crystallized FeSO₄.7H₂O. The yield increases sharply, then slightly with the increase of concentration.

Effect of 1-propanol to liquor volumetric ratio:-

From the data presented in figures (1-14), it is shown that, increasing the volumetric ratio increases the recovery of $FeSO_4.7H_2O$. It is clear that, increasing volumetric ratio from 1:1 to 2:1 increases the recovery largely, but from 2:1 to 3:1 the recovery increases slightly. The increase of recovery with the increase of volumetric ratio may be attributed to the decrease of solubility of ferrous sulfate heptahydrate with the increase of the amount of 1-propanol in the system C₃H₇OH- H₂O-FeSO₄-H₂SO₄. From all these results, it is economically to use the 2:1 volumetric ratio.

Effect of temperature:-

The effect of temperature was studied at 20 and 30 °C. The results indicate that the increase of the temperature of the crystallization decreases the yield of crystallized ferrous sulfate heptahydrate. The decrease at low ferrous sulfate concentration is greater than that at high concentration. Because of the waste pickle liquor is discarded at about 140 g/l ferrous sulfate, temperature of 20°C is suitable for crystallization of ferrous sulfate heptahydrate.

CONCLUSION:

From the previous discussion of results, the recommended conditions for crystallization of ferrous sulfate heptahydrate are, high ferrous sulfate concentration, low sulfuric acid concentration, 1-propanol to liquor volumetric ratio of 2:1 and temperature of 20 °C.

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Fig.(1): Effect of concentration of sulfuric acid and 1-propanol to liquor volumetric ratio on recovery of ferrous sulfate heptahydrate. Conditions:

Ferrous sulfate conc. : 27 g/l Temperature : 20 °C



Fig.(3): Effect of concentration of sulfuric acid and 1-propanol to liquor volumetric ratio on recovery of ferrous sulfate heptahydrate. Conditions: Ferrous sulfate conc. : 81 g/l Temperature : 20 °C



Fig.(2): Effect of concentration of sulfuric acid and 1-propanol to liquor volumetric ratio on recovery of ferrous sulfate heptahydrate Conditions: Ferrous sulfate conc. : 54 g/l

Temperature : 20 °C



Fig.(4): Effect of concentration of sulfuric acid and 1-propanol to liquor volumetric ratio on recovery of ferrous sulfate heptahydrate Conditions: Ferrous sulfate conc. : 108 g/l Temperature : 20 °C



Fig.(5): Effect of concentration of sulfuric acid and 1-propanol to liquor volumetric ratio on recovery of ferrous sulfate heptahydrate. Conditions:

Ferrous sulfate conc. : 135 g/l Temperature : 20 °C



Fig.(7): Effect of concentration of surfuric acid and 1-propanel to liquor volumetric ratio on recovery of ferrous sulfate heptahydrate. Conditions: Ferrous sulfate conc. : 27 g/l Temperature : 30 °C



Fig.(6): Effect of concentration of sulfuric acid and 1-propanol to liquor volumetric ratio on recovery of ferrous sulfate heptahydrate Conditions:

Ferrous sulfate conc. : 162 g/l Temperature : 20 °C



Fig.(8): Effect of concentration of surffuric acid and 1-propanol to liquor volumetric ratio on recovery of ferrous sulfate heptahydrate Conditions: Ferrous sulfate conc. : 54 g/l Temperature : 30 °C



Fig.(9): Effect of concentration of sulfuric acid and 1-propanol to liquor volumetric ratio on recovery of ferrous sulfate heptahydrate. Conditions:

Ferrous sulfate conc. : 81 g/l Temperature : 30 °C



Fig.(11): Effect of concentration of sulfuric acid and 1-propanol to liquor volumetric ratio on recovery of ferrous sulfate heptahydrate. Conditions:

Ferrous sulfate conc. : 135 g/l Temperature : 30 °C



Fig.(10): Effect of concentration of sulfuric acid and 1-propanol to liquor volumetric ratio on recovery of ferrous sulfate heptahydrate Conditions:

Ferrous sulfate conc. : 108 g/l Temperature : 30 °C



Fig.(12): Effect of concentration of sulfuric acid and 1-propanol to liquor volumetric ratio on recovery of ferrous sulfate heptahydrate Conditions:

