

The present invention relates to a process for the preparation of ferric chloride from ferrous chloride. It relates more particularly to the preparation of ferric chloride in aqueous solution which analyses by titration at between 35 and 45% by weight. This ferric chloride formulation is employed as a flocculating agent in water treatment. Reference may be made to Kirk-Othmer, 3rd edition, volume 24, pages 394-396 (1984) and to volume 10, page 498 (1980), where this application appears. The simplest process consists in digesting iron with concentrated hydrochloric acid; a solution containing approximately 36% by weight of ferrous chloride (FeCl_2) is thus obtained and this is chlorinated to obtain a solution of ferric chloride (FeCl_3) analysing by titration at about 41% by weight and capable of being employed directly as a flocculating agent. This 41% solution is the usual commercial form. A concentrated solution of FeCl_2 must be obtained because FeCl_2 and FeCl_3 can undergo partial hydrolysis in the course of a concentration by evaporation, giving HCl . The presence of HCl in FeCl_3 is a hindrance in water treatment. This process requires the use of concentrated hydrochloric acid.

US Patent 4,066,748 describes a process starting with a solution of FeCl_2 originating from a descaling bath. This process makes it necessary to concentrate the ferrous chloride and requires a two-step chlorination.

US Patent 3,682,592 describes a process similar to the preceding one but the ferrous chloride solution is brought into contact with oxygen.

A very simple process has now been found, which makes it possible to prepare a concentrated solution of ferric chloride by chlorination of a dilute solution of ferrous chloride.

The process of the invention consists in preparing ferric chloride from ferrous chloride and comprises the following steps:

a) chlorine and a solution containing ferrous chloride are brought into contact in the presence of a

solution containing ferric chloride,

b) the product obtained in a) is decompressed,

c) the liquid phase obtained in b) is partially recycled to step a); the remaining part forms the ferric chloride output.

The ferrous chloride is in the form of an aqueous solution, as is the ferric chloride. "In the presence of ferric chloride" means that ferric chloride is injected into step a) in addition to ferrous chloride, this ferrous chloride being then chlorinated to ferric chloride. Liquid or gaseous chlorine, or a gas liquid containing chlorine may be employed. The contact may be effected in any manner; it suffices to ensure a contact between the chlorine and the ferrous chloride. For example, a stirred reactor of a column of the distillation column or absorption column type may be employed. A column equipped with contact devices such as plates or packing rings or with a number of these devices may be advantageously employed. The ferrous chloride solution and the ferric chloride solution feed the column at the top and the chlorine is introduced at the bottom of this column. A little chlorine is collected at the top of this column if an excess thereof (relative to the quantity of ferrous chloride) has been employed, together with the gases which may have accompanied the chlorine and a little water vapour entrained by its vapour pressure. A ferric chloride solution is obtained at the bottom of the column. It would not constitute a departure from the scope of the invention to introduce the ferric chloride and ferrous chloride solutions at a number of points in the column; the same applies in the case of the chlorine, which may be introduced at a number of points.

Step b) consists in decompressing the solution collected in step a). This decompression causes a partial vaporisation of the water present in the ferric chloride solution; a concentration of the ferric chloride solution is thus produced. This decompression is carried out in any storage vessel; it suffices that its geometry should

permit the separation of the vapour phase and the liquid phase.

Step c) of the process of the invention consists in recycling a part of the above liquid phase to step a) and the remaining part which is recovered forms the output of ferric chloride; this quantity corresponds, in the number of moles, to the quantity of ferrous chloride introduced into step a).

This solution is advantageously returned to ambient temperature.

The ferrous chloride solution which feeds step a) may be of any concentration; it may also contain hydrochloric acid. A ferrous chloride solution not containing hydrochloric acid is advantageously employed, and this makes it possible to employ the ferric chloride directly as a flocculating agent for water treatment.

Step a) takes place advantageously at a moderate temperature, that is to say that this temperature, together with the residence time in step a) does not result in hydrolysis of FeCl_2 .

The reaction between the ferrous chloride and chlorine is complete. A ferrous chloride residence time of at least 10 seconds and preferably shorter than 4 hours is advantageously observed.

It is not necessary to chlorinate all the FeCl_2 ; ferric chloride specifications can sometimes permit from 0.1 to 1% by weight of FeCl_2 in the ferric chloride solution. It would not constitute a departure from the scope of the invention to employ an excess of chlorine relative to the stoichiometry.

The temperature of the reaction medium in this step a) is advantageously between 50 and 100°C. Any pressure may be used in step a); for convenience, the operation is carried out between atmospheric pressure and 6 bars, and preferably between atmospheric pressure and 1 bar gauge.

The pressure to which the product obtained in a) is decompressed is related to the quantity of water which

it is intended to evaporate. The greater the decompression, the more water is evaporated off. The quantity of water evaporated off also depends on the flow rate of the solution which is decompressed; the larger this quantity,
5 the more evaporated off. The quantity of ferric chloride which is recycled is advantageously between 1 and 10 times the output.

