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(54) Title: ALCOHOLIC COMPOSITIONS

(57) Abstract: A method for manufacturing alcoholic liquid compositions, the method comprises the steps of: • providing an alcoholic liquid composition; • contacting unhydrated hydrocolloid with the alcoholic liquid composition to provide a hydrocolloid composition; • shearing the resulting hydrocolloid composition at a temperature of 60°C or below to hydrate the hydrocolloid.



Alcoholic Compositions

This invention relates generally to alcoholic compositions and to alcoholic compositions in which particles may be suspended. More specifically, although not exclusively, this invention relates to method for manufacturing alcoholic compositions and alcoholic compositions in which particles may be suspended.

There are a range of applications in which it is desired to provide an alcoholic composition, for example personal care products. In some alcoholic compositions it is desired to suspend particles therein. Example applications for alcoholic compositions containing suspended particles include use in cosmetics and personal care products, or in cleansers and toners containing abrasive particles for skin exfoliation. Other applications for alcoholic liquid compositions containing suspended particles may include pharmaceutical delivery systems, cleaning agents, and adhesives.

It is also known to suspend particles, such as gold leaf, edible beads, or fruit, in alcoholic beverages to enhance the aesthetic appeal to the customer. It is desirable for the suspended particles to remain evenly distributed throughout the beverage over a period of time, without sedimentation of particles at the bottom of the beverage, agglomeration of particles or particles floating to the top of the beverage. This enhances the visual appeal during display of the beverage in a transparent container, such as a glass bottle.

Several methods are reported for achieving an even distribution of particles in an alcoholic beverage, including the use of hydrocolloids. Hydrocolloids are compounds that swell when in the presence of water to form a gel-like material. The solid hydrocolloid may be heated in water to form the gel structure; the concentration and treatment conditions may be varied to achieve the desired properties for a specific application.

One such method that utilises a hydrocolloid for suspending particles in an alcoholic liquid composition is described in US2012/0107468A1. Specifically, gellan gum is used. The method described is a two-step process requiring the initial preparation of a non-alcoholic pre-gel solution containing deionised water, gellan gum, and sodium citrate, which is heated to 85 or 90°C under agitation. A second solution is prepared, which contains citric acid and alcohol. The two solutions are mixed together at a temperature of between 21 to 48°C, followed by rapid cooling and addition of the desired particles for suspension in the alcoholic liquid composition. It is clear that this method has a number of significant drawbacks

including the use of high temperatures (85 or 90°C), which is not energy efficient, the pre-gel solution has to be maintained above the setting point of the gellan, and the requirement for the preparation of two separate solutions before mixing to form the final composition.

5 Accordingly, there is a desire to have a method for manufacturing alcoholic liquid compositions, which may be used for suspending particles, using hydrocolloids, which does not require the use of high temperatures (such as between 85 to 90°C), and is preferably a single step process.

10 A first aspect of the invention provides a method for manufacturing alcoholic liquid compositions, the method comprising the steps of:

- a) providing an alcoholic liquid composition;
- b) contacting unhydrated hydrocolloid with the alcoholic liquid composition to provide a hydrocolloid composition;

15 c) shearing the hydrocolloid composition at a temperature of 60°C or below to hydrate the hydrocolloid.

There may be provided a further step d) in which a plurality of particles are added to the alcoholic liquid composition and/or the hydrocolloid composition.

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A second aspect of the invention provides a method for suspending particles in an alcoholic liquid composition, the method comprising the steps of:

- a) providing an alcoholic liquid composition;
- b) adding unhydrated hydrocolloid to the alcoholic liquid composition to provide a hydrocolloid composition;
- 25 c) shearing the hydrocolloid composition at a temperature of 60°C or below to hydrate the hydrocolloid; and
- d) adding a plurality of particles to the alcoholic liquid composition and/or the hydrocolloid composition.

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The order in which the step d) is performed is not limited to that which is alphabetically specified. For example, step d) may be performed before, during or after step b) or before, during or after step c).

It has been surprisingly found that hydrocolloids may be hydrated in an alcoholic liquid composition, for example an aqueous alcoholic solution, at low temperatures (at or below 60°C, for example, at or below 50°C, say 45 °C, at or below 40°C, say 35°C and in embodiments at or below 30°C) using the method of the present invention. In some
5 embodiments the temperature is maintained above 10°C, say above 11, 12, 13, 14, 15, 16, 17, 18, 19, 20°C during step c). Preferably the temperature is maintained for the entire period of shearing. Surprisingly, the method may be performed at a temperature which is less than the temperature at which the hydrocolloid(s) used in the process would usually hydrate in the absence of sequestrants (*i.e.* the hydration temperature). Advantageously,
10 the formation of hydrated gels at low temperatures, instead of above say 60°C, leads to a different gel structure with beneficial properties for a number of applications.

Advantageously, the particles in the gel structure are such that the gel has low yield stress. Upon application of a yield stress, the gel undergoes shear thinning, in which the viscosity
15 of the gel decreases. This behaviour is advantageous for many applications of the fluid gel including, for example, personal care products such as hand gels and/or enables the gel to be pourable upon application of a shear stress.

The alcoholic liquid composition of step a) may be an aqueous composition comprising
20 alcohol and water. Most typically, this is a mixture of water and ethanol. However, it is within the scope of the invention that other solvents may be present, for example, in cosmetics and personal care products. These solvents may include acetone, ethyl acetate, isopropanol, and/or hydrophobic substances such as mineral oils to form biphasic solutions. Emulsifiers may be present to form emulsions with hydrophobic substances. One or more
25 of flavourants, sweeteners (for example sugars), preservatives, buffers and/or colourants may be provided.

There is no particular restriction on the type of alcohol used. The alcohol may be one or more of methanol, ethanol, propan-1-ol, propan-2-ol, *n*-butanol, *sec*-butanol, isobutanol,
30 *tert*-butanol, isomers of one or more of pentanol, hexanol, heptanol, octanol, nonanol and decanol. The alcohol may be a diol (*e.g.* ethylene glycol) or a triol. Typically, the alcohol is ethanol. One type of alcohol may be present in the alcoholic liquid composition, or a mixture of two or more may be used in combination.

The concentration of the alcohol may be more than 4 or 5% by weight in the alcoholic liquid composition. Typically, the concentration of alcohol is more than 10% by weight, more typically this is more than 20% by weight, and even more typically is 30% by weight in the alcoholic liquid composition. Most typically, the concentration of alcohol is more than 40%
5 by weight in the alcoholic liquid composition. In some embodiments, the concentration of alcohol is more than 50% by weight in the alcoholic liquid composition. The target alcohol concentration in the final composition may be from 4 to 50 v/v%, for example from 4 to 15 v/v % or from 15 to 30 v/v% or say from 30 to 50 or 35 to 45v/v%. A comestible composition with an alcohol concentration in the range of 30 to 40 or 50 v/v% may be termed a spirit. A
10 comestible composition with an alcohol concentration in the range of from 4 to 15 v/v% may include pre-mixed drinks comprising a spirit and a mixer. Other comestible compositions may have alcohol concentrations in the range of from 15 to 30 v/v%.

The term "hydrocolloid" as used herein is intended to take a typical meaning, that being a
15 material which is hydrophilic and which, in the presence of water, swells to form particles. Typically, the hydrocolloid forms gels, which may form ion bridges. The hydrocolloid may be selected from one or more polymers. In some embodiments, the hydrocolloid may be cross-linked or cross-linkable. For comestible products, we prefer non-crosslinked gels.

20 The hydrocolloid may be a naturally occurring hydrocolloid such as, for example, a polysaccharide or may be a synthetic hydrocolloid such as, for example, polyacrylates, and polyethylene glycol. The hydrocolloid species may be selected from one or more of agar, agarose, arabinoxylan, carrageenan, gelatin, gellan gum, glucan, curdlan, pectin, xanthan gum, gum arabic, guar gum, locust bean gum, gum tragacanth, gum karaya, cellulose and
25 derivatives thereof, alginate, fibrin, or starch, or combinations thereof. Other suitable hydrocolloid species include chitosan, dextran, collagen and hyaluronic acid. Examples of cellulose derivatives may be those compounds wherein one or more of the hydroxyl groups have been functionalised. These groups may be reacted to form alkoxy groups, alkoxycarboxylic acid groups, alkoxyesters, alkoxyethers, or combinations thereof. Typical
30 cellulose derivatives include carboxymethyl cellulose, methyl cellulose and ethyl cellulose.

In embodiments, the hydrocolloid may be selected from carrageenan, gelatin, gellan gum, agar, alginate, cellulose, cellulose derivatives and combinations thereof.

In one embodiment, carrageen is used and, in another embodiment, kappa carrageenan and/or iota carrageenan are used. Carrageenans are natural products derived from seaweed (red algae). The three molecular forms are iota, kappa and lambda, which are all linear polysaccharides consisting of repeating galactose and 3,6 anhydrogalactose units, the main difference being the extent of sulphation of the disaccharide repeating unit; kappa carrageenan has a single sulphate per disaccharide, iota has two sulphates per disaccharide and lambda has three sulphates per disaccharide. Kappa and iota carrageenan form a helical structures in a gel state. However, lambda carrageenan does not gel.

In a further embodiment, gellan gum is used, and in another embodiment, low acyl gellan gum is used, although high acyl gellan gum may be used. Gellan gum is a naturally occurring anionic hydrocolloid. The structure is a repeating tetrasaccharide unit (1,3- β -D-glucose, 1,4- β -D-glucuronic acid, 1,4- β -D-glucose, 1,4- α -L-rhamnose). Low acyl gellan gum is produced via alkaline hydrolysis of the acyl groups along the native backbone of high acyl gellan gum, which results in a polyanionic chain of carboxylate moieties.

Kappa carrageenan, iota carrageenan, gellan gum, and agar are classed as physical gels because they form thermoreversible interactions such as hydrogen bonds, hydrophobic associations, and cation mediated crosslinking. This is in contrast with synthetic polymers, which form permanent interactions such as covalent bonds.

In this invention we prefer to use physical gels.

The concentration of the hydrocolloid in the alcoholic liquid composition may be any suitable concentration for the properties desired and depending on the application. The amount of hydrocolloid used may be less than 25% by weight of the alcoholic liquid composition, for example, less than 20% by weight, and say less than 10% by weight. The amount of hydrocolloid used may be less than 5% by weight of the alcoholic liquid composition, and may be less than 3% by weight, say less than 2% by weight or less than 1% by weight of the alcoholic liquid composition. In comestible products such as beverages it is preferred that the amount of hydrocolloid present is as low as possible.

A single hydrocolloid species may be used. However, it is also possible for more than one hydrocolloid species to be used in a mixture or in combination.

The rate of shearing may be tailored to achieve the desired properties for a specific application. There is no particular restriction on the shear rate used. The only limitation that may be imposed on the shear rate is if it is desirable to perform step d), to add a plurality of particles, before step c), to shear the solution at a temperature of 60°C or below. In this case, the shear rate may be determined by the conditions that the chosen particles is able to tolerate. However, in the case of robust particles such as gold leaf, which are not easily damaged by shear forces, higher shear rates can be used.

Accordingly, the shear rate in step c) may be less than 2000 s⁻¹, for example, less than 1800 s⁻¹, say less than 1500 s⁻¹, for example, less than 1000 s⁻¹. In embodiments, the shear rate may be less than 800 s⁻¹, and may be equal to or less than 500 s⁻¹. Although there is no lower limit on the shear rate, as the shear rate decreases, the process takes longer. Accordingly, it is preferred that the shear rate is greater than 100 s⁻¹, say greater than 200 s⁻¹ and for example greater than 400 s⁻¹.

The duration of the shearing in step c) is not particularly limited and may vary depending on the temperature, choice of hydrocolloid, and if any other additives are present in the solution. Often, the shearing is conducted until no further change in the solution is observed. This may be less than one hour, say less than 30 minutes, and may be less than 10 minutes.

The shear force may be applied to the solution by any one of a number of processes that will be known to a skilled person in the art. For example, a rheometer may be used to impart the shear force. This could be a dynamic shear rheometer, pipe or capillary rheometer, rotational cylinder rheometer or cone or plater rheometer, linear rheometer or a combination thereof.

The hydration temperature of the hydrocolloid used in the present invention may be greater than 30°C, say greater than 40°C, for example greater than 50°C, 60°C, 70°C, 80°C, 90°C or greater than 100°C. In this specification, where we specify the hydration temperature, as above, it is intended to mean the hydration temperature of the hydrocolloid in a de-ionised water and alcohol mixture, in the absence of sequestrants, such as sodium citrate.

