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(54) Title: EDIBLE OIL-SOLUBLE BLACK SUSPENSION

(57) Abstract: A black oil-based suspension, comprising carbo vegetabilis as a black pigment and further at least one emulsifier and at least one vegetable oil; and method for producing the oil-based suspension comprising dispersing the at least one emulsifier in the at least one vegetable oil to provide a vegetable oil dispersion and adding the carbo vegetabilis pigment to the vegetable oil dispersion and mixing said dispersion. The oil-based suspension may be used as a coloring agent in the manufacture of e.g. food products and pharmaceutical products.



EDIBLE OIL-SOLUBLE BLACK SUSPENSION

TECHNICAL FIELD

The present invention relates to a black oil-based suspension, comprising carbo vegetabilis as a black pigment. The oil-based suspension may be used as a coloring agent in the

5 manufacture of e.g. food products and pharmaceutical products.

BACKGROUND

Coloring agents containing natural or synthetic coloring substances are commonly used as additives in the manufacturing of food products and pharmaceutical products. A wide range of such coloring agents is commercially available making it possible for the manufacturer, where

10 a particular color tone is desired, to select a single coloring agent having the desired color or a mixture of coloring agents which in an appropriate combination impart the desired color to the product.

The commercially available coloring agents can contain synthetic substances including substances that are also normally referred to as dyes or azodyes, or such agents can contain

15 pigments or other coloring substances of natural origin, e.g. in the form of plant material containing a coloring substance, or as more or less purified coloring substances separated from plant, animal or microbial materials naturally containing such substances. There is a constant need for natural coloring substances covering all color tones and also for improving any desired property of a particular color substance. One particular color, which is not

20 commonly found in nature, is the color black. A source for obtaining a coloring substance providing a black color to e.g. food or pharmaceutical products is "carbo vegetabilis". This pigment substance is produced by the carbonization of vegetable material.

The present technology is based on carbo vegetabilis (also called vegetable carbon, E 153), which is a pigment in the form of a dusty powder. Carbo vegetabilis has a high surface-area-to-volume ratio and provides an intense black color. Carbo vegetabilis is, in its pure form, a

25 very light fine black powder which is extremely difficult to handle in industrial settings e.g. production of food or confectionary. It is easily spread by currents in the air and is very difficult to clean off surfaces, as it is very hydrophobic and not soluble in any normal cleaning agents, including acids, alkalis and organic solvents. When mixed in a liquid, carbo

30 vegetabilis has a tendency to agglomerate, and form sediments.

Presently, coloring substances containing carbo vegetabilis are produced by dispersing the pigment powder in glucose syrups or solutions of cellulose derivatives, thus, obtaining an paste which can be used "as is" or which can be processed to provide a further coloring composition. Commercially-available substances comprising carbo vegetabilis are not well suited for all applications particularly due to either:

- High viscosity or
- Low coloring strength
- or both

The problems associated with the above properties are among others that the high viscosity makes it difficult to move the products in a production plant by pumping and accordingly they must be transported manually. In addition, the low concentration of carbo vegetabilis makes it necessary to add increased volumes of the coloring substance in any particular application. Thus, large volumes of water need to be added possibly resulting in unwanted content of water and/or sugar in the food or pharmaceutical product.

RU 2548964 discloses a food coloring using vegetable coal in a water-soluble system.

One object of the present technology is therefore to provide a form of carbo vegetabilis which is suitable for industrial handling in e.g. food or pharmaceutical applications. In particular, the technology should provide an oil-miscible form of carbo vegetabilis, so that it can be used as a component of e.g. fat-containing foods. The suspension should not agglomerate or sediment over time. Furthermore, the dye suspension should provide an intense dark shade, which does not have "off" notes (smell or taste). Advantageously, the suspension should also be heat and oxidation stable.

SUMMARY

In a first aspect the present invention relates to an oil-based suspension for coloring of food or pharmaceutical products, said oil-based suspension comprising:

- carbo vegetabilis particles;
- at least one emulsifier;
- at least one vegetable oil;

wherein the carbo vegetabilis particles are dispersed in said vegetable oil.

A method is also provided for producing the oil-based suspension described herein, said method comprising the steps of:

- a. dispersing the at least one emulsifier in the at least one vegetable oil to provide a vegetable oil dispersion;
- b. adding the carbo vegetabilis pigment to the vegetable oil dispersion, and mixing said dispersion;

5 thereby providing the oil-based suspension according to the invention.

The use of the oil-based suspension of the invention for coloring of a food product or a pharmaceutical product is also provided, as is a food product or a pharmaceutical product comprising the oil-based suspension of the invention.

