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(54) Title: ADDITIVE FOR MINERALLY SETTING AQUEOUS MORTAR COMPOSITIONS

(57) Abstract: An additive for minerally setting aqueous mortar compositions, wherein the additive consists essentially of a) a water-redispersible poly-mer powder, and b) a cellulose-ether powder, and use of the additive in the preparation of a minerally setting aqueous mortar composition, and the minerally setting mortar composition obtained with the additive.

Additive for minerally setting aqueous mortar compositions

The invention relates to an additive for minerally setting aqueous mortar compositions, the use of the additive in the preparation of a minerally setting aqueous mortar composition, and the minerally setting mortar composition obtained with the additive.

Throughout the first third of the 20th century any kind of minerally setting aqueous mortar compositions were mixed from several raw materials immediately for use on the construction site. In steel troughs or motorized mixing drums first the dry compounds such as mineral binder, for example, cement, and filler for example sand, and further additives, for example thickener, were mixed and then brought to the desired consistency by the addition of water.

Since then, the quality requirements for building materials have drastically increased and today industrially produced dry mortars are used for the preparation of building materials. Dry mortar is the term for mortars whose dry components were mixed in the factory of a building materials manufacturer based on fixed instructions.

The basic ingredients of such prefabricated dry mortars are mineral binders such as cement, gypsum or lime, and fillers such as sand, thickeners for the adjustment of rheology, accelerators and retarders for adjusting workability, and water-redispersible polymer powders for the improvement of mechanical properties of the hardened mortar layer.

For improvement of such dry mortar compositions, further additives are incorporated into the dry mortar composition: WO 03/106369 A1 describes an additive for improving rheological flow behavior and which comprises a water-soluble ether of cellulose and a polycarboxylate. EP 2297060 B1 discloses a rheology influencing additive which contains a combination of dispersing agents and stabilizers. WO 2011/073224 A1 describes the use of an additive, containing a plasticizer, to improve the adhesion of a mortar to a building substrate. WO 2011/098412 A1 discloses the hydrophobization of mortars with a rosin additive.

Such dry mortars are delivered in bags or loose (in silos) to the construction site. Before processing the dry mortar is mixed without further ingredients with only a defined amount of water. The advantages of dry-mortars are the factory backed product quality, and the high storage stability as no freezable liquid components are included.

The precondition for obtaining all the above-mentioned advantages of the application of dry mortars, is a highly developed infrastructure, which ensures a reliable transportation of the dry mortars from the factory to the building site, and which ensures a regionwide distribution of such products via hardware stores and specialized retailers. In regions with a lower developed infrastructure, cement, sand and water (the basic ingredients for mortars) are immediately available in local stores, but not dry mortars. In consequence, building materials which fulfill the quality standards obtainable with dry mortar compositions are not available.

Taking this background into account, the object was to provide means which enable the preparation of high quality building materials, starting with universally available basic materials like cement, sand and water, and achieving mortars which show a workability and a mechanical strength of the hardened mortar, which is similar to those obtained with factory made dry mortars.

Surprisingly, it was found that the object can be attained by means of an additive for minerally setting aqueous mortar compositions, wherein the additive consists essentially of

a) a water-redispersible polymer powder, and

b) a cellulose-ether powder.

Water-redispersible polymer powders are generally obtained by drying the corresponding aqueous polymer dispersions in the presence of a drying aid (generally a protective colloid) and of an antiblocking agent. Because of the protective colloid content, irreversible adhesion of the polymer particles is firstly prevented in the course of the drying operation, since the polymer particles are encased by the water-soluble protective colloid particles. Secondly, this protective colloid matrix, which redissolves when the polymer powder is dispersed in water, has the effect that the polymer particles are again present in the aqueous redispersion with the

particle size of the starting dispersion (TIZ-Fachberichte, 1985, Vol. 109 (9), 698).

