Title: CYCLODEXTRIN INCLUSION COMPLEXES AND METHODS OF PREPARING SAME

Abstract: Cyclodextrin inclusion complexes, guest stabilizing systems, and methods for preparing and using the same. Some embodiments of the present invention provide a method for making a guest stabilizing system. The method can include mixing cyclodextrin, a solvent and a guest to form a cyclodextrin inclusion complex. The method can further include adding uncomplexed cyclodextrin to the cyclodextrin inclusion complex to form a guest stabilizing system. Some embodiments of the present invention provide a method for making a beverage that can include mixing uncomplexed cyclodextrin, a guest and a solvent to form a beverage.
CROSS-REFERENCE TO RELATED APPLICATIONS

[0001] Priority is hereby claimed to U.S. Provisional Patent Application No. 60/690,181, filed June 13, 2005, the entire contents of which are incorporated herein by reference.

BACKGROUND

[0002] The following U.S. Patents disclose the use of cyclodextrins to complex various guest molecules, and are hereby fully incorporated herein by reference: U.S. Pat. Nos. 4,296,137, 4,296,138 and 4,348,416 to Borden (flavoring material for use in chewing gum, dentifrices, cosmetics, etc.); 4,265,779 to Gandolfo et al. (suds suppressors in detergent compositions); 3,816,393 and 4,054,736 to Hyashi et al. (prostaglandins for use as a pharmaceutical); 3,846,551 to Mifune et al. (insecticidal and acaricidal compositions); 4,024,223 to Noda et al. (menthol, methyl salicylate, and the like); 4,073,931 to Akito et al. (nitro-glycerine); 4,228,160 to Szjelti et al. (indomethacin); 4,247,535 to Bernstein et al. (complement inhibitors); 4,268,501 to Kawamura et al. (anti-asthmatic actives); 4,365,061 to Szjelti et al. (strong inorganic acid complexes); 4,371,673 to Pitha (retinoids); 4,380,626 to Szjelti et al. (hormonal plant growth regulator), 4,438,106 to Wagu et al. (long chain fatty acids useful to reduce cholesterol); 4,474,822 to Sato et al. (tea essence complexes); 4,529,608 to Szjelti et al. (honey aroma), 4,547,365 to Kuno et al. (hair waving active- complexes); 4,596,795 to Pitha (sex hormones); 4,616,008 Hirai et al. (antibacterial complexes); 4,636,343 to Shibanai (insecticide complexes), 4,663,316 to Ninger et al. (antibiotics); 4,675,395 to Fukazawa et al. (hinokitiol); 4,732,759 and 4,728,510 to Shibanai et al. (bath additives); 4,751,095 to Karl et al. (aspartamane); 4,560,571 (coffee extract); 4,632,832 to Okonogi et al. (instant creaming powder); 5,571,782, 5,660,845 and 5,635,238 to Trinh et al. (perfumes, flavors, and pharmaceuticals); 4,548,811 to Kubo et al. (waving lotion); 6,287,603 to Prasad et al. (perfumes, flavors, and pharmaceuticals); 4,906,488 to Pera (olfactants, flavors, medicaments, and pesticides); and 6,638,557 to Qi et al. (fish oils).

[0003] Cyclodextrins are further described in the following publications, which are also incorporated herein by reference: (1) Reineccius, T.A., et al. "Encapsulation of flavors using cyclodextrins: comparison of flavor retention in alpha, beta, and gamma types." Journal of

SUMMARY

[0004] Some embodiments of the present invention provide a method for preparing a cyclodextrin inclusion complex. The method can include mixing cyclodextrin and an emulsifier to form a dry blend, and mixing a solvent and a guest with the dry blend to form a cyclodextrin inclusion complex.

[0005] In some embodiments of the present invention, a method for preparing a cyclodextrin inclusion complex is provided. The method can include mixing cyclodextrin and an emulsifier to form a first mixture, mixing the first mixture with a solvent to form a second mixture, and mixing a guest with the second mixture to form a third mixture.

[0006] Some embodiments of the present invention provide a method for preparing a cyclodextrin inclusion complex. The method can include dry blending cyclodextrin and pectin to form a first mixture, mixing the first mixture with water to form a second mixture, and mixing diacetyl with the second mixture to form a third mixture.

[0007] In some embodiments of the present invention, a method for making a guest stabilizing system is provided. The method can include mixing cyclodextrin and an emulsifier to form a mixture, mixing a solvent and a guest with the mixture to form a cyclodextrin inclusion complex, and adding uncomplexed cyclodextrin to the cyclodextrin inclusion complex to form a guest stabilizing system.

[0008] Some embodiments of the present invention provide a method for making a guest stabilizing system. The method can include mixing cyclodextrin, a solvent and a guest to form a cyclodextrin inclusion complex. The guest can be added in an excess molar ratio of
guest to cyclodextrin. The method can further include adding uncomplexed cyclodextrin to the cyclodextrin inclusion complex to form a guest stabilizing system. The uncomplexed cyclodextrin can be added in an excess molar ratio of total cyclodextrin to guest to increase the ratio of complexed guest to free guest in the guest stabilizing system to further stabilize the guest from degradation.

[0009] In some embodiments of the present invention, a method for making a beverage is provided. The method can include mixing uncomplexed cyclodextrin, a guest and a solvent to form a beverage. The guest can have a positive log (P) value. The cyclodextrin can be added to the beverage in a weight percentage of cyclodextrin to the beverage ranging from about 0.05 wt % to about 0.3 wt %.

[0010] Other features and aspects of the invention will become apparent by consideration of the detailed description and accompanying drawings.

BRIEF DESCRIPTION OF THE DRAWINGS

[0011] FIG. 1 is a schematic illustration of a cyclodextrin molecule having a cavity, and a guest molecule held within the cavity.

[0012] FIG. 2 is a schematic illustration of a nano-structure formed by self-assembled cyclodextrin molecules and guest molecules.

[0013] FIG. 3 is a schematic illustration of the formation of a diacetyl-cyclodextrin inclusion complex.

[0014] FIG. 4 is a schematic illustration of a nano-structure formed by self-assembled cyclodextrin molecules and diacetyl molecules.

[0015] FIG. 5 is a schematic illustration of the formation of a citral-cyclodextrin inclusion complex.

[0016] FIG. 6 is a schematic illustration of a nano-structure formed by self-assembled cyclodextrin molecules and citral molecules.

[0017] FIG. 7 illustrates a degradation mechanism for citral.
FIG. 7A is a schematic illustration of a three-phase model used to represent a guest-cyclodextrin-solvent system.

FIGS. 8-11 illustrate the effect of cyclodextrin on levels of citral and off-notes formed according to Example 20.

FIGS. 12-15 illustrate the effect of cyclodextrin on levels of citral and off-notes formed according to Example 21.

FIGS. 16-17 illustrate the results of a sensory analysis described in Example 34.

FIGS. 18-19 illustrate the effect of cyclodextrin on levels of key note flavors and off-notes formed according to Examples 35-37.

FIG. 20 shows the results of the experiment set forth in Example 38.

DETAILED DESCRIPTION

Before any embodiments of the invention are explained in detail, it is to be understood that the invention is not limited in its application to the details of construction and the arrangement of components set forth in the following description or illustrated in the following drawings. The invention is capable of other embodiments and of being practiced or of being carried out in various ways. Also, it is to be understood that the phraseology and terminology used herein is for the purpose of description and should not be regarded as limiting. The use of "including," "comprising," or "having" and variations thereof herein is meant to encompass the items listed thereafter and equivalents thereof as well as additional items. Unless specified or limited otherwise, the terms "mounted," "connected," "supported," and "coupled" and variations thereof are used broadly and encompass both direct and indirect mountings, connections, supports, and couplings. Further, "connected" and "coupled" are not restricted to physical or mechanical connections or couplings.

It also is understood that any numerical range recited herein includes all values from the lower value to the upper value. For example, if a concentration range is stated as 1% to 50%, it is intended that values such as 2% to 40%, 10% to 30%, or 1% to 3%, etc., are expressly enumerated in this specification. These are only examples of what is specifically intended, and all possible combinations of numerical values between the lowest value and the highest value enumerated are to be considered to be expressly stated in this application.
[0026] The present invention is generally directed to cyclodextrin inclusion complexes and methods of forming them. Some cyclodextrin inclusion complexes of the present invention provide for the encapsulation of volatile and reactive guest molecules. In some embodiments, the encapsulation of the guest molecule can provide at least one of the following: (1) prevention of a volatile or reactive guest from escaping a commercial product which may result in a lack of flavor intensity in the commercial product; (2) isolation of the guest molecule from interaction and reaction with other components that would cause off note formation; (3) stabilization of the guest molecule against degradation (e.g., hydrolysis, oxidation, etc.); (4) selective extraction of the guest molecule from other products or compounds; (5) enhancement of the water solubility of the guest molecule; (6) taste or odor improvement or enhancement of a commercial product; (7) thermal protection of the guest in a microwave and conventional baking applications; (8) slow and/or sustained release of flavor or odor (e.g., in embodiments employing diacetyl as the guest molecule in cyclodextrin inclusion complex, it can provide the perception of melting butter); and (9) safe handling of guest molecules.

[0027] As used herein and in the appended claims, the term “cyclodextrin” can refer to a cyclic dextrin molecule that is formed by enzyme conversion of starch. Specific enzymes, e.g., various forms of cycloglycosyltransferase (CGTase), can break down helical structures that occur in starch to form specific cyclodextrin molecules having three-dimensional polyglucose rings with, e.g., 6, 7, or 8 glucose molecules. For example, α-CGTase can convert starch to α-cyclodextrin having 6 glucose units, β-CGTase can convert starch to β-cyclodextrin having 7 glucose units, and γ-CGTase can convert starch to γ-cyclodextrin having 8 glucose units. Cyclodextrins include, but are not limited to, at least one of α-cyclodextrin, β-cyclodextrin, γ-cyclodextrin, and combinations thereof. β-cyclodextrin is not known to have any toxic effects, is World-Wide GRAS (i.e., Generally Regarded As Safe) and natural, and is FDA approved. α-cyclodextrin and γ-cyclodextrin are also considered natural products and are U.S. and E.U. GRAS.

[0028] The three-dimensional cyclic structure (i.e., macrocyclic structure) of a cyclodextrin molecule 10 is shown schematically in FIG. 1. The cyclodextrin molecule 10 includes an external portion 12, which includes primary and secondary hydroxyl groups, and which is hydrophilic. The cyclodextrin molecule 10 also includes a three-dimensional cavity 14, which includes carbon atoms, hydrogen atoms and ether linkages, and which is
hydrophobic. The hydrophobic cavity 14 of the cyclodextrin molecule can act as a host and hold a variety of molecules, or guests 16, that include a hydrophobic portion to form a cyclodextrin inclusion complex.

[0029] As used herein and in the appended claims, the term “guest” can refer to any molecule of which at least a portion can be held or captured within the three dimensional cavity present in the cyclodextrin molecule, including, without limitation, at least one of a flavor, an olfactant, a pharmaceutical agent, a nutraceutical agent (e.g., creatine), and combinations thereof.

[0030] Examples of flavors can include, without limitation, flavors based on aldehydes, ketones or alcohols. Examples of aldehyde flavors can include, without limitation, at least one of: acetaldehyde (apple); benzaldehyde (cherry, almond); anisic aldehyde (licorice, anise); cinnamic aldehyde (cinnamon); citral (e.g., geranial, alpha citral (lemon, lime) and neral, beta citral (lemon, lime)); decanal (orange, lemon); ethyl vanillin (vanilla, cream); heliotropine, i.e. piperonal (vanilla, cream); vanillin (vanilla, cream); a-amyl cinnamaldehyde (spicy fruity flavors); butyraldehyde (butter, cheese); valeraldehyde (butter, cheese); citronellal (modifies, many types); decenal (citrus fruits); aldehyde C-8 (citrus fruits); aldehyde C-9 (citrus fruits); aldehyde C-12 (citrus fruits); 2-ethyl butyraldehyde (berry fruits); hexenal, i.e. trans-2 (berry fruits); tolyl aldehyde (cherry, almond); veratreraldehyde (vanilla); 2-6-dimethyl-5-heptenal, i.e. Melonal™ (melon); 2,6-dimethyloctanal (green fruit); 2-dodecenal (citrus, mandarin); and combinations thereof.

[0031] Examples of ketone flavors can include, without limitation, at least one of: d-carvone (caraway); 1-carvone (spearmint); diacetyl (butter, cheese, "cream"); benzophenone (fruity and spicy flavors, vanilla); methyl ethyl ketone (berry fruits); maltol (berry fruits) menthone (mints), methyl amy1 ketone, ethyl butyl ketone, dipropyl ketone, methyl hexyl ketone, ethyl amy1 ketone (berry fruits, stone fruits); pyruvic acid (smokey, nutty flavors); acetanisole (hawthorn heliotrope); dihydrocarvone (spearmint); 2,4-dimethylacetophenone (peppermint); 1,3-diphenyl-2-propanone (almond); acetocumene (orris and basil, spicy); isojasmone (jasmine); d-isomethylionone (orris like, violet); isobutyl acetooacetate (brandy-like); zingerone (ginger); pulegone (peppermint-camphor); d-piperitone (minty); 2-nonanone (rose and tea-like); and combinations thereof.
Examples of alcohol flavors can include, without limitation, at least one of anisic alcohol or p-methoxybenzyl alcohol (fruity, peach); benzyl alcohol (fruity); carvacrol or 2-p-cymenol (pungent warm odor); carveol; cinnamyl alcohol (floral odor); citronellol (rose like); decanol; dihydrocarveol (spicy, peppery); tetrahydrogeraniol or 3,7-dimethyl-1-octanol (rose odor); eugenol (clove); p-mentha-1,8-dien-7-O-β or perillyl alcohol (floral-pine); alpha terpineol; mentha-1,5-dien-8-ol 1; mentha-1,5-dien-8-ol 2; p-cymen-8-ol; and combinations thereof.

Examples of olfactants can include, without limitation, at least one of natural fragrances, synthetic fragrances, synthetic essential oils, natural essential oils, and combinations thereof.

Examples of the synthetic fragrances can include, without limitation, at least one of terpenic hydrocarbons, esters, ethers, alcohols, aldehydes, phenols, ketones, acetics, oximes, and combinations thereof.

Examples of terpenic hydrocarbons can include, without limitation, at least one of lime terpene, lemon terpene, limonen dimer, and combinations thereof.

Examples of esters can include, without limitation, at least one of γ-undecalactone, ethyl methyl phenyl glycidate, allyl caproate, amyl salicylate, amyl benzoate, amyl acetate, benzyl acetate, benzyl benzoate, benzyl salicylate, benzyl propionate, butyl acetate, benzyl butyrate, benzyl phenylacetate, cedryl acetate, citronellyl acetate, citronellyl formate, p-cresyl acetate, 2-t-pentyl-cyclohexyl acetate, cyclohexyl acetate, cis-3-hexenyl acetate, cis-3-hexenyl salicylate, dimethylbenzyl acetate, diethyl phthalate, 8-deca-lactone dibutyl phthalate, ethyl butyrate, ethyl acetate, ethyl benzoate, fenchyl acetate, geranyl acetate, γ-dodecalatone, methyl dihydrojasmonate, isobornyl acetate, β-isopropoxyethyl salicylate, linalyl acetate, methyl benzoate, o-t-butylycyclohexyl acetate, methyl salicylate, ethylene brassylate, ethylene dodecanolate, methyl phenyl acetate, phenylethyl isobutyrate, phenylethylphenyl acetate, phenylethyl acetate, methyl phenyl carbinyl acetate, 3,5,5-trimethylhexyl acetate, terpinyl acetate, triethyl citrate, p-t-butylycyclohexyl acetate, vetiver acetate, and combinations thereof.

Examples of ethers can include, without limitation, at least one of p-cresyl methyl ether, diphenyl ether, 1,3,4,6,7,8-hexahydro-4,6,7,8,8-hexamethyl cyclopenta-β-2-benzopyran, phenyl isoamyl ether, and combinations thereof.
Examples of alcohols can include, without limitation, at least one of n-octyl alcohol, n-nonyl alcohol, β-phenylethyl(dimethyl carbinol, dimethyl benzyl carbinol, carbitol dihydromycenol, dimethyl octanol, hexylene glycol linalool, leaf alcohol, nerol, phenoxyethanol, γ-phenyl-propyl alcohol, β-phenylethyl alcohol, methylphenyl carbinol, terpineol, tetraphydroalloccimenol, tetrahydrolinalool, 9-decen-1-ol, and combinations thereof.

