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(54) **Title:** STABLE AND HOMOGENOUS ANTHRAQUINONE COMPOSITIONS AND PREPARATIONS THEREOF

(57) **Abstract:** Substantially homogeneous and stable suspensions of anthraquinone (AQ) compositions and methods of preparation are disclosed. The AQ compositions are utilized in pulping processes.



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STABLE AND HOMOGENOUS ANTHRAQUINONE COMPOSITIONS AND PREPARATIONS THEREOF

[0001] The present application claims priority to United States Provisional Application No. 62/130,950, filed March 10, 2015, the disclosure of which is incorporated by reference herein in its entirety.

Background

[0002] Anthraquinone (AQ) is a solid that is insoluble in water. There is a need to provide a stable and homogeneous suspension of anthraquinone to be used in pulping processes to provide pulp having good papermaking quality. "Pulping" generally refers to the reduction or break-down of a bulk fiber source material into its component fibers. Wood and other plant materials used to make pulp generally contain three main components (apart from water): cellulose fibers (desired for papermaking), lignin (a three-dimensional polymer that binds the cellulose fibers together) and hemicelluloses (shorter branched carbohydrate polymers). Fiber sources include wood fibers, e.g., soft woods and hard woods and mixtures thereof, and non-wood fibers obtained from agricultural productions, such as without limitation, bagasse, wheat straw, rice straw, corn stover (stalks, leaves and husks), soy residuals, coconut tissues, cotton stalks, palm baskets, kenaf, industrial hemp, seed flax straw, textile flax straw, sisal, hesperaloe, rye grass, and mixtures thereof.

Summary of Drawings

[0003] Figure 1 is a block flow diagram showing the preparation of an AQ composition according to an embodiment of the disclosure.

[0004] Figure 2 is a visual showing the stability over time of an AQ composition prepared according to an embodiment of the disclosure.

[0005] Figure 3 is a visual showing two AQ compositions prepared with and without magnesium chloride and calcium chloride.

[0006] Figure 4 is a visual showing the effect of a polyglycol in an AQ composition.

[0007] Figure 5 is a visual showing the effect of mean particle size on the stability of the AQ composition prepared according to an embodiment of the present disclosure.

Detailed Description

[0008] A more detailed description of the present disclosure will be rendered by reference to specific embodiments thereof. Other features and advantages of the present disclosure will become apparent from the following detailed description. It should be understood, however, that the detailed description and the specific examples, while indicating the preferred embodiments of the present disclosure, are given by way of illustration only, since various changes and modifications within the spirit and scope of the present disclosure will become apparent to those skilled in the art from this detailed description.

[0009] One aspect of the present disclosure relates to an AQ composition comprising AQ, one or more metal ions, a dispersant, a polyglycol, and water, wherein the AQ composition is a substantially homogeneous and stable suspension. One embodiment is a composition comprising anthraquinone, one or more calcium salts, one or more magnesium salts, a salt of diisopropylnaphthalene sulfonate, polyethylene glycol, and water, wherein the composition is a substantially homogeneous and stable suspension.

[0010] As used herein, the term “substantially homogeneous and stable” means the AQ in the “substantially homogeneous and stable” suspension is dispersed evenly and the AQ does not settle during extended storage for at least 24 hours. The settling of AQ in the suspension is determined by measuring a % Transparency, as described in the Examples section. A % Transparency of about 25% or less, about 20% or less, about 15% or less, or about 10% or less, is indicative of a substantially homogeneous and stable suspension.

[0011] As used herein, the term “about” means a range of $\pm 10\%$, $\pm 5\%$ or $\pm 1\%$.

[0012] The mean particle size of AQ in the AQ composition is about 150 μm or less, about 135 μm or less, about 125 μm or less, about 100 μm or less, about 75 μm or less, about 50 μm or less, about 25 μm or less, about 20 μm or less, about 15 μm or less, about 10 μm or less, about 8 μm or less, about 5 μm or less, about 3 μm or less, about 1 μm or less, 100 μm to about 1 μm , about 75 μm to about 1 μm , about 50 μm to about 1 μm , about 25 μm to about 1 μm , about 20 μm to about 1 μm , about 15 μm to about 1 μm , about 10 μm to about 1 μm , about 8 μm to about 1 μm , about 5 μm to about 1 μm , about 3 μm to about 1 μm , about 100 μm to about 3 μm , about 75 μm to about 3 μm , about 50 μm to about 3 μm , about 25 μm to about 3 μm , about 20 μm to about 3 μm , about 15 μm to about 3 μm , about 10 μm to about 3 μm , about 8 μm to about 3 μm , about 5 μm to about 3 μm , about 100 μm to about 5 μm , about 75 μm to about 5 μm , about 50 μm to about 5 μm , about 25 μm to about 5 μm , about 20 μm to about 5 μm , about 15 μm to about 5 μm , 10 μm to about 5 μm , about 8 μm to about 5 μm , about 100 μm to about 8 μm , about 75 μm to about 8 μm , about 50 μm to about 8 μm , about 25 μm to about 8 μm , about 20 μm to about 8 μm , about 15 μm to about 8 μm , about 10 μm to about 8 μm , about 100 μm to about 10 μm , about

