

(12)

## Oversættelse af europæisk patentskrift

#### Patent- og Varemærkestyrelsen

(51)Int.Cl.: D 04 H 13/00 (2006.01) C 04 B 30/02 (2006.01) D 04 H 1/64 (2012.01)

C 04 B 111/10 (2006.01) E 04 B 1/76 (2006.01)

D 04 H 1/4209 (2012.01) E 04 B 1/78 (2006.01)

Oversættelsen bekendtgjort den: 2023-06-06 (45)

(80)Dato for Den Europæiske Patentmyndigheds

bekendtgørelse om meddelelse af patentet: 2023-04-26

Europæisk ansøgning nr.: 14184412.6 (86)

(86)Europæisk indleveringsdag: 2007-01-25

Den europæiske ansøgnings publiceringsdag: 2015-01-21 (87)

(62)Stamansøgningsnr: 07704143.2

Designerede stater: AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HU IE IS IT LI LT LU LV MC NL (84)PL PT RO SE SI SK TR

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(54)Benævnelse: Fremgangsmåde til fremstilling af mineralfiberisoleringsprodukt

(56)Fremdragne publikationer:

EP-A-0714754

EP-A- 1 522 642

EP-A- 1 655 400

EP-A- 1 741 726

WO-A-99/27206

WO-A-2006/044302

### **DESCRIPTION**

[0001] This invention relates to a mineral fibre insulating product having a low formaldehyde or formaldehyde free binder.

**[0002]** Industry standard binders used for fibre insulation, for example glass wool and rock wool insulation are based on phenol formaldehyde. Whilst such binders can provide suitable properties to the insulating products there has for some time been a desire to move away from the use of phenol formaldehyde, particularly due to environmental considerations.

[0003] Traditional polyester based binder systems have previously been proposed but have not gained acceptance in the insulation industry, particularly as their strength in holding the mineral fibres together, especially when exposed to moisture or weathering, has been perceived as insufficient.

**[0004]** To date, only one low formaldehyde based mineral insulation binder system has been used on an industrial scale on glass wool insulation; this is based on polyacrylic acid and supplied by Rohm&Haas. Unfortunately, the highly acid nature of these types of binders can cause excessive corrosion of manufacturing plant unless significant investment is made in acid resistant equipment. US patent 5,977,232 discloses a formaldehyde free binder for glass wool insulation based on a polycarboxylic acid. European patent application EP1698598A discloses use of a corrosion meter to try to mitigate problems associated with polycarboxylic acid-based fibreglass binder resins. In addition, whilst the strength of these binders is acceptable for some applications it is not as good as the commonly used phenol formaldehyde based binders.

[0005] It has not been thought possible to provide a formaldehyde free binder system useable on an industrial scale that will confer required characteristics, including strength, to mineral wool insulating products without encountering the difficulties associated with highly acidic liquid binder systems.

**[0006]** WO2006/0044302A discloses polyester binding compositions; EP0714754A discloses a method a making an insulation assembly.

**[0007]** According to one aspect, the present invention provides a method as defined in claim 1. Preferred and/or alternative features are defined in the dependent claims.

[0008] As used herein, the term formaldehyde free means that the composition is substantially free from formaldehyde, preferably does not liberate substantial formaldehyde as a result of drying or curing and/or preferably comprises less than one part per million by weight of formaldehyde.

[0009] Desired characteristics to be conferred by the binder on some mineral wool insulation product can be assessed by measuring Recovered Thickness and/or Ordinary Parting Strength

and/or Weathered Parting Strength. The procedures for measuring these characteristics are set out below. This is particularly the case for low and medium density insulating products, for example, having a density in the range 5-40kg/m<sup>3</sup>, for example roll insulation and/or glass wool thermal insulation for lofts and/or cavity walls.

