

(19) **DANMARK**



Patent- og
Varemærkestyrelsen

(10) **DK/EP 2390284 T4**

(12) **Oversættelse af ændret
europæisk patentskrift**

-
- (51) Int.Cl.: *C 09 C 1/02 (2006.01)* *C 09 C 1/42 (2006.01)* *C 09 C 3/04 (2006.01)*
D 21 H 17/00 (2006.01) *D 21 H 17/67 (2006.01)* *D 21 H 19/00 (2006.01)*
D 21 H 19/38 (2006.01)
- (45) Oversættelsen bekendtgjort den: **2017-06-19**
- (80) Dato for Den Europæiske Patentmyndigheds
bekendtgørelse om opretholdelse af patentet i ændret form: **2017-03-15**
- (86) Europæisk ansøgning nr.: **10164211.4**
- (86) Europæisk indleveringsdag: **2010-05-28**
- (87) Den europæiske ansøgnings publiceringsdag: **2011-11-30**
- (84) Designerede stater: **AL AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HR HU IE IS IT LI LT LU LV
MC MK MT NL NO PL PT RO SE SI SK SM TR**
- (73) Patenthaver: **Omya International AG, Baslerstrasse 42, 4665 Oftringen, Schweiz**
- (72) Opfinder: **Buri, Matthias, Mätteliweg 20, 4852 Rothrist, Schweiz**
Gane, Patrick A.C., Studenweg 8, 4852 Rothrist, Schweiz
- (74) Fuldmægtig i Danmark: **Zacco Denmark A/S, Arne Jacobsens Allé 15, 2300 København S, Danmark**
- (54) Benævnelse: **Fremgangsmåde til fremstilling af suspensioner af mineralmaterialer med højt faststofindhold**
- (56) Fremdragne publikationer:
EP-A1- 0 108 842
EP-A1- 2 143 688
EP-A1- 2 194 103
EP-A2- 1 160 201
WO-A1-97/10309
US-A1- 2009 111 906

DESCRIPTION

[0001] The present invention relates to a process for manufacturing high solids suspensions of mineral materials, the high solids aqueous suspension of mineral materials obtained by this process, as well as the use of such suspensions.

[0002] In the manufacturing method of a sheet of paper, cardboard or analogous product, one skilled in the art increasingly tends to replace part of the expensive cellulose fibres by cheaper mineral matter in order to reduce the cost of the paper while improving its properties such as opacity and/or brightness.

[0003] Such well-known mineral materials, comprise for example natural calcium carbonate, synthetic calcium carbonate, and miscellaneous analogous fillers containing calcium carbonates such as dolomite or mixed carbonate based fillers; various matter such as talc or analogues; mica, clay, titanium dioxide, etc.

[0004] For reasons of applicability, transport, storage and drying cost, it is especially useful to produce the mineral material in the form of high solids suspensions, i.e. containing only little water, which, however, generally is only possible by adding a high amount of dispersing agents or grinding aids.

[0005] Thus, for a long time, it has been common to use in a wet grinding process, as dispersing agents, water soluble polymers based on partially or totally neutralised polyacrylic acids or their derivatives (EP 0 046 573, EP 0 100 947, EP 0 100 948, EP 0 129 329, EP 0 261 039, EP 0 516 656, EP 0 542 643, EP 0 542 644, EP 0 717 051) to provide aqueous mineral suspensions that meet the desired refinement and low viscosity criteria. These dispersing agents, however, have to be used in a high quantity, which is not only not desirable from the economical view, but also disadvantageous with respect to the capability of the final product of developing scattering visible light as required by the end user in paper application.

[0006] Consequently, there are several approaches in the prior art to provide high solids suspensions of mineral materials, wherein the amount of the dispersing agents above is decreased, and the mentioned drawbacks are avoided or reduced.

[0007] For example, WO 02/49766, EP 0 850 685, WO 2008/010055, WO 2007/072168 disclose processes for manufacturing aqueous suspensions of refined mineral material, with a dry matter concentration that can be high, while having a low Brookfield™ viscosity that remains stable over time. In this respect, the use of specific dispersants like copolymers of acrylic acid with maleic acid, of a particular rate of neutralization, or the use of inorganic fluorine compounds to be put into aqueous suspensions of the mineral particles issuing from the mechanical and/or thermal concentration step following a step of wet grinding at a low solid content without the use of dispersing agent nor grinding aid, are mentioned.

