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(54) **METHOD OF MANUFACTURING CARBON FIBER DISPERSION AND METHOD OF MANUFACTURING CARBON PAPER**

(57) A method of manufacturing a carbon fiber dispersion, a carbon fiber dispersion manufactured by the method, a carbon paper manufacturing method, and a carbon paper manufactured by the carbon paper manufacturing method are disclosed. The method includes a first step of preparing a solution containing hydrophilic carbon fibers by adding carbon fibers and a wetting agent

to a solvent, followed by stirring, and a second step of dispersing the hydrophilic carbon fibers by adding a copolymer solution containing a copolymer to the solution containing the hydrophilic carbon fibers. The copolymer includes acrylic acid-based monomer units and acrylamide-based monomer units.

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Description

CROSS REFERENCE TO RELATED APPLICATION

5 **[0001]** The present application claims priority to Korean Patent Application No. 10-2022-0081800, filed July 4, 2022, the entire contents of which is incorporated herein for all purposes by this reference.

BACKGROUND OF THE INVENTION

10 1. Field of the Invention

[0002] The present disclosure relates to a method of manufacturing a carbon fiber dispersion, a method manufacturing a carbon paper, a carbon fiber dispersion manufactured by the carbon fiber dispersion manufacturing method, and a carbon paper manufactured by the carbon paper manufacturing method. The present disclosure has an advantage of
15 improving the dispersion stability of carbon fibers and the performance (for example, tensile strength, etc.) of a carbon paper.

2. Description of the Related Art

20 **[0003]** Fiber reinforced composites are reinforcing members manufactured using fibers such as glass fibers, carbon fibers, aramid fibers, and silicon carbide fibers. Among these, carbon fiber reinforced plastics (CFRP) manufactured using carbon fibers are widely used in aircraft and rotor blades of wind turbines because they use carbon fibers that are lightweight and have excellent physical properties such as high specific strength that is about six times the strength of steel and high specific modulus that is about three times the modulus of steel.

25 **[0004]** In addition, with the recent rise in environmental concerns, there is a growing effort to recover and recycle carbon fibers from waste carbon fiber reinforced composites that are discarded after use, and the related market is growing.

[0005] To manufacture a carbon fiber reinforced composite by using fresh carbon fibers and recycled carbon fibers, a method of making a composite material by mixing fibers in the form of a fabric, a non-woven fabric, or short fibers with
30 a resin can be used. To make a high-strength product, it is necessary to make a composite material using a non-woven fabric.

[0006] When a non-woven fabric is manufactured from fibers, the physical properties of the fabric vary depending on the thickness, length, arrangement, shaping method, etc. of the fibers. To manufacture a non-woven fabric, a method of regularly arranging fibers and a method of randomly dispersing fibers can be used, and the advantages each have
35 advantages and disadvantages. The random arrangement method is advantageous over the regular arrangement method in terms of facility cost, mobility, energy savings, and environmental friendliness. However, the random arrangement method has the disadvantage of producing non-woven fabrics with poor mechanical physical properties due to phenomena such as agglomeration and kinking of fibers. The poor mechanical physical properties are attributable to poor dispersibility of long fibers because the long fibers agglomerate to be bundled due to attraction force between the fibers.
40 For this reason, to increase the physical properties of non-woven fabrics, it is necessary to conduct research on a method of uniformly dispersing long fibers.

[0007] However, cellulosic polymers such as CMC, which have been used as conventional dispersants to prepare carbon fiber dispersions, have a problem of poorly dispersing carbon fibers with a predetermined length or longer.

45 **[0008]** Accordingly, there is a need for the development of a method of uniformly dispersing carbon fibers with a predetermined length or longer without degrading performance.

Document of Related Art

[0009] Korean Patent Application Publication No. 10-2022-0055337
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SUMMARY OF THE INVENTION

[0010] It is an objective of the present disclosure to provide a method of manufacturing a carbon fiber dispersion in which carbon fibers are uniformly dispersed in water, a method of manufacturing a carbon paper, a carbon fiber dispersion
55 with good dispersion stability manufactured by the carbon fiber dispersion manufacturing method, and a carbon paper with good physical properties manufactured by the carbon paper manufacturing method.

[0011] In particular, the purpose of the present disclosure is to strengthen the mechanical properties of a carbon paper by dispersing long carbon fibers having average lengths of 12 mm to 25 mm in water.

