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RESEARCH DISCLOSURE, no. 203, March 1981, pages 132,133, disclosure no. 20336, Industrial Opportunities Ltd, Hampshire, GB; S. A. COLBY et al.: "Method for making paper with polymer-encapsulated sizing agent"

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ABSTRACT BULLETIN OF THE INSTITUTE OF PAPER CHEMISTRY, vol. 53, no. 11, May 1983, page 1323, abstract no. 12331, Appleton, Wisconsin, US

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Courier Press, Leamington Spa, England.

EP 0 220 941 B1

Description

This invention relates to stable solid compositions comprising ketene dimers and to processes for their production.

Ketene dimers find use as ingredients of paper sizing compositions. Sizing compositions are manufactured in the form of relatively concentrated aqueous dispersions, which are diluted before use in the paper sizing process. These concentrated aqueous dispersions suffer from the disadvantage that their efficiency as paper sizing agents deteriorates on prolonged storage, apparently because of the hydrolysis of the ketene dimer. In order to be useful as paper sizes, ketene dimers are dispersed in water in the presence of a surfactant and/or an emulsifier, present in an amount from 0.2% to 2.5% by weight of the weight of ketene dimer. Attempts to dry such dispersions in the past have not been successful; if such a dispersion is dried slowly, the ketene dimer tends to hydrolyse; if the dispersion is dried at high temperatures, the ketene dimer tends to char. If such a dispersion is spray dried, the material dries to a sticky mass which is difficult to handle.

US—A—3311532 (Kulick et al) describes in Example 9 a ketene dimer composition in which hexadecyl ketene dimer, stearic acid, sodium lignosulphonate and a water-soluble cationic starch are ball-milled to form a powder which subsequently emulsifies when it is added slowly with intense agitation to water 90°C. The starch is stated to be present as a supplementary inert extender.

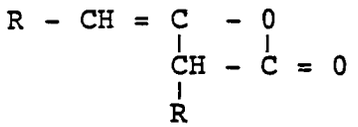
S. A. Colby et al describe in Research Disclosure 20336 (March 1981) a polymer-encapsulated paper sizing agent which comprises ketene dimer encapsulated within anionic polymer shells.

GB—A—1405751 (BPB Industries) discloses a composition for use in paper manufacture, the composition being formed by spray-drying an aqueous slurry of particulate filler and from 1 to 20% by weight of organic polymeric material.

US—A—2865734 (Weisgerber) discloses a ketene dimer paper sizing composition in which the ketene dimer is dispersed on a carrier of finely-divided amorphous silica to form a free-flowing powder for incorporation into an aqueous pulp slurry.

It has been discovered that solid particulate compositions can be manufactured, comprising ketene dimers and a stabilising agent, which are readily dispersible in water, as is required in paper sizing processes, and which exhibit improved stability upon prolonged storage. Solid water-dispersible compositions commprising solid particles of a ketene dimer and a stabilising agent are believed to be novel and constitute one aspect of this information.

The ketene dimers which are useful as paper sizing agents and which are used in carrying out this invention are compounds having the molecular formula:



wherein R is as defined below; the compositions of the invention comprise:

(a) a ketene dimer of this formula, and

(b) a water-soluble stabilising agent, the stabilising agent and the ketene dimer being present in the form of a water-dispersible homogeneous particulate solid.

In the above formula, R represents an alkyl, alkenyl, aryl or alkaryl group having from 8 to 24 carbon atoms. Preferably, R represents an alkyl group having from 8 to 20 carbon atoms. These ketene dimer products are normally prepared from fatty acids obtained from natural sources and they therefore comprise a mixture of dimeric compounds, wherein the number of carbon atoms in the substituent group R varies. Mixtures of ketene dimers wherein R has an average value of from 8 to 24 are useful in carrying out this invention. Preferably, the group R contains an average of from 12 to 18 carbon atoms. Examples of ketene dimers include octyl, decyl, dodecyl, tetradecyl, hexadecyl, octadecyl eicosyl, docosyl, tetracosyl, phenyl, benzyl, beta-naphthyl and cyclohexyl ketene dimers, as well as the ketene dimers prepared by known methods from montanic acid, naphthenic acid, 9,10-decylenic acid, 9-10-dodecylenic acid, palmitoleic acid, oleic acid, ricinoleic acid and eleostearic acid, as well as ketene dimers prepared from naturally-occurring mixtures of fatty acids, such as those found in coconut oil, babassu oil, palm kernel oil, palm oil, olive oil, peanut oil, rape oil, beef tallow, lard (leaf) and whale blubber. Mixtures of any of the above-named fatty acids with each other may also be used.

