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- (54) **Title:** METHOD OF MEASURING VISCOSITY OF GUMS

(57) **Abstract:** A method of determining the viability of a gum for use in making a product has been achieved by (a) mixing a sample of the gum in a solvent comprising one or more hydroxy moiety containing solvent compound to form a solution with the gum; (b) measuring the viscosity of the solution and comparing against a predetermined target viscosity for a product; and (c) comparing the viscosity of the solution and the predetermined target viscosity and discarding gum which is outside the predetermined target viscosity; and (d) using the gum which has a viscosity within the predetermined target to prepare a product; wherein another hydroxy moiety containing solvent is used if water is used as a solvent. Also described are compositions comprising the gum and solvent comprising one or more hydroxy moiety.



METHOD OF MEASURING VISCOSITY OF GUMS

BACKGROUND

[0001] Gums (also known as hydrocolloids) are well known thickening agents, gelling agents and binding agents and have also been used as emulsifiers, foaming or whipping agents, encapsulating agents, film forming agents, flocculating or clarifying agents and crystallization inhibitors. See *Carbohydrates in Food*, ed. Ann-Charlotte Eliasson, pg. 285 (1996); see also *Food Chemistry*, ed. Owen Fennema, pg. 186-190 (1996).

[0002] Given their widespread utility, the gums are often consumed in vast quantities in industry. A problem associated with the use of gums is how to quickly ascertain a bad lot or batch of gum material provided by a bulk manufacturer of gums. Far too often, the unsuitability of the gum material is not detected until after the final product has been produced.

[0003] Complicating this problem is that feedstock for gums from natural origin are often the source of attribution to account for different viscosity patterns for gums received from different suppliers. Moreover, in order to obtain specific viscosity properties for their gums, suppliers often rely on proprietary mixing techniques to form their products.

[0004] Given the stringent regulatory laws and rules governing pharmaceutical and food compositions, it would be beneficial to detect any potential problems with a gum purchased from a supplier as early in the process of making a product as possible.

[0005] The comparison of viscosities are especially critical when using different suppliers or changing suppliers of the thickener or emulsion stabilizer compounds.

[0006] A common method of testing a gum sample is the use of gel permeation chromatography (GPC) with or without a size exclusion chromatography (SEC) system. A test sample is injected into the chromatographic system and the properties of the gum are determined via a detector system comprised of one or more of a refractive index detector and a viscometer.

[0007] The viscometric data generated can form a Mark-Houwink plot, the curvature of which can provide information about properties of the tested gum material.

[0008] However, the GPC/SEC system, in addition to being time consuming, suffers from needing expensive and sensitive equipment more suited for the small scale, controlled environment of a university laboratory rather the rigorous conditions of a factory floor.

[0009] Other methods for determining viscosity and the homogeneity of a gum include a rheological method (creep and recovery test) and the use of NIR (near infrared) spectroscopy.

[0010] The creep and recovery test, a sample reacts to constant shear stress for a certain period of time by deforming (“creep”). The sample is then relieved of the shear stress so that it can recover. See ASTM-D2990 (creep test); ASTM-D2991 (stress relaxation test)

[0011] However, this method also suffers from the problems of requiring high-cost test equipment (rheometer) and being a lengthy test process when measuring the time from sample preparation to final analysis of the tested sample.

[0012] The use of NIR spectroscopy also requires high-cost test equipment and its accuracy in determining viscosity, and therefore, the homogeneity of the gum, is uncertain.

[0013] Water has also been used with gums for the purposes of viscosity assessment by forming a slurry, dispersion or solution of the gum in water. However, this form of the gum often leads to incomplete dispersion or solubilization of the gum in water and to the formation of lumps which skews the viscosity measurements.

[0014] In addition, formation of such slurries, dispersion or solutions in water may require 24-48 hours for the gum to swell and impart its optimum viscosity.

[0015] Therefore, a need still exists in the art to quickly and accurately determine the viability of using a gum.