Examples of aldehydes can include, without limitation, at least one of n-nonyl aldehyde, undecylene aldehyde, methylnonyl acetalddehyde, anisaldehyde, benzaldehyde, cyclamenaldehyde, 2-hexylhexenal, ahexylcinnamic aldehyde, phenyl acetaldehyde, 4-(4-hydroxy-4-methylpentyl)-3-cyclohexene-1-carboxyaldehyde, p-t-butyl-a-methylhydrocinnamic aldehyde, hydroxycitronellal, α-amylcinnamic aldehyde, 3,5-dimethyl-3-cyclohexene-1-carboxyaldehyde, and combinations thereof.

Examples of phenols can include, without limitation, methyl eugenol.

Examples of ketones can include, without limitation, at least one of 1-carvone, α-damascon, ionone, 4-t-pentylcyclohexanone, 3-amy-4-acetoxytetrahydropyran, menthone, methylionone, p-t-amycyclohexanone, acetyl cedrene, and combinations thereof.

Examples of the acetals can include, without limitation, phenylacetaldhydratedimethyl acetal.

Examples of oximes can include, without limitation, 5-methyl-3-heptanon oxime.

A guest can further include, without limitation, at least one of fatty acids, lactones, terpenes, diacetyl, dimethyl sulfide, proline, furanecol, linalool, acetyl propionyl, natural essences (e.g., orange, tomato, apple, cinnamon, raspberry, etc.), essential oils (e.g., orange, lemon, lime, etc.), sweeteners (e.g., aspartame, neotame, etc.), sabine, p-cymene, p,a-dimethyl styrene, and combinations thereof.

FIG. 3 shows a schematic illustration of the formation of a diacetyl-cyclodextrin inclusion complex, and FIG. 5 shows a schematic illustration of the formation of a citral-cyclodextrin inclusion complex.

As used herein and in the appended claims, the term “log (P)” or “log (P) value” is a property of a material that can be found in standard reference tables, and which refers to the
material's octanol/water partition coefficient. Generally, the log (P) value of a material is a representation of its hydrophilicity/hydrophobicity. P is defined as the ratio of the concentration of the material in octanol to the concentration of the material in water. Accordingly, the log (P) of a material of interest will be negative if the concentration of the material in water is higher than the concentration of the material in octanol. The log (P) value will be positive if the concentration is higher in octanol, and the log (P) value will be zero if the concentration of the material of interest is the same in water as in octanol. Accordingly, guests can be characterized by their log (P) value. For reference, Table 1A lists log (P) values for a variety of materials, some of which may be guests of the present invention.

Table 1A. Log (P) values for a variety of guests

<table>
<thead>
<tr>
<th>Material</th>
<th>CAS#</th>
<th>log P</th>
<th>molecular wt</th>
</tr>
</thead>
<tbody>
<tr>
<td>creatine</td>
<td>57-00-1</td>
<td>-3.72</td>
<td>131</td>
</tr>
<tr>
<td>proline</td>
<td>147-85-3</td>
<td>-2.15</td>
<td>115</td>
</tr>
<tr>
<td>diacetyl</td>
<td>431-03-8</td>
<td>-1.34</td>
<td>86</td>
</tr>
<tr>
<td>methanol</td>
<td>67-66-1</td>
<td>-0.74</td>
<td>32</td>
</tr>
<tr>
<td>ethanol</td>
<td>64-17-5</td>
<td>-0.30</td>
<td>46</td>
</tr>
<tr>
<td>acetone</td>
<td>67-64-1</td>
<td>-0.24</td>
<td>56</td>
</tr>
<tr>
<td>maltol</td>
<td>118-71-8</td>
<td>-0.19</td>
<td>126</td>
</tr>
<tr>
<td>ethyl lactate</td>
<td>97-64-3</td>
<td>-0.18</td>
<td>118</td>
</tr>
<tr>
<td>acetic acid</td>
<td>64-19-7</td>
<td>-0.17</td>
<td>60</td>
</tr>
<tr>
<td>acetaldehyde</td>
<td>75-07-0</td>
<td>-0.17</td>
<td>44</td>
</tr>
<tr>
<td>aspartame</td>
<td>22839-47-0</td>
<td>0.07</td>
<td>294</td>
</tr>
<tr>
<td>ethyl levulinate</td>
<td>539-88-8</td>
<td>0.29</td>
<td>144</td>
</tr>
<tr>
<td>ethyl maltol</td>
<td>4940-11-8</td>
<td>0.30</td>
<td>140</td>
</tr>
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<td>furaneol</td>
<td>3658-77-3</td>
<td>0.82</td>
<td>128</td>
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<td>dimethyl sulfide</td>
<td>75-18-3</td>
<td>0.92</td>
<td>62</td>
</tr>
<tr>
<td>vanillin</td>
<td>121-33-5</td>
<td>1.05</td>
<td>152</td>
</tr>
<tr>
<td>benzyl alcohol</td>
<td>100-51-6</td>
<td>1.05</td>
<td>108</td>
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<tr>
<td>raspberry ketone</td>
<td>5471-51-2</td>
<td>1.48</td>
<td>164</td>
</tr>
<tr>
<td>benzaldehyde</td>
<td>100-52-7</td>
<td>1.48</td>
<td>106</td>
</tr>
<tr>
<td>ethyl vanillin</td>
<td>121-32-4</td>
<td>1.50</td>
<td>166</td>
</tr>
<tr>
<td>phenethyl alcohol</td>
<td>60-12-8</td>
<td>1.57</td>
<td>122</td>
</tr>
<tr>
<td>cis-3-hexenol</td>
<td>928-96-1</td>
<td>1.61</td>
<td>100</td>
</tr>
<tr>
<td>trans-2-hexenol</td>
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<tr>
<td>whiskey fusel oils</td>
<td>mixture</td>
<td>1.75</td>
<td>74</td>
</tr>
<tr>
<td>ethyl isobutyrate</td>
<td>97-62-1</td>
<td>1.77</td>
<td>116</td>
</tr>
<tr>
<td>ethyl butyrate</td>
<td>105-54-4</td>
<td>1.85</td>
<td>116</td>
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<tr>
<td>hexanol</td>
<td>111-27-3</td>
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<td>102</td>
</tr>
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<td>130</td>
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<td>2.26</td>
<td>130</td>
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<tr>
<td>isoamyl acetate</td>
<td>123-92-2</td>
<td>2.26</td>
<td>130</td>
</tr>
<tr>
<td>nutmeg oil</td>
<td>mixture</td>
<td>2.90</td>
<td>164</td>
</tr>
<tr>
<td>methyl isoeugenol</td>
<td>93-16-3</td>
<td>2.95</td>
<td>164</td>
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<tr>
<td>gamma undecalactone</td>
<td>104-67-6</td>
<td>3.06</td>
<td>184</td>
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<tr>
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<td></td>
<td></td>
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<tr>
<td>------------</td>
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<td>------</td>
</tr>
<tr>
<td>alpha terpineol</td>
<td>98-55-5</td>
<td>3.33</td>
<td>154</td>
</tr>
<tr>
<td>chlorocyclohexane (CCH)</td>
<td>542-18-7</td>
<td>3.36</td>
<td>118</td>
</tr>
<tr>
<td>linalool</td>
<td>78-70-6</td>
<td>3.38</td>
<td>154</td>
</tr>
<tr>
<td>citral</td>
<td>5392-40-5</td>
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<td>geraniol</td>
<td>106-24-1</td>
<td>3.47</td>
<td>154</td>
</tr>
<tr>
<td>citronellol</td>
<td>106-22-9</td>
<td>3.56</td>
<td>154</td>
</tr>
<tr>
<td>p-cymene</td>
<td>99-87-6</td>
<td>4.10</td>
<td>134</td>
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<tr>
<td>limonene</td>
<td>138-86-3</td>
<td>4.83</td>
<td>136</td>
</tr>
</tbody>
</table>

[0047] Examples of guests having a relatively large positive log (P) value (e.g., greater than about 2) include, but are not limited to, citral, linalool, alpha terpineol, and combinations thereof. Examples of guests having a relatively small positive log (P) value (e.g., less than about 1 but greater than zero) include, but are not limited to, dimethyl sulfide, furaneol, ethyl maltol, aspartame, and combinations thereof. Examples of guests having a relatively large negative log (P) value (e.g., less than about -2) include, but are not limited to, creatine, proline, and combinations thereof. Examples of guests having a relatively small negative log (P) value (e.g., less than 0 but greater than about -2) include, but are not limited to, diacetyl, acetaldehyde, maltol, and combinations thereof.

[0048] Log (P) values are significant in many aspects of food and flavor chemistry. A table of log (P) values is provided above. The log (P) values of guests can be important to many aspects of an end product (e.g., foods and flavors). Generally, organic guest molecules having a positive log (P) can be successfully encapsulated in cyclodextrin. In a mixture comprising several guests, competition can exist, and log (P) values can be useful in determining which guests will be more likely to be successfully encapsulated. Maltol and furaneol are examples of two guests that have similar flavor characteristics (i.e., sweet attributes), but which would have different levels of success in cyclodextrin encapsulation because of their differing log (P) values. Log (P) values may be important in food products with a high aqueous content or environment. Compounds with significant and positive log (P) values are, by definition, the least soluble and therefore the first to migrate, separate, and then be exposed to change in the package. The high log (P) value, however, may make them effectively scavenged and protected by addition cyclodextrin in the product.

[0049] As mentioned above, the cyclodextrin used with the present invention can include α-cyclodextrin, β-cyclodextrin, γ-cyclodextrin, and combinations thereof. In embodiments in which a more hydrophilic guest (i.e., having a smaller log (P) value) is used, α-cyclodextrin may be used (i.e., alone or in combination with another type of cyclodextrin) to improve the encapsulation of the guest in cyclodextrin. For example, a combination of α-cyclodextrin and
β-cyclodextrin can be used in embodiments employing relatively hydrophilic guests to improve the formation of a cyclodextrin inclusion complex.

[0050] As used herein and in the appended claims, the term “cyclodextrin inclusion complex” refers to a complex that is formed by encapsulating at least a portion of one or more guest molecules with one or more cyclodextrin molecules (encapsulation on a molecular level) by capturing and holding a guest molecule within the three dimensional cavity. The guest can be held in position by van der Waal forces within the cavity by at least one of hydrogen bonding and hydrophilic-hydrophobic interactions. The guest can be released from the cavity when the cyclodextrin inclusion complex is dissolved in water. Cyclodextrin inclusion complexes are also referred to herein as “guest-cyclodextrin complexes.” Because the cavity of cyclodextrin is hydrophobic relative to its exterior, guests having positive log (P) values (particularly, relatively large positive log (P) values) will encapsulate easily in cyclodextrin and form stable cyclodextrin inclusion complexes in an aqueous environment, because the guest will thermodynamically prefer the cyclodextrin cavity to the aqueous environment. In some embodiments, when it is desired to complex more than one guest, each guest can be encapsulated separately to maximize the efficiency of encapsulating the guest of interest.

[0051] As used herein and in the appended claims, the term “uncomplexed cyclodextrin” generally refers to cyclodextrin that is substantially free of a guest and has not formed a cyclodextrin inclusion complex. Cyclodextrin that is “substantially free of a guest” generally refers to a source of cyclodextrin that includes a large fraction of cyclodextrin that does not include a guest in its cavity.

[0052] As used herein and in the appended claims, the term “hydrocolloid” generally refers to a substance that forms a gel with water. A hydrocolloid can include, without limitation, at least one of xanthan gum, pectin, gum arabic (or gum acacia), tragacanth, guar, carrageenan, locust bean, and combinations thereof.

[0053] As used herein and in the appended claims, the term “pectin” refers to a hydrocolloidal polysaccharide that can occur in plant tissues (e.g., in ripe fruits and vegetables). Pectin can include, without limitation, at least one of beet pectin, fruit pectin (e.g., from citrus peels), and combinations thereof. The pectin employed can be of varying molecular weight.
Cyclodextrin inclusion complexes of the present invention can be used in a variety of applications or end products, including, without limitation, at least one of foods (e.g., beverages, soft drinks, salad dressings, popcorn, cereal, coffee, cookies, brownies, other desserts, other baked goods, seasonings, etc.), chewing gums, dentifrices, candy, flavorings, fragrances, pharmaceuticals, nutraceuticals, cosmetics, agricultural applications (e.g., herbicides, pesticides, etc.), photographic emulsions, and combinations thereof. In some embodiments, cyclodextrin inclusion complexes can be used as intermediate isolation matrices to be further processed, isolated and dried (e.g., as used with waste streams).

Cyclodextrin inclusion complexes can be used to enhance the stability of the guest, convert it to a free flowing powder, or otherwise modify its solubility, delivery or performance. The amount of the guest molecule that can be encapsulated is directly related to the molecular weight of the guest molecule. In some embodiments, one mole of cyclodextrin encapsulates one mole of guest. According to this mole ratio, and by way of example only, in embodiments employing diacetyl (molecular weight of 86 Daltons) as the guest, and β-cyclodextrin (molecular weight 1135 Daltons), the maximum theoretical retention is \((86/(86+1135)) \times 100 = 7.04\text{ wt }\%\).

In some embodiments, cyclodextrin can self-assemble in solution to form a nano-structure, such as the nano-structure 20 illustrated in FIG. 2, that can incorporate three moles of a guest molecule to two moles of cyclodextrin molecules. For example, in embodiments employing diacetyl as the guest, a 10.21 wt % retention of diacetyl is possible, and in embodiments employing citral as the guest, a wt % retention of citral of at least 10 wt % is possible (e.g., 10-14 wt % retention). FIG. 4 shows a schematic illustration of a nano-structure than can form between three moles of diacetyl molecules and two moles of cyclodextrin molecules. FIG. 6 shows a schematic illustration of a nano-structure than can form between three moles of citral molecules and two moles of cyclodextrin molecules. Other complex enhancing agents, such as pectin, can aid in the self-assembly process, and can maintain the 3:2 mole ratio of guest:cyclodextrin throughout drying. In some embodiments, because of the self-assembly of cyclodextrin molecules into nano-structures, a 5:3 mole ratio of guest:cyclodextrin is possible.

Cyclodextrin inclusion complexes form in solution. The drying process temporarily locks at least a portion of the guest in the cavity of the cyclodextrin and can produce a dry, free flowing powder comprising the cyclodextrin inclusion complex.
[0058] The hydrophobic (water insoluble) nature of the cyclodextrin cavity will preferentially trap like (hydrophobic) guests most easily at the expense of more water-soluble (hydrophilic) guests. This phenomenon can result in an imbalance of components as compared to typical spray drying and a poor overall yield.

[0059] In some embodiments of the present invention, the competition between hydrophilic and hydrophobic effects is avoided by selecting key ingredients to encapsulate separately. For example, in the case of butter flavors, fatty acids and lactones form cyclodextrin inclusion complexes more easily than diacetyl. However, these compounds are not the key character impact compounds associated with butter, and they will reduce the overall yield of diacetyl and other water soluble and volatile ingredients. In some embodiments, the key ingredient in butter flavor (i.e., diacetyl) is maximized to produce a high impact, more stable, and more economical product. By way of further example, in the case of lemon flavors, most lemon flavor components will encapsulate equally well in cyclodextrin. However, terpenes (a component of lemon flavor) have little flavor value, and yet make up approximately 90% of a lemon flavor mixture, whereas citral is a key flavor ingredient for lemon flavor. In some embodiments, citral is encapsulated alone. By selecting key ingredients (e.g., diacetyl, citral, etc.) to encapsulate separately, the complexity of the starting material is reduced, allowing optimization of engineering steps and process economics.

[0060] In some embodiments, the inclusion process for forming the cyclodextrin inclusion complex is driven to completion by adding a molar excess of the guest. For example, in some embodiments (e.g., when the guest used is diacetyl), the guest can be combined with the cyclodextrin in a 3:1 molar ratio of guest: cyclodextrin. In some embodiments, using a molar excess of guest in forming the complex not only drives the formation of the cyclodextrin inclusion complex, but can also make up for any loss of guest in the process, e.g., in embodiments employing a volatile guest.