75 μm to about 10 μm , about 50 μm to about 10 μm , about 25 μm to about 10 μm , about 20 μm to about 10 μm , about 15 μm to about 10 μm , about 100 μm to about 15 μm , about 75 μm to about 15 μm , about 50 μm to about 15 μm , about 25 μm to about 15 μm , about 20 μm to about 15 μm , about 100 μm to about 20 μm , about 75 μm to about 20 μm , about 50 μm to about 20 μm , about 25 μm to about 20 μm , about 100 μm to about 25 μm , 75 μm to about 25 μm , about 50 μm to about 25 μm , about 100 μm to about 50 μm , about 75 μm to about 50 μm , or about 100 μm to about 75 μm .

[0013] The AQ composition can include a concentration of AQ by weight (wt %) of about 50% or higher, about 30% to about 70%, about 40% to about 60%, about 45% to about 55%, about 50% to about 70%, about 50% to about 55%, or about 50%.

[0014] The metal ions, the dispersant, and the polyglycol are water soluble. The one or more metal ions may be monovalent, divalent, trivalent, tetravalent, their salts thereof, and mixtures thereof. The divalent metal ion salts include, without limitation, calcium salt(s) and magnesium salt(s), and they may be any water soluble salt, e.g. chlorides, bromides, nitrates.

[0015] In one embodiment, molar ratio of calcium to magnesium in the AQ composition is about 1:1, about 1.1:0.9, or about 0.9:1.1.

[0016] The dispersant may be a cationic dispersant, an anionic dispersant, or a nonionic dispersant. In one embodiment, the anionic dispersant is, for example and without limitation, an aromatic sulfonate, such as a naphthalene sulfonate, and preferably a dialkyl naphthalene sulfonate dispersant. Examples of dialkyl naphthalene sulfonate include, without limitation, sodium salts of dialkyl naphthalene sulfonic acid. In one embodiment, the dispersant is sodium

diisopropylnaphthalene sulfonate, for example Naxan® DIL dispersant, purchased from the Nease Corporation.

[0017] The concentration of the dispersant in the AQ composition is from about 0.1% to about 5%, from about 0.2% to about 3% or from about 0.5 to about 2%.

[0018] The polyglycol is a polymer or copolymer of alkylene oxides. In preferred embodiments, the alkylene oxides are ethylene oxide, propylene oxide and butylene oxide. The polyglycols are polyethylene glycol, polypropylene glycol, polybutylene glycol, and mixtures thereof.

[0019] The polyglycol comprises a polyglycol having an average molecular weight of from about 200 to about 5,000, from about 500 to about 3,000, from about 750 to about 2,000, or about 1,000. The concentration of a polyglycol in the AQ composition is from about 1% to about 10%, from about 2% to about 8%, from about 3% to about 6%, or about 5%. In one embodiment of the present disclosure, the polyglycol is polyethylene glycol.

[0020] In one embodiment, the molar ratio of AQ to the total calcium in the AQ composition is about 50:1, about 51:1 to about 49:1, or about 55:1 to about 45:1.

[0021] The AQ composition comprising 50% AQ (w/w) as disclosed herein has a viscosity of about 500 to about 10,000 cP at 25°C, about 1,000 to about 7,000 cP at 25°C, or less than about 2,000 to about 5,000 cP at 25°C.

[0022] The AQ composition as disclosed herein remains stable at room temperature in a closed container for about 24 hours or longer, about 1 week or longer, about 1 month or longer, about 3 months or longer, or about 6 months or longer. In some embodiments, the AQ composition is stable up to about a year. The AQ composition is generally more stable than a composition comprising the same components without the metal ion salts.

