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SIZING COMPOSITION FOR MINERAL WOOL BASED ON HYDROGENATED SUGAR AND INSULATING PRODUCTS OBTAINED

5 The present invention relates to the field of thermal and/or acoustic insulating products based on mineral wool, in particular of glass or of rock, and on a formaldehyde-free organic binder.

 The invention more particularly relates to a sizing composition capable of crosslinking thermally to form said organic binder, which includes at least one
10 hydrogenated sugar and at least one polyfunctional crosslinking agent, and to the insulating products which result therefrom.

 The manufacture of insulating products based on mineral wool generally comprises a stage of manufacture of the wool itself, which can be carried out by various processes, for example according to the known technique of fiberizing by internal or
15 external centrifugation.

 Internal centrifugation consists in introducing the molten mineral material (glass or rock) into a centrifugal device comprising a multitude of small orifices, the material being projected towards the peripheral wall of the device under the action of the centrifugal force and escaping therefrom in the form of filaments. On leaving the
20 centrifugal device, the filaments are drawn and carried toward a receiving member by a gas stream having a high temperature and a high speed, in order to form a web of fibers (or mineral wool).

 External centrifugation consists, for its part, in pouring out the molten material at the external peripheral surface of rotating members, known as rotors, from where the melt is ejected under the action of the centrifugal force. Means for drawing by gas
25 stream and for collecting on a receiving member are also provided.

 In order to provide for the assembly of the fibers together and to make it possible for the web to have cohesion, a sizing composition comprising a thermosetting resin is projected onto the fibers, on the route between the outlet of the centrifugal
30 device and the receiving member. The web of fibers coated with the size is subjected to a heat treatment, at a temperature generally of greater than 100°C, in order to bring about the polycondensation of the resin and to thus obtain a thermal and/or acoustic insulating product having specific properties, in particular dimensional stability, tensile strength, thickness recovery after compression and homogeneous color.

The sizing composition to be projected onto the mineral wool is generally provided in the form of an aqueous solution including the thermosetting resin and additives, such as a catalyst for the crosslinking of the resin, an adhesion-promoting silane, a dust-preventing mineral oil, and the like. The sizing composition is generally applied to the fibers by spraying.

The properties of the sizing composition depend largely on the characteristics of the resin. From the viewpoint of the application, it is necessary for the sizing composition to exhibit good sprayability and to be able to be deposited at the surface of the fibers in order to efficiently bind them.

The resin has to be stable for a given period of time before being used to form the sizing composition, which composition is generally prepared at the time of use by mixing the resin and the additives mentioned above.

At the regulatory level, it is necessary for the resin to be regarded as non-polluting, that is to say for it to comprise - and for it to generate during the sizing stage or subsequently - as little as possible in the way of compounds which may be harmful to human health or to the environment.

The thermosetting resins most commonly used are phenolic resins belonging to the family of the resols. In addition to their good crosslinkability under the abovementioned thermal conditions, these resins are soluble in water, have a good affinity for mineral fibers, in particular glass fibers, and are relatively inexpensive.

These resols are obtained by condensation of phenol and formaldehyde, in the presence of a basic catalyst, in a formaldehyde/phenol molar ratio of greater than 1, so as to promote the reaction between the phenol and the formaldehyde and to reduce the level of residual phenol in the resin. The condensation reaction between the phenol and the formaldehyde is carried out while limiting the degree of condensation of the monomers, in order to avoid the formation of long, relatively water-insoluble, chains which reduce the dilutability. Consequently, the resin comprises a certain proportion of unreacted monomer, in particular formaldehyde, the presence of which is undesirable because of its known harmful effects.

For this reason, resol-based resins are generally treated with urea, which reacts with the free formaldehyde by trapping it in the form of nonvolatile urea-formaldehyde condensates. The presence of urea in the resin in addition brings a certain economic advantage as a result of its low cost because it is possible to introduce it in a relatively large amount without affecting the operating qualities of the resin, in

particular without harming the mechanical properties of the final product, which significantly lowers the total cost of the resin.

Nevertheless, it has been observed that, under the temperature conditions to which the web is subjected in order to obtain crosslinking of the resin, the urea-formaldehyde condensates are not stable; they decompose with restoration of the formaldehyde and urea (in its turn at least partially decomposed to give ammonia) which are released into the atmosphere of the factory.

