Jan. 29, 1924. 1,482,327

J. A. THIBAULT

PRODUCTION OF METAL OXIDES, ESPECIALLY LEAD OXIDES

Filed Jan. 5, 1920

FIG. I.

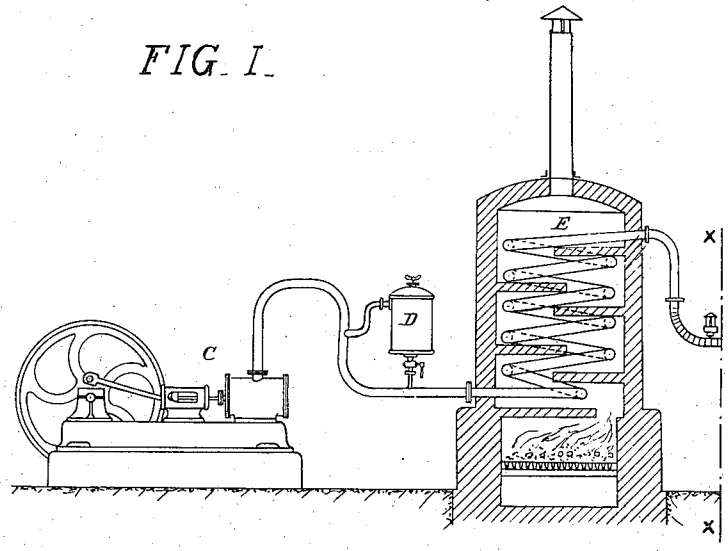
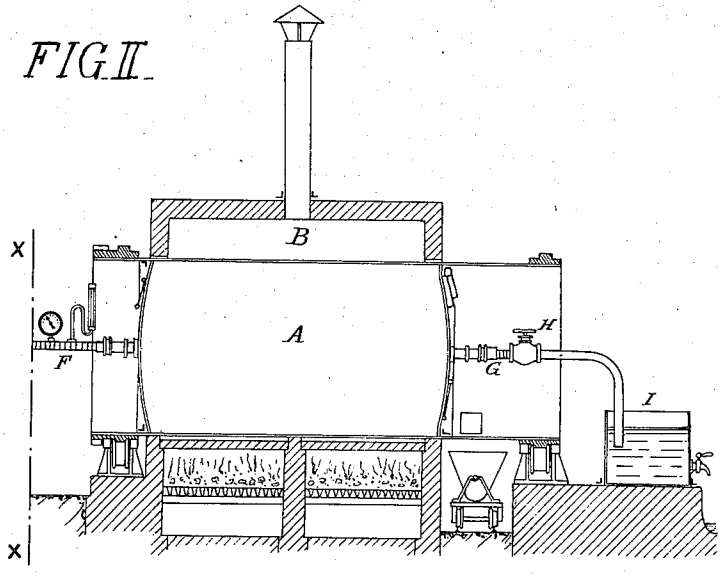


FIG. II.



Inventor:
Jacques Antoine Thibault,
by Bynum, Townsend & Brickenstein,
Attys.

Patented Jan. 29, 1924. 1,482,327

UNITED STATES PATENT OFFICE.

JACQUES ANTOINE THIBAULT, OF PARIS, FRANCE.

PRODUCTION OF METAL OXIDES, ESPECIALLY LEAD OXIDES.

Application filed January 5, 1920. Serial No. 349,548.

To all whom it may concern:

Be it known that I, JACQUES ANTOINE THIBAULT, a citizen of the Republic of France, residing in Paris, France, have invented a new and useful Improvement in the Production of Metal Oxides, Especially Lead Oxides, of which the following is a specification.

This invention relates to a new technical method for converting metallic lead into its oxides such as lead protoxide (litharge) and red-lead.

In the methods as hitherto used, lead protoxide (litharge) has been produced by calcining the molten metal in contact with air in a cupel or reverberatory furnace or by heating the molten lead in the presence of nitrate of soda. In this latter case the oxide produced is freed from nitrite, and crushed by the aid of millstones in the presence of water, in which operation the metallic lead, which has not be attacked, is separated from the oxide.

In order to obtain a better conversion of the lead into lead protoxide, it has been proposed to pulverize the molten lead by impact or the like and to simultaneously cause it to be oxidized by the aid of a current of air and water-vapor.

The current of air then causes a selection to take place, by the impact, between the oxidized particles and the lead which has not been converted; but even in the most improved methods, metallic lead remains in some proportion in the litharge which is obtained.

This metallic lead must be separated by lixiviation or by calcination in reverberatory furnaces, or otherwise it would interfere with the following operation which is necessary for converting the litharge into minium.

In all these known methods, in which the operation is carried out at a temperature higher than 100° C., the oxidation is effected on the molten lead.

The red lead or minium is produced by heating the lead protoxide at a high temperature in reverberatory or muffle-furnaces, or in mechanical or other furnaces.

The red lead further is generally produced, when operating upon metallic lead, in two phases and in a succession of apparatuses.

In the first phase, the lead is converted into lead protoxide (litharge) and in the second phase the litharge is oxidized into minium.

The method according to this invention allows the conversion of the lead into its oxygenized products without any melting operation and, contrary to the usual methods can be carried out in a single apparatus; further it secures the production of oxides which are free from any metallic lead without the necessity of crushing the product during the oxidizing operation or after this operation.

With these objects in view, the improved method essentially consists in oxidizing the lead in the solid state, in a closed vessel and in a hot and damped atmosphere, preferably although not necessarily under pressure and in the presence of an oxidizing agent.

In the practical application of the invention, the temperature is determined in such a manner that, in the production of the protoxide, the lead remains in the solid state and, in the production of minium, the litharge does not melt.