Ferrous sulfate conc. : 162 g/l Temperature : 30 °C



Fig.(13): Effect of concentration of sulfuric acid and temperature on recovery of ferrous sulfate heptahydrate. Conditions:

1-propanol to liquor ratio : 2:1 Ferrous sulfate concentration : 81 g/l



Fig.(14): Effect of concentration of sulfuric acid on recovery of ferrous sulfate heptahydrate at different ferrous sulfate concentrations.. Conditions:

1-propanol to liquor ratio	:	2:1
Гетрегатиге	:	20 °C

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Kineticrof formation of sodium chromate from El-Baramiya chromite ore concentrate with soda ash pellets.

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ABSTRACT: In Egypt, about seventeen occurrences of chromite ore are located among El-Baramiya region in Eastern desert. El-Baramiya ore shows that dressing is required to obtain concentrate with high chromium content. Egypt imports sodium chromate for local industries from abroad and this is at the expense of our foreign currency. Thus the converts of El-Baramiya concentrate to produce sodium chromate is very essential to the local industries. This work is concerned with the factors affecting on the pelletization of El-Baramiya chromite ore concentrate with soda ash and the kinetics of roasting process for the production of sodium chromate from pellets.

The results obtained can be summarized in the following spots:-

1-The optimum factors affecting on formation of green pellets are residence time which was found to be 15 min. and 22% water addition.

2-The maximum amount of chromic oxide recovery was obtained at the optimum condition of pelletization.

3-The percentage of chromic oxide recovery was found to depend on the size of the roasted pellets and reached to maximum value for pellet size -14+11.2mm.

4-The recovery of chromic oxide from roasted pellets was found to depend on the temperature and time of roasting.

5-The reaction kinetics of sodium chromate formation was controlled by solid state diffusion.

1-INTRODUCTION

In Egypt, sufficient reserves of chromite ores are present mainly at El-Baramia region in the Eastern desert. Chemical analysis of El-Baramia ore shows that dressing is required to obtain concentrates with high chromium content. Converts of this concentrate to high and low carbon ferro-chrome and/or to produce chrome chemicals such as sodium chromate and chromium metal are very essential to local industries.

Dichromate and chromic acid are used for the production of some chemical compounds, such as the formation of ketosteroid and other pharmaceutical products; the oxidation of acenaphathene to naphathelic acid; the conversion of o-toluennesulfonamide to \leq

o-sulfonamidobenzoic acid in saccharin manufacture, benzoic acid from toluene, and quinone from aniline(Kirk 1964).

A small amount of dichromate is employed as oxidant in the bleaching of fats, oils, and waxes. The largest consumption in this application is in bleaching of the montan wax derived from lignite (Marsel, 1956). Dichromate oxidant mixtures also find limited uses in the purification of crude carbon dioxide, acetic acid from wood distillation, acetylene, and phosphorus, in the cleaning of the glassware.

Sodium chromate and dichromate are manufactured by roasting chrome ore with soda ash (Kirk 1964) as follows:- The chrome ore is crushed, dried and ground in ball mill to a fineness of 90-98 % less than 200 mesh. The pulverized ore is mixed with soda ash and with a diluent. The proportion of soda ash is such as to obtain the optimum economy in recovery of chromium values from the ore. The chemical reaction of sodium chromate production is as follows:

 $2Cr_2O_3 + 4Na_2CO_3 + 3O_2 \rightarrow 4Na_2CrO_4 + 4CO_2$

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The production of sodium dichromate is produced from sodium chromate solution with sulphuric acid at pH = 3.

Some investigators (Vilnyansku et al 1947, Farbenind 1926, Ei-Tawil et al 1991), indicated that the higher chromate recoveries were achieved at temperature ranging from 850-1200 °C. Some other authors (Tripathy et al 1994, Gorochova et al 1973), illustrated that oxygen is included as a reactant and the rate of reaction is proportional to the oxygen partial pressure.

It was found that the reaction between Na₂CO₃ and Cr₂O₃ is not a solid reaction

(Tripathy et al 1994).But (Slan et al 1992), suggested that the production of Cr (VI)oxide from domestic chromite concentrates by alkali fusion is limited by the product layer mass transfer control.