Regulations with regard to environmental protection, which are becoming more restrictive, are forcing manufacturers of insulating products to look for solutions which make it possible to further lower the levels of undesirable emissions, in particular of formaldehyde.

Solutions in which resols are replaced in sizing compositions are known and are based on the use of a carboxylic acid polymer, in particular an acrylic acid polymer.

In US 5 340 868, the size comprises a polycarboxylic polymer, a β -hydroxyamide and an at least trifunctional monomeric carboxylic acid.

Provision has been made for sizing compositions comprising a polycarboxylic polymer, a polyol and a catalyst, which catalyst is a phosphorus-comprising catalyst (US 5 318 990, US 5 661 213, US 6 331 350, US 2003/0008978), a fluoroborate (US 5 977 232) or else a cyanamide, a dicyanamide or a cyanoguanidine (US 5 932 689).

A description has also been given of sizing compositions comprising an alkanolamine including at least two hydroxyl groups and a polycarboxylic polymer (US 6 071 994, US 6 099 773, US 6 146 746) in combination with a copolymer (US 6 299 936).

In US 2002/0091185, the polycarboxylic polymer and the polyol are used in amounts such that the ratio of the number of equivalents of OH groups to the number of equivalents of COOH groups varies from 0.6/1 to 0.8/1.

In US 2002/0188055, the sizing composition comprises a polycarboxylic polymer, a polyol and a cationic, amphoteric or nonionic surfactant.

In US 2004/0002567, the sizing composition includes a polycarboxylic polymer, a polyol and a coupling agent of silane type.

In US 2005/0215153, a description is given of a size formed from a prebinder comprising a carboxylic acid polymer and a polyol, and from a dextrin as cobinder.

A description is given, in WO 2006/120523, of a sizing composition which comprises (a) a poly(vinyl alcohol), (b) a polyfunctional crosslinking agent chosen from

nonpolymeric polyacids or their salts, the anhydrides and (c) optionally a catalyst, the (a):(b) ratio by weight varying from 95:5 to 35:65 and the pH being at least equal to 1.25.

In addition, WO 2008/053332 discloses a sizing composition which comprises
5 an adduct (a) of a sugar polymer and (b) of a polyfunctional crosslinking agent chosen from monomeric polyacids or their salts, and their anhydrides, which is obtained under conditions such that the (a):(b) ratio by weight varies from 95:5 to 35:65.

Mention may be made, among the disadvantages exhibited by the sizing
10 compositions which have just been mentioned, of the high costs, a high viscosity, a low pH, which causes problems of acid corrosion, and a high crosslinking temperature.

An aim of the present invention is to provide a sizing composition for insulating products based on mineral wool which is devoid of formaldehyde, thus making it possible to have available an alternative to the sizing compositions based on resols.

Another aim is to provide a sizing composition prepared from natural
15 compounds resulting from renewable sources, in particular plant sources.

Another aim is to provide a sizing composition which makes it possible to manufacture insulating products which are white in color.

In order to achieve these aims, the present invention provides a sizing composition for insulating products based on mineral wool such as defined in claim 1.

20 "Hydrogenated sugar" is understood here to mean all the products resulting from the reduction, in whatever way, of a sugar chosen from monosaccharides, oligosaccharides and polysaccharides which are linear, cyclic or branched, and the mixtures of these products, in particular starch hydrolyzates.

The starch hydrolyzates according to the invention are obtained in a way
25 known per se, for example by enzymatic and/or acid hydrolysis. The degree of hydrolysis of the starch is generally characterized by the dextrose equivalent (DE), defined by the following relationship:

$$DE = 100 \times \left[\frac{\text{number of glycoside bonds cleaved}}{\text{number of glycoside bonds in the starting starch}} \right]$$

30 The DE of starch hydrolyzates varies according to the method of hydrolysis used (type of enzyme(s), for example) and the degree of hydrolysis: the distribution of products with different degrees of polymerization can vary within wide limits.

The preferred starch hydrolyzates have a DE of between 5 and 99 and

advantageously between 10 and 80.

The sugar can be hydrogenated by known methods operating under conditions of high hydrogen pressure and high temperature in the presence of a catalyst chosen from Groups IB, IIB, IVB, VI, VII and VIII of the Periodic Table of the Elements, preferably from the group consisting of nickel, platinum, palladium, cobalt, molybdenum and their mixtures. The preferred catalyst is Raney nickel. The hydrogenation converts the sugar or the mixtures of sugars (starch hydrolyzate) to the corresponding polyols.