[0023] Another aspect of the present disclosure relates to an AQ composition comprising AQ having a mean particle size of about 150 μm or less, one or more metal ions, a dispersion, a polyglycol, and water. One embodiment is a composition comprising AQ having a mean particle size of about 150 μm or less, one or more calcium salts, one or more magnesium salts, a salt of diisopropylnaphthalene sulfonate, polyethylene glycol, and water. In certain embodiments, the mean particle size of AQ in the AQ composition is about 150 μm or less, about 135 μm or less, about 125 μm or less, about 100 μm or less, about 75 μm or less, about 50 μm or less, about 25 μm or less, about 20 μm or less, about 15 μm or less, about 10 μm or less, about 8 μm or less, about 5 μm or less, about 3 μm or less, about 1 μm or less, 100 μm to about 1 μm , about 75 μm to about 1 μm , about 50 μm to about 1 μm , about 25 μm to about 1 μm , about 20 μm to about 1 μm , about 15 μm to about 1 μm , about 10 μm to about 1 μm , about 8 μm to about 1 μm , about 5 μm to about 1 μm , about 3 μm to about 1 μm , about 100 μm to about 3 μm , about 75 μm to about 3 μm , about 50 μm to about 3 μm , about 25 μm to about 3 μm , about 20 μm to about 3 μm , about 15 μm to about 3 μm , about 10 μm to about 3 μm , about 8 μm to about 3 μm , about 5 μm to about 3 μm , about 100 μm to about 5 μm , about 75 μm to about 5 μm , about 50 μm to about 5 μm , about 25 μm to about 5 μm , about 20 μm to about 5 μm , about 15 μm to about 5 μm , 10 μm to about 5 μm , about 8 μm to about 5 μm , about 100 μm to about 8 μm , about 75 μm to about 8 μm , about 50 μm to about 8 μm , about 25 μm to about 8 μm , about 20 μm to about 8 μm , about 15 μm to about 8 μm , about 10 μm to about 8 μm , about 100 μm to about 10 μm , about 75 μm to about 10 μm , about 50 μm to about 10 μm , about 25 μm to about 10 μm , about 20 μm to about 10 μm , about 15 μm to about 10 μm , about 100 μm to about 15 μm , about 75

μm to about 15 μm , about 50 μm to about 15 μm , about 25 μm to about 15 μm , about 20 μm to about 15 μm , about 100 μm to about 20 μm , about 75 μm to about 20 μm , about 50 μm to about 20 μm , about 25 μm to about 20 μm , about 100 μm to about 25 μm , 75 μm to about 25 μm , about 50 μm to about 25 μm , about 100 μm to about 50 μm , about 75 μm to about 50 μm , or about 100 μm to about 75 μm .

[0024] Yet another aspect of the invention relates to a method for preparing the AQ composition as disclosed herein, comprising:

a. providing a first solution comprising one or more metal ion, a dispersant, and a polyglycol; and

b. adding AQ with agitation to the first solution at a speed such that the composition remains a substantially homogeneous and stable suspension, and the mean particle size of AQ remains about 150 μm or less.

[0025] The AQ composition is stable after being left at room temperature in a closed container for at least about 24 hours, greater than about 1 week, greater than about 1 month, greater than about 3 months, or about a year.

[0026] The metal ion salts (e.g., calcium and magnesium salts), the dispersant (e.g., a salt of diisopropylnaphthalene sulfonate), and the polyglycol (e.g., polyethylene glycol), may be added separately or together into water to provide the first solution. Alternatively, the metal ion salts, the dispersant, and the polyglycol may be provided as individual solutions and then mixed together to provide the first solution.

[0027] AQ may be added as a powder having a mean particle size of about 150 μm or less, about 135 μm or less, about 125 μm or less, about 100 μm or less, about 75 μm or less, about 50 μm or less, about 25 μm or less, about 20 μm or less, about 15 μm or less, about 10 μm or less, about 8 μm or less, about 5 μm or less, about 3 μm

or less, about 1 μm or less, 100 μm to about 1 μm , about 75 μm to about 1 μm , about 50 μm to about 1 μm , about 25 μm to about 1 μm , about 20 μm to about 1 μm , about 15 μm to about 1 μm , about 10 μm to about 1 μm , about 8 μm to about 1 μm , about 5 μm to about 1 μm , about 3 μm to about 1 μm , about 100 μm to about 3 μm , about 75 μm to about 3 μm , about 50 μm to about 3 μm , about 25 μm to about 3 μm , about 20 μm to about 3 μm , about 15 μm to about 3 μm , about 10 μm to about 3 μm , about 8 μm to about 3 μm , about 5 μm to about 3 μm , about 100 μm to about 5 μm , about 75 μm to about 5 μm , about 50 μm to about 5 μm , about 25 μm to about 5 μm , about 20 μm to about 5 μm , about 15 μm to about 5 μm , 10 μm to about 5 μm , about 8 μm to about 5 μm , about 100 μm to about 8 μm , about 75 μm to about 8 μm , about 50 μm to about 8 μm , about 25 μm to about 8 μm , about 20 μm to about 8 μm , about 15 μm to about 8 μm , about 10 μm to about 8 μm , about 100 μm to about 10 μm , about 75 μm to about 10 μm , about 50 μm to about 10 μm , about 25 μm to about 10 μm , about 20 μm to about 10 μm , about 15 μm to about 10 μm , about 100 μm to about 15 μm , about 75 μm to about 15 μm , about 50 μm to about 15 μm , about 25 μm to about 15 μm , about 20 μm to about 15 μm , about 100 μm to about 20 μm , about 75 μm to about 20 μm , about 50 μm to about 20 μm , about 25 μm to about 20 μm , about 100 μm to about 25 μm , 75 μm to about 25 μm , about 50 μm to about 25 μm , about 100 μm to about 50 μm , about 75 μm to about 50 μm , or about 100 μm to about 75 μm . In certain embodiments, AQ is added slowly in small portions with agitation to enable rapid dispersion of AQ in the first solution.