[0008] Furthermore, unpublished European patent application No. 08 172 465 discloses the use of lithium-neutralised water-soluble organic polymers acting as grinding and/or dispersing capacity enhancers in order to achieve aqueous mineral material suspensions with the required properties while minimizing the dispersant and/or grinding aid agent demand without decreasing the properties of the final products like the optical properties of the paper.

[0009] A further approach is mentioned in EP 0 614 948 or in EP 0 857 763 relating to a process for comminuting in the wet state alkaline earth metal pigments, in particular, but not exclusively, calcium carbonate pigments, by preparing an aqueous suspension of a particulate alkaline earth metal compound, subjecting this suspension to attrition grinding with a particulate grinding medium under conditions such as to yield a product having a particle size distribution such that at least 90% by weight of the particles have an equivalent spherical diameter smaller than 2 μm ; and allowing water to evaporate from the suspension under the action of heat contained in the suspension until the percentage by weight of dry alkaline earth metal compound in the suspension has increased to at least 70% by weight, at an initial solids concentration of from 40 to 70 wt%. It can be taken from the Examples that the use of a suspension having an initial solids content of above 70 wt% is not suitable for the claimed process using 0.7 wt%. Thus, EP 0 614 948 describes a process for grinding mineral material at a high solids content clearly indicating that this process is only suitable for suspension having an initial solids content of not more than 70 wt%.

[0010] Thus, the processes known in the prior art either still use a relatively high amount of dispersant, or are only suitable for suspensions of mineral materials having a relatively low solids content, and thus being not very efficient.

[0011] Consequently, it is one object of the present invention to provide a process for manufacturing high solids aqueous suspensions of mineral material using a reduced amount of dispersant and having a low Brookfield viscosity.

[0012] This object is achieved by a process for manufacturing high solids aqueous suspensions of mineral material(s) as defined in claim 1 comprising the steps of:

1. a) providing at least one mineral material,
2. b) preparing an aqueous suspension comprising the at least one mineral material of step a),
3. c) grinding the mineral material of the resulting aqueous suspension of step b),
4. d) concentrating the aqueous suspension of ground mineral material of step c) by flash cooling,

characterised in that

- at least one dispersing agent is added to the aqueous suspension of step b), wherein the at least one dispersing agent is added completely before grinding step c), or stepwise before and during grinding step c), or before and during and/or after grinding step c), wherein the at least one dispersing agent is independently selected from the

group comprising homopolymers or copolymers of polycarboxylic acids such as acrylic, methacrylic acids or maleic acid; and/or their salts, e.g. partially or completely neutralized acid salts with sodium, lithium, potassium, ammonium, calcium, magnesium, strontium and/or aluminium or mixtures thereof, preferably sodium, calcium and magnesium; or derivatives of such acids such as esters based on, e.g., acrylic acid, methacrylic acid, maleic acid, fumaric acid, itaconic acid, e.g. acryl amide or acrylic esters such as methylmethacrylate, or mixtures thereof; alkali polyphosphates; or mixtures thereof, and wherein

- before, during, or after step c), but before step d), at least one earth alkali oxide and/or hydroxide is added to the aqueous suspension of step b), and wherein
- the final solids content of the aqueous suspension obtained after step d) is from 80 to 85 wt%.

[0013] Mineral materials suitable for the use in the process according to the present invention are preferably selected from the group comprising natural calcium carbonate (GCC) such as marble, chalk, limestone; precipitated calcium carbonate (PCC) such as aragonitic PCC, vateritic PCC and/or calcitic PCC, especially prismatic, rhombohedral or scalenohedral PCC; surface modified calcium carbonate; dolomite; talc; bentonite; clay; magnesite; satin white; sepiolite, huntite, diatomite; silicates; and mixtures thereof.

[0014] It is preferred that from 40 to 85 wt%, preferably from 45 to 80 wt%, more preferably from 50 to 75 wt%, most preferably from 60 to 70 wt%, e.g. 65 wt% of the at least one mineral material particles provided in step a) have an equivalent spherical diameter of $< 10 \mu\text{m}$, measured by means of a Sedigraph 5100 device from the company Micromeritics, USA, in an aqueous solution of 0.1 wt-% $\text{Na}_4\text{P}_2\text{O}_7$, wherein the samples were dispersed using a high-speed stirrer and ultrasound.