[0061] In some embodiments, the viscosity of the suspension, emulsion or mixture formed by mixing the cyclodextrin and guest molecules in a solvent is controlled, and compatibility with common spray drying technology is maintained without other adjustments, such as increasing the solids content. An emulsifier (e.g., a thickener, gelling agent, polysaccharide, hydrocolloid) can be added to maintain intimate contact between the cyclodextrin and the guest, and to aid in the inclusion process. Particularly, low molecular
weight hydrocolloids can be used. One preferred hydrocolloid is pectin. Emulsifiers can aid in the inclusion process without requiring the use of high heat or co-solvents (e.g., ethanol, acetone, isopropanol, etc.) to increase solubility.

[0062] In some embodiments, the water content of the suspension, emulsion or mixture is reduced to essentially force the guest to behave as a hydrophobic compound. This process can increase the retention of even relatively hydrophilic guests, such as acetaldehyde, diacetyl, dimethyl sulfide, etc. Reducing the water content can also maximize the throughput through the spray dryer and reduce the opportunity of volatile guests blowing off in the process, which can reduce overall yield.

[0063] In some embodiments of the present invention, a cyclodextrin inclusion complex can be formed by the following process, which may include some or all of the following steps:

[0064] (1) Dry blending cyclodextrin and an emulsifier (e.g., pectin);

[0065] (2) Combining the dry blend of cyclodextrin and the emulsifier with a solvent such as water in a reactor, and agitating;

[0066] (3) Adding the guest and stirring (e.g., for approximately 5 to 8 hours);

[0067] (4) Cooling the reactor (e.g., turning on a cooling jacket);

[0068] (5) Stirring the mixture (e.g., for approximately 12 to 36 hours);

[0069] (6) Emulsifying (e.g., with an in-tank lightning mixer or high shear drop-in mixer); and

[0070] (7) Drying the cyclodextrin inclusion complex to form a powder.

[0071] These steps need not necessarily be performed in the order listed. In addition, the above process has proved to be very robust in that the process can be performed using variations in temperature, time of mixing, and other process parameters.

[0072] In some embodiments, step 1 in the process described above can be accomplished using an in-tank mixer in the reactor to which the hot water will be added in step 2. For example, in some embodiments, the process above is accomplished using a 1000 gallon
reactor equipped with a jacket for temperature control and an inline high shear mixer, and the reactor is directly connected to a spray drier. In some embodiments, the cyclodextrin and emulsifier can be dry blended in a separate apparatus (e.g., a ribbon blender, etc.) and then added to the reactor in which the remainder of the above process is completed.

[0073] A variety of weight percentages of an emulsifier to cyclodextrin can be used, including, without limitation, an emulsifier:cyclodextrin weight percentage of at least about 0.5 %, particularly, at least about 1 %, and more particularly, at least about 2 %. In addition, an emulsifier:cyclodextrin weight percentage of less than about 10 % can be used, particularly, less than about 6 %, and more particularly, less than about 4 %.

[0074] Step 2 in the process described above can be accomplished in a reactor that is jacketed for heating, cooling, or both. In some embodiments, the combining and agitating can be performed at room temperature. In some embodiments, the combining and agitating can be performed at a temperature greater than room temperature. The reactor size can be dependent on the production size. For example, a 100 gallon reactor can be used. The reactor can include a paddle agitator and a condenser unit. In some embodiments, step 1 is completed in the reactor, and in step 2, hot deionized water is added to the dry blend of cyclodextrin and pectin in the same reactor.

[0075] Step 3 can be accomplished in a sealed reactor, or the reactor can be temporarily exposed to the environment while the guest is added, and the reactor can be re-sealed after the addition of the guest. Heat can be added when the guest is added and during the stirring of step 3. For example, in some embodiments, the mixture is heated to about 55-60 degrees C.

[0076] Step 4 can be accomplished using a coolant system that includes a cooling jacket. For example, the reactor can be cooled with a propylene glycol coolant and a cooling jacket.

[0077] The agitating in step 2, the stirring in step 3, and the stirring in step 5 can be accomplished by at least one of shaking, stirring, tumbling, and combinations thereof.

[0078] In step 6, the mixture of the cyclodextrin, emulsifier, water and guest can be emulsified using at least one of a high shear mixer (e.g., a ROSS-brand mixer (e.g., at 10,000 RPM for 90 seconds), or a SILVERSTON-brand mixer (e.g., at 10,000 RPM for 5 minutes)),
a lightning mixer, or simple mixing followed by transfer to a homogenization pump that is part of a spray dryer, and combinations thereof.

[0079] Step 7 in the process described above can be accomplished by at least one of air drying, vacuum drying, spray drying (e.g., with a nozzle spray drier, a spinning disc spray drier, etc.), oven drying, and combinations thereof.

[0080] The process outlined above can be used to provide cyclodextrin inclusion complexes with a variety of guests for a variety of applications or end products. For example, some of the embodiments of the present invention provide a cyclodextrin inclusion complex with a guest comprising diacetyl, which can be used for various food products as a butter flavoring (e.g., in microwave popcorn, baked goods, etc.). In addition, some embodiments provide a cyclodextrin inclusion complex with a guest comprising citral, which can be used for acid stable beverages. Furthermore, some embodiments provide a cyclodextrin inclusion complex with a combination of flavor molecules as the guest that can mimic the butter flavoring of diacetyl. For example, the cyclodextrin inclusion complex can alternatively include at least one of dimethyl sulfide (a volatile sulfur compound), proline (an amino acid) and furaneol (a sweetness enhancer) as the guest. This diacetyl-free cyclodextrin inclusion complex can be used to provide a butter flavoring to food products, such as those described above. For cyclodextrin inclusion complexes that can be used in microwavable products, the very close association of guests enhances, for example, maillard and browning reactions, which can generate new and distinct aromas.

[0081] As mentioned above, the encapsulation of the guest molecule can provide isolation of the guest molecule from interaction and reaction with other components that would cause off note formation; and stabilization of the guest molecule against degradation (e.g., hydrolysis, oxidation, etc.). Stabilization of the guest against degradation can improve or enhance the desired effect or function (e.g., taste, odor, etc.) of a resulting commercial product that includes the encapsulated guest.

[0082] Many guests can degrade and create off-notes that can detract from a main or desired effect or function. For example, many flavors or olfactants can degrade and create off-note flavors or odors that can detract from the desired flavor or odor of a commercial product. Guests can also be degraded by means of photo-oxidation. By way of example, FIG. 7 shows the degradation mechanism of citral. The rate of degradation of the guest (i.e.,
the rate of formation of off-note(s)) is generally governed by the following generic kinetic rate equation:

\[
Rate \approx \frac{[\text{offnote}]^x}{[\text{guest}]^y \cdot [RC]^z}
\]

where [guest] refers to the molar concentration of guest in a solution, [RC] refers to the molar concentration of a reactive compound in a solution responsible for reacting with and degrading the guest (e.g., an acid), and [offnote] refers to the molar concentration of off-notes formed. The powers x, y and z represent kinetic order, depending on the reaction that occurs between a guest of interest and the corresponding reactive compound(s) present in solution to produce off-notes. Thus, the rate of degradation of the guest is proportional to the product of the molar concentrations of the guest and any reactive compounds, raised to a power determined by the kinetic order of the reaction.

[0083] For example, the following equation represents the degradation of citral in an acidic solution to form off-notes at any given temperature and concentration:

\[
\frac{[\text{offnote}]^x}{[\text{citral}]^y \times [H^+]^z} = \kappa
\]

[0084] where, based on the degradation mechanism of citral shown in FIG. 7,

\[
[\text{offnote}] = \sum \kappa[p-\text{menthadien}-8-\text{ol}]^{p_\text{p}} + \kappa[p-\text{cymene}-8-\text{ol}]^{p_\text{p}} + ...
\]

\[
+ \kappa[p-\text{methylacetophenone}]^{p_\text{p}}
\]

[0085] Any of the above-mentioned guests can be protected and stabilized in this manner. For example, cyclodextrin can be used to protect and/or stabilize a variety of guest molecules to enhance the desired effect or function of a product, including, but not limited to, the following guest molecules: citral, benzaldehyde, alpha terpineol, vanillin, aspartame, neotame, acetaldehyde, creatine, and combinations thereof. An example of this phenomenon is described in Example 21 and shown in Table 2 and FIGS. 12-15. Specifically, this phenomenon was demonstrated by comparing samples 1BH3, 1BH4, and 1BH5, all with added citral; and samples 3FH3, 3FH4 and 3FH5, all with water-soluble rosemary (WSR) with the BCD samples. Mentha 1,5-dien-8-ol was converted to p-cymene-8-ol in the 1BH and 3FH samples, and it was observed that the of concentration of mentha 1,5-dien-8-ol, for example, decreased, and the concentration of p-cymene-8-ol increased. However, this did not occur in the BCD samples.
A “guest stabilizing system” can refer to any system which stabilizes a guest (or guests) of interest and protects the guest from degradation. The present invention includes several embodiments of guest stabilizing systems, as will be described in greater detail below.

Citral (log (P) = 3.45) is a citrus or lemon flavor that can be used in various applications, such as acidic beverages. Acidic beverages can include, but are not limited to lemonade, 7UP® lemon-lime flavored soft drink (registered trademark of Dr Pepper/Seven-Up, Inc.), SPRITE® lemon-lime flavored soft drink (registered trademark of The Coca-Cola Company, Atlanta, GA), SIERRA MIST® lemon-lime flavored soft drink (registered trademark of Pepsico, Purchase, NY), tea (e.g., LIPTON® and BRISK®, registered trademarks of Lipton), alcoholic beverages, and combinations thereof. Alpha terpineol (log (P) = 3.33) is a lime flavor that can be used in similar products as those listed above with respect to citral.

Benzaldehyde (log (P) = 1.48) is a cherry flavor that can be used in a variety of applications, including acidic beverages. An example of an acidic beverage that can be flavored with benzaldehyde includes, but is not limited to CHERRY COKE® cherry-cola flavored soft drink (registered trademark of The Coca-Cola Company, Atlanta, GA).

Vanillin (log (P) = 1.05) is a vanilla flavor that can be used in a variety of applications, including, but not limited to, vanilla-flavored beverages, baked goods, etc., and combinations thereof.

Aspartame (log (P) = 0.07) is a non-sucrose sweetener that can be used in a variety of diet foods and beverages, including, but not limited to, diet soft drinks. Neotame is also a non-sucrose sweetener that can be used in diet foods and beverages.

Acetaldehyde (log (P) = -0.17) is an apple flavor that can be used in a variety of applications, including, but not limited to, foods, beverages, candies, etc., and combinations thereof.

Creatine (log (P) = -3.72) is a nutraceutical agent that can be used in a variety of applications, including, but not limited to, nutraceutical formulations. Examples of nutraceutical formulations include, but are not limited to, powder formulations that can be combined with milk, water or another liquid, and combinations thereof.
The protection and/or stabilization of a guest can be accomplished by providing an excess of cyclodextrin (e.g., uncomplexed cyclodextrin) to the final powder product of the cyclodextrin inclusion complex. In other words, dry blending uncomplexed cyclodextrin with the dry powder that is formed in step 7 of the process described above can produce a dry, free-flowing powder (referred to herein as “guest-cyclodextrin/cyclodextrin blend”) with a desired amount of guest and cyclodextrin (i.e., including excess uncomplexed cyclodextrin) that can be used in a variety of applications or commercial products. The proportion of a guest-cyclodextrin complex in a guest-cyclodextrin/cyclodextrin blend depends on the potency (e.g., flavor value if the guest is a flavor) of the guest, and the desired effect in the final product. The excess uncomplexed cyclodextrin in the guest-cyclodextrin/cyclodextrin blend acts to protect and/or stabilize the guest (including from photo-oxidation) when the guest-cyclodextrin/cyclodextrin blend is added to, or used in, a product of interest. For example, a flavor powder including a guest-cyclodextrin/cyclodextrin blend can be effective in decreasing the rate of degradation of the flavor in beverage applications while providing an appropriate flavor profile to that beverage.

A variety of systems can be employed to add excess uncomplexed cyclodextrin for protection and/or stabilization of the guest. In some embodiments, the guest-cyclodextrin/cyclodextrin blend is added as a dry powder to a final product (e.g., in a weight percentage of ranging from about 0.05 wt % to about 0.50 wt % of guest-cyclodextrin/cyclodextrin blend to product, particularly, from about 0.15 wt % to about 0.30 wt %, and more particularly, about 0.2 wt %).

In some embodiments, if solubility of the powder permits, the guest-cyclodextrin/cyclodextrin blend is added to a liquid product, emulsion or emulsion-compatible product (e.g., a flavor emulsion), which is then added to the final product (e.g., in a weight percentage of ranging from about 0.05 wt % to about 0.50 wt % of guest-cyclodextrin/cyclodextrin blend to product, particularly, from about 0.15 wt % to about 0.30 wt %, and more particularly, about 0.2 wt %, such that the weight percentage of the guest achieves a desired flavor level in the final product. In some embodiments, the excess uncomplexed cyclodextrin can be added to the composition comprising the cyclodextrin inclusion complex that is formed in step 6, thereby skipping step 7 (the drying step) and forming a stable emulsion or emulsion-compatible product that can be added to the final product in the range of weight percentages listed above. The emulsion-compatible product
can be added to another final product (e.g., a beverage, a salad dressing, a dessert, and/or a seasoning, etc.). In some embodiments, the emulsion-compatible product can be provided in the form of, or be added to, a syrup or a coating mix, which can be sprayed onto a substrate as a stable coating (e.g., a flavor emulsion sprayed onto cereal, a dessert, a seasoning, nutritional bars, and/or snack foods such as pretzels, chips, etc.).

[0097] Providing the cyclodextrin inclusion complex in a liquid form can, but need not, have several advantages. First, the liquid form can be more familiar and user friendly for beverage customers who are accustomed to adding flavor compositions to their beverages in the form of a liquid concentrate. Second, the liquid form can be easily sprayed onto dry food products including those listed above to achieve an evenly-distributed and stable coating that includes the flavor composition. Unlike existing spray-on applications, the sprayed-on flavor composition comprising the cyclodextrin inclusion complex would not require the typical volatile solvents or additional coatings or protective layers to maintain the flavor composition on that dry substrate. Third, cyclodextrin can extend the shelf-life of such food products, because cyclodextrin is not hygroscopic, and thus will not lead to staleness, flatness, or reduced freshness of the base food product or beverage. Fourth, drying processes can be costly, and some guest (e.g., free guest or guest present in a cyclodextrin inclusion complex) can be lost during drying, which can make the drying step difficult to optimize and perform economically. For these reasons and others that are not specifically mentioned here, providing the cyclodextrin inclusion complex in a liquid form in some embodiments can be beneficial. The emulsion form of the cyclodextrin inclusion complex can be added to a final product (e.g., a beverage or food product) to impart the appropriate guest profile (e.g., flavor profile) to the final product, while ensuring that the cyclodextrin in the final product is within the legal limits for that given product (e.g., no greater than 0.2 wt % of some products, or no greater than 2 wt % of some products).

[0098] Improving the manufacturability of a cyclodextrin inclusion complex, including the formation of a liquid or emulsion form comprising the cyclodextrin inclusion complex, is the subject matter of co-pending U.S. Patent Application Serial No. ____________, filed on the same day herewith, the entire contents of which are incorporated herein by reference.

[0099] Because there is an equilibrium that is established between encapsulation of the guest with the cyclodextrin and free (or uncomplexed) guest molecules and cyclodextrin molecules, adding excess uncomplexed cyclodextrin to a system can force the equilibrium to
encapsulation of the guest. As described above, decreasing the amount of free guest in a
system decreases the rate of degradation of the guest and the rate of formation of off-notes.
In addition, especially in beverage or other liquid applications, the guest may prefer,
thermodynamically and/or kinetically, to be encapsulated in cyclodextrin over being
unencapsulated. This phenomenon can be exaggerated by adding excess uncomplexed
cyclodextrin. It is also possible that the small amount of off-note molecules that are formed,
if any, may become encapsulated in cyclodextrin, and become essentially “masked” from the
final product. In other words, in some embodiments, because of the chemical makeup of the
off-notes, the off-notes may bind very stably with cyclodextrin, which can lead to a masking
effect of any off-notes that may be formed. Thus, in some embodiments, the excess
uncomplexed cyclodextrin may act as a scavenger to mask or isolate other water-miscible
components in a system that may interfere with desired effects or functions of a product.

