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MINERAL FIBRE MATS COMPRISING A BINDER BASED ON LIGNOSULFONATE AND A CARBONYL COMPOUND

The present invention relates to the field of products comprising mineral fibers, in particular glass fibers, bonded by a formaldehyde-free organic binder.

- 5 The present description more particularly relates to an aqueous binder capable of crosslinking thermally which includes at least one lignosulfonate and at least one carbonyl compound, and also to the products based on mineral fibers which result therefrom.

The products based on mineral fibers to which the invention more particularly
10 relates are mats of mineral fibers, in particular glass fibers, (also known as “nonwovens” or “veils”) which are manufactured according to known processes proceeding by the dry route or the wet route.

In the dry-route process, molten matter present in a furnace is conveyed toward a set of bushings from which filaments flow out by gravity and are drawn by a
15 gaseous fluid. The continuous mineral filaments are collected on a conveyor, where they become entangled, forming a mat.

A binder is applied to the upper face of the mat thus formed using an appropriate device, generally proceeding by curtain coating, and the excess binder is removed by suction on the opposite face. The mat subsequently enters a device
20 containing hot air, the temperature of which, of the order of 200 to 250 °C, is adjusted in order to remove the water and to crosslink the binder in a very short time, of the order of approximately ten seconds to 1 minute, and then the mat of mineral fibers is collected in the form of a roll.

In the wet-route process, the mat is obtained from an aqueous dispersion of cut
25 mineral fibers which is deposited, by means of a forming head, on a conveyor provided with perforations and the water is extracted through the conveyor by virtue of a suction box. The cut mineral fibers remaining on the conveyor form a mat which is treated under the same conditions as those described for the dry-route process.

In the abovementioned processes, the role of the binder is to bind the mineral
30 fibers to one another and to confer, on the mat containing them, mechanical

properties suitable for the desired use, in particular sufficient stiffness to allow it to be easily handled without the risk that it may be torn.

The binder to be applied to the mineral fibers is generally in the form of an aqueous solution containing at least one thermosetting resin and additives, such as a catalyst
5 for the crosslinking of the resin, an adhesion-promoting silane, a water repellent, etc.

The most commonly used thermosetting resins are formaldehyde-based resins, in particular phenolic resins belonging to the family of the resols, urea-formaldehyde resins and melamine-formaldehyde resins. These resins have good crosslinkability under the abovementioned thermal conditions, are soluble in water, have a good
10 affinity for the mineral fibers and are furthermore relatively inexpensive.

However, these resins are liable to contain free formaldehyde, the presence of which is not desired because of the undesirable effects in terms of human health and the environment. For some years, regulations regarding environmental protection have become more restrictive and are forcing manufacturers of resin
15 and fibrous products to seek solutions which make it possible to further lower the content of free formaldehyde.

Solutions for replacing formaldehyde-based resins for bonding mineral fibers are known and are based on the use of a carboxylic acid polymer in combination with a β -hydroxyamide and an at least trifunctional monomeric carboxylic acid (US 5 340 868).
20 Adhesive compositions comprising an alkanolamine containing at least two hydroxyl groups and a polycarboxylic polymer (US 6 071 994, US 6 099 773, US 6 146 746, US 2002/091185) have been proposed.

Adhesive compositions which comprise a polycarboxylic polymer, a polyol and a catalyst, which catalyst contains phosphorus (US 5 318 990, US 5 661 213, US 6 331
25 350, US 2003/0008978), a fluoroborate (US 5 977 232) or else a cyanamide, a dicyanamide or a cyanoguanidine (US 5 932 689), have also been described.

Furthermore, adhesive compositions based on saccharides capable of crosslinking under the effect of heat are known.

In US 5 895 804, the adhesive composition comprises a polycarboxylic polymer having at least two carboxylic acid functional groups and a molecular weight at least equal to 1.000, and a polysaccharide having a molecular weight at least equal to 10.000.

- 5 CN 103 031 108 A discloses a method for manufacturing an adhesive composition comprising glyoxal and alkali metal lignosulfonate, in the form of an aqueous solution. FR 2 208 963 A1 discloses an aqueous adhesive composition comprising alkali metal or alkaline earth metal lignosulfates and furfural.

- US 2014/113123 A1 discloses an aqueous binder composition for mineral fiber
10 mats, comprising a lignosulfate and thermosetting phenol-formaldehyde resin.

