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[54] **DENTURE SOAK TABLET**
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[56] **References Cited**

UNITED STATES PATENTS
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ABSTRACT: This disclosure relates to the process of preparing a water-soluble effervescent denture soak tablet free of capping or picking tendencies, which comprises the thorough blending of the water-soluble ingredients including a carbonate, organic acid and at least one oxidizing agent, the terminal addition and blending of a water-soluble detergent to the above formulation, subjecting the homogenous mixture to normal pressures in a tableting device, yielding tablets with clean surfaces and sharp edges. The terminal addition of the water-soluble detergent to the formulation lubricates the dies and permits easy and clean ejection of the tablet.

DENTURE SOAK TABLET

The present invention relates to the production of a water-soluble effervescent denture soak tablet having clean surfaces and sharp edges and free of capping and picking tendencies, and more particularly to a tablet which is completely water-soluble even though it contains a die lubricant.

The prior art is replete with disclosures of cleansing tablets and processes for their manufacture, useful for myriad purposes inclusive of denture soak formulations. Problems commonly encountered in the manufacture of tablets include (1) sticking of the tablet to the compression die, (2) tablets that chip, cap or break easily. These are well recognized in the art and have been solved in many different ways, inclusive of using specific granulation methods with or without limiting the granules to specific mesh sizes, hydrating the tablets, subsequent heating treatments, specific compounding steps whereby a portion of the ingredients are moistened prior to compression, coating the tablets with specific products such as amides or a urea complex, etc. However, aforesaid solutions have been useful for specific formulations and only partially effective in certain procedures.

Accordingly, it is an object of this invention to overcome tableting processing difficulties and provide a completely soluble effervescent tablet having clean surfaces and sharp edges.

It has now been found that picking and capping difficulties normally encountered in tableting procedures has been substantially completely eliminated by the particular procedure of adding at least one water-soluble detergent as a final step in the formulations prior to the tableting step. The detergent acts as a lubricant and permits clean and easy ejection of the tablet from the compression die. The ingredients may be in the form of powders or granules of varying sizes. The water-soluble ingredients consisting essentially of at least one oxidizing agent, a carbonate and an organic acid are thoroughly mixed and blended prior to the addition of the water-soluble detergent. All other optional ingredients inclusive of flavor oils, flavor carriers, binders, dyes, etc. are added to the basic formulation above prior to the addition of the water-soluble detergent. The final procedural step must constitute the addition and mixing of the water-soluble detergent. The thoroughly blended mixture is then fed to the die cavity of the tableting machine, where it is subjected to normal pressures (about 3-5 tons). The tablets having clean surfaces and sharp edges are ejected from the mold with ease, leaving clean surfaces in the die cavity for continuous tablet formation.

More specifically, this invention relates to a process of preparing a completely water-soluble, neutral pH, effervescent tablet having clean surfaces and sharp edges, and comprising about 5%-75% of a water-soluble carbonate, about 5%-50 % of a water-soluble organic acid and about 5-50% of at least one oxidizing agent, the improvement which comprises the terminal addition of 0.1-5% of at least one water-soluble detergent to the above formulation and forming a homogeneous mixture, and molding said mixture into a tablet whereby said detergent lubricates the mold and said tablet is easily released therefrom with clean surfaces and sharp edges.

As a source of one effervescent gas, namely carbon dioxide, there may be used a water-soluble alkali-metal carbonate compound selected from the group consisting of sodium bicarbonate, sodium carbonate, sodium sesquicarbonate, potassium carbonate, potassium bicarbonate, and mixtures thereof, which reacts with a solid organic acid selected from the class consisting of tartaric acid, citric acid, malic acid, maleic acid, fumaric acid, succinic acid, etc. and mixtures thereof, in the presence of water to produce effervescence (bubbles). The carbonate content and acid content may each vary from about 5%-75.35%-50% by weight of the total composition. However, maximum effervescence level is attained when 3 parts carbonate to 2 parts acid concentration is utilized.