BRIEF SUMMARY

[0016] A method of determining the viability of a gum for use in making a product has been achieved by (a) mixing a sample of the gum in a solvent comprising one or more hydroxy moiety containing solvent compound to form a solution containing the gum; (b) measuring the viscosity of the solution and comparing against a predetermined target viscosity for a product; and (c) comparing the viscosity of the solution and the predetermined target viscosity and discarding gum which is outside the predetermined target viscosity; and (d) using the gum which has a viscosity within the predetermined target to prepare a product; wherein another hydroxy moiety containing solvent is used if water is used as a solvent.

[0017] Also described are compositions comprising the gum and solvent comprising one or more hydroxy moiety.

[0018] Further areas of applicability of the present invention will become apparent from the detailed description provided hereinafter. It should be understood that the detailed description and specific examples, while indicating the preferred embodiment of the invention, are intended for purposes of illustration only and are not intended to limit the scope of the invention.

DETAILED DESCRIPTION

[0019] The following description of the preferred embodiment(s) is merely exemplary in nature and is in no way intended to limit the invention, its application, or uses.

[0020] As used throughout, ranges are used as shorthand for describing each and every value that is within the range. Any value within the range can be selected as the terminus of the range.

[0021] In addition, all references cited herein are hereby incorporated by referenced in their entireties. In the event of a conflict in a definition in the present disclosure and that of a cited reference, the present disclosure controls.

[0022] Unless otherwise specified, all percentages and amounts expressed herein and elsewhere in the specification should be understood to refer to percentages by weight. The amounts given are based on the active weight of the material.

[0023] One embodiment of the invention is directed toward a method of determining the viability of a thickener or emulsion stabilizer compound lot for use in making an oral care product which comprises:

- (a) mixing a sample of the gum in a solvent comprising one or more hydroxy moiety containing solvent compound to form a solution containing the gum;
 - (b) measuring the viscosity of the solution and comparing against a predetermined target viscosity for a product; and
 - (c) comparing the viscosity of the solution and the predetermined target viscosity and discarding gum which is outside the predetermined target viscosity; and
 - (d) using the gum which has a viscosity within the predetermined target to prepare a product;
- wherein another hydroxy moiety containing solvent is used if water is used as a solvent.

[0024] One of ordinary skill in the art would be able to ascertain what constitutes the predetermined target viscosity based on their knowledge of the product that is intended to be produced.

[0025] The predetermined viscosity is determined by preparing a solution containing the thickening or emulsion stabilizer compound and measuring the solution in a viscometer. The measurement is repeated and the average of the first and second measurement is recorded as the final result.

[0026] If the final results is an unacceptable standard for establishing a predetermined viscosity suitable for the compound lot to be used in an oral care product, the amount of: (1) water and polyhydric alcohol or polyethylene glycol; or (2) thickening or emulsion stabilizer compound can be adjusted until a suitable predetermined viscosity is achieved.

[0027] In one embodiment of the method, the concentration of the gum in step (a) is 1-60% by weight, based on the weight of the solution and the gum is selected from the group consisting of modified celluloses, seed galactomannans, microbial polysaccharides, seaweed extracts/sulfated galactans, seaweed extracts/poly(uronic) acid, citrus peel extracts/poly(uronic) acid, exudate gums, starch, agar, collagen, egg whites, furcellaran, gelatin, casein and mixtures thereof.

[0028] In another embodiment of the method, the concentration of the gum in step (a) is 2-50% by weight, based on the weight of the solution and the gum is selected from the group consisting of carboxymethylcellulose, methylcellulose, hydroxypropylmethylcellulose, microcrystalline cellulose, guar gum, locust bean gum, xanthan gum, carrageenan, alginates, pectins, gum arabic, starch, agar, collagen, egg whites, furcellaran, gelatin, casein and mixtures thereof.

[0029] In one embodiment of the method, the gum is a carboxymethylcellulose or a carrageenan.

[0030] In one embodiment of the method, the solvent containing one or more a solvent comprising one or more hydroxy moiety is selected from the group consisting of a polyhydric alcohol, a monohydric alcohol, polyethylene glycol, polypropylene glycol, water and mixtures thereof.

[0031] The polyhydric alcohols include, but are not limited to, glycol, glycerol (glycerin), propylene glycol, ethylene glycol, erythritol, threitol, arabitol, xylitol, ribitol, mannitol, sorbitol, galactitol, fucitol, iditol, inositol and mixtures thereof.

