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### POTENTIOMETRIC INVESTIGATION OF THE OXIDATION OF FERROUS CHLORIDE BY OXYGEN

by  
V. V. Lyul'ko

Khimiya i Khimicheskaya Technologiya, 6,  
911-915 (1962)

Translated from the Russian  
by Walter Urusky

August 1966

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The oxidation of ferrous chloride by oxygen proceeds very slowly at room temperature. The acceleration of this process and the study of its kinetics can be of great value in obtaining non-ferrous metals. This is the case when removing iron impurities from zinc concentrates.

The effects of various catalyzers, and hydrochloric acid and iron chloride concentrations on the oxidation rate of bivalent Fe ions at room temperature are discussed.

In literature there is a relatively large number of articles on the oxidation of ferrous sulfate<sup>1,2,3,4,5,6,7</sup> and very few articles on the oxidation of ferrous chloride<sup>8</sup>. The majority of these investigations were conducted at high temperatures and the results of many of these are contradictory. Thus, Michelson<sup>1</sup>, Shishakov<sup>2</sup>, Lamb and Elder<sup>3</sup>, and Byelopol'skiy and Urusov<sup>4</sup> studied the effect of sulfuric acid concentrations on the oxidation rate of FeSO<sub>4</sub>. Michelson, Shishakov, and Byelopol'skiy and Urusov believe that increasing the H<sub>2</sub>SO<sub>4</sub> concentration impedes the oxidation rate of ferrous sulfate. Lamb and Elder assume that decreasing the sulfuric acid concentration from 3.00 mol/l to 0.23 mol/l does not effect the oxidation rate of FeSO<sub>4</sub>, and that decreasing the acid concentration will further accelerate the process.

Posner<sup>8</sup> studied the effects of hydrochloric acid concentrations on the oxidation of ferrous chloride. He discovered that the oxidation rate of FeCl<sub>2</sub> is immeasurably small when the acid concentration is below 4N and that the oxidation rate increases with an increase of the acid concentration. The accelerating effect of hydrochloric acid is explained by the generation of a complex HFeCl<sup>2+</sup> ion.

Gromov<sup>5</sup>, Lamb and Elder<sup>3</sup>, and Snurnikov and Ponomarev<sup>6</sup> studied the effect of cupric sulfate on the oxidation rate of FeSO<sub>4</sub> and discovered that the occurrence of Cu<sup>3+</sup> ions accelerates the process. Gromov<sup>5</sup> ascertained that the catalytic action of cupric sulfate professes to be best with the addition of CuSO<sub>4</sub> in the amount of 1 g/l and the oxidation process made in a medium close to neutral at a pH of not less than 5. Snurnikov and Ponomarev<sup>6</sup> assume that the Fe<sup>2+</sup> ions begin to oxidize at pH 2.5 and the oxidation process of Fe<sup>2+</sup> is accelerated by increasing the pH to 5.2. Lamb and Elder<sup>3</sup> discovered that oxidation occurs in an acid medium (a solution of 0.1 M of H<sub>2</sub>SO<sub>4</sub>). Increasing the CuSO<sub>4</sub> from 0.1 mol/l to 1 mol/l will accelerate the process.

## EXPERIMENTAL PART

Ferrous chloride was obtained by reducing ferric chloride with hydrogen. The container for the hydrogen electrode was filled with 12.5 ml of a 0.5 M ferric chloride solution in 0.025 N of hydrochloric acid. Hydrogen was then passed through this solution until it became colorless.

Hydrogen and oxygen were obtained by electrochemical means. The quality reaction with potassium thiocyanate and the solution decolorization indicated the absence of  $\text{Fe}^{3+}$  ions. Oxygen was passed through the reduced solution at a rate of 10 ml/min. The platinated platinum plate which was half submerged in the experimental solution was measured for potential at preset time intervals. Thereafter, according to Peters<sup>9</sup>, the concentration of bivalent and trivalent Fe ions was determined. Rate constant was calculated according to the reaction equations of the first, second, and third order. The most stable constant appeared when calculated by the reaction equation of the first order (Table I).

Table I.

Oxidation of ferrous chloride by oxygen occurring with an  
activated carbon (0.502 N  $\text{FeCl}_2$ , 0.527 N  $\text{HCl}$ \*,  
20 g/l of carbon, temperature 18° C)

Start of test, hours	Concentration $\text{Fe}^{3+}$		Rate Constant		
	%	g-equi/l	$k_1 \cdot 10^5$	$k_2 \cdot 10^4$	$k_3 \cdot 10^4$
61.0	15.3	0.077	4.53	0.932	2.63
70.1	19.8	0.099	5.20	1.16	2.60
118.6	26.8	0.135	4.41	1.03	2.47
166.9	33.8	0.170	4.11	1.02	3.00
187.0	38.8	0.195	4.35	1.13	2.96
212.6	41.1	0.206	4.15	1.09	2.95
233.8	46.2	0.232	4.42	1.22	3.84
284.7	55.8	0.280	4.78	1.47	4.97
334.9	60.2	0.302	4.60	1.50	4.71
381.1	60.4	0.304	4.08	1.34	4.70
mean value			$4.46 \cdot 10^{-5}$	$1.19 \cdot 10^{-4}$	$3.48 \cdot 10^{-4}$

\*The reduced acid concentration in this case and all others pertains to the beginning of the test.