Further aspects of the invention are provided in the dependent claims.

10 DETAILED DISCLOSURE

The present inventors have found a way of preparing a stable, fat soluble carbo vegetabilis coloring composition which is water free and for which no heating step is required during processing. Creating a stable fat soluble, water free dispersion of a highly hydrophobic pigment such as carbo vegetabilis, which is a very porous material and therefore has a very large, very hydrophobic surface area is a challenge.

In the present context the term "carbo vegetabilis" and "carbo" are used interchangeably to denote the black pigment powder which can be produced by e.g. slow pyrolysis, the heating of wood, sugar, bone char, or other substances in the absence of oxygen. The terms "carbo vegetabilis", "carbo vegetalis", "vegetable black" or "vegetable carbon", are specifically used in legislation for identification of substances which can be used as food coloring substances. Under the current EU legislation carbo vegetabilis is identified by the E-number E153.

The present technology uses at least one emulsifier to stabilize the carbo vegetabilis in suspension and prevent agglomeration and sedimentation. The addition of an emulsifier allows the product to stabilize as a thick paste with no agglomeration or sedimentation over time.

Therefore, an oil-based suspension for coloring of food or pharmaceutical products is provided, said oil-based suspension comprising:

- a. carbo vegetabilis particles;
- b. at least one emulsifier;
- c. at least one vegetable oil;

wherein the carbo vegetabilis particles are dispersed in said vegetable oil.

This technology is based on the principle of the dispersion of carbo vegetabilis in oil using co-surfactants (emulsifier) to protect them from agglomerating due to their higher affinity with each other than their affinity with oil.

- 5 It should be noted that all percentages of the different materials/substances of the oil-based suspension (e.g. carbo vegetabilis) are calculated as w/w percentage.

The carbo vegetabilis particles may be present in an amount of between 0.5-25 wt%, preferably between 10-20 wt%, more preferably between 12-16 wt% based on the total suspension. In a particular embodiment of the present invention the amount is below 22,
10 such as below 20, such as below 18 wt%. In a particular embodiment of the present invention the amount is above 1 wt%, such as above 2, 4, 6, 8 wt%.

There is an interrelationship between particle size, surface area and resulting viscosity of a given dispersion. Thus, a high content of carbo vegetabilis in the oil-based suspension is more readily achieved with larger particle sizes without compromising the viscosity. However,
15 too high amounts of carbo vegetabilis, the oil-based suspension becomes too viscous.

Surprisingly, the present inventor has found that the use of an emulsifier makes it possible to disperse high amounts of carbo vegetabilis (even with a particle size of less than 10 μm) and – at the same time – obtain an excellent (low) viscosity as compared to known products.

The oil-based suspension typically, therefore, has a viscosity of between 500 cP and 6000 cP,
20 determined using a Brookfield DV-2T RV with spindle SC4-34 and a rotor speed of 100 RPM and at a temperature of 20 °C. Preferred viscosities are between 1000 cP and 5000 cP, more preferably between 2000 and 4000 cP under these conditions.

As previously mentioned, the carbo vegetabilis pigment powder particles have a very large total surface area due to the inherent nature of the carbon. The size of the carbo vegetabilis
25 powder particles is decisive for the surface area and hence the viscosity of the resulting oil-based suspension. A reduction in particle size results in increased surface area and thus an increase in viscosity when the pigment powder is dispersed. Color properties such as intensity and darkness of compositions tend to increase with decreasing pigment particle size. Viscosity and stability can also be affected with decreasing pigment particle size. Thus,
30 the average pigment particle size of the oil-based suspension of the invention is preferably less than 10 μm . Smaller particle size may be preferred for particular applications; such particle sizes include an average particle size of less than 6 μm , such as less than 4 μm .

However, an oil-based suspension with a larger particle size is also included by the present invention. Such larger particle sizes include an average size of 15 μm such as 20 μm , 30 μm , 40 μm , 50 μm , or even higher such as an average particle size of 60 μm , 70 μm or even 100 μm . The particle size is measured as D(4,3).

- 5 The at least one emulsifier is present in an amount of between 0.01-15 wt%, preferably between 0.1 – 10 wt%, more preferably between 0.1 – 5 wt% based on the total suspension. If more than one emulsifier is present, the amounts described herein refer to the total amount of all emulsifiers.