[00100] FIG. 7A illustrates a three-phase model that represents a guest-cyclodextrin-
solvent system. The guest used in FIG. 7A is citral, and the solvent used is water, but it
should be understood that citral and water are shown in FIG. 7A for the purpose of
illustration only. One of ordinary skill in the art, however, will understand that the three-
phase model shown in FIG. 7A can be used to represent a wide variety of guests and solvents.
Additional information regarding a three-phase model similar to the one illustrated in FIG. 7
can be found in Lantz et al., “Use of the three-phase model and headspace analysis for the
facile determination of all partition/association constants for highly volatile solute-
cyclodextrin-water systems,” Anal Bioanal Chem (2005) 383: 160-166, which is incorporated
herein by reference.

[00101] This three-phase model can be used to explain the phenomena that occur (1)
during formation of the cyclodextrin inclusion complex, (2) in a beverage application of the
cyclodextrin inclusion complex, and/or (3) in a flavor emulsion. The flavor emulsion can
include, for example, the slurry formed in step 5 or 6 in the process described above prior to
or without drying, or a slurry formed by resuspending a dry powder comprising a
cyclodextrin inclusion complex in a solvent. Such a flavor emulsion can be added to a
beverage application (e.g., as a concentrate), or sprayed onto a substrate, as described above.

[00102] As shown in FIG. 7A, there are three phases in which the guest can be present,
namely, the gaseous phase, the aqueous phase, and the cyclodextrin phase (also sometimes
referred to as a “pseudophase”). Three equilibria, and their associated equilibrium constants (i.e., $K_H$, $K_{P1}$ and $K_{P2}$) are used to describe the presence of the guest in these three phases:

$$S_{(g)} \xrightarrow{K_H} S_{(aq)}; \quad K_H = \frac{C_{aq}}{P_s} \quad \text{(based on Henry’s Law: } K_H = \frac{C_s}{P_s}) \quad (1)$$

$$S_{(g)} \xrightarrow{K_{P1}} S_{(CD)}; \quad K_{P1} = \frac{C_{CD}^S}{P_s} \quad (2)$$

$$S_{(aq)} \xrightarrow{K_{P2}} S_{(CD)}; \quad K_{P2} = \frac{C_{CD}^{aq}}{C_{aq}^S} \quad (3)$$

$$K_H = \frac{K_{P1}}{K_{P2}} \quad (4)$$

wherein “S” represents the solute (i.e., the guest) of the system in the corresponding phase of the system which is denoted in the subscript, “g” represents the gaseous phase, “aq” represents the aqueous phase, “CD” represents the cyclodextrin phase, “$C_s$” represents the concentration of the solute in the corresponding phase (i.e., aq or CD, denoted in the superscript), and “$P_s$” represents the partial pressure of the solute in the gaseous phase.

To account for all of the guest in the three-phase system shown in FIG. 7A, it follows that the total number of moles of guest ($n_s^{total}$) can be represented by the following equation:

$$n_s^{total} = n_s^g + n_s^{aq} + n_s^{CD}. \quad (5)$$

To account for any loss of the guest in a product (e.g., a beverage or flavor emulsion) at steady state, the total number of moles of guest available for sensation ($n_s^{taste}$, e.g., for taste in a beverage or flavor emulsion) can be represented by the following equation:

$$n_s^{taste} = n_s^g + n_s^{aq} + n_s^{CD} - f(P) \quad (6)$$

wherein $f(P)$ is a partitioning function that represents any migration (or loss) of the guest, for example, through a barrier or container (e.g., a plastic bottle formed of
polyethylene or polyethylene terephthalate (PET)) in which the beverage of flavor emulsion is contained.

[00113] For guests having a large positive log (P) value, encapsulation of the guest in cyclodextrin will be thermodynamically favored (i.e., $K_{P1}$ and $K_{P2}$ will be greater than 1), and the following relationship will occur:

$$n_{s}^{CD} \gg n_{s}^{aq} > n_{s}^{g} > f_{(P)}$$  \hspace{1cm} (7)

[00115] such that the majority of the guest present in the system will be in the form of a cyclodextrin inclusion complex. Not only will the amount of free guest in the aqueous and gaseous phases be minimal, but also the migration of guest through the barrier or container will be minimized. Accordingly, the majority of the guest available for sensation will be present in the cyclodextrin phase, and the total number of moles of guest available for sensation ($n_{s}^{true}$) can be approximated as follows:

$$n_{s}^{true} \approx n_{s}^{CD}$$  \hspace{1cm} (8)

[00117] The formation of the cyclodextrin inclusion complex in solution between the guest and the cyclodextrin can be more completely represented by the following equation:

$$S_{(aq)} + CD_{(aq)} \xrightarrow{K_{P1}} S \cdot CD_{(aq)}; \quad K_{P2} = \frac{[S \cdot CD]_{(aq)}}{[S]_{(aq)}[CD]_{(aq)}}$$  \hspace{1cm} (9)

[00119] Empirically, the data supporting the present invention has shown that the log (P) value of the guest can be a factor in the formation and stability of the cyclodextrin inclusion complex. That is, empirical data has shown that the equilibrium shown in equation 9 above is driven to the right by the net energy loss accompanied by the encapsulation process in solution, and that the equilibrium can be at least partially predicted by the log (P) value of the guest of interest. It has been found that log (P) values of the guests can be a factor in end products with a high aqueous content or environment. For example, guests with relatively large positive log (P) values are typically the least water-soluble and can migrate and separate from an end product, and can be susceptible to a change in the environment within a package. However, the relatively large log (P) value can make such guests effectively scavenged and protected by the addition of cyclodextrin to the end product. In other words, in some
embodiments, the guests that have traditionally been the most difficult to stabilize can be easy to stabilize using the methods of the present invention.

[00120] To account for the effect of the log \((P)\) value of the guest, the equilibrium constant \((K_{P2}')\) that represents the stability of the guest in a system can be represented by the following equation:

\[
K_{P2}' = \log(P) \frac{[S \bullet CD]_{aq}}{[S]_{aq} [CD]_{aq}}
\]  (10)

[00121] wherein log \((P)\) is the log \((P)\) value for the guest \((S)\) of interest in the system.

Equation 10 establishes a model that takes into account a guest's log \((P)\) value. Equation 10 shows how a thermodynamically stable system can result from first forming a cyclodextrin inclusion complex with a guest having a relatively large positive log \((P)\) value. For example, in some embodiments, a stable system (i.e., a guest stabilizing system) can be formed using a guest having a positive log \((P)\) value. In some embodiments, a stable system can be formed using a guest having a log \((P)\) value of at least about +1. In some embodiments, a stable system can be formed using a guest having a log \((P)\) value of at least about +2. In some embodiments, a stable system can be formed using a guest having a log \((P)\) value of at least about +3. Furthermore, one can see how a thermodynamically stable system can result not only by using a guest having a positive log \((P)\) value, but also by adding additional, uncomplexed cyclodextrin to that cyclodextrin inclusion complex to further favor the right side of the equilibrium shown in equation 9 above, and to increase the ratio of complexed guest to free, or uncomplexed, guest to further stabilize the guest from degradation.

[00123] While log\((P)\) values can be good empirical indicators and are available from several references, another important criteria is the binding constant for a particular guest (i.e., once a complex forms, how strongly is the guest bound in the cyclodextrin cavity). Unfortunately, the binding constant for a guest is determined experimentally. In the case of limonene and citral, for example, citral can form a much stronger complex, even though the log\((P)\) values are similar. As a result, even in the presence of high limonene concentrations, citral is preferentially protected until consumption, because of its higher binding constant. This is an unexpected benefit and is not directly predicted from the current scientific literature.
[00124] In some embodiments of the present invention, as supported by equation 10, the
guest is added to a product, system or application (e.g., a beverage) in an uncomplexed form,
and uncomplexed cyclodextrin is added to that same product, system or application. As
suggested by equation 10, the stability of the guest in such a system (and the guest's
protection from degradation) will be at least partially dependent on the log (P) value of the
guest. For example, a guest can be added to a system to obtain a desired concentration of
guest in the system, and uncomplexed cyclodextrin can be added to the system to stabilize the
guest and protect the guest from degradation. In some embodiments, the concentration of the
guest in the system is at least about 1 ppm, particularly, at least about 5 ppm, and more
particularly, at least about 10 ppm. In some embodiments, the concentration of the guest in
the system is less than about 200 ppm, particularly, less than about 150 ppm, and more
particularly, less than about 100 ppm. In some embodiments, the overall concentration of
citrus components, for example, can exceed 1000 ppm (e.g., when limonene is present).
However, this has not proved an impediment to the stabilization/protection scheme of the
present invention.

[00125] In some embodiments, the cyclodextrin is added to the system in a molar ratio of
cyclodextrin:guest of greater than 1:1. As shown in equation 10, stabilization of the guest in
the system by cyclodextrin can be predicted by the log (P) value of the guest. In some
embodiments, the guest chosen has a positive log (P) value. In some embodiments, the guest
has a log (P) value of greater than about +1. In some embodiments, the guest has a log (P)
value of greater than about +2. In some embodiments, the guest has a log (P) value of greater
than about +3.

[00126] Whether the product, system or application includes a free/uncomplexed guest, or
a cyclodextrin-encapsulated guest, the guest can be added to achieve a desired concentration
of the guest in the final product, system or application, and the uncomplexed cyclodextrin can
be added to the product, system or application to maintain the total weight percentage of
cyclodextrin within legal limits. For example, in some embodiments, the weight percentage
of cyclodextrin to the system ranges from about 0.05 wt % to about 0.50 wt %, particularly,
from about 0.15 wt % to about 0.30 wt %, and more particularly, about 0.2 wt %. In some
embodiments, the uncomplexed cyclodextrin is combined with the guest and then added to
the system. In some embodiments, the uncomplexed cyclodextrin is added directly to the
system separately from the guest. Example 20 illustrates the stabilizing effects of
uncomplexed α-cyclodextrin or β-cyclodextrin added to a solution comprising citral. As explained in Example 20, the citral is protected from degradation and off-note formation is inhibited. Equation 10 suggests that the stabilizing effect of citral can be at least partially due to the relatively large log (P) value of citral (i.e., 3.45).

[00127] By taking into account the log (P) of the guest, it is possible to predict the stability of the guest in a system that comprises cyclodextrin. By exploiting the thermodynamics of the complexation in solution, a protective and stable environment can be formed for the guest, and this can be driven further by the addition of excess uncomplexed cyclodextrin. Release characteristics of a guest from the cyclodextrin can be governed by \( K_H \), the guest’s air/water partition coefficient. \( K_H \) can be large compared to log (P) if the system comprising the cyclodextrin inclusion complex is placed in a non-equilibrium situation, such as the mouth. One of ordinary skill in the art will understand that more than one guest can be present in a system, and that similar equations and relationships can be applied to each guest of the system.

[00128] In embodiments in which the guest is a flavor and the commercial product is a beverage (or other liquid), the cyclodextrin can protect the flavor from degradation in the liquid product, but can release the flavor from encapsulation when the liquid is allowed to contact taste buds in the mouth. Thus, the desired flavor or essence of the product can be maintained, and the appropriate flavor or essence profile can be delivered, while preventing degradation of that flavor or essence, and while supplying a legally allowable amount of cyclodextrin to the beverage. This phenomenon is further described in Examples 21-22 and further illustrated in Tables 2 and 3 and FIGS. 7-10.

[00129] Various features and aspects of the invention are set forth in the following examples, which are intended to be illustrative and not limiting. All of the examples were performed at atmospheric pressure, unless stated otherwise. Examples 1-19A, 20-23, 25, 28, 29, 31, 34-37 are working examples. Examples 19B, 24A, 24B, 26, 27, 30, 32 and 33 are prophetic examples.
EXAMPLE 1: CYCLODEXTRIN INCLUSION COMPLEX WITH β-CYCLODEXTRIN AND DIACETYL, PECTIN AS AN EMULSIFIER, AND PROCESS FOR FORMING SAME

[00130] At atmospheric pressure, in a 100 gallon reactor, 49895.1600 g (110.02 lb) of β-cyclodextrin was dry blended with 997.9 g (2.20 lb) of beet pectin (2 wt % of pectin: β-cyclodextrin; XPQ EMP 5 beet pectin available from Degussa-France) to form a dry blend. The 100 gallon reactor was jacketed for heating and cooling, included a paddle agitator, and included a condenser unit. The reactor was supplied with a propylene glycol coolant at approximately 40 °F (4.5 °C). The propylene glycol coolant system is initially turned off, and the jacket acts somewhat as an insulator for the reactor. 124737.9 g (275.05 lb) of hot deionized water was added to the dry blend of β-cyclodextrin and pectin. The water had a temperature of approximately 118 °F (48 °C). The mixture was stirred for approximately 30 min. using the paddle agitator of the reactor. The reactor was then temporarily opened, and 11226.4110 g (24.75 lb) of diacetyl was added (as used hereinafter, “diacetyl” in the examples refers to diacetyl purchased from Aldrich Chemical, Milwaukee, WI). The reactor was resealed, and the resulting mixture was stirred for 8 hours with no added heat. Then, the reactor jacket was connected to the propylene glycol coolant system. The coolant was turned on to approximately 40 °F (4.5 °C), and the mixture was stirred for approximately 36 hours. The mixture was then emulsified using a high shear tank mixer, such as what is typically used in spray dry operations. The mixture was then spray dried on a nozzle dryer having an inlet temperature of approximately 410 °F (210 °C) and an outlet temperature of approximately 221 °F (105 °C). A percent retention of 12.59 wt % of diacetyl in the cyclodextrin inclusion complex was achieved. The moisture content was measured at 4.0 %. The cyclodextrin inclusion complex included less than 0.3 % surface diacetyl, and the particle size of the cyclodextrin inclusion complex was measured as 99.7 % through an 80 mesh screen. Those skilled in the art will understand that heating and cooling can be controlled by other means. For example, diacetyl can be added to a room temperature slurry and can be automatically heated and cooled.
EXAMPLE 2: CYCLODEXTRIN INCLUSION COMPLEX WITH \(\alpha\)-CYCLODEXTRIN AND DIACETYL, PECTIN AS AN EMULSIFIER, AND PROCESS FOR FORMING SAME

[00131] The \(\beta\)-cyclodextrin of example 1 was replaced with \(\alpha\)-cyclodextrin and dry blended with 1 wt % pectin (i.e., 1 wt % of pectin: \(\beta\)-cyclodextrin; XPQ EMP 5 beet pectin available from Degussa-France). The mixture was processed and dried by the method set forth in Example 1. The percent retention of diacetyl in the cyclodextrin inclusion complex was 11.4 wt %.

EXAMPLE 3: CYCLODEXTRIN INCLUSION COMPLEX WITH \(\beta\)-CYCLODEXTRIN AND ORANGE ESSENCE, PECTIN AS AN EMULSIFIER, AND PROCESS FOR FORMING SAME

[00132] Orange essence, an aqueous waste stream from juice production, was added as the aqueous phase to a dry blend of \(\beta\)-cyclodextrin and 2 wt % pectin, formed according to the process set forth in Example 1. No additional water was added, the solids content was approximately 28 %. The cyclodextrin inclusion complex was formed by the method set forth in Example 1. The dry inclusion complex contained approximately 3 to 4 wt % acetaldehyde, approximately 5 to 7 wt % ethyl butyrate, approximately 2 to 3 wt % linalool and other citrus enhancing notes. The resulting cyclodextrin inclusion complex can be useful in top-noting beverages.

EXAMPLE 4: CYCLODEXTRIN INCLUSION COMPLEX WITH \(\beta\)-CYCLODEXTRIN AND ACETYL PROPIONYL, PECTIN AS AN EMULSIFIER, AND PROCESS FOR FORMING SAME

[00133] A molar excess of acetyl propionyl was added to a dry blend of \(\beta\)-cyclodextrin and 2 wt % pectin in water, following the method set forth in Example 1. The percent retention of acetyl propionyl in the cyclodextrin inclusion complex was 9.27 wt %. The mixture can be useful in top-noting diacetyl-free butter systems.
EXAMPLE 5: ORANGE OIL FLAVOR PRODUCT AND PROCESS FOR FORMING SAME

[00134] Orange oil (i.e., Orange Bresil; 75 g) was added to an aqueous phase comprising 635 g of water, 403.75 g of maltodextrin, and 21.25 g of beet pectin (available from Degussa – France, product no. XPQ EMP 5). The orange oil was added to the aqueous phase with gentle stirring, followed by strong stirring at 10,000 RPM to form a mixture. The mixture was then passed through a homogenizer at 250 bars to form an emulsion. The emulsion was dried using a NIRO-brand spray drier having an inlet temperature of approximately 180 °C and an outlet temperature of approximately 90 °C to form a dried product. The percent flavor retention was then quantified as the amount of oil (in g) in 100 g of the dried product, divided by the oil content in the starting mixture. The percent retention of orange oil was approximately 91.5%.

