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(54) Air freshener gel

(57) A gel for the sustained release of an active ingredient, eg. fragrance, therefrom comprises an alginate cross-linked with borate ions. It may also contain a further polysaccharide and xanthan gum. The gel can be prepared without heating, is plant-derived and exhibits advantageous physical properties.

The claims were filed later than the filing date within the period prescribed by Rule 25(1) of the Patents Rules 1990.

This print takes account of replacement documents submitted after the date of filing to enable the application to comply with the formal requirements of the Patents Rules 1990.

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GEL

The present invention relates to a gel, and more particulary a gel comprising an alginate suitable for use as an air freshener.

Various gel compositions containing alginates have been described in the prior art for use as air fresheners.

US 4755377 describeds a gel, suitable for use as an air freshener which comprises a gelling agent (such as carrageenan, algin, amylose or agar) water, perfume, surfactant and thickener. The compositions are prepared by preparing the gel at a temperature above its glass transition temperature, injecting gas, shearing to form a foam and cooling below the glass transition temperature.

JP 53149183 describes a carrageenan gel containing sodium alginate and a polyvinyl alcohol for use as a gelling agent for an air freshener. The carrageenan gum and potassium chloride were mixed at high temperature with water and cooled to a gel.

JP 53032134 describes slow-releasing fragrance compositions prepared by mixing polysaccharides obtained from tamarind seed, sodium alginate, polyvinylpyrrolidone and agar, and dissolving the mixture in water by heating. A mixture of perfume, lower monohydric alcohol and alkylene glycol were added to the solution which was solidified to form a gel.

We have now developed a gel suitable for use as an air freshener which can be prepared without the application of heat. A gel according to the present invention is

also desirable in that it is plant derived and is therefore environmentally acceptable, and also exhibits advantageous physical properties such as reduced syneresis, rapid setting time and the like.

According to the present invention there is provided a gel for the sustained release of an active ingredient therefrom, which gel comprises an alginate cross-linked with borate ions.

Various alginates useful in this invention are described in detail by I.W. Cottrell and P. Kovacs in "Alginates," as Chapter 2 of Davidson, ed., Handbook of Water-Soluble Gums and Resins (1980). Most preferred herein are naturally derived algal sodium alginates, such as those sold commercially under the trademarks KELTEX, KELGIN and KELTONE™ by Kelco Division of Merck & Co., Inc.

Alginates also include "bioalgin", microbially produced polysaccharides produced by both Pseudomonas and Azotobacter Strains as described, for example, in Jarman et al., United States patent 4,235,966. These alginates are polysaccharides consisting of a partially acetylated variable block copolymer of D-mannuronic and L-guluronic acid residues. Jarman et al. state that the polysaccharide produced is similar to that produced from seaweed except that the molecule is partially acetylated.

A particularly preferred sodium alginate is that available under the trade mark KELTONE (KELTONE is a trade mark of Kelco Division of Merck & Co., Inc.). The use of this preferred alginate is advantageous in terms of the gelation properties of the resulting gel according

to the present invention (reduced slumping and syneresis) and also the colour and odorant properties thereof.

It is generally preferred that a light coloured gel is produced, and that the alginate is substantially odourless. The latter property is particularly advantageous when the gel is required for use as an air freshener, wherein the odour of the alginate should not interfere with that of the fragrance employed.

In the case where a firm gel is required, a high concentration of a low viscosity alginate is preferred. However, in the case of elastic gels, a lower concentration of a high viscosity alginate can be used.

As used herein, the terms low and high viscosity respectively denote viscosity measurements of the order of 2 to 6 (typically 4) m Pa.s and 295 to 305 (typically 300) m Pa.s, based on the viscosity of a 1% alginate solution. The terms low and high concentration as used herein to refer to the alginate concentration respectively denote 0.1 to 1% (typically 0.5%) alginate and 4 to 6% (typically not more than 5%) alginate, based on the weight of the final gel.