10 The at least one emulsifier may comprise a C4-C28 fatty acid monoglyceride or diglyceride or derivative thereof. In particular, the at least one emulsifier may comprise a C4-C6 organic acid ester of a C4-C28 fatty acid monoglyceride or diglyceride. Particular emulsifiers of interest are citric acid esters of mono and diglycerides of fatty acids (E472c, Lecithin E322, Mono- And Diglycerides Of Fatty Acids E 471, Sorbitan monooleate E494 and diacetyl tartaric acid ester of mono- and diglycerides (E472e). Particularly preferred is DATEM. These
15 emulsifiers have the correct hydrophilic-lipophilic balance (HLB) and dispersive effects for suspension of carbo vegetabilis in oil. Preferably, the at least one emulsifier is not derived from palm oil.

The at least one emulsifier has a hydrophilic-lipophilic balance (HLB) of 13 or less, 12 or less, or 11 or less. Suitably, the at least one emulsifier has a hydrophilic-lipophilic balance (HLB)
20 of 2 or more, 3 or more, 4 or more, or 5 or more. Emulsifiers having a HLB which is too high or too low risk not providing the required suspension stability.

The vegetable oil provides the "base", it is the carrier in which the other ingredients are mixed. Typically, therefore, it makes up the balance of weight in the oil-based suspension. To be able to make a suspension the oil is in liquid form. The at least one vegetable oil may be
25 selected from sunflower oil, canola oil, sunflower oil, canola oil, MCT oil, corn oil or rapeseed oil. Mixtures of these oils may also be possible. These vegetable oils in particular avoid undesired aroma or taste and provide a consistent oil-based suspension. Preferably, the at least one vegetable oil is a MCT coconut oil. In a similar manner to the emulsifier, the at least one vegetable oil is preferably not derived from palm oil. In a particular embodiment of the
30 present invention the suspension does not comprise palm oil. The vegetable oil is present in an amount of between 35-98.9 wt%, such as between 55-97wt%, preferably between 70-95 wt%, more preferably between 75 – 90 wt% based on the total suspension. In a particular embodiment the suspension comprises more than 50% wt vegetable oil, such as more than 60% wt vegetable oil, such as more than 70% wt or even more than 80% wt vegetable on
35 the total suspension. In one particular aspect, the oil-based suspension comprises

- a. 0.5-25 wt% carbo vegetabilis particles;
- b. 0.01-10 wt% emulsifier(s);
- c. balance vegetable oil;

in which the emulsifier and vegetable oil are as described herein.

- 5 In a particular embodiment of the present invention the suspension consists of Carbo vegetabilis, emulsifier and vegetable oil, preferably palm oil free vegetable oil.

The present technology also provides a method for producing the oil-based suspension as described herein, in which said method comprises the steps of:

- 10 a. dispersing the at least one emulsifier in the at least one vegetable oil to provide a vegetable oil dispersion;
- b. adding the carbo vegetabilis pigment to the vegetable oil dispersion, and mixing said dispersion;

thereby providing the oil-based suspension according to the invention.

The preparation of the oil-based suspension was done at ambient temperature.

- 15 It has been shown that the emulsifier must be dispersed in the vegetable oil before the carbo vegetabilis powder is added, in order to have optimum stabilization.

- 20 In the method, mechanical stirring and the use of a high shear mixer were tested and compared. High shear mixing does not add to the quality of the product. A stable intense black shade could be reached by simple mechanical stirring. Counterintuitively, it has been discovered that high shear mixing does not help reduce particle size in the suspension of this invention. Therefore, the mixing step (b) in the method of the invention is preferably not a high-shear mixing step. Low shear mixing is characterized by blending the substances without reducing the particle sizes during the method.

- 25 Most coloring agents in powder form are subjected to milling/grinding after suspension. This can provide improved properties with respect to stability or color. In the present technology, however, grinding does not provide positive effects on the particle size of the carbo vegetabilis in suspension. Accordingly, the method of the invention does not comprise a grinding step during or after step b.

The mixing time has also been shown to be significant: the more the product is mixed, the more intense the shade becomes. Suitably, therefore, the mixing step (b) takes place for a period of at least 30 minutes, preferably at least 60 minutes, more preferably at least 75 minutes.

- 5 The oil-based suspension of the invention can be used as all other coloring substances for providing a black color tone to any edible product intended for human or animal consumption. Additionally, the oil-based suspension may be used in the manufacturing of pharmaceutical products. The oil-based suspension of the invention may be blended or mixed with any other oil-based suspension/s to provide a desired color tone.
- 10 The present invention thus provides the use of oil-based suspensions described herein for coloring of a food product or a pharmaceutical product. The present technology also provides a food product or a pharmaceutical product comprising the oil-based suspension described herein. Of particular interest is a fondant paste comprising the oil-based suspension described herein and sugar.
- 15 Particular useful applications of the oil-based suspension of the present invention is the coloring of seasonings, spice blends, marinades, dressings, chocolate, fat based coatings, cream fillings for chocolate, cookies e.g. oreos, coloring of licorice tubes, flavored spreads, fondants, chewing gum and ice-cream.