Advantageously, hydrocolloids that have high hydration temperatures, for example, greater than 80°C, may be used in the present invention without the need to heat the hydrocolloid in the de-ionised water and alcohol mixture to a temperature above 60°C, and may be performed at a much lower temperature, for example, 30°C.

5

The size of the hydrocolloid particles may be in the range of 1µm to 1000µm, for example 1µm to 500µm, 1µm to 100µm or 30µm to 50µm, or 1 to 50 µm.

10

In embodiments of the invention for use in comestible products, the suspended particles may be edible, for example, an edible metal such as gold leaf (E175) or silver leaf (E174). Other food-grade pigment particles may be used, for example pigments based on titanium, dioxide (E171) and/or iron oxides (E172) and/or potassium aluminium silicate (E555). The suspended particles may be comprised of a food product, for example, fruit pieces or flakes, a starch-based glitter, tapioca pearls, or gelatin spheres.

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In embodiments of the invention for use in, for example, cosmetic products, the suspended particles may be any acceptable particle type including non-toxic metallic particles including gold, silver, copper, platinum, schlagmetal, or may be natural particles including sand, or synthetic particles including microbeads, or plastic-based glitter.

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The suspended particles may be any shape, for example, spherical or near spherical. The suspended particles may comprise a very thin foil or metallic leaf.

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The size of the suspended particles in the alcoholic liquid composition depends on the shape, weight and/or density of each particle. This is the only limitation imposed on the size of the particles that may be suspended in the alcoholic liquid composition. The suspended particles may be visible to the naked eye and can have a diameter of more than 50µm, say 100µm, 200µm, 300µm, 500µm or 1000µm (1mm). In embodiments, the suspended particles may be nominally or substantially spherical and may be up to 5mm in diameter, say 4mm, 3mm, 2mm or 1mm. In embodiments, the suspended particles may be a foil or leaf, and may have a dimension of up to 5mm across a major surface, for example say 4mm, 3mm, 2mm, 1mm, the foil may be thin, for example say 0.1µm or 0.2µm thin.

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The amount by weight of suspended particles present in the alcoholic liquid composition will depend on the application. The suspended particles may comprise under 5% by weight

of the alcoholic liquid composition, say less than 1% by weight, for example less than 0.5% by weight, say less than 0.1% of the alcoholic liquid composition.

Typically, the alcoholic liquid composition has an ionic strength of close to zero, or of zero,
5 with no dissolved buffer components or acidic/basic, or anionic/cationic species present.

In some embodiments, the alcoholic liquid composition may be provided with a buffer composition for regulating the pH, or to aid hydration of the hydrocolloid. Suitable buffers include citric acid/sodium citrate, Dulbecco's Modified Eagle's medium (DMEM), and 4-(2-
10 hydroxyethyl)-1-piperazineethanesulfonic acid (HEPES).

Additives may be added to the alcoholic liquid composition. Typical additives for comestible products include colourings, flavourings, natural and artificial sweeteners, stabilisers, preservatives, and vitamins and minerals. There is no limitation on the types of additives
15 that may be added to the alcoholic liquid composition, as long as these are compatible with composition and do not interfere with its operation.

The inventors have surprisingly found that stable fluid gels may be prepared via hydration of hydrocolloids in alcohol and deionised water, by applying shear forces at low temperature
20 (60°C or below), in the absence of charged species.

Without wishing to be bound by theory, it is believed that, in the presence of ethanol, and during shearing at a low temperature (60°C or below), the hydrocolloid molecules undergo conformational ordering and gelation over a limited period of time to produce discrete
25 particles. Advantageously, this results in a system of gel particles suspended in a non-gelled continuous medium, which are pourable and spreadable.

This is in contrast to quiescent gels, which are formed when the hydrocolloid molecules aggregate to form bridges between particles, which imparts the quiescent gel with
30 properties such as yield stress. This is undesirable for certain applications.

A further aspect of the invention provides an alcoholic composition comprising alcohol, water and a hydrocolloid gel, wherein the hydrocolloid gel is substantially non-quiescent and formed of discrete hydrocolloid particles dispersed within the composition.

A yet further aspect of the invention provides an alcoholic composition formed in accordance with one or other of the above methods.

Discrete particles may be dispersed throughout the composition.

5

The composition may be pourable.

The composition may also display 'pourable' qualities when shear is applied, for example, when the bottle is shaken or agitated. In this specification, the term 'pourable' means that
10 once located in a 70cl bottle, upon inversion of the bottle to angle of 20° beyond horizontal (or more) the composition will flow from the neck of the bottle.

In an embodiment the alcohol is ethanol.

15 Advantageously, particles may be suspended in the alcoholic liquid compositions of the present invention, which has a range of applications in, for example, the alcoholic beverage industry, in cosmetics and personal care products, pharmaceutical delivery systems, cleaning agents, and adhesives.

20 The alcoholic compositions of the invention are also useful when particles are not suspended therein, for example as hand sanitisers and other personal care products. In those embodiments, the alcohol will be chosen as appropriate.

In order to better understand the invention, and by way of non-limiting example only,
25 reference is made to the following drawings, in which:

Figure 1 is the chemical structure of the repeating unit of kappa carrageenan;

Figure 2 is the chemical structure of the repeating unit of gellan gum;

Figure 3 shows kappa carrageenan (3% by weight) in range of concentrations of
30 ethanol (20%, 22%, 23%, 25%, 30%, and 40% by weight) in deionised water, hydrated at 30°C;

Figure 4 is a graphical representation of the percentage by weight of hydrated kappa carrageenan at the range of concentrations of ethanol in deionised water shown in Figure 3;

Figure 5 shows kappa carrageenan at a range of concentrations (1%, 2%, 3%, 4% and 5% by weight) in ethanol (20% by weight) and deionised water, hydrated at 30°C;

Figure 6A shows the differential scanning calorimetry (DSC) heating profiles of kappa carrageenan (3% by weight) in ethanol (20% by weight) and deionised water;

Figure 6B shows the differential scanning calorimetry (DSC) cooling profiles of kappa carrageenan (3% by weight) in ethanol (20% by weight) and deionised water;

Figure 7 shows kappa carrageenan (3% by weight) in ethanol (20% by weight) and deionised water, hydrated at a range of temperatures (10°C, 20°C, 30°C, 40°C);

Figure 8 shows kappa carrageenan (3% by weight) in ethanol (30% by weight) and deionised water, hydrated at a range of temperatures (10°C, 20°C, 30°C, 40°C);

Figure 9 shows kappa carrageenan (3% by weight) in ethanol (40% by weight) and deionised water, hydrated at a range of temperatures (10°C, 20°C, 30°C, 40°C);

Figure 10 is a graphical representation of the percentage by weight of hydrated kappa carrageenan at the range of hydration temperatures shown in Figures 7 to 9;

Figure 11 shows kappa carrageenan (1% by weight) in ethanol (20% by weight) and deionised water, hydrated at a range of temperatures (10°C, 20°C, 30°C, 40°C);

Figure 12 shows kappa carrageenan (1% by weight) in ethanol (30% by weight) and deionised water, hydrated at a range of temperatures (10°C, 20°C, 30°C, 40°C);

Figure 13 shows kappa carrageenan at a range of concentrations (1%, 2%, 3%, 4% and 5% by weight) in methanol (20% by weight) and deionised water, hydrated at 30°C;

Figure 14A shows the differential scanning calorimetry (DSC) heating profiles of kappa carrageenan at a range of concentrations (1%, 2%, 3%, 4% by weight) in methanol (20% by weight) and deionised water;

Figure 14B shows the differential scanning calorimetry (DSC) cooling profiles of kappa carrageenan at a range of concentrations (1%, 2%, 3%, 4% by weight) in methanol (20% by weight) and deionised water;

Figure 15 shows kappa carrageenan at a range of concentrations (1%, 2%, 3%, 4% and 5% by weight) in propanol (20% by weight) and deionised water, hydrated at 30°C;

Figure 16A shows the differential scanning calorimetry (DSC) heating profiles of kappa carrageenan (3% by weight) in propanol (20% by weight) and deionised water;

Figure 16B shows the differential scanning calorimetry (DSC) cooling profiles of kappa carrageenan (3% by weight) in propanol (20% by weight) and deionised water;

Figure 17 shows gellan gum (3% by weight) in range of concentrations of ethanol (15%, 18%, 25%, and 30% by weight) in deionised water, hydrated at 30°C;

Figure 18A shows the differential scanning calorimetry (DSC) heating profiles of a standard gel of the prior art method containing kappa carrageenan (3% by weight) in ethanol (20% by weight) and deionised water;

Figure 18B shows the differential scanning calorimetry (DSC) cooling profiles of a standard gel of the prior art method containing kappa carrageenan (3% by weight) in ethanol (20% by weight) and deionised water;

Figure 19A shows the differential scanning calorimetry (DSC) heating profiles three repeats of the standard gel of the prior art of kappa carrageenan (3% by weight) in ethanol (20% by weight) and deionised water upon heating the gels for the first time;

Figure 19B the differential scanning calorimetry (DSC) heating profiles three repeats of the standard gel of the prior art of kappa carrageenan (3% by weight) in ethanol (20% by weight) and deionised water upon heating the gels for a second time;

Figure 20 shows the heating profile used in the DSC measurements of Figures 19A and 19B.

Referring to Figures 1 and 2 there is shown the chemical structure of the repeating unit of the hydrocolloid kappa carrageenan 1 and the chemical structure of the repeating unit of the hydrocolloid gellan gum 2 respectively. In respect of Figure 2, the * and ** indicate the location of acetate group and glycerate group respectively for high acyl gellan gum.

Hydrocolloids including but not limited to, for example, kappa carrageenan 1 and low acyl gellan gum 2, form a gel, initially by molecular ordering into a helical structure, followed by aggregation and gelation. This is dependent on the ionic environment in aqueous media. For example, under certain conditions, kappa carrageenan binds monovalent ions (particularly potassium ions) to form the random coil confirmation of the carrageenan into a double helix, which in turn aggregate to form a gel.

To further exemplify the invention, reference is made to the following non-limiting examples:

Example 1 – Effect of ethanol concentration using 3% kappa carrageenan

Referring to Figure 3 there is shown six alcoholic liquid compositions containing kappa carrageenan (3% by weight) in range of concentrations of ethanol; 3a. 20%; 3b. 22%; 3c. 23%; 3d. 25%; 3e. 30%; and 3f. 40%; by weight, in deionised water. The alcoholic liquid compositions 3a-3f were prepared using the method below with a Kinexus rheometer (Malvern Instruments, UK) with cup and vane geometry:

- i. Prepare stock solutions of 20%, 22%, 23%, 25%, 30% and 40% by weight of ethanol in deionised water.
- ii. Add 29.1 g of the appropriate stock solution to the rheometer cup.
- 10 iii. Add 0.9 g of kappa carrageenan powder to the rheometer cup. For all compositions in Example 1, the total weight of the composition in the rheometer cup is 30 g.
- iv. Immediately begin the shearing process using a sequence of 30 minutes and a single shear rate of 500 s^{-1} , maintaining the temperature at 30°C , for 30 minutes.
- 15 v. Stop the shear process, transfer the composition from the rheometer cup to a sample pot. Leave the sample to stand in the sample pot for 6 hours.
- vi. Photograph for analysis (as shown in Figure 3).

Referring to Figure 4 there is shown a graphical representation of the percentage by weight of hydrated kappa carrageenan of the alcoholic liquid compositions 3a-3f of Example 1. The graph shows that as the concentration of ethanol in water increases in the samples, the weight percentage of hydrated kappa carrageenan decreases.

It is known that at low temperatures kappa carrageenan cannot be hydrated in pure deionised water and usually requires the addition of an ionic species, such as a buffer, to form a gel. As shown by the graph in Figure 4, the inventor has surprisingly found that kappa carrageenan can be hydrated effectively in deionised water containing ethanol at a concentration of above 18% by weight, in the absence of charged species, at 30°C using a shearing process.

Example 2 – Effect of increasing the concentration of kappa carrageenan in 20% by weight ethanol and deionised water

Referring to Figure 5 there is shown five alcoholic liquid compositions containing kappa carrageenan in a range of concentrations; 5a. 1%; 5b. 2%; 5c. 3%; 5d. 4%; 5e. 5%; by weight; in 20% by weight ethanol in deionised water. The alcoholic liquid compositions 5a-

5e were prepared using the method below with a Kinexus rheometer (Malvern Instruments, UK) with cup and vane geometry:

- i. Prepare a stock solution of 20% by weight of ethanol in deionised water.
- ii. Add the appropriate weight of the stock solution to the rheometer cup.
- 5 iii. Add the appropriate amount of kappa carrageenan powder to the rheometer cup. For example, a 3% by weight kappa carrageenan solution in 20% by weight ethanol and deionised water would contain 29.1 g of stock solution and 0.9 g of kappa carrageenan. For all compositions in Example 2, the total weight of the composition in the rheometer cup is 30 g.
- 10 iv. Immediately begin the shearing process using a sequence of 30 minutes and a single shear rate of 500 s^{-1} , maintaining the temperature at 30°C , for 30 minutes.
- v. Stop the shear process and leave the sample to stand for 6 hours.
- vi. Photograph for analysis (as shown in Figure 5).