[0012] The objectives to be achieved by the present disclosure are not limited to the ones mentioned above, and other objectives not mentioned above can be clearly understood by those skilled in the art from the following description.

[0013] According to one aspect of the present disclosure, there is provided a method of manufacturing a carbon fiber dispersion, the method including: preparing a solution containing hydrophilic carbon fibers by adding carbon fibers and a wetting agent to a solvent, followed by stirring; and dispersing the hydrophilic carbon fibers by adding a copolymer solution to the solution containing the hydrophilic carbon fibers. The copolymer includes acrylic acid-based monomer units and acrylamide-based monomer units.

[0014] According to another aspect of the present disclosure, there is provided a carbon fiber dispersion manufactured by the method of the present disclosure, in which the carbon fiber dispersion has a pH of 2 or more and 12 or less and a viscosity of 10 cp or more and 200 cp or less.

[0015] According to a further aspect of the present disclosure, there is provided a method of manufacturing a carbon paper, the method including: shaping the carbon fiber dispersion prepared by the method of the present disclosure and drying the shaped carbon fiber dispersion.

[0016] The carbon fiber dispersion manufacturing method and the carbon paper manufacturing method of the present disclosure have the advantage of effectively dispersing carbon fibers (particularly, long carbon fibers) in water, thereby producing a carbon fiber dispersion with good dispersion stability and a carbon paper having good properties.

[0017] Particularly, since it is possible to effectively disperse relatively long carbon fibers in water, the mechanical properties of a carbon paper manufactured can be improved.

BRIEF DESCRIPTION OF THE DRAWINGS

[0018]

FIG. 1 is a photograph illustrating a carbon fiber dispersion in which rope-type agglomerates are formed; and

FIG. 2 is a photograph illustrating the dispersibility of carbon fibers in a sheet of carbon paper, which is measured with a 2D lab formation sensor device.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

[0019] Hereinafter, the actions and effects of the present disclosure will be described in more detail with reference to specific examples of the present disclosure. However, the examples are presented only on the illustrative purposes, and the scope of the present disclosure is not limited thereby.

[0020] Prior to giving the following detailed description of the present disclosure, it should be noted that the terms and words used in the specification and the claims should not be construed as being limited to ordinary meanings or dictionary definitions but should be construed in a sense and concept consistent with the technical idea of the present disclosure, on the basis that the inventor can properly define the concept of a term to describe its invention in the best way possible.

[0021] The features of exemplary embodiments described herein are presented for illustrative purposes and do not exhaustively represent the technical spirit of the present disclosure. Accordingly, it should be appreciated that there will be various equivalents and modifications that can replace the exemplary embodiments at the time at which the present application is filed.

[0022] As used herein, the singular forms "a", "an", and "the" are intended to include the plural forms as well unless the context clearly indicates otherwise. It will be further understood that the terms "comprising", "including", or "having" when used in the present disclosure specify the presence of stated features, regions, integers, steps, operations, elements and/or components, but do not preclude the presence or addition of one or more other features, regions, integers, steps, operations, elements, components and/or combinations thereof.

[0023] As used herein to refer to numerical ranges, "a to b" and "a-b" are defined as "being equal to or greater than a ($\geq a$) and being equal to or less than b ($\leq b$)".

[0024] According to one aspect of the present disclosure, there is provided a method of manufacturing a carbon fiber dispersion, the method including: a first step of preparing a solution containing hydrophilic carbon fibers by adding carbon fibers and a wetting agent to a solvent, followed by stirring; and a second step of dispersing the hydrophilic carbon fibers by adding a copolymer solution to the solution containing the hydrophilic carbon fibers.

[0025] The copolymer may include acrylic acid-based monomer units and acrylamide-based monomer units.

[0026] The solvent is water.

[0027] The copolymer is a random copolymer or a block copolymer depending on a synthesis process.

[0028] The first step in the carbon fiber dispersion manufacturing method refers to a step of providing a hydrophilic group capable of forming a hydrogen bond with water to the surface of hydrophobic carbon fibers, thereby enabling dispersion in water.

[0029] In addition, the second step refers to a step of inducing dispersion of the carbon fibers by electrostatic repulsion

and preventing re-agglomeration of the carbon fibers by steric hindrance. In this step, the copolymer solution obtained by dissolving a copolymer in a solvent is mixed with the carbon fibers that are adequately hydrophilized in the first step, and a predetermined amount of the solvent is added so that the hydrophilized carbon fibers can be properly dispersed.