[0028] In one embodiment, the AQ composition is prepared according to the flow chart shown in Figure 1, comprising the following steps:

- a. adding water of a pre-determined amount into a tank equipped with an agitator;
- b. adding the dispersant (e.g., a dialkyl naphthalene sulfonate, such as sodium diisopropylnaphthalene sulfonate) of a pre-determined amount into the water, and stirring until the dispersant dissolves;
- c. adding metal ion salt(s) of a pre-determined amount into the dispersant solution prepared in step b., and stirring until all salt(s) dissolve, wherein the metal ion salt(s) may be added together or separately;
- d. adding a polyglycol (e.g., polyethylene glycol) of a pre-determined amount into the solution prepared in step c., and stirring until the polyglycol dissolves;
- e. adding AQ of a pre-determined amount into the solution prepared in step (d) slowly and in small portions with agitation to provide a substantially homogeneous AQ composition; and
- f. optionally, adding a dye of a pre-determined amount into the substantially homogeneous AQ composition of step e. to provide a substantially homogeneous AQ composition with dye.

[0029] The above steps b., c., d., and f., can be added to the water in any order, and preferably AQ is added in the final step to avoid the formation of clumps. Other compounds that can be used in a pulping process can be added to the substantially homogeneous AQ composition.

Examples

Materials:

AQ, PEG 1000, NAXAN-DIL dispersant, Zetasperse® 179 and 3600 dispersant, DOWFAX 2A1 and 8390 dispersant, and C6L Petro ULF dispersant,

Aerosol® OS dispersant, and CaCl_2 and $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ were purchased from commercially available sources.

Sample preparation and % Transparency:

[0030] After mixing, samples of suspensions of the different AQ compositions were slowly poured into scintillation vials (27.5 mm outer diameter, 72.7 mm height without cap). The headspace above the surface of the sample remained clear. The vial was capped tightly. A measurement was taken on Day 0, as well as periodically over weeks and months, after letting the samples sit at room temperature.

[0031] A % Transparency was calculated. The % Transparency is the fraction of the sample in the vial that is transparent or clear (i.e., the supernatant). Measuring the total height of the AQ composition and the height of the supernatant will yield % Transparency.

$$\% \text{ Transparency} = 100 \times (\text{supernatant height} / \text{total height of the AQ composition})$$

Example 1. Preparation of AQ compositions (50 wt%)

[0032] The present application claims priority to United States Provisional Application No. 61/474,035, filed April 11, 2011, the disclosure of which is incorporated by reference herein in its entirety. AQ compositions having a formulation listed in Table 1 were prepared according to the block flow diagram in Figure 1, and the following protocol:

1. Naxan DIL dispersant (34-36% actives in aqueous solution from Nease Co. Ohio, USA) was added into water of the desired amount. The mixture was stirred for about 10 to 15 minutes until the Naxan DIL dissolved completely;

2. Magnesium chloride hexahydrate was added to the mixture of step 1 with agitation, and stirred for about 5 to about 10 minutes until the magnesium chloride dissolved completely.

3. Anhydrous calcium chloride was added to the mixture of step 2 with agitation, and stirred for about 5 to about 10 minutes until the calcium chloride dissolved completely.

4. PEG 1000 (50% aqueous solution, ~1,000 average molecular weight) was added to the mixture of step 3 with agitation, and stirred for about 10 to about 15 minutes until the PEG 1000 dissolved completely.

5. AQ powder was added slowly in small portions with agitation to provide a well-dispersed suspension, and stirred for about 15 to about 20 minutes to ensure the AQ was well-dispersed. The dispersion obtained was visually homogeneous.

Table 1. AQ composition formulations

	% (by weight)	g of individual component per 100 g batch size
Anthraquinone (AQ)	50.00	50
Water	35.61	35.61
PEG 1000, aq. 50%	10.00	10
NAXAN DIL, aq. 35%	2.86	2.86
CaCl ₂	0.54	0.54
MgCl ₂ · 6H ₂ O	0.99	0.99

[0033] The calcium chloride and magnesium chloride used in the formulation can be anhydrous or hydrates. The amount of water used in the formulation is adjusted accordingly.

Example 2. Test of Stability of AQ composition

[0034] An AQ composition was prepared according to the method described in Example 1. Figure 2 shows the AQ composition soon after it was prepared (Day 0), and at 18 days and 5.5 months (Day 165). The AQ composition is substantially stable after 18 days, and even after 165 days, of storage in a closed container at room temperature. The % Transparency at Day 0, Day 18, and Day 165 was 0%, about 7%, and about 7%, respectively.

Example 3. Effect of dispersants

[0035] Several AQ compositions were prepared according to the method described in Example 1, except that the NAXAN DIL dispersant was replaced with other dispersants listed in Table 2. The % of each dispersant is shown in Table 2 and was kept the same in the AQ compositions.

