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COMPOSITION

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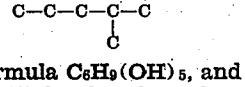
This invention relates to compositions comprising a hexahydric alcohol selected from the group consisting of sorbitol, iditol, and talitol, and a substantial proportion of a crystallization inhibitor therefor.

An object of the invention is the production of a composition of matter distinguished by resistance to gelation or crystallization in concentrated aqueous solution, desirable humectant properties, high hydroxyl content, and freedom from objectionable color, taste and odor.

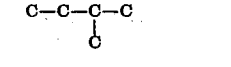
Other objects of the invention will hereinafter more fully appear.

Although the sparingly water-soluble hexahydric alcohols, mannitol and dulcitol, are not suited to humectant or conditioning applications, it has heretofore been proposed to use the readily water-soluble, straight chain aliphatic hexahydric alcohols, namely sorbitol, iditol and talitol as conditioning or softening agents for textiles, leather, tobacco, paper, glue, etc. However, when these water-soluble hexahydric alcohols are applied in aqueous solution to textiles, paper, etc., they tend to change from the fluid to a solid state, gelatinizing or crystallizing to form a harsh, horny film, and thus lose their softening or humectant power.

In accordance with the present invention, conditioning syrups are prepared in which one or more of the readily water-soluble hexahydric alcohols of the group aforesaid are admixed in aqueous solution with a crystallization inhibitor taken from the group consisting of branched chain desoxy hexitols of carbon skeleton



and empirical formula $C_6H_9(OH)_5$, and branched chain desoxy pentitols of carbon skeleton



and empirical formula $C_5H_8(OH)_4$. In the preferred embodiment of the invention the conditioning syrup is a mixture comprising at least three components, having a first portion comprising one or more of the readily water-soluble hexahydric alcohols selected from the group consisting of sorbitol, iditol and talitol, a second portion comprising one or more of the branched chain desoxy compounds referred to above, and a third portion comprising one or more of the readily water-soluble anhydro derivatives of the aliphatic straight chain hexahydric alcohols (including the anhydro derivatives mannitan, mannide, dulcitan, and dulcide which are materially different from their parent alcohols in that they are readily adapted to humectant applications).

The desoxy hexitols and pentitols inhibit crystallization of the hexahydric alcohol portion when admixed therewith, and in addition, these desoxy compounds are water-soluble and polyhydric in character thereby preserving to the composite syrup the conditioning and humectant powers desired. The anhydro derivatives referred to above likewise are readily water-soluble, polyhydric in character, and do not therefore detract from the humectant value of the syrup but rather, due to their hygroscopicity, impart to the syrup their conditioning and humectant values. If desired, the composition may also contain up to 30% sodium salts of hydroxy acids, such as lactic acid, the saccharinic acids $C_6H_7(OH_4).COOH$, and other four, five and six carbon atom, aliphatic hydroxy acids. Although the compositions of the present invention can be induced to gelatinize or crystallize under special conditions, under conditions of ordinary humectant use they retain enough water to prevent crystallization and hence do not dry out, harden and lose humectant power.

The compositions of the invention, due to their hydrophile character, are good softeners or conditioning agents for textiles, paper, regenerated cellulose, leather, tobacco, glues and gelatin compositions, urea formaldehyde and phenol formaldehyde plastics, and the like. The compositions are insoluble in hydrophile solvents with but few exceptions among which are acetic acid, methyl alcohol, pyridine, phenol and acetamide, which is advantageous in many applications where resistance to organic solvents is of importance.

Although the product of the invention is complex, the physical and chemical properties of its components are so similar, that the composition may be put through a variety of processes without undergoing sensible changes in composition due to selective removal of one of the components by a step in the process. Thus, all components of the mixture are absorbed to approximately the same extent by cellulosic materials from dilute aqueous solution, so that a conditioning bath containing the product of the invention does not undergo appreciable change in composition during use.

The product is polyhydroxylic, and sufficiently heat stable to permit its use as a starting material for the production of various technically valuable esters, acetals, and ethers, metallic complexes, and volatile anhydro products, by reacting with the appropriate reagents.