The quantity of water to be evaporated off depends on the concentration of the FeCl_2 solution and on
10 the desired concentration for the FeCl_3 produced. The quantity of FeCl_3 which is recycled also makes it possible to control the temperature; the higher the recycle flow rate, for the same quantity of FeCl_2 , the lower the temperature at the outlet of a). The heat energy supplied
15 by the FeCl_2 chlorination reaction is essentially consumed by the decompression, which is reflected in a lowering of temperature. If it is desired to evaporate off more water, energy must be supplied; the product obtained in a) is advantageously heated before the decompression. The
20 heating advantageously does not exceed 110°C , so as not to decompose the FeCl_3 . The heating-decompression cycle can also be repeated a number of times; it is obviously sufficient to recompress the liquid phase after the decompression, for example with the aid of a pump.
25 Advantageously, the decompression is taken to an absolute pressure of between 0.05 and 0.3 bars. The vacuum is provided, for example, by a pump or a steam ejector.

According to another form of the invention, the ferric chloride solution may also be heated after the
30 decompression and before it is recycled into step a), but quite obviously after the withdrawal of the output, and this makes it possible to provide heat energy at a low level and hence to make full use of the heat energy and also not to raise the temperature of FeCl_3 too much.

35 According to another preferred form of the invention, and provided that the temperature levels are compatible, the heat energy may be transferred from the output of ferric chloride to the ferrous chloride

solution feeding step a), and this makes it possible to preheat it. This recovery enables some heat energy to be gained and hence more water to be evaporated off during the decompression and less additional heat energy to be supplied before this decompression.

The advantage of the present invention is that it makes it possible to employ a dilute solution of ferrous chloride to produce a concentrated solution of ferric chloride, while avoiding the use of evaporators.

The attached single plate shows an example of embodiment of the invention. (1) shows a column in which step a) is carried out, (3) is the decompression pot, (2) and (4) show pumps. The ferrous chloride solution is introduced at (5), the recycled ferric chloride at (6) and the chlorine at (7). The product is led by means of the pipe (8) via the exchanger (11) towards the decompression pot (3). The pot (3) is connected to the vacuum system by the pipe (9). The liquid phase is removed from the decompression pot by the pump (4). The output is recovered at the pipe (10). The inerts introduced with the chlorine are collected at (12).

The following examples illustrate the invention without limiting it.

EXAMPLE 1

A device in accordance with the attached single plate is employed, in which the column (1) is made of glass, with an inner diameter of 0.35 m and has a packing height of 10 m.

A solution of 296 kg/h of FeCl_2 and 704 kg/h of water at 80°C is introduced at (5) and a flow of 82.7 kg/h of chlorine and 5 kg/h of inerts at (7). The 5 kg/h of inerts are collected at (12) and a ferric chloride solution at 85°C at (8). The column (1) operates at a pressure of 1.1 bars absolute. The ferric chloride is reheated from 85 to 94°C by the exchanger (11) and it is then decompressed to a pressure of 0.25 bars absolute. A solution containing 2272 kg/h of FeCl_3 and 3269 kg/h of water is recycled at (6) and 379 kg/h of FeCl_3 diluted in

545 kg/h of water are withdrawn at (10).

EXAMPLE 2

5 The procedure is as in Example 1, but introducing a more dilute solution of FeCl_2 at (5), that is to say 296 kg/h FeCl_2 in 963 kg/h of water. The FeCl_3 solution at (8) is at 76°C ; it is reheated to 106°C and is then decompressed to 0.15 bars absolute; the temperature of the liquid phase drops to 62°C . The same concentrated solution of FeCl_3 is obtained at (10).

10 EXAMPLE 3

15 The procedure is as in Example 1, but in a column 0.4 m in diameter and with a more dilute solution of FeCl_2 being introduced at (5), that is to say 296 kg/h of FeCl_2 in 963 kg/h of water. The FeCl_3 solution at (8) is at 69°C ; it is reheated to 92°C and is then decompressed to 0.15 bars absolute. The same concentrated solution of FeCl_3 is obtained at (10); on the other hand, the recycle flow rate at (6) is 3790 kg/h of FeCl_3 diluted in 5450 kg/h of water.

CLAIMS

1. Process for the preparation of ferric chloride from ferrous chloride, characterised in that it comprises the following steps:

5 a) chlorine and a solution containing ferrous chloride are brought into contact in the presence of a solution containing ferric chloride,

b) the product obtained in a) is decompressed,

10 c) the liquid phase obtained in b) is partially recycled to step a); the remaining part forms the ferric chloride output.

2. Process according to Claim 1, characterised in that step b) is preceded by heating.

15 3. Process according to Claim 1 or 2, characterised in that the quantity of ferric chloride which is recycled is between 1 and 10 times the output.

20 4. Process according to one of Claims 1 to 3, characterised in that the ferric chloride output is employed for preheating the ferrous chloride solution upstream of step a).