An acid anhydride may be utilized in addition to or as a partial substitute for the organic acid. Such anhydrides are par-

ticularly effective in producing sustained effervescence when dissolved in water and also function as an internal desiccant in the prevention of premature evolution of CO₂. The acid anhydrides utilized herein are nontoxic, solid water-soluble inorganic or organic materials and capable of hydrolyzing and subsequently reacting with the inorganic carbonate to evolve CO₂. Specific examples of acid anhydrides include boric anhydrides, succinic anhydride, adipic anhydride, fumaric anhydride, tartaric anhydride, citric anhydride, malic anhydride, maleic anhydride and the like. The acid anhydride content may vary from about 1%-60% and preferably from 5%-10% by weight of the total composition.

The oxidizing agents offer in addition to active oxygen another source of effervescence (liberation of molecular oxygen) and are preferably the persalts such as the alkali-metal perborates, persulfates, percarbonates, perphosphates and the like. The simple addition of these water-soluble peroxygen compounds to water produces a highly effective source of active oxygen which is particularly useful in oxidation reactions, thereby providing considerably improvements in applications where peroxygen compounds are employed such as for antiseptic, cleansing and bleaching activity. The degree of effervescence which relates to both the volume and speed is dependent on the particular peroxygen compound employed. For example, sodium perborate monohydrate has three times as much active oxygen as potassium persulfate, but the latter has a stronger oxidation potential (stronger oxidizing agent) and is less effected by moisture. Consequently, it is preferred to use a mixture of persalts in order to obtain the combined advantages of both oxidizing agents. The total amount of oxidizing agents utilized herein may vary from 5%-50 % and preferably 20%-40% by weight of the total composition, the distribution between the various agents being dependent on the desired end result. More specifically, a composition having less active oxygen but yielding stronger oxidizing action will utilize a greater amount of the persulfate salt, whereas a formulation requiring a greater and quick release of active oxygen will employ a greater amount of the perborate. Other suitable solid peroxygen compounds include ammonium persulfate, sodium persulfate, sodium pyrophosphate peroxide, sodium carbonate peroxide, etc.

In the formation of tablets from the aforescribed effervescent formulation, the mixture is preferably in the granular form in order to be easily fed to the die cavity of the tableting machine. However, this does not preclude the direct feeding of the powdered mixture to the compression molding machine in the formulation of tablets. One satisfactory granulation method constitutes in the wet granulation of the mixture by the addition thereto of a solution of a binding material such as polyvinylpyrrolidone in an anhydrous solvent such as isopropanol, breaking it up into granules via an oscillator or like apparatus, screening and drying. The ingredients may be separately granulated and the granulations mixed prior to compression molding, or the powdered mixture of ingredients may be granulated prior to tableting. Polyvinylpyrrolidone is a polymer of vinylpyrrolidone having a molecular weight between 10,000 and 360,000, soluble in water and in organic solvents such as aliphatic alcohols (methyl, ethyl, propyl, isopropyl, butyl alcohols), ketones (acetone, methyl ethyl ketone), chlorinated hydrocarbons (methylene chloride, ethylene dichloride, trichloroethylene, trichloroethane, chloroform, carbon tetrachloride), etc. The amount of polyvinylpyrrolidone in the tablet may vary from 0.14% to 5%.

Another method of granulation is slugging whereby the powdered mixture is fed into a tableting device in order to make a poor grade tablet which is easily broken up into granules and screened.

Still another granulation method involves the formation of sheets from the powdered mixture by passing the mixture between two rollers, breaking the sheets into granules and screening.

A lubricant must be added to the dry effervescent mixture, whether in the form of powder or granules, as a final addition

step prior to compression molding into tablets. This lubricates the dies in the tableting device and permits the clean and easy ejection or removal of the tablets therefrom. No sticking or breakage occurs, thereby permitting the tableting machine to run continuously and efficiently. The art is replete with myriad lubricants inclusive of fatty acids, soaps, fatty acid salts, etc.