Ketene dimer products are manufactured in the form of waxy solids comprising the ketene dimers per se, in admixture with up to 20% by weight of impurities. These products are normally used as such, i.e. without separation of the impurities. For the purpose of this disclosure, all references to ketene dimers include mixtures thereof with these impurities and all proportions based on the weight of ketene dimer are based upon the weight of the manufactured product.

The stabilising agent also present in the compositions of the invention should be selected so as to be effective in enabling an aqueous dispersion, containing a ketene dimer and such a stabilising agent, to be dried to form a product which is redispersible in water and is thus useful in paper sizing processes. The nature of the stabilising agent should be such that it is compatible with the other components of the

resultant spray-dried product and such that it is not degraded during the drying process. Examples of compounds which are useful as stabilising agents are: gelatin, tristearin, gum arabic, sugars, ethyl cellulose, carboxymethylcellulose, polyacrylamides, water-soluble silicates, polyethers, polyesters, polyamides, starches, especially cationic starches, and polyvinyl alcohol.

The amount of stabilising agent which is employed is desirably proportional to the amount of ketene dimer in the resultant dispersion and will normally be adjusted so as to be just sufficient to enable the dispersion to be dried. The use of excess quantities of stabilising agent is less preferred. Normally the quantity of stabilising agent added to the dispersion is in the range from 25% to 100%, preferably from 25% to 75% by weight of the weight of ketene dimer present in the dispersion, i.e. the weight ratio of ketene dimer to the stabilising agent is in the range from 1:0.25 to 1:1 and, preferably, from 1:0.25 to 1:0.75. A preferred class of compounds used as stabilising agents are those which are known to be useful as ingredients of paper sizing compositions. Such agents may be employed in larger quantities than are required in order to cooperate successfully with the dimer and enable the desired stable water-dispensible particulate solid product to be formed. Such extra quantities make a contribution to the efficiency of the dried product when it is used in the paper sizing process. Examples of such agents are starches, such as cationic starches. A particularly preferred class of stabilising agents are those which are known to disperse in cold water, preferred examples being certain cationic starches such as those sold under the Trade Marks Solvitose NX and Perfectamyl PLV. The stabilising agent can be added to any aqueous dispersion of the ketene dimer. However, it is strongly preferred to add the stabilising agent to a dispersion which is itself useful as a paper size, so that redispersion of the dried product produces a dispersion which is useful as a paper size, without the need to disperse any additional components in order to form the size.

Ketene dimer dispersions normally comprise at least one surfactant and the presence of a surfactant in the composition from which the dried product is made and so in the dried product itself facilitates redispersion of the particulate solid product in the paper sizing process. Examples of suitable surfactants are the sodium salts of naphthalene formadehyde sulphonate condensates, esters of polyoxyethylene sorbitan or a sodium lignosulphonate.

The amount of surfactant added will usually be in the range 0.3% to 3.5% by weight of the ketene dimer, preferably in the range 1.25 to 2.5%.

The ketene dimer dispersions used in the spray drying processes may further comprise other conventional ingredients such as emulsifiers, stabilisers, retention aids and slimicides although the use of compounds which are sensitive to heat is preferably avoided.

In particular the ketene dimer dispersions preferably comprise a cationic polymer such as a polyamine in order to increase the efficiency of the dispersion as a paper size. Examples of polymers useful in this application are water soluble polyamino polyamideepichlohydrin resins, alkylene polyamine epichlorhydrin resins, poly(diallylamin)-epichlorhydrin resins, poly dialkyl dimethyl ammonium chloride and polyethyleneimines.

The amount of polymer present may be up to a weight ratio of ketene dimer to polymer of 1.0:1.0. However as the quantity of polymer increases there is an increasing tendency to produce a relatively sticky product and it is preferred that the weight ratio of ketene dimer to polymer is in the range 1.0:0.2 to 1.0:0.5.

