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(54) Title: HYBRID PIGMENT COMPOSITIONS COMPRISING CLAY AND NATURAL DYES

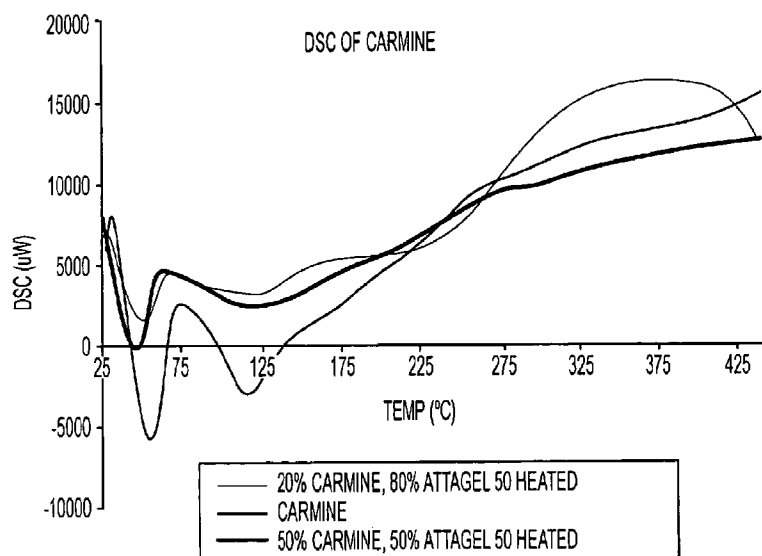


FIG. 2

(57) Abstract: A hybrid pigment composition is provided comprising a fibrous clay or charged bentonite and a natural dye selected from carmine, beta carotene, carmine hydrosoluble, turmeric, beet, and annato. The fibrous clay is a palygorskite, sepiolite, or a combination thereof. The natural dye is present in the composition in a range of 0.01 wt.% to about 75 wt.% based on the total weight of the hybrid pigment composition. The composition optionally includes water and a biodegradable polymer.

Hybrid Pigment Compositions Comprising Clay and Natural Dyes

RELATED APPLICATIONS

This application claims the benefit of priority of U.S. Provisional Application Serial No. 61/021,783, filed January 17, 2008. This application discloses subject matter similar to that disclosed in U.S. Patent Nos. 7,052,541; 7,425,235; and 7,429,294; and U.S. Application Serial No. 11/424,758, filed June 16, 2006; and U.S. Application Serial No. 12/068,115 filed February 1, 2008, the contents of each herein incorporated by reference in their entirety.

FIELD OF THE INVENTION

This invention relates to hybrid pigment compositions comprising clay and natural dyes and methods of preparing the hybrid pigments.

BACKGROUND

Colored minerals, earths and ochers, have been used throughout human history. Natural earth minerals lend themselves to a wide range of decorations, from body paint to painting on natural or constructed walls. The colors are extremely stable, as can be seen in ancient paintings that have lasted to this day. The use of colored earth pigments is found even in the oldest civilizations.

In the scientific literature, the term Maya blue refers to a “turquoise” brilliant shade of blue that is found on murals and archaeological artifacts, for example, throughout Mesoamerica. It is described in the literature as being composed of palygorskite clay and indigo, that when mixed and heated, produce the stable brilliant blue color similar to that found in Mesoamerica.

Proposed methods of preparation were performed with the intent of trying to replicate the blue color found at the historical sites and to reproduce the techniques employed by the original Maya.

H. Van Olphen, Rutherford Gettens, Edwin Littman, Anna Shepard, and Luis Torres were involved in the examination of organic/inorganic complex paint from the 1960's to the 1980's. In early studies, Littman and Van Olphen published information specifically on the synthesis of the Mayan organic/inorganic complex (Littman, *Amer. Antiquity*, 45:87-101, 1980; Littman, *Amer. Antiquity*, 47:404-408, 1982; Olphen, *Amer. Antiquity*, 645-646, 1966; Olphen, *Science*, 154:645-646, 1966). Their work did not describe the technique for making the colorant, nor explain the stability of the organic/inorganic complex. However, the results of their two decades of studies with respect to the ancient paint laid a foundation of knowledge for future investigators.

