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UNITED STATES PATENT OFFICE.

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MANUFACTURE OF ELECTROLYTIC IRON.

No Drawing. Application filed October 12, 1921. Serial No. 507,352.

To all whom it may concern:

Be it known that I, AUGUSTE BOUCHAYER, a citizen of the Republic of France, and resident of Grenoble, France, post-office address 8 Rue Lesdiguières, have invented new and useful Improvements in the Manufacture of Electrolytic Iron, which improvements are fully set forth in the following specification.

The present invention has for its object a process for the commercial manufacture of electrolytic iron. In order that such manufacture may be conducted on a truly commercial scale, it is necessary to be able to operate with a current of high density, while obtaining a product of very good quality and a maximum yield.

Consequently, it is necessary to observe various special conditions.

First of all, the electrolyte is prepared, which is constituted by a solution of ferrous chloride or of ferrous sulfate or a mixture of ferrous salts. If the electrolyte is used without any previous treatment, and which has then a green color, there will be obtained merely a deposit of wholly irregular form and without commercial value.

In order to obtain an even, compact and homogeneous deposit capable of being utilized after a slight reheating, it is advisable at the start to agitate the electrolyte in a receptacle, which may or may not be specially constructed, by merely rotating the anode, for example, in such a way as to produce an oxidizing action of the air at the same time as the phenomenon of electrolysis properly speaking. As the iron salts are very easily oxidized in air, the composition of the bath becomes changed and there is formed, in particular, oxychloride of iron, which reacts on the hydrogen bubbles collected on the cathode, and thereby eliminates the cause of the poor quality of the deposits.

Concurrently with this formation of oxychloride, the oxygen of the air likewise produces ferric chloride which, at the expense of the apparatus itself or of the deposit of iron on the cathode, is again converted into ferrous chloride. In order to avoid this loss of yield, as well as the corrosion of the apparatus, the electrolyte is caused to circulate through a mass of iron filings in a separate receptacle.

It will be observed that the solution is in good condition when it has attained a bright brown color and no longer foams. It can then be used commercially in the regular way; this foaming being due to the oxidation of the ferrous salts to ferric.

It is, moreover, necessary to observe the following operating conditions:

1. To rotate the cathode at a tangential speed which varies according to the density of the current. When operating under very low amperages, it is possible to rotate the electrode very slowly or even not at all; but with high current densities, it is necessary to increase the speed; the more the amperage increases the faster it is necessary to turn the cathode, without, however, exceeding a practical speed in order to avoid complications of apparatus, the inconveniences of centrifugal force, etc. By way of example, it may be stated that at 800 amperes per square meter the advisable tangential speed is 120 meters per minute.

2. To regulate the temperature according to the density of the particular current used and to maintain this temperature absolutely constant. It is possible to obtain a good yield operating in the cold with a very low current density, but if the amperage increases, it is necessary to equally increase the temperature, without, however, reaching the boiling point of the liquid, for at that moment there are formed in the interior of the electrolyte bubbles of gas or steam which interfere with the regularity of the deposit. At 1000 amperes per square meter, for example, the proper temperature is from 75 to 77° C. Moreover, and above all, it is necessary to maintain the temperature absolutely constant in order to obtain an extremely regular deposit; in fact, electrolytic deposits always contain a certain proportion of different gases which give rise to rather high tensions in the body of the metal itself. By varying the temperature these internal tensions are caused to vary, and there very often results a cracking and separation of the deposit which renders it worthless.

3. To regulate the concentration in accordance with the depolarizing power of the liquid and to maintain this concentration absolutely constant. In order to regulate the concentration, it is necessary to take into account the depolarizing power of the

liquid. In fact, when the concentration of the liquid varies, there is a variable electrolysis of the water; in other words, the quantity of hydrogen varies at the cathode, and this hydrogen has an action which is harmful or not according to the depolarizing power of the liquid. It is necessary, then, for any particular apparatus, to adopt a suitable concentration, for example, 20° Bé., and to maintain this concentration constant; otherwise, irregularities will take place in the deposit and the iron is no longer utilizable commercially. It is possible to replace the losses of liquid by continuously circulating in the apparatus a stream of fresh liquid instead of adding this liquid to the electrolyte from time to time and in relatively unimportant quantities.

4. Finally, it is advisable to maintain a speed of circulation as high as possible for the electrolyte around the anode. It has been established, indeed, that the more the speed of circulation increases, the more the phosphorus content is lowered.

By observing the above-mentioned conditions, excellent results are obtained.