The dispersion of the ketene dimer and stabilising agent can be dried by a variety of techniques. However exposure of the dimer to high temperatures for long periods can lead to its degradation either by charring or by hydrolysis and is best avoided. Techniques such as freeze drying and spray drying are preferably employed. We prefer to utilise spray drying although the conditions under which the drying is carried out must be monitored in order to minimise degradation of the ketene dimer. The spray drying of the ketene dimer dispersions can be carried out using conventional equipment. The temperature of the drier should be controlled so as to avoid any significant amount of charring or degradation of the components of the emulsion. Typically the temperature at the inlet to the dryer should be in the range 220° to 350°C and that at the outlet of the dryer 110° to 125°C. The drying process is carried out so as to produce a powdered product comprising less than 0.1% by weight of water.

The powder products must be dispersed in an aqueous medium in order to be useful in paper sizing processes. Typically the powder will be diluted with up to ten times its weight of water. Adequate dispersion is preferably obtained using vigorous agitation. We prefer to wet and/or slurry the powder with a small amount of water, e.g. about 5 to 30% by weight of the total dilution water is added to the powder, and to agitate those slurries with additional water to the desired content, usually about 10% by weight.

The invention is illustrated by the following examples:

Example 1

A dispersion of a stearic/palmitic alkyl ketene dimer having the following composition (Composition 1) was prepared.

Water	800 Kgs
Alkyl ketene dimer	100 Kgs
*Orotan SN	1.24 Kgs
a quaternised polyamine	100 Kgs
Slimicide	400 mls

*the sodium salt of a naphthalene formaldehyde sulphonate condensate.

A cationic starch product as sold under the Trade Mark Perfectamyl PLV was added to one sample of the dispersion in a quantity such that the ratio of the weight of ketene to starch was 1:1 (Composition 2). The same cationic starch product was added to a second sample of the dispersion in a quantity such that the ratio of the weight of ketene dimer to starch was 1:0.5 (Composition 3).

Compositions 2 and 3 were then spray dried using a Niro Production Minor Spray Drier set for Rotary Atomisation using an atomiser speed of 24,000 rpm. The liquor feed temperature was 15°C, the drier inlet temperature was 250°C and the drier outlet temperature 125°C. The product was a white powder.

The sizing efficiency of these powders 2 and 3 and dispersion 1 was assessed in a series of trials.

Trial A

Composition	1 minute Cobb Value (gsm)	
	off drier 100 °C	Natural Cure
1	42.6	20.9
2	39.3	20.6
3	71.5	23.4

Sizing condition

Furnish	Brown Waste
Retention aid	0.02% solids/fibre
Size addition level	0.2% KD/Fibre

Trial B

Example	1 minute Cobb Value (gsm)	
	off drier 100 °C	Natural Cure
1	40.4	35.3
2	46.0	35.5
3	42.9	32.0

Sizing Conditions

Furnish	Bleached sulphite
Retention aid	0.02% solids/fibre
Size addition level	0.1% KD/Fibre

Trial C

Example	1 minute Cobb Value (gsm)	
	off drier 100 °C	Natural Cure
1	69.2	38.8
2	37.8	25.0
3	32.2	25.5

Sizing Conditions

Furnish	Brown waste
Retention aid	0.02% solids/fibre
Size addition level	0.2% KD/Fibre

Trial D

Example	1 minute Cobb Value (gsm)	
	off drier 100 °C	Natural Cure
1	127.7	117.9
2	96.4	102.9
3	103.6	96.6

Sizing Conditions

Furnish	Bleached sulphite
Retention aid	0.02% solids/fibre
Size addition level	0.1% KD/Fibre

Trial E

Example	1 minute Cobb Value (gsm)	
	off drier 100 °C	Natural Cure
1	86.3	40.5
2	118.9	33.5
3	120.5	38.1

Sizing Conditions

Furnish	Brown waste
Retention aid	0.02% solids/fibre
Size addition level	0.2% KD/Fibre

Trial F

Example	1 minute Cobb Value (gsm)	
	off drier 100 °C	Natural Cure
1	90.7	81.7
2	32.6	25.3
3	30.3	26.7

Sizing Conditions

Furnish	Bleached sulphite
Retention aid	0.02% solids/fibre
Size addition level	0.2% KD/Fibre

Trials G and H are based on a product (Composition 4) with a surfactant content on ketene dimer of 0.62% but which are otherwise identical with and are manufactured under the same conditions as composition 3 described above.