Table 2 – Observations of AQ compositions using various dispersants

Dispersant Brand	Source	% Dispersant in the commercial product	Comments
ZetaSpense® 179 (alkoxylated alcohol, non-ionic)	Air Products	52%	Dispersion was easy, like NAXAN DIL; no foaming; final dispersion was more viscous.
ZetaSpense® 3600 (hydrophobic anionic polymer)	Air Products	52%	Addition of salts resulted in a gummy, insoluble precipitate.
DOWFAX® 2A1 (branched 12-carbon hydrophobe)	The Dow Chemical Co.	46%	Difficult to break up AQ lumps, needed to increase stirring speed; a fair amount of foaming.
DOWFAX® 8390 (linear 16-carbon hydrophobe)	The Dow Chemical Co.	32.50%	Wetted AQ particles slowly, forms clumps, needed to increase stirring speed. Foamed, small bubbles.
DOWFAX® C6L (linear 6-carbon hydrophobe)	The Dow Chemical Co.	46%	Wetted AQ particles slowly, needed to increase stirring speed; foamed a lot.
Petro ULF Liquid (anionic)	AkzoNobel	50%	AQ dispersed similar to the AQ compositions using NAXAN DIL
Aerosol® OS (anionic)	Cytec	75% (powder)	No specific observations
NAXAN DIL (anionic)	Nease Co.	35%	AQ dispersed quickly into solution without clumping or foaming

[0036] Table 2 shows that dispersants affected the homogeneity and viscosity of the resulting AQ compositions. AQ dispersed quickly into solution without clumping or foaming with the desired viscosity using the anionic dispersant, e.g., sodium diisopropylnaphthalene sulfonate (NAXAN DIL dispersant). Another anionic dispersant, Petro ULF Liquid dispersant, had similar effect as the NAXAN DIL dispersant. Aerosol OS dispersant, a powder, is also an anionic dispersant and, while no specific observations were made, it should have similar effect as the NAXAN DIL dispersant. Compared to the AQ composition with NAXAN DIL dispersant, the AQ composition with the non-ionic dispersant, ZetaSpers® 179 dispersant, was similar except it was more viscous.

Example 4. Effect of salts

[0037] Two samples (Samples A and B) were prepared according to the method described in Example 1, except that in one sample (Sample B) the calcium and magnesium salts were left out. The difference was made up with water (i.e., 37.14 g H₂O instead of 35.61 g H₂O). The majority of AQ particles that settled happened in the first 7 days after preparation. The AQ composition showed insignificant changes (less than 5% change of the % Transparency) at various months (e.g., 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, or 12 months) from the eighth day after preparation.

[0038] In Figure 3, the sample on the left is Sample B (which contained the ingredients listed in Table 1 minus the calcium chloride and magnesium chloride), and the sample on the right is Sample A which contained all the ingredients listed in Table 1. The sample with the calcium chloride and magnesium chloride (Sample A) remained stable from Day 0 and even after several months, demonstrating that magnesium and calcium salts kept the AQ composition substantially homogeneous

and stable. The sample with the salts (Sample A) had a % Transparency of 6.3% while the sample without the salts (Sample B) had a % transparency of 30.3%.

Example 5: Role of PEG 1000

[0039] AQ compositions were prepared according to Example 1, except that the PEG 1000 was left out, and the level of dispersant was increased. In Example 1, the AQ composition formulation contained 1% of the active ingredient contained in NAXAN DIL dispersant (i.e., the AQ composition formulation contains 2.86% NAXAN DIL dispersant and at a 35% aqueous solution, the final concentration of the active ingredient is 1%). The amount of active ingredient in the AQ compositions was increased to 2% (DIL-2) and 5% (DIL-5), respectively, in order to determine if the dispersants could compensate for the absence of PEG1000. The two samples (DIL-2-A and DIL-2-B) on the left in Figure 4 contained 2% of the active ingredient present in NAXAN DIL and the two samples (DIL-5-A and DIL-5-B) on the right contained 5% of the active ingredient present in NAXAN DIL. This visual was taken 30 minutes after preparation of the formulations. The AQ compositions were not stable in the absence of PEG 1000 as demonstrated by their % Transparency of greater than 26% for each of the samples DIL-2-A, DIL-2-B, DIL-5-A, and DIL-5-B. The % Transparency decreased with the addition of PEG 1000 to the test samples DIL-2 and DIL-5.

Example 6: AQ mean particle size

[0040] AQ compositions were prepared according to Example 1 using AQ of different particle sizes. The AQ used had mean particle sizes of 3 μm , 5 μm , and 135 μm , respectively. Figure 5 shows the three AQ compositions one day after

preparation. The stability of the dispersions was dependent on the particle size, and compositions containing smaller particle size of AQ were more stable. The composition with AQ of 3 μm mean particle size had a % Transparency of 6.1%, the composition with AQ of 5 μm mean particle size had a % Transparency of 9.7%, and composition with AQ of 135 μm mean particle size had a % Transparency of 25%.