**[0010]** Desired characteristics for some mineral wool insulation product can be assessed by measuring Ordinary Compression Strength and/or Weathered Compression Strength. The procedures for measuring these characteristics are set out below. This is particularly for higher density insulating products, for example, insulating boards or materials adapted for use as: a fire barrier; a fire protection; cladding for a building; a ceiling tile; a roof board; thermal insulation for high temperature machinery for example, generators, ovens and industrial plant. Such products may be made of rock wool.

**[0011]** The pH of the binder when applied may be substantially neutral or alkaline; this may facilitate handling and avoid significant corrosion and/or environmental problems. Its pH when applied may be: greater than or equal to 7 and/or less than or equal to 10; between 7 and 10; between 8 and 10.

[0012] An important aspect of the invention is the pH of the binder in liquid form when applied to the fibres as this is the form in which the binder will have significant contact with manufacturing equipment as freshly prepared and in a wash water system. The binder may change its pH as it cures; it may become more acidic as it cures. Nevertheless, once cured, the binder has less direct contact with the manufacturing equipment. Furthermore, where the cured binder is substantially insoluble in water, which is preferably the case, there is little risk of acid contamination from the cured binder.

[0013] It is surprising that binders of this type at 15% or less by weight can confer the desired characteristics on the insulating product. This amount of binder is comparable with the binder contents commonly used with phenol formaldehyde based binders. The cured binder content may be 12% or less or 10% or less; it may be within the range of 3-8%, particularly 3.5-6% by weight. The binder content may be determined by loss on ignition. Such binder contents are particularly suitable for low and medium density products. Particularly for higher density products, the cured binder content may be in the range 0.5-5% by weight.

#### [0014] The binder disclosed herein may:

- be based on a reducing sugar; and/or
- be based on reductosis; and/or
- be based on an aldehyde containing sugars/and/or
- include at least one reaction product of a carbohydrate reactant and an amine reactant;
  and/or
- include at least one reaction product of a reducing sugar and an amine reactant; and/or
- include at least one reaction product of a carbohydrate reactant and a polycarboxylic acid ammonium salt reactant; and/or

include at least one reaction product from a Maillard reaction.

[0015] According to the invention, the binder is based on a reducing sugar and includes at least one Maillard reaction product.

**[0016]** The binder may be based on a combination of a polycarboxylic acid, for example citric acid, a sugar, for example dextrose, and a source of ammonia, for example ammonia solution. It may be based on a combination of ammonium citrate and dextrose. Where the binder is based on sugars and/or citric acid and or comprises significant -OH groups, it is particularly surprising that such levels of Weathered Parting Strength can be achieved. It would have been thought that the -OH groups for example in the sugars and/or citric acid would be readily subject to hydrolysis and that the binder would consequently loose significant strength in humid and/or weathering conditions.

**[0017]** The binder may comprise a silicon containing compound, particularly a silane; this may be an amino-substituted compound; it may be a silyl ether; it may facilitate adherence of the binder to the mineral fibres.

[0018] The binder may comprise melanoidins; it may be a thermoset binder; it may be thermally curable.

[0019] The binder may be one of those disclosed in International patent application  $n^{\circ}$  PCT/US2006/028929.

**[0020]** The insulating material is packaged or provided in the form of a package; the package may comprise one or more mineral wool insulating products arranged and/or bound together, for example to facilitate transport; it may comprise an enveloping film, for example of a plastics material. The package may comprise or consist of a roll of insulating material or an assembly of individual slabs of insulating material.

[0021] The insulating material, particularly when it is a low or medium density product, may have

- a nominal thickness in the range 60-260mm; and/or
- a thermal resistance R of R≥ 3 m²K/W, preferably R≥ 4 m²K/W at a thickness or 200mm;
  and/or
- a density in the range 5-40 kg/m<sup>3</sup>, particularly 5-18 kg/m<sup>3</sup> or 7-12 kg/m<sup>3</sup>, for example for low density roll products.