Trial G

Example	1 minute Cobb Value (gsm)	
	off drier 100 °C	Natural Cure
1	32.0	25.8
4	43.9	28.9

Sizing Conditions

Furnish	Bleached sulphite + 5% chalk
Retention aid	0.02% solids/fibre
Size addition level	0.1% KD/Fibre

Trial H

Example	1 minute Cobb Value (gsm)	
	off drier 100 °C	Natural Cure
1	36.0	23.3
4	34.5	25.0

Furnish	Brown waste
Retention aid	0.02% solids/fibre
Size addition level	0.2% KD/Fibre

Trial J is based on a product (Composition 5) with a surfactant content on ketene dimer of 2.8% but which is otherwise identical with and is manufactured under the same conditions as composition 3.

Trial J

Example	1 minute Cobb Value (gsm)	
	off drier 100 °C	Natural Cure
1	37.6	27.8
5	44.6	32.6

Size conditions

Furnish	Bleached sulphite + 10% chalk
Retention aid	0.02% solids/fibre
Size addition level	0.5% KD/Fibre

Example 2

A dispersion (composition 6) of a stearic/palmitic alkyl ketene dimer having the following composition was prepared:

Alkyl ketene dimer	100 kgs
Orotan SN	0.62 kgs
Quaternised Polyamine	50 kgs
Slimicide	400 mls
Water	850 kgs

A cationic starch product as sold under the Trade Mark Solvitose NX was added to this dispersion in a quantity such that the ratio of the weight of ketene dimer to starch was 1:0.5 (composition 7).

Composition 7 was then spray dried using the conditions set forth in Example 1. The efficiency of sizing of compositions 6 and 7 was then assessed at that time and after storage at ambient temperatures for 3.5 and 4.5 months. The results were as follows:

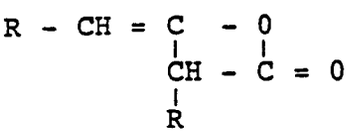
Composition	1 minute cobb value (gsm)	
	off drier 100 °C	Natural Cure
6	28.2	24.2
7	31.6	25.4
after 3½ months		
6	26.2	27.0
7	33.6	26.5
after 4½ months		
6	62.6	40.8
7	33.6	26.5

Sizing conditions

Furnish	Bleached sulphite
Retention Aid	0.02 solids/fibre
Size Addition	0.1 KD/fibre

Claims

1. A stable solid composition which is dispersible in water to form a paper sizing agent and comprises (1)a ketene dimer of the formula:



wherein R represents an alkyl, alkenyl, aryl or alkaryl group having from 8 to 24 carbon atoms and (2) a water-soluble stabilising agent, the stabilising agent and ketene dimer being present in the form of a water-dispersible homogeneous particulate solid.

2. A composition according to claim 1, the particulate solid being freeze- or spray-dried.

3. A composition according to claim 1 or 2, wherein the group R has an average value of from 12 to 18 carbon atoms.

4. A composition according to any preceding claim, wherein the stabilising agent is selected from gelatin, tristearin, gum arabic, sugars, ethylcellulose, carboxymethylcellulose, polyacrylamides, water-soluble silicates, polyethers, polyesters, polyamides, starches and polyvinyl alcohol.

5. A composition according to claim 4, wherein the stabilising agent is a cationic starch.

6. A composition according to any preceding claim, wherein the weight ratio of ketene dimers to the stabilising agent is in the range from 1:0.25 to 1:1.

7. A composition according to any preceding claim, wherein a polymeric cationic dispersing agent is present.

8. A composition according to claim 7, wherein the dispersing agent is selected from water-soluble alkylene polyamine epichlorhydrin resins and poly(diallylamine)-epichlorhydrin resins.

9. A composition according to claim 7 or 8, wherein the weight ratio of ketene dimer to the dispersing agent is in the range from 1:0.5 to 1:1.

10. A composition according to any preceding claim, wherein at least one surfactant is present.

11. A composition according to claim 10, wherein the surfactant is selected from salts of naphthalene formaldehyde sulphonate condensates, esters of polyoxyethylene sorbitan and salts of lignosulphonic acids.

12. A composition according to claim 10 or 11, wherein the weight ratio of surfactant to ketene dimer is in the range from 0.3% to 3.5%.

13. A process for the production of a stable solid composition which is dispersible in water to form a paper sizing agent, in which process:

(a) a ketene dimer having the formula defined in claim 1 and a water-soluble stabilising agent are formed into an aqueous dispersion and

(b) the aqueous dispersion is dried to form a homogeneous particulate water-dispersible solid.

