

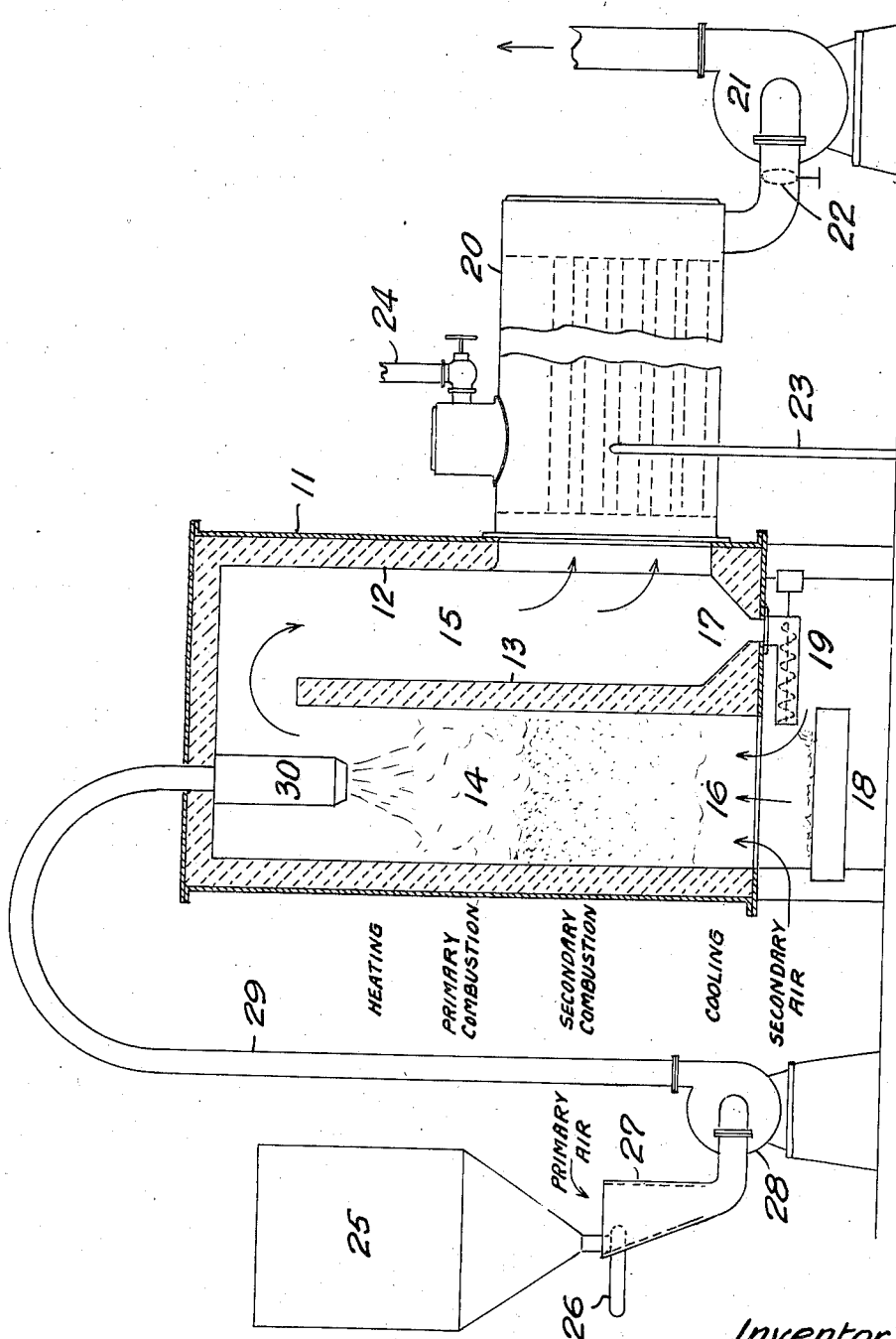
Feb. 11, 1936.