[0032] The monohydric alcohols include, but are not limited to, alkyl alcohols (e.g. methanol, ethanol, propanol, butanol (all forms), pentanol (all forms), etc.), stearyl alcohols, fatty alcohols (e.g. a C₁₂-C₂₂ fatty alcohol) and mixtures thereof.

[0033] Polyethylene glycol has the chemical formula H-(O-CH₂CH₂)_nOH and polypropylene glycol has the chemical formula H-(O-CH(CH₃)CH₂)_nOH. These alcohols include, but are not

limited to glycols wherein n is selected from a range consisting of 2 - 2000, 10 - 5000, 20 - 2000, 200 - 1000 and 400 - 800.

[0034] In one embodiment of the invention, the solvent is a mixture of one or more of the specific polyhydric alcohols, monohydric alcohols, polyethylene glycols, polypropylene glycols described above and water.

[0035] In one embodiment of the method the solvent is selected from the group consisting of polyethylene glycol wherein n is 200 - 1000, glycerin, water and mixtures thereof.

[0036] In one embodiment of the method, the solvent comprising one or more hydroxy moiety containing solvent compound is water and a polyhydric alcohol and the ratio of water to polyhydric alcohol is from 2:1 to 1:2.

[0037] In one embodiment of the method, the solvent comprising one or more hydroxy moiety containing solvent compound is water and a polyhydric alcohol and the ratio of water to polyhydric alcohol is from 4:3 to 3:4.

[0038] In one embodiment of the method, the solution has a concentration selected from the group consisting of 10-50 wt.%, 25-45 wt.%, 1-6 wt.%, 2.5-4.5 wt.% and 3-4 wt.% of the gum.

[0039] In one embodiment of the method, the solvent is water and the polyhydric alcohol glycerin.

[0040] In one embodiment of the method, the solvent is polyethylene glycol where n is 400-800.

[0041] In one embodiment of the method, the gum is first mixed with the polyhydric alcohol to form an intermediate compound/alcohol solution and subsequently, the intermediate compound/alcohol solution is mixed with water to form the solution containing the gum.

[0042] In one embodiment of the method, the predetermined target viscosity is between 50,000 and 200,000 cP after testing a solution containing 1.5 - 6 wt.% of the gum between 30 minutes and 90 minutes after formation of the solution.

[0043] In one embodiment of the method, the predetermined target viscosity is between 100,000 and 200,000 cP after testing a solution containing 4 wt.% of the gum between 50 minutes and 70 minutes after formation of the solution or between 50,000 and 75,000 cP after testing a solution containing 3 wt.% of the gum between 50 minutes and 70 minutes after formation of the solution.

[0044] In one embodiment of the method, the predetermined target viscosity is between 225,000 and 600,000 cP after testing a solution containing 1.5 – 6 wt.% of the gum between 18 hours and 30 hours after formation of the solution.

[0045] In one embodiment of the method, the predetermined target viscosity is between 450,000 and 600,000 cP after testing a solution containing 4 wt.% of the gum between 22 hours and 26 hours after formation of the solution or between 225,000 and 300,000 cP after testing a solution containing 3 wt.% of the gum between 22 hours and 26 hours after formation of the solution.

[0046] In another embodiment of the method, the gum is used if the viscosity measurement is within a range selected from the group consisting of $\pm 35\%$, $\pm 30\%$ and $\pm 25\%$ of the predetermined target viscosity after the initial measurement.

[0047] In one embodiment of the method, the product is selected from the group consisting of oral care product, a home care product, a personal care product, a pharmaceutical product, a food product, a lubricant product, a paint product, a detergent product, a textile sizing product and a paper product.

[0048] In one embodiment of the method, the oral care product is a toothpaste, mouthwash, mouthrinse, dental floss and strips.

[0049] Another embodiment of the invention is a solution consisting of a gum and a solvent comprising one or more hydroxy moiety containing solvent compound to form a solution containing the gum wherein the solution has a concentration selected from the group consisting of 1-60 wt.%, 2-50 wt.%, 10-50 wt.%, 25-45 wt.%, 1-6 wt.%, 2.5-4.5 wt.% and 3-4 wt.% of the gum wherein another hydroxy moiety containing solvent compound if water is present.