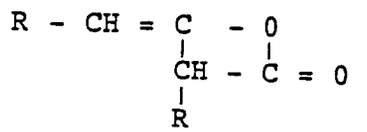
14. A process according to claim 13, wherein the dispersion is dried by passage through a spray or freeze drier.

15. A process according to claim 14, wherein the dispersion is spray-dried and temperature at the inlet to the drier is in the range from 220°C to 350°C and the temperature at the outlet from the drier is in the range from 110°C to 125°C.

16. A process for the production of sized paper or paper board, wherein a sizing agent is added to the paper or paper stock, characterised in that the sizing agent comprises an aqueous dispersion of a particulate solid according to any of claims 1 to 12 or which has been produced by a process according to any of claims 13 to 15.

Patentansprüche

1. Stabile, feste Zubereitung, die in Wasser dispergierbar ist zur Bildung eines Schlichtemittels für Papier, enthaltend
(1) ein Dimeres der Formel:



in der R eine Alkyl-, Alkenyl-, Aryl- oder Alkarylgruppe mit 8 bis 24 Kohlenstoffatomen bedeutet, und
(2) ein wasserlösliches Stabilisierungsmittel, wobei das Stabilisierungsmittel und das Keten-Dimere in Form einer in Wasser dispergierbaren homogenen teilchenförmigen festen Substanz vorliegt.

2. Zubereitung nach Anspruch 1, wobei die teilchenförmige feste Substanz gefriergetrocknet oder sprühgetrocknet ist.

3. Zubereitung nach den Ansprüchen 1 oder 2 wobei die Gruppe R einen Durchschnittswert von 12 bis 18 Kohlenstoffatomen hat.

4. Zubereitung nach den vorhergehenden Ansprüchen, wobei das Stabilisierungsmittel ausgewählt ist aus Gelatine, Tristearin, Gummi Arabicum, Zuckern, Ethylcellulose, Carboxymethylcellulose, Polyacrylamiden, wasserlöslichen Silikaten, Polyethern, Polyestern, Polyamiden, Stärken und Polyvinylalkohol.

5. Zubereitung nach Anspruch 4, wobei das Stabilisierungsmittel eine kationische Stärke ist.

6. Zubereitung nach den vorhergehenden Ansprüchen, wobei das Gewichtsverhältnis Keten-Dimeres zum Stabilisierungsmittel im Bereich von 1:0,25 bis 1:1 liegt.

7. Zubereitung nach den vorhergehenden Ansprüchen, wobei ein polymeres kationisches Dispergiermittel vorhanden ist.

8. Zubereitung nach Anspruch 7, wobei das Dispergiermittel ausgewählt ist aus wasserlöslichen Alkylenpolyamin-Epichlorhydrinharzen und Poly(diallylamin)-Epichlorhydrinharzen.

9. Zubereitung nach dem Ansprüchen 7 oder 8, wobei das Gewichtsverhältnis Keten-Dimeres zum Dispergiermittel im Bereich von 1:0,5 bis 1:1 liegt.

10. Zubereitung nach den vorhergehenden Ansprüchen, wobei wenigstens ein oberflächenaktives Mittel vorhanden ist.

11. Zubereitung nach Anspruch 10, wobei das oberflächenaktive Mittel ausgewählt ist aus Salzen von Naphthalin-Formaldehyd-Sulfonat-Kondensaten, estern von Polyoxyethylen-sorbit und Salzen von Lignosulfonsäuren.

12. Zubereitung nach den Ansprüchen 10 oder 11, wobei das Gewichtsverhältnis oberflächenaktives Mittel zu Keten-Dimer im Bereich von 0,3% bis 3,5% liegt.

13. Verfahren zur Herstellung einer stabilen festen Zubereitung, die in Wasser dispergierbar ist zur Bildung eines Schlichtemittels für Papier, in welchem Verfahren:

(a) ein Keten-Dimeres der in Anspruch 1 genannten Formel und ein wasserlösliches

Stabilisierungsmittel in eine wäßrige Dispersion gebracht werden und

(b) die wäßrige Dispersion getrocknet wird, um eine homogene, teilchenförmige in Wasser dispergierbare feste Substanz zu bilden.

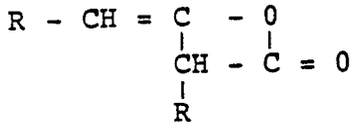
14. Verfahren nach Anspruch 13, worin die Dispersion mittels eines Durchgangs durch eine Sprühtrockner oder Gefriertrockner getrocknet wird.