Although not being preferred, the hydrogenation can be carried out in the absence of hydrogenation catalyst, in the presence of a source of hydrogen other than hydrogen gas, for example an alkali metal borohydride, such as sodium borohydride.

Mention may be made, as examples of hydrogenated sugars, of glycerol, erythritol, arabitol, xylitol, sorbitol, mannitol, iditol, maltitol, isomaltitol, lactitol, cellobitol, palatinitol, maltotritol and the products from the hydrogenation of starch hydrolyzates, in particular sold by Roquette under the name Polysorb[®]. Preferably, use is made of the products from the hydrogenation of starch hydrolyzates.

The hydrogenated sugar in accordance with the invention has a number-average molar mass of less than 100 000, preferably of less than 50 000, advantageously of less than 5000 and better still of greater than 180.

The hydrogenated sugar in accordance with the invention can comprise reducing sugars in a low proportion which does not exceed 5% by weight (on a dry basis), preferably 1% by weight and better still 0.5% by weight.

The polyfunctional crosslinking agent is capable of reacting with the hydroxyl groups of the hydrogenated sugar under the effect of heat to form acid bonds, which result in a polymeric network being obtained in the final binder. Said polymer network makes it possible to establish bonds at the junction points of the fibers in the mineral wool.

The polyfunctional crosslinking agent is chosen from organic polycarboxylic acids or the salts of these acids, the anhydrides and the polyaldehydes.

“Organic polycarboxylic acid” is understood to mean an organic acid comprising at least two carboxyl functional groups, preferably at most 300 carboxyl functional groups, advantageously at most 70 carboxyl functional groups and better still at most 15 carboxyl functional groups.

The organic polycarboxylic acid can be a nonpolymeric or polymeric acid; it exhibits a number-average molar mass generally of less than or equal to 50 000,

preferably of less than or equal to 10 000 and advantageously of less than or equal to 5000.

The nonpolymeric organic polycarboxylic acid is a saturated or unsaturated and linear or branched alicyclic acid, a cyclic acid or an aromatic acid.

5 The nonpolymeric organic polycarboxylic acid can be a dicarboxylic acid, for example oxalic acid, malonic acid, succinic acid, glutaric acid, adipic acid, pimelic acid, suberic acid, azelaic acid, sebacic acid, malic acid, tartaric acid, tartronic acid, aspartic acid, glutamic acid, fumaric acid, itaconic acid, maleic acid, traumatic acid, camphoric acid, phthalic acid and its derivatives, in particular comprising at least one boron or
10 chlorine atom, tetrahydrophthalic acid and its derivatives, in particular comprising at least one chlorine atom, such as chlorendic acid, isophthalic acid, terephthalic acid, mesaconic acid and citraconic acid; a tricarboxylic acid, for example citric acid, tricarballic acid, 1,2,4-butanetricarboxylic acid, aconitic acid, hemimellitic acid, trimellitic acid and trimesic acid; or a tetracarboxylic acid, for example 1,2,3,4-butane-
15 tetracarboxylic acid and pyromellitic acid.

Mention may be made, as examples of polymeric organic polycarboxylic acids, of homopolymers of unsaturated carboxylic acids, such as (meth)acrylic acid, crotonic acid, isocrotonic acid, maleic acid, cinnamic acid, 2-methylmaleic acid, fumaric acid, itaconic acid, 2-methylitaconic acid, α,β -methylene glutaric acid and unsaturated
20 dicarboxylic acid monoesters, such as C₁-C₁₀ alkyl maleates and fumarates, and copolymers of at least one abovementioned unsaturated carboxylic acid and at least one vinyl monomer, such as unsubstituted styrene or styrene substituted by alkyl, hydroxyl or sulfonyl groups or by a halogen atom, (meth)acrylonitrile, unsubstituted (meth)acrylamide or (meth)acrylamide substituted by C₁-C₁₀ alkyl groups, alkyl
25 (meth)acrylates, in particular methyl (meth)acrylate, ethyl (meth)acrylate, n-butyl (meth)acrylate and isobutyl (meth)acrylate, glycidyl (meth)acrylate, butadiene and a vinyl ester, in particular vinyl acetate.

Preferably, the sizing composition comprises at least one nonpolymeric organic polycarboxylic acid having a number-average molar mass of less than or equal to 1000,
30 preferably of less than or equal to 750 and advantageously of less than or equal to 500, optionally as a mixture with at least one polymeric organic acid.