- Finally, WO 2012/172252 describes an aqueous formaldehyde-free binder for mats of fibers which comprises (in parts by weight): a lignosulfonic acid salt (20 to 95 parts), an oligosaccharide (5 to 80 parts) and a crosslinking catalyst selected from phosphorus-containing compounds and sulfates (5 to 20 parts per
15 100 parts of lignosulfonic acid salt and of oligosaccharides).

The present invention more particularly relates to fibrous products based on mineral fibers, in particular glass fibers, which are in the form of mats, according to claim 1.

In particular, the targeted mats are intended for the manufacture of leaktight bituminous roofing membranes.

- 20 The present disclosure aims to propose an alternative to binders liable to contain or to generate formaldehyde and which make it possible to confer good mechanical properties on mats based on mineral fibers, in particular an improved breaking stress.

This aim is achieved by virtue of the aqueous binder for mineral fibers, in particular glass fibers, which comprises

- 25 - at least one ammonium lignosulfonate or an alkali metal or alkaline earth metal salt of lignosulfonic acid, and
- at least one carbonyl compound of formula



in which:

- R represents a saturated or unsaturated and linear, branched or cyclic hydrocarbon radical, a radical containing one or more aromatic rings which consist of 5 or 6 carbon atoms, a radical containing one or more aromatic heterocycles containing 4 or 5 carbon atoms and an oxygen, nitrogen or sulfur atom, it being possible for the R radical to contain other functional groups, especially hydroxyl or alkoxy groups, in particular methoxy groups,
 - R₁ represents a hydrogen atom or a C₁-C₁₀ alkyl radical, et
 - x varies 1 to 10,
- 10 said binder being devoid of hydrogenated sugar and of melamine.

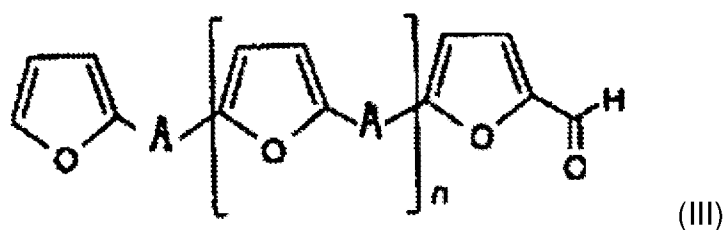
Ammonium lignosulfonate is a byproduct resulting from the treatment of wood for the manufacture of paper pulp according to the "sulfite" process. The treatment of the paper pulp with ammonium sulfite or ammonium bisulfite makes it possible to obtain ammonium lignosulfonates.

- 15 In addition, ammonium lignosulfonate makes it possible to confer fire resistance properties on the binder.

- The alkali metal or alkaline earth metal salts of sulfonic acid are generally complex mixtures of several lignosulfonic acids in the salified form, commonly denoted "lignosulfonates". Lignosulfonates are byproducts resulting from the treatment of
- 20 wood for the manufacture of paper pulp according to the abovementioned "sulfite" process which employs a sulfite or a bisulfite. Depending on the nature of the counterion of the sulfite or bisulfite employed, alkali metal or alkaline earth metal salts of lignosulfonic acid are obtained in particular. In the present invention, the preferred alkali metal salts of lignosulfonic acid are sodium or potassium,
- 25 advantageously sodium, lignosulfonates, and the preferred alkaline earth metal salts of lignosulfonic acid are magnesium or calcium lignosulfonates.

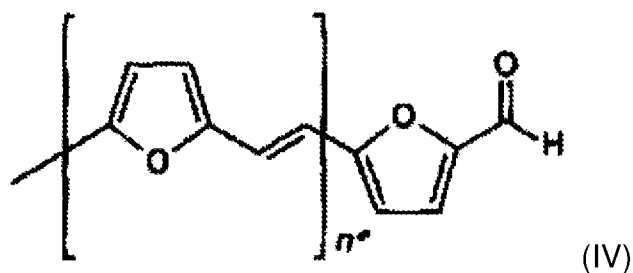
The carbonyl compound is a monofunctional or polyfunctional aldehyde selected from the group consisting of acetaldehyde, propionaldehyde, dimethoxyethanal, butyraldehyde, in particular n-butyraldehyde, glyoxal, malonaldehyde,