A particularly preferred gel comprises an alginate present in an amount of 1 to 5% by weight, based on the weight of the gel. More preferably the alginate is present in an amount of 2.5 to 3.5% by weight, based on the weight of the gel.

A typical source of borate ions comprises borax, advantageously together with a calcium salt, such as calcium lactate. The salt is typically employed to

enhance the cross-linking and gelation properties of the gel.

A particularly preferred source of borate ions comprises calcium tetraborate and/or sodium tetraborate. The use of this particularly preferred source of borate ions is beneficial in that it aids mixing thereof with the active ingredient.

Preferably the source of borate ions, typically calcium tetraborate, is present in the gel at a level of 0.3 to 1% by weight based on the weight of the gel. Advantageously the source of borate ions is present at a level of 0.5 to 0.6% by weight, based on the weight of the gel.

Preferably the gel further comprises a promoter for enhancing the gelation and cross-linking of the components of the gel. Preferred promoters include salts, such as potassium and/or calcium salts. The salts are typically present in an amount of 1.5 to 2.5% by weight, based on the weight of the gel. In a preferred embodiment, about 1.75 to 1.85% by weight, based on the weight of the gel, of potassium chloride is included in the gel. Levels of salt outside the above ranges were found to be detrimental, either in being present at too low a level to promote gelation resulting in gel products which slumped or when excess salt was present in promoting a fast reaction resulting in a sheared gel causing syneresis.

A gel according to the invention preferably further comprises a preservative. Suitably the preservative is

present in a minor amount in the gel, such as 0.01 to 0.15% by weight.

In a preferred embodiment the gel includes at least one further gellable polysaccharide in addition to an alginate. A particularly preferred further gellable polysaccharide being xanthan gum, an example of such a xanthan gum being available under the trade mark KELZAN (KELZAN is a trade mark).

Xanthan gum is an extracellularly produced gum made by the heteropolysaccharide-producing bacterium Xanthomonas campestris by the whole culture fermentation of a variety of conditions of medium comprising a fermentable carbohydrate, a nitrogen source and other appropriate nutrients.

Processes for producing xanthan gum are well known in the art and are described in a number of patents including U.S. patents Nos. 4,316,012, 4,352,882, 4,375,512, 3,671,398, 3,433,708, 3,271,267, 3,594,280, 3,591,578, 3,391,061, 3,020,206, 3,481,899 and 3,391,060 as well as British Patent No. 1,448,645.

The further gellable polysaccharide is typically present in an amount of 1 to 5% by weight, preferably 2.5 to 3.5% by weight, based on the weight of the gel.

According to a preferred aspect of the present invention, the gel includes an active ingredient for the sustained release thereof to a surrounding environment. A particularly preferred active ingredient comprises a fragrance. Typically the alginate and borate ions form a

solid matrix for receiving a fragrance emulsion which can be subsequently released from the gel.

A fragrance is typically included in the gel at a level of 1 to 5% by weight based on the weight of the gel. Preferably the fragrance is present in an amount of 2.5 to 3.5% by weight based on the weight of the gel. A gel according to the present invention is therefore particularly suitable for use as an air freshener, and is advantageous in that it exhibits environmentally desirable properties.

There is therefore further provided by the present invention an air freshener comprising a gel comprising an alginate cross-linked with borate ions, wherein said gel contains a fragrance suitable for sustained release therefrom

According to a further aspect of the present invention, there is provided a method of preparing a gel, which method comprises blending, at ambient temperature, an alginate with a source of borate ions in an aqueous environment so as to form an intimate mixture thereof, and allowing the mixture to set at ambient temperature to form a gel.

A method according to the present invention is particularly suitable for preparing a gel required for the sustained release of an active ingredient, such as a fragrance, therefrom substantially as hereinbefore described. The method preferably further involves blending an active ingredient, such as a fragrance, with an alginate and source of borate ions substantially as described above.

The present method is particularly beneficial in achieving blending and setting of the gel ingredients at ambient temperature, without the requirement of the application of heat. The cold setting (that is at ambient temperature) is advantageously rapid (approximately 10 to 20 minutes). The method is therefore energetically favourable, and as appreciated by a skilled addressee, is surprising in achieving blending and gelation at ambient temperature within the time limit specified above.