[0050] In one embodiment of the solution, the gum is selected from the group consisting of selected from the group consisting of modified celluloses, seed galactomannans, microbial polysaccharides, seaweed extracts/sulfated galactans, seaweed extracts/poly(uronic) acid, citrus peel extracts/poly(uronic) acid, exudate gums, starch, agar, collagen, egg whites, furcellaran, gelatin, casein and mixtures thereof and the the solvent comprising one or more hydroxy moiety containing solvent compound is selected from the group consisting of a polyhydric alcohol, a monohydric alcohol, polyethylene glycol, polypropylene glycol, water and mixtures thereof.

[0051] In one embodiment of the solution, the gum is selected from the group consisting of carboxymethylcellulose, methylcellulose, hydroxypropylmethylcellulose, microcrystalline cellulose, guar gum, locust bean gum, xanthan gum, carrageenan, alginates, pectins, gum arabic,

starch, agar, collagen, egg whites, furcellaran, gelatin, casein and mixtures thereof, and the solvent comprising one or more hydroxy moiety containing solvent compound is selected from the group consisting of glycol, glycerol (glycerin), propylene glycol, ethylene glycol, erythritol, threitol, arabitol, xylitol, ribitol, mannitol, sorbitol, galactitol, fucitol, iditol, inositol, methanol, ethanol, propanol, butanol, pentanol, stearyl alcohols, a C₁₂-C₂₂ fatty alcohol, a polyethylene glycol or polypropylene glycol where n is selected from a range consisting of 2 - 6000, 50 - 3000, 100 - 2000, 200 - 1000, and 400 - 800 and water, wherein another hydroxy moiety containing solvent compound if water is present. At high MW, the polypropylene glycol is melted into liquid form before mixing with the gum.

[0052] In another embodiment of the solution,

- (a) the gum is carboxymethylcellulose and the solvent is water and glycerin; or
- (b) the gum is carboxymethylcellulose or carrageenan and the solvent is a polyethylene

[0053] Embodiments of the present invention are further described in the following examples. The examples are merely illustrative and do not in any way limit the scope of the invention as described and claimed.

EXAMPLES

Example 1 - Preparation of 3% CMC sample solution in water and glycerin

[0054] Equipment – Viscometer: Brookfield Model RVDV-II+ equipped with Helipath Stand and T-Bar Spindle T-C; Overhead Stirrer: IKA Model Eurostar power control – visc equipped with two-bladed centrifugal stirrer or four bladed propeller stirrer.

[0055] In a 600 mL beaker, 242.50 g of glycerin (A) was weighed. In a separate container, 242.50 g of water (B) was weighed. In a separate container, 15.00 g sample of sodium CMC (C) was weighed. Using the overhead stirrer, glycerin (A) was agitated at 1000 rpm. Sodium CMC (C) was added to the glycerin (A) under agitation at 1000 rpm and allowed to mix for 5 minutes. Water (B) is added to the mixture of (A) + (B) under agitation at 1000 rpm. The mixing speed is then raised to 2000 rpm and the agitation of the mixture of (A) + (B) + (C) continues for 15 minutes. The container containing the mixture of (A) + (B) + (C) is covered with plastic film and placed in a 25°C temperature controlled environment until further use.

Example 2 - Measurement of viscosity after 1 hour and 24 hours

[0056] One hour after the preparation of the 3% CMC sample solution of Example 1, the viscometer is set to spindle type S93 (T-C), a 1 minute stop time and a speed of 2 rpm. The spindle (T-C) is inserted in to the 3% CMC sample solution using the disengaging lever of the Helipath stand.

[0057] The spindle shaft is entirely immersed in the test sample and the reversing rod of the helipath is fully pressed down. The helipath stand is turned on and measurement is started with the viscometer. The viscosity in cP and % torque is recorded upon completion of the measurement.

[0058] The measurement steps described above is repeated by inserting the spindle at a different point of the 3% CMC sample solution. The average between the first and second readings is recorded as the final viscosity result. The 3% CMC sample solution is covered with plastic film and placed in a 25°C temperature controlled environment until further use.

[0059] 24 hours after the preparation of the 3% CMC sample solution of Example 1, viscosity is measured by repeating the steps described above for one hour after preparation.