- succinaldehyde, glutaraldehyde, 2-hydroxyglutaraldehyde, 3-methylglutaraldehyde, adipaldehyde, suberaldehyde, sebacaldehyde, malealdehyde, fumaraldehyde, poly(acroleins), dialdehyde starch, furfural (2-furancarboxaldehyde), 5-methylfurfural (2-methyl-5-furancarboxaldehyde), hydroxymethylfurfural (2-hydroxymethyl-5-furancarboxaldehyde), 2,5-furancarboxydialdehyde, vanillin and vanillin polymers, in particular bis-vanillin, cinnamaldehyde and cinnamaldehyde polymers, phthalaldehyde, isophthalaldehyde, terephthalaldehyde and the oligomers of following formulae (III) and (IV):



- 10 in which:

A represents a divalent $-\text{CH}_2-$, $-\text{CH}(\text{OH})-$ or $-\text{CH}_2-\text{O}-\text{CH}_2-$ radical, n varies from 1 to 8,



in which n' varies from 1 to 9.

Hydroxymethylfurfural is particularly preferred.

- 15 In the binder, the ammonium lignosulfonate or the alkali metal or alkaline earth metal salt of lignosulfonic acid generally represents 30 to 95 % of the weight of the mixture consisting of the ammonium lignosulfonate or the alkali metal or alkaline earth metal salt of lignosulfonic acid and the carbonyl compound of formula (I), preferably 40 to 80 % and advantageously 50 to 70 %.
- 20 The binder may further comprise:

- at least one vinyl acetate homopolymer or a copolymer of vinyl acetate and of at least one hydrophobic monomer such as ethylene, propylene, butylene, styrene or vinyl chloride, preferably ethylene. The abovementioned homopolymer and copolymer are generally present in an amount at most equal to 15 parts by weight per 100 parts by weight of ammonium lignosulfonate or of alkali metal or alkaline earth metal salt of lignosulfonic acid and of carbonyl compound of formula (I), and
- at least one polysaccharide, for example a starch. The polysaccharide is generally present in an amount at most equal to 50 parts by weight per 100 parts by weight of ammonium lignosulfonate or of alkali metal or alkaline earth metal salt of lignosulfonic acid and of carbonyl compound (I).

When the binder contains ammonium lignosulfonate, it can further contain at least one compound selected from phosphorus-containing compounds and ammonium sulfate.

- Mention may be made, by way of examples of phosphorus-containing compounds, of alkali metal hypophosphite salts, alkali metal phosphites, alkali metal polyphosphates, alkali metal hydrogen phosphates, phosphoric acids and alkylphosphonic acids, in which the alkali metal is preferably sodium or potassium, and ammonium phosphates, in particular diammonium phosphate. The content of abovementioned compound is at most equal to 20 parts by weight per 100 parts by weight of ammonium lignosulfonate and of carbonyl compound of formula (I).

The binder in accordance with the invention can also comprise the conventional additives below in the following proportions, calculated on the basis of 100 parts by weight of ammonium lignosulfonate or of alkali metal or alkaline earth metal salt of lignosulfonic acid and of carbonyl compound of formula (I):

- 0 to 1 part by weight of silane, preferably 0,1 to 0,5 part,
- 0 to 5 parts by weight of a silicone, of a vegetable oil or of a fluorinated compound, preferably 0,1 to 1 part, and
- 0 to 5 parts of a plasticizing agent, in particular glycerol.

The role of the additives is known and briefly restated: the silane is an agent for coupling between the fibers and the binder, and also acts as antiaging agent; the silicone, the vegetable oil and the fluorinated compound are water repellents, the role of which is to reduce the absorption of water by the mat of mineral fibers.

- 5 The binder is provided in the form of an aqueous solution.

When the binder contains ammonium lignosulfonate, the preferred silane is an aminosilane.

- The binder containing an alkali metal or alkaline earth metal salt of lignosulfonic acid is devoid of nitrogenous compounds, which is advantageous as for this
- 10 reason the gaseous emissions generated during the heat treatment targeted at crosslinking the binder do not contain nitrogen. The treatment of the waste products at the outlet of the oven in industrial plants is thus easier to carry out.

The binder is more particularly intended to be applied to mineral fibers consisting of glass or of a rock, in particular basalt, and preferably of glass, in order to form a mat.

- 15 Conventionally, the binder is deposited on a mat of mineral fibers (formed by the dry route or the wet route) and then the mat is treated at a temperature enabling the crosslinking of the binder, which then becomes infusible. The crosslinking of the binder is generally carried out under conditions comparable to those of a conventional formaldehyde-containing resin, generally at a temperature which varies from 200 to
- 20 220 °C and for a very short period of time, of the order of a few seconds to 1 minute.