In order to keep the liquid at the proper degree of oxidation,—which is readily recognized, as has already been stated above, by the bright brown color and the absence of foaming,—it has been usual, heretofore, to blow air into the liquid, regulating the blast by means of a valve. In this way, the difficulties are avoided which result from the employment of sealed vessels—i. e., vessels absolutely deprived of air—which necessitate the constant addition of a special depolarizer, as well as those resulting from the employment of open vessels which leave the entire surface of the electrolytic liquid in contact with atmospheric air.

By regulating the action of the air, the formation of a certain quantity of oxychloride of iron results, which acts as a depolarizer; and on the other hand, the formation of an excess of ferric salts, which would lower the yield, is avoided. It is possible, under these conditions, to operate at a current density of 1000 amperes and above per square meter, and to obtain an extremely homogeneous product, with a mean yield of 98% of the amperes.

Now, the above-described process, which is primarily that disclosed in United States patent to Anthelme Boucher, No. 1,086,132, granted February 3, 1914, and assigned to Société "Le Fer," assignee of the present case, is open to the objection that it requires the supply of a large volume of air, which necessitate the provision of considerable motive power and which, moreover, entails the further drawback of cooling the bath.

The present invention completely overcomes the disadvantages above indicated, by dispensing with the air blast or controlled action of the air and replacing the soluble anodes in one or more of the vats or vessels by insoluble anodes, graphite for example; the soluble anodes in the remaining vats being left unchanged. Due to the use of these insoluble anodes, the replenishment of the metal of the electrolyte is prevented, whereby the conversion of the ferrous salt to ferric, produced by its oxidation consequent upon the rotation of the anode, is augmented. The ferric electrolytic solution thus obtained is caused to circulate through a mass of iron filings contained in a separate receptacle, whereby ferric hydroxide ($Fe(OH)_3$) is formed which is thereupon utilized to effect a depolarizing action which is extremely similar to that produced by the controlled introduction of air. All of the other phases of the process, however, are retained.

What I claim and desire to secure by Letters Patent is:

1. The process for the industrial manufacture of electrolytic iron which consists in preparing an electrolyte solution of soluble ferrous salts, stirring the same in the presence of air until it assumes a brown colour and ceases to foam, and electrolyzing said solution by means of insoluble anodes.

2. The process for the industrial manufacture of electrolytic iron which consists in preparing an electrolyte solution of soluble ferrous salts, stirring the same in the presence of air until it assumes a brown colour and ceases to foam, electrolyzing said solution by means of insoluble anodes, and allowing access of air thereto during electrolysis.

3. The process for the industrial manufacture of electrolytic iron which consists in preparing an electrolyte solution of soluble ferrous salts, stirring the same in the presence of air until it assumes a brown colour and ceases to foam, electrolyzing said solution by means of insoluble anodes, and maintaining the temperature and concentration of said solution at a constant value during electrolysis.

4. The process for the industrial manufacture of electrolytic iron which consists in preparing an electrolyte solution of soluble ferrous salts, stirring the same in the presence of air until it assumes a brown colour and ceases to foam, electrolyzing said solution by means of insoluble anodes, and, maintaining at a constant value the temperature and concentration of said solution during electrolysis while allowing access of air thereto.

5. The process for the industrial manufacture of electrolytic iron which consists in preparing an electrolyte solution of soluble ferrous salts, stirring the same in the presence of air until it assumes a brown colour and ceases to foam, electrolyzing said

solution by means of insoluble anodes, and causing said solution to circulate around said anodes at as high a speed as possible.

6. The process for the industrial manufacture of electrolytic iron which consists in preparing an electrolyte solution of soluble ferrous salts, stirring the same in the presence of air until it assumes a brown colour and ceases to foam, electrolyzing said solution by means of insoluble anodes, circulating said solution around said anodes at as high a speed as possible, and allowing access of air to said solution during electrolysis.

7. The process for the industrial manufacture of electrolytic iron which consists in preparing an electrolyte solution of soluble ferrous salts, stirring the same in the presence of air until it assumes a brown colour and ceases to foam, electrolyzing said solution by means of insoluble anodes, circulating said solution around said anodes at as high a speed as possible, and maintaining at a constant value the temperature and concentration of said solution during electrolysis while allowing access of air thereto.

In testimony whereof I have signed this specification in the presence of two subscribing witnesses.

AUGUSTE BOUCHAYER.

Witnesses:
JULIAN KEMBLE LUEDERS,
LUCIENNE BRUERLT.