Although the compositions of the invention may be prepared in any suitable manner, such as by assembling and admixing the various com-

ponents, it has been found that the compositions may be readily produced by the electrolytic reduction of a monosaccharide, such as glucose, fructose or a mixture thereof such as inverted sucrose, under specified conditions as to temperature, time of reduction, alkalinity and current strength, said reduction being followed by the removal of undesirable by-products of the reaction by the recovery process described and claimed in my co-pending application Serial No. 100,390, filed September 11, 1936, now Patent No. 2,116,665.

In the following examples of electrolytic preparation of the products of the invention, a cell such as that described in U. S. Letters Patent to H. J. Creighton No. 1,990,582 was employed. The conditions of reduction are tabulated in Table I for the main process variables. In each case, the catholyte was made up originally to 325 gm. of sugar and 80 gm. sodium sulfate per liter, while the anolyte acidity was kept at 300 to 425 gm. sulfuric acid per liter. The cathode ratio referred to is the square decimeters of gross cathode surface facing the anode divided by the volume of the catholyte in liters. The current density employed in each reduction was reduced from approximately 1.0 to 0.5 ampere per square decimeter at the time approximately 65% of the sugar was reduced.

Table I

Example	1	2	3	4	5	6
Sugar	Glucose	Glucose	Glucose	Glucose	Invert	Glucose
Catholyte Alkalinity gms. Na OH/liter	10–20	2	10–15	0–20	0–2	0–2
Catholyte temperature °C	18–21	23–30	21–24	18–19	20–21	18–21
Cathode ratio dm^2/L	1.25	2.5	2.5	2.0	8	2.0
Reduction time, hours	178	184	141	237	55	311
Percent reduction	90	99	90	90	90	97

After reduction, the water soluble compositions of the present invention were recovered from the catholyte of Examples 1, 3 and 4 as follows:

The alkaline catholyte was neutralized with sulfuric acid and evaporated under vacuum at 70° C. to a thick sludge. The sludge was extracted with hot 81½% by volume denatured alcohol, using 0.25 gal. per lb. of reduction product, at 70° C. for ½ to 1 hour, the salt filtered off, and washed three times with 81½% alcohol, the rinsings being equal in volume to one-third of the amount of the first extraction.

The alcoholic solution of the reaction product, containing mannitol, was then cooled at the rate of about 3° C. every two hours down to 20° C. Slow agitation was used for five minutes every hour. After 20° C. was reached, agitation was continued for one hour, when the mannitol was filtered off on a centrifugal machine, washing with strong alcohol.

The alcoholic filtrate was evaporated, the residual syrup diluted to 50% by addition of water, and treated with 2% of a decolorizing carbon. The residual sugar was then fermented out with yeast, using an amount of the latter equal to one-fifth of the sugar present, a temperature of 27° to 30° C., and a pH of 5.0 to 5.5. After fermentation, the syrup was treated again with decolorizing carbon, and finally evaporated to a convenient moisture content—about 15% by weight.

In Example 2, the recovery was carried out as in Examples 1, 3 and 4, except that fermentation was omitted since reduction was carried to virtual completion. In Examples 5 and 6 the recovery was carried out as in Examples 1, 3 and 4, except that, there being substantially no mannitol present, the mannitol crystallization was omitted.

Preferably, the compositions are substantially free of inorganic salt and sugar, but for many humectant purposes the presence of up to 2% of sodium sulfate and of reducing sugar is not only harmless but even beneficial. For other uses, such as the making of print roll compositions wherein ash-containing glue is used, it is obviously unnecessary to prepare an ash-free composition, but, sugar must be absent to prevent tanning of the glues. In other cases, where the composition is used as a starting material for high temperature esterification products, small amounts of sugars can be tolerated, if the ash is low enough. Hence, where the compositions are recovered from the catholyte in the electrolytic reduction of sugars, the amount of residual ash and sugar is regulated according to the technical and economic requirements of the use to which the product is to be put. The essential feature of the invention resides in the complex composition of matter containing a plurality of polyhydroxy compounds.