Example 3 – Analysis using differential scanning calorimetry (DSC)

Referring now to Figure 6A there is shown differential scanning calorimetry (DSC) heating profiles of the hydrated gel (corresponding to alcoholic liquid composition 5c of Example 2) of kappa carrageenan (3% by weight) in ethanol (20% by weight) and deionised water.

Referring also to Figure 6B there is shown a differential scanning calorimetry (DSC) cooling profiles of the hydrated gel (corresponding to alcoholic liquid composition 5c of Example 2) of kappa carrageenan (3% by weight) in ethanol (20% by weight) and deionised water.

Two heating and cooling steps were undertaken to the alcoholic liquid composition 5c. The first heating a cooling step is shown as follows. In Figure 6A there is shown the first heating profile 61 of the first heating step, in Figure 6B there is shown the first cooling profile 63 first cooling step. The alcoholic liquid composition 5c underwent a subsequent second heating and cooling step. In Figure 6A there is shown the second heating profile 62 of the second heating step, in Figure 6B there is shown the second cooling profile 64 of the second cooling step.

Differential scanning calorimetry (DSC) measures the change in heat capacity of a material as the temperature increases or decreases. The peaks signal a phase change in the gel structure. The enthalpy and temperature of thermal transition were measured for the gel

phase using a μ DSC evo Dynamic Scanning Calorimeter (DSC) (Setaram Instrumentation, France) in the following procedure:

- i. Hydrated gel sample - Add 650 mg ($\pm$ 5 mg) of the hydrated gel 5c containing 3% kappa carrageenan in 20% ethanol and deionised water to a screw top 'closed batch cell'.
- ii. Reference sample - Add 650 mg ($\pm$ 5 mg) of deionised water to a screw top 'closed batch cell'.
- iii. Taking the hydrated gel sample initially, hold the sample isothermally at 5°C for 30 minutes.
- iv. Heat the sample to 90°C at a rate of 1.2°C/min to obtain first heating profile 61
- v. Cool the sample to 5°C at a rate of 1.2°C/min to obtain first cooling profile 62.
- vi. Hold the sample isothermally at 5°C for 30 minutes.
- vii. Heat the sample to 90°C at a rate of 1.2°C/min to obtain second heating profile 63.
- viii. Cool the sample to 5°C at a rate of 1.2°C/min to obtain second cooling profile 64.
- ix. Record the graphs of Figures 6A and 6B.

Referring to both Figures 6A and 6B, it appears that the first heating profile 61 differs from the second heating profile 62. Without wishing to be bound by theory, it is believed that preparation of the hydrated gel sample of Example 2, at a low temperature (e.g. 30°C) in conjunction with a shearing process, leads to a hydrated gel with a homogeneous structure.. This imparts advantageous rheological properties to the alcoholic liquid composition 5c, such as greater flowability or pourability, which is beneficial for use in alcoholic beverages. More advantageously, the use of kappa carrageenan in this method for alcoholic beverages does not alter the organoleptic properties of the beverage such as the taste and mouth feel. Yet more advantageously, the compositions are stable over a period of months to years.

Example 4 – Effect of temperature using 3% by weight kappa carrageenan in ethanol and deionised water

Referring now to Figure 7 there is shown four alcoholic liquid compositions containing 3% by weight kappa carrageenan in 20% by weight ethanol and deionised water, which have been hydrated and sheared at a range of temperatures: 7a. 10°C; 7b. 20°C; 7c. 30°C; 7d. 40°C. Referring also to Figure 8 there is shown four alcoholic liquid compositions containing

3% by weight kappa carrageenan in 30% by weight ethanol and deionised water, which have been hydrated and sheared at a range of temperatures: 8a. 10°C; 8b. 20°C; 8c. 30°C; 8d. 40°C. Referring also to Figure 9 there is shown four alcoholic liquid compositions containing 3% by weight kappa carrageenan in 40% by weight ethanol and deionised water, which have been hydrated and sheared at a range of temperatures: 9a. 10°C; 9b. 20°C; 9c. 30°C; 9d. 40°C. The alcoholic liquid compositions of Figures 7, 8 and 9 were prepared using the method below with a Kinexus rheometer (Malvern Instruments, UK) with cup and vane geometry:

- i. Prepare stock solutions of 20%, 30% and 40% by weight of ethanol in deionised water.
- ii. Add 29.1 g of the appropriate stock solution to the rheometer cup.
- iii. Add the 0.9 g of kappa carrageenan powder to the rheometer cup. For all compositions of Example 4, the total weight of the composition in the rheometer cup is 30 g.
- iv. Immediately begin the shearing process using a sequence of 30 minutes and a single shear rate of 500 s^{-1} , maintaining the temperature during this step as appropriate for each composition, e.g. 10°C, 20°C, 30°C, or 40°C, for 30 minutes.
- v. Stop the shear process, transfer the composition from the rheometer cup to a sample pot. Leave the sample to stand in the sample pot for 6 hours.
- vi. Photograph for analysis (as shown in Figure 7 to 9).

Referring now to Figure 10 there is shown a graphical representation of the percentage by weight of hydrated kappa carrageenan at the range of hydration temperatures shown in Figures 7 to 9. The graph shows data points for alcoholic liquid compositions 7a-7d (20% ethanol in water) 101 (squares), alcoholic liquid compositions 8a-8d (30% ethanol in water) 102 (circles), and alcoholic liquid compositions 9a-9d (30% ethanol in water) 103 (triangles). The graph shows that as the temperature increases, the percentage of hydrated kappa carrageenan also increases, at a constant concentration of ethanol in deionised water. This effect can also be observed in the photographs of Figures 7 to 9, at low temperature (10°C and 20°C) the kappa carrageenan in the alcoholic liquid compositions, 7a, 7b, 8a, 8b, 9a, 9b, is less than fully hydrated.

As expected from the results shown in the graph of Figure 4, the percentage of hydrated kappa carrageenan is lower at higher concentrations of ethanol in deionised water.

Example 5 – Effect of temperature using 1% by weight kappa carrageenan in ethanol and deionised water

Referring now to Figure 11 there is shown four alcoholic liquid compositions containing 1% by weight kappa carrageenan in 20% by weight ethanol and deionised water, which have been hydrated and sheared at a range of temperatures: 11a. 10°C; 11b. 20°C; 11c. 30°C; 11d. 40°C. Referring now to Figure 12 there is shown four alcoholic liquid compositions containing 1% by weight kappa carrageenan in 30% by weight ethanol and deionised water, which have been hydrated and sheared at a range of temperatures: 12a. 10°C; 12b. 20°C; 12c. 30°C; 12d. 40°C. The alcoholic liquid compositions of Figures 11 and 12 were prepared using the method below with a Kinexus rheometer (Malvern Instruments, UK) with cup and vane geometry:

- i. Prepare stock solutions of 20% and 30% by weight of ethanol in deionised water.
- ii. Add 29.7 g of the appropriate stock solution to the rheometer cup.
- iii. Add the 0.3 g of kappa carrageenan powder to the rheometer cup. For all compositions of Example 5, the total weight of the composition in the rheometer cup is 30 g.
- iv. Immediately begin the shearing process using a sequence of 30 minutes and a single shear rate of 500 s^{-1} , maintaining the temperature during this step as appropriate for each composition, e.g. 10°C, 20°C, 30°C, or 40°C, for 30 minutes.
- v. Stop the shear process, transfer the composition from the rheometer cup to a sample pot. Leave the sample to stand in the sample pot for 6 hours.
- vi. Photograph for analysis (as shown in Figure 11 and 12).

As shown in Figure 11, the kappa carrageenan is fully hydrated at a concentration of 1% by weight in alcoholic liquid composition 11c and 11d (20% ethanol at 30°C and 40°C respectively).

Example 6 – Effect of kappa carrageenan concentration in methanol and deionised water

Referring now to Figure 13 there is shown five alcoholic liquid compositions containing kappa carrageenan in a range of concentrations; 13a. 1%; 13b. 2%; 13c. 3%; 13d. 4%; 13e. 5%; by weight; in 20% by weight methanol in deionised water, at 30°C. The alcoholic liquid

compositions 13a-13e were prepared using the method below with a Kinexus rheometer (Malvern Instruments, UK) with cup and vane geometry:

- i. Prepare a stock solution of 20% by weight of methanol in deionised water.
- ii. Add the appropriate weight of the appropriate stock solution to the rheometer cup.
- iii. Add the appropriate amount of kappa carrageenan powder to the rheometer cup. For example, a 3% by weight kappa carrageenan solution in 20% by weight methanol and deionised water would contain 29.1 g of stock solution and 0.9 g of kappa carrageenan. For all compositions in Example 6, the total weight of the composition in the rheometer cup is 30 g.
- iv. Immediately begin the shearing process using a sequence of 30 minutes and a single shear rate of 500 s^{-1} , maintaining the temperature at 30°C , for 30 minutes.
- v. Stop the shear process, transfer the composition from the rheometer cup to a sample pot. Leave the sample to stand in the sample pot for 6 hours.
- vi. Photograph for analysis (as shown in Figure 13).

As shown in Figure 13, the kappa carrageenan was fully hydrated at concentrations at and below 3% by weight. Additionally, an acceptable level of hydration was achieved in concentrations from above 3% to 5% kappa carrageenan by weight.

Example 7 – Analysis of 13a-13d using differential scanning calorimetry (DSC)

Referring now to Figure 14A there is shown differential scanning calorimetry (DSC) heating profiles of kappa carrageenan at a range of concentrations as prepared in Example 6: 13a. 1%; 13b. 2%; 13c. 3%; and 13d. 4%; by weight; in 20% by weight methanol in deionised water. Referring now to Figure 14B there is shown a differential scanning calorimetry (DSC) cooling profiles of kappa carrageenan at a range of concentrations as prepared in Example 6: 13a. 1%; 13b. 2%; 13c. 3%; and 13d. 4%; by weight; in 20% by weight methanol in deionised water. The enthalpy and temperature of thermal transition were measured for the gel phase using a μDSC evo Dynamic Scanning Calorimeter (DSC) (Setaram Instrumentation, France) in the following procedure:

- i. Add 650 mg (± 5 mg) of the gel to be analysed (13a-13d) to a screw top 'closed batch cell'.
- ii. Reference sample - Add 650 mg (± 5 mg) of deionised water to a screw top 'closed batch cell'.

- iii. Taking the hydrated gel sample initially, hold the sample isothermally at 5°C for 30 minutes.
- iv. Heat the sample to 90°C at a rate of 1.2°C/min.
- v. Cool the sample to 5°C at a rate of 1.2°C/min.
- 5 vi. Hold the sample isothermally at 5°C for 30 minutes.
- vii. Heat the sample to 90°C at a rate of 1.2°C/min.
- viii. Cool the sample to 5°C at a rate of 1.2°C/min.
- ix. Record the graphs of Figures 14A and 14B.

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Example 8 – Effect of kappa carrageenan concentration in propanol and deionised water

Referring now to Figure 15 there is shown five alcoholic liquid compositions containing kappa carrageenan in a range of concentrations; 15a. 1%; 15b. 2%; 15c. 3%; 15d. 4%; 15e. 5%; by weight; in 20% by weight propanol in deionised water, at 30°C. The alcoholic liquid compositions were prepared using the method described in Example 6 by substituting methanol for propanol in Step i.

15

Example 9 – Effect of kappa carrageenan concentration in propanol and deionised water

20

Referring now to Figure 16A there is shown differential scanning calorimetry (DSC) heating profiles of the alcoholic liquid composition 15c comprising kappa carrageenan (3% by weight) in propanol (20% by weight) and deionised water.

25

Referring also to Figure 16B there is shown a differential scanning calorimetry (DSC) cooling profiles of the alcoholic liquid composition 15c comprising of kappa carrageenan (3% by weight) in propanol (20% by weight) and deionised water.

30

The procedure of Example 6A and 6B was followed by substituting the samples used in Step i for the sample prepared in Example 8 (15c). Two heating and cooling steps were undertaken to the alcoholic liquid composition 15c. The first heating a cooling step is shown as follows. In Figure 16A there is shown the first heating profile 161 of the first heating step, in Figure 16B there is shown the first cooling profile 163 first cooling step. The alcoholic liquid composition 15c underwent a subsequent second heating and cooling step. In Figure

16A there is shown the second heating profile 162 of the second heating step, in Figure 16B there is shown the second cooling profile 164 of the second cooling step.