The present invention is further illustrated by the following example.

20

EXAMPLES

Example 1

- 0.5 wt% emulsifier (E472e) mixed glycerol esters of mono- and diacetyltartaric acid and fatty acids of food fats was dispersed in 85 wt% MCT coconut oil. Then the carbo vegetabilis (14.5 wt%) was slowly added to the oil phase under mixing.
- 25

The product was then mixed by simple mechanical stirring (minimum 1h at ambient temperature 20-25°C). A gentle vortex was obtained around the mixer head, showing good mixing of the powder, in order to settle and stabilize the suspension.

In the first trials, a high shear (HS) mixer (Ultra Turax T25 digital from IKA at 16000 RPM) was used as well as a mechanical stirring. This is because high shear mixing is usually helpful to reduce particle size of the suspension, therefore providing a more stable system.

The product was mixed, then stirred mechanically for 10 minutes, then high shear (HS) stirring was performed for 10 minutes.

The L value, which represents the darkness of the sample, therefore the intensity of the black shade, was measured in a data color throughout the process. This was done by mixing 0.1% (i.e. 0.1 grams of oil suspension for 100 grams of the sugar and oil combination) of the oil product in 28.6 grams of MCT oil and 71.4 grams of icing sugar. The mixture was thoroughly mixed before measurement. The oil fondant described above was placed in a plastic measuring cup with lid. The sample was placed in front of a light source on a colorimeter, a Datacolor 650. The sample was measured in reflection mode with a large opening. The result was given on the LCh color scale, but since the color was black, only the L (lightness) value was useful to indicate the darkness of the sample.

For particle size, the sample was measured by laser diffraction on a Malvern Mastersizer 3000 with the Hydro MV cell, using a Fraunhofer model. The $d(3;2)$, $d(4;3)$ and $d(90)$ were measured.

The particle size of the suspension was followed through the process. The results are shown in Table 1:

	0.1% in Oil Fondant			
	L value	$d(3;2)$ μm	$d(4;3)$ μm	$d(90)$ μm
Stir 5 minutes	53.46			
Stir 10 minutes	52.76	2.388	3.733	7.028
High Shear 10 minutes	54.35	2.426	3.880	7.393

In Table 1, the L value represents the lightness of the product from black to white, therefore for a black product it represents the intensity of black obtained, whether it is more dark grey or intense black. The lower the L value, the darker the product is in the oil fondant.

$d(3;2)$ represents the median value of the particle size of the samples $d(4;3)$ represents the average particle size of the sample and $d(90)$ represents the value for which 90% of the volume distribution is below this value, 90% of the particles are below that value. This helps

determine the risk of sedimentation, as the higher the d(90), the higher the risk that some particles will sediment at the bottom of the sample.

It seems that the use of high shear mixing did not help reduce particle size; in fact it is quite the opposite since particle size increased. This is reflected in that the L value went up again, showing a lighter shade. This test did show that longer stirring times seemed to improve the dispersion and the darkness of the shade.

Another trial was then made to see how much mixing time could affect the L value and particle size. Again, the product was mixed as described previously and left to stir with only mechanical stirring for up to 2 hours.

The results are shown in Table 2:

	L	d(3;2)	d(4;3)	d(90)
stirring 15 minutes	57.08	2.524	3.809	7.718
Stirring 30 minutes	56.22	2.498	3.797	7.452
stirring 60 minutes	55.91	2.466	3.777	7.028
Stirring 75 minutes	55.01	2.399	3.658	6.876
stirring 120 minutes	54.95	2.386	3.612	6.842

It seems that longer stirring time provided a darker shade (lower L value) and better dispersion (lower particle size, especially d(90) which represents bigger particles). After 2 hours, a level was reached where neither shade or particle size evolved.

The samples from these trials were sent for testing in fat filling (Oreo filling type) and fat compound (white chocolate type). The application tests showed a very good performance of the product in both applications, with a good dispersibility of the product in the different matrices (no spots or undispersed particles).

The present invention has been described with reference to a number of examples, aspects and embodiments. However, the skilled person may combine features from various examples, aspects and embodiments while remaining within the scope of the appended claims.

CLAIMS

1. An oil-based suspension for coloring of food or pharmaceutical products, said oil-based suspension comprising:

- a. carbo vegetabilis particles;
- b. at least one emulsifier;
- c. at least one vegetable oil;

wherein the carbo vegetabilis particles are dispersed in said vegetable oil.