[0022] The insulating material, particularly when it is an insulating board or a higher density product, may have

- a nominal thickness in the range 20 to 200 mm; and/or
- a thermal resistance R of R≥1.7 m<sup>2</sup>K/W, preferably R≥2 m<sup>2</sup>K/W at a thickness or 100mm; and/or
- a density in the range 100 to 200 kg/m<sup>3</sup>, particularly 130 to 190 kg/m<sup>3</sup>.

[0023] The mineral fibres may be glass wool or rock wool; the fibres may have an average diameter between 2 and 9 microns or be microfibres of smaller diameter; they may have an average length between 8 and 80mm.

[0024] The mineral fibres may be crimped.

[0025] According to a further aspect, the present invention provides a mineral fibre insulating material having at least one of the following features:

- · the insulating material having cut edges;
- the insulating material having a facing provided on at least one of its major surface, for example comprising a moisture penetration barrier and/or a Kraft paper and/or an aluminium foil and/or a plastics layer and/or a laminate sheet comprising a plurality of individual layers and/or a woven or non-woven fabric; a facing may be provided on each major surface of the insulating material;
- the insulating material being a packaged insulating material held under compression for example by one or more packaging components, for example by an enveloping packaging film; the insulating material may be compressed to 80% or less of its noncompressed thickness;
- the insulating material being in the form of pipe insulation having a length of greater than 30cm; the cross-section may be substantially annular;
- the insulating material being in the form of a compressed roll of material;
- the insulating material being in the form of a compressed slab of material;
- the insulating material being a roll or slab having a length of greater than or equal to 1 m, preferably greater than or equal to 2m;
- the insulating material being a roll or slab having a width of greater than or equal to 0.3 m, preferably greater than or equal to 0.5 m;
- the insulating material having a nominal thickness of at least 45 mm, preferably at least 50 mm, and a density in the range 5-40 kg/m<sup>3</sup>

[0026] The insulating material may have any combination of these features; these features may be combined with other features and/or aspects described herein.

[0027] Non-limiting examples of the invention are described below with reference to Fig 1

which shows the form of samples used for testing parting strength.

[0028] An aqueous binder was prepared by mixing together:

	Approximate % by weight		
Powdered dextrose monohydrate	12.9%		
Powdered anhydrous citric acid	2.3%		
28% aqueous ammonia	2.6%		
Silane A-1100	0.05%		
water	82.1%		

[0029] This binder was used in the manufacture of a fibre glass insulating product on a standard manufacturing line, the binder being sprayed onto glass fibres just after fiberising using internal spinners and the coated fibres being collected, assembled in to a mat and cured in the usual way.

[0030] The binder had a pH of about 8 when applied to the glass fibres.

[0031] The cured glass fibre insulating product had:

- a binder content of about 5% by weight as determined by loss on ignition
- a thickness of about 150 mm
- a density of about 9 kg/m<sup>3</sup>

[0032] This is suitable as a low density residential roll insulation product; it was packaged in a roll under compression.

[0033] Desired characteristics and results achieved are set out in Table 1:

Table 1

	Units	Acceptance limit			Most preferred	Result achieved
Recovered Thickness	:	≥95	≥100	≥110	≥120	103
Ordinary Parting Strength	g/g	≥95	≥100	≥150	≥200	122
Weathered Parting strength	g/g	≥75	≥80	≥100	≥150	112

#### <u>Testing of Recovered Thickness:</u>

**[0034]** Recovered Thickness is tested and measured in accordance with Annex A of British standard BS EN 823: 1995 (incorporated herein by reference) and expressed as a % of the nominal or announced thickness for the product measured.

#### <u>Testing of Ordinary Parting Strength and Weathered Parting Strength:</u>

**[0035]** Parting strength is a measure of the tensile strength of mineral fibre mats determined by placing an O shaped sample over cylindrical jaws, separating the jaws and measuring the load to break the fibres. Although it can be measured in Newtons per gram, the parting strength is expressed in grams/gram being the total breaking load of six test specimens divided by their total weight.