The composition of the six examples on a water-free, ash-free, sugar-free basis is given in Table II. The hexitol component in these examples is substantially all sorbitol.

Table II

Example	1	2	3	4	5	6
Hexitol	67.7	55.0	53.9	34.22	49.75	52.2
Desoxy hexitol	16.5	28.0	18.3	21.7	22.75	18.6
Desoxy pentitol	4.4	5.25	7.38	4.58	7.0	3.0
Anhydrohexitol	4.4	7.25	7.33	25.6	20.0	1.66
Organic sodium salt	7.0	4.50	13.1	13.9	0.5	24.5

In the foregoing examples, sorbitol is the predominating component of that portion of the composition comprising one or more materials taken from the group consisting of sorbitol, iditol, talitol and the readily water-soluble anhydro derivatives of the aliphatic, straight chain hexahydric alcohols. It is to be understood, however, that the invention is not to be taken as limited to compositions which are predominantly sorbitol, and that iditol or talitol may predominate in the composition if desired.

No single component of the portion of the composition comprising one or more hexahydric alcohol taken from the group consisting of sorbitol, iditol and talitol is present in the composition in an amount in excess of 70% by weight of the total composition on a water-free, ash-free, sugar-free basis, in order to avoid any tendency of the composition to gelatinize or crystallize. Preferably, no single component of the portion of the composition comprising one or more materials taken from the group consisting of desoxy hexitols and desoxy pentitols is present in the composition in an amount in excess of 50% by

weight of the total composition on a water-free, ash-free, sugar-free basis. While the latter portion of the composition acts to inhibit crystallization of the first portion, it is preferred that no single desoxy component be present in excess of 50% since if present in such an excess it tends to gelatinize or crystallize itself, thereby decreasing the humectant and conditioning value of the syrup. In addition, it is preferred that no single component of the portion of the composition comprising one or more of the readily water-soluble anhydro derivatives of the hexitols should be present in an amount in excess of 50% by weight of the total composition. It will be understood that where any single portion contains a substantial amount of each of two or more components, such portion may be present in amounts greater than where said portion contains only one component for the reason that the increased complexity within the portion tends to prevent crystallization or gelatinization of the components.

Although several methods of preparing the compositions of the present invention have been disclosed, it is to be understood that the compositions may be prepared by other methods if so desired. For example, complex compositions containing sorbitol in excess of 70% and desoxy hexitols and pentitols may be adjusted to the desired proportions by removing sorbitol preferentially from the mixture by crystallization or other means, thus, in effect, increasing the amount of other polyhydric products of the composition. Or, the desired components of the composition may be assembled from different sources and blended in the desired proportions.

As can be seen from the foregoing, the proportions of the ingredients of the composition may vary over wide limits so long as the polyhydric components are present in sufficient complexity and amount to inhibit crystallization or gelatinization of the composition. Accordingly, the invention is not to be limited to the above disclosure, but is to be limited only as set forth in the appended claims.

In the claims the percentages are based upon the total weight of solids in the composition upon a water-free, ash-free, sugar-free basis.

Having described my invention, what I claim is:

1. A substantially mannitol-free and substantially alkali sulfate-free water soluble conditioning and humectant composition having a substantial proportion of a first portion consisting of at least one component selected from the group consisting of sorbitol, iditol and talitol, any single component of said first portion being present in an amount not greater than 70% by weight of the total solids in the composiiton, and a substantial proportion of a second portion consisting of at least one component selected from the group consisting of branched chain desoxy hexitols and pentitols, any single component of said second portion being present in an amount not greater than 50% by weight of the total solids in the composition, said composition having the various components present in sufficient complexity and amount to inhibit crystallization or gelatinization of the composition.

2. A substantially mannitol-free and substantially alkali sulfate-free water soluble conditioning and humectant composition comprising at least three components and having a substantial proportion of a first portion consisting of at least one component selected from the group consisting of sorbitol, iditol and talitol, any single component of said first portion being present in an amount not greater than 70% by weight of the total solids in the composition, and a substantial proportion of a second portion consisting of at least one component selected from the group consisting of branched chain desoxy hexitols and pentitols, any single component of said second portion being present in an amount not greater than 50% by weight of the total solids in the composition, said composition having the various components present in sufficient complexity and amount to inhibit crystallization or gelatinization of the composition.

