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PREPARATION OF ALKALI PHOSPHATES

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It is known that ferro-phosphorus can be converted by calcining roasting or fusing in the presence of alkali sulphates or alkali carbonates and in this way alkali phosphates be produced. A process is also known, according to which iron phosphate is first obtained from ferro-phosphorus by roasting and the iron phosphate then converted with alkali carbonate under pressure. A further process is known according to which phosphides are heated in the presence of aqueous alkalies to temperatures above 100° C. particularly to temperatures of from 300 to 350° C. whereby alkali phosphates and free hydrogen are formed. All these known processes, however, are only of slight practical importance since they require high temperatures, costly furnaces or pressure vessels and sometimes involve several stages of operation and can consequently only be carried out in a troublesome and difficult manner. As a rule also, the yields are deficient, particularly if the wet decomposition process at temperatures of below 300° C. is employed.

According to this invention it has been found that, by proceeding in a certain manner, alkali phosphates can be obtained in a simple manner and in one operation from phosphorus-containing materials, particularly phosphorus alloys, such as ferro-phosphorus or the like and in this way the aforementioned disadvantages and difficulties of the known methods be overcome. This is effected according to the invention by carrying out the conversion, in the preparation of alkali phosphates from phosphorus-containing materials in the presence of aqueous alkali hydroxides or carbonates at elevated temperatures, with the aid of oxidizing agents such as oxygen or oxygen-containing gases.

In this way the advantages are obtained over the afore-mentioned processes, particularly over the wet decomposition process, which is carried into effect without the addition of oxygen, of a considerable improvement in the yields and above all that the ferro-phosphorus can be decomposed in relatively simple apparatus at low temperatures and pressures. The invention accordingly affords an economy in plant and running costs for the apparatus as well as a considerable economy in fuel. Furthermore when treating ferro-phosphorus according to the invention the iron is obtained in the form of iron oxide, which can, for example, be used as a pigment.

The conversion is with advantage carried out under pressure at temperatures above 100° C. preferably at 150° to 300° C. Instead of using alkali hydroxides or carbonates for the conversion, the operation can also be carried out with the aid of other substances, which yield alkali under the conditions of the reaction, such as alkali silicates, alkali aluminates, organic alkali salts or the like. The resulting liquors can be worked up in known manner to alkali phosphates after the completion of the process and separation of the solid constituents.

It has been further found according to this invention that it is particularly advantageous to carry out the conversion by subjecting the preferably finely comminuted matter to thorough mechanical mixing, for example in shaking or stirring autoclaves. In this way not only can the entire process be advantageously accelerated and carried to completion but the plant and working costs can also be further considerably reduced.

As oxidizing agents there may be employed all substances which react in the aforesaid manner during the conversion. The most advantageous oxidizing agent for the purpose is oxygen as such or in the form of air or other oxygen-containing gases, the use of which affords not only the advantage of very great economy but also that of extremely simple application. The introduction of oxygen is most suitably effected by passing a continuous current of oxygen or oxygen-containing gases during the conversion process, preferably under a pressure, which is higher than the prevailing vapour tension, through the conversion container.

The carbon dioxide formed or set free during the conversion process is with advantage continuously or discontinuously removed from the conversion container. This can, for example, also be effected with the aid of a current of gas. The removal of the carbon dioxide can thus be in a very advantageous manner so combined with the afore-mention passage of oxygen or oxygen-containing gases that the carbon dioxide set free is simultaneously carried away with the residual discharging gases. The removed carbon dioxide may then be further worked up in known manner to liquid or solid carbon dioxide and be in this way obtained in a completely pure form.

The temperatures and pressures employed for the conversion may vary within relatively wide limits. The reaction, however, in general proceeds the more rapidly the higher the temperature and the partial pressure of the oxygen.

According to a particular modification of the invention the conversion process is carried out in the presence of alkali-yielding phosphates, for example, trisodium phosphate, as alkali-yield-

ing substance, it being however, of course, also possible, if desired, to use in conjunction therewith other alkali-yielding substances of the hereinbefore mentioned kind. When employing such phosphates, phosphates poorer in alkali, such as disodium phosphate, are obtained after the conversion, which can then be re-generated and be thereafter again used for the conversion process. This regeneration can, for example, be effected by treating the phosphates in question with aqueous alkali carbonate or the like at elevated temperature, preferably above 100° C. and elevated pressure. In this case also the material is with advantage thoroughly mechanically mixed, for example, by stirring, and the carbon dioxide set free thereby removed.