H. FREEMAN

2,030,627

PROCESS OF MAKING IRON OXIDE AND SULPHUR DIOXIDE FROM IRON SULPHIDE ORES

Filed March 5, 1930



Inventor
Horace Freeman
By *R. W. Allen*
Attorney

UNITED STATES PATENT OFFICE

2,030,627

PROCESS OF MAKING IRON OXIDE AND
SULPHUR DIOXIDE FROM IRON SULPHIDE
ORES

Horace Freeman, Shawinigan Falls, Quebec, Canada, assignor to Nichols Engineering & Research Corporation of Canada, Limited, a corporation of Canada

Application March 5, 1930, Serial No. 433,471

10 Claims. (Cl. 23—200)

This invention relates to improvements in the treatment of sulphide iron ores, such as pyrite and pyrrhotite and one object of the invention is to efficiently produce simultaneously sulphur dioxide and an iron oxide product, preferably magnetic and of high iron content and low sulphur content suitable for production of metallic iron.

Various other objects, features and advantages of the invention may be ascertained from the following description.

This application comprises a continuation in part of my copending application, filed January 17, 1929, Ser. No. 333,222, now Patent No. 1,812,397, granted June 30, 1931, the present application embodying certain alternative and additional features and improvements.

In the past, sulphide iron ores have been largely used in the production of sulphur dioxide by roasting or burning the ore with air. Of late, this has been accomplished usually in roasters in which the ore is caused by mechanical means to move slowly counter current to air. This type of roaster is well illustrated in multiple hearth apparatus in which the ore is introduced on the top hearth and moved by rakes across the hearth to fall onto the hearth below, the operation being repeated on each successive hearth. In this and other types of roasters in general use, the combustion of the sulphide is comparatively slow. While considerable heat is generated, the temperature of the burning sulphide rarely exceeds 1,000° C. and usually does not exceed 700° to 800° C. The iron oxide product resulting is in the form of unfused and unagglomerated small particles varying from red to purple in colour and is not magnetic to any appreciable or significant degree. In the operation of these mechanical roasters, the ore is cold as it enters the roaster, and thus cools the gases leaving the roaster, so that a considerable part of the sulphur dioxide is oxidized to sulphur trioxide. At no time during passage through the roaster is it ordinarily attempted to heat the ore sufficiently for fusion to result and the temperature in any part of the furnace is substantially the same as the temperature of the ore in that part. The solid residual product of this treatment is unfused iron oxide containing varying amounts of sulphur, usually from 2% to 6% depending on the ore, the type of roaster and the manner of operation. For example, if it is desired to produce from such roasters gas of high sulphur dioxide content, this can be done only by using a minimum of air and the result is a higher temperature in the

roaster causing somewhat excessive deterioration of the apparatus and a high sulphur residue in the iron oxide product.

According to this invention, the ore, reduced to finely divided form, is burned in suspension in air during slow passage through a combustion chamber, the amount of air being not materially in excess of that necessary for oxidation of all the sulphur and iron. In the absence of cooling by excess of air and by reason of the suspension of the particles, relatively widely separated, in air and their slow passage through the combustion chamber, a very rapid and complete combustion is brought about with liberation of heat which is largely taken up by the particles and causes fusion thereof, with consequent puffing and bursting.

In greater detail, the process is as follows:—The ore particles entering the combustion chamber pass first through hot gases flowing to the gas outlet and are thereby heated to the temperature of ignition and of fusion. The hot particles then pass into contact with air and combustion ensues at the surfaces of the particles. In the finely divided state in which the ore is treated, the ratio of surface to volume is very high and the particles being relatively widely separated and suspended in the air almost the whole volume of each particle, is presented at one time for oxidation, so that almost every particle burns completely and all the particles burn very rapidly. The removal of sulphur is more complete than is possible with mechanical roasters. The rapid oxidation liberates heat which is absorbed by the particles and serves to complete and maintain a state of fusion. In all probability the temperature of the fused particles is higher than can be obtained by other methods of roasting. This is evidenced by the fact that the resulting iron oxide is found to be fused, black in colour and highly magnetic. It is known that if red iron oxide, which is non-magnetic, is heated to or above 1650° C., it is fused and converted to a black magnetic oxide and from this fact it is concluded that, although temperatures of the order of 1650° C. in the combustion chamber may not be recorded, nevertheless the individual particles have been heated by their heat of combustion to this or higher temperature and are fused. The oxidized ore particles finally emerge from the combustion chamber through the cool entering air and are thereby chilled. The produced oxide may be subjected to magnetic separation for elimination of non-magnetic material such as incompletely oxidized particles and silica and other impurities which have en-