Example 3 - Comparison of CMC lots with known CMC lots with unacceptable viscosity

[0060] 3% CMC sample solution from CMC lot #50011 (high viscosity) and CMC lot #67109 (low viscosity) were measured for viscosity against 3% CMC sample solution from different CMC lots (#149911 and #146811) using the methods described in Example 2. The results of these measurements is presented in Table 1 below.

[0061] Table 1

CMC Lot	Viscosity after 1 hr (cPs)	Viscosity after 24 hrs (cPs)
CMC Lot # 50011 (high viscosity)	90,000	340,000
CMC Lot # 67109 (low viscosity)	40,000	190,000
CMC Lot # 149911	55,000	275,000
CMC Lot # 146811	60,000	260,000

[0062] Toothpastes prepared with CMC from Lot # 50011 and # 67109 had unacceptable viscosities especially when compared with toothpastes prepared with CMC from Lot # 149911 and Lot # 146811, which presented an expected viscosity behavior.

Example 4 - Comparison of CMC lots with known CMC lots with unacceptable viscosity

[0063] 4% CMC sample solution from CMC lot #50011 (high viscosity) and CMC lot #67109 (low viscosity) were measured for viscosity against 4% CMC sample solution from different CMC lots (#149911 and #146811) using the methods described in Example 2. The 4% CMC sample solutions were prepared by using the methods described in Example 1 and increasing the amount of CMC sample taken. The results of these measurements is presented in Table 2 below.

[0064] Table 2

CMC Lot	Viscosity after 1 hr (cPs)	Viscosity after 24 hrs (cPs)
CMC Lot # 50011 (high viscosity)	200,000	655,000
CMC Lot # 67109 (low viscosity)	70,000	380,000
CMC Lot # 149911	155,000	530,000
CMC Lot # 146811	165,000	555,000
CMC Lot # 189811	165,000	520,000
CMC Lot # 126011	160,000	474,000
CMC Lot # 101511	180,000	530,000

[0065] Toothpastes prepared with CMC from Lot # 50011 and # 67109 had unacceptable viscosities for consumer use especially when compared with toothpastes prepared with CMC from Lot # 149911, 146811, 189811, 126011 and 101511.

Example 5 – Preparation of CMC sample in polyethylene glycol (PEG 600)

[0066] A silica based dentifrice was prepared which had the formula outlined in Table 3 below:

Table 3

Ingredient	% w/w
SORBITOL 70% Sol	50.000 – 85.00
PEG 600	0.500 – 2.000
CMC TMS (carboxymethylcellulose)	0.500 – 2.000
SACCHARIN SODIUM	0.100 – 0.500
SMFP (sodium monofluorophosphate)	0.500 – 1.000
FD & C RED 40 (CI16035)	0.001 – 0.010
THICKENER SILICA	5.000 – 10.000
AMORPHOUS SILICA	5.000 – 10.000
SLS GRANULES (sodium lauryl sulfate)	1.000 – 5.000
BETAINE, 30% SOLUTION	0.500 – 2.500
FLAVORING	0.500 – 2.000

OUTBURST FILM	0.050 – 0.250
CP PURIFIED WATER	q.s.
TOTAL	100.000

[0067] During the manufacturing process of this dentifrice, the carboxymethylcellulose supplied by Supplier #1 resulted in a dentifrice product density of about 13 bku on average initially (bku = British kinetic unit; 1 bku x 10,000 = cps). The same dentifrice product was made with carboxymethylcellulose supplied by Supplier #2 resulted in a dentifrice product density of 18.55 bku initially (see Table 4 below)

Table 4 (viscosity of dentifrice in bku)

Batch No. of CMC used	Initial	Day 1	Day 2	Day 3	Day 4	Day 5	Day 6	Day 7	% incr.
6371-206 Supplier #1	12.42	14.67	16.84	16.09				16.76	34.9
6371-210 Supplier #1	13.48	15.58				19.45		22.97	70.4
6371-208 Supplier #2	18.55	26.07	27.19				31.14	34.48	85.9
% difference between Supplier #1 and Supplier #2	49.3 37.6	77.7 67.3						105.7 50.1	

[0068] As can be seen in Table 4, the gums used in the process of making the dentifrice resulted in vastly different dentifrice properties despite being formed with ostensibly the same compound, i.e. carboxymethylcellulose (CMC). Although, the data shows that viscosity buildup was worse for the Supplier #1 product both initially and over seven days relative to the Supplier #2 product, it would have still been beneficial to see a more immediate indicator that the Supplier #1 lot should not have been used in the formation of the dentifrice.

