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The Effect of Ferric Iron Upon the Current Efficiency and the Deposition of Copper in the Electrowinning of Copper from Copper Sulphate Solutions

James L. Owings
THE EFFECT OF FERRIC IRON UPON THE CURRENT EFFICIENCY AND THE DEPOSITION OF COPPER IN THE ELECTROWINNING OF COPPER FROM COPPER SULPHATE SOLUTIONS

by

James L. Owings

A thesis
Submitted to the Department of Metallurgy
in Partial Fulfillment of the
Requirements for the Degree of
Bachelor of Science in Metallurgical Engineering

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PER SULPHATE SOLUTIONS

INTRODUCTION

In the hydrometallurgy of copper, probably the most widely used method 
for the precipitation of copper is by electrolysis using insoluble anodes. 
The process is similar in many respects to the electrolytic refining of copper; 
one of the main differences is the use of insoluble anodes in electrowinning 
as compared to the use of impure copper anodes in electrorefining. Impurities, 
however, are common to both processes. The nature of these impurities differ 
and the problem of purifying the electrolyte is also different.

Ferric iron is a harmful impurity to both processes. It is well known 
that the ferric iron oxidizes copper at the cathode according to the following 
reaction:

\[ 2\text{Fe}^{++} + \text{Cu} = 2\text{Fe}^{++} + \text{Cu}^{++} \]

When conditions are right, the reduced iron can migrate to the anode and be 
oxidized to the ferric state as is shown in the reaction which follows:

\[ \text{Fe}^{++} - (e) = \text{Fe}^{+++} \]

Thus, the previous reaction is nullified, and a cycle proceeds in which power 
is consumed without the desired deposition of copper. When ferric iron is 
present in sufficient quantity, copper is actually lost from the cathode faster 
than it can be deposited.

There are three methods available, or means in practice today whereby 
the ferric iron can be removed from the electrolyte, or made harmless to the 
process:

1. Enclose the anode in a diaphragm or cotton bag. Such a pro-
   cedure will keep the reduced iron from being oxidized back to 
   the ferric state.
2. Bring the solution in contact with SO₂ gas in reaction towers. The reaction involved will reduce the ferric iron to ferrous iron and generate sulphuric acid. The reaction involved is as follows: 

\[
\text{Fe}_2(\text{SO}_4)_3 + \text{SO}_2 + 2\text{H}_2\text{O} \rightarrow 2\text{FeSO}_4 + 2\text{H}_2\text{SO}_4
\]

3. Pass the solution over cement copper. All but a trace of the ferric iron is removed; however, no sulphuric acid is generated. The reaction is as follows:

\[
\text{Fe}_2(\text{SO}_4)_3 + \text{Cu} \rightarrow 2\text{FeSO}_4 + \text{CuSO}_4
\]

Although ferric iron can be removed by such methods, the question arises as to if it is feasible to add to the expense of the operation by giving the electrolyte additional treatment to remove the ferric iron. Also, at what concentration of ferric iron is its removal necessary?

In this investigation, an attempt is made to determine the allowable concentration of ferric iron that can be present for any desired current efficiency. Conditions of electrolysis are on an experimental basis and can not be compared, with accuracy, with those in practice. Work functions of electrolysis, such as current density, concentrations, temperatures, etc., are comparable to those in practice. A low and a high (approximate to data from practice) copper concentration and current density are used with varied concentrations of ferric iron. Data are recorded for each test made, and from the collection of data curves are drawn and discussions are made.

**APPARATUS AND EQUIPMENT**

For this investigation, the experimental electrolytic cell is put in series with a coulometer so that the ampere-hours used for each run can be accurately measured. Power for the electrolysis is supplied by a 6-volt
battery. Included in the circuit are an ammeter so that the current can be read and a close control on current density is maintained; a voltmeter to measure the impressed voltage from anode to cathode; and a variable resistor to regulate current and voltage. Figure 1 shows a wiring diagram of the apparatus. Figure 2 shows a photograph of the equipment and apparatus.