tered with the ore. The sulphur content of the product is very low, ranging from 0.3% to 2% according to the fineness of the ore. Owing to limitation of the air supply to that necessary in practice to effect complete combustion of the sulphur and iron, the amount of air admixed with the sulphur dioxide leaving the combustion chamber is much less than in the case of mechanical roasting. Also, owing to the absence of cooling by excess of air and to the high temperature attained by rapid and complete combustion of the ore, the sulphur dioxide leaves the combustion chamber at a temperature well above that at which it oxidizes to sulphur trioxide. The exit gases are preferably passed through a steam boiler wherein the sulphur dioxide is chilled so rapidly through the temperature range at which it oxidizes to sulphur trioxide that practically none of this latter gas is formed. Also, owing to limitation of the air supply, the sulphur dioxide produced by this process is less diluted than that produced from mechanical roasters.

It is found most convenient and practical to introduce the finely divided ore vertically downwardly into the top of a combustion chamber by means of a gentle current of air in amount much less than is necessary for combustion of the ore, and to supply the further air necessary for combustion by drawing the same upwardly through the combustion chamber. By adjusting the velocities of the two opposed air currents relatively to one another and to the force of gravity acting on the particles, the period of time occupied by the particles in descending through the combustion chamber may be regulated to provide ample time for combustion. In other words, the descent of the particles, due to their initial downward velocity and the acceleration of gravity, may be retarded by the upwardly moving current of air.

A form of apparatus which has been found suitable for carrying out the process, but to use of which the invention is not confined as other forms of apparatus may be used, is illustrated more or less diagrammatically in the accompanying drawing.

Referring more particularly to the drawing, 11 designates a gas-tight furnace shell, preferably of metal, having a refractory lining 12 and an internal refractory division wall 13 extending from the bottom of the furnace nearly to the top thereof. The dimensions of the furnace are such that the division wall constitutes two chambers 14 and 15, the greatest dimension of each of which is its height. The chamber 14, which is the combustion chamber, is open to the atmosphere at the bottom 16 while the chamber 15, which is a settling chamber, is closed to the atmosphere at its bottom 17. Any suitable means 18 for collecting and removing the solid furnace product is provided at such distance beneath the chamber 14 that it does not interfere with the free entrance of air into the chamber. Any suitable means 19 is provided at the bottom of the chamber 15 for removing solid furnace product while excluding air. Conveniently, the means 19 may discharge into the means 18 but this is not obligatory. A gas cooler 20, preferably in the form of a fire tube steam boiler, is connected to the furnace to receive gases from the lower part of the chamber 15. Preferably the boiler shell is connected directly to the furnace shell, as shown, and the furnace wall formed with a large exit opening giving free access to the boiler. A suction device 21, preferably a fan or centrifugal blower, is con-

nected to the end of the boiler remote from the furnace in such manner as to draw gases from the furnace through the boiler. A damper 22 may be provided between the blower 21 and the boiler. The boiler is provided with the usual water supply pipe 23 and steam delivery pipe 24.

Ore to be treated is delivered in finely divided form into a hopper bottom bin 25 from which it is fed through a regulating device 26 into a hopper 27 open to the atmosphere and connected at its bottom to the suction inlet of a fan or centrifugal blower 28. The discharge pipe 29 of the blower leads downward into the top of the combustion chamber 14 and terminates in any suitable sort of heat resisting nozzle 30.