The concentration logarithm of the bivalent Fe ions linear dependency on time also demonstrates that the oxidation of ferrous chloride into ferric chloride is a reaction of the first order (Figures 1 and 2).

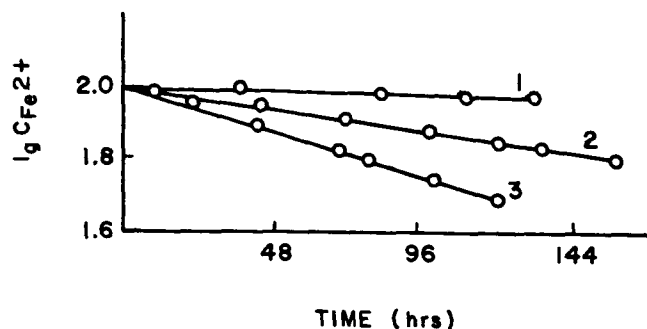


Figure 1. The change in the logarithm of concentration percentage of bivalent Fe ions with a change of time for various hydrochloric acid concentrations, g-equi/l: 1 - 0.525; 2 - 6.26; 3 - 8.52.

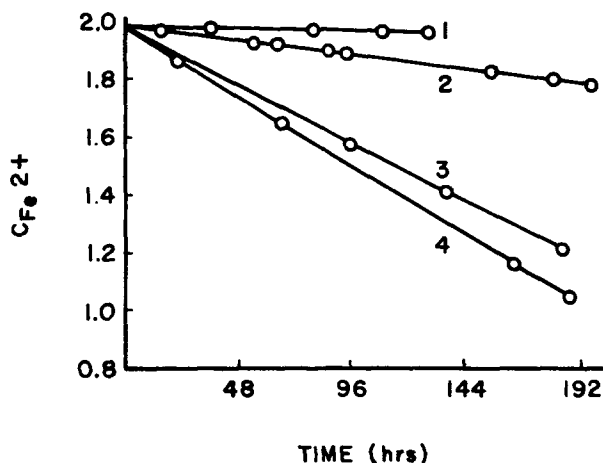


Figure 2. The change in the logarithm of concentration percentage of bivalent Fe ions with a change of time occurring with different amounts of activated carbon, g/l: 1 - without carbon, 2 - 20, 3 - 200, 4 - 300.

The tests were conducted under a condition of full saturation by the oxygen. Changing the amount of passing oxygen from 7 ml/min to 14 ml/min does not effect the oxidation rate of ferrous chloride.

In order to study the influence of catalyzers on the oxidation rate the tests were made using silver, activated carbon, platinated asbestos,

platinated platinum, platinated platinum and cobaltous chloride, platinated platinum and cupric sulphate, and platinated platinum and activated carbon. The platinum and silver catalyzers were in sheet form measuring 1 by 1.5 by 0.1 cm. These sheets were half submerged in the oxidizing solution. The activated carbon was added in an amount of 20 g/l; 1 to 2 g/l of cobaltous chloride; 1, 2, 25 g/l of cupric sulphate, and 3 g/l of platinated asbestos solution. The effect of the various catalyzers on the oxidation of ferrous chloride is shown in Figure 3.

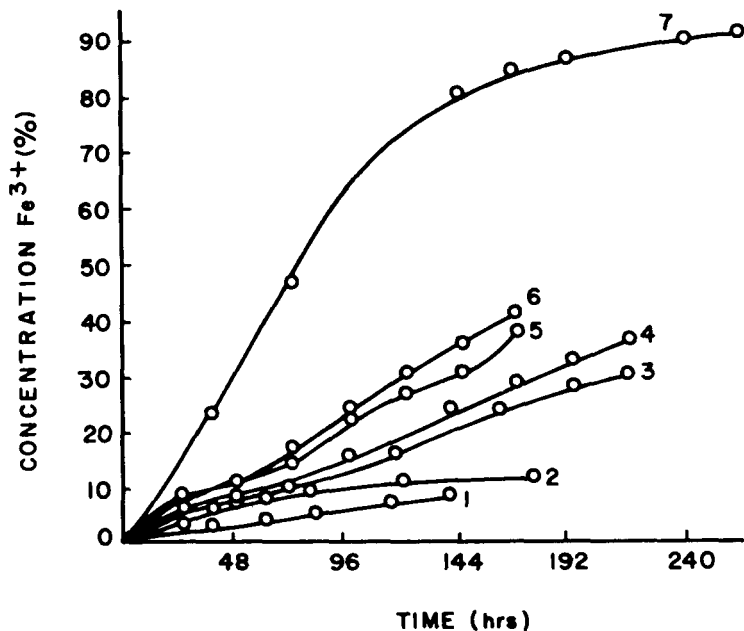


Figure 3. Change of the concentration percentage of trivalent Fe ions with the change in time occurring with various catalyzers. 1 - without catalyzer, 2 - silver, 3 - 3 g/l of platinated asbestos, 4 - platinated platinum and 1 g/l of  $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ , 5 - platinated platinum, 6 - platinated platinum and 1 g/l of  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ , 7 - platinated platinum and 200 g/l of activated carbon.