[0015] According to step b) of the process of the present invention, an aqueous suspension is prepared from the mineral material provided in step a).

[0016] This aqueous suspension, before it is subjected to step d), preferably has a solids content of from above 70 to 80 wt%, especially from 72 to 79 wt%, more preferably from 74 to 78 wt%, and most preferably from 76 to 78 wt% based on the total weight of the suspension.

[0017] Subsequently, the aqueous suspension of mineral material(s) is subjected to a grinding step, which may be performed in any of the known grinding equipment with which those skilled in the art are familiar for grinding mineral materials.

[0018] Especially conventional attritor mills such as those distributed by the company Dynomill are suitable in this respect, preferably using grinding balls made of glass, porcelain, and/or metal; especially preferably, however, grinding balls are used made of, e.g., zirconium silicate, zirconium dioxide and/or baddeleyite with a diameter of 0.2 to 5 mm, preferably 0.2 to 2 mm,

but also 0.5 to 5 mm, e.g., 1 to 2 mm. Quartz sand having an equivalent spherical diameter of 0.1 to 2 mm may also be used.

[0019] In a special embodiment, at least one dispersing agent, which preferably also act as a deflocculation agent, is added to the aqueous suspension of step b), wherein, the at least one dispersing agent may be completely added before grinding step c), or stepwise before and during grinding step c), and optionally also before and during and/or after grinding step c).

[0020] Useful amounts of such dispersing agents added to the aqueous suspension during step b), during and after step b), but before step c), are from 0.01 to 1.25 wt%, preferably from 0.01 to 0.1 wt%, more preferably from 0.02 to 0.07 wt%, most preferably from 0.03 to 0.05 wt% based on the dry weight of the mineral material.

[0021] Preferred amounts of such de-flocculation agents additionally added during step c) are from 0.05 to 1 wt%, more preferably from 0.1 to 0.7 wt%, even more preferably from 0.15 to 0.55, e.g. 0.3 wt% based on the dry weight of the mineral material.

[0022] The total amount of the at least one dispersing agent added before grinding step c), or before and during step c), or before and during and/or after grinding step c), preferably is from about 0.01 to 1.25 wt%, more preferably 0.05 to 1 wt%, even more preferably 0.1 to 0.7 wt%, most preferably 0.3 to 0.5 wt% based on the dry weight of the mineral material.

[0023] In another preferred embodiment, the dispersing agent is added in an amount such that, with respect to the specific surface area, measured by BET, of the ground material after step c), it is present in amount of below 0.15 mg/m², e.g. 0.05 to 0.08 mg/m², if it is added before grinding step c), and, in an amount of from 0.05 mg/m² to 1.5 mg/m², e.g. 0.5 to 0.8 mg/m², if it is added during grinding step c).

[0024] Dispersing agents, which may be used with respect to step b) and c) may be those selected from the group comprising homopolymers or copolymers of polycarboxylic acids such as acrylic or methacrylic acids or maleic acid; and/or their salts, e.g. partially or completely neutralized acid salts with sodium, lithium, potassium, ammonium, calcium, magnesium, strontium and/or aluminium or mixtures thereof, preferably sodium, calcium and magnesium; or derivatives of such acids such as esters based on, e.g., acrylic acid, methacrylic acid, maleic acid, fumaric acid, itaconic acid, e.g. acryl amide or acrylic esters such as methylmethacrylate, or mixtures thereof; alkali polyphosphates; or mixtures thereof.

[0025] The molecular weight Mw of such products is preferably in the range of from 1000 to 15000 g/mol, more preferably in the range of from 3000 to 7000 g/mol, e.g. 3500 g/mol, most preferably in the range of from 4000 to 6000 g/mol, e.g. 5500 g/mol.

[0026] In a preferred embodiment, the earth alkali oxide and/or hydroxide added to the aqueous suspension of step b) before, during, or after step c), but before step d), is lime

and/or burnt dolomite, wherein lime in the context of the present invention comprises calcium oxide and calcium hydroxide.

[0027] Only small amounts of earth alkali oxide and/or hydroxide are sufficient to be used during grinding, such as amounts of from about 0.001 to 0.1 wt%, preferably 0.005 to 0.07 wt%, more preferably 0.007 to 0.05 wt%, most preferably 0.01 to 0.03 wt%, e.g. 0.02 wt% based on the dry weight of the mineral material.

