UNITED STATES PATENT OFFICE.

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PROCESS OF PRODUCING PHOSPHORIC ACID.

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To all whom it may concern:

Be it known that we, ERNEST W. GUERNSEY and JEW YAM YEE, citizens of the United States, and residents of Washington, District of Columbia, have invented an Improvement in Processes of Producing Phosphoric Acid, of which the following is a specification.

The subject of this invention is a process of producing phosphoric acid.

The main object of the invention is the provision of a process for liberating phosphoric acid from natural and artificial phosphates of the metals by means of silica and carbon at temperatures below fusion and in a more simple and economical manner than has heretofore been possible.

In order that our invention may be more clearly understood, and that it may be more easily differentiated from the prior art the following facts are submitted.

It is well known that phosphorus can be practically completely expelled by intense heating of a properly proportioned mixture of phosphatic material, sand and carbonaceous material. The reaction which takes place may be summarized by the equation:

$$Ca_3(PO_4)_2+3SiO_2+5C=3CaSiO_3+2P+5CO.$$

It has generally been believed that a practical liberation of P_2O_5 by this reaction could be obtained only at the temperature at which a fluid melt is obtained. This temperature can be easily attained only in electric furnaces. As is well known, the use of electric current for smelting purposes is very expensive. In fact, we have made a calculation on the cost of phosphoric acid produced in this way and have found that with electric power at $.005 per K. W. H. the cost of electricity for heating is more than 50% of the total cost of the phosphoric acid produced. A further disadvantage and expense connected with the use of an electric furnace is the lining replacement charges due to high temperatures employed. Moreover, a relatively large amount of high grade labor is required to operate an electric furnace. Finally, an expensive and complicated auxiliary electrical installation is required.

We have made a large number of experiments and have confirmed the general belief that a practical liberation of P_2O_5 can not be obtained from a viscous melt containing $Ca_3(PO_4)_2$, SiO_2 and C. If it is desired to practically expel P_2O_5 from a molten bath a quite fluid melt must be obtained.

We have discovered however, that, contrary to the current belief, phosphorus may be practically completely expelled from an intimate mixture in proper proportions of phosphate rock, sand and carbon at temperatures considerably below those which have heretofore been employed, provided the temperature is maintained just under the point of fusion of the charge. This temperature depends on the exact composition of the charge but is in the neighborhood of 1300° C., a temperature which can be attained in relatively inexpensive and simply and cheaply operated fuel fired furnaces.

Our invention may be carried out in any apparatus in which solid material may be conveniently handled and efficiently heated to 1300° C. under conditions sufficiently non-oxidizing to prevent the burning of the carbon in the charge before it can perform its function of reducing calcium phosphate. An internally fired rotary kiln heated with a non-oxidizing flame meets these requirements. We do not, however, wish to be limited to the employment of a rotary kiln in carrying out our processes. When we use an internally fired rotary kiln, we prefer to briquette the charge in order to prevent the dusting of the powder charge and in order to furnish an additional safeguard against the burning out of the carbon in the charge.

As an example of our invention we first finely grind phosphate rock, sand and coal. We prefer to grind these materials as fine as practicable since the rate at which the desired reaction takes place depends largely on this factor. The materials are often mixed before grinding or are ground separately and are then intimately mixed in such proportions that combined CaO, and silica, SiO_2, are in the molecular ratio of 1:1 and carbon is present in at least 50% excess of that required by the equation:

$$Ca_3(PO_4)_2+3SiO_2+5C=3CaSiO_3+2P+5CO.$$

The actual proportions of phosphate rock, sand and coal will vary according to the composition of these materials as will be understood by those skilled in the art. This

mixed charge is preferably formed into briquettes according to known methods, and is then fed into an internally fired kiln in which the fuel and air are so proportioned that a reducing or at least not-strongly oxidizing atmosphere is maintained. The heat is so regulated that the charge is brought only to incipient clinkering or slightly under. Under these conditions practically all of the phosphorus is evolved in the elementary state. The duration of the heating necessary to secure complete expulsion of the P_2O_5 depends on the fineness and intimacy of mixing of the materials on the physical structure of the phosphate rock, on the exact composition of the charge, etc. For an ordinary Florida or Tennessee phosphate rock the time of heating need not exceed one hour at the most if the materials are ground to a fineness under 100 mesh. The gases from the kiln, containing the expelled phosphorus may be burned under boilers or other heat conserving devices. The oxides of phosphorus contained in the gases from the burners may be collected in any suitable manner as for instance by a Cottrell precipitator or they may be absorbed in any well known and usual manner.

It is obvious that those skilled in the art may vary the details without departing from the spirit of the invention and therefore, we do not wish to be limited by the above disclosure except as required by the claims.

Having described our invention what we claim and desire to secure by Letters Patent, is:

1. The process of making phosphoric acid, comprising forming an intimate mixture of phosphate rock, siliceous material and carbonaceous material in such proportions that the molecular ratio of combined CaO to silica will be 1:1 heating the mixture at a temperature not to exceed incipient clinkering until practically all the phosphorus has been expelled forming oxides of the expelled phosphorus and recovering such oxides.

2. The process of making phosphoric acid, comprising forming an intimate mixture of phosphate rock, siliceous material and carbonaceous material in such proportions that the molecular ratio of combined CaO to silica will be 1:1 and the carbon present will be at least 50% in excess of that required by the equation

$$Ca_3(PO_4)_2+3SiO_2+5C=3CaSiO_3+2P+5CO,$$

heating the mixture at a temperature not to exceed that of incipient clinkering until practically all the phosphorus has been expelled, forming oxides of the expelled phosphorus and recovering such oxides.

3. The process of making phosphoric acid, comprising forming an intimate mixture of phosphate rock, silicious material and carbonaceous material in such proportions that the carbon present will be at least 50% in excess of that required by the equation

$$Ca_3(PO_4)_2+3SiO_2+5C=3CaSiO_3+2P+5CO,$$

heating the mixture at a temperature not to exceed that of incipient clinkering until practically all the phosphorus has been expelled, forming oxides of the expelled phosphorus and recovering such oxides.

4. The process of making phosphoric acid, comprising forming an intimate mixture of phosphate rock, silicious material and carbonaceous material in such proportions that the molecular ratio of the combined CaO to the silica will be 1:1, forming the material so mixed into briquettes, heating the briquettes at a temperature not to exceed incipient clinkering until all the phosphorus has been expelled, forming oxides of the expelled phosphorus and recovering such oxides.

5. The process of making phosphoric acid, comprising forming an intimate mixture of phosphate rock, silicious material and carbonaceous material in such proportions that the molecular ratio of silica to combined CaO will be at least 50% in excess of that required by the equation

$$Ca_3(PO_4)_2+3SiO_2+5C=3CaSiO_3+2P+5CO,$$

heating the mixture at a temperature not to exceed that of incipient clinkering until practically all the phosphorus has been expelled, forming oxides of the expelled phosphorus and recovering such oxides.

6. The process of making phosphoric acid, comprising forming an intimate mixture of phosphate rock, silicious material and carbonaceous material in such proportions that the carbon present will be at least 50% in excess of that required by the equation

$$Ca_3(PO_4)_2+3SiO_2+5C=3CaSiO_3+2P+5CO,$$

forming the materials so mixed into briquettes, heating the briquettes at a temperature not to exceed that of incipient clinkering until practically all the phosphorus has been expelled, forming oxides of the expelled phosphorus and recovering such oxides.

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