As shown in Figure 16A, it appears that the first heating profile 61 differs from the second heating profile 62. However, these differences are not thought to be significant, and it is thought that the gel structures on the first heating profile 161 and the second heating profile 163 are substantially the same. This is also seen in the first cooling profile 163 and the second cooling profile 164. As with Figures 6A and 6B, the hysteresis effects between the gel setting temperature and the gel melting temperature are observed.

Example 10 – Effect of gellan gum in ethanol and deionised water

Referring now to Figure 17 there is shown four alcoholic liquid compositions containing low acyl gellan gum (3% by weight) in range of concentrations of ethanol; 17a. 15%; 17b. 18%; 17c. 25%; 17d. 30%; by weight, in deionised water, hydrated at 30°C. The alcoholic liquid compositions 17a-17d were prepared using the method below with a Kinexus rheometer (Malvern Instruments, UK) with cup and vane geometry:

- i. Prepare stock solutions of 15%, 18%, 25%, and 30% by weight of ethanol in deionised water.
- ii. Add 29.1 g of the appropriate stock solution to the rheometer cup.
- iii. Add 0.9 g of low acyl gellan gum powder to the rheometer cup.
- iv. Immediately begin the shearing process using a sequence of 30 minutes and a single shear rate of 500 s^{-1} , maintaining the temperature at 30°C, for 30 minutes.
- v. Stop the shear process, transfer the composition from the rheometer cup to a sample pot. Leave the sample to stand in the sample pot for 6 hours.
- vi. Photograph for analysis (as shown in Figure 17).

As shown in Figure 17, at the low shear rates employed, the gellan gum is hydrated at low concentrations of ethanol. However, the gellan gum appears to form clumps of unhydrated powder at high concentrations of ethanol (above 25% by weight) in deionised water. We believe that an increase in shear and/or a moderate increase in temperature would be sufficient to hydrate at least more of the gellan gum.

Comparative Example 1 – Gel structures using standard gel method

A standard gel comprising kappa carrageenan (3% by weight) in 20% ethanol and water was prepared using the standard method.

- i. Prepare a stock solution of 20% by weight ethanol in deionised water.
- 5 ii. Add 29.1 g of stock solution and 0.9 g of kappa carrageenan to a vessel.
- iii. Heat the mixture to 65°C whilst stirring using a hot plate stirrer.
- iv. Maintain the temperature at 65°C until the kappa carrageenan has dissolved.
- v. Transfer the resulting mixture to a rheometer cup.
- vi. Cool to from 65°C to 10°C at a rate of 2°C/min whilst sheering at a rate of 500
10 s⁻¹.
- vii. Leave to stand for 24 hours.

Analysis of the standard gel was performed using differential scanning calorimetry (DSC). The enthalpy and temperature of thermal transition were measured for the gel phase using
15 a µDSC evo Dynamic Scanning Calorimeter (DSC) (Setaram Instrumentation, France) in the following procedure.

- i. Hydrated gel sample - Add 650 mg ($\pm$ 5 mg) of the hydrated gel containing 3% kappa carrageenan in 20% ethanol and deionised water to a screw top 'closed batch cell'.
- 20 ii. Reference sample - Add 650 mg ($\pm$ 5 mg) of deionised water to a screw top 'closed batch cell'.
- iii. Hold the sample isothermally at 5°C for 30 minutes.
- iv. Heat the sample to 90°C at a rate of 2°C/min to obtain first heating profile 181.
- v. Cool the sample to 5°C at a rate of 2°C/min to obtain first cooling profile 183.
- 25 vi. Hold the sample isothermally at 5°C for 30 minutes.
- vii. Heat the sample to 90°C at a rate of 2°C/min to obtain second heating profile 182.
- viii. Cool the sample to 5°C at a rate of 2°C/min to obtain second cooling profile 184.
- ix. Record the graphs of Figures 18A and 18B.

30 Figure 18A shows the enthalpy of thermal transition of the standard gel upon melting. There is shown the first heating profile 181 representing the first melt, and the second heating profile 182 representing the second melt. Figure 18B shows the enthalpy of thermal transition of the standard gel upon reformation. There is shown the first cooling profile 183
35 and the second cooling profile 182.

As is clearly seen from Figure 18A, the first heating profile 181, which represents the first melt of the standard gel, is significantly different to the second heating profile 182, which represents the second melt of the standard gel. However, it appears that the first cooling profile 183 and the second cooling profile 184 are substantially the same.

The effect on the structure of heating the gel is not fully understood. Without wishing to be bound by theory, it is believed that the standard gel, which is obtained using the method of the prior art, initially has a non-homogenous structure. Upon heating, the gel structure equilibrates to a homogeneous structure. This is shown in the first heating profile 181, which contains a shoulder within the peak representing the enthalpy of thermal transition. This gel cools to produce the first cooling profile 183. Upon heating the standard gel for a second time, the second heating profile 182 is produced, which is comparable to the first and second heating profiles of the gel of the present invention as shown in Figure 6A. The standard gel cools for a second time to produce second cooling profile 184, which is identical to first cooling profile 183, showing that the gel structure obtained after the first heating and the second heating are substantially the same, but differ from the gel structure obtained after the standard method of gel formation described above in Comparative Example 1.

Figure 19A shows the enthalpy of thermal transition upon melting of the standard gel for the first time. Three repeats (191, 192, 193) of the DSC procedure as described above were performed for the standard gel only. Figure 19B shows the enthalpy of thermal transition upon melting of the standard gel for the second time. Three repeats (194, 195, 196) of the DSC procedure as described above were performed for the standard gel only.

Figure 20 shows the first (A) and the second (B) heating profiles of the standard gel during the DSC measurements. Heating profile A shows that used for the data shown in Figure 19A, and heating profile B shows that used for the data shown in Figure 19B.

As is clearly shown in Figure 19A, the standard gels 191, 192, 193 do not produce consistent results for the enthalpy and/or temperature of thermal transition upon melting for the first time. As shown in Figure 19B, upon melting for a second time, the standard gels 194, 195, 196 produce consistent results for the enthalpy and temperature of thermal transition.

Without wishing to be bound by theory, we believe that heating the standard gel for the first time (as shown in Figure 19A) causes the gel to order into a homogeneous structure. Once heated, the standard gel is stable, and may be cooled and reheated (as shown in Figure 19B) to produce a consistent enthalpy and temperature of thermal transition across the three repeats 194, 195, 196.

The results shown in Figures 19A and 19B are consistent with the results shown in Figures 18A and 18B.

Therefore, the method of the present invention has a number of advantages over the standard method for the preparation of the standard gel.

Firstly, the standard method is a two-step process, in which the hydrocolloid is dissolved in the alcohol and water solution, and then is transferred to the rheometer cup to cool whilst sheering. The method of the present invention is a one-step method in which the hydrocolloid is mixed and sheered in the rheometer cup in a one-pot process. It has been shown that attempts to dissolve the hydrocolloid in the rheometer cup whilst heating to 65°C are unsuccessful.

Another advantage is that the method of the present invention uses a low temperature to hydrate the hydrocolloid, i.e. below 60°C. The standard method uses temperatures of 65°C or above to dissolve the hydrocolloid. The use of a low temperature in the present invention is made possible using the sheering sequence whilst hydrating the hydrocolloid at a low temperature. The method of the present invention is clearly safer and requires less energy, making it more environmentally friendly.

A further advantage is that the method of the present invention provides a hydrated gel with a homogenous structure, which is reproducible. As shown in Figure 19A, the standard gel structure that results from the standard method produces a gel structure that is neither homogenous, nor reproducible. Upon heating, the gel is reordered to produce a gel that has an identical structure to that which results from the method of the present invention. Therefore, it is clearly advantageous to use the method of the present invention to achieve a consistent and homogeneous gel.

It will be appreciated that the gels of the present invention are capable of suspending a plurality of particles including, for example, gold leaf (E175) or silver leaf (E174), various food-grade pigment particles based on titanium, dioxide (E171) and/or iron oxides (E172) and/or potassium aluminium silicate (E555), or alternatively fruit pieces or flakes, a starch-based glitter, tapioca pearls, or gelatin spheres. Specifically, a plurality of particles may be suspended in an alcoholic beverage, for example, vodka, gin, rum, and/or whiskey.

It will also be appreciated by those skilled in the art that any number of combinations of the aforementioned features and/or those shown in the appended drawings provide clear advantages over the prior art and are therefore within the scope of the invention described herein.

Within the scope of this application it is expressly intended that the various aspects, embodiments, examples and alternatives set out in the preceding paragraphs, in the claims and/or in the following description and drawings, and in particular the individual features thereof, may be taken independently or in any combination. That is, all embodiments and/or features of any embodiment can be combined in any way and/or combination, unless such features are incompatible. For the avoidance of doubt, the terms “may”, “and/or”, “e.g.”, “for example” and any similar term as used herein should be interpreted as non-limiting such that any feature so-described need not be present. Indeed, any combination of optional features is expressly envisaged without departing from the scope of the invention, whether or not these are expressly claimed. The applicant reserves the right to change any originally filed claim or file any new claim accordingly, including the right to amend any originally filed claim to depend from and/or incorporate any feature of any other claim although not originally claimed in that manner.

CLAIMS

1. A method for manufacturing alcoholic liquid compositions, the method comprising the steps of:
 - a) providing an alcoholic liquid composition;
 - b) contacting unhydrated hydrocolloid with the alcoholic liquid composition to provide a hydrocolloid composition;
 - c) shearing the resulting hydrocolloid composition at a temperature of 60°C or below to hydrate the hydrocolloid.
2. A method according to Claim 1, comprising the further step of: comprising the steps of:
 - d) adding a plurality of particles to the alcoholic liquid composition or the hydrocolloid composition.
3. A method according to Claim 2, in which step d) is performed before step c) and/or before step b).
4. A method according to Claim 1, 2 or 3, wherein the alcoholic liquid composition comprises an aqueous solution of alcohol.
5. A method according to Claim 4, wherein the concentration of alcohol in deionised water is 50% by weight or less.
6. A method according to Claim 4 or 5, wherein the concentration of alcohol in deionised water is 40% by weight or less.
7. A method according to Claim 4, 5 or 6, wherein the concentration of alcohol in deionised water is 30% by weight or less, say 20% by weight or less, or 10% by weight or less.
8. A method according to any preceding Claim, wherein the hydrocolloid is selected from one or more of kappa carrageenan, iota carrageenan, low acyl gellan, agar and/or agarose.

9. A method according to any preceding Claim, wherein the concentration of the hydrocolloid is less than or equal to 3 wt. %, for example less than or equal to 1 wt. %.
- 5 10. A method according to any preceding Claim, comprising maintaining the temperature of step c) at or below 40°C, for example at or below 30°C, say at or below 20°C, say 10°C.
- 10 11. A method according to any preceding Claim, comprising performing step c) at shear rate is 1000 s⁻¹ or less.
12. A method according to any preceding Claim, comprising performing step c) for less than one hour, for example for less than 30 minutes.
- 15 13. A method according to Claim 21, wherein a rheometer is used to perform the shearing in step c).
- 20 14. A method according to any preceding Claim, wherein the suspended particles are or comprise one or more of titanium oxide (E171), iron oxide (E172), potassium aluminium silicate (E555), gold (E175), silver (E174), fruit pieces, fruit flakes, starch-based glitter, tapioca pearls, gelatin spheres, non-toxic metallic particles including gold, silver, copper, platinum, schlagmetal, sand, microbeads, or plastic-based glitter.
- 25 15. A method according to any preceding Claim, wherein the suspended particles are spherical or near spherical.
16. A method according to Claim 26, in which the suspended particles a thin foil or metallic leaf.
- 30 17. An alcoholic composition comprising alcohol, water and a hydrocolloid gel, wherein the hydrocolloid gel is substantially non-quiescent and formed of discrete hydrocolloid particles dispersed within the composition.
- 35 18. An alcoholic composition according to Claim 17, wherein particles are dispersed and suspended throughout the composition.