The mineral fibers are both mineral filaments and yarns composed of a multitude of mineral filaments bonded together, in particular by a size, and assemblies of such yarns. The mineral filaments and yarns can be continuous or discontinuous.

- Thus, according to a first embodiment, the mat of mineral fibers is composed of
- 25 discontinuous mineral filaments with a length which can reach 150 mm, preferably varying from 20 to 100 mm and advantageously from 50 to 70 mm, and which have a diameter which can vary within wide limits, for example from 5 to 30 μm .

According to a second embodiment, the mat of mineral fibers is composed of mineral yarns.

The mineral yarns can be yarns composed of a multitude of mineral filaments (or base yarns) or assemblies of these base yarns in the form of rovings.

The abovementioned yarns can be zero-twist yarns or twisted yarns (or textile yarns), preferably zero-twist yarns.

- 5 The mineral yarns, in particular glass yarns, are generally cut to a length which can range up to 100 mm, preferably varying from 6 to 30 mm, advantageously from 8 to 20 mm and better still from 10 to 18 mm.

The diameter of the glass filaments constituting the yarns may vary within wide limits, for example from 5 to 30 μm . In the same way, wide variations can occur
10 in the linear density of the yarn, which can range from 34 to 1.500 tex.

The glass involved in forming the filaments can be of any type, for example C, E, R or AR (alkali-resistant). E or C glass is preferred.

Although the invention relates more particularly to mats consisting of mineral fibers, in particular of glass or rock fibers, as already mentioned, it cannot be ruled
15 out that the mat contains a small proportion of organic fibers, for example representing at most 20 % of the weight of the mineral fibers.

The organic fibers can be synthetic fibers or natural fibers.

Mention may be made, as examples of synthetic fibers, of fibers consisting of a polyolefin, for example of polyethylene or polypropylene, of a polyalkylene
20 terephthalate, for example polyethylene terephthalate, or of a polyester.

Mention may be made, as examples of natural fibers, of plant fibers, for example cotton, coconut, sisal, hemp or flax fibers, and animal fibers, for example silk or wool.

The mat of mineral fibers can, if appropriate, be reinforced by continuous fibers which are generally deposited on the device for conveying the mat in the
25 direction of forward progression of the mat and are distributed over all or part of the width of the mat. These fibers are generally deposited in the thickness of the mat, before the application of the binder.

The reinforcing fibers can be mineral and/or organic fibers of the same chemical nature as the abovementioned fibers involved in forming the mat of fibers according to the invention.

Reinforcing fibers made of glass are preferred.

- 5 The mat of mineral fibers generally has a weight per unit area which varies from 10 to 1.100 g/m², preferably from 30 to 350 g/m² and advantageously from 35 to 75 g/m².

The binder generally represents 5 to 40 % by weight of the mat of mineral fibers, preferably 10 to 30 %.

- 10 The mat of mineral fibers in accordance with the present invention is more particularly intended for the production of leaktight bituminous membranes. However, the mat of mineral fibers can also be used in other applications, for example as wall and/or ceiling covering (to be or not to be painted), surface or sealing covering for gypsum board or cement board, surface covering for thermal and/or sound insulation products, such as a mineral wool or a foam intended
- 15 more particularly for the insulation of roofs, or for producing a floor covering, in particular an acoustic underlayer.

The examples which follow make it possible to illustrate the invention without, however, limiting it.

- 20 In these examples, the breaking stress of a 5 cm x 25 cm sample attached at one end to a tensile testing machine and subjected to a continuous elongation of 40 mm/minute is measured. The breaking stress is expressed in N/5 cm.

- The breaking stress is measured after the manufacture (initial) and after the sample has been treated under the following conditions (a) accelerated aging in a heated chamber at 50 °C under 98 % relative humidity for 3 days, (b) treatment in
- 25 water at 80 °C for 10 minutes. The result is expressed by the percentage retention, which is equal to: (breaking stress after treatment/initial breaking stress) x 100.

Examples 1 to 6

Aqueous binders comprising the constituents appearing in table 1 in proportions expressed as parts by weight of solids are prepared. The reference binder (denoted Ref.) is representative of the state of the art described in WO 2012/172252.