In both the cell and the coulometer, two anodes and one cathode are used. The insoluble anodes for the cell contain eight-per cent antimony and ninety-two per cent lead. Anodes in the coulometer are of starting-sheet copper as are the cathodes in the cell and coulometer. Wooden suspension bars support the electrodes in glass jars (2.5-liter capacity); these glass jars serve as containers for the experimental electrolyte and coulometer solution. The electrodes are suspended in the electrolyte at a predetermined depth so that a definite current density is maintained. An electric stirrer is employed in the cell to keep the electrolyte in constant circulation.

Since iron determinations are made periodically, and since the Permanganate method for iron is well suited for the types of solutions used, a rapid method is needed to reduce the ferric iron to the ferrous state for the titration. A Jones Reducer (suggested by Dr. Koch of the Chemistry Department) meets these requirements because of its simple construction, ease of handling, and speed of operation. The components of this reducer are as follows:

1. Graduated cylinder with stopcock
2. 40cm³ of 20-mesh zinc metal
3. Small wad of cotton

The cotton wad is placed in the neck of the cylinder and the zinc metal placed on top. To operate the reducer, a diluted sample of the solution to be analyzed is introduced into the cylinder in the presence of sulphuric acid, and
FIGURE 1
WIRING DIAGRAM
then the stopcock is opened and the cylinder drained. As the solution comes in contact with the zinc metal, the ferric iron is reduced to ferrous, and thus made ready for the titration with the standard solution of permanganate.

**PROCEDURE**

As mentioned above the working functions of electrolysis are kept as close as possible to those maintained in actual practice. It should be understood, however, that in a procedure of this type, which is carried on experimentally, the results obtained and the conditions maintained can only be compared in proximity to large-scale plant operations. In this work, two major tests were made; each test is composed of 16 runs. Each run was ran for a duration of one hour.

In test number one (Table 1), the average copper concentration was 36 g/liter; the current density was from 8.7 to 12 amp/ft² (11.7 amp/ft² was used in most cases); the concentration of H₂SO₄ ranged from 21 to 76 g/liter; and the ferric iron content was varied from 9.85 to 1.81 g/liter. The average temperature for each run was 25-degrees centigrade; no attempt was made to control this temperature. From anode to cathode, the impressed voltage was from 1.6 to 1.8 volts.

Test number one was made with a comparatively high copper concentration; that is, a concentration that compares to the highest used in practice. Test number two was made with a low copper concentration; a concentration as low as the lowest usually used in industry. In test number two (Table 2), the average copper concentration was 15 g/liter; current density was 11.7 amp/ft²; the H₂SO₄ concentration was the same as that used in test number one; and the ferric iron concentration ranged from 0.85 to 8.76 g/liter. The temperature and voltage were the same as in test number one (25-degrees centigrade and 1.6 to 2.0 volts).
Before and after each run, the ferric iron, ferrous iron, and copper content of the solution was determined by titration. First, the total iron content was determined by running a solution sample through the Jones Reducer and then titrating with standard permanganate solution. To obtain the ferrous iron content, a titration was made directly on a solution sample. The amount of ferrous iron subtracted from the amount of total iron is the ferric iron content. A standard permanganate solution was used for all iron determinations. To determine the copper content, the Short Iodide Flouride method was used as prescribed in the Laboratory Manual For Elementary Quantitative Analysis by Edwin G. Koch, Ph.D., Professor of Chemistry at the Montana School of Mines, Butte, Montana.

After each run, the coulometer cathode and the cell cathode were weighed to determine the amount of copper lost or deposited during electrolysis; from a comparison of these weights, the current efficiency and the effect of the ferric iron was noted.

Glue was added for each test; the concentration of glue in solution was held at approximately 0.1 g/liter, and was only varied to check the effect, if any, of the glue on the action of the ferric iron. The glue addition will be discussed in a following section of this thesis.

**DISCUSSION OF RESULTS**

**Test No. 1:** The disastrous effect of ferric iron on the current efficiency can be seen in Plate 1. With concentrations of ferric iron up to 3.5 g/liter, current efficiencies comparable to those obtained in practice can be reached. When the ferric iron content reaches the vicinity of 6 g/liter, however, the current efficiency is very close to zero; therefore, the curve shows that the decrease in current efficiency with an increase in ferric iron is not gradual, but very rapid.
At high concentrations of ferric iron, copper is actually lost from the cathode. As the concentration of ferric iron increases, the amount of copper lost from the cathode also increases. Plate 2 shows at what approximate ferric iron concentration the amount of copper being deposited is in equilibrium with that being dissolved. It should be remembered that this ferric iron concentration, and those mentioned above in relation to current efficiency have been found while using a copper concentration of approximately 36 g/liter. During test number two, in which the copper content was about 15 g/liter, results of noteworthy difference were obtained.