The operation of the apparatus described is as follows:—Dry, finely divided metal sulphide, such as a flotation concentrate or finely crushed iron pyrites, is delivered to the bin 25 and is fed from the bin at measured rate through the regulator 26 into the open hopper 27, where it is caught by the current of air and drawn into the blower 28 and fed by it vertically downward into the top of the combustion chamber 14. By regulating the feeding device 26 and the speed of the blower 28, the rate of feed of ore into the furnace and the amount and velocity of air entering with the ore may be regulated as desired. The suction of the blower 21 draws air into the bottom of the combustion chamber and upwardly through this chamber and also draws the furnace gases and any residual air downwardly through the chamber 15 and through the boiler 20. The amount and velocity of air entering the bottom of the combustion chamber may be regulated by means of the damper 22 and by regulating the speed of the blower 21. The regulation is preferably such that the upwardly moving current of air offsets the initial downward velocity of the ore particles and partly offsets the acceleration of gravity, so that the descent of the particles through the combustion chamber is retarded. Most of the particles settle upon and are removed by the device 18. Fine particles which are carried over the wall 13 settle readily to the bottom of the chamber 15 and are removed by the device 19.

The process as conducted in the apparatus described is as follows:—The furnace having been suitably preheated, operation is commenced by feeding finely divided sulphide downwardly into the combustion chamber with less air than necessary for combustion of the sulphide and at the same time drawing the additional air necessary in practice for complete combustion of the sulphide upwardly through the combustion chamber. When the temperatures have been built up and the furnace is in full operation, there are three clearly defined zones established, the dimensions of which depend on the amounts and velocities of air and sulphide fed.

The upper zone, or heating zone, extends some distance downwardly from the nozzle 30. The atmosphere inherent to this zone is largely sulphur dioxide and nitrogen rising from the zones below. The temperature is too high to permit oxidation of the sulphur dioxide by the air entering with the sulphide and little, if any, air rises from the zones below. The sulphide particles passing down through this zone are heated to ignition and fusion temperature. If there is free sulphur in the ore, some of it may be volatilized and burned.

The middle zone, or zone of primary combustion, is quite clearly and visibly defined from the zones above and below. This zone extends down-

wardly from the level at which the sulphide particles ignite and is a zone of intense combustion and complete fusion in which most of the sulphur is burnt to sulphur dioxide with the air introduced along with the sulphide and with some of the air drawn in at the bottom of the combustion chamber. Possibly some of the iron is also burned. In this zone the sulphide particles are burned with air sufficient for oxidation of most of the sulphur and are thereby maintained at fusion temperature. The zone is filled with the characteristic flame of burning sulphur.

The bottom zone, or zone of secondary combustion, extends downwardly from the level at which the sulphide particles become incandescent to nearly the bottom of the chamber. In this zone the highly heated particles meet an excess of comparatively undiluted air drawn in at the bottom of the chamber. The particles are individually visible as a shower of brightly sparkling points of light and it is presumed that in this zone most of the iron and the residual sulphur is burned. The intense heat of the individual particles, indicated by the condition of incandescence, is largely absorbed by the particles, at the moment of liberation, in maintaining the fused state of iron oxide so that the temperature of the atmosphere in this zone is probably much below the temperature of the individual particles. The scintillating effect in this zone diminishes toward the bottom of the zone.

Below the bottom zone the fused particles are chilled and solidified in the incoming air and serve in some measure to preheat the air. The solid material collected is in the form of a heavy black powder composed of minute fused particles usually hollow.

From the foregoing description, it will be observed that the sulphide is first burned with less air than is necessary for complete combustion, the amount being, however, sufficient for oxidation of most of the sulphur, and is later burned with perhaps less air than necessary for complete combustion, the secondary air supply being materially in excess of that required for oxidation of the iron and remaining sulphur.

It will also be observed that the sulphide particles are maintained in suspension in air during the entire process of oxidation. By regulating the velocities of the two air supplies, the period of time during which the particles are held in suspension may be materially longer than would be normally occupied in the gravitation of the particles to the bottom of the combustion chamber and, in this way, ample time is provided for very complete combustion.