The binders are prepared by introducing the different constituents into a
5 container containing water at ambient temperature, with moderate stirring.

The solids content (dry matter) of the binders is equal to 20 %.

Use is made of a mat of filaments of E glass having a diameter of 13 μm and a length of 18 mm. The weight per unit area of the mat is equal to 75 g/m^2 .

The mat is submerged in the binder for 30 seconds and then the excess is
10 removed by suction. The mat is subsequently treated in an oven at 210 °C for 60 seconds. Ultimately, the mat contains 20 % by weight of binder.

The properties of each mat are given in table 1.

Examples 1 to 3 have an initial breaking stress and a breaking stress after accelerated aging which are improved with respect to the Reference.

15 In addition, examples 2, 3 and 6, after treatment with water, have a better breaking stress with respect to the Reference.

Table 1

	Ex. 1	Ex. 2	Ex. 3	Ex. 4	Ex. 5	Ex. 6	Ref.
Composition of the binder							
Ammonium lignosulfonate ⁽¹⁾	95	80	60	-	-	-	60
Sodium lignosulfonate ⁽²⁾	-	-	-	80	-	-	-
Magnesium lignosulfonate ⁽³⁾	-	-	-	-	80	-	-
Calcium lignosulfonate ⁽⁴⁾	-	-	-	-	-	80	-
Hydroxymethylfurfural	5	20	40	20	20	20	-
Glucose	-	-	-	-	-	-	40
Diammonium phosphate	-	-	-	-	-	-	5
Properties of the mat							
Breaking stress (N/5 cm)							
- Initial	40	63	80	42	62	48	30
- After accelerated aging (a)	48	61	77	2	3	41	27
% retention	121	98	96	5	5	86	90
- After treatment with water (b)	0	14	33	0	0	29	11
% retention	0	22	41	0	0	60	37

⁽¹⁾ T11N5, sold by Tembec

⁽²⁾ Arbo N18, sold by Tembec

⁽³⁾ Arbo MGLS, sold by Tembec

5 ⁽⁴⁾ C12, sold by Tembec

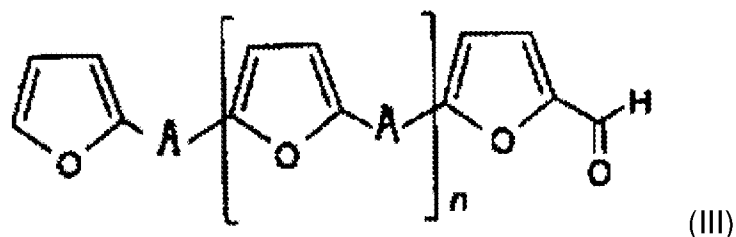
PATENTKRAV

1. Måtte omfattende mineralfibre, **kendetegnet ved, at** fibrene er sammenføjet med et vandigt bindemiddel omfattende

- mindst ét ammoniumligninsulfonat eller et alkalimetal eller

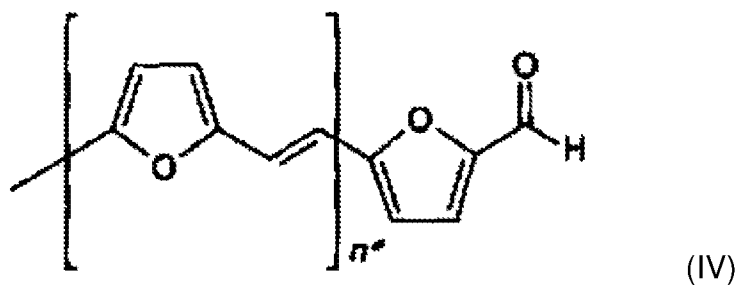
5 jordalkalimetalsalt af ligninsulfonsyre og

- mindst én carbonylforbindelse udvalgt fra gruppen bestående af acetaldehyd, propionaldehyd, dimethoxyethanal, butyraldehyd, navnlig n-butyraldehyd, glyoxal, malonaldehyd, succinaldehyd, glutaraldehyd, 2-hydroxyglutaraldehyd, 3-methylglutaraldehyd, adipaldehyd, suberaldehyd, 10 sebacaldehyd, malealdehyd, fumaraldehyd, poly(acroleiner), dialdehyd-stivelse, furfural (2-furancarboxaldehyd), 5-methylfurfural (2-methyl-5-furancarboxaldehyd), hydroxymethylfurfural (2-hydroxymethyl-5-furancarboxaldehyd), 2,5-furancarboxydialdehyd, vanillin og vanillin-polymerer, navnlig bis-vanillin, cinnamaldehyd og cinnamaldehyd-polymerer, phthalaldehyd, isophthalaldehyd, 15 terephthalaldehyd og oligomererne med følgende formler (III) og (IV):



hvor:

A repræsenterer et divalent $-CH_2-$, $-CH(OH)-$ eller $-CH_2-O-CH_2-$ radikal, n varierer fra 1 til 8,



hvor n' varierer fra 1 til 9,

hvilket bindemiddel er blottet for hydrogeneret sukker og for melamin.