The water-redispersible polymer powders are based in general on polymers of one or more ethylenically unsaturated monomers. Preferred ethylenically unsaturated monomers are selected from the group encompassing vinyl esters, (meth)acrylic esters, vinylaromatics, olefins, 1,3-dienes, and vinyl halides, and optionally further monomers copolymerizable therewith.

Suitable vinyl esters are, for example, those of carboxylic acids having 1 to 15 C atoms. Suitable monomers from the group of acrylic esters or methacrylic esters are, for example, esters of unbranched or branched alcohols having 1 to 15 C atoms. Preferred vinylaromatics are styrene, methylstyrene, and vinyltoluene. A preferred vinyl halide is vinyl chloride. The preferred olefins are ethylene and propylene, and the preferred dienes are 1,3-butadiene and isoprene.

Optionally it is also possible for 0,1 to 10 % by weight of auxiliary monomers to be copolymerized, based on the total weight of the monomer mixture. Preference is given to using 0,1 to 5 % by weight of auxiliary monomers. Examples of auxiliary monomers are ethylenically unsaturated monocarboxylic and dicarboxylic acids, ethylenically unsaturated carboxamides and carbonitriles, and also maleic anhydride, ethylenically unsaturated sulfonic acids and their salts. Other examples for auxiliary monomers are pre-crosslinking comonomers such as polyethylenically unsaturated comonomers, or post-crosslinking comonomers, examples being N-methylolacrylamide (NMA), N-methylolmethacrylamide. Also suitable are epoxide-functional comonomers such as glycidyl methacrylate and silicon-functional comonomers, such methacryloyloxypropyltrialkoxysilanes, vinyltrialkoxysilanes

Examples of suitable homo- and copolymers are vinyl acetate homopolymers, copolymers of vinyl acetate with ethylene, copolymers of vinyl acetate with ethylene and one or more further vinyl esters, copolymers of vinyl acetate with one or more further vinyl esters, copolymers of vinyl acetate with ethylene and acrylic esters, copolymers of vinyl acetate with ethylene and vinyl chloride, copolymers of vinyl chloride and ethylene and optionally one or more further vinyl

esters, styrene-acrylic ester copolymers, styrene-1,3-butadiene copolymers, where the polymers may each also contain the auxiliary monomers mentioned in the amounts mentioned, and the figures in % by weight add up to 100 % by weight in each case.

Preference is given to vinyl acetate homopolymers; copolymers of vinyl acetate with 1 to 50% by weight of one or more further comonomers from the group of the vinyl esters having 1 to 12 carbon atoms in the carboxyl radical, such as vinyl propionate, vinyl laurate, vinyl esters of alpha-branched carboxylic acids having 5 to 12 carbon atoms, such as VeoVa9^R, VeoVa10^R; copolymers of vinyl acetate with 1 to 40% by weight of ethylene; copolymers of vinyl acetate with 1 to 40% by weight of ethylene and 1 to 50% by weight of one or more further comonomers from the group of the vinyl esters having 1 to 12 carbon atoms in the carboxyl radical, such as vinyl propionate, vinyl laurate, vinyl esters of alpha-branched carboxylic acids having 5 to 12 carbon atoms, such as VeoVa9^R, VeoVa10^R; copolymers of vinyl acetate, 1 to 40% by weight of ethylene and preferably 1 to 60% by weight of (meth)acrylic esters of unbranched or branched alcohols having 1 to 15 carbon atoms, especially methyl methacrylate, methyl acrylate, n-butyl acrylate or 2-ethylhexyl acrylate; copolymers with 30 to 75% by weight of vinyl acetate, 1 to 30% by weight of vinyl laurate or vinyl esters of an alpha-branched carboxylic acid having 5 to 12 carbon atoms, and 1 to 30% by weight of (meth)acrylic esters of unbranched or branched alcohols having 1 to 15 carbon atoms, especially methyl methacrylate, methyl acrylate, n-butyl acrylate or 2-ethylhexyl acrylate, which may also contain 1 to 40% by weight of ethylene; copolymers with one or more vinyl esters having 1 to 12 carbon atoms in the carboxyl radical, such as vinyl acetate, vinyl propionate, vinyl laurate, vinyl esters of alpha-branched carboxylic acids having 5 to 12 carbon atoms, such as VeoVa9^R, VeoVa10^R, 1 to 40% by weight of ethylene and 1 to 60% by weight of vinyl chloride; where the polymers may each also contain the auxiliary monomers mentioned in the amounts mentioned, and the figures in % by weight add up to 100 % by weight in each case.