2. The oil-based suspension according to claim 1, wherein the carbo vegetabilis particles are present in an amount of between 0.5-25 wt%, preferably between 10-30 wt%, more preferably between 12-16 wt% based on the total suspension.

3. The oil-based suspension according to any one of the preceding claims, wherein the at least one emulsifier is present in an amount of between 0.01-15 wt%, preferably between 0.1 – 10 wt%, more preferably between 0.1 – 5 wt% based on the total suspension.

4. The oil-based suspension according to any one of the preceding claims, wherein the average particle diameter $d(4,3)$ of the carbo vegetabilis particles is less than 10 μm , preferably less than 6 μm , more preferably less than 4 μm .

5. The oil-based suspension according to any one of the preceding claims, wherein the at least one emulsifier comprises a C4-C28 fatty acid monoglyceride or diglyceride or derivative thereof.

6. The oil-based suspension according to any one of the preceding claims, wherein the at least one emulsifier comprises a C4-C6 organic acid ester of a C4-C28 fatty acid monoglyceride or diglyceride.

7. The oil-based suspension according to any one of the preceding claims, wherein the at least one emulsifier is selected from citric acid esters of mono and diglycerides of fatty acids (Citrem, E472c), Lecithin E322, Mono- And Diglycerides Of Fatty Acids E 471, Sorbitan monooleate E494 and diacetyl tartaric acid ester of mono- and diglycerides (DATEM, E472e), or mixtures thereof, preferably diacetyl tartaric acid ester of mono- and diglycerides (DATEM).

8. The oil-based suspension according to any one of the preceding claims, wherein the at least one emulsifier is not derived from palm oil.

9. The oil-based suspension according to any one of the preceding claims, wherein the at least one emulsifier has a hydrophilic-lipophilic balance (HLB) of 13 or less, preferably 12 or less, or more preferably 11 or less.

10. The oil-based suspension according to any one of the preceding claims, wherein the at least one emulsifier has a hydrophilic-lipophilic balance (HLB) of 2 or more, 3 or more, 4 or more, or 5 or more.

11. The oil-based suspension according to any one of the preceding claims, wherein the at least one vegetable oil is selected from sunflower oil, canola oil, sunflower oil, canola oil, MCT oil, corn oil or rapeseed oil.

12. The oil-based suspension according to any one of the preceding claims, wherein the at least one vegetable oil is a MCT coconut oil.

13. The oil-based suspension according to any one of the preceding claims, wherein the at least one vegetable oil is not derived from palm oil.

14. The oil-based suspension according to any one of the preceding claims, having a viscosity of between 500 cP and 6000 cP, preferably between 1000 cP and 5000 cP, more preferably between 2000 and 4000 cP, determined using a Brookfield DV-2T RV with spindle SC4-34 and a rotor speed of 100 RPM and at a temperature of 20°C.

15. The oil-based suspension according to any one of the preceding claims, comprising

- a. 0.5-25 wt% carbo vegetabilis particles;
- b. 0.01-15 wt% emulsifier(s);
- c. balance vegetable oil;

16. A method for producing the oil-based suspension according to any one of the preceding claims, said method comprising the steps of:

- a. dispersing the at least one emulsifier in the at least one vegetable oil to provide a vegetable oil dispersion;

- b. adding the carbo vegetabilis pigment to the vegetable oil dispersion, and mixing said dispersion;

thereby providing the oil-based suspension according to any one of the preceding claims.

17. The method according to claim 16, which does not comprise a grinding step during or
5 after step b.
18. The method according to any one of claims 16-17, wherein the mixing step (b) is not a high-shear mixing step.
- 10 19. The method according to any one of claims 16-18, wherein the mixing step (b) takes place for a period of at least 30 minutes, preferably at least 60 minutes, more preferably at least 75 minutes.
20. Use of an oil-based suspension according to any of claims 1-15 for coloring of a food
15 product or a pharmaceutical product.
21. A food product or a pharmaceutical product comprising the oil-based suspension according to any one of claims 1-15.
- 20 22. A fondant paste comprising the oil-based suspension according to any one of claims 1-15 and sugar.

INTERNATIONAL SEARCH REPORT

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A. CLASSIFICATION OF SUBJECT MATTER
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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)
A23L C09B C09D G03G

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, FSTA, BIOSIS

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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X	EP 3 566 583 A1 (CUETARA S L [ES]) 13 November 2019 (2019-11-13) example 2	21
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Further documents are listed in the continuation of Box C.



See patent family annex.

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"A" document defining the general state of the art which is not considered to be of particular relevance

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Name and mailing address of the ISA/

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Authorized officer

Muller, Isabelle

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2021/054437

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
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Information on patent family members

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