It is advisable in all cases to employ the starting materials as well as the oxidizing agent in the highest possible concentration.

Examples

1. 2 parts by weight of very finely ground ferro-phosphorus (containing 19% of phosphorus) are mixed with 1.95 parts by weight of sodium carbonate and, after adding 20 parts of water, heated in an autoclave with thorough stirring to 200° to 350° C. until the conversion is completed, the progress of which can be followed by the evolution of carbon dioxide. A current of oxygen or air at a slightly higher pressure than that corresponding to the vapour tension at the said temperature is passed through the apparatus during the conversion, the carbon dioxide being simultaneously removed, the resulting liquor is filtered off from the residue and is then worked up in known manner to sodium phosphate.

2. 2 parts by weight of very finely ground ferro-phosphorus (containing 19% of phosphorus) are mixed with 1.47 parts by weight of sodium hydroxide and, after adding 20 parts of water, heated in an autoclave with thorough stirring to 200° to 350° C. until the conversion is completed. During the conversion oxygen or air at a slightly higher pressure than that corresponding to the vapour tension at the said temperature is passed into the apparatus. The resulting liquor is filtered off from the residue and is then worked up in known manner to sodium phosphate.

3. 60 parts by weight of ferro-phosphorus, 68 parts by weight of caustic potash and 200 parts of water are heated with the addition of oxygen in a shaking autoclave, containing a few steel balls, to a temperature of 150° C. for six hours. The resulting yield of potassium phosphate amounts to 88 to 90%.

What I claim is:—

1. A process for the preparation of phosphates of the alkali-metals consisting in reacting an alloy of phosphorus with an alkali-metal compound which is alkaline in reaction in the presence of water at an increased temperature and in the presence of an oxidizing agent.

2. A process as defined in claim 1 in which the oxidizing agent is present in excess.

3. A process for the preparation of alkali-metal phosphates consisting in reacting an alloy of phosphorus with an alkaline, alkali-metal compound at an increased temperature in the presence of an oxidizing gas.

4. A process for the preparation of phosphates of the alkali-metals consisting in reacting an alloy of phosphorus with an alkali-metal hydroxide in the presence of water at an increased temperature and in the presence of an oxidizing agent.

5. A process for the preparation of phosphates of the alkali-metals consisting in reacting an alloy of phosphorus with a carbonate of an alkali metal in the presence of water at an increased temperature and in the presence of an oxidizing agent.

6. A process for the preparation of phosphates of the alkali metals consisting in reacting an alloy of phosphorus with a phosphate of the alkali metal in the presence of water at an increased temperature and in the presence of an oxidizing agent.

7. A process for the preparation of phosphates of the alkali metals consisting in treating an alloy of phosphorus with tri-sodium phosphate in the presence of water at an increased temperature and in the presence of an oxidizing agent.

8. A process as defined in claim 6 in which the phosphate, which is reacted with the alloy of phosphorus, is regenerated subsequent to its decomposition and again used in the process.

9. A process as defined in claim 6 in which the alkali metal phosphate which is reacted with the alloy of phosphorus is regenerated with an alkaline, alkali-metal compound at an increased temperature and under increased pressure, and then utilized again in the process.

10. A process as defined in claim 1 in which the reaction of the alkaline alkali metal compound with the said alloy of phosphorus resulting in the decomposition of the latter is carried out at a temperature in excess of 100° C. and at an increased pressure.

11. A process as defined in claim 1 in which the reaction of the alkaline alkali metal compound and the said alloy of phosphorus is accompanied by mechanical agitation of the reaction materials.

12. A process for the preparation of alkali metal phosphates consisting in reacting an alloy of phosphorus with an alkaline alkali-metal compound in the presence of water at an increased temperature, a current of an oxidizing gas being continuously introduced into the reaction mixture so that it travels through a substantial portion of said reaction mixture.

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