2. Måtte ifølge krav 1, **kendetegnet ved, at** ammoniumligninsulfonatet eller alkalimetallet eller jordalkalimetalsaltet af ligninsulfonsyre repræsenterer 30 til 95 % af vægten af blandingen, der består af ammoniumligninsulfonatet eller
5 alkalimetallet eller jordalkalimetalsaltet af ligninsulfonsyre og carbonylforbindelsen med formlen (I), fortrinsvis 40 til 80 % og med fordel 50 til 70 %.
3. Måtte ifølge krav 1 eller 2, **kendetegnet ved, at** den endvidere omfatter:
 - mindst én vinylacetat-homopolymer eller en copolymer af vinylacetat og af mindst én hydrofob monomer og
10 - mindst ét polysaccharid.
4. Måtte ifølge krav 3, **kendetegnet ved, at** homopolymeren eller copolymeren af vinylacetat og af mindst én hydrofob monomer er til stede i en mængde på højst lig med 15 vægtdele pr. 100 vægtdele ammoniumligninsulfonat eller alkalimetal eller jordalkalimetalsalt af ligninsulfonsyre og carbonylforbindelse med formlen (I).
- 15 5. Måtte ifølge krav 3, **kendetegnet ved, at** polysaccharidet er til stede i en mængde på højst lig med 50 vægtdele pr. 100 vægtdele ammoniumligninsulfonat eller alkalimetal eller jordalkalimetalsalt af ligninsulfonsyre og carbonylforbindelse med formlen (I).
6. Måtte ifølge et af kravene 1 til 5, **kendetegnet ved, at** den indeholder et
20 ammoniumligninsulfonat og mindst én forbindelse udvalgt fra phosphorholdige forbindelser og ammoniumsulfat.
7. Måtte ifølge krav 6, **kendetegnet ved, at** den phosphorholdige forbindelse og ammoniumsulfatet er til stede i en mængde på højst lig med 20 vægtdele pr. 100 vægtdele ammoniumligninsulfonat og carbonylforbindelse med formlen (I).
- 25 8. Måtte ifølge et af kravene 1 til 7, **kendetegnet ved, at** den endvidere omfatter nedenstående konventionelle additiver i følgende andele beregnet på basis af 100 vægtdele ammoniumligninsulfonat eller alkalimetal eller jordalkalimetalsalt af ligninsulfonsyre og carbonylforbindelse med formlen (I):

- 0 til 1 vægt del silan, fortrinsvis 0,1 til 0,5 del,
- 0 til 5 vægtdele af en silikone, af en vegetabilsk olie eller af en fluoreret forbindelse, fortrinsvis 0,1 til 1 del og
- 0 til 5 dele af et blødgøringsmiddel, navnlig glycerol.

5 9. Måtte ifølge et af kravene 1 til 8, **kendetegnet ved, at** den indeholder et alkalimetal eller jordalkalimetalsalt af ligninsulfonsyre **og ved, at** den er blottet for nitrogenforbindelser.

10. Måtte ifølge et af kravene 1 til 9 omfattende sten- eller glasmineralfibre.

10 11. Måtte ifølge et af kravene 1 til 10, **kendetegnet ved, at** den har en vægt pr. arealenhed, der varierer fra 10 til 1100 g/m², fortrinsvis fra 30 til 350 g/m² og med fordel fra 35 til 75 g/m².

12. Måtte ifølge et af kravene 1 til 11, **kendetegnet ved, at** bindemidlet repræsenterer 5 til 40 vægt-% af måtten, fortrinsvis 10 til 30 %.

15 13. Måtte ifølge et af kravene 1 til 12, **kendetegnet ved, at** den indeholder organiske fibre i en andel på højst lig med 20 % af mineralfibrenes vægt.