The velocity of the converting operation depends upon the following conditions: (1°) the pressure which is maintained in the apparatus, (2°) the oxidizing agent and (3°) the degree of moisture which is maintained in the apparatus as a consequence of the evaporization of water.

The converting operation can be effected at atmospheric pressure but as the velocity of the operation is accelerated by the pressure which is maintained in the apparatus, it is preferably effected under pressure. This pressure is conveniently produced by aid of compressed air and through the vaporization of water which is introduced into the apparatus.

The less expensive oxidizing agent, air, may be employed, but oxygen, ozone or oxygenated water can also be used as also suitable vehicles of oxygen such as, for example, lead-peroxide which easily gives up its oxygen to the metal.

The converting operation proceeds to some extent under the action of the current of air, as in the usual methods, but the fact that the reaction takes place in the presence of water-vapor greatly improves the action of the oxidizing agent and consequently allows the shortening of the operation.

The improved method secures an impalpable product, which need not be pulverized and which, when issuing from the apparatus, shows the bright coloration of the finest commercial products.

In order to have the invention fully understood, it will be described hereinafter by way of examples with reference to the annexed drawings which show diagrammatically an apparatus especially adapted for its practical application, and wherein:

Fig. 1 is an elevation, partly in section, of the air and water supplying and heating device, and

Fig. 2 is a diagrammatic sectional view of the oxidizing chamber proper and its adjacent parts.

This apparatus essentially comprises an oxidizing vessel such as a rotatably mounted and tightly closed drum A arranged in a heating chamber B adapted to be externally heated either by hot air or gas coming from a furnace or in any other suitable manner. This drum is connected to a compressor C, Fig. 1, by a pipe which is provided with a water distributing device D. A portion of the pipe is formed into a coil E arranged in a heating device so as to vaporize the water carried from the distributor D by the current of air. The compressed air, mixed with the water-vapor, enters the drum A through the hollow shaft F and escapes therefrom at the other end through the shaft G.

The pressure in the drum A can be controlled by a cock or valve H; further a suitable device I, to which the discharged air, steam etc. from the shaft G are led allows the collection of the particles which are carried away by the current of air.

Example I.—A quantity of say: 3.000 kgs. of lead in form of pieces, balls, sticks or powder, but preferably granulated, is introduced into the drum A or other suitable closed vessel adapted to continuously renew the contacts between the lead and the oxidizing agents which are used, and which will withstand a given pressure. (Granulated lead is preferably used because this form, owing to the great number of contact points, secures a rapid conversion into lead protoxide.)

The drum A having been set in motion (rotation), a current of compressed air produced by the compressor C is charged with a small quantity of water from the distributor D. This water, whilst circulating with the air in the coil E, is vapourized and enters the drum A in which the pressure is maintained at about 4 kgs. by adjustment of the valve H.

After 6 hours of operation, the lead is entirely converted into a protoxide of a yellow ochre colour, of extra fine nature and very unctuous. 3.230 kgs. of this protoxide are thus collected, including a small quantity of dust which is carried into the collecting device I.

Example II.—A quantity of say 3.000 kgs. of lead, preferably of granulated form, is charged into the drum A and treated as already described.

As soon as this lead has been converted into lead protoxide, the temperature is raised to about 425° C., which is below the melting point of the oxide, whilst maintaining the current of damped air and a pressure of about 4 kilogs. After 4 hours of operation, the whole charge of protoxide of lead is converted into red lead having a bright red color and yielding 33 to 34% of lead peroxide.

The product is impalpable and unctuous and need not be pulverized. 3.308 kgs. of minium (including the dust collected in the device I) are thus obtained.

The technical and industrial advantages of this improved method are very important.

The handling of the lead oxides, which is always dangerous for the workmen, is reduced to a minimum. Further the oxidizing operation is effected in a closed vessel, at temperatures which can be easily controlled, and always below the melting points of the products which are to be oxidized. Finally this oxidizing operation takes place in a damped atmosphere which secures amorphous products, which are impalpable and unctuous, having a good covering power, of bright colors and which do not require a pulverizing operation. The operation further is effected in a relatively short time which secures a great economy of time, power and labour.

Although the invention has been described as specially applied to the treatment of lead, it could also be applied usefully to other metals such as zinc, for example, in the manufacture of oxides and colors.

Having thus described my invention, what I claim is:

1. The improved method for the production of metal oxides which consists in charging the solid metal in a divided state in a closed vessel, heating the said metal at a temperature below its melting point, introducing a current of air under pressure into the vessel together with water-vapor, agitating the metal so as to improve its contact with the air and water-vapour, simultaneously maintaining a given pressure of air and water vapour in the vessel and finally raising the temperature above the melting point of the metal but below the melting point of an intermediary oxide.

2. The improved method for the production of lead oxides which consists in heating solid lead in a divided state in a closed vessel in the presence of an oxidizing agent and water-vapor, at a temperature below the

melting point of the lead, whereby an intermediary oxide is produced, and then raising the temperature above the melting point of the metal but below the melting point of said intermediary oxide while maintaining the action of the oxidizing agent and water vapor, whereby red lead is finally produced.

3. The improved method for the production of lead oxides which consists in charging solid lead in a divided state in a closed vessel, heating at a temperature below the melting point of the lead, introducing a current of air and water vapor under pressure into the vessel, controlling the escape of the compressed air and water-vapor from the vessel whereby the lead is subjected therein to a suitable pressure and is converted into lead protoxide, then raising the temperature above the melting point of the metal but below the melting point of the lead protoxide while maintaining the action of the current of air and water-vapor under pressure, whereby red lead is finally produced.

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

JACQUES ANTOINE THIBAULT.

Witnesses:
PIERRE GAURICHON,
EMILE LOUIS POTAY.