Littman synthesized indigo-attapulgitic complexes and verified that his synthetic version was indistinguishable from the original pigments found in the pre-Hispanic murals and artifacts (Littman, *Amer. Antiquity*, 45:87-101, 1980; Littman, *Amer. Antiquity*, 47:404-408, 1982). The prepared samples had the same physical and chemical characteristics as the authentic Maya blue examined. Littman concluded that the remarkable stability of the attapulgitic was due to the heat treatment the attapulgitic received during the synthesis. Others have also synthesized compounds similar to that of Maya blue by a number of routes (Torres, *Maya Blue: How the Mayas Could Have Made the Pigment*, Mat. Res. Soc. Symp., 1988). They employed the Gettens test to determine whether the laboratory synthesis of Maya blue was indeed authentic with the same chemical resistant properties (Gettens, *Amer. Antiquity*, 27:557-564, 1962). The test was necessary because initial attempts of simply mixing the palygorskite clay produced the color of

Maya blue but the mixture did not possess the same chemical properties as the original organic/inorganic complex samples.

Previous literature discussions of pH pertain to the alkaline pH required to reduce the indigo prior to contacting it with the clay (Littman, *Amer. Antiquity*, 45:87-101, 1980; Littman, *Amer. Antiquity*, 47:404-408, 1982). Moreover, there was a lack of understanding regarding the chemistry for producing stable and nontoxic paint systems by combining dyes and pigments with fibrous clays. U.S. Patent 3,950,180 describes color compositions that involve cationic organic basic colored compounds complexed to alkali-treated inorganic substances.

More recently, several patents and patent applications discussed indigo and related organic dyes complexed in an ionic interaction with inorganic supports. PCT Publication No. WO 01/04216 also describes ionic interactions in color compositions, wherein organic dyes undergo ion exchange with charged inorganic clays.

U.S. Patent 3,950,180 covers a method of manufacturing color compositions that include zeolite and montmorillonite. U.S. Patent 5,061,290 covers a method of using indigo derivatives as a dyeing agent. U.S. Patent 4,246,036 covers the method of manufacturing color compositions that are comprised of asbestos-cement. U.S. Patent 4,640,862 covers color compositions that are used for coating an expanded polystyrene "drop-out" ceiling tile. U.S. Patent 4,868,018 covers color compositions that are used with a mixture of epoxy resin, epoxy resin hardener, and portland cement to form a coating which can be applied to a surface to form simulated marble products. U.S. Patent 4,874,433 covers a method for encapsulating color compositions in and/or to a zeolite. U.S. Patent 5,574,081 covers a method of manufacturing waterborne clay-containing emulsion paints with improved application performance using color compositions. U.S. Patent 5,972,049 covers the method of manufacturing and using color compositions to form dye carriers used in the dyeing process for hydrophobic textiles. U.S.

Patent 5,993,920 covers the method of manufacturing and using color compositions with stone powder and/or cement powder, fine sawdust and/or the heart of a kaoliang stalk and other materials to form an incombustible artificial marble. U.S. Patent 6,339,084 covers the method of manufacturing thiazine-indigo pigments. U.S. Patent 6,402,826 covers the method and manufacturing of color compositions for paper coating.

U.S. Patents 7,052,541 and 7,429,294 describe color compositions comprising neutral indigo derivative pigments and dyes complexed to the surface of inorganic clays. These materials are useful as paints and coatings for artistic and industrial purposes, including use in cements, plastics, papers and polymers. Upon grinding and heating the organic and inorganic component as solid mixtures or in aqueous solutions, the resulting color compositions have unprecedented stability relative to the original starting materials. U.S. Patent No. 7,425,235 describes the use of similar starting materials in methods that rely on UV-light for preparing color compositions.