Increasing the amount of  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  from 1 g/l to 25 g/l only slightly accelerates the reaction speed (Table II), which does not agree with the Gromov<sup>5</sup> work on sulfuric acid solutions.

The best results were obtained using platinated platinum and activated carbon. It is in agreement with the Shishakov<sup>2</sup> and Lamb and Elder<sup>3</sup> research on sulfuric acid solutions and Posner's<sup>8</sup> research on chloride solutions.

Upon increasing the amount of activated carbon from 20 g/l to 200 g/l the oxidation rate of ferrous chloride increases by approximately

three to four times, and the amount of oxidized Fe increases from 20-30% to 70-75%, correspondingly. A further increase in the amount of activated carbon accelerates the oxidation process of ferrous chloride to a lesser degree (Table III).

Table II

Oxidation of ferrous chloride by oxygen in the presence of various amounts of copper sulfate (0.525 N HCl, 0.500 N FeCl<sub>2</sub>, 18° C)

Values $k_1 \cdot 10^4$ upon the addition of CuSO <sub>4</sub> · 5H <sub>2</sub> O (g/l)		
1	2	25
0.431	0.486	0.530

Table III

Oxidation of ferrous chloride in the presence of various amounts of activated carbon (0.500 N FeCl<sub>2</sub>, 0.525 N HCl, 18° C)

Values $k_1 \cdot 10^4$ with a specific amount of activated carbon (g/l)			
20	200	280	320
with platinum			
0.450	1.50	1.68	1.95
without platinum			
0.296	1.20	1.36	1.66

The data in Table III show that the application of activated carbon allows oxidation to be conducted without platinated platinum.

During the oxidation of bivalent Fe ions without a catalyzer 5 to 8% of the Fe<sup>2+</sup> becomes oxidized in 144 hours; thereafter the process sharply decelerates and practically ceases; in 320 g/l of carbon 90 to 95% of the Fe<sup>2+</sup> becomes oxidized.



The lasting effect of the activated carbon is obtained by washing and drying it after each use as a catalyzer. The results of the investigations show that the catalyzing effect of the activated carbon was barely reduced after repeated use.

Because literature shows contradictory data on the effect of acid on the oxidation rate of bivalent Fe ions, tests were conducted in 0.525 N, 2.14 N, 2.42 N, 6.26 N, and 8.52 N solutions of hydrochloric acid (Table IV).

Table IV

The oxidation of ferrous chloride at various concentrations of acid (0.500 N FeCl<sub>2</sub>, 18° C)

Values $k_1 \cdot 10^4$ at concentration HCl (g-equi/l)				
0.525	2.14	2.42	6.26	8.52
without activated carbon				
0.0713	0.0616	0.0612	0.351	2.29
with 320 g/l of activated carbon				
1.66	1.63	1.56	1.78	1.81

The oxidation rate of the Fe<sup>2+</sup> ions does not change when the acid concentration changes from 0.525 N to 2.42 N, which corresponds with the data of Lamb and Elder<sup>3</sup> and contradicts the data of Byelopol'skiy and Urusov<sup>4</sup> and Michelson<sup>1</sup>.

In 6.26 N hydrochloric acid the oxidation rate significantly increases; the rate constant increases by almost six times. In a period of 144 hours 7 to 8% of the Fe<sup>2+</sup> becomes oxidized in the 0.525 N - 2.42 N HCl solutions, and from 25 to 30% in 6.26 N HCl. In 8.52 N hydrochloric acid the acceleration of the reaction becomes even greater; the rate constant increases another six times and in 144 hours from 90 to 98% of the Fe<sup>2+</sup> becomes oxidized. This is in complete agreement with Posner's<sup>8</sup> data.

If oxidation is conducted using activated carbon the change of the acid concentration professes to be negligible.

It was established by subsequent experiments that a change in the FeCl<sub>2</sub> concentration from 0.50 to 0.75 mol/l has no effect on the rate constant of this oxidation of bivalent Fe ions.

## CONCLUSIONS

1. In the oxidation reaction of ferrous chloride by oxygen of the air the following catalyzers were investigated: platinated platinum, platinated asbestos, cupric sulfate, cobaltous chloride, and activated carbon. Activated carbon proved to be the best catalyzer.

2. It was shown that upon increasing the amount of activated carbon from 20 g/l to 200 g/l the oxidation rate of ferrous chloride increased by approximately four times. A further increase in the amount of activated carbon increases the reaction rate to a lesser degree.

3. It was shown that without a catalyzer the change of the HCl concentration from 0.50 N to 2.42 N has no effect on the oxidation rate of  $\text{FeCl}_2$ . In concentrated solutions of 6.26 N and 8.52 N hydrochloric acid the oxidation rate sharply increases.

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