Test No. 2: As mentioned before, the procedure used in this test was the same as that used in number one; except, that the copper concentration was reduced and held at approximately 15 g/liter.

Plate No. 3 shows that the effect of ferric iron on the current efficiency is not quite so intense as in test number one. The curve is more gradual, and a zero-current efficiency is not reached until the ferric iron content approaches 9 g/liter. As compared to test number one, the limit for ferric iron in order to maintain reasonable current efficiencies has dropped from 3.5 g/liter to around 2.5 g/liter for a copper concentration of 15 g/liter. Plate No. 4 shows at what ferric iron content copper is lost from the cathode with the conditions prescribed in test number two. From the path of the curve, several conclusions can be drawn:

1. Curve A shows the zero point to be at 6 g/liter ferric iron. This conclusion compares very closely with the zero point shown in Plate No. 2 while using a copper concentration of 36 g/liter.

2. Curve B shows the zero point to be at 9 g/liter.
One does not have to hesitate to say that no definite conclusion can be drawn from Plate No. 4. An approximate conclusion can be drawn, however, in saying that with over 6 g/liter ferric iron, copper is being dissolved from the cathode very rapidly. The undeterminable results and the nonreproducibility of the data for this investigation is probably due, in part, to the variables that were introduced into the procedure without a proper knowledge of their probable effect. These variables are discussed in the following section.

VARIABLES OF ELECTROLYSIS

Variables of electrolysis is a term which has been chosen to represent the controllable functions of electrolysis such as current density, addition agents, agitation, temperature, etc. From the collected data for this investigation, it is felt that the below mentioned variables had a major influence on the results that were obtained.

**Current Density**: The current density of 11.7 amp/ft² which was used in most cases throughout this work, is just under the average current density used in practice. By keeping this current density constant, it should not be considered to have any appreciable influence on the general effect of ferric iron in copper solutions of from 15 to 36 g/liter, and with current densities from 5.5 to 20 amp/ft². A slight change in the current density while experimenting, however, will cause nonreproducibility in results. The above statement was made on the following grounds:

With a higher current density, not only will the copper deposit more rapidly, but also, the ferric iron is reduced more quickly, and thus the current efficiency is increased. This effect can be seen on Table No. 2, Run No. 15. It is shown that the ferric iron concentration is 8.76 g/liter as compared to 7.62 g/liter in Run No. 14. With an increase in current density (12.5 amp/ft² in Run
No. 15, and 11.7 amp/ft² in Run No. 14), a 11.7% current efficiency was reported in Run No. 15, and in Run No. 14, copper was lost even though the concentration of ferric iron was higher in Run 15 than that in Run 14.

Addition of Glue: At the beginning of this investigation, it was decided that glue should be added in small amounts so that a finer and more uniform deposit of copper could be obtained. The average concentration of glue was kept in the vicinity of 0.1 g/liter except in Test No. 2, Run No. 16, where the glue concentration was increased to 1.25 g/liter so that its effect, if any, might be noted. In reference to Table No. 2, Runs 14 and 16, it can be seen that the ferric iron concentration is 7.62 g/liter in Run No. 14 and 8.53 g/liter in Run No. 16; other than this, the other conditions of electrolysis compare very closely. To check the effect of glue, the glue concentration was increased to 1.25 g/liter in Run No. 16 as compared to 0.1 g/liter of glue in Run No. 14. The result was quite obvious. The current efficiency found for Run No. 16 was 5.7%; whereas, in Run No. 14 copper was lost from the cathode.

If the results of this test are sufficient, glue in ample quantity has some effect on the action of ferric iron; that is, glue must in some manner surpress the action of ferric iron. Reference materials that give any detail about the effects of glue or gelatin were not available to this writer.