The monomer selection and the selection of the weight fractions of the comonomers are preferably made here so as to result in a glass transition temperature, Tg, of 50°C to +50°C, more preferably –40°C to +40°C, most preferably -20°C to

+30°C. The glass transition temperature Tg of the polymers can be determined in a known way by means of Differential Scanning Calorimetry (DSC). The Tg may also be calculated approximately in advance using the Fox equation. According to Fox T. G., Bull. Am. Physics Soc. 1, 3, page 123 (1956) the following is the case: $1/Tg = x1/Tg1 + x2/Tg2 + ... + xn/Tgn$, where xn stands for the mass fraction (wt%/100) of the monomer n, and Tgn is the glass transition temperature, in kelvins, of the homopolymer of the monomer n. Tg values for homopolymers are listed in Polymer Handbook 2nd Edition, J. Wiley & Sons, New York (1975).

The polymers are prepared generally in an aqueous medium and preferably by the emulsion or suspension polymerization process, as described for example in WO 2010/057888 A1. The polymers in that case are obtained in the form of aqueous dispersions. In the polymerization it is possible to use the customary protective colloids and/or emulsifiers, as described in WO 2010/057888 A1. As protective colloids preference is given to partially hydrolyzed or fully hydrolyzed polyvinyl alcohols, having a degree of hydrolysis of 80 to 100 mol-%, more particularly to partially hydrolyzed polyvinyl alcohols having a degree of hydrolysis of 80 to 94 mol-% and a Höppler viscosity, in 4% strength aqueous solution, of 1 to 30 mPas (Höppler method at 20°C, DIN 53015). The stated protective colloids can be obtained by methods known to the skilled person, and are added generally in an amount of in total 1 to 20 % by weight, based on the total weight of the monomers, in the polymerization.

The polymers in the form of aqueous dispersions may be converted by spray-drying into corresponding powders that are redispersible in water, as described in WO 2010/057888 A1, for example. In that case it is usual to add a drying aid in a total amount of 3 to 30 % by weight, based on the polymeric constituents of the dispersion. A preferred drying aid are the abovementioned polyvinyl alcohols. Additionally antiblocking agent maybe added during or after the drying step.

The water-redispersible polymer powders are also commercially available. For example Vinnapas^{R} dispersion powders of Wacker Chemie AG.

One or more cellulose ethers can be used. They are preferably selected from the group of alkyl cellulose ethers, hydroxyalkyl cellulose ethers, carboxyalkyl cellu-

lose ethers, and of mixed ethers with at least two different substituents from the group of alkyl, hydroxyalkyl, carboxyalkyl with in each case alkyl groups with 1 to 10 carbon atoms. The alkyl groups are preferably methyl, ethyl and/or propyl groups and the hydroxyalkyl groups are preferably hydroxymethyl, hydroxyethyl and/or hydroxypropyl groups. The Brookfield viscosity of the cellulose ether measured at 20 rpm and as a 2 % aqueous solution at 20°C is in general 100 to 100000 mPas, preferably 1000 to 75000 mPas and in particularly preferred 5000 to 50000 mPas.

Particularly preferred cellulose ethers are methyl cellulose (MC), ethyl cellulose (EC), methyl hydroxyethyl cellulose (MHEC), ethyl hydroxyethyl cellulose (EHEC), methyl ethyl hydroxyethyl cellulose (MEHEC), hydroxyethyl cellulose (HEC), hydroxypropyl cellulose (HPC), methyl hydroxypropyl cellulose (MHPC), hydroxypropyl methyl cellulose (HPMC), hydroxyethyl methyl cellulose (HEMC), carboxymethyl cellulose (CMC).