EXAMPLE 6: ORANGE OIL FLAVOR PRODUCT AND PROCESS FOR FORMING SAME

[00135] Orange oil (75 g) was added to an aqueous phase comprising 635 g of water, 297.50 g of maltodextrin, and 127.50 g gum arabic (available from Colloïds Naturels International). The orange oil was added to the aqueous phase and dried following the method set forth in Example 5. The percent flavor retention was approximately 91.5%.

EXAMPLE 7: ORANGE OIL FLAVOR PRODUCT AND PROCESS FOR FORMING SAME

[00136] Orange oil (75 g) was added to an aqueous phase comprising 635 g of water, 297.50 g of maltodextrin, 123.25 g gum arabic (available from Colloïds Naturels International), and 4.25 g of depolymerized citrus pectin. The orange oil was added to the aqueous phase and dried following the method set forth in Example 5. The percent flavor retention was approximately 96.9%.

EXAMPLE 8: ORANGE OIL FLAVOR PRODUCT AND PROCESS FOR FORMING SAME

[00137] Orange oil (75 g) was added to an aqueous phase comprising 635 g of water, 297.50 g of maltodextrin, 123.25 g gum arabic (available from Colloïds Naturels International).
International), and 4.25 g of beet pectin (available from Degussa – France, product no. XPQ EMP 5). The orange oil was added to the aqueous phase and dried following the method set forth in Example 5. The percent flavor retention was approximately 99.0 %.

EXAMPLE 9: ORANGE OIL FLAVOR PRODUCT AND PROCESS FOR FORMING SAME

[00138] Orange oil (75 g) was added to an aqueous phase comprising 635 g of water, 403.75 g of maltodextrin, and 21.25 g of depolymerized citrus pectin. The orange oil was added to the aqueous phase and dried following the method set forth in Example 5. The percent flavor retention was approximately 90.0 %.

EXAMPLE 10: ORANGE OIL FLAVOR PRODUCT AND PROCESS FOR FORMING SAME

[00139] Orange oil (75 g) was added to an aqueous phase comprising 635 g of water, 340.00 g of maltodextrin, and 85.00 g gum arabic (available from Colloids Naturels International). The orange oil was added to the aqueous phase and dried following the method set forth in Example 5. The percent flavor retention was approximately 91.0 %.

EXAMPLE 11: ORANGE OIL FLAVOR PRODUCT AND PROCESS FOR FORMING SAME

[00140] Orange oil (75 g) was added to an aqueous phase comprising 635 g of water and 425.00 g of maltodextrin. The orange oil was added to the aqueous phase and dried following the method set forth in Example 5. The percent flavor retention was approximately 61.0%.

EXAMPLE 12: ORANGE OIL FLAVOR PRODUCT AND PROCESS FOR FORMING SAME

[00141] Orange oil (75 g) was added to an aqueous phase comprising 635 g of water, 420.75 g of maltodextrin, and 4.25 g of pectin. The orange oil was added to the aqueous phase and dried following the method set forth in Example 5. The percent flavor retention was approximately 61.9 %.
EXAMPLE 13: ORANGE OIL FLAVOR PRODUCT AND PROCESS FOR FORMING SAME

[00142] Orange oil (75 g) was added to an aqueous phase comprising 635 g of water, 403.75 g of maltodextrin, and 21.50 g of pectin. The orange oil was added to the aqueous phase and dried following the method set forth in Example 5. The percent flavor retention was approximately 71.5 %.

EXAMPLE 14: ORANGE OIL FLAVOR PRODUCT AND PROCESS FOR FORMING SAME

[00143] Orange oil (75 g) was added to an aqueous phase comprising 635 g of water, 420.75 g of maltodextrin, and 4.75 g of depolymerized citrus pectin. The orange oil was added to the aqueous phase and dried following the method set forth in Example 5. The percent flavor retention was approximately 72.5 %.

EXAMPLE 15: ORANGE OIL FLAVOR PRODUCT AND PROCESS FOR FORMING SAME

[00144] Orange oil (75 g) was added to an aqueous phase comprising 635 g of water, 420.75 g of maltodextrin, and 4.75 g of beet pectin (available from Degussa-France, product no. XPQ EMP 5). The orange oil was added to the aqueous phase and dried following the method set forth in Example 5. The percent flavor retention was approximately 78.0 %.

EXAMPLE 16: ORANGE OIL FLAVOR PRODUCT AND PROCESS FOR FORMING SAME

[00145] Orange oil (75 g) was added to an aqueous phase comprising 635 g of water, 414.40 g of maltodextrin, and 10.60 g of depolymerized citrus pectin. The orange oil was added to the aqueous phase and dried following the method set forth in Example 5. The percent flavor retention was approximately 85.0 %.

EXAMPLE 17: ORANGE OIL FLAVOR PRODUCT AND PROCESS FOR FORMING SAME

[00146] Orange oil (75 g) was added to an aqueous phase comprising 635 g of water, 414.40 g of maltodextrin, and 10.60 g of beet pectin (available from Degussa-France, product
no. XPQ EMP 5). The orange oil was added to the aqueous phase and dried following the method set forth in Example 5. The percent flavor retention was approximately 87.0%.

EXAMPLE 18: CYCLODEXTRIN INCLUSION COMPLEX WITH β-CYCLODEXTRIN AND CITRAL, PECTIN AS AN EMULSIFIER, AND PROCESS FOR FORMING SAME

[00147] At atmospheric pressure, in a 1-L reactor, 200 g of β-cyclodextrin was dry blended with 4.0 g of beet pectin (2 wt % of pectin: β-cyclodextrin; XPQ EMP 5 beet pectin available from Degussa-France) to form a dry blend. 500 g of deionized water was added to the dry blend of β-cyclodextrin and pectin to form a slurry or mixture. The 1-L reactor was set up for heating and cooling via a lab-scale water bath heating and cooling apparatus. The mixture was heated at 55-60 degrees C for 5 hours and agitated by stirring. 27 g of citral (natural citral, SAP No. 921565, Lot No. 10000223137, available from Citrus & Allied) was added. The reactor was sealed, and the resulting mixture was stirred for 5 hours at about 55-60 degrees C. The cooling portion of the heating and cooling lab apparatus was then turned on, and the mixture was stirred overnight at about 5-10 degrees C. The mixture was then spray dried on a BUCHI B-191 lab spray dryer (available from Buchi, Switzerland) having an inlet temperature of approximately 210 degrees C and an outlet temperature of approximately 105 degrees C. A percent retention of 11.5 wt % of citral in the cyclodextrin inclusion complex was achieved. The resulting dry powder included 0.08 wt % surface oils (free citral).

EXAMPLE 19A: FLAVOR COMPOSITION COMPRISING CYCLODEXTRIN-ENCAPSULATED CITRAL AND EXCESS UNCOMPLEXED CYCLODEXTRIN

[00148] Encapsulated citral was produced according to the method set forth in Example 18. The resulting dry powder including the cyclodextrin-encapsulated citral was dry blended with additional β-cyclodextrin to achieve a wt % of about 1 wt % of citral in the resulting dry powder mixture (“citral-β-cyclodextrin/cyclodextrin blend”). The citral-cyclodextrin/cyclodextrin blend was added to an acidic beverage in a wt % of about 0.2 wt % of the dry powder mixture (i.e., β-cyclodextrin-encapsulated citral plus additional β-cyclodextrin) to the total weight of the beverage. This provided 10-15 ppm of citral and about 0.2 wt % of β-cyclodextrin to the acidic beverage.
EXAMPLE 19B: FLAVOR COMPOSITION COMPRISING CYCLODEXTRIN-ENCAPSULATED CITRAL AND EXCESS UNCOMPLEXED CYCLODEXTRIN

[00149] Encapsulated citral is produced according to the method set forth in Example 18. The resulting dry powder including the cycloextrin-encapsulated citral is dry blended with additional β-cyclodextrin to achieve a wt % of about 0.1 wt % of citral in the resulting dry powder mixture ("citral-cyclodextrin/cyclodextrin blend"). The citral-cyclodextrin/cyclodextrin blend is added to a beverage as a topnote. The citral-cyclodextrin/cyclodextrin blend is added in a wt % of about 0.2 wt % of the dry powder mixture (i.e., β-cyclodextrin-encapsulated citral plus additional β-cyclodextrin) to the total weight of the beverage.

EXAMPLE 20: STABILIZATION OF CITRAL WITH CYCLODEXTRIN

[00150] Citral (natural citral, SAP No. 921565, Lot No. 10000223137, available from Citrus & Allied) was cut in ethanol and diluted in citric acid to obtain a desired flavor level (e.g., 3mL (1% citral in EtOH) per 2L 0.6% citric acid; designated as "control" or "control freshly made" in Table 1B). Then, 0.1 wt % and 0.2 wt % of α-cyclodextrin or β-cyclodextrin was added to the control and maintained at 40 degrees F or 90 degrees F for 18 hours, 36 hours, or 48 hours to simulate various shelf lives. The raw area counts of various forms of citral or character-impact citrus flavor compounds (i.e., neral, geranial, and citral total, the sum of neral and geranial), and a variety of other compounds, including common citrus flavor off-note chemicals (e.g., carveol, p-cymene or p-cymene-8-ol, p,a-dimethyl styrene, mentha-1,5-dien-8-ol 1, and mentha-a,5-dien-8-ol 2) and chlorocyclohexane internal standard (designated as "CCH int std" in Table 1B) were measured for each permutation of the experiment, as shown in Table 1B. As used herein, the term "raw area counts" is used to refer to the area under the curve of a corresponding portion of a gas chromatogram when the samples are analyzed using a gas chromatography – mass spectrometry analysis, namely, a PEGASUS II Time-of-flight mass spectrometer (TOF-MS; available from LECO Corp., St. Joseph, Michigan). The chlorocyclohexane internal standard was included at 10 ppm per beverage to attempt to normalize the raw area counts of the other compounds of interest. As shown in Table 1B, the addition of cyclodextrin (and particularly, β-cyclodextrin) increased the amount of citral in the solution, and decreased the amount of off-notes formed. Specifically, this phenomenon was observed as simulated shelf-life increased (i.e., a greater distinction was observed between solutions containing cyclodextrin, and particularly, β-
cycloextrin and the control as time and temperature increased). This can be seen by comparing FIG. 8 and FIG. 9, which illustrate the inhibition of off-note formation with the addition of β-cycloextrin. This can further be seen by comparing FIG. 10 and FIG. 11, which illustrate a sustained citral (and other character-impact citrus flavor) contribution to the beverage at later time intervals and lack of off-notes at later time intervals with the addition of β-cycloextrin.

Table 1B. Stability/Method Development of Citral-Cycloextrin

<table>
<thead>
<tr>
<th>sample</th>
<th>ID</th>
<th>nerol</th>
<th>geranial</th>
<th>citral total</th>
<th>carvone</th>
<th>p-cymene</th>
<th>p,a-dimethyldurene</th>
<th>CCH</th>
<th>mentha-1,5-dien-8-ol 1</th>
<th>mentha-1,5-dien-8-ol 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>rool freshly made</td>
<td>time = 0</td>
<td>26,300,000</td>
<td>74,304,000</td>
<td>100,634,000</td>
<td>1,113,100</td>
<td>5,886,300</td>
<td>1,082,800</td>
<td>22,201,000</td>
<td></td>
<td></td>
</tr>
<tr>
<td>rool + 0.1% alpha CD</td>
<td>time = 0</td>
<td>21,285,000</td>
<td>82,820,000</td>
<td>84,105,000</td>
<td>1,022,500</td>
<td>6,633,200</td>
<td>939,659</td>
<td>1,299,300</td>
<td></td>
<td></td>
</tr>
<tr>
<td>rool + 0.2% alpha CD</td>
<td>time = 0</td>
<td>21,291,000</td>
<td>82,999,000</td>
<td>83,990,000</td>
<td>981,550</td>
<td>1,646,000</td>
<td>949,770</td>
<td>20,362,000</td>
<td></td>
<td></td>
</tr>
<tr>
<td>rool + 0.1% beta CD</td>
<td>time = 0</td>
<td>20,897,000</td>
<td>79,297,000</td>
<td>100,194,000</td>
<td>838,400</td>
<td>1,488,200</td>
<td>836,691</td>
<td>9,211,700</td>
<td></td>
<td></td>
</tr>
<tr>
<td>rool + 0.2% beta CD</td>
<td>time = 0</td>
<td>20,945,000</td>
<td>84,485,000</td>
<td>85,530,000</td>
<td>728,010</td>
<td>1,259,100</td>
<td>954,970</td>
<td>14,376,000</td>
<td></td>
<td></td>
</tr>
<tr>
<td>rool refrig</td>
<td>time = 16 hrs @ 90°F</td>
<td>14,874,000</td>
<td>35,523,000</td>
<td>50,397,000</td>
<td>0</td>
<td>5,180,200</td>
<td>2,193,100</td>
<td>18,990,000</td>
<td>2,277,700</td>
<td></td>
</tr>
<tr>
<td>rool + 0.1% alpha CD</td>
<td>time = 16 hrs @ 90°F</td>
<td>16,053,000</td>
<td>39,960,000</td>
<td>66,013,000</td>
<td>0</td>
<td>4,190,200</td>
<td>1,027,400</td>
<td>19,398,000</td>
<td>2,038,400</td>
<td></td>
</tr>
<tr>
<td>rool + 0.2% alpha CD</td>
<td>time = 16 hrs @ 90°F</td>
<td>19,840,000</td>
<td>44,449,000</td>
<td>64,289,000</td>
<td>0</td>
<td>1,655,400</td>
<td>1,015,700</td>
<td>15,954,000</td>
<td>1,745,000</td>
<td></td>
</tr>
<tr>
<td>rool + 0.1% beta CD</td>
<td>time = 16 hrs @ 90°F</td>
<td>11,154,000</td>
<td>44,480,000</td>
<td>55,634,000</td>
<td>405,540</td>
<td>1,182,000</td>
<td>754,470</td>
<td>13,798,000</td>
<td>1,880,000</td>
<td></td>
</tr>
<tr>
<td>rool + 0.2% beta CD</td>
<td>time = 16 hrs @ 90°F</td>
<td>23,414,000</td>
<td>82,972,000</td>
<td>106,387,000</td>
<td>290,580</td>
<td>1,427,000</td>
<td>891,120</td>
<td>19,802,000</td>
<td></td>
<td></td>
</tr>
<tr>
<td>rool refrigerated</td>
<td>time = 56 hrs @ 90°F</td>
<td>4,090,600</td>
<td>10,646,000</td>
<td>14,735,600</td>
<td>0</td>
<td>12,616,000</td>
<td>2,446,900</td>
<td>17,876,000</td>
<td>3,179,000</td>
<td></td>
</tr>
<tr>
<td>rool + 0.1% alpha CD</td>
<td>time = 56 hrs @ 90°F</td>
<td>4,346,905</td>
<td>11,343,000</td>
<td>15,689,000</td>
<td>0</td>
<td>10,842,000</td>
<td>2,182,200</td>
<td>17,016,000</td>
<td>3,195,000</td>
<td></td>
</tr>
<tr>
<td>rool + 0.2% alpha CD</td>
<td>time = 56 hrs @ 90°F</td>
<td>5,829,100</td>
<td>13,944,000</td>
<td>19,773,000</td>
<td>0</td>
<td>12,381,000</td>
<td>2,376,400</td>
<td>17,896,000</td>
<td>3,231,300</td>
<td></td>
</tr>
<tr>
<td>rool + 0.1% beta CD</td>
<td>time = 56 hrs @ 90°F</td>
<td>7,127,100</td>
<td>16,486,000</td>
<td>23,612,100</td>
<td>0</td>
<td>3,773,300</td>
<td>1,104,100</td>
<td>14,947,000</td>
<td>1,932,000</td>
<td></td>
</tr>
<tr>
<td>rool + 0.2% beta CD</td>
<td>time = 56 hrs @ 90°F</td>
<td>8,901,400</td>
<td>19,720,000</td>
<td>28,621,400</td>
<td>0</td>
<td>2,326,700</td>
<td>901,610</td>
<td>13,433,000</td>
<td></td>
<td></td>
</tr>
<tr>
<td>rool refrigerated</td>
<td>time = 36 hrs @ 40°F</td>
<td>17,124,000</td>
<td>46,714,000</td>
<td>63,838,000</td>
<td>604,400</td>
<td>1,523,500</td>
<td>876,520</td>
<td>16,247,000</td>
<td></td>
<td></td>
</tr>
<tr>
<td>rool refrigerated a</td>
<td>time = 48 hrs @ 40°F</td>
<td>26,435,000</td>
<td>61,091,000</td>
<td>87,526,000</td>
<td>0</td>
<td>1,293,700</td>
<td>855,960</td>
<td>19,397,000</td>
<td></td>
<td></td>
</tr>
<tr>
<td>rool refrigerated b</td>
<td>time = 48 hrs @ 40°F</td>
<td>1,446,800</td>
<td>3,652,100</td>
<td>5,098,900</td>
<td>762,610</td>
<td>18,504,000</td>
<td>2,659,000</td>
<td>18,165,000</td>
<td>3,743,000</td>
<td></td>
</tr>
</tbody>
</table>