19. An alcoholic composition according to Claim 18, wherein the suspended particles comprise less than 5% by weight of the alcoholic composition, say less than 1%, or less than 0.1% by weight.
- 5 20. An alcoholic composition according to Claim 18 or 19, wherein the suspended particles are or comprise one or more of titanium oxide (E171), iron oxide (E172), potassium aluminium silicate (E555), gold (E175), silver (E174), fruit pieces, fruit flakes, starch-based glitter, tapioca pearls, gelatin spheres, non-toxic metallic particles including gold, silver, copper, platinum, schlagmetal, sand, microbeads, or
10 plastic-based glitter.
21. An alcoholic composition according to any one of Claims 17 to 20, wherein the hydrocolloid comprises less than 3% by weight, say less than 2% by weight and preferably less than 1 % by weight of the alcoholic composition.
- 15 22. An alcoholic composition according to any one of Claims 17 to 21, wherein the concentration of alcohol is 4 to 50 w/w %, for example from 4 to 15 w/w % or from 15 to 30 w/w % or say from 30-40 w/w %.
- 20 23. An alcoholic composition according to any one of Claims 17 to 22, wherein the composition is pourable.
24. An alcoholic composition according to any of Claims 17 to 23, further comprising one or more of a colouring, flavouring, natural and artificial sweeteners, stabilisers,
25 preservatives, and vitamins and minerals.