It has now been found that the addition of at least one water-soluble detergent lubricant to the effervescent mixture as a terminal addition step in the tableting process, is productive of clean, sharp-edged tablets, readily and completely soluble in water. In addition to its lubricant properties, the detergent assists in the cleansing action of the composition. Both the anionic and nonionic detergents are effective lubricants in amounts of about 0.1% to 5.0% by weight of the total composition. Although one surface active agent performs the required lubrication, excessive foaming sometimes occurs upon dissolution of the formulation in water, thereby requiring the adjunct of a foam depressant such as a silicone, etc. However, the use of a combination of anionic and nonionic detergents eliminates excessive foaming, thereby obviating the necessity of adding a foam depressant.

Suitable anionic detergents include water soluble salts of organic sulfox compounds having in their molecular structure an alkyl or acyl radical of carbon atom content within the range of about 8 to about 18 and a sulfonic acid or a sulfuric acid ester radical. Important examples of these anionic detergents are: sodium or potassium alkyl benzene sulfonate in which the alkyl group contains from about 9 to about 15 carbon atoms in either a straight chain or a branched chain which is derived from polymers of propylene; sodium and potassium alkyl glyceryl ether sulfonates, especially those ethers of higher fatty alcohols derived from the reduction of coconut oil; the reaction product of higher fatty acids with sodium or potassium isethionate, where, for example, the fatty acids are derived from coconut oil; sodium or potassium alkyl sulfonates and sulfates, especially those alkyl sulfates derived by the sulfation of coconut or tallow fatty alcohols and mixtures of such alkyl sulfates; dialkyl esters of sodium or potassium salts of sulfosuccinic acid, for example, the dihexyl ester; sodium and potassium salts of sulfated or sulfonated monoglycerides derived, for example, from coconut oil; sodium or potassium salts of higher fatty alcohol esters of sulfacarbonylic acids, for example, the sodium salt of the lauryl alcohol ester of sulfoacetic acid; sodium or potassium salts of a higher fatty acid amide of methyl taurine in which the higher acyl radical for example, are derived from coconut oil. Examples of other useful anionic detergents are acyl sarcosinates, e.g. sodium N-lauroyl sarcosinate.

Nonionic detergents include a water solubilizing polyoxyethylene group in chemical combination with an organic hydrophobic compound such as polyoxypropylene, alkyl phenol, the reaction product of an excess of propylene oxide and ethylene diamine, and aliphatic alcohols. The nonionic synthetic detergents have a molecular weight in the range of from about 800 to about 11,000. Other nonionic synthetic detergents useful in the present invention are: condensation products of 10 to 30 moles of ethylene oxide with one mole of an alkyl phenol containing 6 to 12 carbon atoms, either in a straight or branched chain, in the alkyl group (e.g. nonyl or octylphenol); condensation products of 10 to 30 moles of ethylene oxide with 1 mole of an aliphatic straight or branched chain alcohol containing 8 to 18 carbon atoms (e.g. lauryl alcohol or tallow fatty alcohol); condensation products of ethylene oxide and the reaction product of propylene oxide and ethylene diamine wherein the reaction product has a molecular weight of 2500-3000, for example and the condensation product has a polyoxyethylene content of 40% to 80%.

The effervescent denture soak composition may contain certain water-soluble dyes which act as timing indicators by turning colorless at the completion of the cleansing action. The initial coloration of the dye, upon dissolution of the dry composition in water, is bleached out in a period of time approximating the time required for cleaning the denture. The

type and amount of dye selected is dependent on the desired completion time of the cleansing activity as well as on the composition and pH of the denture product. It has been found that a period of at least 10-15 minutes and up to 30 minutes is a desirable cleansing time. Accordingly, at substantially neutral pH, the azo dyes have been found particularly desirable due to their ability to be readily oxidized to a colorless state in the required time. A specific example of an azo dye is acid red 014 (ext D and C red 010) which is the disodium salt of 2-(4-sulfo-1-naphthyl-azo) 1-naphthol-4-sulfonic acid. Other dyes with suitable fading times are FD and C Blue 02, FD and C Green 01, FD and C Green 02, FD and C Violet 01, D and C Yellow 010, Ext D and C Violet 02 and Ext D and C Yellow 03. The amount of dye incorporated in the composition may vary from about 0.005% to 0.5% and preferably from 0.01%-0.04% by weight of the total.