Because of limitation of the air supply as nearly to the theoretical requirements as is consistent with efficient operation, relatively very little air leaves the combustion chamber, so that the sulphur dioxide is but little diluted with air. The high temperatures of the gases in the combustion and settling chambers preclude oxidation of the sulphur dioxide to trioxide and the rapid cooling of the gases in the boiler to below 350° C. so reduces the time during which the sulphur dioxide is passing through the temperature range of oxidation that practically no sulphur trioxide is formed.

With the process as carried out in the above described apparatus, the temperature and time of treatment of the ore in the various zones of the roasting chamber may be very accurately regulated. There are several important features contributing to this possibility, among which the

following may be particularly noted. The free entrance of air substantially unobstructed by the roasted product at the bottom air inlet 16 is such that air may be drawn in over the entire area through which the treated ore falls out of the furnace. Thus there is free access of air to all parts of the lower portion of the furnace and substantially uniformly to all areas through which the treated particles fall and are chilled during their passage out of the furnace. Yet despite the fact that this entrance of air may take place over a considerable area, the quantity of air thus admitted may be carefully regulated by regulation of the damper 22 on the suction fan 21, this regulation being effected without disturbing the uniformity of the draft at one part of the furnace bottom as compared with any other part. In the treatment of many forms of ores, it is of advantage to use the suction method here disclosed, of drawing the auxiliary air into the bottom of the roasting chamber in lieu of forcing the air into the bottom of the chamber, as indicated in my prior patent above referred to. That is, by the suction method, I have found that the suspension of the ore particles during their passage through the furnace may be made more uniform at each level in the furnace by using the suction draft at the top of the furnace instead of forcing the air in at the bottom of the furnace. Also by using the suction fan 21 in lieu of forcing the air in at the bottom of the furnace, the travel of the gases through the settling chamber 15 and the boiler 20 may be made somewhat more uniform. In short, by properly regulating the damper 22 and by properly adjusting the flow of the ore into the apparatus by the regulator 26, the desirable roasting conditions within the furnace may be readily achieved for various grades of ores and so that the various zones of treatment above described are obtained.

With proper regulation of the roasting conditions, as above described, the relatively free admission of air into the bottom of the furnace also has the advantage that the ore particles are individually chilled somewhat while still in suspension and before the same reach the conveyor or other carrying device 18. With the individual fused particles thus chilled in suspension, any substantial agglomeration thereof on the conveyor may be avoided.

While the only oxidizing medium mentioned is air, it will be obvious that any gas mixture which contains the requisite free oxygen and no components which will react undesirably with the sulphide or formed oxides may be substituted for air. In the following claims, the term "air" is to be construed as including such gas mixtures.

While the sulphide introducing current of gas has been stated to be air, it will be understood that a non-oxidizing gas may be substituted and all the oxidizing effect obtained from a counter-current of oxidizing gas or, alternatively, both of the countercurrents of gas may be non-oxidizing and serve merely to maintain the sulphide particles in suspension and exposed to an oxidizing gas introduced separately from the two countercurrent streams.

While the invention has been described in detail with respect to particular preferred examples, it will be understood by those skilled in the art after understanding the invention, that various changes and further modifications may be made without departing from the spirit and scope of the invention, and it is intended therefore in

the appended claims to cover all such changes and modifications.

What is claimed as new and desired to be secured by Letters Patent of the United States is:—