[0028] Grinding step c) may be carried out once or several times. For example, the aqueous suspension may be partially ground in a grinder, the suspension of partially ground mineral material may then be fed to a second grinder for further grinding, etc., until the desired particle size is obtained. The desired particle size can also be obtained in one step by adjusting the residence time in the grinder.

[0029] It is possible to grind the mineral material such that from 20 to 70 wt%, preferably from 36 to 68 wt%, more preferably from 40 to 60 wt%, e.g. 50 wt% of the at least one mineral material particles have a particle size of $< 2 \mu\text{m}$ after step c).

[0030] In an even more preferred embodiment, the mineral material may be ground such that from 10 to 90 wt%, preferably from 20 to 80 wt%, more preferably from 36 to 75 wt%, especially from 40 to 70 wt%, most preferably from 50 to 65 wt%, e.g. 55 wt% of the at least one mineral material particles have a particle size of $< 1 \mu\text{m}$ after step c).

[0031] Furthermore, the aqueous suspension of ground mineral material may also be subjected to a separation step after grinding, wherein a finer fraction is separated from a coarser fraction, just as desired within the above-mentioned equivalent spherical diameter ranges of the mineral material.

[0032] The separation may take place by means of well-known techniques such as screening, filtration, or centrifugation, wherein for the most applications centrifugation will be preferred, using commonly known equipment.

[0033] In this respect, it is also possible that the separated coarse fraction is recirculated into any one of one to several grinders of step c).

[0034] Subsequently, in step d), the aqueous suspension of the ground, and optionally separated, mineral material is concentrated by flash cooling. Flash cooling in the context of the present invention is carried out by allowing water to evaporate from the aqueous suspension under the action of heat contained in the suspension.

[0035] This may take place by the heat which has been generated in the suspension as a direct result of the grinding. For example, during the grinding step the temperature of the suspension may rise to the boiling point of the aqueous phase of the suspension, and the evaporation of water occurs naturally under the action of the heat contained in the suspension.

In this respect, the temperature generated by grinding may also be below the boiling point of the water contained in the suspension. The heat contained in the suspension as a result of the energy dissipated in the suspension during grinding may be sufficient to increase the percentage by weight of dry alkaline earth metal compound in the suspension to within the range from 79 to 85 wt% as mentioned below.

[0036] Alternatively or additionally, the suspension may be heated by a heating means such as a heat exchanger to a temperature within the range of from 50 °C to the boiling point of the suspension, preferably to within the range of from 60 to 90 °C, more preferably from 70 to 85 °C, e.g. from 81 to 83 °C.

[0037] For instance, heat may be supplied to the suspension by passing the suspension through one side of a non-contact heat exchanger through the other side of which is passed a hot fluid, preferably at a temperature in the range from 50 to 100 °C.

[0038] Basically, known flash cooler systems consist of a (vacuum) chamber, into which the suspension is fed. Depending upon the temperature and the vacuum, the water in the suspension will evaporate. The evaporation results in an increase of the solids content. Cooling will take place simultaneously.

[0039] In this respect, the temperature of the aqueous suspension at the inlet of the grinder may be from 20 to 80°C, preferably from 20 - 50°C, and at the outlet of the grinder, preferably of from 80 to 105°C.

[0040] Thus, the temperature of the aqueous suspension at the inlet of the flash cooler may be from 70 to 105°C, e.g. 95°C and at the outlet of the flash cooler less than 60 to 30°C, e.g. 35°C.

[0041] Advantageously, the aqueous suspension is exposed to reduced pressure, preferably to a pressure of from 200 to 500 mbar, more preferably of from 250 to 400 mbar, most preferably a pressure of from 288 to 360 mbar, e.g. of from 300 to 350 mbar.

[0042] The final solids content of the aqueous suspension obtained after step d) preferably is from 79 to 85 wt%, especially from 79.5 to 84 wt%, more preferably from 80 to 83 wt%, e.g. 82 wt%.

[0043] With respect to the initial solids content of step b), it is thus preferred that the final solids content of the aqueous suspension after step d) is at least 1 wt%, preferably at least 2 wt%, more preferably at least 3 wt% higher than the initial solids content of the aqueous suspension obtained in step b).

