

UNITED STATES PATENT OFFICE

2,356,799

PROCESS FOR PRODUCING INSOLUBLE
SODIUM METAPHOSPHATEGeorge E. Taylor and Allen G. Erdman, Anniston,
Ala., assignors to Monsanto Chemical Com-
pany, a corporation of DelawareNo Drawing. Application May 27, 1943,
Serial No. 488,776

8 Claims. (Cl. 23-106)

This invention relates to a process for producing insoluble sodium metaphosphate.

An object of this invention is to provide a process whereby insoluble sodium metaphosphate may be produced without the simultaneous production of excessive amounts of soluble forms of sodium metaphosphate.

The use of the insoluble sodium metaphosphate as a dentifrice was first disclosed by Kuever in U. S. 2,019,142.

In the commercial manufacture of insoluble sodium metaphosphate which manufacture is carried out by heating mono-sodium orthophosphate to temperatures in the range of from 400° C. to 460° C., it has been found that relatively large amounts of the soluble metaphosphates are simultaneously formed. Since insoluble sodium metaphosphate produces an ideal polishing ingredient either for tooth powders or tooth pastes and moreover since the soluble variety of sodium metaphosphate has been found to have a deleterious effect upon tooth enamel, it is highly desirable that the insoluble sodium metaphosphate be produced without, at the same time producing the soluble variety.

While it is possible to overcome the effect of minor proportions of soluble sodium metaphosphate by adding various alkaline or alkaline earth compounds such as magnesium or calcium salts to the insoluble sodium metaphosphate when formulating a dentifrice composition, as suggested by Long in U. S. Patent No. 2,216,821, nevertheless the soluble variety of sodium metaphosphate in the insoluble sodium metaphosphate represents a deleterious constituent which must either be counteracted or must be removed from the composition as by leaching, etc.

Previous methods of producing insoluble sodium metaphosphate have consisted in heating either the crystalline or the finely ground mono-sodium orthophosphate to metaphosphate forming temperatures, the heating being carried out in trays or pans. Such heating was necessarily carried out as a batch operation, the heating period being of the order of 14 hours.

I have now found that if finely ground crystalline mono-sodium orthophosphate in the anhydrous form be first formed into pellets by compression, that the pellets thus formed may be heated to the metaphosphate forming temperature in a rotary kiln or shaft furnace or other heating device and a good yield of insoluble sodium metaphosphate obtained which is surprisingly free of soluble metaphosphates.

A satisfactory pellet of mono-sodium ortho-

phosphate is made by merely briquetting the orthophosphate powder as such or the powder may first be somewhat moistened utilizing up to 5% by weight of water as the binding agent, after which the slightly moist orthophosphate is then briquetted. I have found that from 1% to 2% of water is usually sufficient for making a strong briquette from the powdered orthophosphate. If desirable, the water may also be added by adding the equivalent amount of the mono- or dihydrate of mono-sodium orthophosphate.

The strength of the briquette should be such that it will maintain its shape during the subsequent treatment wherein the briquette is heated to convert the orthophosphate to the insoluble metaphosphate. The size of the compressed pellet produced is not particularly critical except that the pellet should be small enough so that it may be thoroughly heated to the metaphosphate conversion temperature by circulating hot gases in a relatively short time. We have found that a circular pellet up to 1 inch diameter and from 1/4" to 3/8" in thickness may conveniently be employed. On the other hand, if desired, the pellet size may be decreased to that size now used for aspirin tablets. In general, pellets weighing between 0.5 gram and 25 grams are preferred and may be used satisfactorily. The anhydrous mono-sodium orthophosphate employed for producing the pellets may vary in particle size over a considerable range but we have found that 40 mesh is close to the upper limit of particle size to give a satisfactory product. Material which passes a 100 mesh screen is definitely preferable and particle sizes as fine as 350 or 400 mesh may be used quite satisfactorily.

Pellets produced as described above may now be heated in a rotary kiln or converter in the well known manner. A suitable device consists of a rotary kiln provided with a gas burner located at the discharge end thereof and supplied at the entrance to the kiln with hot gas at a temperature of about 500° C. The pellets of orthophosphate are introduced at the inlet or upper end of the inclined rotary kiln and due to the inclination and rotation of the kiln are caused to move downwardly through the kiln countercurrently to the heated gases. The pellets upon encountering gas of this temperature are heated gradually to a temperature which should not be higher than about 450° C. to 460° C. Conversion to the insoluble metaphosphate takes place between the limits of from 300° C. to 460° C. The inclination of the axis of the kiln should be such that the entire heating of the stream

of pellets takes place in from 1 to 1½ hours or possibly 2 hours. At all events the heating should be completed in less than 3½ or 4 hours.

Alternatively, the pellets may be heated by passing them countercurrent to a flow of hot gases, through a vertical shaft kiln. The pellets are passed through such a kiln by removing the heated and converted product from the bottom of the kiln, while adding fresh pellets at the top of the kiln. The time of heating in such a device is thus governed by the rate of discharge from the lower portion of the kiln and may be adjusted to conform with the time of heating obtained in the rotary kiln, as given above.

The hot pellets discharged from the kiln are allowed to cool to room temperature and then are crushed and finely ground to produce a powder fine enough to pass a 350 mesh screen.

Using the herein described process we have found that the water soluble salt content of the product is maintained under 4% and generally under 2%. In many cases the soluble salt content will be found to be below a fraction of 1%.

What we claim is:

1. The method for producing insoluble sodium metaphosphate comprising compressing finely monosodium orthophosphate into pellets, heating said pellets to a temperature favorable to the formation of insoluble sodium metaphosphate and grinding said pellets to a fine powder.

2. The method for producing insoluble sodium metaphosphate comprising compressing finely divided monosodium orthophosphate into pellets, heating said pellets to a temperature between 300° C. and 460° C. to form insoluble sodium

metaphosphate and grinding said pellets to a fine powder.

3. The method for producing the insoluble metaphosphate of sodium which comprises forming pellets by compressing a mixture of monosodium orthophosphate and water in an amount less than 5% and then heating said pellets in a stream of air to a temperature between 300° C. and 460° C. to form insoluble sodium metaphosphate, and then grinding said pellets to a fine powder.

4. The process defined in claim 3 in which the time of heating is between one hour and four hours.

5. The process defined in claim 3 in which the pellets flow counter-current to the stream of air.

6. The process defined in claim 3 in which the particle size of the mono-sodium phosphate before pelleting is fined than 40 mesh.

7. The process defined in claim 3 in which the particle size of the monosodium phosphate before pelleting is finer than 100 mesh.

8. The method for producing the insoluble metaphosphate of sodium, which comprises forming pellets from finely divided monosodium orthophosphate, said orthophosphate having a screen size of less than 40 mesh and said pellets weighing between 0.5 gram and 25 grams each, heating said pellets in a stream of air to a temperature between 300° C. and 460° C. for a period of from 1 to 4 hours and then crushing said pellets to a fine powder.

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CERTIFICATE OF CORRECTION.

Patent No. 2,356,799.

GEORGE E. TAYLOR, ET AL.

August 29, 1944.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 2, first column, line 27, claim 1, before the word "monosodium" insert --divided--; and second column, line 19, claim 6, for "fined" read --finer--; and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 24th day of October, A. D. 1944.

Leslie Frazer

(Seal)

Acting Commissioner of Patents.