The polyfunctional crosslinking agent can be an anhydride, in particular maleic anhydride, succinic anhydride or phthalic anhydride. However, the addition of an anhydride to the sizing composition brings about a major fault in the pH, which causes

problems of corrosion of the equipment in the line for the manufacture and hydrolysis of the hydrogenated sugar. The introduction of a base makes it possible to bring the pH of the sizing composition to a value sufficient to prevent these problems. The cost related to the supplementary addition of the base means that the use of anhydride is not preferred.

The polyfunctional crosslinking agent can also be a polyaldehyde.

“Polyaldehyde” is understood to mean an aldehyde comprising at least two aldehyde functional groups.

Preferably, the polyaldehyde is a nonpolymeric dialdehyde, for example glyoxal, glutaraldehyde, 1,6-hexanedial or 1,4-terephthalaldehyde.

Polyaldehydes have a very high reactivity with regard to the hydroxyl groups of the hydrogenated sugar but also to hydroxyl groups in general, which can present disadvantages, in particular a reduction in the stability and/or a pregelling of the sizing composition before the thermal crosslinking treatment. In order to prevent these disadvantages, the aldehyde functional groups of the polyaldehyde are advantageously masked, to prevent the reaction with the constituents present in the sizing composition, before the mineral wool enters the oven. Mention may be made, as example of agent which makes it possible to mask the aldehyde functional groups, of urea and cyclic ureas.

In the sizing composition, the hydrogenated sugar represents at least 30 % of the weight of the mixture composed of hydrogenated sugar and the polyfunctional crosslinking agent, and better still from 30 to 80%.

The sizing composition can additionally comprise an acid or basic catalyst which has in particular the role of adjusting the temperature at which crosslinking begins.

The catalyst can be chosen from Lewis bases and acids, such as clays, colloidal or noncolloidal silica, organic amines, quaternary amines, metal oxides, metal sulfates, metal chlorides, urea sulfates, urea chlorides and catalysts based on silicates.

The catalyst can also be a phosphorus-comprising compound, for example an alkali metal hypophosphite salt, an alkali metal phosphite, an alkali metal polyphosphate, an alkali metal hydrogenphosphate, a phosphoric acid or an alkylphosphonic acid. Preferably, the alkali metal is sodium or potassium.

The catalyst can also be a compound comprising fluorine and boron, for example tetrafluoroboric acid or a salt of this acid, in particular an alkali metal

tetrafluoroborate, such as sodium tetrafluoroborate or potassium tetrafluoroborate, an alkaline earth metal tetrafluoroborate, such as calcium tetrafluoroborate or magnesium tetrafluoroborate, a zinc tetrafluoroborate and an ammonium tetrafluoroborate.

5 Preferably, the catalyst is sodium hypophosphite, sodium phosphite and the mixtures of these compounds.

The amount of catalyst introduced into the sizing composition can represent up to 20% of the weight of the hydrogenated sugar and polyfunctional crosslinking agent, preferably up to 10%, and advantageously is at least equal to 1%.

10 The sizing composition in accordance with the invention can additionally comprise the conventional additives below in the following proportions, calculated on the basis of 100 parts by weight of hydrogenated sugar and polyfunctional crosslinking agent:

- from 0 to 2 parts of silane, in particular an aminosilane,
- from 0 to 20 parts of oil, preferably from 4 to 15 parts,
- 15 - from 0 to 5 parts of a hydrophobic agent, in particular silicone,
- from 0 to 20 parts of a polyol other than the hydrogenated sugars,
- from 0 to 30 parts of urea, preferably from 0 to 20 parts,
- from 0 to 30 parts of an "extender" chosen from lignin derivatives, such as ammonium lignosulfonate (ALS) or sodium lignosulfonate, and
- 20 animal or plant proteins.

The role of the additives is known and is briefly restated: the silane is an agent for coupling between the fibers and the binder, and also acts as anti-aging agent; the oils are dust-preventing and hydrophobic agents;

25 the urea acts as plasticizer and in addition makes it possible to adjust the gel time of the sizing composition, in order to prevent problems of pregelling; the "extender" is an organic filler, soluble or dispersible in the sizing composition, which makes it possible in particular to reduce the cost of the latter.

The polyol added as additive is necessarily different from the hydrogenated sugar; in particular, polyols provided in the form of polymers comprising nonsaccharide units, such as vinyl alcohol polymers and copolymers, are ruled out.