[0036] The test is carried out on mineral fibre mats as received for testing (Ordinary Parting Strength) and after an accelerated weathering test as explained below (Weathered Parting Strength).

[0037] A first set of six samples of the form and dimensions shown in Fig 1 are cut from the mineral fibre mat to be tested; the long axis of the samples should be parallel to the conveyor direction and the samples should be taken across the full width of the mineral mat. A second set of six samples is then taken in the same way. The dimensions in Fig 1 are in mm.

[0038] The total weight of the first group of six samples W1 in grams is recorded.

**[0039]** The total weight of the second group of six samples W2 in grams is recorded; these samples are then placed in a preheated autoclave and conditioned on a wire mesh shelf away from the bottom of the chamber under wet steam at 35kN/m<sup>2</sup> for one hour. They are then removed, dried in an oven at 100°C for five minutes and tested immediately for parting strength.

**[0040]** To test the parting strength, each sample is mounted in turn on the jaws of the tensile strength machine and the maximum breaking load in grams or Newtons is recorded. If the breaking load is measured in Newtons it is converted to grams by multiplying it by 101.9. Six results in grams are obtained for each set of samples: G1 G2 G3 G4 G5 and G6 for the first set of samples and G7 G8 G9 G10 G11 and G12 for the second set of samples.

**[0041]** The Ordinary Parting Strength is calculated from the first set of samples using the formula Ordinary Parting Strength = (G1+G2+G3+G4+G5+G6)/W1.

[0042] The Weathered Parting Strength is calculated from the second set of samples using the

formula Weathered Parting Strength = (G7+G8+G9+G10+G11+G12)/W2.

[0043] In another example, an aqueous binder was prepared by mixing together:

	Approximate % by weight		
Powdered dextrose monohydrate	19.1%		
Powdered anhydrous citric acid	3.4%		
28% aqueous ammonia	2.6%		
Silane A-1100	0.07%		
Water	73.5%		

**[0044]** This binder was used in the manufacture of a rock wool roof board on a standard manufacturing line, the binder being sprayed onto the fibres just after fiberising and the coated fibres being collected, assembled in to a mat, compressed and cured in the usual way.

[0045] The cured roof board had:

- a binder content of about 3% by weight as determined by loss on ignition
- a thickness of about 80 mm
- a density of about 150 kg/m<sup>3</sup>

[0046] It was packaged as part of a stack of insulation boards.

[0047] Desired characteristics and results achieved are set out in Table 2:

Table 2

	Units	Acceptance limit	Preferred		Most preferred	Result achieved
Ordinary Compression Strength	kPa	≥60	≥70	≥80	≥90	72.3
Weathered Compression Strength	kPa	≥25	≥30	≥40	≥50	54.6

#### <u>Testing of Ordinary Compression Strength and Weathered Compression Strength:</u>

[0048] Ordinary Compression Strength is measured according to British Standard BS EN 826 : 1996 (incorporated herein by reference).

**[0049]** Weathered Compression Strength is measured according to British Standard BS EN 826: 1996 on samples that have been subjected to the following accelerated weathering procedure: samples are cut to size and then placed in a preheated autoclave and conditioned on a wire mesh shelf away from the bottom of the chamber under wet steam at 35kN/m² for one hour. They are then removed, dried in an oven at 100°C for five minutes and tested immediately for compression strength.

**[0050]** In both cases, compression strength is determined in the direction of the thickness of the product; the dimensions of face of the samples in contact with the compression test apparatus are preferably 200mm x 200 mm.