[0030] For example, in the second step, the solution containing the hydrophilic carbon fibers prepared in the first step (i.e., solution containing carbon fibers, a solvent, a wetting agent, a binder, etc.) may be mixed with the copolymer solution (containing a copolymer (dispersant) and a solvent (for example, water) for diluting the copolymer). In this step, the weight of the copolymer solution used is about 1 to 50 times the total weight of the carbon fibers.

[0031] On the other hand, the carbon fiber may be selected from among PAN-based fibers, pitch-based fibers, cellulose fibers, recovered carbon fibers obtained by recycling prepreg by chemically or physically treating the prepreg, and various combinations thereof.

[0032] In one embodiment, in the first step, a binder (a binder fiber or a binder polymer) may be additionally added.

[0033] The binder fiber or the binder polymer is used to increase the bonding strength of the carbon paper after the drying step when manufacturing carbon paper. Examples of the binder fiber or binder polymer include polyvinyl alcohol (PVA) fibers, low-melting-point polyesters (LMPETs), and polyvinyl alcohol resins.

[0034] In one embodiment, in the first step, a shear force is applied to stir the solution at a speed of 500 to 2,000 rpm. The application of the shear force is to minimize the damage to the fibers contained in the carbon fiber dispersion. Equipment capable of applying a high shear force, such as a pulper, an overhead stirrer, and a knife beater is used to apply the shear force.

[0035] In one embodiment, in the second step, the hydrophilic carbon fibers can be stirred at a speed of 1,000 rpm or less so as to be easily dispersed.

[0036] The stirring speed may be, for example, 1,000 rpm or less, 800 rpm or less, 500 rpm or less, or 300 rpm or less.

[0037] In this case, the carbon fibers can be dispersed and stabilized with the use of an impeller designed to minimize vortex generation.

[0038] In one embodiment, the average length of the carbon fibers is 5 mm or more. For example, the average length of the carbon fibers is 6 mm or more, 10 mm or more, 12 mm or more, 15 mm or more, or 20 mm or more.

[0039] In one embodiment, the wetting agent is a cationic surfactant, an anionic surfactant, a nonionic surfactant, or any combination thereof.

[0040] For example, the wetting agent is an ethylene oxide (EP)/propylene oxide (PO) copolymer surfactant, a polyethylene oxide (PEO)-based surfactant, a polyethylene glycol (PEG)-based surfactant, or any combination thereof.

[0041] In one embodiment, the copolymer is included in an amount of 0.1 % by weight or more and 10% by weight or less, and the carbon fibers are included in an amount of 0.01 % by weight or more and 10% by weight or less, based on 100% by weight of the total content of the carbon fiber dispersion.

[0042] When the content of the copolymer exceeds the range, the viscosity of the dispersion is excessively high. Therefore, it is difficult to dehydrate and dry carbon paper in the process of manufacturing the carbon paper. When the content of the copolymer is lowered, the dispersibility of the fibers decreases.

[0043] The suitable content of the carbon fibers is determined according to the desired basis weight of paper when forming a web. When the content of the carbon fibers is above or below the range, the damage caused by friction increases, or the wetting is not properly performed.

[0044] In one embodiment, the binder (binder fiber) is included in an amount of 0.001% by weight or more and 5% by weight or less, based on 100% by weight of the total content of the carbon fiber dispersion.

[0045] When the content of the binder fiber falls below the range, a suitable strength cannot be obtained.

[0046] The wetting agent is included in an amount of 0.01% by weight or more and 5% by weight or less, based on 100% by weight of the total content of the carbon fiber dispersion.

[0047] In one embodiment, based on 100% by mole of the total content of the copolymer, the ratio of the mol% of the acrylic acid-based monomer units to the mol% of the acrylamide-based monomer units is in the range of from 10:90 to 90:10.

[0048] For example, the ratio of the mol% of the acrylic acid-based monomer units to the mol% of the acrylamide-based monomer units may be 90:10, 80:20, 70:30, 60:40, 50:50, 40:60, 30:70, 20:80, or 10:90. The mol% ratio is in the range of from 30:70 to 70:30.

[0049] In the case of using a homopolymer produced through polymerization of only acrylic acid-based monomer units or only acrylamide-based monomer units as a dispersant, the carbon fibers are poorly dispersed.