Circulation of the Electrolyte: Agitation of the electrolyte can cause an increase or decrease in current efficiency. When the electrolyte is in constant circulation, ions of copper and iron are brought into the immediate area of the electrodes more readily; therefore, the current is more efficient for depositing copper, reducing iron, and oxidizing iron.
In the experimental work done on this thesis, there was the question of whether or not the cell solution was receiving uniform agitation. Since the electrical stirrer was introduced at the head of the cell, one can say that the solution in the opposite end was not being agitated as effectively. If this is so, the ions of copper and iron were not being distributed at a uniform rate throughout the cell; thus, the side of the cathode which was closest to the stirrer would be subject to greater corrosion by the ferric iron because the ferric iron is reduced faster with faster circulation. An indication of this was shown by the formation of brown rings or stains on the side of the cathode facing the stirrer; the brown discoloring is due to the resolution of copper by ferric iron.

CONCLUSIONS AND RECOMMENDATIONS

From the experimental work and results of this thesis, several conclusions can be drawn as to the effect of ferric iron on the current efficiency and copper deposition in the electrowinning of copper from a copper sulphate solution.

1. It is conclusive to the investigator and writer of this thesis that copper concentration has an effect or influence on the action of ferric iron. If one does not consider the points below the zero line on Plates 2 and 4, and only considers the curves on Plate 1 and Plate 3, it can be seen that the current efficiencies reported for any one ferric iron concentration, while using different copper concentrations, are not in agreement. With a lower copper concentration, the curves seem to level off in contrast to the steepness displayed with high copper concentrations.
2. Current efficiencies in practice are usually in the range of 65 per cent. With this in mind and with reference to Plates 1 and 3, it can be said that a ferric iron concentration of from 2 to 3 g/liter is the maximum limit for safe and economical operation.

3. With the use of higher current densities, the allowable amount of ferric iron in solution is increased.

4. As a matter of interest, and without - I believe - practical value, an increase in the glue concentration with high ferric iron concentrations has a tendency to suppress the action of ferric iron and thus increase current efficiency.

5. A high rate of circulation can either increase or decrease current efficiency. As explained in the above section on circulation, with high circulation the current is more efficient for depositing copper, reducing ferric iron, and oxidizing ferrous iron.

The writer of this thesis does not feel that his work has added to the general knowledge of this subject. He does feel, however, that further work on this problem is yet necessary. If in the future more work is directed along this line, the author can recommend the following:

1. Most important is to take care in the number of variables that are added to the system, and only add additional variables in order to check their effect on the system.

2. Maintain a very careful control on current density and copper concentration.

3. The effect of increased or decreased temperature should be investigated.
These recommendations have been made in hope that future investigators working on this problem might progress where this worker has faltered.
TABLES AND GRAPHS
TABLE NO. 1

TEST NO. 1

<table>
<thead>
<tr>
<th>Run No.</th>
<th>Current Density (amp/ft²)</th>
<th>Current Efficiency (%)</th>
<th>Time (min.)</th>
<th>Copper Deposited in cell (grams)</th>
<th>Copper Deposited in Coulometer (grams)</th>
<th>Current (amp)</th>
<th>Voltage (volts)</th>
<th>Fe+++ (g/l)</th>
<th>Fe++ (g/l)</th>
<th>Cu++ (g/l)</th>
<th>Temp (°C)</th>
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PLATE 1
Copper Concentration = 36 g/liter

Per Cent
Current
Efficiency

Fe^{++}, g/liter
PLATE 2
Copper Concentration = 36 g/liter
Time of Run = 1 hour

Fe^{+++}, g/liter
<table>
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<th>Run No.</th>
<th>Current Density (amp/ft²)</th>
<th>Current Efficiency (%)</th>
<th>Time (min.)</th>
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<th>Copper Deposited in Coulometer (grams)</th>
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<th>Voltage (volts)</th>
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PLATE 3
Copper Concentration = 15 g/liter

Per Cent Current Efficiency
PLATE 4

Copper Concentration = 15 g/liter
Time of Run = 1 hour

Fe^{+++}, g/liter

Grams Copper Deposited

Grams Copper Lost

zero line
BIBLIOGRAPHY


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