[0044] In an especially preferred embodiment, the initial solids content of an aqueous suspension of mineral material of 76 to 78 wt% is raised to 80 to 81 wt% by the process of the present invention, wherein the mineral material preferably is comminuted such that while

initially 66 wt% of the mineral material particles have an equivalent spherical diameter of $< 10 \mu\text{m}$, 36 to 65 wt% of the final mineral material produced according to the process of the present invention have an equivalent spherical diameter of $< 2 \mu\text{m}$, and preferably even $< 1 \mu\text{m}$.

[0045] Furthermore, in an especially preferred embodiment, the Brookfield viscosity of the final aqueous suspension after step d) measured at 23°C after 1 minute of stirring by the use of a RVT model Brookfield™ viscosimeter at room temperature and a rotation speed of 100 rpm with the appropriate spindle is from 50 to 1000 mPa·s, preferably 100 to 750 mPa·s, more preferably from 150 to 600 mPa·s, most preferably from 200 to 460 mPa·s, e.g. 300 mPa·s.

[0046] A second aspect of the present invention is the provision of a high solids aqueous suspension of mineral material(s) obtained by the process as described above.

[0047] In this respect, a high solids aqueous suspension of mineral material obtained by the process of the present invention especially preferably has a solids content of from 80 to 81 wt%, wherein 36 to 65 wt% of the mineral material have an equivalent spherical diameter of $< 2 \mu\text{m}$, and preferably even $< 1 \mu\text{m}$.

[0048] Furthermore, a third aspect of the present invention is the use of the high solids aqueous suspension of mineral materials obtained by the process as described above in paper, paper coating colours, paints and plastics.

[0049] The following examples and experiments serve to illustrate the present invention and should not restrict it in any way.

EXAMPLES:

Comparative Example 1:

[0050] Natural ground calcium carbonate (limestone from Orgon, France), 66 wt% of which having an equivalent spherical diameter of $< 10 \mu\text{m}$, were suspended in water together with 0.07 wt%, based on the dry weight of calcium carbonate, of a sodium polyacrylate of $M_w = 3500 \text{ g/mol}$ until a solids content of 76 wt% based on the total weight of the suspension was reached.

[0051] The resulting aqueous suspension was ground in a vertical cylindrical 7 m^3 volume wet mill, using 15 tons of zircon dioxide grinding media having a medium diameter of about 1 - 2 mm, using additional dispersant (polyacrylate of $M_w = 5500 \text{ g/mol}$, wherein 70 mol% of the carboxylic groups are neutralized by sodium, and 30 mol% by calcium) in a quantity of 0.15 wt% , based on the dry weight of calcium carbonate, until 62 wt% of the calcium carbonate

particles had an equivalent spherical diameter of $< 2 \mu\text{m}$, and 36 wt% $< 1 \mu\text{m}$, respectively. The specific surface area of the dry calcium carbonate, measured by BET, was $7.1 \text{ m}^2/\text{g}$.

[0052] Subsequently, the ground calcium carbonate suspension was continuously centrifuged in a conventional continuous centrifuge at a speed of 1200 rpm.

[0053] The centrifuged aqueous suspension having a solids content of 76 wt% was fed into a continuously running flash cooler. The feeding speed was adjusted to reach approximately 80 wt% slurry solids at the outlet and concentrated at the following conditions:

•	Pressure at the top of the flash cooler:	288 mbar
•	Pressure at the bottom of the flash cooler:	350 mbar
•	Temperature at the entry of the flash cooler:	83°C
•	Temperature at the exit of the flash cooler:	39°C

[0054] The final solids content after the concentration step was determined to be 80 wt% by drying 20 g of the suspension in an oven at 110°C to weight constancy $\pm 0.1 \text{ wt\%}$.

[0055] The Brookfield viscosity of the final aqueous suspension measured after 1 minute of stirring by the use of a RVT model Brookfield™ viscosimeter at room temperature and a rotation speed of 100 rpm with a spindle No. 3 at the exit of the flash cooler was $150 \text{ mPa}\cdot\text{s}$; $153 \text{ mPa}\cdot\text{s}$ after 24 hours, and $162 \text{ mPa}\cdot\text{s}$ after 3 days.

Example 2:

[0056] Natural ground calcium carbonate (limestone from Orgon, France), 66 wt% of which having an equivalent spherical diameter of $< 10 \mu\text{m}$, were suspended in water together with 0.07 wt%, based on the dry weight of calcium carbonate, of a sodium polyacrylate of $M_w = 3500 \text{ g/mol}$ until a solids content of 78 wt% based on the total weight of the suspension was reached.