## REFERENCES CITED IN THE DESCRIPTION

Cited references

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#### Patent documents cited in the description

- <u>US5977232A</u> [0004]
- EP1698598A [0004]
- WO20060044302A [0006]
- EP0714754A [0006]
- US2006028929W [0019]

#### **Patentkrav**

- 1. Fremgangsmåde til fremstilling af et pakket mineralfiberisoleringsmateriale valgt fra gruppen bestående af:
  - (i) materiale med

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- a) en genvindingstykkelse på mindst 95%; og
- b) en normalseparationsstyrke på mindst 95 g/g; og
- c) en forvitringsseparationsstyrke på mindst 75 g/g; og
- (ii) materiale med
  - a) en normalkompressionsstyrke på mindst 60 kPa; og
- 10
- b) en forvitringskompressionsstyrke på mindst 25 kPa; omfattende trinnene
  - i) påføring af et organisk, formaldehydfrit bindemiddel i vandig opløsning på mineralfibrene med en pH på mere end 5, hvor bindemidlet er baseret på et reducerende sukker og inkluderer mindst et Maillard-
- 15 reaktionsprodukt;
  - ii) hærdning af produktet således at det indeholder en mængde på mindre end 15 vægtprocent bindemiddel; og
  - iii) pakning af isoleringsmaterialet.
- 20 **2.** Fremgangsmåden ifølge krav 1, hvor
  - a) mineralfiberisoleringsmaterialet har en genvindingstykkelse på mindst omtrent 95%;
  - b) mineralfiberisoleringsmaterialet har en normalseparationsstyrke på mindst omtrent 95 g/g; og
- 25 c) mineralfiberisoleringsmaterialet har en forvitringsseparationsstyrke på mindst omtrent 75 g/g.
  - 3. Fremgangsmåden ifølge krav 2, hvor genvindingstykkelsen er mindst omtrent 100%.

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4. Fremgangsmåden ifølge et hvilket som helst af krav 2 eller 3, hvor normalseparationsstyrken er mindst omtrent 100 g/g.

- **5.** Fremgangsmåden ifølge et hvilket som helst af kravene 2 til 4, hvor forvitringsseparationsstyrken er mindst omtrent 80 g/g.
- 6. Fremgangsmåden ifølge et hvilket som helst af kravene 2 til 5, hvor
  mineralfiberisoleringsmaterialet er et rullet produkt med en massefylde mellem omtrent 5 og omtrent 40 kg/m³, og mineralfiberisoleringsmaterialet pakkes under kompression.
- **7.** Fremgangsmåde ifølge krav 1, hvor mineralfiberisoleringsmaterialet har en massefylde i området 100 til 200 kg/m³, og hvor
  - a) mineralfiberisoleringsmaterialet har en normalkompressionsstyrke på mindst omtrent 60 kPa,
  - b) mineralfiberisoleringsmaterialet har en forvitringskompressionsstyrke på mindst omtrent 25 kPa.

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- **8.** Fremgangsmåden ifølge krav 7, hvor normalkompressionsstyrken er mindst omtrent 70 kPa.
- **9.** Fremgangsmåden ifølge et hvilket som helst af kravene 7 til 8, hvor 20 forvitringskompressionsstyrken er mindst omtrent 30 kPa.
  - **10.** Fremgangsmåden ifølge et hvilket som helst foregående krav, hvor det organiske, formaldehydfrie bindemiddel er en vandig opløsning med en pH på mindre end omtrent 11, når det påføres mineralfibrene.

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- **11.** Fremgangsmåden ifølge et hvilket som helst foregående krav, hvor det organiske, formaldehydfrie bindemiddel er en vandig opløsning med en pH mellem omtrent 6 og omtrent 10, når det påføres mineralfibrene.
- 30 **12.** Fremgangsmåden ifølge et hvilket som helst foregående krav, hvor bindemiddelindholdet er mellem omtrent 2 vægtprocent og omtrent 8 vægtprocent af mineralfiberisoleringsmaterialet.

**13.** Fremgangsmåden ifølge et hvilket som helst foregående krav, hvor den vandige opløsning omfatter citronsyre, ammoniak og dextrose.

# **DRAWINGS**

Fig 1