15. Verfahren nach Anspruch 14, worin die Dispersion sprühgetrocknet wird und die Temperatur am Einlaß des Trockners im Bereich von 220°C bis 350°C liegt und die Temperatur am Auslaß des Trockners im Bereich von 110°C bis 125°C begrägt.

16. Verfahren zur Herstellung eines geschlichteten Papiers oder einer geschlichteten Pappe, wobei ein Schlichtemittel zum Papier oder zur Papiermasse zugesetzt wird, dadurch gekennzeichnet, daß das Schlichtemittel eine wäßrige Dispersion einer teilchenförmigen festen Substanz gemäß den Ansprüchen 1 bis 12 enthält oder daß es gemäß einem verfahren nach den Ansprüchen 13 bis 15 hergestellt worden ist.

Revendications

1. Composition solide stable, qui est dispersable dans l'eau pour former un agent d'encollage du papier et comprend (1) un dimère de cétène, de formule:



dans laquelle R représente un groupe alkyle, alcényle, aryle ou alcaryle, ayant de 8 à 24 atomes de carbone; et (2) un agent stabilisant hydrosoluble, l'agent stabilisant et le dimère de cétène étant présents sous la forme d'un solide particulaire homogène et dispersble dans l'eau.

2. Composition selon la revendication 1, le solide particulaire étant séché par lyophilisation ou par atomisation.

3. Composition selon la revendication 1 ou 2, dans laquelle le groupe R a une teneur moyenne de 12 à 18 atomes de carbone.

4. Composition selon l'une quelconque des revendications précédentes, dans laquelle l'agent stabilisant est choisi parmi la gélatine, la tristéarine, la gomme arabique, des sucres, l'éthylcellulose, de la carboxyméthylcellulose, des polyacrylamides, des silicates hydrosolubles, des polyéthers, des polyesters, des polyamides, des amidons et du poly(alcool vinylique).

5. Composition selon la revendication 4, dans laquelle l'agent stabilisant est un amidon cationique.

6. Composition selon l'une quelconque des revendications précédentes, dans laquelle le rapport pondéral entre le dimère de cétène et l'agent stabilisant se situe entre 1:0,25 et 1:1.

7. Composition selon l'une quelconque des revendications précédentes, dans laquelle est présent un agent dispersant cationique polymère.

8. Composition selon la revendication 7, dans laquelle l'agent dispersant est choisi parmi des résines hydrosolubles alkylène polyamine-épichlorhydrine et des résines de poly(diallylamine)-épichlorhydrine.

9. Composition selon la revendication 7 ou 8, dans laquelle le rapport pondéral entre le dimère de cétène et l'agent dispersant se situe entre 1:0,5 et 1:1.

10. Composition selon l'une quelconque des revendications précédentes, dans laquelle est présent au moins un surfactif.

11. Composition selon la revendication 10, dans laquelle le surfactif est choisi parmi des sels de produits de condensation de naphtalène formaldéhyde sulfonates, des esters de polyoxyéthylène sorbitanne et des sels d'acides lignosulfoniques.

12. Composition selon la revendication 10 ou 11, dans laquelle le rapport pondéral du surfactif au dimère de cétène se situe entre 0,3% et 3,5%.

13. Procédé pour la production d'une composition solide stable, qui est dispersable dans l'eau pour former un agent d'encollage du papier, procédé dans lequel:

(a) on met un dimère de cétène, répondant à la formule définie dans la revendication 1, et un agent stabilisant hydrosoluble, sous forme d'une dispersion aqueuse et

(b) on sèche la dispersion aqueuse pour former un solide particulaire homogène disperable dans l'eau.

14. Procédé selon la revendication 13, dans lequel on sèche la dispersion en la faisant passer dans un sécheur par atomisation ou par lyophilisation.

15. Procédé selon la revendication 14, dans lequel on sèche la dispersion par atomisation, et la température à l'entrée du sécheur se situe entre 220°C et 350°C, et la température à la sortie du sécheur se situe entre 110°C et 125°C.

16. Procédé pour la production de papier ou de carton encollés, dans lequel on ajoute un agent d'encollage au papier ou à la pâte à papier, procédé caractérisé en ce que l'agent d'encollage comprend une dispersion aqueuse d'un solide particulaire selon l'une quelconque des revendications 1 à 12 ou qui a été produite par un procédé selon l'une quelconque des revendications 13 à 15.