[0057] The resulting aqueous suspension was ground in a vertical cylindrical 7 m^3 volume wet mill, using 15 tons of zircon dioxide grinding media having a medium diameter of about 1-2 mm, using dispersant (polyacrylate of $M_w = 5500 \text{ g/mol}$, wherein 70 mol% of the carboxylic groups are neutralized by sodium, and 30 mol% by calcium) in a quantity of 0.15 wt%, based on the dry weight of calcium carbonate. Then, a second grinding pass was carried out in a vertical cylindrical 7 m^3 volume wet mill, using 15 tons of zircon dioxide grinding media having a medium diameter of about 0.7 - 1.5 mm, using additional dispersant (polyacrylate of $M_w = 5500 \text{ g/mol}$, wherein 50 mol% of the carboxylic groups are neutralized by sodium, and 50 mol% by magnesium) in a quantity of 0.55 wt%, based on the dry weight of calcium carbonate, and 0.02 wt% based on the dry weight of calcium carbonate of lime ($> 97 \text{ wt\% Ca(OH)}_2$), until

65 wt% of the calcium carbonate particles had an equivalent spherical diameter of $< 1 \mu\text{m}$. The specific surface area of the dry calcium carbonate, measured by BET, was $12.5 \text{ m}^2/\text{g}$.

[0058] Subsequently, the ground calcium carbonate suspension was continuously centrifuged in a conventional continuous centrifuge at a speed of 1300 rpm.

[0059] The centrifuged aqueous suspension having a solids content of 78 wt% was fed into a continuously running flash cooler. The feeding speed was adjusted to reach approximately 80 wt% slurry solids at the outlet and concentrated at the following conditions:

•	Pressure at the top of the flash cooler:	300 mbar
•	Pressure at the bottom of the flash cooler:	360 mbar
•	Temperature at the entry of the flash cooler:	81 °C
•	Temperature at the exit of the flash cooler:	36 °C

[0060] The final solids content after the concentration step was determined to be 80 wt% by drying 20 g of the suspension in an oven at 110 °C to weight constance $\pm 0.1 \text{ wt\%}$.

[0061] The Brookfield viscosity of the final aqueous suspension measured after 1 minute of stirring by the use of a RVT model Brookfield™ viscosimeter at room temperature and a rotation speed of 100 rpm with a spindle No. 3 at the exit of the flash cooler was 460 mPa·s; and 575 mPa·s after 6 days.

REFERENCES CITED IN THE DESCRIPTION

This list of references cited by the applicant is for the reader's convenience only. It does not form part of the European patent document. Even though great care has been taken in compiling the references, errors or omissions cannot be excluded and the EPO disclaims all liability in this regard.

Patent documents cited in the description

- [EP0046573A](#) **[0005]**
- [EP0100947A](#) **[0005]**
- [EP0100948A](#) **[0005]**
- [EP0129329A](#) **[0005]**
- [EP0261039A](#) **[0005]**
- [EP0516656A](#) **[0005]**
- [EP0542643A](#) **[0005]**

- EP0542644A [0005]
- EP0717051A [0005]
- WO0249766A [0007]
- EP0850685A [0007]
- WO2008010055A [0007]
- WO2007072168A [0007]
- EP08172465A [0008]
- EP0614948A [0009] [0009]
- EP0857763A [0009]