A further beneficial feature of the cold setting aspect of the present invention, is the avoidance of any detrimental effect on the active ingredient by the application of heat. This is particularly the case where the active ingredient comprises a fragrance.

The method preferably involves dispersing an alginate in an aqueous carrier, typically water, prior to blending with the source of borate ions. Preferably the source of borate ions is similarly dispersed in an aqueous carrier prior to blending with the alginate.

Preferably the alginate comprises a naturally derived sodium alginate substantially as hereinbefore described with reference to a gel according to the present invention. Preferably the alginate is employed in an amount substantially as hereinbefore described, based on the weight of a resultant gel.

It is preferred that the source of borate ions comprises calcium tetraborate and/or sodium tetraborate, although borax together with a calcium salt such as calcium lactate could be employed as discussed above. Preferably

the source of borate ions is employed at a level substantially as hereinbefore described with reference to a gel according to the present invention.

In a preferred embodiment sodium tetraborate is initially mixed with a calcium salt in an aqueous environment. Preferably calcium tetraborate is subsequently added to the resulting aqueous mixture.

The method preferably comprises intimately mixing the source of borate ions with at least one further gellable polysaccharide other than alginate, prior to blending with the latter. Preferably the further gellable polysaccharide comprises xanthan gum employed in an amount substantially as hereinbefore described.

It is further preferred that, prior to blending with an alginate, the source of borate ions is intimately mixed with an active ingredient such as a fragrance. Preferably the active ingredient is added to a resulting intimate mixture of the source of borate ions and further gellable polysaccharide prepared as above.

It is preferred that following dispersal of an alginate in an aqueous carrier, the alginate is subjected to substantially continuous agitation during the method. Preferably further ingredients such as gelation promoters, typically a potassium salt, are added to the aqueous alginate dispersion prior to blending with the source of borate ions. Addition of gelation promoters to the alginate dispersion, rather than the source of borate ions is advantageous in improving the mixing properties of an intermediate prepared from the source of borate ions.

It is preferred that the method employs high energy, high shear agitation of the aqueous alginate dispersion during addition of the source of borate ions thereto. Advantageously the high shear mixing has a speed of 5000rpm when employing a laboratory paddle mixer. The agitation is preferably such as to achieve intimate mixing of the alginate dispersion and source of borate ions within about 5 seconds. Typically aliquots of an aqueous dispersion of borate ions are added, suitably by injection, to the highly agitated alginate dispersion.

The resulting mix is poured into a suitable container and allowed to set at ambient temperature to form a gel. Gelation time is typically 10 to 20 minutes.

The present invention will now be further illustrated by the following example which does not limit the scope of the invention in any way.

Example

Ingredients

Part 1	% w/w	g
Tap water	70.70	141.40
sodium alginate	3.00	6.00
Trisodium citrate $2H_2O$	0.18	0.36
Potassium chloride	1.80	3.60
Preservative	0.05	0.10
Part 2		
Tap water	14.70	29.40
1% xanthan gum solution	3.00	6.00
Calcium tetraborate	0.54	1.08
Fragrance	3.00	6.00
Emulsifier	3.00	6.00
Preservative	0.05	0.10

Preparation

Part 1

The preservative was initially added to water, following dispersal of the alginate (available under the trade mark KELTONE, a trade mark of Kelco Division Merck & Co., Inc.) in the resulting aqueous mixture. Trisodium citrate was added to the aqueoue mixture which was continuously stirred until full hydration was achieved. The potassium chloride was added, and the mixture stirred for 30 minutes.

Part 2

A 1% solution of xanthan gum (available under the trade mark KELZAN, a trade mark of Merck & Co., Inc.) was prepared in tap water. Calcium tetraborate, the preservative and the xanthan gum solution were dispersed in tap water. A separate blend containing the emulsifier and fragrance was prepared, and the resulting blend added to the tetraborate mixture.