In the Middle Ages, new products derived from animal, vegetable, and mineral, appeared and were substituted for the ancient colors. Dyeing had always depended greatly on vegetable sources. Almost any shade of color could be produced from plants except green. But, the vegetable colors were not very light or water fast, and faded quickly. Because of the poor light-fastness clothing was dyed with plants from woods and heaths, mulberry and huckleberry for blues; sea or land lichen for mauves; weld, broom and berberis for yellow; wild madder, patience dock, and alkanet for red; walnut and chestnut bark for browns; sumac and gall nut for blacks. These colors produced a palette that was very dull in comparison to brighter, synthesized colors and were lacking in properties that are seen in synthesized dyes. For this reason, synthesized dyes are used in most applications.

Many natural dyes derived from plants and foods provide color and nutritional value. As consumer demand for natural products grow, natural colorants will likely replace synthetic colorants in many applications, especially food. Natural dyes, however, are dull in color, non-applicable in many plastic resins, and are very expensive.

SUMMARY

According to an embodiment of the present invention, natural dyes are reacted with a naturally occurring, FDA approved clay to produce a line of natural pigments containing unique colors and improved properties at a lower cost. The natural dyes are reacted onto the surface of the clay to provide a novel pigment with enhanced stability. The ability to produce stable colors with natural pigments in plastic resins makes them useful for food and food packaging applications. Several plastics / resins are now commercially available that are biodegradable.

In certain embodiments of the present disclosure, a hybrid pigment composition is provided comprising a fibrous clay and a natural dye selected from the group consisting of carmine, beta carotene, carmine hydrosoluble, turmeric, beet, and annato. In certain embodiments, the fibrous clay is a palygorskite, sepiolite, or a combination thereof. In certain embodiments, the composition comprises the natural dye in a range of 0.01 wt.% to about 75 wt.% based on the total weight of the hybrid dye composition. The hybrid pigment composition can further comprise water or a biodegradable polymer.

In certain embodiments of the present disclosure, a hybrid pigment composition is provided comprising a charged bentonite and a natural dye. In certain embodiments, the natural dye is selected from the group consisting of natural indigo, carmine, beta carotene, carmine hydrosoluble, turmeric, beet, and annato. In certain embodiments, the composition comprises the natural dye in a range of 0.01 wt.% to about 75 wt.% based on the total weight of the hybrid

pigment composition. The hybrid pigment composition can further comprise water or a biodegradable polymer.

In certain embodiments of the present disclosure, a method of forming a hybrid pigment is provided comprising the steps of providing a natural dye selected from the group consisting of carmine, beta carotene, carmine hydrosoluble, turmeric, beet, and annato and a fibrous clay, and mixing the natural dye and fibrous clay. In certain embodiments, the method further comprises heating the mixture of the natural dye and fibrous clay to react the natural dye with the fibrous clay. In certain embodiments, the method further comprises grinding the fibrous clay or natural dye prior to mixing the clay and natural dye, or grinding the natural dye and clay together after mixing the fibrous clay and natural dye. In certain embodiments, the method further comprises heating the fibrous clay and organic-inorganic hybrid pigment to a temperature of about 90 °C to about 200 °C for about 10 minutes to about 24 hours to react the natural dye with the fibrous clay.

In certain embodiments of the present disclosure, a method of forming a hybrid pigment is provided comprising the steps of providing a natural dye and a charged bentonite, and mixing the natural dye and charged bentonite. In certain embodiments, the natural dye is selected from the group consisting of natural indigo, carmine, beta carotene, carmine hydrosoluble, turmeric, beet, and annato. In certain embodiments, the method further comprises heating the mixture of the natural dye and charged bentonite to react the natural dye with the charged bentonite. In certain embodiments, the method further comprises grinding the charged bentonite or natural dye prior to mixing the charged bentonite and natural dye, or grinding the natural dye and charged bentonite together after mixing the natural dye and charged bentonite. In certain embodiments, the method further comprises heating the charged bentonite and natural dye to a temperature of

about 90 °C to about 200 °C for about 10 minutes to about 24 hours to react the natural dye with the charged bentonite.