3. A substantially mannitol-free and substantially alkali sulfate-free water soluble conditioning and humectant composition having a substantial proportion of a first portion consisting of at least one component selected from the group consisting of sorbitol, iditol and talitol, any single component of said first portion being present in an amount not greater than 70% by weight of the total solids in the composition, and a substantial portion of a second portion consisting of desoxy hexitol and desoxy pentitol, each present in an amount not greater than 50% by weight of the total solids in the composition, said composition having the various components present in sufficient complexity and amount to inhibit crystallization or gelatinization of the composition.

4. A substantially mannitol-free and substantially alkali sulfate-free water soluble conditioning and humectant composition having a substantial proportion of a first portion consisting of at least one component selected from the group consisting of sorbitol, iditol and talitol, any single component of said first portion being present in an amount not greater than 70% by weight of the total solids in the composition, a substantial proportion of a second portion consisting of at least one component selected from the group consisting of branched chain desoxy hexitols and pentitols, any single component of said second portion being present in an amount not greater than 50% by weight of the total solids in the composition, and a substantial proportion of a third portion consisting of at least one of the readily water soluble anhydro derivatives of the aliphatic, straight chain hexahydric alcohols, any single component of said third portion being present in an amount not greater than 50% by weight of the total solids in the composition, said composition having the various components present in sufficient complexity and amount to inhibit crystallization or gelatinization of the composition.

5. A substantially mannitol-free and substantially alkali sulfate-free water soluble conditioning and humectant composition having a substantial proportion of a first portion consisting of at least one component selected from the group consisting of sorbitol, iditol and talitol, any single component of said first portion being present in an amount not greater than 70% by weight of the total solids in the composition, a substantial proportion of a second portion consisting of at least one component selected from the group consisting of branched chain desoxy hexitols and pentitols, any single component of said second portion being present in an amount not greater than 50% by weight of the total solids in the composition, and not more than 30% by weight of total solids of sodium salts of hydroxy acids taken from the group consisting of lactic acid

and four, five and six carbon atom aliphatic hydroxy carboxylic acids containing only carbon, hydrogen and oxygen, said composition having the various components present in sufficient complexity and amount to inhibit crystallization or gelatinization of the composition.

6. A substantially mannitol-free and substantially alkali sulfate-free water soluble conditioning and humectant composition having a substantial proportion of a first portion consisting of at least one component selected from the group consisting of sorbitol, iditol and talitol, any single component of said first portion being present in an amount not greater than 70% by weight of the total solids in the composition, a substantial proportion of a second portion consisting of at least one component selected from the group consisting of branched chain desoxy hexitols and pentitols, any single component of said second portion being present in an amount not greater than 50% by weight of the total solids in the composition, a substantial proportion of a third portion consisting of at least one of the readily water-soluble anhydro derivatives of the aliphatic, straight chain hexahydric alcohols, any single component of said third portion being present in an amount not greater than 50% by weight of the total solids in the composition and not more than 30% by weight of total solids of sodium salts of hydroxy acids taken from the group consisting of lactic acid and four, five and six carbon atom aliphatic hydroxy carboxylic acids, containing only carbon, hydrogen and oxygen, said composition having the various components present in sufficient complexity and amount to inhibit crystallization or gelatinization of the composition.

7. A composition as set forth in claim 1 and in which the first portion is predominantly sorbitol.

8. A composition as set forth in claim 2 and in which the first portion is predominantly sorbitol.

9. A composition as set forth in claim 3 and in which the first portion is predominantly sorbitol.

10. A composition as set forth in claim 4 and in which the first portion is predominantly sorbitol.

11. A composition as set forth in claim 5 and in which the first portion is predominantly sorbitol.

12. A composition as set forth in claim 6 and in which the first portion is predominantly sorbitol.

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