[0069] Various components of the dentifrice were combined with the CMC and the combination of CMC with PEG 600 resulted in a clear distinction between the CMC used.

Table 5 (40 wt % CMC in PEG 600)

Supplier →	Supplier #2	Supplier #1					
Lot number	111015015	1210118	1210119	1210120	02	03	04
Viscosity (cps)	968	400	424	416	464	424	464
% increase in Supplier #2 product		142	128	133	109	128	109

relative to Supplier #1							
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[0070] As can be seen from the data in Table 5, the use of PEG 600 provided an instantaneous distinction between the CMC lots provided by Supplier #2 and Supplier #1.

[0071] Similar tests were run for two different suppliers of CMC

Table 6 (30 wt % CMC in PEG 600)

Supplier →	Supplier #3	Supplier #4
Lot number	AA6275042	21991
Viscosity (cps)	1144	1020
% increase in Supplier #3 relative to Supplier #4		12.1

[0072] As can be seen from the data in Table 6, the viscosities of the different products were within 12.1% of each other which could make them suitable for interchangeable use in the production of a dentifrice product assuming a permissible variance in viscosity of $\pm 25\%$.

Example 6 – Preparation of carrageenan sample in polyethylene glycol (PEG 600)

[0073] The test method from Example 5 as exemplified by the data in Table 5 was repeated except that carrageenan was used as the gum instead of CMC.

Table 7 (40 wt % carrageenan in PEG 600)

Supplier →	FMC	Shemberg		
Lot number	30110111	N1013-1	N1213-3	N1113-1
Viscosity (cps)	1496	1320	1336	1952
% increase in Supplier #2 product relative to Supplier #1		13.3	12.0	-23.3

[0074] As can be seen from the data in Table 6, the viscosities of the different products are suitable for interchangeable use in the production of a dentifrice product assuming a permissible variance in viscosity of $\pm 25\%$.

[0075] As those skilled in the art will appreciate, numerous changes and modifications may be made to the embodiments described herein without departing from the spirit of the invention. It is intended that all such variations fall within the scope of the appended claims.

CLAIMS

WHAT IS CLAIMED IS:

1. A method of determining the viability of a gum for use in making a product which comprises:
 - (a) mixing a sample of the gum in a solvent comprising one or more hydroxy moiety containing solvent compound to form a solution containing the gum;
 - (b) measuring the viscosity of the solution and comparing against a predetermined target viscosity for a product; and
 - (c) comparing the viscosity of the solution and the predetermined target viscosity and discarding gum which is outside the predetermined target viscosity; and
 - (d) using the gum which has a viscosity within the predetermined target to prepare a product; wherein another hydroxy moiety containing solvent is used if water is used as a solvent.
2. The method of claim 1, wherein concentration of the gum in step (a) is 1 - 60% by weight, based on the weight of the solution and the gum is selected from the group consisting of modified celluloses, seed galactomannans, microbial polysaccharides, seaweed extracts/sulfated galactans, seaweed extracts/poly(uronic) acid, citrus peel extracts/poly(uronic) acid, exudate gums, starch, agar, collagen, egg whites, furcellaran, gelatin, casein and mixtures thereof.
3. The method of claim 1 or 2, wherein the concentration of the gum in step (a) is 2 - 50% by weight, based on the weight of the solution and the gum is selected from the group consisting of carboxymethylcellulose, methylcellulose, hydroxypropylmethylcellulose, microcrystalline cellulose, guar gum, locust bean gum, xanthan gum, carrageenan, alginates, pectins, gum arabic, starch, agar, collagen, egg whites, furcellaran, gelatin, casein and mixtures thereof.
4. The method of any one of claims 1-3, wherein the gum is a carboxymethylcellulose or a carrageenan.