Brief Description of the Drawings

Fig. 1 are differential scanning calorimetry curves of embodiments according to the present disclosure comprising natural indigo.

Fig. 2 are differential scanning calorimetry curves of embodiments according to the present disclosure comprising carmine.

DETAILED DESCRIPTION

In certain embodiments of the present disclosure, a hybrid pigment composition is provided comprising a fibrous clay or charged bentonite and a natural dye selected from the group consisting of natural indigo, carmine, beta carotene, carmine hydrosoluble, turmeric, beet, and annato. In certain embodiments, the fibrous clay is a palygorskite, sepiolite, or a combination thereof. The palygorskite can be Attagel 50, an FDA approved palygorskite. The charged bentonite is also known as bleaching earth, activated clay, Fuller's earth, and spent earth.

In certain embodiments, the composition comprises the natural dye in a range of about 0.01 wt.% to about 75 wt.% based on the total weight of the hybrid pigment composition. In certain embodiments, the composition comprises the natural dye in a range of about 1 wt.% to about 50 wt.% based on the total weight of the hybrid pigment composition. In certain embodiments, the composition comprises the natural dye in a range of about 10 wt.% to about 35 wt.% based on the total weight of the hybrid pigment composition.

In certain embodiments of the present disclosure, the hybrid pigment is used to color biodegradable polymers. Biodegradable polymers that can be colored by the hybrid pigment comprising a natural dye include: polylactic acid made from corn starch, poly(3-hydroxybutyric acid) made from sugar cane, and poly hydroxyl alkanooates made from starch.

The hybrid pigment composition according to the present disclosure can be prepared by mixing a natural dye, such as natural indigo, carmine, beta carotene, carmine hydrosoluble, turmeric, beet, and annato; and a fibrous clay or charged bentonite.

The natural dye can be reacted with the clay. In certain embodiments, the method of the present disclosure further comprises heating the mixture of the natural dye and fibrous clay to react the natural dye with the fibrous clay or charged bentonite. The natural dye can be reacted onto the surface of the fibrous clay or charged bentonite to provide a pigment with enhanced stability. To react the natural dye and the clay, the mixture can be heated to a temperature of about 90 °C to about 200 °C. In certain embodiments, the natural dye and clay are heated to a temperature of about 120 °C to about 180 °C. In certain other embodiments, the natural dye and clay are heated to a temperature of about 150 °C to about 170 °C. The mixture can be heated for about 10 minutes to about 24 hours. In certain embodiments, the natural dye and clay mixture is heated for about 1 to about 9 hours. In certain other embodiments, the natural dye and clay mixture is heated for about 2 to about 4 hours.

The natural dye and/or clay can be ground prior to mixing in certain embodiments. In other embodiments, the natural dye and clay are mixed together and then ground. Grinding can be performed by any suitable means, including mortar and pestle, coffee grinder, or kitchen blender.

In certain embodiments of the disclosure, the natural dyes are soluble in water and will only react in the presence of water. In such cases, water can be added to the mix of natural dye and clay to create a paste.

Examples

The natural dye and clay were ground. Water was added to mixtures with water soluble dyes, beta carotene, carmine hydrosoluble, beet, and annatto, to form a paste. The mixtures, both with and without water, were reacted by heating a mixture of clay and natural dye. Samples of the mixtures of dye and clay, both with and without water, were heated in a static oven at 90 °C, 150 °C, and 170 °C for about 3 hours. A change of color was observed at all temperatures indicating the natural dyes reacted with the clay, as shown in Table 1.