The present work was undertaken to investigate the basic factors influencing the pelletization process, the roasting process of chromium concentrate with soda ash in air or oxygen to produce sodium chromate, and also the kinetics of roasting processes for the production of sodium chromate from pellets in a fixed bed reactor.

It is hoped that the results of this work may contribute to the long line of efforts expended to choose an adequate local process for the roasting of local chromite ore concentrate to recover chromium as sodium chromate on an industrial scale.

2-Materials and Experiments

2.1 Materials

A representative sample of chromite concentrate from El-Baramiya region was used in this investigation. The chemical analysis of the chromite ore concentrate was $Cr_2O_3 = 44.28\%$, $Al_2O_3 = 16.9\%$ MgO=18%, FeO =11.73%, and $SiO_2 = 6.9\%$ and L.O.1. = 0.79%. Soda ash was delivered from Masr Chemical Co. (Alexandria) containing 90% Na₂CO₃.

2.2 Method of pelletization

The chromite ore concentrate sample with soda ash (having size -0.074mm) was fed to the laboratory disc pelletizer 40 cm diameter and 10 cm depth. The predetermined amount of added water was then sprayed onto the rolling bed of the material in the pelletizer. At the end of the test, the sample was collected and screened to collect the -16+8mm pellets. Then the pellets (-16+8 mm diameter) were roasted in the thermal balance furnace in gas atmosphere (air or oxygen) at constant temperature for certain time. The roasted material was ground and dissolved in hot water and then chromic oxide was determined.

The chromic oxide recovery was determined as follows:

Chromic oxide recovery = (chromic oxide in sodium chromate/ Chromic oxide in the feed).

3-RESULTS AND DISCUSSION

There are many factors affecting the green pellets formation such as:-

- 1- The amount of water added to the charge. disc pelletizer.
- 2- The residence time of the charge material in the

3- The disc inclination angle.

4- The disc rotating speed.

5- Particle size of the charge.

In this work the disc inclination angle, disc rotating speed, particle size of the charge and the feed charge weight were kept constant at the following values 60° , 17 rpm, 0.074 mm and 300 grams respectively. The amount of water added to the charge and the residence time of the charge material in the disc pelletizer were studied.

From the previous work (Shalabi et al 1999), it was found that the optimum chromic oxide recovery for a mixture of soda ash and chromite ore concentrate from the same ore was reached at the ratio $(Na_2CO_3:Cr_2O_3 =$ 2.5). Thus the pelletization process was carried out for these mixtures at the above-mentioned ratio.

3.1 Effect of residence time of the charge on the productivity and quality of the produced pellets.

Sastry et al 1973 reported that the residence time varies linearly with pellets diameter. Thus, series of experiments were carried out to determine the optimum residence time of the charge mixture while the amount of water added was kept constant at 22%.

Table 1 shows the effect of residence time of raw materials in disc pelletizer on the productivity of green pellets and their strength. It is noticed that the productivity of pellets(+8-16mm) diameter, average compressive strength and average drop number are increased as the residence time of the material in the disc pelletizer increased till it reached 15 min. and approached 66.5, 5752 gm/pellets and 37.5 drop number respectively. This may be due to the growth in the formed nuclei of pellets. On the other hand, the decrease in the productivity of pellets, average drop number and average compressive strength for more than 15 min. residence time, may be due to the flushing of some moisture content during their growth (Naque, et al 1993, Ahmed 1993).

Table1 Effect of residence time of raw materials in disc pelletizer on the productivity of green pellets and their strength.

Parameters	Parameters Residence time, min.		in.
	10	15	20
Productivity of pellets (-16+8 mm),%	34	66.5	43
Average drop number	40	73.5	50
Average crushing strength, gm/pellet	3100	5752	5100

3.2 Effect of amount of water added on the productivity and the quality of the produced pellets.

The amount of water added for the production of suitable pellets is quite critical and the best way to determine the optimum amount of water for any given material can be achieved by practical experiments (Ball, et al 1973). Series of experiments were carried out for determining the suitable amount of water for pelletization. The pelleization processes were done at residence time of material in disc pelletizer equal to 15 min., while the amount of water added was changed.