EXAMPLE 21: STABILITY OF CYCLODEXTRIN-ENCAPSULATED CITRAL IN ACID SOLUTION

[00151] As shown in Table 2, four different versions of a sample acid beverage were analyzed. The four sample beverages were formed by adding various forms of citral to a low pH lemonade base, or an “acid-sugar” solution (e.g., 0.5% citric acid and 8% sugar in water). The first beverage, referred to in Table 2 as “no citral,” was formed by adding a non-citral citrus flavor component to the acid-sugar solution. The second beverage, “add citral,” was formed by adding 3mL (1% citral in EtOH) per 2L 0.6% citric acid (the citral used was natural citral, SAP No. 921565, Lot No. 10000223137, available from Citrus & Allied) to the acid-sugar solution to achieve a citral concentration of about 10-15 ppm. The third beverage, “0.2% BCD-citral,” was formed by adding 0.2 wt% of the citral-cycloextrin/cycloextrin blend formed in Example 19A to the acid-sugar solution to achieve a citral concentration of about 10-15 ppm. The fourth beverage, “0.2% WSR,” was formed by adding 0.2 wt% of water-soluble rosemary to the second beverage, while maintaining a citral concentration of
about 10-15 ppm. Water soluble rosemary ("WSR") as used herein refers to the industry standard used in stabilizing water-miscible flavorings.

[00152] The raw area counts of various forms of citral or character-impact citrus flavor compounds (i.e., sabinene, p-cymene, neral, and geranial), and a variety of other compounds, including common citral off-note chemicals (e.g., p,a-dimethyl styrene, p-cymene-8-ol, and mentha-1,5-dien-8-ol 1) were measured for each of the four beverages. Measurements were taken after 1 day at 40 degrees F, 1 day at 88 degrees F, 2 days at 40 degrees F, 2 days at 88 degrees F, 7 days at 40 degrees F, 7 days at 100 degrees F, 14 days at 40 degrees F, 14 days at 100 degrees F, 21 days at 40 degrees F, and 21 days at 100 degrees F to simulate various shelf lives. In addition, the raw area counts of the above compounds in a can of Country Time®-brand lemonade were determined.

[00153] As shown in Table 2, FIG. 12 and FIG. 13, at warmer temperatures (i.e., 88 degrees F and 100 degrees F), the third beverage included similar raw area counts of citral and other citrus flavor compounds as the other beverages (see FIG. 12), but with the lowest raw area counts of off-notes formed at all time intervals (see FIG. 13). As shown in FIGS. 14 and 15, at a colder temperature (i.e., 40 degrees F), the third beverage included similar raw area counts of citral and other citrus flavor compounds as the other beverages (see FIG. 14), but with lower raw area counts of off-notes formed at all time intervals than the second and third beverages and the same raw area counts of off-notes formed in the first beverage to which no citral was added (see the “Offnotes Combined” column in Table 2 and FIG. 15).

[00154] As shown in Table 2, mentha-1,5-dien-8-ol is the first off-note to form from unprotected citral, which further degrades to p-cymen-8-ol over time. However, neither off-note was present in the third beverage, which includes the citral-cyclodextrin/cyclodextrin blend. Also, the 0.2% BCD-citral was better at stabilizing citral and other citrus flavor compounds than the industry standard WSR.
Table 2: Stability comparisons of four beverages containing various amounts and forms of citral and cyclodextrin

<table>
<thead>
<tr>
<th>Example</th>
<th>ID</th>
<th>p-cymene</th>
<th>nerol</th>
<th>geranial</th>
<th>P-a-dimethyl styrene</th>
<th>p-CYME-8-OL</th>
<th>nEROL-1,5-dien-5-ol</th>
<th>Olfines Combined</th>
<th>TOTAL AREA</th>
</tr>
</thead>
<tbody>
<tr>
<td>TAC1</td>
<td></td>
<td>0.587,000</td>
<td>1.546,000</td>
<td>1.510,000</td>
<td>0.587,000</td>
<td>1.510,000</td>
<td>0.000</td>
<td>0.000</td>
<td>0.000</td>
</tr>
<tr>
<td>TAC2</td>
<td></td>
<td>0.587,000</td>
<td>1.546,000</td>
<td>1.510,000</td>
<td>0.587,000</td>
<td>1.510,000</td>
<td>0.000</td>
<td>0.000</td>
<td>0.000</td>
</tr>
<tr>
<td>TAC3</td>
<td></td>
<td>0.587,000</td>
<td>1.546,000</td>
<td>1.510,000</td>
<td>0.587,000</td>
<td>1.510,000</td>
<td>0.000</td>
<td>0.000</td>
<td>0.000</td>
</tr>
<tr>
<td>TAC4</td>
<td></td>
<td>0.587,000</td>
<td>1.546,000</td>
<td>1.510,000</td>
<td>0.587,000</td>
<td>1.510,000</td>
<td>0.000</td>
<td>0.000</td>
<td>0.000</td>
</tr>
<tr>
<td>TAC5</td>
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<td>0.587,000</td>
<td>1.546,000</td>
<td>1.510,000</td>
<td>0.587,000</td>
<td>1.510,000</td>
<td>0.000</td>
<td>0.000</td>
<td>0.000</td>
</tr>
<tr>
<td>TAC6</td>
<td></td>
<td>0.587,000</td>
<td>1.546,000</td>
<td>1.510,000</td>
<td>0.587,000</td>
<td>1.510,000</td>
<td>0.000</td>
<td>0.000</td>
<td>0.000</td>
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<tr>
<td>TAC7</td>
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<td>0.587,000</td>
<td>1.546,000</td>
<td>1.510,000</td>
<td>0.587,000</td>
<td>1.510,000</td>
<td>0.000</td>
<td>0.000</td>
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<tr>
<td>TAC8</td>
<td></td>
<td>0.587,000</td>
<td>1.546,000</td>
<td>1.510,000</td>
<td>0.587,000</td>
<td>1.510,000</td>
<td>0.000</td>
<td>0.000</td>
<td>0.000</td>
</tr>
<tr>
<td>TAC9</td>
<td></td>
<td>0.587,000</td>
<td>1.546,000</td>
<td>1.510,000</td>
<td>0.587,000</td>
<td>1.510,000</td>
<td>0.000</td>
<td>0.000</td>
<td>0.000</td>
</tr>
<tr>
<td>TAC10</td>
<td></td>
<td>0.587,000</td>
<td>1.546,000</td>
<td>1.510,000</td>
<td>0.587,000</td>
<td>1.510,000</td>
<td>0.000</td>
<td>0.000</td>
<td>0.000</td>
</tr>
</tbody>
</table>

EXAMPLE 22: STABILITY OF CYCLODEXTRIN-ENCAPSULATED CITRAL IN ACID

[00155] A first beverage, referred to as “.3% BCD” in the ID column of Table 3, was formed by adding 0.3 wt % of the citral-cyclodextrin/cyclodextrin blend formed in Example 19A to the acid-sugar solution to achieve a citral concentration of about 20 ppm. A second beverage, “.3% WSR,” was formed by adding 0.3 wt % of WSR to the second beverage of Example 21, while maintaining a citral concentration of about 10-15 ppm. The raw area counts of various forms of citral or citrus flavor compounds (i.e., sabine, p-cymene, nerol, and geranial), and a variety of other compounds, including common citral off-note chemicals (e.g., p-a-dimethyl styrene, p-cymene-8-ol, and mentha-1,5-dien-8-ol) were measured for each of the two beverages. Measurements were taken after 7 days at 40 degrees F, 7 days at 100 degrees F, 14 days at 40 degrees F, 14 days at 100 degrees F, 21 days at 40 degrees F and 21 days at 100 degrees F to simulate various shelf lives. As shown in Table 3, at the warmer
temperature and the colder temperature, the first beverage included similar maintenance of citral (and other character-impact citrus flavor) contribution as the other beverage, but enhanced inhibition of the formation of off-notes at all time intervals. A general decrease in volatiles was noted due to interactions with the beverage container. However, the very strong complexes that formed between citral and β-cyclodextrin may be partially responsible for the reduction in headspace values for citral. Citral is, nevertheless, available for taste, as shown in the sensory analyses (Example 34 and FIGS. 16 and 17), and as previously described.

Table 3: Stability comparisons of two beverages containing various amounts and forms of citral and cyclodextrin

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>t = 7 day, 3% BCD</th>
<th>t = 14 day, 3% BCD</th>
<th>t = 21 day, 3% BCD</th>
<th>t = 7 day, 3% RCD</th>
<th>t = 14 day, 3% RCD</th>
<th>t = 21 day, 3% RCD</th>
<th>t = 7 day, 3% VRD</th>
<th>t = 14 day, 3% VRD</th>
<th>t = 21 day, 3% VRD</th>
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</thead>
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<td>2EC3</td>
<td>3,108,100</td>
<td>11,474,000</td>
<td>4,500,000</td>
<td>1,027,100</td>
<td>318,040</td>
<td>210,780,000</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2EC4</td>
<td>3,275,800</td>
<td>17,390,000</td>
<td>3,470,400</td>
<td>1,473,100</td>
<td>609,940</td>
<td>223,890,000</td>
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<td></td>
<td></td>
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<tr>
<td>2EC5</td>
<td>2,546,200</td>
<td>12,714,000</td>
<td>2,565,500</td>
<td>923,012</td>
<td>416,269</td>
<td>192,210,000</td>
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<tr>
<td>2EH3</td>
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<td>10,886,000</td>
<td>824,890</td>
<td>158,860,000</td>
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</tr>
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<td>2EH4</td>
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<td>2EH5</td>
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<td>3,247,000</td>
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<td>265,040,000</td>
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<td></td>
<td></td>
</tr>
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<td>747,810</td>
<td>723,560,000</td>
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<tr>
<td>3GC5</td>
<td>1,227,300</td>
<td>1,055,200</td>
<td>896,860</td>
<td>392,450</td>
<td>365,790</td>
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<td>6,000,000</td>
<td>1,402,900</td>
<td>1,227,700</td>
<td>490,040</td>
<td>191,800,000</td>
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<tr>
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<td>1,519,100</td>
<td>1,141,300</td>
<td>439,800</td>
<td>62,800,000</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

EXAMPLE 23: CYCLODEXTRIN INCLUSION COMPLEX WITH β-CYCLODEXTRIN AND LEMON OIL 3X, PECTIN AS AN EMULSIFIER, AND PROCESS FOR FORMING SAME

[00156] At atmospheric pressure, in a 1-L reactor, 400 g of β-cyclodextrin was dry blended with 8.0 g of beet pectin (2 wt % of pectin: β-cyclodextrin; XPO EMP 5 beet pectin available from Degussa-France) to form a dry blend. 1 L of deionized water was added to the dry blend of β-cyclodextrin and pectin to form a slurry or mixture. The 1-L reactor was set up for heating and cooling via a lab-scale water bath heating and cooling apparatus. The mixture was stirred for about 30 min. 21 g of 3X (i.e., 3-fold) California Lemon Oil, available from Citrus & Allied) was added. The reactor was sealed, and the resulting mixture was stirred for 4 hours at about 55-60 degrees C. The cooling portion of the heating and cooling lab apparatus was then turned on, and the mixture was stirred overnight at about 5-10 degrees C. The mixture was then spray dried on a BUCHI B-191 lab spray dryer (available from Buchi, Switzerland) having an inlet temperature of approximately 210 degrees C and an
outlet temperature of approximately 105 degrees C. A percent retention of 4.99 wt % of lemon oil 3X in the cyclodextrin inclusion complex was achieved.

EXAMPLE 24A: FLAVOR COMPOSITION COMPRISING CYCLODEXTRIN-ENCAPSULATED LEMON OIL 3X AND EXCESS UNCOMPLEXED CYCLODEXTRIN USED IN BEVERAGE PRODUCT

[00157] The dry powder resulting from Example 23 including the cyclodextrin-encapsulated lemon oil 3X is dry blended with additional β-cyclodextrin to achieve a wt % of about 1 wt % of lemon oil 3X in the resulting dry powder mixture ("lemon oil 3X-cyclodextrin/cyclodextrin blend"). The lemon oil 3X-cyclodextrin/cyclodextrin blend is then added to a beverage in a wt % ranging from about 0.05 wt % to about 0.30 wt % of the dry powder mixture (i.e., β-cyclodextrin-encapsulated citral plus additional β-cyclodextrin) to the total weight of the beverage. This is expected to provide 20-30 ppm of lemon oil 3X and from about 0.05 wt % to about 0.30 wt % of β-cyclodextrin to the beverage, depending on the amount of dry powder mixture added to the beverage.

EXAMPLE 24B: FLAVOR COMPOSITION COMPRISING CYCLODEXTRIN-ENCAPSULATED LEMON OIL 3X AND EXCESS UNCOMPLEXED CYCLODEXTRIN USED IN BEVERAGE PRODUCT

The combination of the dry powder from Example 24 mixed with the citral-cyclodextrin inclusion complex from Example 18 is blended (5 parts citral / 3 parts 3X lemon) and blended with additional β-cyclodextrin to achieve a 1 % active flavor in cyclodextrin. The mixture is useful in delivering a stable peely, fresh lemon character in spices and condiments with a high acid content (acetic) or in beverage where a more opaque, juice like appearance is desired, with high stability.

EXAMPLE 25: CYCLODEXTRIN INCLUSION COMPLEX WITH β-CYCLODEXTRIN AND ALPHA-TOCOPHEROL, PECTIN AS AN EMULSIFIER, AND PROCESS FOR FORMING SAME

[00158] At atmospheric pressure, in a 1-L reactor, 200 g of β-cyclodextrin was dry blended with 4.0 g of beet pectin (2 wt % of pectin: β-cyclodextrin; XPQ EMP 5 beet pectin available from Degussa-France) to form a dry blend. 500 g of deionized water was added to the dry blend of β-cyclodextrin and pectin to form a slurry or mixture. The 1-L reactor was
set up for heating and cooling via a lab-scale water bath heating and cooling apparatus. The mixture was stirred for about 30 min. 23 g of D,L-alpha-tocopherol (Kosher, SAP# 1020477, available from BASF) was added. The reactor was sealed, and the resulting mixture was stirred overnight at about 55-60 degrees C. The cooling portion of the heating and cooling lab apparatus was then turned on, and the mixture was stirred overnight at about 5-10 degrees C. The mixture was then spray dried on a BUCHI B-191 lab spray dryer (available from Buchi, Switzerland) having an inlet temperature of approximately 210 degrees C and an outlet temperature of approximately 105 degrees C. A percent retention of 10.31 wt % of alpha-tocopherol in the cyclodextrin inclusion complex was achieved. A 1:1 mole ratio of alpha tocopherol in β-cyclodextrin would correspond to 27.52 wt %, however, the literature reports this to be an oily paste. The 10.31 wt % product is a dry, free flowing powder that can easily be dispersed in water. The 10.31 wt % alpha tocopherol complex easily disperses in water when used at 0.1% (i.e., cut in excess uncomplexed β-cyclodextrin).