Table 1 - Color Change of Pigments When Reacted With Clay

EXAMPLE	NATURAL DYE	CLAYS	COLOR BEFORE HEATING	COLOR AFTER HEATING
1	Natural Indigo	Palygorskite, Sepiolite, Charged Bentonite	Dark Blue	Turquoise
2	Carmine	Palygorskite, Sepiolite, Charged Bentonite	Pink	Dark wine red
3	Beta Carotene	Palygorskite, Sepiolite, Charged Bentonite	Orange	Light Blue
4	Carmine Hydrosoluble	Palygorskite, Sepiolite, Charged Bentonite	Dark Blue	Reddish/Purple
5	Turmeric	Palygorskite, Sepiolite, Charged Bentonite	Yellow	Brown
6	Beet	Palygorskite, Sepiolite, Charged Bentonite	Pink	Dark Pink
7	Annato	Palygorskite, Sepiolite, Charged Bentonite	Light Brown	Dark Tan

Differential Scanning Calorimetry (DSC) was performed on the samples to measure the improvements in thermal stability. These DSC data demonstrate that samples according to the present disclosure provide hybrid pigments having a unique color space that can be used in resins and applications where natural dyes alone are not practical. Fig. 1 illustrates DSC curves of natural indigo; 20 wt.% natural indigo, 80 wt.% Attagel 50 (palygorskite) unheated; and 20 wt.% natural indigo, 80 wt.% Attagel 50 (palygorskite) heated. Fig. 2 illustrates DSC curves of natural carmine; 20 wt.% natural carmine, 80 wt.% Attagel 50 (palygorskite) heated; and 50 wt.% natural carmine, 50 wt.% Attagel 50 (palygorskite) heated.

The embodiments illustrated in the instant disclosure are for illustrative purposes only. They should not be construed to limit the claims. As is clear to one of ordinary skill in the art, the instant disclosure encompasses a wide variety of embodiments not specifically illustrated herein. While the compositions and methods of this disclosure have been described in terms of exemplary embodiments, it will be apparent to those of skill in the art that variations may be applied to the compositions and methods and in the steps or in the sequence of steps of the method described herein without departing from the concept, spirit and scope of the invention.

What is claimed is:

1. A hybrid pigment composition comprising a fibrous clay and a natural dye selected from the group consisting of carmine, beta carotene, carmine hydrosoluble, turmeric, beet, and annato.
2. The hybrid pigment composition of claim 1, wherein the fibrous clay is a palygorskite, sepiolite, or a combination thereof.
4. The hybrid pigment composition of claim 1, wherein the composition comprises said natural dye in a range of 0.01 wt.% to about 75 wt.% based on the total weight of the hybrid pigment composition.
5. The hybrid pigment composition of claim 1, further comprising water.
6. The hybrid pigment composition of claim 1, further comprising a biodegradable polymer.
7. A hybrid pigment composition comprising a charged bentonite and a natural dye.
8. The hybrid pigment composition of claim 7, wherein the natural dye is selected from the group consisting of natural indigo, carmine, beta carotene, carmine hydrosoluble, turmeric, beet, and annato.
9. The hybrid pigment composition of claim 7, wherein the composition comprises said natural dye in a range of 0.01 wt.% to about 75 wt.% based on the total weight of the hybrid dye composition.
10. The hybrid pigment composition of claim 7, further comprising water.
11. The hybrid pigment composition of claim 7, further comprising a biodegradable polymer.

12. A method of forming a hybrid pigment comprising the steps of providing a natural dye selected from the group consisting of carmine, beta carotene, carmine hydrosoluble, turmeric, beet, and annato; and a fibrous clay; and mixing the natural dye and fibrous clay.

13. The method of forming a hybrid pigment according to claim 12, further comprising heating the mixture of the natural dye and fibrous clay to react the natural dye with the fibrous clay.

14. The method of forming a hybrid pigment composition according to claim 12, further comprising grinding the fibrous clay or natural dye prior to mixing the clay and natural dye, or after mixing the natural dye with the clay, grinding the natural dye and clay together.

15. The method of forming a hybrid pigment composition according to claim 12, further comprising heating the fibrous clay and dye to a temperature of about 90 °C to about 200 °C for about 10 minutes to about 24 hours to react the natural dye with the fibrous clay.

16. A method of forming a hybrid pigment provided comprising the steps of providing a natural dye and a charged bentonite, and mixing the natural dye and charged bentonite.