Various other adjuvant materials may also be incorporated in the present denture soak preparation. Minor amounts of flavor oils such as oils of spearmint, peppermint and wintergreen may be included to impart a mild and pleasant flavor and odor to the cleaned denture. However, when adding a flavor oil in amounts not exceeding 0.25%, it has been found desirable to also incorporate minor amounts about 1.00% of a flavor retainer or carrier such as magnesium carbonate to avoid the separation of the oil droplets from the rest of the dry composition. Other flavor retainers may be utilized provided they readily absorb the flavor oils and prevent flavor weeping. Minor amounts of other suitable water-soluble additives may be included such as sweeteners, foam depressants, preservatives, buffers, fillers, diluents, binders, etc., provided they do not adversely affect the properties and characteristics of the denture soak tablet.

The denture cleansing tablets of the instant invention may be packaged in bulk in assorted containers or individually wrapped in tin foil, polyethylene, cellophane or other foils. It is desirable that aforesaid product completely dissolve in water to yield a clear solution having a substantially neutral pH, at which carbon dioxide and some oxygen become readily available, thereby providing the effervescence required for the proper cleansing of dentures. An alkaline solution has the disadvantage of hastening the corrosion of metal parts of the dentures, whereas acid solutions tend to etch the dentures.

The term denture includes all kinds of orthodontic appliances such as false teeth, removable dental plates and bridges, artificial teeth and the like.

The following specific examples are further illustrative of the nature of the present invention, but it is to be understood that the invention is not limited thereto. The compositions are prepared in the usual manner as indicated, and all amounts of the various ingredients are by weight unless otherwise specified.

Example I

Ingredients	%
Na H CO ₃	30.00
Citric acid	20.00
KH SO ₄ 28.00	
Na BO ₂ H ₂ O ₄	15.00
Polyvinylpyrrolidone (M.W. 40,000)	1.10
Flavor oils	0.25
Mg CO ₃	1.00
FD and C Blue 02	0.015
Na H CO ₃	2.385
Sodium Lauryl Sulfate	0.25
Sodium benzoate	2.00

The sodium bicarbonate, citric acid, potassium persulfate, sodium perborate and a 1.1% solution of polyvinylpyrrolidone in isopropanol are thoroughly blended. The flavor oils are thoroughly mixed with magnesium carbonate and subsequently blended with the first mixture. The dye is mixed

with 2.385% sodium bicarbonate and subsequently added to the flavored mixture. The sodium lauryl sulfate and the sodium benzoate is finally added and thoroughly mixed (about 1 minute) with the dye and flavored composition to produce a homogeneous mixture which is fed to a tableting machine and compressed under normal pressure (3-5 tons). The tablets do not cap or pick or break easily, but have sharp edges and clean surfaces.

Example II

The flavor content of Example I is increased to 1.0%, and 0.04% FD and C Violet 01 in 1.71 sodium bicarbonate is substituted for the blue dye. This mixture is subjected to normal pressure in a tableting device, yielding cap-free, pick-free tablets.

Example III

Sodium salt of dodecyl benzene sulfonate is substituted for sodium lauryl sulfate in Example I. The resultant tablets are sharp-edged and clean.

EXAMPLE IV

Ingredients	%
Tartaric acid	21.50
Acid red 014	.01
Na H CO ₃	34.14
KH SO ₃	28.00
Na BO ₂ H ₂ O ₂	15.00
Sodium salt of dodecyl benzene sulfonate	0.1
Flavor	0.25
Mg CO ₃	1.00

Three separate granulations are made; the tartaric acid and the Acid Red dye are wetted with an aqueous isopropanol solution and dried, the Na H CO₃ is wetted with an aqueous isopropanol solution and dried, the KH SO₃ and Na BO₂ H₂O₂ are wetted and dried as above. The flavor is blended with the magnesium carbonate and mixed with the three granulations. The sodium salt of dodecyl benzene sulfonate is added to and mixed with the above granulated mixture for 1 minute and then subjected to compression molding at normal pressure in a tableting device. The tablets have clean surfaces and sharp edges and are readily and completely soluble in water as an efficient denture cleanser.