EXAMPLE 26: COMPOSITION COMPRISING CYCLODEXTRIN-ENCAPSULATED ALPHA-TOCOPHEROL AND EXCESS UNCOMPLEXED CYCLODEXTRIN USED IN BEVERAGE PRODUCT

The dry powder resulting from Example 25 that includes the cyclodextrin-encapsulated alpha-tocopherol is dry blended with additional β-cyclodextrin to achieve a wt % of about 1 wt % of alpha-tocopherol in the resulting dry powder mixture (“alpha-tocopherol-cyclodextrin/cyclodextrin blend”). The alpha-tocopherol-cyclodextrin/cyclodextrin blend is then added to a beverage as an antioxidant and/or a nutraceutical to an A.C.E. beverage (i.e., A = vitamin A, C = vitamin C, and E = vitamin E) in a wt % of about 0.2 wt % of the dry powder mixture (i.e., β-cyclodextrin-encapsulated alpha-tocopherol plus additional β-cyclodextrin) to the total weight of the beverage. This is expected to provide 10 ppm of alpha-tocopherol and about 0.2 wt % of β-cyclodextrin to the acidic beverage.

EXAMPLE 27: FLAVOR COMPOSITION COMPRISING CYCLODEXTRIN-ENCAPSULATED ALPHA-TOCOPHEROL AND EXCESS UNCOMPLEXED CYCLODEXTRIN USED IN BEVERAGE PRODUCT

The dry powder resulting from Example 25 including the cyclodextrin-encapsulated alpha-tocopherol is combined with other flavor compositions (e.g., the citral-β-
cycloextrin formed according to Example 18, and/or the lemon oil 3X-β-cycloextrin formed according to Example 23) and then dry blended with additional β-cycloextrin to achieve the desired level of flavor components and alpha-tocopherol in the resulting dry powder mixture. The resulting dry powder mixture is then added to a beverage as an antioxidant/nutraceutical/flavor composition. This is expected to deliver the appropriate amount of antioxidant/nutraceutical and flavor profile to the beverage, and an appropriate amount of β-cycloextrin to the beverage (e.g., 0.2 wt %). In beverages, such a combination is expected to provide flavor, cloud (i.e., juice-like appearance), added stability to citrus components, and demonstrates the advantage of being able to blend flavor level, cloud and functionality. It is anticipated that such a system is highly effective in salad dressing and seasoning mixes, at least partially because of the enhanced citrus protection coupled with added lipid protection.

EXAMPLE 28: CYCLODEXTRIN INCLUSION COMPLEX WITH β-CYCLODEXTRIN AND LEMON LIME OILS, PECTIN AS AN EMULSIFIER AND XANTHAN GUM AS A THICKENER, AND PROCESS FOR FORMING SAME

[00161] In a 1-L reactor, 400 g of β-cycloextrin (W7 β-cycloextrin, available from Wacker), 8 g of beet pectin (2 wt % of pectin: β-cycloextrin; XPQ EMP 4 beet pectin available from Degussa-France), and 1.23 g xanthan gum (KELTROL xanthan gum, available from CP Kelco SAP No. 15695) were dry blended together to form a dry blend. 800 mL of deionized water were added to the dry blend to form a slurry or mixture. The 1-L reactor was set up for heating and cooling via a lab-scale water bath heating and cooling apparatus. The mixture was agitated by stirring for about 30 min. 21 g of lemon lime flavor 043-03000 (SAP# 1106890, available from Degussa Flavors & Fruit Systems), were added. The reactor was sealed, and the resulting mixture was stirred for 4 hours at about 55-60 degrees C. The cooling portion of the heating and cooling lab apparatus was then turned on, and the mixture was stirred overnight at about 5-10 degrees C. The mixture was then spray dried on a BUCHI B-191 lab spray dryer (available from Buchi, Switzerland) having an inlet temperature of approximately 210 degrees C and an outlet temperature of approximately 105 degrees C. A percent retention of about 4.99 wt % of lemon lime oils in the cycloextrin inclusion complex was achieved.
EXAMPLE 29: CYCLODEXTRIN INCLUSION COMPLEX WITH β-CYCLODEXTRIN AND LEMON LIME OILS, PECTIN AS AN EMULSIFIER AND XANTHAN GUM AS A THICKENER, AND PROCESS FOR FORMING SAME

[00162]  In a 1-L reactor, 300 g of β-cyclodextrin (W7 β-cyclodextrin, available from Wacker), 6 g of beet pectin (2 wt % of pectin: β-cyclodextrin; XPQ EMP 4 beet pectin available from Degussa-France), and 1.07 g xanthan gum (KELTROL xanthan gum, available from CP Kelco SAP No. 15695) were dry blended together to form a dry blend. 750 mL of deionized water were added to the dry blend to form a slurry or mixture. The 1-L reactor was set up for heating and cooling via a lab-scale water bath heating and cooling apparatus. The mixture was agitated by stirring for about 30 min. 16 g of lemon lime flavor 043-03000 (SAP# 1106890, available from Degussa Flavors & Fruit Systems), were added. The reactor was sealed, and the resulting mixture was stirred for 4 hours at about 55-60 degrees C. The cooling portion of the heating and cooling lab apparatus was then turned on, and the mixture was stirred overnight at about 5-10 degrees C. The mixture was then emulsified using a high shear tank mixer (HP 51PQ mixer, available from Silverston Machines Ltd., Chesham England). A percent retention of about 5.06 wt % of lemon lime oils in the cyclodextrin inclusion complex was achieved.

EXAMPLE 30: FLAVOR COMPOSITION COMPRISING CYCLODEXTRIN-ENCAPSULATED LEMON LIME OILS AND EXCESS UNCOMPLEXED CYCLODEXTRIN USED IN BEVERAGE PRODUCT

[00163]  The dry powder resulting from Example 28, and/or the emulsion resulting from Example 29 including the cyclodextrin-encapsulated lemon lime oils is dry blended with additional β-cyclodextrin to achieve a wt % of about 1 wt % of lemon lime oils in the resulting dry powder mixture (“lemon lime oils-cyclodextrin/cyclodextrin blend”). The lemon lime oils-cyclodextrin/cyclodextrin blend is then added to a beverage in a wt % ranging from about 0.05 wt % to about 0.30 wt % of the dry powder mixture (i.e., β-cyclodextrin-encapsulated lemon lime oils plus additional β-cyclodextrin) to the total weight of the beverage. This is expected to provide 50-100 ppm of lemon lime oils and from about 0.05 wt % to about 0.30 wt % of β-cyclodextrin to the beverage, depending on the amount of dry powder mixture added to the beverage.
EXAMPLE 31: CYCLODEXTRIN INCLUSION COMPLEX WITH β-CYCLODEXTRIN AND CITRAL, PECTIN AS AN EMULSIFIER AND XANTHAN GUM AS A THICKENER, AND PROCESS FOR FORMING SAME

[00164] In a 1-L reactor, 300 g of β-cyclodextrin (W7 β-cyclodextrin, available from Wacker), 6 g of beet pectin (2 wt % of pectin: β-cyclodextrin; XPQ EMP 4 beet pectin available from Degussa-France), and 0.90 g xanthan gum (KELTROL xanthan gum, available from CP Kelco SAP No. 15695) were dry blended together to form a dry blend. 575 mL of deionized water were added to the dry blend to form a slurry or mixture. The 1-L reactor was set up for heating and cooling via a lab-scale water bath heating and cooling apparatus. The mixture was agitated by stirring for about 30 min. 18 g of citral (natural citral, SAP No. 921565, Lot No. 10000223137, available from Citrus & Allied), were added. The reactor was sealed, and the resulting mixture was stirred for 4 hours at about 55-60 degrees C. The cooling portion of the heating and cooling lab apparatus was then turned on, and the mixture was stirred over the weekend at about 5-10 degrees C. The mixture was then divided into two halves. One half was emulsified neat using a high shear tank mixer (HP 5 1PQ mixer, available from Silverston Machines Ltd., Chesham England). 1 wt % gum acacia was added to the other half, and the resulting mixture was emulsified using the same high shear tank mixer. A percent retention of about 2.00 wt % of citral in the cyclodextrin inclusion complex was achieved.

EXAMPLE 32: FLAVOR EMULSION COMPRISING CYCLODEXTRIN-ENCAPSULATED CITRAL USED IN FOOD OR BEVERAGE PRODUCT

[00165] One or both of the resulting emulsions from Example 31 including the cyclodextrin-encapsulated citral is added directly to a food or beverage product to obtain a stable product with the appropriate flavor profile. The emulsions are added directly to a food or beverage product, or sprayed onto a food substrate.

EXAMPLE 33: FLAVOR EMULSION COMPRISING CYCLODEXTRIN-ENCAPSULATED CITRAL AND EXCESS UNCOMPLEXED CYCLODEXTRIN USED IN A BEVERAGE PRODUCT

[00166] One (or a mixture of both) of the resulting emulsions formed according to Example 31 including the cyclodextrin-encapsulated citral is combined with additional β-
cycloextrin to achieve a wt % of about 1 wt % of citral in the resulting flavor emulsion ("citral-cycloextrin/cycloextrin emulsion"). The citral-cycloextrin/cycloextrin emulsion is added to a beverage in a wt % ranging from about 0.05 wt % to about 0.30 wt % of the flavor emulsion (i.e., β-cyclodextrin-encapsulated citral plus additional β-cyclodextrin) to the total weight of the beverage. This is expected to provide 10-20 ppm of citral and from about 0.05 wt % to about 0.30 wt % of β-cyclodextrin to the beverage, depending on the amount of flavor emulsion added to the beverage. One of ordinary skill in the art will recognize that the excess uncomplexed β-cyclodextrin need not first be added to the flavor emulsion, but rather the excess uncomplexed β-cyclodextrin and a flavor emulsion formed according to Example 31 can be added simultaneously to a beverage product.

EXAMPLE 34: SENSORY ANALYSIS OF LEMONADE BEVERAGE COMPRISING CYCLODEXTRIN-ENCAPSULATED CITRAL VS. CONTROL LEMONADE BEVERAGE

[00167] Encapsulated citral was produced according to the method set forth in Example 18. The resulting dry powder including the cycloextrin-encapsulated citral was dry blended with additional β-cyclodextrin to achieve a wt % of about 1 wt % of citral in the resulting dry powder mixture ("citral-cycloextrin/cycloextrin blend"). The citral-cycloextrin/cycloextrin blend then blended with standard spray-dried lemon oil flavor 073-00531 (32.0 parts) (Degussa Flavors & Fruit Systems) to form a flavor composition. The flavor composition was added to a lemonade beverage base in a wt % of about 0.2 wt % of the dry powder mixture (i.e., β-cyclodextrin-encapsulated citral plus additional β-cyclodextrin) to the total weight of the beverage. The lemonade beverage base included 10.5 g of the flavor composition, 0.54 g of sugar, 0.04 g of citric acid, 0.13 g of sodium benzoate, and 88.79 g water. This provided 10 ppm of citral and about 0.2 wt % of β-cyclodextrin to the acidic beverage. This beverage was identified as "CD" for the sensory analysis illustrated in FIGS. 16 and 17.

[00168] A first control flavor composition was prepared by combining a spray-dried citral (natural citral, SAP No. 921565, Lot No. 10000223137, available from Citrus & Allied) and spray-dried lemon oil flavor 073-00531 (32.0 parts) (Degussa Flavors & Fruit Systems). The spray-dried forms of the flavors were prepared according to standard spray-dry procedures known to those of ordinary skill in the art. The first control flavor composition was added to the same lemonade base beverage as described above to create a first control lemonade
beverage having a citral flavor level of 10 ppm. The results of the sensory analysis comparing the first control lemonade beverage with the CD beverage are shown in FIG. 16. The sensory analysis was performed after the beverages had been stored in the dark at 110 degrees F for 3 weeks to simulate an aged beverage. The sensory analysis was a descriptive analysis performed by a trained sensory panel of six expert tasters, using a consensus approach and reference standards. As shown in FIG. 16, the CD beverage had a similar overall flavor intensity, a similar peely flavor, a higher fresh lemon flavor, and a lower fatty/waxy, oxidized, phenolic, acetophenone and camphoraceous flavor than the first control lemonade beverage. This sensory analysis illustrates the ability of cyclodextrin in stabilizing the key note flavor, citral, and in preventing the formation of off-note flavors that detract from and diminish the fresh lemon flavor of a lemonade beverage.

[00169] A second control flavor composition was prepared by combining an emulsion of citral (natural citral, SAP No. 921565, Lot No. 10000223137, available from Citrus & Allied) and lemon oil flavor 073-00531 (Degussa Flavors & Fruit Systems). The emulsion was prepared according to standard emulsifying procedures known to those of ordinary skill in the art. The second control flavor composition was added to the same lemonade base beverage as described above to create a second control lemonade beverage having a citral flavor level of 10 ppm. The results of the sensory analysis comparing the second control lemonade beverage with the CD beverage are shown in FIG. 17. The sensory analysis was performed after the beverages had been stored in the dark at 110 degrees F for 3 weeks to simulate an aged beverage. The sensory analysis was a descriptive analysis performed by a trained sensory panel of six expert tasters, using a consensus approach and reference standards. As shown in FIG. 17, the CD beverage had a similar overall flavor intensity, a similar peely flavor, a higher fresh lemon flavor, and a lower fatty/waxy, oxidized, phenolic, acetophenone and camphoraceous flavor than the second control lemonade beverage. This sensory analysis illustrates the ability of cyclodextrin in stabilizing the key note flavor, citral, and in preventing the formation of off-note flavors that detract from and diminish the fresh lemon flavor of a lemonade beverage. As illustrated by comparing FIGS. 16 and 17, the second control lemonade beverage had higher perceived levels of oxidized and acetophenone flavors than the first control lemonade beverage. This could be because the second control flavor composition was in a liquid form, which could have led to a more accelerated degradation of key note flavors and off-note formation.
EXAMPLE 35: CYCLODEXTRIN INCLUSION COMPLEX WITH β-CYCLODEXTRIN AND CITRAL, PECTIN AS AN EMULSIFIER AND XANTHAN GUM AS A THICKENER, AND PROCESS FOR FORMING SAME

[00170] In a 5-L reactor a base formula of 86.25 g of β-cyclodextrin (W7 β-cyclodextrin, available from Wacker), 1.70 g of beet pectin (2 wt % of pectin: β-cyclodextrin; XPQ EMP 4 beet pectin available from Degussa-France), and 0.35 g xanthan gum (KELTROL xanthan gum, available from CP Kelco SAP No. 15695) were dry blended together to form a dry blend. 216.50 mL of deionized water were added to the dry blend to form a slurry or mixture. The 5-L reactor was set up for heating and cooling via a lab-scale water bath heating and cooling apparatus. The mixture was stirred for about 30 min. 11.7 g of citral (natural citral, SAP No. 921565, Lot No. 10000223137, available from Citrus & Allied) were added. This base formulation was scaled to produce 2200 g. The reactor was sealed, and the resulting mixture was stirred for 4 hours at about 55-60 degrees C. The cooling portion of the heating and cooling lab apparatus was then turned on, and the mixture was stirred overnight at about 5-10 degrees C. The mixture was then spray dried on a Niro Basic Lab Dryer (Niro Corp. Columbia, Maryland) having an inlet temperature of approximately 210 degrees C and an outlet temperature of approximately 105 degrees C. A percent retention of about 11.5 wt % of citral in the cyclodextrin inclusion complex was achieved.