17. The method of forming a hybrid pigment according to claim 16, wherein the natural dye is selected from the group consisting of natural indigo, carmine, beta carotene, carmine hydrosoluble, turmeric, beet, and annato.

18. The method of forming a hybrid pigment according to claim 16, further comprising heating the mixture of the natural dye and charged bentonite to react the natural dye with the charged bentonite.

19. The method of forming a hybrid pigment composition according to claim 16, further comprising grinding the charged bentonite or natural dye prior to mixing the charged bentonite and natural dye, or after mixing the natural dye with the charged bentonite, grinding the natural dye and charged bentonite together.

20. The method of forming a hybrid pigment composition according to claim 16, further comprising heating the charged bentonite and natural dye to a temperature of about 90 °C to about 200 °C for about 10 minutes to about 24 hours to react the natural dye with the charged bentonite.

1/2

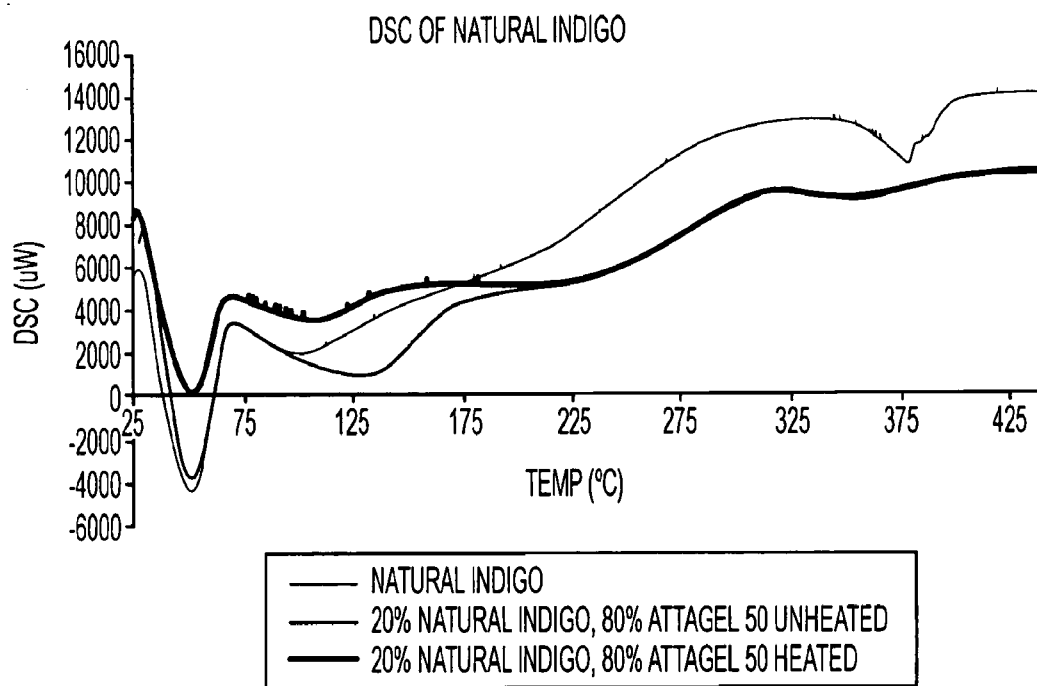


FIG. 1

2/2

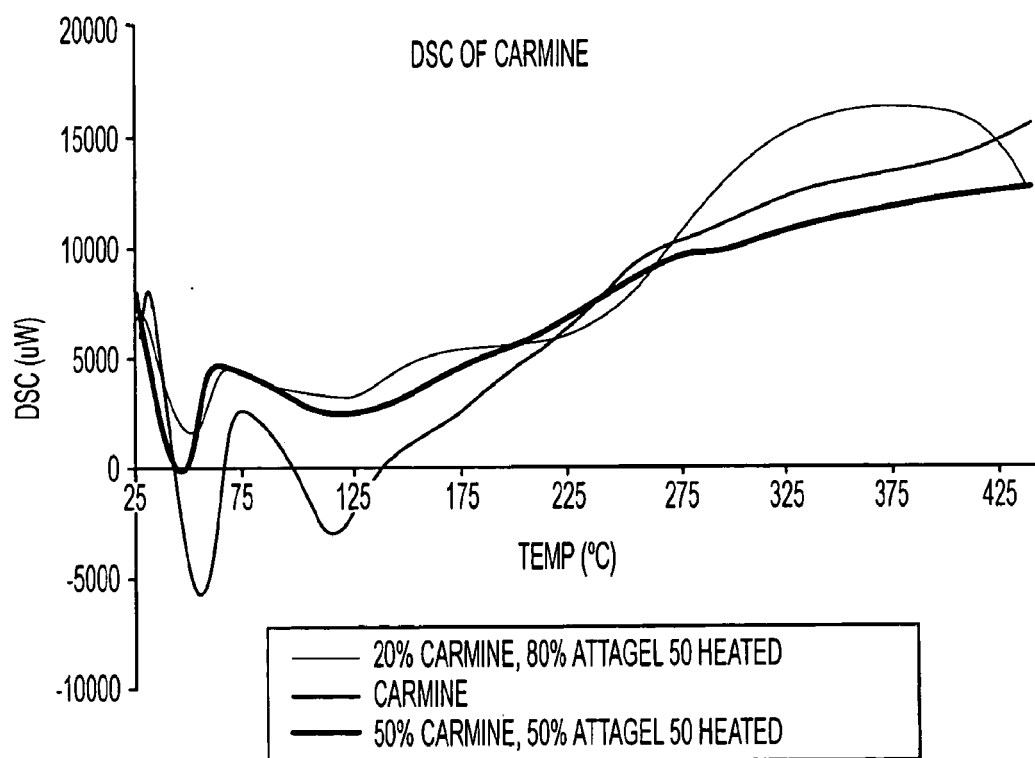


FIG. 2

INTERNATIONAL SEARCH REPORT

International application No.

PCT/US 09/31572

A. CLASSIFICATION OF SUBJECT MATTER

IPC(8) - C04B 14/00, 14/04 (2009.01)

USPC - 106/401,487

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)

IPC(8) - C04B 14/00, 14/04 (2009.01)

USPC - 106/401,487

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

IPC(8) - C04B 14/00, 14/04 (2009.01) (text search only - see search history printout)

USPC - 106/401,487 (text search only - see search history printout)

