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METHOD OF PRODUCING MONOCALCIUM PHOSPHATE CONTAINING A HIGH P₂O₅ CONTENT

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This invention relates to a granular, free flowing, substantially, non-caking monocalcium phosphate having a high P₂O₅ content and to a process for producing same.

Monocalcium phosphate containing an excess of P₂O₅ corresponding to 1% by weight of free H₃PO₄ is very hygroscopic and tends to cake and lose its dry free flowing character in a relatively short time. If the P₂O₅ content of this product is increased to a value corresponding to 5% free acid, its hygroscopicity is markedly increased with the result that, upon storage, it rapidly sets to a solid crystalline mass.

To offset these objectionable properties it has been the practice to either neutralize the excess acidity or to so proportion the raw materials used in the preparation of monocalcium phosphate that the product contains less than 1% by weight of free acid. These methods of stabilization serve the intended purpose very well except that the potential acidity and also the solubility of the monocalcium phosphate are too low for certain applications. For example, in the beverage industry there has recently been a demand for a granular, free flowing, monocalcium phosphate of high acidity and solubility which may be used in partial or complete substitution of fruit acids such as citric, tartaric, and malic acid and the like. In view of its low acidity and solubility in aqueous media, commercially available monocalcium phosphate is not suitable for this purpose and, so far as I am aware, these properties cannot be improved by the prior art methods without, at the same time, excessively increasing the hygroscopicity and caking tendency of the product.

It is therefore the object of the present invention to provide a process for making a granular, free flowing, substantially non-caking monocalcium phosphate which is readily soluble in aqueous media and which contains P₂O₅ in an amount corresponding to a free acid (H₃PO₄) content of from 5% to 18.5% and preferably from about 10% to about 18.2% by weight.

In carrying out the present invention, phosphoric acid is thoroughly mixed with granular monocalcium phosphate in the proportions calculated to yield a product containing up to about 18% free H₃PO₄, which operation takes place within a period of from about 1.0 to about 5 minutes. At the end of the mixing step, the product is passed through a vibrating screen to break up any lumps which are formed, whereupon it is dried, milled and then screened to the desired size. In this manner, a readily soluble dry granular, free flowing and substantially non-caking monocalcium phosphate of high acidity is produced which is adapted for use as a fruit acid substitute in the production of powdered beverages.

For a more complete understanding of the pres-

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ent invention, reference is made to the following specific examples.

EXAMPLE I

36 lbs. of 80% H₃PO₄ was gradually added and mixed in a Baker-Perkins mixer with 100 lbs. of spray dried monocalcium phosphate having the following screen analysis

	Per cent
+80	0.8
-80+200	20.4
-200	79.6

After about 3 to 5 minutes, the mixing operation was completed and then the product was put through a ¼ inch vibrating screen to break up any lumps which were formed. The screened product was dried in a stainless steel rotary drier heated with hot air at 90° C. to 100° C., whereupon it was milled in a hammer mill and finally passed through a 10 mesh screen. The resulting product was granular, free flowing and substantially non-caking in character and on analysis was found to have the following P₂O₅, free H₃PO₄ and moisture content:

	Per cent
P ₂ O ₅	60.69
Free H ₃ PO ₄	15.74
Loss at 105° C.	2.92

EXAMPLE II

36 lbs. of 80.7% of H₃PO₄ was thoroughly mixed with 100 lbs. of spray dried monocalcium phosphate oversize using a mixing time of about 50 seconds. The resulting mixture was passed through a ¼ inch vibrating screen to break up a relatively small proportion of lumps and then dried in a rotary air drier at a temperature of 95° C. The dried mixture was milled in a hammer mill and finally put through a 10 mesh screen. The product thus obtained was granular, free flowing and substantially non-caking in character and was further characterized by the following properties.

Chemical analysis

$$\frac{P_2O_5}{CaO} = \frac{59.18}{18.57} = 3.20$$

Free H₃PO₄ = 15.91%

Ignition loss = 22.01%

Screen analysis

R10	Trace
CR20	per cent 16.2
CR50	do 92.0
CR80	do 97.2
S80	do 2.8

EXAMPLE III

The procedure set forth in Example II was repeated using 43.5 lbs. instead of 36 lbs. of 80.7% H₃PO₄.