United States Patent Office 3,016,299 Patented Jan. 9, 1962

3,016,299

FUMARIC ACID COMPOSITION

Stanley P. Raffensperger, Palos Park, and Thomas T. Takashima, La Grange, Ill., assignors to General Foods Corporation, White Plains, N.Y., a corporation of Delaware

No Drawing. Filed Feb. 9, 1959, Ser. No. 791,862
12 Claims. (Cl. 99—78)

The following invention relates to an improved method of increasing the solubility rate of fumaric acid in water and, in particular, the solubility rate of fumaric acid in cold water.

In the past, citric acid has been employed as an acidulant in dehydrated beverage powders capable of being rehydrated in cold water. In addition to citric acid, such powders usually contain other hydroscopic materials such as sugars and the like. Such compositions are relatively unstable if stored for extended periods of time since they readily absorb moisture and cake upon standing. The storage problem encountered with such dehydrated beverage powders has always been of great concern to those skilled-in-the-art and in particular the storage problems encountered in warm, humid climates. Fumaric acid has many properties which make it desirable for commercial use in such products. However, such uses are limited due to the fact that fumaric acid has a very low rate of solubility in cold water. While the dry beverage powders of commerce are rehydratable in cold water in less than one minute, the use of fumaric acid in such powders in the past has been impossible due to the fact that the fumaric acid does not dissolve rapidly in cold water, periods as long as 24 hours at times not being sufficient to put all of the fumaric acid into solution.

It is an object of this invention to prepare a fumaric acid composition which has an increased rate of solubility in cold water. It is a further object of this invention to prepare a fumaric acid composition which when used in cold water soluble beverage powders will not absorb substantial amounts of moisture upon standing and will be readily and easily soluble in cold water. Further objects of this invention will be apparent from a reading of the specification.

It has now been discovered that the rate of solubility of fumaric acid may be increased by wetting crystalline sugar with a quantity of water insufficient to destroy the crystalline character of all of the sugar but sufficient to create a proportion of sugar solution capable of aggregating fumaric acid powder and undissolved sugar crystals; such a wetted sugar will generally have a moisture content in excess of 5% and for the more typical sugar, sucrose, a moisture content of at least 7%. The fumaric acid, on the other hand, is ground to a particle size whereat it will, upon addition to an aqueous liquid, neither float at the top of said liquid nor drop to the bottom of a body of said liquid, but rather will be suspended throughout, such that the fumaric acid powder can be combined by admixture with the wetted sugar and aggregate with the sugar crystals thereof. This aggregate is eventually dried under conditions of time and temperature insufficient to melt the sugar crystals but sufficient to reduce the moisture content of said aggregates to a stable level, say less than 1%, wherebelow the product will not lump later in storage. Finally, the dried product is broken up to a particle size suitable for handling.

In achieving such particle size reduction of the fumaric acid powder and combining this powder with the wetted sugar, the sugar and fumaric acid powder should be in sufficient proportion to one another to permit the sugar solution to coat the fumaric acid powder and the sugar crystals whereby the aforesaid aggregation occurs. Granulation of the fumaric acid powder may be practiced before combination thereof with the wetted sugar or the wetted sugar can be ground with the fumaric acid powder during which operation the aggregate is subdivided to the particle size desired. The most suitable particle size of the fumaric acid and sugar will not be the same for all methods of preparing the fumaric acid composition of the present invention. While the invention is not to be limited to any specific proportion of sugar to fumaric acid powder, it has generally been found that a 1:1 by weight ratio produces an acceptable proportion of ingredients although still higher proportions of sugar may be practiced, ranging upwards to the neighborhood of 4 to 5 parts by weight of sugar per part by weight of fumaric acid powder. The sugar crystals should, as indicated above, be wetted to the extent that a small portion of the sugar crystals are in solution. In any event, the sugar should be in a partially hydrous state, the degree of hydration depending upon the sugar involved. In the case of sucrose, a moisture level of at least 7% by weight of the sugar and fumaric acid combination should be employed. Some desired results are obtained at moisture levels of below 7%; but below moistures of 5% by weight the degree of wetting is insufficient to produce the desired aggregates.

While sugars such as the monosaccharides, disaccharides, polyaccharides or the sugar alcohols may be employed, it is preferred to employ disaccharides such as sucrose or monosaccharides such as dextrose. The sugars employed should be of such nature that when the fumaric acid composition of the present invention is dried, the sugar will not remain in a syrupy state and will be substantially dry and crystalline.

While it is preferred to use water as the moisture source, other solvents in which both the sugar and fumaric acid are soluble may be employed.

In producing the fumaric acid compositions according to certain embodiments of this invention, fumaric acid having a particle size of commercial 40 to 230 U.S. Standard mesh and preferably about commercial 100 U.S. Standard mesh is dry blended with a sugar having a particle size of commercial 18 to 100 U.S. Standard mesh and preferably commercial 30 U.S. Standard mesh. Where sucrose is employed, then 0.5 to 4.5 parts by weight of sucrose to one part by weight of fumaric acid may be employed and preferably one part by weight of sucrose to one part by weight of fumaric acid. The mixture is blended until a homogeneous mass is obtained. Blending for an excessive period of time causes the particles to segregate and therefore it has been found preferable to blend until uniformity is obtained. The dry mixture of fumaric acid and sugar is moistened by the addition of 5–10% moisture and preferably 7%. A moisture content of the moistened mixture of less than 5% produces a product which is not sufficiently wet to obtain the desired end product and a moisture content of more than about 10% where sucrose is employed permits the undesirable inversion of sucrose to take place when the wetted mixture is heated in the presence of fumaric acid, frequently resulting in an invert syrup which canot be dried. The moisture content of the mixture must be between those upper and lower limits at which the particles "pack" or adhere together as a friable mass of aggregates. The moistened mixture is blended to obtain a uniform distribution of the added water and to wet all the dry powder particles thereby permitting the wetted sugar to adhere to and coat the fumaric acid. The wetted mixture of fumaric acid and sugar is then dried to less than about 1% and preferably