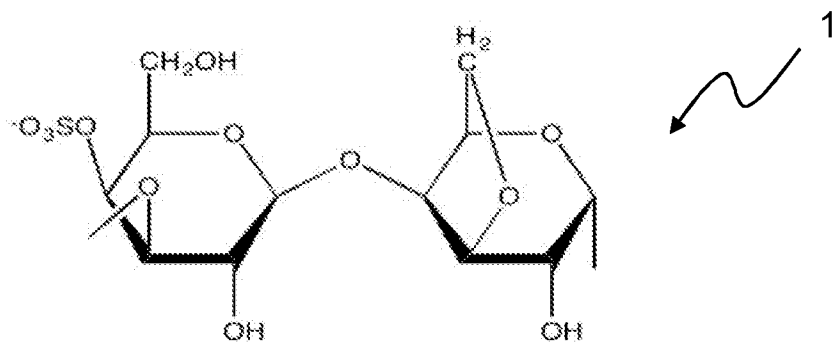


FIGURE 1

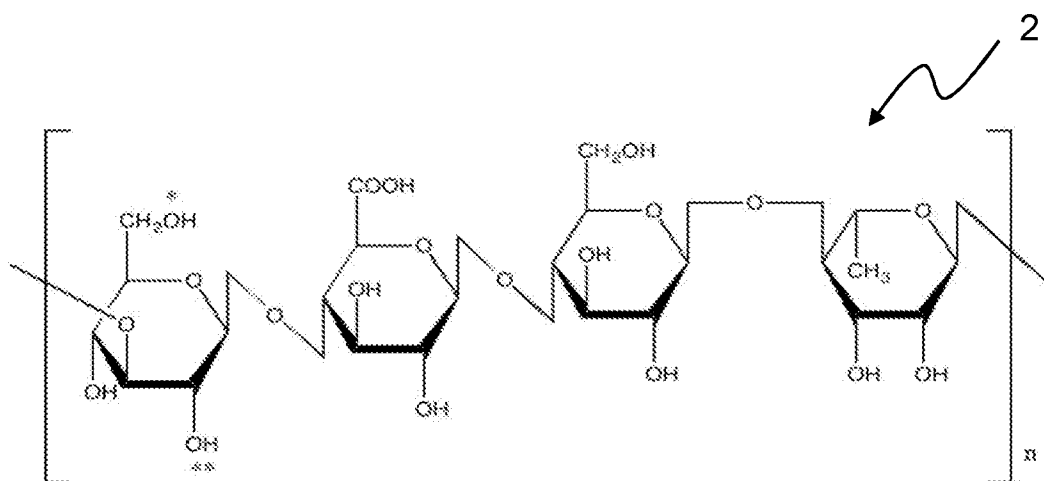


FIGURE 2

2/15

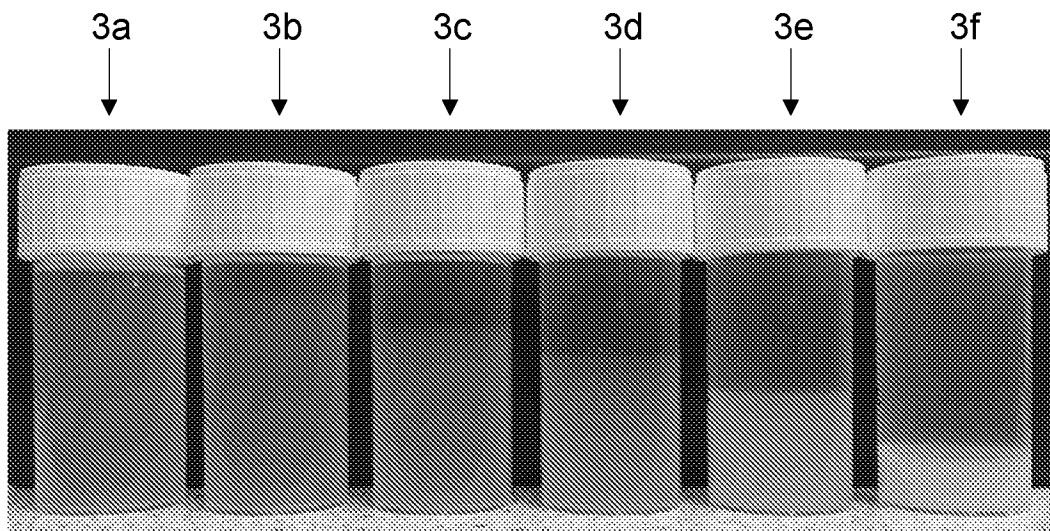


FIGURE 3

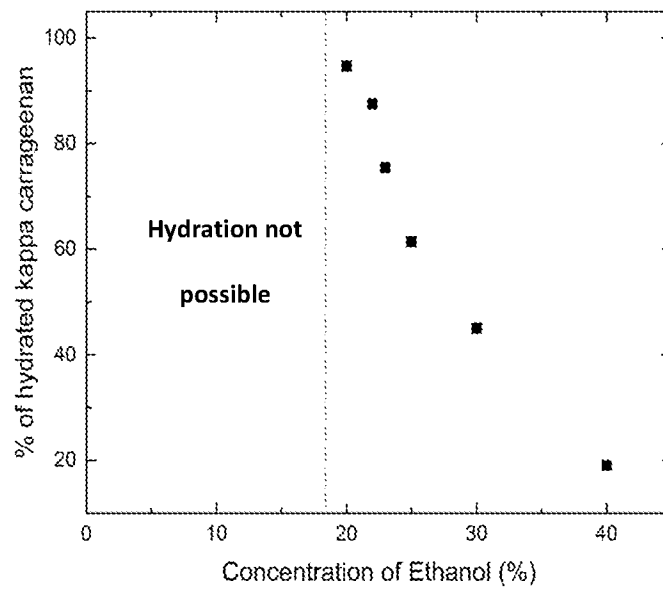


FIGURE 4

3/15

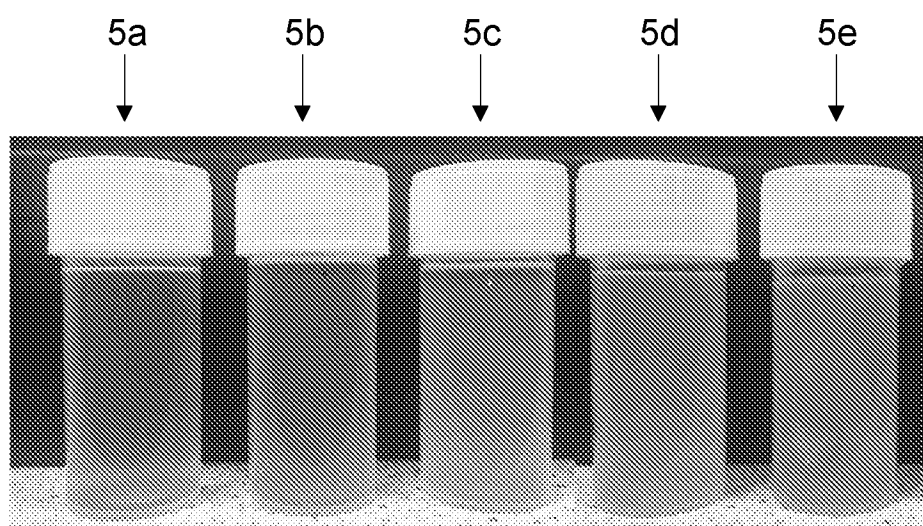


FIGURE 5

4/15

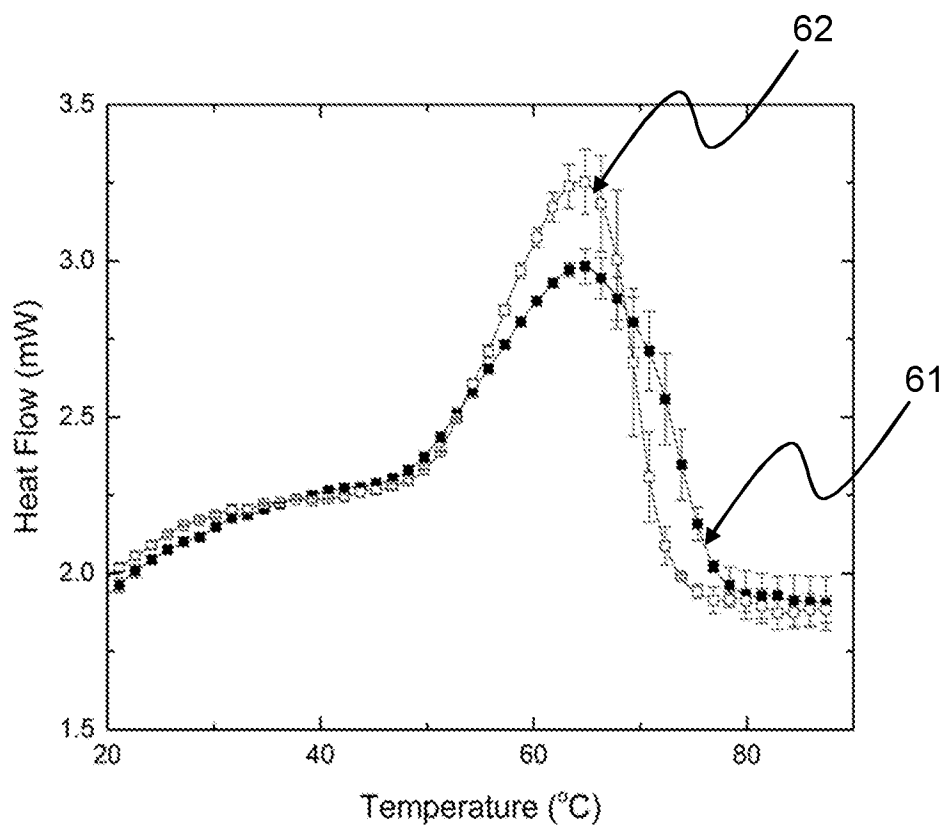


FIGURE 6A

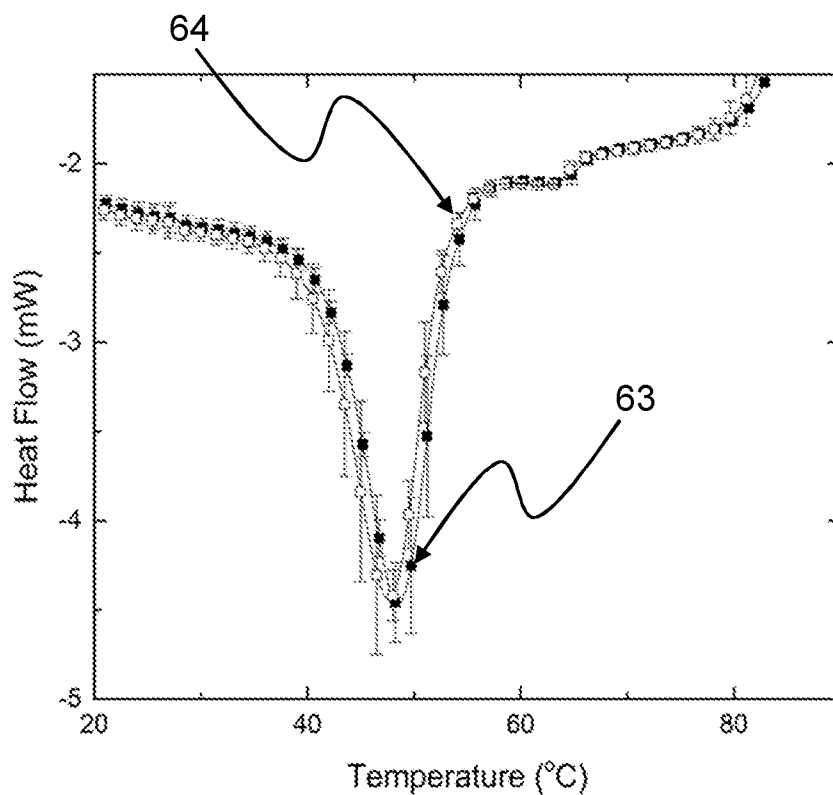


FIGURE 6B

5/15

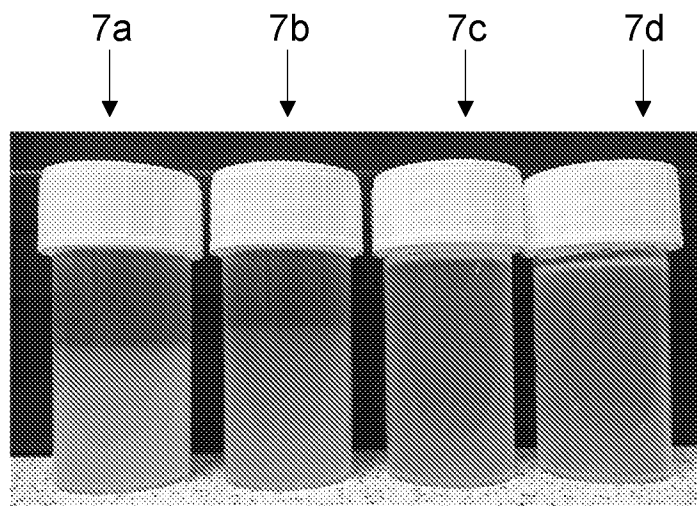


FIGURE 7

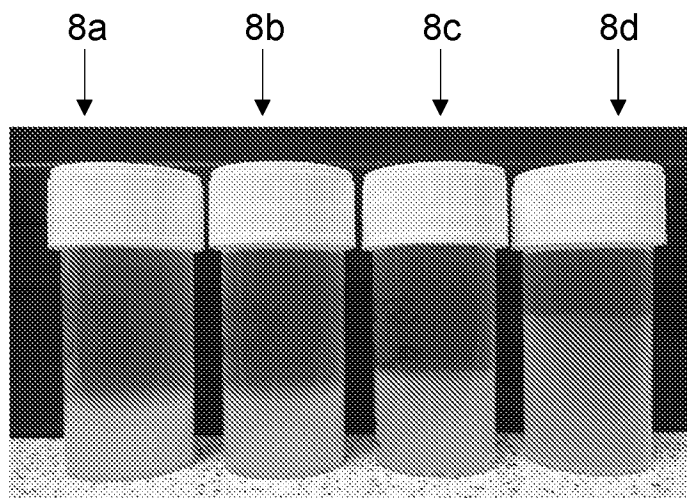


FIGURE 8

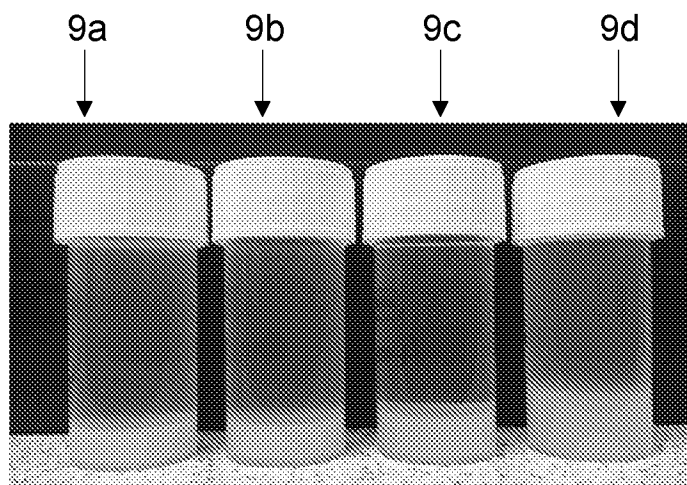


FIGURE 9

6/15

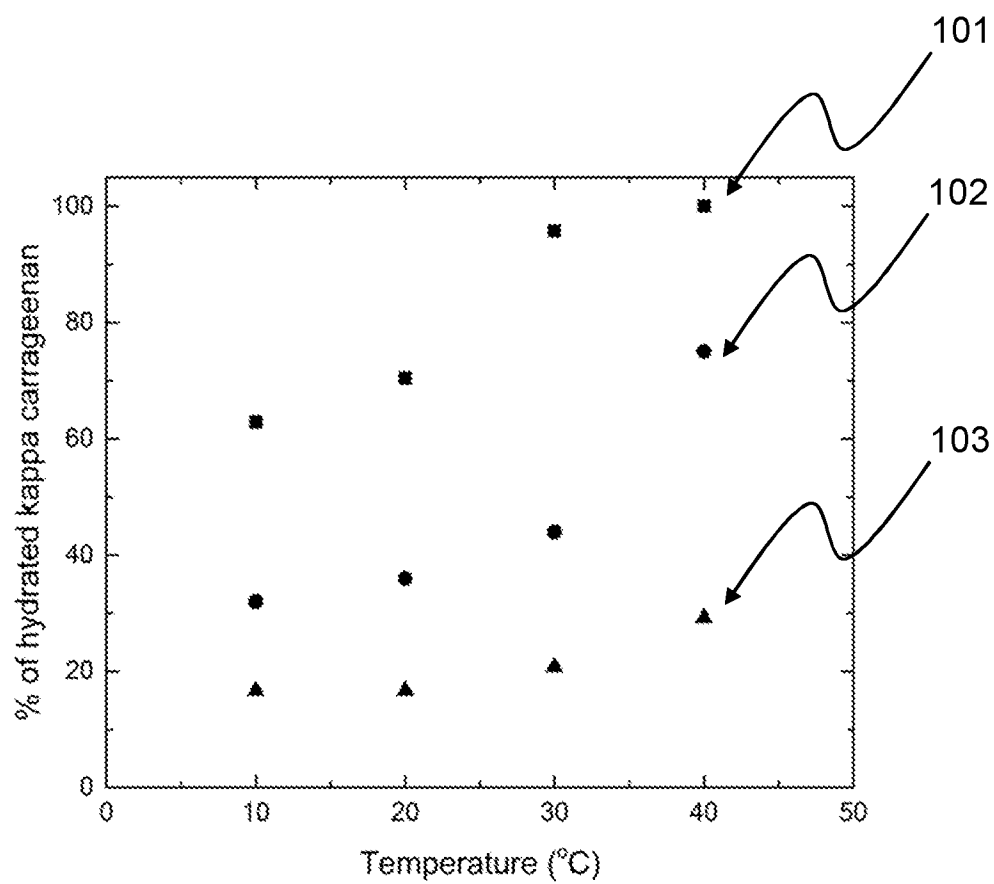


FIGURE 10

7/15

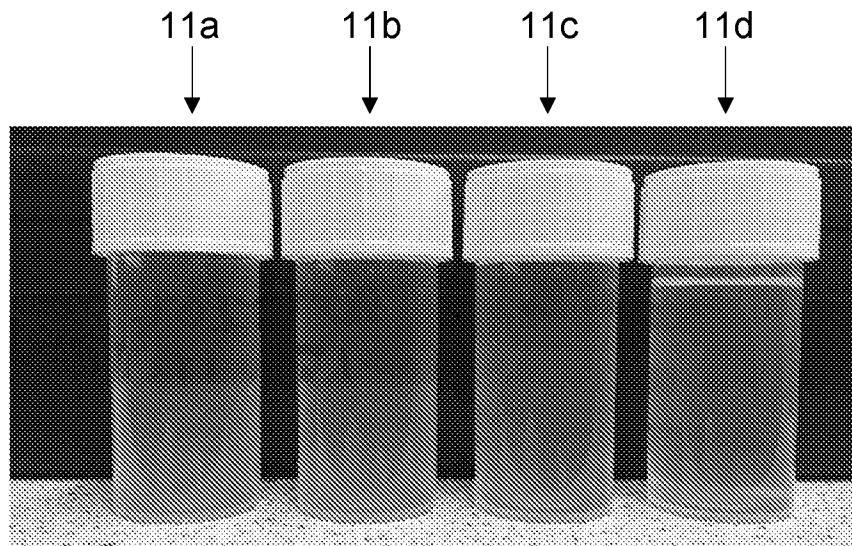


FIGURE 11

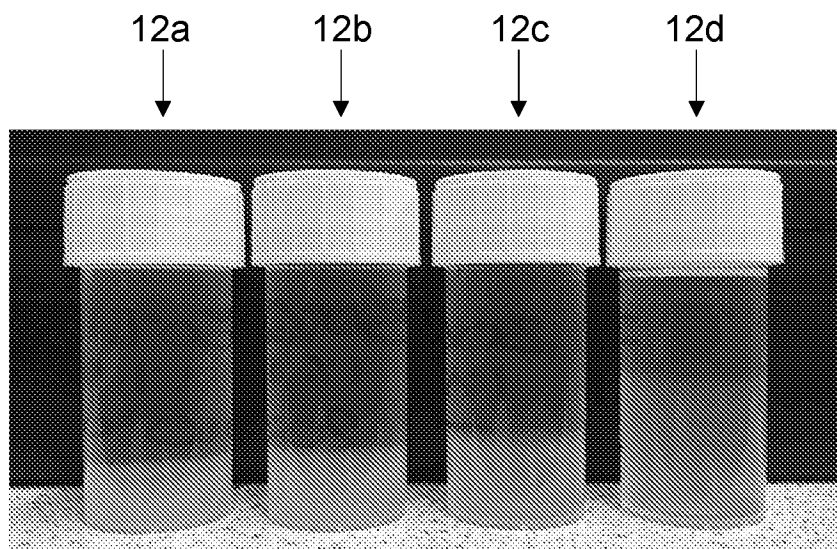


FIGURE 12

8/15

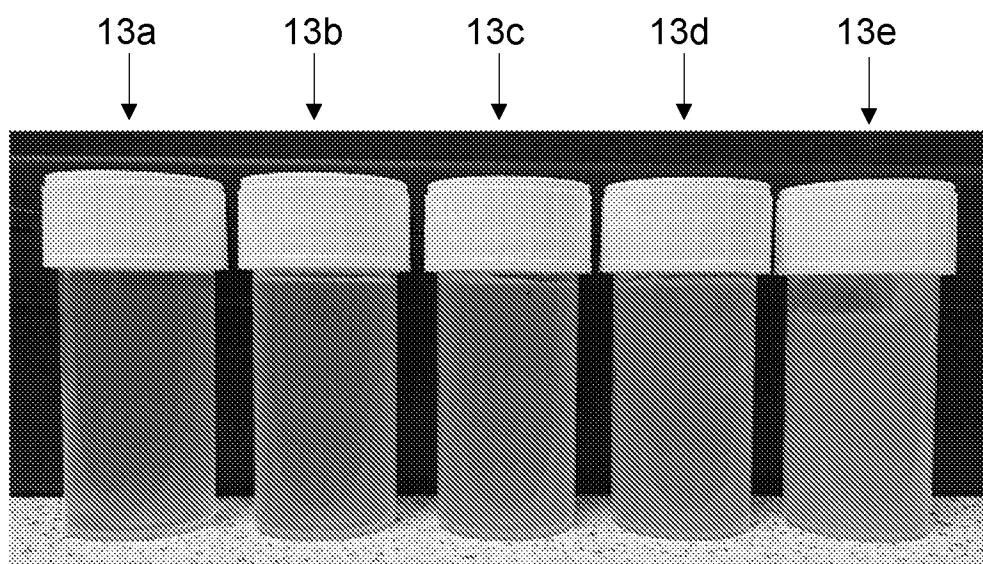


FIGURE 13

9/15

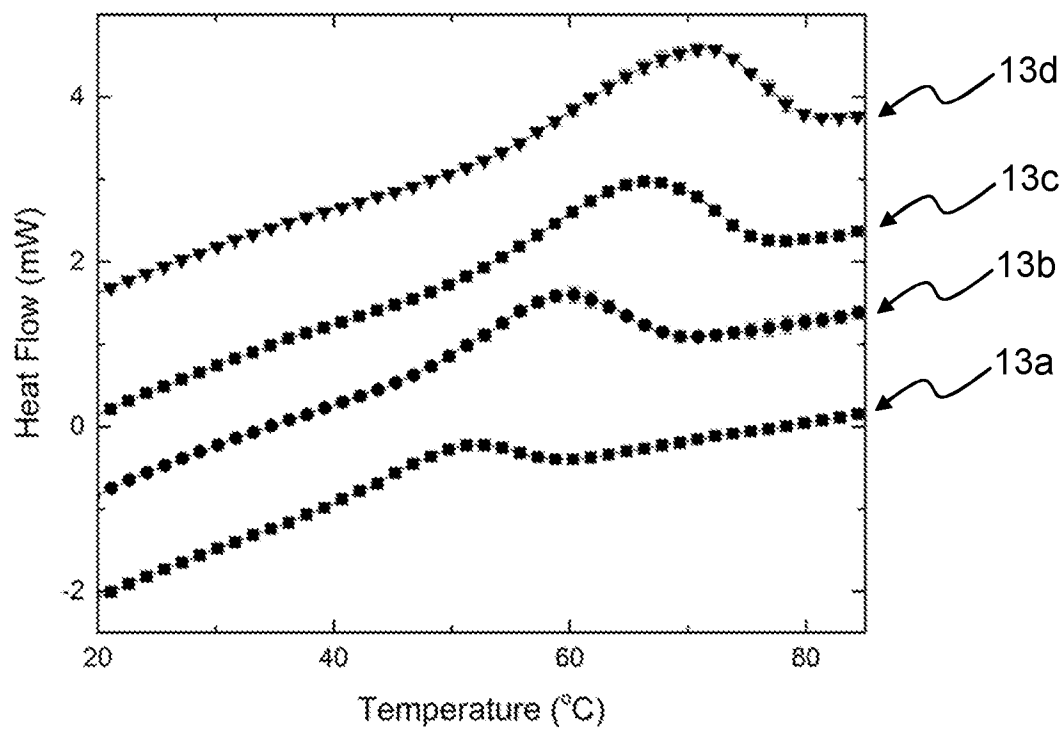


FIGURE 14A

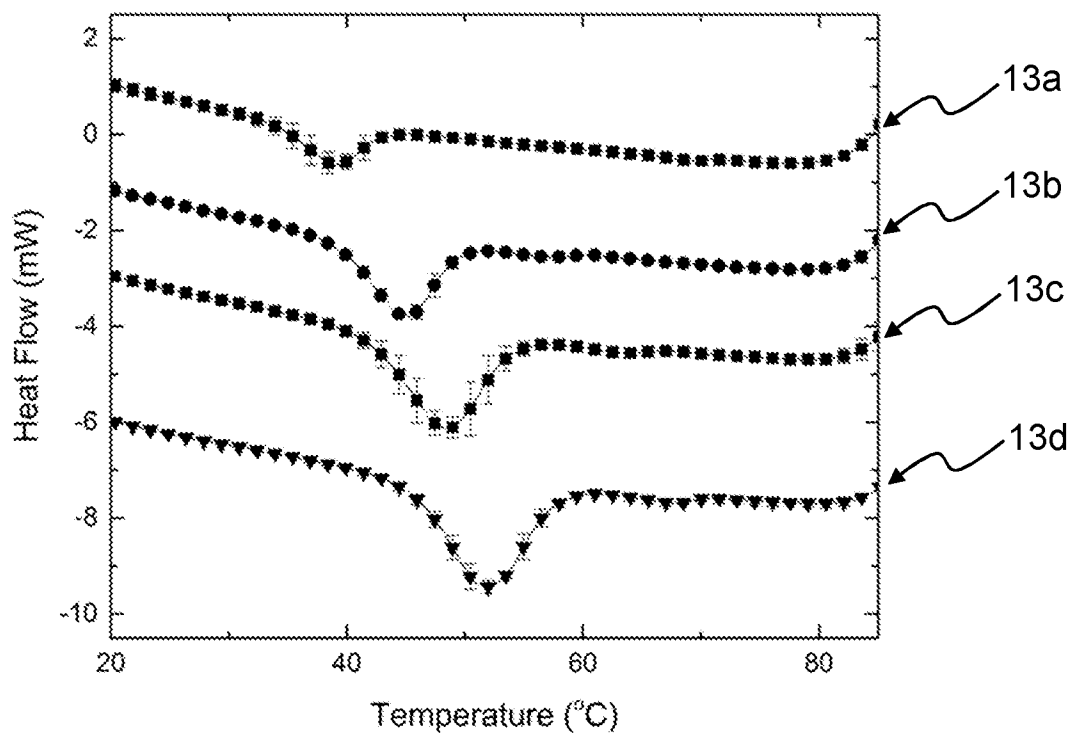


FIGURE 14B

10/15

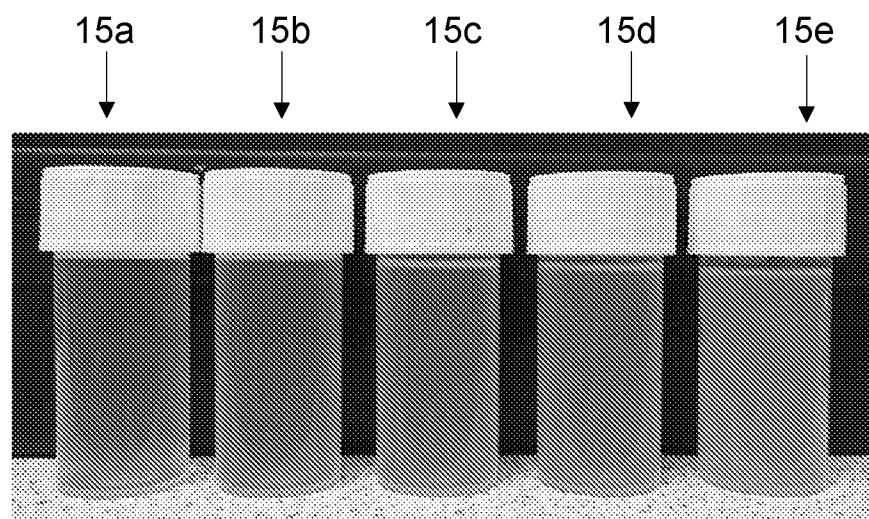


FIGURE 15

11/15

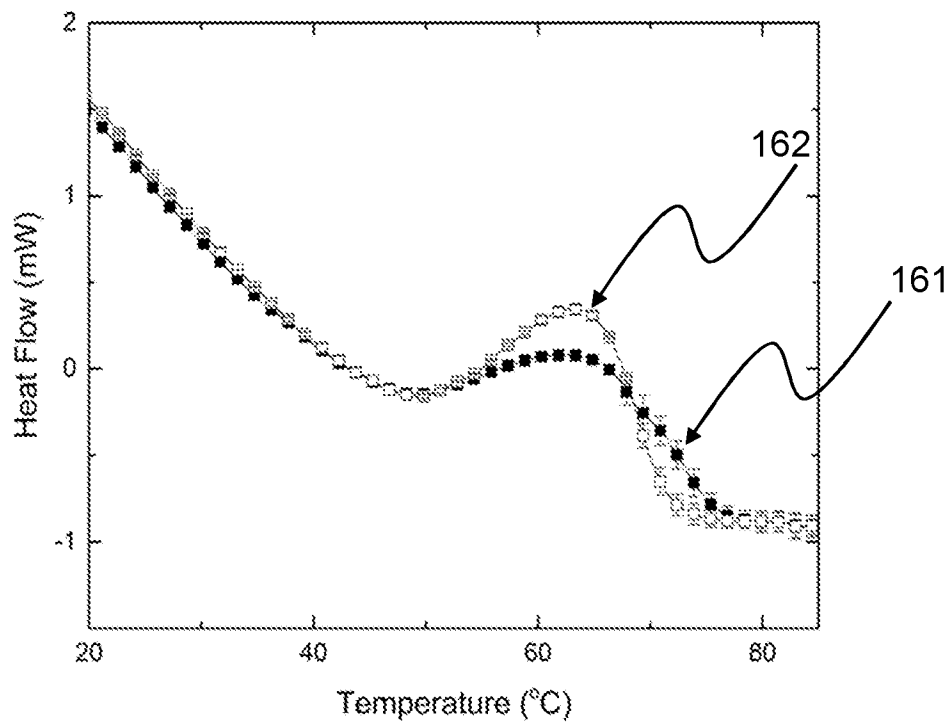


FIGURE 16A

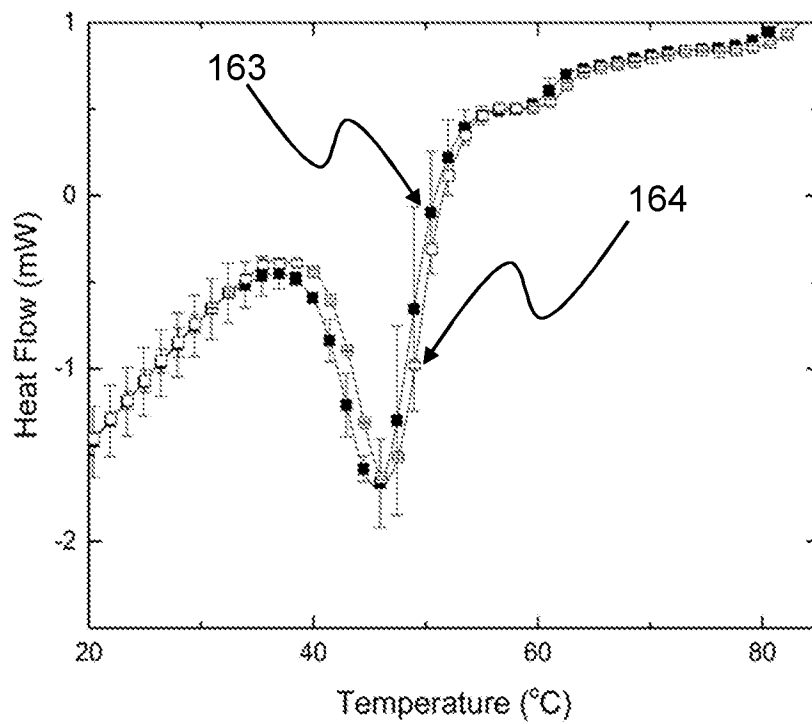


FIGURE 16B

12/15

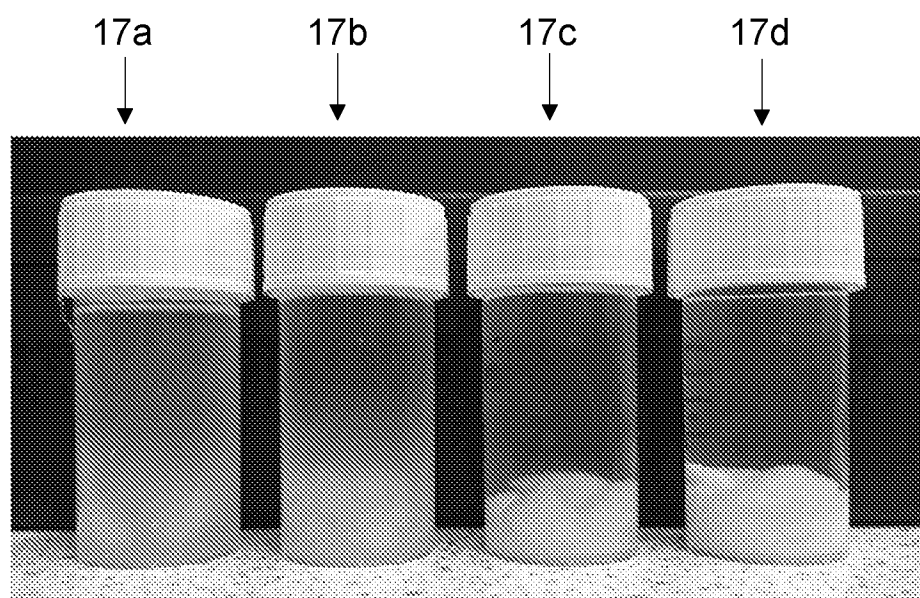


FIGURE 17

13/15

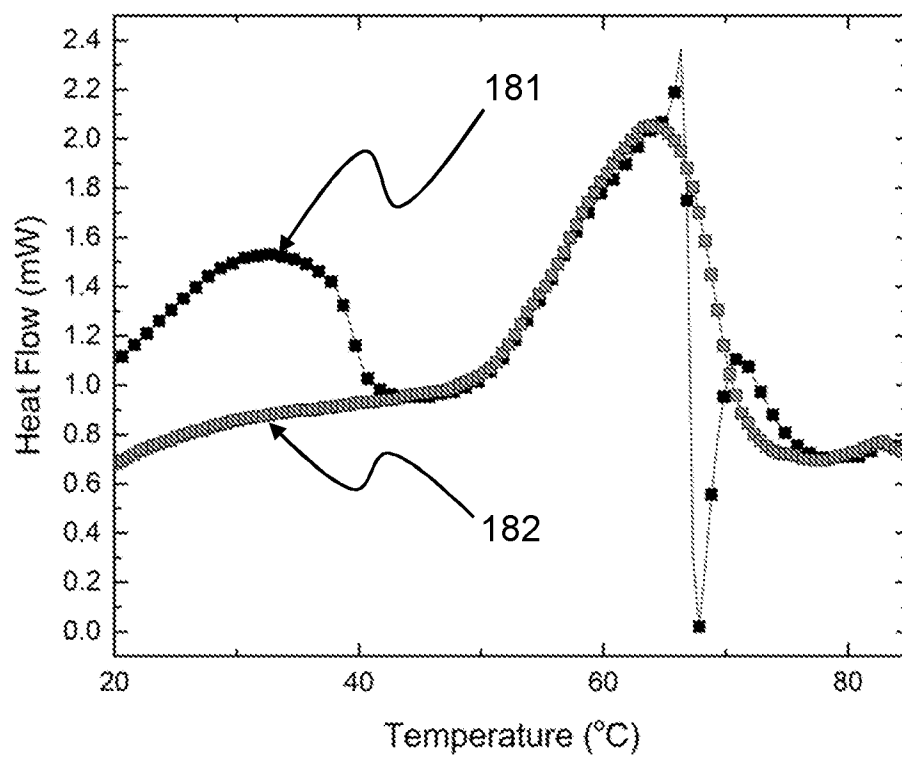


FIGURE 18A

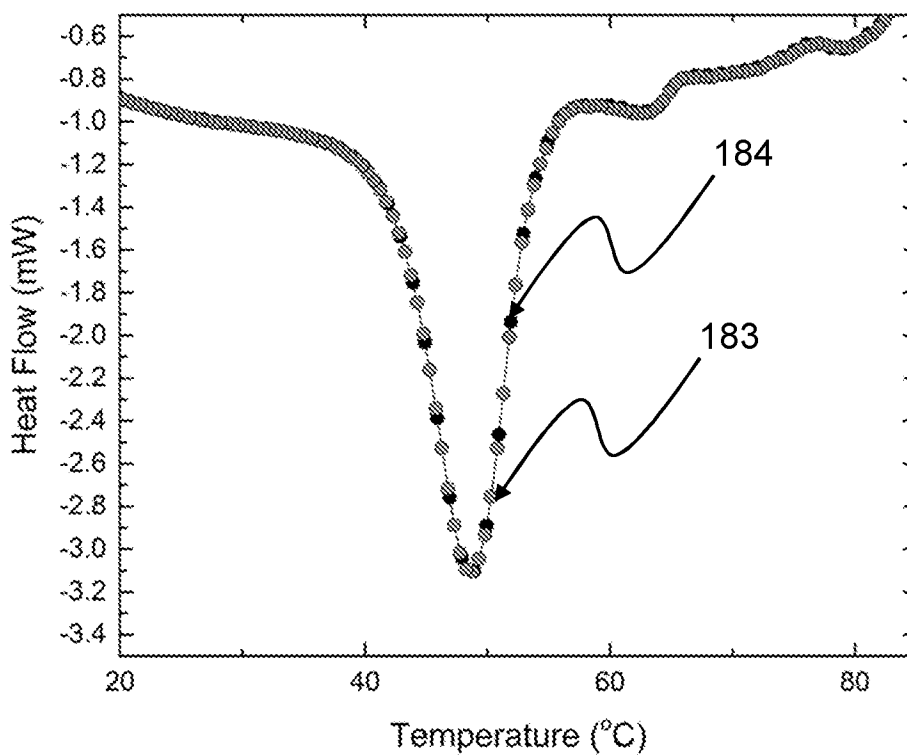


FIGURE 18B

14/15

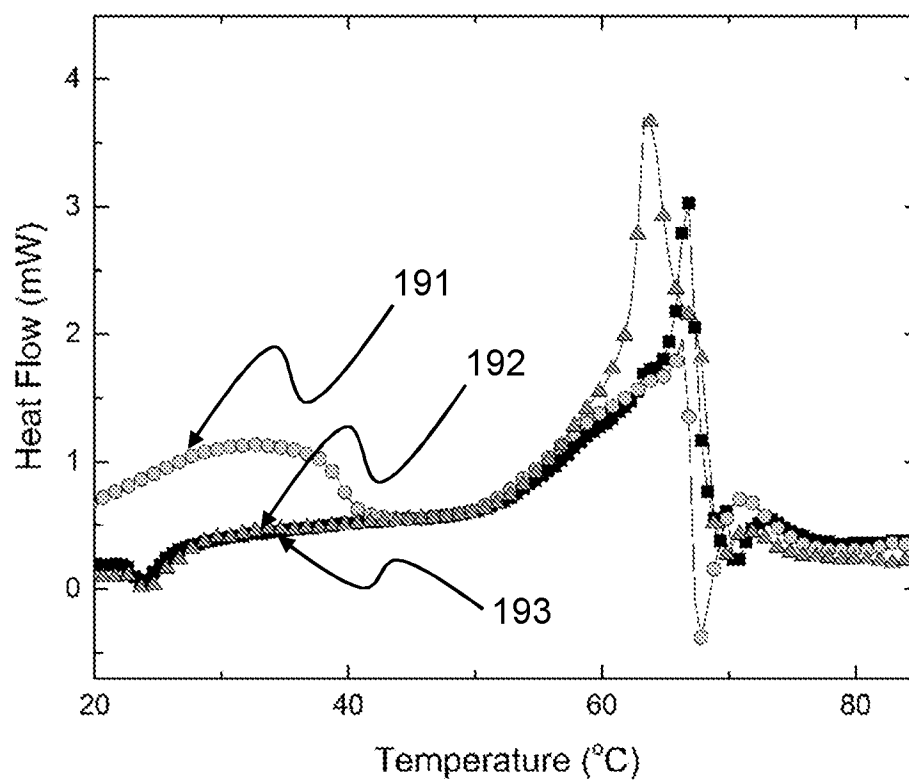


FIGURE 19A

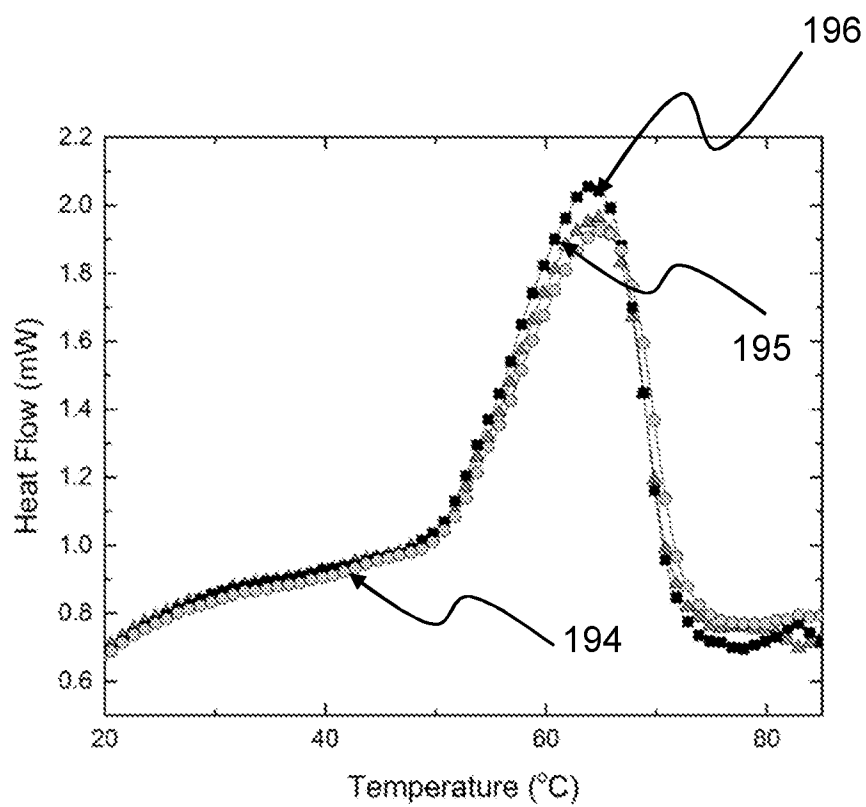


FIGURE 19B

15/15