30 The sizing composition is prepared by simple mixing of the abovementioned constituents.

The sizing composition obtained exhibits an acidic pH, of the order of 1 to 4, which is preferably maintained at a value at least equal to 2, advantageously at least

equal to 3, so as to limit problems of corrosion of the line for the manufacture of insulating products based on mineral wool. The pH can be adjusted by adding a base to the sizing composition, in particular a nitrogenous base, such as triethanolamine, or ammonium hydroxide or sodium hydroxide or potassium hydroxide.

5 When the polyfunctional crosslinking agent is a nonpolymeric polyacid, it can be advantageous to subject the sizing composition to a heat treatment, so as to cause a portion of the hydrogenated sugar to react with said polyacid. By virtue of this heat treatment, the content of free polyacids with a low molar mass in the sizing composition is reduced, which has the effect of limiting the gaseous emissions generated during the
10 curing of the size in the oven. The heat treatment is carried out at a temperature which can range from 40 to 130°C.

The sizing composition is intended to be applied to mineral fibers, in particular glass or rock fibers.

Conventionally, the sizing composition is projected onto the mineral fibers at
15 the outlet of the centrifugal device and before they are collected on the receiving member in the form of a web of fibers which is subsequently treated at a temperature which makes possible the crosslinking of the size and the formation of an infusible binder. The crosslinking of the size according to the invention takes place at a temperature comparable to that of a conventional formaldehyde-phenol resin, at a
20 temperature of greater than or equal to 110°C, preferably of greater than or equal to 130°C and advantageously of greater than or equal to 140°C.

The acoustic and/or thermal insulating products obtained from these sized fibers also constitute a subject matter of the present invention.

These products are generally provided in the form of a mat or felt of mineral
25 wool, of glass or of rock, or also of a veil of mineral fibers, also of glass or of rock, intended in particular to form a surface coating on said mat or felt. These products exhibit a particularly advantageously white color.

In addition, the insulating products exhibit great resistance to the growth of
30 microorganisms, in particular of molds, which is due to the nonfermentable nature of the hydrogenated sugar.

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

In these examples, the following are measured:

- On the sizing composition

- the crosslinking start temperature (T_c) and the crosslinking rate (R) by the Dynamic Mechanical Analysis (DMA) method, which makes it possible to characterize the viscoelastic behavior of a polymeric material. The procedure is as follows: a sample of Whatman paper is impregnated with the sizing composition (content of organic solids of the order of 40%) and is then fixed horizontally between two jaws. An oscillating component equipped with a device for measuring the stress as a function of the strain applied is positioned on the upper face of the sample. The device makes it possible to calculate the modulus of elasticity E' . The sample is heated to a temperature varying from 20 to 250°C at a rate of 4°C/min. The curve of variation in the modulus of elasticity E' (in MPa) as a function of the temperature (in °C) is plotted from the measurements, the general appearance of the curve being given in figure 1. The values corresponding to the crosslinking start temperature (T_c), in °C, and the slope corresponding to the crosslinking rate (R), in MPa/°C, are determined on the curve.

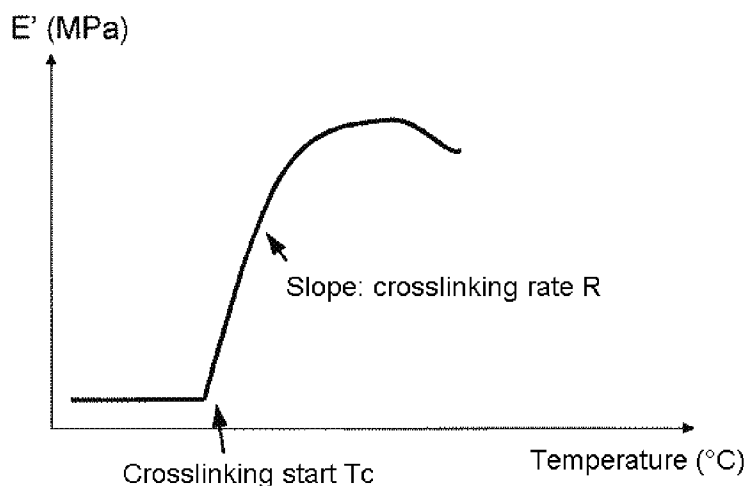


Figure 1

15 ➤ On the insulating product

- the tensile strength according to the standard ASTM C 686-71T on a sample cut out by stamping from the insulating product. The sample has the shape of a torus with a length of 122 mm, a width of 46 mm, a radius of curvature of the cut-out of the outer edge equal to 38 mm and a radius of curvature of the cut-out of the inner edge equal to 12.5 mm.