[0050] In one embodiment, the monomer of the acrylic acid-based monomer units of the copolymer is acrylic acid, methacrylic acid, itaconic acid, 2-acrylamido-2-methylpropane sulfonic acid, maleic acid, or any combination thereof.

[0051] In addition, the monomer of the acrylamide-based monomer units of the copolymer is acrylamide, methacrylamide, or any combination thereof.

[0052] The copolymer has a pH of 5 or higher and 10 or lower and a viscosity of 100 cp or higher and 20,000 cp or lower.

[0053] For example, the pH of the copolymer is in the range of 5 or higher and 9 or lower. When the pH of the carbon fiber dispersion is above or below the range, the dispersant may degrade over time or the dispersion performance of

the carbon fibers may decrease.

[0054] An alkali metal, an alkaline earth metal, an amine compound, or any combination thereof is used to adjust the pH of the copolymer.

[0055] In particular, the amine compound is an amine compound having a pH of 9 or higher.

[0056] For example, sodium hydroxide, potassium hydroxide, ammonium hydroxide, lithium hydroxide, dimethylamine, trimethylamine, monoethanolamine, diethanolamine, triethanolamine, arginine, tromethamine, diisopropanolamine, triisopropanolamine, etc. may be used to control the pH of the copolymer.

[0057] In addition, when the viscosity of the copolymer exceeds the range, since the viscosity is excessively high, it is difficult to use the copolymer. When the viscosity is below the range, the carbon fibers may not be properly dispersed.

[0058] The copolymer may have a weight average molecular weight of 10 or higher and 500 or lower.

[0059] When the weight molecular weight of the copolymer exceeds the range, since the viscosity is high, it is difficult to use the copolymer. When the molecular weight is below the range, the carbon fibers cannot be properly dispersed.

[0060] In addition, copolymers with relatively low molecular weights are suitable for the dispersion of short fibers, and copolymers with relatively high molecular weights are suitable for the dispersion of long fibers.

[0061] A carbon fiber dispersion according to another aspect of the present disclosure is prepared by the carbon fiber dispersion manufacturing method described above. The carbon fiber dispersion has a pH of 2 or higher and 12 or lower and a viscosity of 10 cp or higher and 200 cp or lower.

[0062] A method of manufacturing a carbon paper according to a further aspect of the present disclosure includes a third step of shaping the carbon fiber dispersion prepared by the carbon fiber dispersion manufacturing method described above and a fourth step of drying the shaped carbon fiber dispersion.

[0063] In the third step, the carbon fiber dispersion prepared in the second step is the process of shaping the dispersion into the form of paper using a wet process. For example, a wet-laid non-woven fabric maker, a sheet machine, or the like is used to shape the carbon fiber dispersion into the form of paper.

[0064] In the fourth step, drying is performed to produce a carbon paper as the end product.

[0065] A carbon paper according to a yet further aspect of the disclosure is manufactured by the carbon fiber manufacturing method described above, and the carbon paper has a tensile strength of 25 kgf/cm² or higher.

[0066] For example, the tensile strength of the carbon paper is 25 kgf/cm² or more, 30 kgf/cm² or more, or 40 kgf/cm² or more.

[0067] The carbon paper has a basis weight of 16 g/m² and a thickness of 120 to 150 μm, and the uniformity (dispersibility) of the carbon fibers in the carbon paper measured by a 2D lab formation sensor device is 320 or less.

[0068] For example, the uniformity (dispersibility) of the carbon paper is 320 or less, 310 or less, or 290 or less.

[0069] Hereinafter, the present disclosure will be described in more detail with reference to examples, but the scope of the present disclosure is not limited thereby.

Preparation Example 1. Preparation of polymer

[0070] Acrylic acid monomer (AA), acrylamide monomer (AM), and water were added to a three-neck flask reactor, a thermometer was fitted to the reactor, nitrogen purging was performed, and the reactor was heated until the temperature of the reactor reached 70°C.

[0071] The weight ratio of the total weight of the acrylic acid monomer (AA) and acrylamide monomer (AM) to the weight of the water was 2:8.

[0072] Next, ammonium persulfate (APS) dissolved in water was then added dropwise to the reactor for 30 minutes. The amount of the ammonium persulfate (APS) used was 0.1 parts by weight per 100 parts by weight of the total weight of the acrylic acid monomer (AA), the acrylamide monomer (AM), and the water.