5. The method of any one of claims 1-4, wherein the solvent comprising one or more hydroxy moiety containing solvent compound is selected from the group consisting of a polyhydric alcohol, a monohydric alcohol, polyethylene glycol, polypropylene glycol, water and mixtures thereof.

6. The method of any one of claims 1-5, wherein the solvent comprising one or more hydroxy moiety containing solvent compound is selected from the group consisting of glycol, glycerol (glycerin), propylene glycol, ethylene glycol, erythritol, threitol, arabitol, xylitol, ribitol, mannitol, sorbitol, galactitol, fucitol, iditol, inositol, methanol, ethanol, propanol, butanol, pentanol, stearyl alcohols, a C₁₂-C₂₂ fatty alcohol, a polyethylene glycol or polypropylene glycol where n is selected from a range consisting of 2 - 6000, 50 - 3000, 100 - 2000, 200 - 1000, and 400 - 800 and water.

7. The method of any one of claims 1-6, wherein the solvent is selected from the group consisting of polyethylene glycol wherein n is 400 - 800, glycerin, water and mixtures thereof.

8. The method of any one of claims 1-7, wherein the solvent comprising one or more hydroxy moiety containing solvent compound is water and a polyhydric alcohol and the ratio of water to polyhydric alcohol is selected from the group of ratios consisting of 2:1 to 1:2 and 4:3 to 3:4.

9. The method of any one of claims 1-8, wherein the solution has a concentration of 10-50 wt.%, 25-45 wt.%, 1-6 wt.%, 2.5-4.5 wt.% and 3-4 wt.% of the gum of the gum.

10. The method of any one of claims 1-9, wherein the thickening or emulsion stabilizer compound is first mixed with the polyhydric alcohol to form an intermediate compound/alcohol solution and subsequently, the intermediate compound/alcohol solution is mixed with water to form the solution containing the thickening or emulsion stabilizer compound.

11. The method of any one of claims 1-10, wherein the predetermined target viscosity is between 50,000 and 200,000 cP after testing a solution containing 1.5 - 6 wt.% of the thickening

or emulsion stabilizer compound between 30 minutes and 90 minutes after formation of the solution.

12. The method of any one of claims 1-11, wherein the predetermined target viscosity is between 100,000 and 200,000 cP after testing a solution containing 4 wt.% of the thickening or emulsion stabilizer compound between 50 minutes and 70 minutes after formation of the solution or between 50,000 and 75,000 cP after testing a solution containing 3 wt.% of the thickening or emulsion stabilizer compound between 50 minutes and 70 minutes after formation of the solution.

13. The method of any one of claims 1-12, wherein the predetermined target viscosity is between 225,000 and 600,000 cP after testing a solution containing 1.5 – 6 wt.% of the thickening or emulsion stabilizer compound between 18 hours and 30 hours after formation of the solution.

14. The method of any one of claims 1-13, wherein the predetermined target viscosity is 450,000 and 600,000 cP after testing a solution containing 4 wt.% of the thickening or emulsion stabilizer compound between 22 hours and 26 hours after formation of the solution or between 225,000 and 300,000 cP after testing a solution containing 3 wt.% of the thickening or emulsion stabilizer compound between 22 hours and 26 hours after formation of the solution.

15. The method of any one of claims 1-14, wherein the gum is used if the viscosity measurement is within a range selected from the group consisting of $\pm 35\%$, $\pm 30\%$ and $\pm 25\%$ of the predetermined target viscosity after the initial measurement.

16. The method of any one of claims 1-15, the product is selected from the group consisting of oral care product, a home care product, a personal care product, a pharmaceutical product, a food product, a lubricant product, a paint product, a detergent product, a textile sizing product and a paper product.

17. The method of any one of claims 1-16, wherein the product is an oral care product which is selected from the group consisting of toothpaste, mouthwash, mouthrinse, dental floss and strips.