Claims

1. A composition comprising:
 - anthraquinone;
 - one or more metal ions;
 - a dispersant;
 - a polyglycol; and
 - water,wherein the composition is a substantially homogeneous and stable suspension.
2. The composition according to claim 1, wherein the composition is stable after being left at room temperature in a closed container for about 24 hours to about 1 year.
3. The composition according to claim 1, wherein the mean particle size of anthraquinone is about 150 μm or less, about 100 μm to about 1 μm , about 75 μm to about 1 μm , about 50 μm to about 1 μm , about 25 μm to about 1 μm , about 20 μm to about 1 μm , about 15 μm to about 1 μm , about 10 μm to about 1 μm , about 8 μm to about 1 μm , about 5 μm to about 1 μm , or about 3 μm to about 1 μm .
4. The composition according to claim 1, wherein the one or more metal ions are divalent cations, trivalent cations, tetravalent cations, or mixtures thereof.
5. The composition according to claim 4, wherein the divalent metal ions are magnesium and calcium, or salts thereof.
6. The composition according to claim 5, wherein the molar ratio of calcium to magnesium in the composition is about 1:1.
7. The composition according to claims 4 or 5, wherein the at least one or more metal ions are water soluble.
8. The composition according to Claim 1, wherein the dispersant is an anionic dispersant, a cationic dispersant, or a neutral dispersant.
9. The composition according to Claim 8, wherein the dispersant is water soluble.
10. The composition according to claim 8, wherein the dispersant is a naphthalene sulfonate dispersant.

11. The composition according to claim 10, wherein the dispersant is a salt of diisopropylnaphthalene sulfonate.
12. The composition according to claim 1, wherein the polyglycol is polyethylene glycol, polypropylene glycol, or polybutylene glycol, or mixtures thereof.
13. The composition according to claim 12, wherein the polyglycol is water soluble.
14. The composition according to claim 12, wherein the average molecular weight of polyethylene glycol in the polyethylene glycol composition is from about 200 to about 5,000, from about 500 to about 3,000, from about 750 to about 2,000, or about 1,000.
15. The composition according to claim 1, having a viscosity of about 500 to about 10,000 cP at 25°C, about 1,000 to about 7,000 cP at 25°C, or about 2,000 to about 5,000 cP at 25°C, wherein the composition contains 50% by mass anthraquinone.
16. The composition according to claim 1, wherein the molar ratio of anthraquinone to metal ion is about 50:1.
17. The composition according to claim 1, wherein the concentration of anthraquinone by weight is at least about 20%, at least about 30%, at least about 40%, or about 50%.
18. The composition according to claim 1, wherein the concentration of the dispersant by weight is from about 0.1% to about 5%, from about 0.2% to about 3%, or from about 0.5% to about 2%.
19. The composition according to claim 1, wherein the concentration of the polyglycol by weight is from about 1% to about 10%, from about 2% to about 8%, from about 3% to about 6%, or about 5%.
20. The composition according to claim 1, further comprising a dye or other additives.
21. A composition comprising:
 - anthraquinone;
 - one or more calcium salts;
 - one or more magnesium salts;
 - a salt of diisopropylnaphthalene sulfonate;

polyethylene glycol; and

water,

wherein the composition is a substantially homogeneous and stable suspension.

22. A composition comprising:

anthraquinone having a mean particle size of about 150 μm or less;

one or more metal ions;

a dispersant;

a polyglycol; and

water.

23. The composition according to claim 22, wherein the composition is a substantially homogeneous and stable suspension.

24. The composition according to claim 22, wherein the composition is stable after being left at room temperature in a closed container for about 24 hours to 1 year.

25. The composition according to claim 22, wherein the mean particle size of anthraquinone is 100 μm to about 1 μm , about 75 μm to about 1 μm , about 50 μm to about 1 μm , about 25 μm to about 1 μm , about 20 μm to about 1 μm , about 15 μm to about 1 μm , about 10 μm to about 1 μm , about 8 μm to about 1 μm , about 5 μm to about 1 μm , about 3 μm to about 1 μm .

26. The composition according to claim 22, wherein the one or more metal ions are divalent cations, trivalent cations, or tetravalent cations, or mixtures thereof.

27. The composition according to claim 26, wherein the divalent metal ions are magnesium and calcium, or salts thereof.

28. The composition according to claim 27, wherein the molar ratio of calcium to magnesium in the composition is about 1:1.

29. The composition according to claims 26 or 27, wherein the at least one or more metal ions are water soluble.

30. The composition according to Claim 22, wherein the dispersant is an anionic dispersant, a cationic dispersant, or a neutral dispersant.