[0073] After the dropwise addition was completed, the solution was maintained at 70°C for 7 hours, followed by cooling. Next, the pH of the solution was adjusted to 7.0 using NaOH, so that a polymer having a solid content of 25% and a weight average molecular weight of 100,000 g/mol was obtained.

[0074] In the total content (100 mol%) of the polymer, the ratio of the mol% of the acrylic acid monomer (AA) to the mol% of the acrylamide monomer (AM) was adjusted to 70:30 (D1), 30:70 (D2), 100:0 (D3), and 0:100 (D4) to prepare four polymers.

Preparation Example 2. Preparation of carbon fiber dispersion

[0075] Carbon fiber (manufactured by Zolteck), polyvinyl alcohol (PVA) fiber (VPB-101, Kuraray Co., Ltd.), and a nonionic surfactant-based wetting agent (HPW-500, Hansol) in respective amounts shown in Table 1 were added to a balanced amount of a solvent, followed by mixing. After the mixing, pre-wetting was performed at 1,000 rpm using a pulper. The pre-wetting is the process of preparing a solution containing hydrophilic carbon fibers.

[0076] Subsequently, the solution containing hydrophilic carbon fibers resulting from the pre-wetting was added to

each of the polymer solutions prepared by dissolving the respective polymers (dispersants) prepared according to Preparation Example 1 in 2000 g of a solvent (water), followed by stirring at a speed of 300 rpm or lower using an impeller that minimally causes vortex generation.

[0077] The average lengths of the carbon fibers used were 6, 12, and 20 mm, as shown in Table 1 below.

[0078] Carbon fiber dispersions each were prepared according to Preparation Example 2, using each of the polymers D1, D2, D3, and D4 prepared according to Preparation Example 1 as a dispersant. In Examples 1 to 3 and Comparative Example 6, the polymer D1 was used as the dispersant. In Example 4, the polymer D2 was used. In Comparative Example 1, the polymer D3 was used. In Comparative Example 2, the polymer D4 was used.

[0079] On the other hand, in Comparative Examples 3 to 5, respective carbon fiber dispersions were prepared according to Preparation Example 2 by using carboxymethyl cellulose ((CMC), manufactured by Duksan Science, Mw: 21,000 to 500,000) as the dispersant.

[Table 1]

	Carbon fiber		PVA fiber	Wetting agent (% by weight)	Dispersant (% by weight)
	Average length (mm)	Content (% by weight)	Content (% by weight)		
Example 1	6	0.05	0.005	0.05	D1 (0.2% by weight)
Example 2	12			0.05	D1 (0.2% by weight)
Example 3	20			0.05	D1 (0.2% by weight)
Example 4	12			0.05	D2 (0.2% by weight)
Comparative Example 1	12			0.05	D3 (0.2% by weight)
Comparative Example 2	12			0.05	D4 (0.2% by weight)
Comparative Example 3	6			0	CMC (0.2% by weight)
Comparative Example 4	12			0	CMC (0.2% by weight)
Comparative Example 5	12			0.05	CMC (0.2% by weight)
Comparative Example 6	12			0	D1 (0.2% by weight)

[0080] For each of the carbon fiber dispersions of Examples 1 to 4 and Comparative Examples 1 to 6, the time taken until rope-type agglomerated carbon fibers illustrated in FIG. 1 disappeared was measured to determine the dispersion speed.

[0081] In addition, after the carbon fibers in the carbon fiber dispersions of Examples 1 to 4 and Comparison Examples 1 to 6 were dispersed, the dispersions were left for 2 hours, and the dispersion stability of each of the dispersions was determined by visually checking whether re-agglomeration or settling of the carbon fibers occurred.

[0082] The measured dispersion speed and dispersion stability of each of the carbon fiber dispersions are shown in Table 2 below.

[Table 2]

Carbon fiber dispersion	Dispersion speed	Dispersion stability
Example 1	3 minutes or less	2 hours or more
Example 2	3 minutes or less	2 hours or more

(continued)

Carbon fiber dispersion	Dispersion speed	Dispersion stability
Example 3	5 minutes or less	2 hours or more
Example 4	5 minutes or less	2 hours or more
Comparative Example 1	2 hours or more	Poor dispersion
Comparative Example 2	2 hours or longer	Poor dispersion
Comparative Example 3	30 minutes or less	30 minutes
Comparative Example 4	2 hours or more	Poor dispersion
Comparative Example 5	2 hours or more	Poor dispersion
Comparative Example 6	2 hours or more	Poor dispersion

[0083] It was confirmed that the carbon fiber dispersions of Examples 1 to 4 in which a copolymer of acrylic acid monomer (AA) and acrylamide monomer (AM) was used as the dispersant exhibited faster dispersion speed (i.e., shorter dispersion time) and higher dispersion stability than the carbon fiber dispersions of Comparative Examples 1 and 2 in which a homopolymer of only an acrylic acid monomer (AA) or only an acrylamide monomer was used as the dispersant.

