

UNITED STATES PATENT OFFICE.

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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 RUYM-
BEKE, a subject of the King of Belgium, resid-
ing at Chicago, in the county of Cook and
State of Illinois, have invented a certain new
and useful Improvement in Processes of Mak-
ing 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 sul-
phuric and nitric acid. This treatment pro-
duces a sirupy solution, which may be evapo-
rated to dryness, if desired.

My present invention relates to a new pro-
cess for obtaining this persulphate by treat-
ing 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 pro-
duct to a high degree of heat, as will be here-
inafter 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 ac-
tion takes place rapidly, attended by the pro-
duction 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 com-
pleted, 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

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.

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:

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