The cellulose ethers are commercially available. For example Berocoll^{R} of AkzoNobel NV, Walocel^{R} of Wolff Cellulosics GmbH & Co. KG, Methocel^{R} or Cellosize^{R} of Dow Chemical Company, Tylose^{R} of SE Tylose GmbH&Co.KG, Culminal^{R} or Natrosol^{R} of Ashland Inc..

Preferably the additive consists essentially from 50 to 95 % by weight of one or more water-redispersible polymer powders a), and from 5 to 50 % by weight of one or more cellulose-ether powder b), based on the dry weight of the additive composition. More preferred the additive consists essentially from 50 to 75 % by weight of one or more water-redispersible polymer powders a), and from 25 to 50 % by weight of one or more cellulose-ether powder b), based on the dry weight of the additive composition. The additive may also include further ingredients in minor amounts from 1 to 10 % by weight, based on the dry weight of the additive composition in each case, and the figures in % by weight add up to 100 % by weight in each case. Examples for further ingredients are fillers such as sand, quartz powder and/or calcium carbonate, and/or setting retarders such as saccharose, and/or setting accelerators such as calcium formate, and/or defoamer, and/or water reppellants such as fatty acid esters, and/or stabilizers such as polyvinyl alcohol.

For the preparation of the additive the components a) and b) and optionally further ingredients are mixed with one another in powder form using commercially available mixers. If one of the further ingredients is in liquid form the components a) and b) are mixed as powders with the further ingredient in liquid form. Liquid ingredients can also be applied absorbed on a solid, powdery carrier material.

The additive according to the present invention makes it possible to obtain high quality aqueous mortars by starting with universally available basic materials such as cement, sand and water. To achieve this goal even in the case of small job-sites, and additionally for simplifying dosing of the additive, the additive is packed in relatively small portions. Preferably the additive is distributed in portions of 50 g to 10 kg, more preferred in portions of 50 g to 1 kg. In smaller packaging size the portions of the additive maybe packed in sachets. For bigger packaging size the portions of the additive maybe packed in buckets.

For the preparation of the minerally setting aqueous mortar composition modified with the additive according to the invention, in a first step the additive is admixed with the mineral binder and the filler in a dry mixing step. In the second step the additive-containing dry mix is admixed with water for obtaining the minerally setting aqueous mortar composition.

Preferably from 1 to 10 % by weight, more preferred from 1 to 5 % by weight of the additive, in each case based on the dry weight of the mineral binder, is added to the mortar composition.

As preferred mineral binders, which are universally available, can be used cement, particularly Portland cement and/or calcium-aluminate cement, and/or gypsum, and/or lime, and/or puzzolanes such as volcanic slag and/or volcanic tuft and/or fly ash and/or blast-furnace slag.

As preferred filler materials, which are universally available, can be used for example sand, and/or chalks, and/or clay.

The minerally setting aqueous mortar composition modified with the additive comprises in general from 5 to 80 parts by weight of mineral binder, and from 20 to 95 parts by weight of filler, and water in an amount for reaching the desired consistency of the aqueous mortar. In the given framework the compositions of the aqueous mortar depends on the application of the mortar. For example, the minerally setting aqueous mortar composition modified with the additive can be formulated as plaster, or as flow bed mortar, or as repair mortar, or as tile grout, or as floor screed.

Examples:

A tile adhesive obtained with a premixed dry mortar mixture (comparison example 1) was compared with a tile adhesive obtained by mixing cement, sand, the inventive additive and water (example 2)

Comparison example 1:

Dry mortar mixture with
300,0g cement (OPC CEM 42.5,MilkeZement GmbH&Co. KG)
686,5 g quartzsand (Quarzsand F31/F36,1:1, Quarzwerke GmbH)
3,5 g cellulose ether (TyloseMB3003P4, ShinEtsu SE)
5,0 g redispersible powder (polyvinyl-alcohol-stabilized vinyl acetate ethylene copolymer, Vinna pas 4023N of Wacker Chemie AG)
5,0 g Calciumformiat (Fluka GmbH)

The dry mortar formulation was mixed with 265 g water to form a tile adhesive.