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

(USPT, PGPB, EPAB, JPAB); Google Patent, Google Scholar, Dialog Web

Search terms on extra sheet

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 2004/0011254 A1 (Chianelli et al.) 22 Jan 2004 (22.01.2004) (Abstract, para [0011], [0014], [0016], [0019], [0026], [0038], [0043], [0047]).	7-11, 16-20
Y		1-2, 4-6, 12-15
Y	US 2007/0189996 A1 (Weber et al.) 16 Aug 2007 (16.08.2007) (para [0020], [0061]).	1-2, 4-6, 12-15
A	US 5,962,553 A (Ellsworth) 05 Oct 1999 (05.10.1999) (Col.1 lines 34-38), teaches that bentonite is inherently negatively charged.	1-20

☐ Further documents are listed in the continuation of Box C.

* Special categories of cited documents:

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

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"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"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

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search

18 Feb 2009 (18.02.2009)

Date of mailing of the international search report

03 MAR 2009

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INTERNATIONAL SEARCH REPORT

International application No.

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continued from box B

Search terms:

palygorskite, sepiolite, attagel, dye, pigment, dye, fibrous clay, carmine, beta carotene, carmine
hydrosoluble, turmeric, beet, annato, water, biodegradable, polylactic, polyhydroxyl alkanoate,
polyhydroxyl ethanoate, polyhydroxyl propanoate, polyhydroxyl butanoate, polyhydroxyl pentanoate,
heating, polymer, charged bentonite, bleaching earth, activated clay, fullers earth, spent earth