The sample is positioned between two cylindrical mandrels of a test machine, one of which is movable and is moved at a constant rate. The breaking force F (in gram-

force) of the sample is measured and the tensile strength TS, defined by the ratio of the breaking force F to the weight of the sample, is calculated.

The tensile strength is measured after manufacture (initial tensile strength) and after accelerated aging in an autoclave at a temperature of 105°C under 100% relative humidity for 15 minutes (TS 15).

- the initial thickness of the insulating product and the thickness after compressing for 24 hours and 12 days with a degree of compression (defined as being the ratio of the nominal thickness to the thickness under compression) equal to 5/1. The thickness measurements, expressed as %, make it possible to evaluate the good dimensional behavior of the product.

- the water absorption under the conditions of the standard EN 1609, expressed as kg of water absorbed per m² of insulating product. The insulating products for which the water absorption is less than 1 kg/m² are regarded as having a low short-term water absorption (24 hours) and belong to the "WS" category according to the ACERMI certification.

- the thermal conductivity coefficient λ according to the standard EN 13162, expressed in W/(m × °K).

EXAMPLES 1 TO 4

Sizing compositions are prepared which comprise the constituents appearing in Table 1, expressed as parts by weight.

The hydrogenated sugar is the product of the hydrogenation of a starch hydrolysate (solids content: 70%) which comprises 12% by weight of maltitol and 12% by weight of sorbitol.

The sizing compositions are prepared by successively introducing, into a vessel, the hydrogenated sugar, citric acid and sodium hypophosphite (catalyst) with vigorous stirring until the constituents have completely dissolved.

The properties of the sizing compositions which appear in the following table 1 are evaluated in comparison with a conventional sizing composition including a formaldehyde-phenol resin and urea (Reference) prepared in accordance with example 2, test 1, of WO 01/96254 A1.

Table 1

	Ex. 1	Ex. 2	Ex. 3	Ex. 4	Ref.
Composition					
Hydrogenated sugar (Polysorb® 70/12/12; Roquette)	58	58	58	57	-
Citric acid	42	42	42	31	-
Acrylic homopolymer (Acusol® 445; Röhm & Haas)	-	-	-	12	-
Sodium hypophosphite	5	5	5	5	-
Glycerol	-	10	-	-	-
Silicone	-	-	1	-	-
Properties					
Crosslinking start temperature T_c (°C)	155	153	n.d.	152	151
Crosslinking rate R (MPa/°C)	94	87	n.d.	85	161
Viscosity at 25°C (mPa·s) ⁽¹⁾	9.0	8.7	n.d.	11.0	8.0
Viscosity at 50°C (mPa·s) ⁽¹⁾	6.0	5.9	n.d.	6.7	6.0

⁽¹⁾ solution with a solids content of 40%

n.d.: not determined

The sizing compositions of examples 1 to 4 have similar properties to those of the Reference in terms of crosslinking start temperature (T_c) and of viscosity. The crosslinking rate (R) remains lower than that of the Reference.

The compositions of examples 1 to 4, and also the formaldehyde-phenol resin (Reference), are used to form insulation products based on glass wool.

Glass wool is manufactured by the internal centrifugation technique in which the molten glass composition is converted into fibers by means of a tool, referred to as centrifuging disk, comprising a basket forming a chamber for receiving the molten composition and a peripheral band pierced by a multitude of orifices: the disk is rotated about its vertically positioned axis of symmetry, the composition is ejected through the orifices under the effect of the centrifugal force and the material escaping from the orifices is drawn into fibers with the assistance of a drawing gas stream.

Conventionally, a size spraying ring is positioned beneath the fiberizing disk so

as to uniformly distribute the sizing composition over the glass wool which has just been formed.

The mineral wool, thus sized, is collected on a belt conveyor equipped with internal extraction boxes which hold the mineral wool in the form of a felt or web at the surface of the conveyor. The conveyor subsequently moves through an oven maintained at 290°C where the constituents of the size polymerize to form a binder. The insulating product obtained exhibits a density of 17.5 kg/m³, a thickness of approximately 82 mm immediately after manufacture and a loss on ignition of the order of 5%.