P a t e n t k r a v

1. Fremgangsmåde til fremstilling af vandige suspensioner af mineralmateriale(r) med højt faststofindhold, omfattende trinnene med:
- 5 a) tilvejebringelse af mindst et mineralmateriale,
b) fremstilling af en vandig suspension, omfattende det i det mindste ene mineralmateriale i trin a)
c) formaling af mineralmaterialet af den resulterende vandige suspension i trin b),
10 d) koncentring af den vandige suspension af formalet mineralmateriale i trin c) ved flash-køling,
- kendetegnet ved, at**
- mindst et dispergeringsmiddel tilsættes den vandige suspension i trin b), hvorved det i det mindste ene dispergeringsmiddel tilsættes helt før formalingstrin c), eller trinvis før og under formalingstrin c), eller før og under og/eller efter formalingstrin c), hvorved det i det mindste ene dispergeringsmiddel uafhængigt udvælges fra gruppen, der omfatter homopolymerer eller copolymerer af polycarboxylsyrer såsom acryl-, methacrylsyre eller maleinsyre; og/eller deres salte, f.eks. delvist eller helt neutraliserede syresalte med
- 15 natrium, lithium, kalium, ammonium, calcium, magnesium, strontium og/eller aluminium eller blandinger deraf, foretrukket natrium, calcium og magnesium; eller derivater af sådanne syrer såsom estere på basis af f.eks. acrylsyre, methacrylsyre, maleinsyre, fumarsyre, itaconsyre f.eks. acrylamid eller acrylestere såsom methylmethacrylat eller blandinger deraf; alkali-
- 20 lipolyphosphater; eller blandinger deraf, og hvorved
- der før, under eller efter trin c), men før trin d), tilsættes mindst et jordalkalioxid og/eller hydroxid til den vandige suspension i trin b), og hvor
- slutfaststofindholdet af den vandige suspension opnået efter trin d) er fra
- 25 80 til 85 vægtprocent.
- 30
2. Fremgangsmåde ifølge krav 1, **kendetegnet ved, at** det i det mindste ene mineralmateriale udvælges fra gruppen, omfattende naturligt calciumcarbonat (GCC) såsom marmor, kridt, kalksten; præcipiteret calciumcarbonat (PCC) såsom aragonitisk PCC, vateritisk PCC og/eller calcitisk PCC, især
- 35 prismatisk romboedrisk eller scalenoedrisk PCC; overflademodificeret calci-

umcarbonat; dolomit; talk; bentonit; ler; magnesit; satinhvidt; sepiolit, huntit, diatomit; silikater; og blandinger deraf.

- 5 3. Fremgangsmåde ifølge et hvilket som helst af kravene 1 eller 2, **kendetegnet ved, at** fra 40 til 85 vægtprocent, foretrukket fra 45 til 80 vægtprocent, mere foretrukket fra 50 til 75 vægtprocent, mest foretrukket fra 60 til 70 vægtprocent, f.eks. 65 vægtprocent af den i det mindste ene mineralmaterialepartikel i trin a) har en ækvivalent sfærisk diameter på $< 10 \mu\text{m}$.
- 10 4. Fremgangsmåde ifølge et hvilket som helst af kravene 1 til 3, **kendetegnet ved, at** den vandige suspension før trin d) har et faststofindhold på over 70 til 80 vægtprocent, foretrukket fra 72 til 79 vægtprocent, mere foretrukket fra 74 til 78 vægtprocent, mest foretrukket fra 76 til 78 vægtprocent på basis af den totale vægt af suspensionen.
- 15 5. Fremgangsmåde ifølge krav 4, **kendetegnet ved, at** den totale mængde af det i det mindste ene dispergeringsmiddel, der tilsættes før formalingstrin c) eller før og under trin c) eller før og under og/eller efter formalingstrin c), er fra omtrent 0,01 til 1,25 vægtprocent, foretrukket 0,05 til 1 vægtprocent, mere foretrukket fra 0,1 til 0,7 vægtprocent, mest foretrukket fra 0,3 til 0,5 vægtprocent på basis af mineralmaterialelets tørvægt.
- 20 6. Fremgangsmåde ifølge et hvilket som helst af de foregående krav, **kendetegner ved, at** molekylvægten M_w af det i det mindste ene dispergeringsmiddel ligger inden for området 1000 til 15000 g/mol, foretrukket inden for området 3000 til 7000 g/mol, f.eks. 3500 g/mol, mest foretrukket inden for området fra 4000 til 6000 g/mol, f.eks. 5500 g/mol.
- 25 7. Fremgangsmåde ifølge et hvilket som helst af de foregående krav, **kendetegnet ved, at** jordalkalioxid og/eller hydroxid, der tilsættes til den vandige suspension i trin b) før, under eller efter trin c), men før trin d), er kalk og/eller brændt dolomit.
- 30 8. Fremgangsmåde ifølge et hvilket som helst af de foregående krav, **kendetegnet ved, at** jordalkalioxid og/eller hydroxid tilsættes i en mængde fra
- 35

omtrent 0,001 til 0,1 vægtprocent, foretrukket 0,005 til 0,7 vægtprocent, mere foretrukket 0,007 til 0,5 vægtprocent, mest foretrukket 0,01 til 0,03 vægtprocent, f.eks. 0,02 vægtprocent på basis af mineralmaterialets tørvægt.