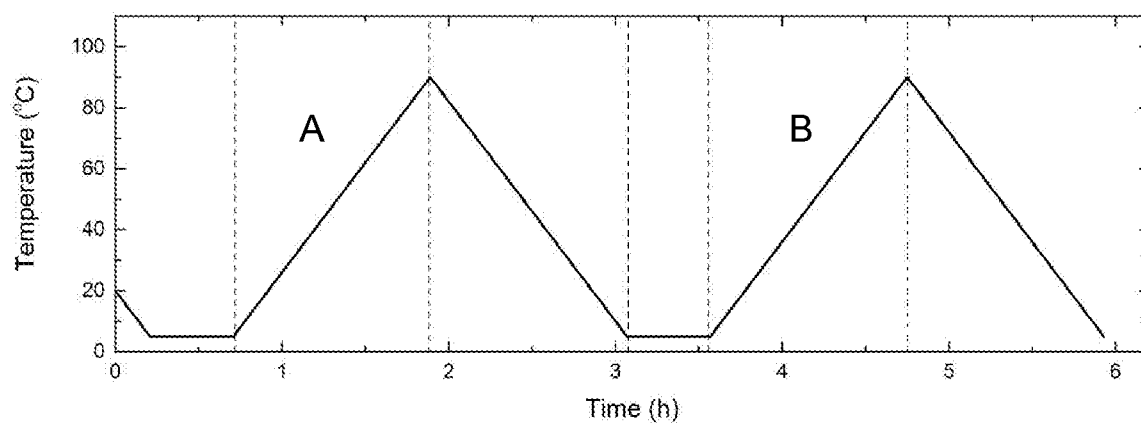


FIGURE 20

INTERNATIONAL SEARCH REPORT

International application No
PCT/GB2018/051838

A. CLASSIFICATION OF SUBJECT MATTER
INV. C12G3/04 A61L26/00 A23L29/269
ADD.

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)
C12G A61L A61K A23L

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)

EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 6 623 727 B2 (WELLA AG [DE]) 23 September 2003 (2003-09-23) claims 1, 15; example 5 -----	1-24
X	US 2012/107468 A1 (DOYLE DAVID [CA] ET AL) 3 May 2012 (2012-05-03) claims 1, 22 -----	1-24
A	WO 2016/003894 A1 (FMC CORP [US]) 7 January 2016 (2016-01-07) the whole document -----	1-24
A	WO 2007/048193 A1 (MEDIHONEY PTY LTD [AU]; MOLONEY ANTHONY PETER [AU]) 3 May 2007 (2007-05-03) the whole document -----	1-24
	-/--	



Further documents are listed in the continuation of Box C.



See patent family annex.

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"&" document member of the same patent family

Date of the actual completion of the international search

5 September 2018

Date of mailing of the international search report

24/09/2018

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Authorized officer

Picout, David

INTERNATIONAL SEARCH REPORT

International application No

PCT/GB2018/051838

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 97/15200 A1 (BUSH BOAKE ALLEN INC [US]) 1 May 1997 (1997-05-01) the whole document -----	1-24