18. A solution consisting of a gum and a solvent comprising one or more hydroxy moiety containing solvent compound to form a solution containing the gum wherein the solution has a concentration selected from the group consisting of 1-60 wt.%, 2-50 wt.%, 10-50 wt.%, 25-45 wt.%, 1-6 wt.%, 2.5-4.5 wt.% and 3-4 wt.% of the gum wherein another hydroxy moiety containing solvent compound is present if water is present;

wherein the gum is selected from the group consisting of selected from the group consisting of modified celluloses, seed galactomannans, microbial polysaccharides, seaweed extracts/sulfated galactans, seaweed extracts/poly(uronic) acid, citrus peel extracts/poly(uronic) acid, exudate gums, starch, agar, collagen, egg whites, furcellaran, gelatin, casein and mixtures thereof; and

the solvent comprising one or more hydroxy moiety containing solvent compound is selected from the group consisting of a polyhydric alcohol, a monohydric alcohol, polyethylene glycol, polypropylene glycol, water and mixtures thereof.

19. The solution of claim 18, wherein the gum is selected from the group consisting of carboxymethylcellulose, methylcellulose, hydroxypropylmethylcellulose, microcrystalline cellulose, guar gum, locust bean gum, xanthan gum, carrageenan, alginates, pectins, gum arabic, starch, agar, collagen, egg whites, furcellaran, gelatin, casein and mixtures thereof, and the solvent comprising one or more hydroxy moiety containing solvent compound is selected from the group consisting of glycol, glycerol (glycerin), propylene glycol, ethylene glycol, erythritol, threitol, arabitol, xylitol, ribitol, mannitol, sorbitol, galactitol, fucitol, iditol, inositol, methanol, ethanol, propanol, butanol, pentanol, stearyl alcohols, a C₁₂-C₂₂ fatty alcohol, a polyethylene glycol or polypropylene glycol where n is selected from a range consisting of 2 - 2000, 10 - 5000, 20 - 2000, 200 - 1000, and 400 - 800 and water, wherein another hydroxy moiety containing solvent compound is present if water is present.

20. The solution of claim 19, wherein:

- (a) the gum is carboxymethylcellulose and the solvent is water and glycerin; or
- (b) the gum is carboxymethylcellulose or carrageenan and the solvent is a polyethylene glycol.

INTERNATIONAL SEARCH REPORT

International application No

PCT/US2014/068753

A. CLASSIFICATION OF SUBJECT MATTER

INV. C08L1/28 C08L5/00
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)

C08L

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, BIOSIS, EMBASE, MEDLINE, 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 2011/274811 A1 (HINES MARK EDWARD [US] ET AL) 10 November 2011 (2011-11-10) examples 1-3	1-20
X	----- Yasuhiko Sato: "Evaluation of Additives Required for Periodontal Disease Formulation Using Basic Fibroblast Growth Factor", 1 December 2010 (2010-12-01), XP055172260, Retrieved from the Internet: URL:https://www.jstage.jst.go.jp/article/cpb/58/12/58_12_1582/pdf [retrieved on 2015-02-26] page 1582, column 2, paragraph 4 - page 1583, column 2, paragraph 1 ----- -/--	1-20



Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents :

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"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

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Date of the actual completion of the international search

24 March 2015

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INTERNATIONAL SEARCH REPORT

International application No

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C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 2010/183697 A1 (SWANICK THOMAS M [US] ET AL) 22 July 2010 (2010-07-22) example 4	18-20
A	----- Peter S Francis: "Solution Properties of Water-Soluble Polymers. I. Control of Aggregation of Sodium Carboxymethylcellulose (CMC) by Choice of Solvent and/or Electrolyte", 1 January 1961 (1961-01-01), pages 261-270, XP055172404, Retrieved from the Internet: URL: http://onlinelibrary.wiley.com/doi/10.1002/app.1961.070051503/epdf [retrieved on 2015-02-26] the whole document -----	1-20

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/US2014/068753

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 2011274811 A1	10-11-2011	CA 2796434 A1	10-11-2011
		CN 102905550 A	30-01-2013
		EP 2566343 A1	13-03-2013
		JP 2013527768 A	04-07-2013
		US 2011274811 A1	10-11-2011
		WO 2011140440 A1	10-11-2011

US 2010183697 A1	22-07-2010	CN 102753118 A	24-10-2012
		EP 2490618 A1	29-08-2012
		JP 2013508033 A	07-03-2013
		US 2010183697 A1	22-07-2010
		WO 2011049833 A1	28-04-2011