Example V

Malic acid is substituted for the tartaric acid in Example IV, yielding similarly improved tablets.

Example VI

Succinic acid is substituted for the tartaric acid of Example IV, resulting in improved tablets.

Example VII

Ingredients	Parts
Malic acid	8.77
Succinic anhydride	5.00
Na H CO ₃	22.88
KH SO ₃ 28.00	
Na BO ₂ H ₂ O ₂	15.00
Sodium salt of dodecyl benzene sulfonate	0.8
Soap chips	0.2

The malic acid, succinic anhydride, Na H CO₃, KH SO₃ and Na BO₂ H₂O₂ are thoroughly blended. The sulfonate and the soap chips are added to and mixed with the above composition

for 1 minute and then fed to the tablet press. The tablets do not stick to the machine.

Example VIII

Ingredients	%
Malic Acid	19.27
Boric anhydride	5.00
Na H CO ₃	45.00
KH SO ₃	18.67
Na BO ₂ H ₂ O ₂	10.00
Methyl naphthalene sodium sulfonate	1.60
Alkylphenoxypoly(ethyleneoxy)ethanol	0.20
Acid Red 014-0.01%-Flavor oils-0.25%	

The ingredients may either be mixed and made into a slug for tableting purposes or may be prepared into a wet granulation and then subjected to compression molding in a tableting device.

Example IX

Maleic anhydride is substituted for the boric anhydride of Example VIII. The formulation is adequately lubricated to eliminate sticking in the tableting device.

Example X

0.01% of Acid Red 014 dye is preblended with 7.73% malic acid and 12.26% Na H CO₃ and thoroughly blended with the formulation in Example VIII along with 0.25% flavor oils. The mixture is subjected to normal pressure in a tableting device yielding improved tablets in accordance with this invention.

Although the present invention has been described with reference to particular embodiments and examples, it will be apparent to those skilled in the art that variations and modifications of this invention can be made and that equivalents can be substituted therefor without departing from the principles and true spirit of the invention.

I claim:

1. In a process of preparing a water-soluble neutral pH, effervescent tablet having clean surfaces and sharp edges, and consisting essentially of about 5%-75% of a water-soluble alkali-metal carbonate, about 5%-50% of a nontoxic water-soluble organic acid, and about 5%-50% of at least one inorganic peroxygen compound, said carbonate, organic acid, and peroxygen compound constituting a major portion of said tablet, the improvement which comprises the terminal addition of 0.1%-5% of at least one water-soluble anionic or nonionic detergent to the above formulation and forming a homogeneous mixture, and molding said mixture into a tablet whereby said detergent lubricates the mold and said tablet is easily released therefrom with clean surfaces and sharp edges.

2. In a process in accordance with claim 1, wherein the water-soluble detergent is anionic.

3. In a process in accordance with claim 1, wherein a mixture of anionic and nonionic detergents are used.

4. In a process in accordance with claim 1, wherein the tablet contains a maximum of 0.25% flavor oils.

5. In a process in accordance with claim 1, wherein the tablet contains 0.005%-0.5% of a nontoxic water-soluble dye capable of being oxidized to its colorless state within 10 to 30 minutes.

6. In a process in accordance with claim 1, wherein the water-soluble carbonate is a bicarbonate.

7. In a process in accordance with claim 1, wherein the effervescent tablet contains 1%-60% of a nontoxic solid, water-soluble acid anhydride.

8. In a process in accordance with claim 1, wherein the tablet contains 0.14% to 5% polyvinylpyrrolidone, molecular weight 10,000 to 360,000.