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/GB2018/051838

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 6623727	B2	23-09-2003	AT 380050 T 15-12-2007
		DE 10051955 A1 02-05-2002	
		EP 1199064 A2 24-04-2002	
		JP 3859203 B2 20-12-2006	
		JP 2002167312 A 11-06-2002	
		US 2002076387 A1 20-06-2002	
US 2012107468	A1	03-05-2012	AU 2010359182 A1 21-02-2013
		BR 112013003465 A2 21-06-2016	
		CA 2806356 A1 23-02-2012	
		CN 103068258 A 24-04-2013	
		DK 2448429 T3 14-03-2016	
		EP 2448429 A1 09-05-2012	
		EP 3023484 A1 25-05-2016	
		ES 2565207 T3 01-04-2016	
		HK 1184643 A1 20-11-2015	
		JP 5964302 B2 03-08-2016	
		JP 2013537420 A 03-10-2013	
		KR 20130137610 A 17-12-2013	
		MX 337826 B 22-03-2016	
		NZ 606578 A 27-02-2015	
		RU 2013111280 A 27-09-2014	
		SG 187681 A1 28-03-2013	
		UA 109022 C2 10-07-2015	
		US 2012107468 A1 03-05-2012	
		WO 2012022375 A1 23-02-2012	
WO 2016003894	A1	07-01-2016	NONE
WO 2007048193	A1	03-05-2007	AU 2006308514 A1 03-05-2007
		EP 1948254 A1 30-07-2008	
		US 2009317467 A1 24-12-2009	
		WO 2007048193 A1 03-05-2007	
WO 9715200	A1	01-05-1997	AT 269001 T 15-07-2004
		AU 713783 B2 09-12-1999	
		CA 2235825 A1 01-05-1997	
		DE 69632742 D1 22-07-2004	
		DE 69632742 T2 07-07-2005	
		EP 0889694 A1 13-01-1999	
		ES 2218605 T3 16-11-2004	
		NZ 321366 A 29-09-1999	
		US 6106883 A 22-08-2000	
		WO 9715200 A1 01-05-1997	