Table 2 illustrates the relationship between productivity of pellets (-16+8 mm) diameter, average crushing strength, drop number and the amount of water added to the charge.

amount of water, %	P,%	S	D	
18	35.5	4806	60.4	
22	67	5750	73.7	
26	46	5625	67.9	
28	33.5	4307	39.1	

Table 2 The relationship between productivity of pellets,(P) (-16+8 mm) diameter and strength(S) gm/pellets and drop number(L) and the amount of water added to the charge.

From Table 2, it is evident that the increase of water content from 18% to 22% increases the productivity of pellets of (-16+8 mm) diameter from 35.5% to 67%. Meanwhile, the average compressive strength and average drop number were increased from 4806 gm/pellet to 5750 gm/pellet and from 60.4 drop number to 73.5 drop number respectively. This is due to the increase of the amount of water to 22% which leads to an increase in the coalescence mechanism between the different conditioners of the charge (Seddik, et al 1978) and an increase in the number of liquid bridges between the charge particles (Betal 1961, Rumph 1977).

Any excess of water content more than 22% leads to an increase in the capillary diameter and this leads to a decrease in the capillary force (Seddik, et al 1978) subsequently the productivity and strength decrease. This may be also due to the fact that the excess water coats the pellets forming coherent film of water and neutralize the capillary effect (Ball, et al 1973).

From the above results it was found that the optimum residence time of the mixture in the disc pelletizer was 15 min. and the optimum amount of water added to the mixture was 22%.

3.3. Effect of Pellet Size on Chromic Oxide Recovery after Pellet Roasting.

The produced pellets (-16 +8 mm diameter) by optimum pelletization conditions were classified into three categories (-11.2 +8, -14 +11.2 and -16 +14 mm). Each was roasted in atmosphere of 1 l/min flow rate for 60 min at temperature 1100 $^{\circ}$ C.

Table 3 shows that the relation between pellet size and chromic oxide recovery, from which it is clear that the pellet size of (-14 + 11.2 mm) diameter gives highest chromic oxide recovery. While the average sizes (-16 + 8 mm) gave chromic oxide recovery near to that of the pellet size (-11.2 + 8 mm) with deviation ~ 1.3%.

The decrease of chromic oxide recovery with the increase of particle size more than (-14 + 11.2) is mainly attributed to the smaller surface area available for the reaction, and is also attributed to the fact that the diffusion path is longer for such pellets (Rumph 1977). While the decrease of chromic oxide recovery for pellet size smaller than (-14 + 11.2 mm) is mainly attributed to the increase of the surface energy which leads to the sintering of particles and decreased the surface area (Sayed 1981).

Table 3 The relationship between the pellet size and chromic oxide recovery.

Pellet size, mm .	chromic oxide ,%
-11.2 + 8	77.7
-14 +11.2	79
-16 +14	76.5
Average size (-16 +8)	77.7

Thus the pellet size (-14 +11.2 mm) was chosen for the kinetic study of Na₂CrO₄ formation .

3.4 The optimum residence time which gives the maximum chromic oxide ratio after roasting.

In these experiments, pellets of (-14+11.2 mm diameter) were roasted at 1100° C in air of flow rate 1 L/min for a period of 60 min. Table 4 shows the relationship between the residence time and the chromic oxide recovery. It is clear that the optimum chromic oxide recovery is at 15min. residence time. The low value of chromic oxide recovery at 10min. residence time may be due to the high porosity of pellets. The recovery of chromic oxide is 66% at 20 min residence time which may be due to flushing of moisture content of pellets which in turn increases the porosity of the pellets, This makes the gas flow inside the pellets faster, thus the reaction decreased.

Table 4 The relationship between the residence time and chromic oxide recovery.

residence time, min.	chromic oxide ,%		
10	58		
15	79		
_20	66		

3.5The optimum moisture added to the raw materials which give the maximum chromic oxide recovery.

Table 5 shows the relationship between the chromic oxide recovery and the amount of water added to the raw mix. The pellets of 14+11.2 mm) diameter produced in disc pelletizer were roasted at 1100° C for 60 min in air flow rate (1 L/min). It is clear that the maximum chromic oxide recovery was achieved when 22% water was added to the mixture of raw material to produce the green pellets. The maximum recovery of chromic oxide at 22% water may be due to the fact that the porosity of such produced pellets is the most suitable for the reaction.