The resultant mixture was homogenised gently for 20 seconds.

Gel preparation

Part 1 was mixed rapidly under high shear, and part 2 injected into the vortex formed by mixing part 1. Parts 1 and 2 were mixed together for approximately 5 seconds. The resultant mixture was poured into a container and allowed to set at ambient temperature. Gelation time was 15 minutes.

CLAIMS:

1. A gel for the sustained release of an active ingredient therefrom, which gel comprises an alginate cross-linked with borate ions.

2. A gel according to Claim 1, which comprises an alginate present in an amount of 1 to 5% by weight, based on the weight of the gel.

3. A gel according to Claim 1, wherein a source of borate ions comprises calcium tetraborate and/or sodium tetraborate.

4. A gel according to Claim 1, wherein a source of borate ions is present in the gel at a level of 0.3 to 1% by weight based on the weight of the gel.

5. A gel according to Claim 1, which further comprises at least one further polysaccharide in addition to an alginate.

6. A gel according to Claim 1, which further comprises xanthan gum.

7. A gel according to Claim 1, which further comprises at least one further polysaccharide in addition to an alginate, wherein said further polysaccharide is present in an amount of 1 to 5% by weight, based on the weight of the gel.

8. A gel according to Claim 1, which further comprises an active ingredient for the sustained release thereof to a surrounding environment.

9. A gel according to Claim 1, which further comprises potassium chloride present in an amount of 1.5 to 2.5% by weight, based on the weight of the gel.

10. A gel according to Claim 1, which comprises:-

(a) 1 to 5% of an alginate;
(b) 0.3 to 1% of calcium tetraborate;
(c) 1.5 to 2.5% of potassium chloride; and
(d) 1 to 5% of xanthan gum;
by weight, based on the weight of the gel.

11. An air freshener comprising a gel comprising an alginate cross-linked with borate ions, wherein said gel contains a fragrance suitable for sustained release therefrom.

12. An air freshener according to Claim 11, wherein said fragrance is included in the gel at a level of 1 to 5% by weight based on the weight of the gel.

13. An air freshener according to Claim 11, which comprises:-

(a) 1 to 5% of an alginate;
(b) 0.3 to 1% of calcium tetraborate;
(c) 1.5 to 2.5% of potassium chloride;
(d) 1 to 5% of xanthan gum; and
(e) 1 to 5% of a fragrance;
by weight, based on the weight of the gel.

14. A method of preparing a gel, which method comprises blending, at ambient temperature, an alginate with a source of borate ions in an aqueous environment so

as to form an intimate mixture thereof, and allowing the mixture to set at ambient temperature to form a gel.

15. A method according to Claim 14, which further involves blending an active ingredient with an alginate and a source of borate ions.

16. A method according to Claim 14, wherein the source of borate ions comprises calcium tetraborate and/or sodium tetraborate.

Patents Act 1977
Examiner's report to the Comptroller under Section 17
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Relevant Technical Fields

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Search Examiner
P N DAVEY

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8 MARCH 1995

Databases (see below)
(i) UK Patent Office collections of GB, EP, WO and US patent specifications.

(ii) ONLINE: WPI

Documents considered relevant following a search in respect of Claims :-
1-16

Categories of documents

X: Document indicating lack of novelty or of inventive step.

Y: Document indicating lack of inventive step if combined with one or more other documents of the same category.

A: Document indicating technological background and/or state of the art.

P: Document published on or after the declared priority date but before the filing date of the present application.

E: Patent document published on or after, but with priority date earlier than, the filing date of the present application.

&: Member of the same patent family; corresponding document.

Category	Identity of document and relevant passages	Relevant to claim(s)
A	WPI Abstract Acc. No.90-102280/14 and JP 020051581 A (TAKASUGI SEIYAKU) see abstract	1 at least

Databases: The UK Patent Office database comprises classified collections of GB, EP, WO and US patent specifications as outlined periodically in the Official Journal (Patents). The on-line databases considered for search are also listed periodically in the Official Journal (Patents).