31. The composition according to Claim 30, wherein the dispersant is water soluble.
32. The composition according to claim 30, wherein the dispersant is a naphthalene sulfonate dispersant.
33. The composition according to claim 32, wherein the dispersant is a salt of diisopropylnaphthalene sulfonate.
34. The composition according to claim 22, wherein the polyglycol is polyethylene glycol, polypropylene glycol, or polybutylene glycol, or mixtures thereof.
35. The composition according to claim 34, wherein the polyglycol is water soluble.
36. The composition according to claim 34, wherein the average molecular weight of polyethylene glycol is from about 200 to about 5,000, from about 500 to about 3,000, from about 750 to about 2,000, or about 1,000.
37. The composition according to claim 22, having a viscosity of about 500 to about 10,000 cP at 25°C, about 1,000 to about 7,000 cP at 25°C, and about 2,000 to about 5,000 cP at 25°C, wherein said composition contains 50% by mass anthraquinone.
38. The composition according to claim 22, wherein the molar ratio of anthraquinone to metal ion is about 50:1.
39. The composition according to claim 22, wherein the concentration of anthraquinone by weight is at least about 20%, at least about 30%, at least about 40%, or about 50%.
40. The composition according to claim 22, wherein the concentration of the dispersant by weight is from about 0.1% to about 5%, from about 0.2% to about 3%, or from about 0.5% to about 2%.
41. The composition according to claim 22, wherein the concentration of the polyglycol by weight is from about 1% to about 10%, from about 2% to about 8%, from about 3% to about 6%, or about 5%.
42. The composition according to claim 22, further comprising a dye or other additives.
43. A composition comprising:
 - anthraquinone having a mean particle size of about 150 μm or less;

one or more calcium salts;
one or more magnesium salts;
a salt of diisopropylnaphthalene sulfonate;
polyethylene glycol; and
water.

44. The composition according to claim 43, wherein the composition is a substantially homogeneous and stable suspension.

45. The composition according to claim 43, wherein the composition is stable after being left at room temperature in a closed container for about 24 hours to 1 year.

46. The composition according to claim 43, wherein the mean particle size of anthraquinone is 100 μm to about 1 μm , about 75 μm to about 1 μm , about 50 μm to about 1 μm , about 25 μm to about 1 μm , about 20 μm to about 1 μm , about 15 μm to about 1 μm , about 10 μm to about 1 μm , about 8 μm to about 1 μm , about 5 μm to about 1 μm , about 3 μm to about 1 μm .

47. A method for preparing a composition, comprising:

- a. providing a solution comprising water, at least one or more metal ions, a dispersant, and a polyglycol ; and
- b. adding anthraquinone to the solution with agitation at a speed such that the composition forms a substantially homogeneous and stable suspension.

48. The method according to claim 47, wherein the metal ions are magnesium and calcium, or salts thereof.

49. The method of claim 47, wherein the anthraquinone has a mean particle size of about 150 μm or less, about 100 μm to about 1 μm , about 75 μm to about 1 μm , about 50 μm to about 1 μm , about 25 μm to about 1 μm , about 20 μm to about 1 μm , about 15 μm to about 1 μm , about 10 μm to about 1 μm , about 8 μm to about 1 μm , about 5 μm to about 1 μm , about 3 μm to about 1 μm .

50. The method according to claim 47, wherein the composition is stable after being left at room temperature in a closed container for at least about 24 hours, greater

than about 1 week, greater than about 1 month, greater than about 3 months, or about a year.

51. A method for preparing a composition comprising:

- a. providing a solution comprising water, a magnesium salt, a calcium salt, a salt of diisopropylnaphthalene sulfonate, and polyethylene glycol; and
- b. adding anthraquinone having a particle size of about 150 μm or less to the solution with agitation at a speed such that the composition forms a substantially homogeneous and stable suspension.

52. The composition according to claim 51, wherein the mean particle size is about 100 μm to about 1 μm , about 75 μm to about 1 μm , about 50 μm to about 1 μm , about 25 μm to about 1 μm , about 20 μm to about 1 μm , about 15 μm to about 1 μm , about 10 μm to about 1 μm , about 8 μm to about 1 μm , about 5 μm to about 1 μm , about 3 μm to about 1 μm .

53. The method of claim 51, wherein wherein the composition is stable after being left at room temperature in a closed container for at least about 24 hours, greater than about 1 week, greater than about 1 month, greater than about 3 months, or about a year

54. The use of the composition according to any one of claims 1, 21, 22, 43, 47, and 51 in a pulping process.

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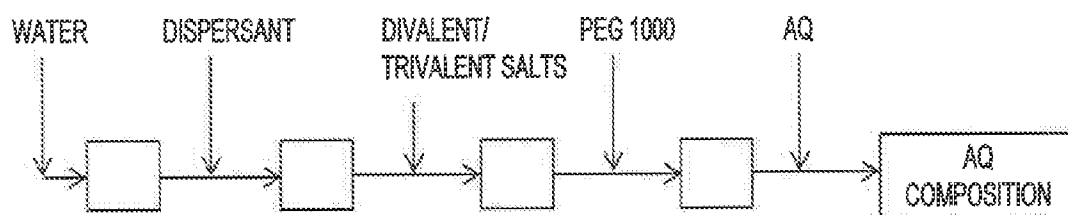


FIGURE 1: BLOCK FLOW DIAGRAM SHOWING THE PREPARATION OF AN AQ COMPOSITION ACCORDING TO AN EMBODIMENT.