1. A process of oxidizing iron sulphide ores by passing the same downwardly through a roasting chamber of substantial height, which comprises introducing the ore in finely divided form into a heated zone at the top of the chamber where the ore is preheated to ignition temperature by contact with rising hot furnace gases, allowing the ore to settle into a second zone in which it is heated to fusion temperature and burned with a deficiency of gaseous oxidizing agent whereby most of the sulphur is oxidized, allowing the ore to settle into a third zone in which it is further heated in a state of fusion and is burned with an excess of gaseous oxidizing agent whereby substantially all of the iron and the remaining sulphur is oxidized, and finally passing the ore through an incoming stream of said gaseous oxidizing agent as the ore passes out at the bottom of the furnace, said incoming stream being of such temperature that the fused particles are individually chilled while in suspension sufficiently to substantially avoid agglomeration.
2. Process for forming magnetic iron oxide in the form of finely divided fused particles, which comprises injecting with a gaseous medium finely divided iron sulphide ore into the top of a hot roasting chamber, allowing said particles to settle in the chamber through a rising current of oxidizing gas, the particles finally passing out at the bottom of the furnace through a relatively cold incoming stream of said oxidizing gas, the movement of the ore particles and the gases within the chamber being so controlled that the sulphur content of the ore is largely burned and the ore particles are individually fused while in suspension and are then chilled in suspension as they pass out through the incoming stream of oxidizing gas.
3. Process according to claim 2 and in which the gaseous products of combustion are withdrawn by suction through an outlet at the top of the chamber.
4. Process according to claim 2 and in which the gaseous products of combustion are withdrawn by suction through an outlet at the top of the chamber, and including regulating the draft through such outlet to produce the desired uniform flow of incoming oxidizing gas at the bottom of the chamber.
5. Process of forming magnetic iron oxide in the form of finely divided fused particles, which comprises roasting finely divided iron sulphide ore particles in suspension, the temperature of the roast being so controlled that the particles are individually fused in suspension, and allowing the particles to gravitate against an upwardly flowing stream of relatively cool oxidizing gas of such temperature that the particles while still in suspension are chilled sufficiently to avoid agglomeration.
6. Process of forming magnetic iron oxide in the form of finely divided particles, which comprises roasting finely divided sulphide ore particles

in suspension in an oxidizing gas, at a temperature causing fusion of the particles, the particles being allowed to gravitate from the area of roasting against an upwardly flowing stream of a relatively cool gaseous medium such that the particles while still in suspension are chilled sufficiently to substantially avoid agglomeration.

7. Process of forming magnetic iron oxide in the form of finely divided particles but substantially free of dust, which comprises roasting finely divided iron sulphide ore particles in suspension in an oxidizing gas, at a temperature causing fusion of the particles, allowing the particles to gravitate from the area of roasting against an upwardly flowing stream of relatively cool gaseous medium of such temperature that the particles while still in suspension are chilled sufficiently to substantially avoid agglomeration, and withdrawing upwardly by suction means from the area of roasting the gaseous products of the roast together with the dusty material.

8. Process of oxidizing metal sulphides which comprises introducing the sulphide in finely divided form carried in a gaseous medium into the top of a hot combustion chamber, introducing a countercurrent of air in an amount sufficient to substantially completely oxidize the sulphides in suspension, withdrawing the gaseous products at the top of the chamber and through a settling chamber by suction means, such a said countercurrent of air being maintained by adjusting said suction means so that the temperature in the combustion chamber and the settling chamber is maintained above that at which sulphur trioxide may be formed, and finally between said settling chamber and suction means passing the gases through means which quickly cools them to a temperature below that at which sulphur trioxide forms.

9. Process of oxidizing metal sulphides which comprises roasting the same in finely divided form in suspension in an oxidizing gas, at a temperature causing fusion of the particles, allowing the particles to gravitate from the area of roasting against an upwardly flowing stream of relatively cool oxidizing gas of such temperature that the particles while still in suspension are chilled sufficiently to substantially avoid agglomeration when collected, withdrawing upwardly from the area of roasting the gaseous products of combustion together with any dust occurring at said area, and then passing said products of combustion and dust downwardly through a settling chamber.

10. The process of producing iron oxide in the form of finely divided fused particles, which comprises oxidizing and roasting finely divided iron sulphide particles in suspension in an atmosphere of oxidizing gas, the temperature of the roasting and oxidizing being so controlled that the particles are individually fused and caused to become hollow while in suspension, and thereafter while the particles are still in suspension, applying air thereto of such temperature and amount that the particles while still in suspension are chilled sufficiently to avoid agglomeration.

HORACE FREEMAN.