- 5 9. Fremgangsmåde ifølge et hvilket som helst af de foregående krav, **kendetegnet ved, at** trin c) udføres flere gange.
- 10 10. Fremgangsmåde ifølge et hvilket som helst af de foregående krav, **kendetegnet ved, at** fra 20 til 70 vægtprocent, foretrukket fra 36 til 68 vægtprocent, mere foretrukket fra 40 til 60 vægtprocent, f.eks. 50 vægtprocent af den i det mindste ene mineralmaterialepartikel har en partikelstørrelse på $< 2 \mu\text{m}$ efter trin c).
- 15 11. Fremgangsmåde ifølge et hvilket som helst af de foregående krav, **kendetegnet ved, at** fra 10 til 90 vægtprocent, foretrukket fra 20 til 80 vægtprocent, mere foretrukket fra 36 til 75 vægtprocent, især fra 40 til 70 vægtprocent, mest foretrukket fra 50 til 65 vægtprocent, f.eks. 55 vægtprocent af den i det mindste ene mineralmaterialepartikel har en partikelstørrelse på $< 1 \mu\text{m}$ efter trin c).
- 20 12. Fremgangsmåde ifølge et hvilket som helst af de foregående krav, **kendetegnet ved, at** den vandige suspension af formalet mineralmateriale efter formalingstrin c) udsættes for et separeringstrin, f.eks. ved hjælp af screening, filtrering eller centrifugering.
- 25 13. Fremgangsmåde ifølge et hvilket som helst af de foregående krav, **kendetegnet ved, at** der tilføres varme i trin d), hvorved det muliggøres, at vand kan fordampe fra den vandige suspension, hvorved denne varme frembringes ved hjælp af det tidligere formalingstrin c) eller tilføres eksternt.
- 30 14. Fremgangsmåde ifølge krav 13,
kendetegnet ved, at temperaturen på den vandige suspension ved indløbet af flash-køleren er fra 70 til 105 °C, f.eks. 95 °C, og ved udløbet af flash-køleren mindre end 60 til 30 °C, f.eks. 35 °C.

15. Fremgangsmåde ifølge et hvilket som helst af de foregående krav, **kendetegnet ved, at** den vandige suspension i trin d) udsættes for reduceret tryk, foretrukket for et tryk fra 200 til 500 mbar, mere foretrukket fra 250 til 400 mbar, mest foretrukket et tryk fra 288 til 360 mbar, f.eks. fra 300 til 350 mbar.
16. Fremgangsmåde ifølge et hvilket som helst af de foregående krav, **kendetegnet ved, at** slutfaststofindholdet af den vandige suspension opnået efter trin d) er fra 82 til 84 vægtprocent, f.eks. 83 vægtprocent.
17. Fremgangsmåde ifølge et hvilket som helst af de foregående krav, **kendetegnet ved, at** slutfaststofindholdet af den vandige suspension opnået efter trin d) er mindst 1 vægtprocent, foretrukket mindst 2 vægtprocent, mere foretrukket mindst 3 vægtprocent højere end udgangsfaststofindholdet af den vandige suspension opnået i trin b).
18. Fremgangsmåde ifølge et hvilket som helst af de foregående krav, **kendetegnet ved, at** Brookfield-viskositeten målt ved 23 °C efter 1 minuts omrøring ved 100 omdrejninger per minut af den endelige vandige suspension efter trin d) er fra 50 til 1000 mPa·s, foretrukket 100 til 750 mPa·s, mere foretrukket fra 150 til 600 mPa·s, mest foretrukket fra 200 til 460 mPa·s, f.eks. 300 mPa·s.
19. Vandig suspension af mineralmateriale(r) med højt faststofindhold opnået ved fremgangsmåden ifølge et hvilket som helst af kravene 1 til 18, hvor slutfaststofindholdet af den vandige suspension opnået efter trin d) er fra 83 til 85 vægtprocent.
20. Anvendelse af den vandige suspension af mineralmaterialer med højt faststofindhold opnået ved fremgangsmåden ifølge et hvilket som helst af kravene 1 til 18 i papir, papir-coating-farver, farver og plaststoffer, hvor slutfaststofindholdet af den vandige suspension opnået efter trin d) er fra 83 til 85 vægtprocent.