[0084] In addition, it was confirmed that the carbon fiber dispersions of Examples 1 to 4 exhibited faster dispersion speed (i.e., shorter dispersion time) and higher dispersion stability than the carbon fiber dispersions of Comparative Examples 3 to 5 in which CMC was used as the dispersant.

[0085] In particular, the carbon fiber dispersions of Examples 2 to 4 containing carbon fibers with relatively long average lengths of 12 mm and 20 mm exhibited high dispersion stability.

[0086] In the case of the carbon fiber dispersions of Comparative Examples 1, 2, and 5 that were the same as the carbon fiber dispersion of Example 2 except that a homopolymer or CMC was used as the dispersant, long carbon fibers with an average length of 12 mm were not easily dispersed, and the dispersion stability was poor.

[0087] In the case of the carbon fiber dispersion of Comparative Example 6 using the same polymer as in Examples 1 to 3 but not containing the wetting agent (i.e., not having undergone the pre-wetting), the dispersion of carbon fibers was not easy, and the dispersion stability was extremely poor.

[0088] That is, it was found that the pre-wetting greatly affected the dispersion of carbon fibers and the dispersion stability of the carbon fiber dispersion.

Preparation Example 3. Manufacturing of carbon paper

[0089] A 25×25-cm² sheet machine was used to produce paper from each of the carbon fiber dispersions of Examples 1 to 4 and Comparative Examples 1 to 6, followed by drying in a 120°C hot air drying oven for 30 minutes. Thus, carbon papers were produced.

[0090] The dispersibility and tensile strength of the carbon papers that were prepared from the respective carbon fiber dispersions of Examples 1 and 4 and Comparative Examples 1 to 6 according to Preparation Example 3 were measured.

[0091] Dispersibility (uniformity) of carbon fibers in each of the carbon papers was measured using a 2D lab formation sensor device (manufactured by Techpap SAS, France). The carbon papers had a basis weight of 16 g/m² and a thickness of 120 to 150 μm.

[0092] The dispersibility in the carbon papers can be evaluated on the basis of the number and distribution of flocs measured with the 2D lab formation sensor device. The lower the measurement value, the better the dispersibility.

[0093] The tensile strength of the carbon papers was measured at a speed of 2 m/min using AGS-X 10N-10KN (manufactured by Shimadzu Corporation).

[0094] As illustrated in FIG. 2, in the case of the carbon papers made from the carbon fiber dispersions of Examples 1 to 4, due to the excellent dispersibility of the carbon fiber dispersions of Examples 1 to 4, the measurement values obtained using the 2D lab formation sensor device were in the range of from 200 to 300, and it was confirmed that the carbon fibers were uniformly dispersed in the carbon papers.

[0095] On the other hand, in the case of the carbon papers respectively made from the dispersions of Comparative Examples 1 to 6, the measurement values obtained using the 2D lab formation sensor device exceeded 320, and it was confirmed that the carbon fibers were not uniformly dispersed in the carbon papers.

[0096] The measurement values of the dispersibility and tensile strength of the carbon papers are shown in Table 3 below.

[Table 3]

Dispersion used	Dispersibility	Tensile strength
Example 1	224.94	67.38 kgf/cm ²
Example 2	235.72	101.19 kgf/cm ²
Example 3	281.24	134.52 kgf/cm ²
Example 4	254.16	95.28 kgf/cm ²
Comparative Example 1	415.21	21.18 kgf/cm ²
Comparative Example 2	486.86	19.85 kgf/cm ²
Comparative Example 3	325.33	28.99 kgf/cm ²
Comparative Example 4	519.26	15.01 kgf/cm ²
Comparative Example 5	420.67	21.23 kgf/cm ²
Comparative Example 6	390.25	23.25 kgf/cm ²

[0097] In the case of the carbon papers made from the carbon fiber dispersions of Examples 1 to 4, the measurement value obtained using the 2D lab formation sensor device slightly increased with increasing length of the carbon fiber, but the measurement value was 300 or less, showing excellent dispersibility.