Table 5 The relationship between the chromic oxide recovery and amount of water added to the raw mix.

Water added,%.	chromic oxide ,%	
18	65	
22	79	
26	70	
28	60	

3.6. The relationship between pellets weight/gas ratio and the chromic oxide recovery.

Figure 1 shows the relationship between the chromic oxide recovery and the solid/gas ratio, for the roasted pellets at 1100 °C for 60 min in 1 L/min air flow rate. It is clear that the recovery of chromic oxide increases markedly with decreasing

the solid/gas ratio. This may be due to the fact that the increase of sample weight means increasing chromite ore concentrate and soda ash, thus this sample needs more oxygen to complete the reaction (Shalabi 1973). Within the experimental range of solid / gas ratio (4.5-18.4 gm/L), the highest value of chromic oxide was obtained at 4.6 gm/L.

3.7. Kinetic of sodium chromate formation using pellets roasted in air and oxygen atmospheres.

The effect of roasting temperature on the kinetic reaction of chromic oxide recovery for pellet was studied where the other parameters (pellet weight 4.6 gm and pellet size (-14 +11.2 mm)) were kept constant.

3.7.1. Reaction kinetic of sodium chromate using pellets in air atmosphere.

The roasting is carried out in an air flow rate of 1 L/min. The results of these experiments are represented graphically in (Figure 2) which shows the relationship between chromic outide recovery and time of roasting at different temperatures. From (Figure 2) it is clear that the increase of roasting temperature at any constant roasting time increases the chromic oxide recovery. Also (Figure 2) can be divided into three slopes the first slope is the highest one, the second is somewhat lower and the third is the slowest.

From the previous results (Shalabi et al 1999) found that the controlling mechanism of sodium chromate formation by the briquetting is controlled by solid state diffusion mechanism. Thus the model of solid diffusion mechanism (K Int = $[(1 - (1-R)^{1/3}]^2)$ (Kroger et al 1954) was applied. Figure 3 confirmed that the reaction up to 60% is controlled by solid state diffusion mechanism.

3.7.2. Sodium chromate kinetic formation using pellets in oxygen atmosphere.

Kinetic formation of chromic oxide recovery in oxygen atmosphere of flow rate 0.21 L/min from pellets was also studied while the other parameters (pellet weight 4.6 gm and pellet size (-14 + 11.2 mm)) were kept constant.

Figure 4 shows the isotherm curve of chromic oxide recovery in oxygen atmosphere. The same trend of the effect of temperature was obtained (i.e. the increases of temperature increases the chromic oxide recovery at constant time). Also it is clear that the final stage of roasting is the slowest one.

The mechanism of solid diffusion was applied (K int = $[(1 - (1 - R)^{1/3})^2)$ and illustrated in(Figure 5).

Figure 5 confirmed that the mechanism of formation of sodium chromate is solid diffusion at the final zone of reaction.

4-CONCLUSIONS

1-The percentage of chromic oxide recovery depends on the size of the roasted pellets, where the maximum chromic oxide recovery percentage is obtained at pellet size (-14 + 11.2 mm) diameter.

2-The recovery of chromic oxide from pellets depends on the temperature of roasting, time of roasting and the amount of oxygen in gas flow rate.

3-The maximum recovery of chromic oxide is achieved by using pure oxygen at roasting temperature 1100 °C for 60 min roasting time.

4-The sample weight/gas ratio (gm/liter) has a pronounced influence on the chemical reaction.

5-The reaction kinetics of sodium chromate formation using pellet composed of a mixture of chromite ore concentrate and soda ash is controlled by solid state diffusion

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Figure 1. Effect of solid /gas flow ratio on the percentage of chromic oxide recovery.



Figure 2. Isotherm curves for chromic oxide recovery when air was used during roasting.



Figure 3. Relationship between $\ln t$ and $(1-(1-R)^{1/3})^2$ for the roasted pellets in air.



Figure 4. Isotherm curves for chromic oxide recovery for pellets roasted in oxygen atmosphere.

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Figure 5. Relationship between $(1-(1-R)^{1/3})^2$ and ln t for the roasting pellets in oxygen atmosphere.