EXAMPLE 36: CYCLODEXTRIN INCLUSION COMPLEX WITH β-CYCLODEXTRIN AND LEMON OIL 3X, PECTIN AS AN EMULSIFIER AND XANTHAN GUM AS A THICKENER, AND PROCESS FOR FORMING SAME

[00171] In a 5-L reactor, a base formulation of 92.95 g of β-cyclodextrin (W7 β-cyclodextrin, available from Wacker), 1.8 g of beet pectin (2 wt % of pectin: β-cyclodextrin; XPQ EMP 4 beet pectin available from Degussa-France), and 0.35 g xanthan gum (KELTROL xanthan gum, available from CP Kelco SAP No. 15695) were dry blended together to form a dry blend. 235.00 mL of deionized water were added to the dry blend to form a slurry or mixture. The 5-L reactor was set up for heating and cooling via a lab-scale water bath heating and cooling apparatus. The mixture was stirred for about 30 min. 4.9 g of 3X California lemon oil (available from Citrus & Allied) were added. The base formula was scaled up to produce 2200 g of product. The reactor was sealed, and the resulting mixture was stirred for 4 hours at about 55-60 degrees C. The cooling portion of the heating and
cooling lab apparatus was then turned on, and the mixture was stirred overnight at about 5-10
degrees C. The mixture was then spray dried on a on a Niro Basic Lab Dryer (Niro Corp.
Columbia, Maryland) having an inlet temperature of approximately 210 degrees C and an
outlet temperature of approximately 105 degrees C. A percent retention of about 5 wt % of
lemon oil 3X in the cyclodextrin inclusion complex was achieved.

EXAMPLE 37: OFF-NOTE FORMATION COMPARISON OF LEMONADE BEVERAGE
COMPRISING CYCLODEXTRIN-ENCAPSULATED CITRAL, CYCLODEXTRIN-
ENCAPSULATED LEMON OIL 3X, AND EXCESS UNCOMPLEXED
CYCLODEXTRIN VS. A CYCLODEXTRIN-FREE CONTROL BEVERAGE

[00172] A lemonade base was prepared by combining 89.79 g water, 9.42 g of granulated
sugar, 0.04 g of finely granulated sodium citrate, and 0.50 g of citric acid (anhydrous, fine).
A preservative was not added to the beverage, but the beverage was subjected to a
pasteurization hot pack. This base was scaled to produce 8L finished beverage.

[00173] A beverage identified as “CD” was formed comprising a citral-cyclodextrin
inclusion complex formed according to Example 35 (“citral-CD”) and a lemon oil 3X-
cyclodextrin inclusion complex formed according to Example 36 (“lemon-CD”). A “CD”
flavor composition was prepared by dry blending 32.00 g of spray-dried lemon oil (073-
00531 available from Degussa Flavors & Fruit System), 5.20 g of citral-CD (073-00339
available from Degussa Flavors & Fruit System), 3.20 g of lemon-CD, and 59.60 g of excess
uncomplexed β-cyclodextrin (W7 β-cyclodextrin, available from Wacker). The CD flavor
composition was blended until uniform and screened using an approximately 30-mesh screen.
The CD beverage was then prepared by adding 0.25 g of the CD flavor composition to the
lemonade base.

[00174] A control flavor composition was prepared by dry blending 32.00 g of spray-dried
lemon oil, 5.20 g of spray-dried citral, and 3.20 g of spray-dried lemon oil 3X with 59.60 g of
maltodextrin (all sprayed on maltodextrin (SAP No. 15433 available from Tate & Lyle).
Each of the spray-dried flavors were spray-dried with maltodextrin according to standard
spray-drying procedures known to those of ordinary skill in the art. The control flavor
composition was completely free of cyclodextrin. A control beverage (referred to as
“Unprotected”) was prepared by adding 0.25 g of the control flavor composition to the
lemonade base.
The flavor retention and off-note formation of the CD beverage was compared to that of the control beverage. The amount of citral and off-notes were determined using Solid Phase Dynamic Extraction (SPDE), which is an analytical headspace technique that allows a high degree of automation and sensitivity with minimal sample preparation time. SPDE has the same sub parts-per-million sensitivity as liquid-liquid extraction and distillation techniques but does not expose the sample to temperature extremes or use large amounts of solvents that can add contaminants and which need to be removed before analysis. SPDE uses a 2 mL static headspace syringe with the inner needle wall coated with an absorbent polymer (carboxen - available from Chromsys, Alexandria VA). The analytical sample is placed in a 10 mL crimp-top vial. By repetitively drawing the headspace, which exists above the analytical sample, over the polymer layer, the organics are trapped in the polymer until thermally desorbed into the injection port of a gas chromatograph (GC) or GC-Mass Spectrometer (a PEGASUS II Time-of-flight mass spectrometer was used in this study (GC/TOF-MS; available from LECO Corp., St. Joseph, Michigan). The GC was an Agilent 6890 and the analysis performed on a 60 meter -x- 0.32 mm – carbowax column with a 1 micron film thickness (available from Restek Bellefonte, PA). Concentration effects on the order of 100,000 to 1,000,000 are easily obtained. In this study, 2 mL of each sample were placed in a 10 mL vial, which was thermostated at 50 degrees C for 10 min. and extracted for 12 min. to obtain sub-parts-per-million sensitivity.

Flavor retention and total off-note growth at 88 degrees F is shown for the Unprotected beverage and the CD beverage in FIG. 18. (The lighter bar represents the key note flavor (i.e., citral), and the darker bar represents the total off-note growth for the Unprotected beverage and the CD beverage.) As shown in FIG. 18, the CD beverage retained the key note flavor (i.e., citral) longer than the Unprotected beverage, and the CD beverage had observably lower total off-note formation than the Unprotected beverage. The formation of four types of off-notes were measured over time (i.e., after 21 days of storage at 88 degrees F, after 33 days of storage at 88 degrees F, and after 42 days of storage at 88 degrees F in both beverages, and the results are shown in FIG. 19. Namely, the four off-notes that were analyzed were p-methyl acetophenone, p-cymen-8-ol, mentha-1,5-dien-8-ol 1 and mentha-1,5-dien-8-ol 2. As shown in FIG. 19, the CD beverage formed lower levels of all four off-notes than the Unprotected beverage, and particularly, formed lower levels of p-cymen-8-ol than the Unprotected beverage.
EXAMPLE 38: PROTECTIVE EFFECTS IN “SUN-STRUCK” PHENOMENON OFFERED BY β-CYCLODEXTRIN.

To study other protective effects offered by the incorporation of cyclodextrins into beverage products preliminary studies into the “Sun–Struck” (photooxidation) phenomenon were undertaken. Specifically, the sun exposure experienced by commercial products was studied. As in EXAMPLE 20, citral (natural citral, SAP No. 921565, available from Citrus & Allied) was diluted in ethanol at a level of 1.0%. Two simulated beverage bases were made: control, 0.6% citric acid in water and protected, 0.6% citric acid and 0.2% β-cyclodextrin in water. The 1.0% citral in ethanol solution was added to each beverage base at 0.1% (10 ppm citral); both simulated beverages were in glass juice bottles and placed in a lab window with south-east exposure that experiences strong sunlight for 5 days. Duplicate bottles of each simulated beverage were placed in an oven and maintained at 110 degrees F. After 5 days each bottle was sampled and analyzed by the same headspace methods employed throughout this research (SPDE). The results are shown graphically in FIG. 20. Very little information is available on citral photo-stability, however, an examination of the offnotes in the unprotected sample shows very similar compounds and concentrations. It is, therefore, assumed that a similar reaction pathway is active in thermal and photo catalyzed degradation in acidic media (see, e.g., FIG. 7). In FIG. 20, the protected sample (labeled BCD) shows no formation of the reactive intermediate offnote p-mentha-dien-8-ol compared to the un-protected (labeled CIT). It is also evident that the formation of p-cymene is much reduced in the protected system.

All patents, publications and references cited herein are hereby fully incorporated by reference. In case of conflict between the present disclosure and incorporated patents, publications and references, the present disclosure should control. Various features and aspects of the invention are set forth in the following claims.
CLAIMS

What is claimed is:

1. A method for making a guest stabilizing system, the method comprising:
   mixing cyclodextrin and an emulsifier to form a mixture;
   mixing a solvent and a guest with the mixture to form a cyclodextrin inclusion complex;
   adding uncomplexed cyclodextrin to the cyclodextrin inclusion complex to form a guest stabilizing system.

2. The method of claim 1, wherein the guest comprises at least one of a flavor, an olfactant, a pharmaceutical, a nutraceutical, an antioxidant, and a combination thereof.

3. The method of claim 1, wherein the guest comprises at least one of diacetyl, citral, benzaldehyde, acetaldehyde, an essential oil, aspartame, creatine, alpha-tocopherol, and a combination thereof.

4. A method of making an end product comprising adding the guest stabilizing system of claim 1 to an end product.

5. The method of claim 4, wherein the end product comprises at least one of a beverage, a food product, a chewing gum, a dentifrice, a candy, a flavoring, a fragrance, a pharmaceutical, a nutraceutical, a cosmetic, an agricultural product, a photographic emulsion, a waste stream system, and a combination thereof.

6. The method of claim 4, wherein the end product comprises a beverage, and wherein the weight percentage of guest stabilizing system to the beverage ranges from about 0.05 wt % to about 0.3 wt % to obtain a desired flavor profile in the beverage, and wherein the weight percentage of cyclodextrin to the beverage ranges from about 0.05 wt % to about 0.3 wt %.

7. The method of claim 1, wherein the guest has a positive log (P) value.
8. The method of claim 1, wherein the guest has a log (P) value of at least about +2.

9. The method of claim 1, wherein the emulsifier comprises pectin, and the solvent comprises water.

10. The method of claim 1, wherein mixing a solvent and a guest with the mixture forms a second mixture comprising the cyclodextrin inclusion complex, and further comprising drying the second mixture to form a dry powder comprising the cyclodextrin inclusion complex.

11. The method of claim 10, wherein drying the second mixture comprises at least one of air drying, vacuum drying, spray drying, oven drying, and a combination thereof.

12. The method of claim 10, wherein the uncomplexed cyclodextrin is dry blended with the dry powder.

13. The method of claim 1, wherein the guest is added in an excess molar ratio of guest to cyclodextrin.

14. The method of claim 1, wherein the cyclodextrin comprises β-cyclodextrin.

15. The method of claim 1, wherein the cyclodextrin comprises a combination of α-cyclodextrin and β-cyclodextrin.

16. A method for making a guest stabilizing system, the method comprising: mixing cyclodextrin, a solvent and a guest to form a cyclodextrin inclusion complex, the guest being added in an excess molar ratio of guest to cyclodextrin; adding uncomplexed cyclodextrin to the cyclodextrin inclusion complex to form a guest stabilizing system, the uncomplexed cyclodextrin being added in an excess molar ratio of total cyclodextrin to guest to increase the ratio of complexed guest to free guest in the guest stabilizing system to further stabilize the guest from degradation.
17. The method of claim 16, wherein the guest comprises at least one of a flavor, an olfactant, a pharmaceutical, a nutraceutical, an antioxidant, and a combination thereof.

18. The method of claim 16, wherein the guest comprises at least one of diacetyl, citral, benzaldehyde, acetaldehyde, an essential oil, aspartame, creatine, alpha-tocopherol, and a combination thereof.

19. The method of claim 16, wherein the guest has a negative log (P) value.

20. A method of making an end product comprising adding the guest stabilizing system of claim 16 to an end product.

21. The method of claim 20, wherein the end product comprises at least one of a beverage, a food product, a chewing gum, a dentifrice, a candy, a flavoring, a fragrance, a pharmaceutical, a nutraceutical, a cosmetic, an agricultural product, a photographic emulsion, a waste stream system, and a combination thereof.

22. The method of claim 20, wherein the end product comprises a beverage, and wherein the weight percentage of guest stabilizing system to the beverage ranges from about 0.05 wt % to about 0.3 wt % to obtain a desired flavor profile in the beverage, and wherein the weight percentage of cyclodextrin to the beverage ranges from about 0.05 wt % to about 0.3 wt %.

23. The method of claim 16, wherein mixing the cyclodextrin, the solvent and the guest forms a mixture comprising the cyclodextrin inclusion complex, and further comprising drying the mixture to form a dry powder comprising the cyclodextrin inclusion complex.

24. The method of claim 23, wherein drying the second mixture comprises at least one of air drying, vacuum drying, spray drying, oven drying, and a combination thereof.

25. The method of claim 23, wherein the uncomplexed cyclodextrin is dry blended with the dry powder.

26. The method of claim 16, wherein the cyclodextrin comprises β-cyclodextrin.
27. A method for making a beverage, the method comprising:
mixing uncomplexed cyclodextrin, a guest and a solvent to form a beverage,
the guest having a positive log (P) value,
the cyclodextrin being added to the beverage in a weight percentage of
cyclodextrin to the beverage ranging from about 0.05 wt % to about 0.3 wt %.

28. The method of claim 27, wherein the cyclodextrin comprises β-cyclodextrin.

29. The method of claim 27, wherein the guest comprises at least one of a flavor, an
olfactant, a pharmaceutical, a nutraceutical, an antioxidant, and a combination thereof.

30. The method of claim 27, wherein the guest comprises at least one of diacetyl,
citral, benzaldehyde, acetaldehyde, an essential oil, aspartame, creatine, alpha-tocopherol,
and a combination thereof.

31. The method of claim 27, wherein the guest has a log (P) value of at least about
+1.

32. The method of claim 27, wherein the cyclodextrin is added in a weight
percentage of cyclodextrin to the beverage ranging from about 0.15 wt % to about 0.2 wt %.

33. The method of claim 27, wherein the cyclodextrin is added in a weight
percentage of cyclodextrin to the beverage of about 0.2 wt %.

34. The method of claim 27, wherein the guest has a concentration in the beverage
ranging from about 5 ppm to about 100 ppm.

35. The method of claim 27, wherein the guest comprises citral, and wherein the
citral has a concentration in the beverage ranging from about 10 ppm to about 15 ppm.

36. The method of claim 27, further comprising forming a cyclodextrin inclusion
complex in the beverage between the uncomplexed cyclodextrin and the guest to stabilize the
guest, the formation of the cyclodextrin inclusion complex being at least partially dependent on the magnitude of the log (P) value of the guest.

37. The method of claim 27, wherein the molar ratio of cyclodextrin:guest in the beverage is greater than 1:1.
FIG. 3

diacetyl

cyclodextrin complex
FIG. 5

cycloextrin + citral → complex

CHO

CHO
FIG. 7

FIG. 7A

\[ \text{gas} \xrightarrow{K_{P1}} \text{pseudophase} \xleftarrow{K_{P2}} \text{solution} \]

\[ K_{H} \]
FIG. 8

FIG. 9
FIG. 10

3mL (1% citral in EtOH) per 2L 0.6% citric

FIG. 11

citral protected with β-CD
FIG. 7

Total Area Count (Hot Samples)

FIG. 12

Offnote Formation (Hot Samples)

FIG. 13
**FIG. 14**

**Total Area Count (Cold Samples)**

**FIG. 15**

**Offnote Formation (Cold Samples)**
FIG. 16

- Control
- CD

Overall Flavor Intensity

Fresh Lemon

Fatty/waxy

Phenolic

Acetophenone

Camphoraceous

Oxidized

Peely
FIG. 17

- Control
- CD

Overall Flavor Intensity

Fresh Lemon
Peely
Fatty/Waxy

Camphoraceous
Acetophenone
Phenolic
Oxidized

70
60
50
40
30
20
10
0
INTERNATIONAL SEARCH REPORT

A. CLASSIFICATION OF SUBJECT MATTER
   IPC: C07G 17/00 (2007.01); C07H 1/00 (2007.01); C08B 37/00 (2007.01)
   According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED
   Minimum documentation searched (classification system followed by classification symbols)
   Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
   Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
   Please see continuation sheet

C. DOCUMENTS CONSIDERED TO BE RELEVANT

<table>
<thead>
<tr>
<th>Category</th>
<th>Citation of document, with indication, where appropriate, of the relevant passages</th>
<th>Relevant to claim No.</th>
</tr>
</thead>
</table>

Further documents are listed in the continuation of Box C.

See patent family annexes:

- "A" document containing the general state of the art which is not considered to be of particular relevance
- "B" earlier application or patent published on or after the international filing date
- "C" document which may throw doubts on priority claims or which is close to enabling the publication date of another Claim or other special reason as specified
- "D" document referring to an oral disclosure, test, exhibition or other means

Document published prior to the international filing data but later than the priority data obtained

Date of the actual completion of the international search: 07 August 2006 (07.08.2006)

Authorized officer: Date: 24 AUG 2006

Devishi Khuro
Telephone No.: 703-306-1235

Form PCT/ISA/210 (second sheet) (April 2005)
Continuation of B. FIELDS SEARCHED Item 3:
CAS online, EAST. Search terms used: cyclodextrin inclusion complexes, guest stabilizing system and methods for preparing and using the same.