[0098] On the other hand, in the case of the carbon papers made from the carbon fiber dispersions of Comparative Examples 1 to 6, the measurement value obtained using the 2D lab formation sensor device greatly increased with increasing length of the carbon fiber. Therefore, it was confirmed that the dispersibility of the carbon paper greatly decreased.

[0099] In addition, the carbon papers made from the carbon fiber dispersions of Examples 1 to 4 exhibited higher tensile strength than the carbon papers made from the carbon fiber dispersions of Comparative Examples 1 to 6 due to the excellent dispersibility thereof.

[0100] Particularly, in the case of the carbon papers made from the carbon fiber dispersions of Examples 1 to 3, it was found that the tensile strength increased with increasing length of the carbon fiber.

[0101] That is, it was confirmed that the copolymers of the acrylic acid monomer (AA) and the acrylamide monomer (AM) could be used as effective dispersants for dispersing carbon fibers having a relatively long average length of 12 mm or 20 mm.

[0102] Since the carbon fiber dispersions disclosed herein have excellent dispersion properties compared to conventional carbon fiber dispersions, it was confirmed that the properties of a carbon paper manufactured from the carbon fiber dispersion of the present disclosure were improved.

[0103] The scope of the present disclosure is defined by the following claims rather than the above detailed description, and all changes or modifications derived from the meaning and scope of the claims and their equivalent concepts should be interpreted to fall within the scope of the present disclosure.

Claims

1. A method of manufacturing a carbon fiber dispersion, the method comprising:

preparing a solution containing hydrophilic carbon fibers by adding carbon fibers and a wetting agent to a solvent, followed by stirring; and

dispersing the hydrophilic carbon fibers by adding a copolymer solution containing a copolymer to the solution containing hydrophilic carbon fibers,

wherein the copolymer comprises an acrylic acid-based monomer unit and an acrylamide-based monomer unit.

2. The method of claim 1, wherein a binder is added in the preparing of the solution containing hydrophilic carbon fibers.

3. The method of claim 1, wherein in the preparing of the solution containing hydrophilic carbon fibers, a shear force is applied for the stirring at a speed of 500 to 2,000 rpm.

4. The method of claim 1, wherein in the dispersing of the hydrophilic carbon fibers, stirring was performed at a speed of 1000 rpm or less.
5. The method of claim 1, wherein the carbon fiber has an average length of 5 mm or more.
6. The method of claim 1, wherein the wetting agent is a cationic surfactant, an anionic surfactant, a nonionic surfactant, or any combination thereof.
7. The method of claim 1, wherein the copolymer is contained in an amount of 0.1% by weight or more and 10% by weight or less, and the carbon fiber is contained in an amount of 0.01% by weight or more and 10% by weight or less, based on 100% by weight of the total content of the carbon fiber dispersion.
8. The method of claim 2, wherein the binder is contained in an amount of 0.001 % by weight or more and 5% by weight or less, based on 100% by weight of the total content of the carbon fiber dispersion.
9. The method of claim 1, wherein a ratio of the mol% of the acrylic acid-based monomer unit to the mol% of the acrylamide-based monomer unit is in a range of from 10:90 to 90:10.
10. The method of claim 1, wherein a monomer of the acrylic acid-based monomer unit of the copolymer is acrylic acid, methacrylic acid, itaconic acid, 2-acrylamido-2-methylpropane sulfonic acid, maleic acid, or any combination thereof, and a monomer of the acrylamide-based monomer unit of the copolymer is acrylamide, methacrylamide, or a combination thereof.
11. The method of claim 1, wherein the copolymer has a pH of 5 or higher and 10 or lower and a viscosity of 100 cp or higher and 20,000 cp or lower.
12. A carbon fiber dispersion manufactured by any one of claims 1 to 11, the carbon fiber dispersion having a pH of 2 or higher and 12 or lower and a viscosity of 10 cp or higher and 200 cp or lower.
13. A method of manufacturing a carbon paper, the method comprising:
shaping the carbon fiber dispersion manufactured by any one of claims 1 to 11; and
drying the shaped carbon fiber dispersion.