The properties of the insulating products are given in table 2 below.

Table 2

Sizing composition	Ex. 1	Ex. 2	Ex. 3	Ex. 4	Ref.
Tensile strength (gf/g)					
Before aging	327	319	258	333	419
After aging	295	269	188	218	351
Loss (%)	9.80	15.70	27.10	34.53	16.22
Thickness (mm)					
1 hour	83.0	81.7	83.2	79.6	78.9
24 hours	78.8	77.8	80.2	77.5	77.6
12 days	77.1	75.3	77.0	76.6	77.0
Water absorption (kg/m²)	8.6	n.d.	0.4	n.d.	2.6
λ (W/(m × °K))	0.0338	0.0339	0.0337	0.0338	0.0336

n.d.: not determined

The insulating products manufactured with the sizing compositions of examples 1 to 4 exhibit similar properties to the reference product in terms of recovery of thickness after compression for 12 days and of thermal conductivity coefficient λ.

The insulating products treated with the sizing composition of examples 1 and 2 exhibit a good tensile strength after aging (loss equal to 9.8 and 15.7% respectively) in comparison with the Reference.

LIMNINGSSAMMENSÆTNING TIL MINERALULD PÅ BASIS AF HYDROGENERET SUKKER OG OPNÅEDE ISOLERINGSPRODUKTER

PATENTKRAV

1. Limningssammensætning til isoleringsprodukter på basis af mineraluld,
5 navnlig af sten eller glas, **kendetegnet ved, at** den omfatter

- mindst et hydrogeneret sukker,
- mindst et polyfunktionelt tværbindingsmiddel, og
- eventuelt mindst en polyol, der er forskellig fra det hydrogenerede sukker,

10 idet det hydrogenerede sukker udgør mindst 30 % af vægten af blandingen af hydrogeneret sukker og polyfunktionelt tværbindingsmiddel, og idet polyolen er til stede i en mængde, der ikke overskrider 20 vægtdele pr. 100 vægtdele af hydrogeneret sukker og polyfunktionelt tværbindingsmiddel.

2. Sammensætning ifølge krav 1, **kendetegnet ved, at** det hydrogenerede sukker er produktet, der fremkommer ved reduktion af et sukker valgt blandt
15 monosacchariderne, oligosacchariderne og de lineære, cykliske eller forgrenede polysaccharider.

3. Sammensætning ifølge krav 1 eller 2, **kendetegnet ved, at** det hydrogenerede sukker er glycerol, erythritol, arabitol, xylitol, sorbitol, mannitol, iditol, maltitol, isomaltitol, lactitol, cellobitol, palatinitol, maltotritol og produkterne af
20 hydrogeneringen af stivelseshydrolysater.

4. Sammensætning ifølge krav 3, **kendetegnet ved, at** det hydrogenerede sukker er et produkt af hydrogeneringen af stivelseshydrolysater.

5. Sammensætning ifølge et af kravene 1 til 4, **kendetegnet ved, at** det hydrogenerede sukker indeholder reducerende sukkere i et forhold, der ikke overskrider
25 5 vægt-% (tørstof), fortrinsvis 1 % og endnu bedre 0,5 %.

6. Sammensætning ifølge et af kravene 1 til 5, **kendetegnet ved, at** det polyfunktionelle tværbindingsmiddel er valgt blandt de organiske polycarboxylsyrer eller saltene af disse syrer, syreanhydrider og polyaldehyder.

7. Sammensætning ifølge krav 6, **kendetegnet ved, at** den organiske
30 polycarboxylsyre omfatter mindst to carboxylsyrefunktioner, fortrinsvis højst 300 carboxylsyrefunktioner, med fordel højst 70 og endnu bedre højst 15.

8. Sammensætning ifølge krav 6 eller 7, **kendetegnet ved, at** den organiske polycarboxylsyre er valgt blandt de ikke-polymere, forgrenede eller ikke-forgrenede,

mættede eller umættede, alicykliske organiske polycarboxylsyre, de cykliske syrer og de aromatiske syrer.

