

UNITED STATES PATENT OFFICE.

JOSEPH VAN RUYMBEKE, OF CHICAGO, ILLINOIS, ASSIGNOR OF ONE-HALF
TO WILLIAM F. JOBBINS, OF SAME PLACE.

PROCESS OF MAKING BASIC PERSULPHATE OF IRON.

SPECIFICATION forming part of Letters Patent No. 453,137, dated May 26, 1891.

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To all whom it may concern:

Be it known that I, JOSEPH VAN RUYMBEKE, a subject of the King of Belgium, residing at Chicago, in the county of Cook and State of Illinois, have invented a certain new and useful Improvement in Processes of Making Persulphate of Iron, which is fully set forth in the following specification.

In the manufacture of persulphate of iron from iron ore heretofore the ore has been treated by heating it with a mixture of sulphuric and nitric acid. This treatment produces a sirupy solution, which may be evaporated to dryness, if desired.

My present invention relates to a new process for obtaining this persulphate by treating with sulphuric acid only.

The invention consists, briefly stated, in treating pulverized iron ore with sulphuric acid under heat, whereby an acid sulphate is obtained, and then submitting the latter product to a high degree of heat, as will be hereinafter specified.

I have discovered that if finely-ground hematite ore is poured into and thoroughly mixed with the proper proportion of sulphuric acid at about 60° Baumé and at a temperature of from 250° to 300° Fahrenheit an energetic action takes place rapidly, attended by the production of considerable heat, and the material rapidly solidifies, giving off considerable vapor and usually assuming a grayish color. The gray color will always appear when the acid used is at or above 60° Baumé and is heated to 300° Fahrenheit, or nearly this temperature. A weaker acid will produce the same reaction, but when it falls below 50° Baumé the action becomes very slow and it is necessary to heat it to a higher temperature than with the stronger acid, and below 45° Baumé it is almost impossible to obtain the reaction. The product is an acid sulphate containing also some free ore and some free acid. I have also discovered that if this gray product, containing yet some free acid and some free oxide of iron, as stated, is submitted to a high degree of heat of from 380° to 500° Fahrenheit it is transformed more or less rapidly, according to the temperature, into a persulphate by the combination of the free ore partly with the free acid and partly with the

acid sulphate. This product is somewhat basic and usually assumes a yellow color, though with some hematites the color is green. It becomes very soft and pulverizes with great ease.

In carrying out my process a hematite ore analyzing, say, fifty per cent. of iron or its equivalent of alumina is first thoroughly dried and then ground to a fine powder. This pulverized ore is mixed with sulphuric acid in about the proportions of about one hundred and fifty pounds of the ore and two hundred and fifty pounds of the acid at 60° Baumé. This acid must be previously heated to about 300° Fahrenheit—say from 250° to 300° Fahrenheit—and the mixing must be done rapidly and thoroughly. The mixture is then thrown upon a suitable floor, preferably of iron, before it solidifies, in which position the reaction continues and the mixture solidifies on the plates of the floor in a few minutes. The purpose of throwing the mixture upon the floor before solidification is simply to facilitate the breaking up of the mass. This product is an acid sulphate with some free ore and some free acid, and, as already stated, is usually of a grayish color. This material is then broken in pieces and is properly crushed in order to facilitate the subsequent application of heat. The pulverized material is placed in a suitable revolving drum heated to about 500° Fahrenheit—say from 380° to 500° Fahrenheit. This application of heat is continued several hours, depending upon the quantity and temperature of the mass treated. If the load of the drum be about two tons of the material and the temperature be kept at nearly 500° Fahrenheit, about six hours will be necessary to complete the operation and the time must be varied from this period according to quantity and temperature. When completely cooked and the second reaction mentioned above as produced by this application of heat is completed, the mass assumes a substantially uniform yellow, or in some other instances green color, as stated above. It is a somewhat basic persulphate of iron and is either in a pulverized condition, due to the revolution of the drum, or is readily pulverized by reason of its softness. A solution of the acid sulphate obtained by the first reaction is yel-

low, while a solution of the basic persulphate obtained by the last reaction is red.

It is to be noted from the description above that my process relates to the treatment of 5 natural oxide. The action of the persulphate on ferric hydroxide is well known in chemistry; but this action is very different from that on an oxide, especially a natural oxide, such as iron ore. It is well known among chemists 10 that hydroxide of iron is very easily dissolved by acids, whereas natural oxides are dissolved with difficulty, and in the case of diluted sulphuric acid are almost absolutely insoluble, even with a prolonged ebullition. The known 15 treatment of ferric hydroxide referred to above will not effect with the natural oxide the result described above, while the process herein set forth is entirely efficient and suc-

cessful in the treatment of such natural oxide. 20

Having thus described my invention, what I claim as new, and desire to secure by Letters Patent, is—

The herein-described process of making basic persulphate of iron, consisting in first 25 mixing sulphuric acid with oxide of iron and then submitting this mixture to the action of heat at from 380° to 500° Fahrenheit until the water has been expelled and a persulphate of requisite basicity is produced, substantially 30 as described.

JOSEPH VAN RUYMBEKE.

Witnesses:

CARRIE FEIGELL,
A. M. BEST.