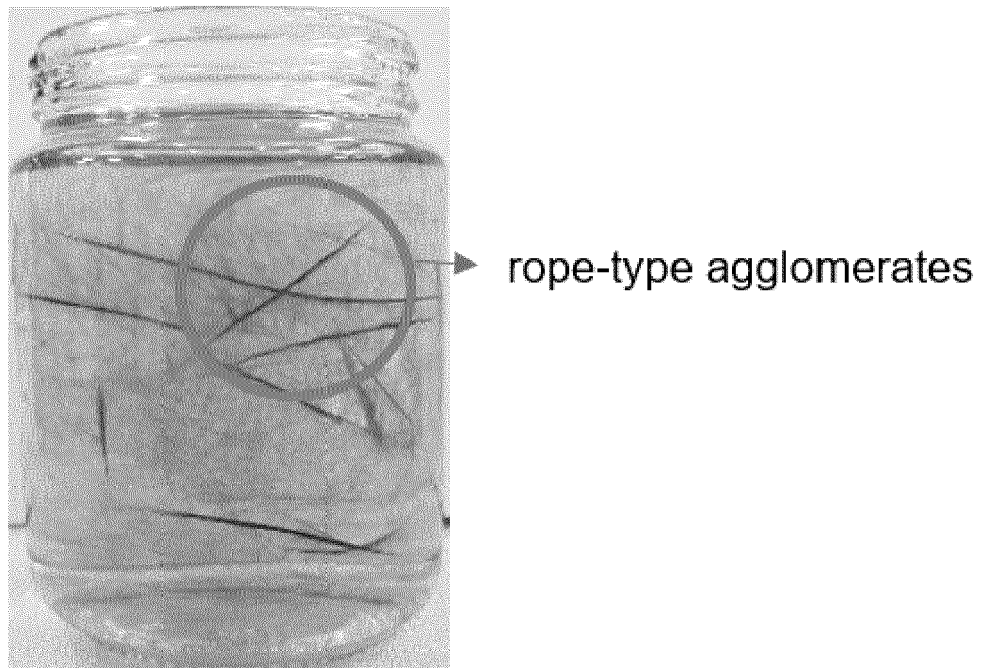


FIG. 1

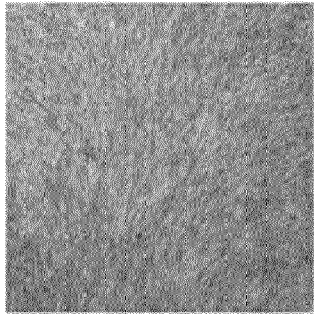
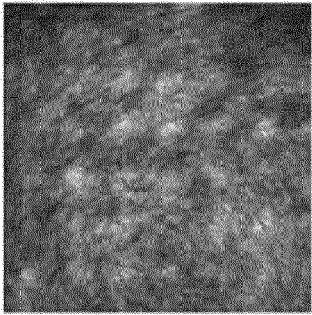
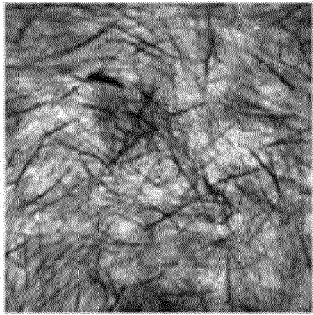
measurement value	200~300	300~400	400 or more
dispersibility (uniformity)			

FIG. 2



EUROPEAN SEARCH REPORT

Application Number

EP 23 18 3053

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
X	KR 2018 0130840 A (KOREA INST IND TECH [KR]) 10 December 2018 (2018-12-10) * paragraphs [0071], [0077]; examples 3-6 *	1-13	INV. D21H13/50 D21H17/37
X	JP 2018 159164 A (MITSUBISHI CHEM CORP) 11 October 2018 (2018-10-11) * paragraphs [0057] - [0060] *	1-13	

TECHNICAL FIELDS SEARCHED (IPC)

D21H

The present search report has been drawn up for all claims

1

Place of search

Munich

Date of completion of the search

23 October 2023

Examiner

Ponsaud, Philippe

CATEGORY OF CITED DOCUMENTS

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5 This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report.
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23-10-2023

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
KR 20180130840 A	10-12-2018	NONE	
JP 2018159164 A	11-10-2018	JP 6825442 B2	03-02-2021
		JP 2018159164 A	11-10-2018

REFERENCES CITED IN THE DESCRIPTION

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Patent documents cited in the description

- KR 1020220081800 [0001]
- KR 1020220055337 [0009]