9. Sammensætning ifølge krav 8, **kendetegnet ved, at** den organiske polycarboxylsyre er valgt blandt dicarboxylsyrerne, navnlig oxalsyre, malonsyre, ravsyre, glutarsyre, adipinsyre, pimelinsyre, suberinsyre, azelainsyre, sebacinsyre, æblesyre, vinsyre, tartronsyre, asparaginsyre, glutaminsyre, fumarsyre, itaconsyre, maleinsyre, traumatinsyre, kamfersyre, phthalsyre og dens derivater, særligt indeholdende mindst et bor- eller chloratom, tetrahydrophthalsyre og dens derivater, særligt indeholdende mindst et chloratom, isophthalsyre, terephthalsyre, mesaconsyre og citraconsyre, tricarboxylsyrerne, navnlig citronsyre, tricarballylsyre, 1,2,4-butantricarboxylsyre, aconitsyre, hemimellitsyre, trimellitsyre og trimesinsyre, og tetracarboxylsyrene, navnlig 1,2,3,4-butantetracarboxylsyre og pyromellitsyre.

10. Sammensætning ifølge krav 6 eller 7, **kendetegnet ved, at** den organiske polycarboxylsyre er valgt blandt de polymere organiske polycarboxylsyrer, særligt homopolymerne af umættet carboxylsyre og copolymererne af mindst en umættet carboxylsyre og mindst en vinylmonomer.

11. Sammensætning ifølge krav 10, **kendetegnet ved, at** den umættede carboxylsyre er (meth)acrylsyre, crotonsyre, isocrotonsyre, maleinsyre, kanelisyre, 2-methylmaleinsyre, fumarsyre, itaconsyre, 2-methylitaconsyre, α,β -methylenglutarsyre og monoesterne af umættede dicarboxylsyrer, og vinylmonomeren er styren substitueret eller ej med alkyl-, hydroxyl- eller sulfonylgrupper eller med et halogenatom, (meth)acrylonitril, (meth)acrylamid substitueret eller ej med C₁-C₁₀-alkylgrupper, alkyl(meth)acrylater, glycidyl(meth)acrylat, butadien og en vinylester.

12. Sammensætning ifølge krav 6, **kendetegnet ved, at** syreanhydridet er maleinsyreanhydrid, ravsyreanhydrid eller phthalsyreanhydrid.

13. Sammensætning ifølge krav 6, **kendetegnet ved, at** polyaldehydet er et ikke-polymert dialdehyd, navnlig glyoxal, glutaraldehyd, 1,6-hexandial eller 1,4-terephthaldialdehyd.

14. Sammensætning ifølge krav 13, **kendetegnet ved, at** polyaldehydets aldehydfunktioner er blokerede med urea eller cykliske ureaer.

15. Sammensætning ifølge et af kravene 1 til 14, **kendetegnet ved, at** det hydrogenerede sukker udgør 30 til 90 % af vægten af blandingen bestående af hydrogeneret sukker og det polyfunktionelle tværbindingmiddel, fortrinsvis 30 til 85 %, og med fordel 30 til 80 %.

16. Sammensætning ifølge et af kravene 1 til 15, **kendetegnet ved**, at den endvidere omfatter en katalysator, der er valgt blandt Lewis-syrer og -baser, phosphorholdige forbindelser og fluor- og borholdige forbindelser.

5 17. Sammensætning ifølge krav 16, **kendetegnet ved**, at katalysatoren udgør op til 20 % af vægten af det hydrogenerede sukker og det polyfunktionelle tværbindingmiddel, fortrinsvis op til 10 % og med fordel mindst 1 %.

18. Sammensætning ifølge et af kravene 1 til 17, **kendetegnet ved**, at den endvidere omfatter følgende additiver i de angivne forhold beregnet på basis af 100 vægtdele hydrogeneret sukker og polyfunktionelt tværbindingmiddel:

- 10
- 0 til 2 dele silan, særligt en aminosilan,
 - 0 til 20 dele olie, fortrinsvis 4 til 15 dele,
 - 0 til 5 dele af et vandskyende middel, særligt en silicone,
 - 0 til 30 dele urea, fortrinsvis 0 til 20 dele,
 - 15 - 0 til 30 dele af et "udstrækningmiddel" valgt blandt ligninderivaterne, såsom ammoniumlignosulfonat eller natriumlignosulfonat, og animalske eller vegetabiliske proteiner.

19. Lyd- og/eller varmeisolerende produkt på basis af mineraluld, navnlig af glas eller sten, som er limet ved hjælp af limningssammensætningen ifølge et af kravene 1 til 18.

20 20. Slør af mineralfibre, navnlig af glas eller sten, som er limet ved hjælp af limningssammensætningen ifølge et af kravene 1 til 18.