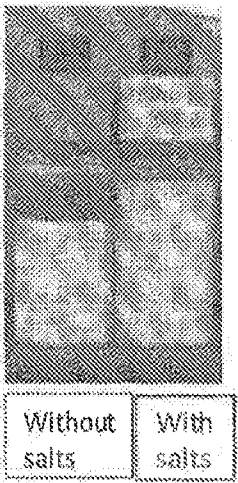
Figure 1

Figure 2



Day 0	Day 18	Day 165 (5.5 mos)
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Figure 3



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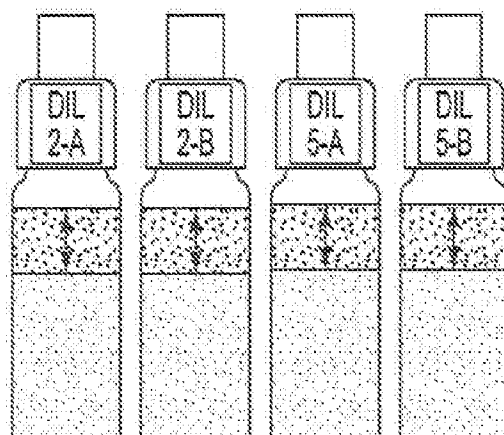
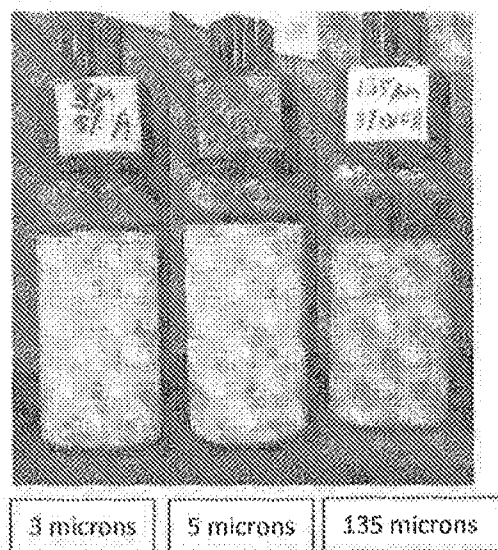
**Figure 4**

FIGURE 5



INTERNATIONAL SEARCH REPORT

International application No.

PCT/US 16/20957

A. CLASSIFICATION OF SUBJECT MATTER

IPC(8) - C07C 46/10; C07C 50/18; D21C 3/00 (2016.01)

CPC - C07C46/10; C07C50/18; D21C3/00

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) - C07C 46/10; C07C 50/18; D21C 3/00 (2016.01)

CPC - C07C46/10; C07C50/18; D21C3/00

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
Patents and NPL (classification, keyword; search terms below)

USPC - 552/208; 162/72

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

PatBase (AU BE BR CA CH CN DE DK EP ES FI FR GB IN JP KR SE TH TW US WO), PubWest, Google Web

search terms: pulping anthraquinone stable homogeneous polyglycol polyethylene glycol calcium magnesium salt dispersant
naphthalene sulfonate diisopropyl naphthalene sulfonate

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X ----- Y	US 5,997,623 A (Lin) 07 December 1999 (07.12.1999), claim 1, 5, 11; col 4, ln 7-10; col 6, ln 55-57; col 9, ln 12-20; col 10, ln 53-55; col 11, ln 4-25; col 13, ln 38-44; col 13, ln 56 to col 14, ln 10; col 14, ln 23-27	1-13, 15-35, 37-46 ----- 14, 36, 47-54
Y	US 2010/0319862 A1 (Rahman) 23 December 2010 (23.12.2010), para [0048], [0082], [0110]	14, 36
Y	US 6,117,224 A (Siegel et al.) 12 September 2000 (12.09.2000), col 5, ln 12-17; col 7, ln 27-40	47-53, 54/(47, 51)
Y	US 2014/0174680 A1 (Hawkins et al.) 26 June 2014 (26.06.2014) para [0012], [0014], [0015]	54
A,P	WO 2015/116564 A1 (CARGILL, INCORPORATED) 06 August 2015 (06.08.2015), para [0005], [0007], [0013], [0014]	1, 4-7, 16, 20-23, 26-29, 38, 43, 44, 48, 51



Further documents are listed in the continuation of Box C.



*

Special categories of cited documents:

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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

05 May 2016

Date of mailing of the international search report

23 MAY 2016

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