Example 2:

300.0 g cement (OPC CEM 42.5, MilkeZement GmbH&Co. KG)
686,5 g quartz sand (Quarzsand F31/F36,1:1, Quarzwerke GmbH)
8,5 g of additive

The additive was obtained by mixing 3,5 g cellulose ether (Tylose MB3003P4) and 5,0 g redispersible powder (Vinnapas 4023N).

The cement, the sand and the additive were mixed, and 265 g water was admixed to form a tile adhesive.

Determination of the tensile bond strengths:

For testing, the tile adhesive compositions of comparison example 1 and example 2 were each applied to concrete paving slabs using a 5 mm toothed trowel. Then 5 tiles (5 x 5 cm) were laid on each slab and weighted down for 30 seconds using a 2 kg weight.
The tensile bond strengths were tested in each case after the following storage conditions:
Storage under standard conditions (SC):
28 days at 23°C and 50% humidity.
Wet storage (WS):
7 days of storage under standard conditions followed by 21 days of storage at 20°C in water.

The tensile bond strengths were determined in accordance with DIN EN 1348, in each case following the abovementioned storage, using a pulloff device from Herion, with a load increase rate of 250 N/s. The measurements, in N/mm^2, represent mean values of 5 measurements.

Example	Tensile Strength (SC)	Tensile Strength (WS)
Comp. Example 1	1,17 N/mm^2	0,97 N/mm^2
Example 2	1,16 N/mm^2	0,93 N/mm^2

Claims:

1. Additive for minerally setting aqueous mortar compositions, wherein the additive consists essentially of
a) a water-redispersible polymer powder, and
b) a cellulose-ether powder.

2. Additive according to claim 1, wherein the water-redispersible polymer powder comprises a polymer of one or more ethylenically unsaturated monomers selected from the group comprising vinyl esters, (meth)acrylic esters, vinylaromatics, olefins, 1,3-dienes, and vinyl halides, and optionally further monomers copolymerizable therewith.

3. Additive according to claim 1, wherein the water-redispersible polymer powder comprises a polymer selected from the group comprising vinyl acetate homopolymers, copolymers of vinyl acetate with ethylene, copolymers of vinyl acetate with ethylene and one or more further vinyl esters, copolymers of vinyl acetate with one or more further vinyl esters, copolymers of vinyl acetate with ethylene and acrylic esters, copolymers of vinyl acetate with ethylene and vinyl chloride, copolymers of vinyl chloride and ethylene and optionally one or more further vinyl esters, styrene-acrylic ester copolymers, styrene-1,3-butadiene copolymers, in each case optionally further monomers copolymerized therewith.

4. Additive according to one of the claims 1 to 3, wherein the cellulose ether powder is selected from the group of alkyl cellulose ethers, hydroxyalkyl cellulose ethers, carboxyalkyl cellulose ethers, and of mixed ethers with at least two different substituents from the group of alkyl, hydroxyalkyl, carboxyalkyl with in each case alkyl groups with 1 to 10 carbon atoms.

5. Additive according to one of the claims 1 to 3, wherein the cellulose ether powder is selected from the group of methyl cellulose (MC), ethyl cellulose (EC), methyl hydroxyethyl cellulose (MHEC), ethyl hydroxyethyl cellulose (EHEC), methyl ethyl hydroxyethyl cellulose (MEHEC), hydroxyethyl cellulose (HEC), hydroxypropyl cellulose (HPC), methyl hy-

droxypropyl cellulose (MHPC), hydroxypropyl methyl cellulose (HPMC), hydroxyethyl methyl cellulose (HEMC), carboxymethyl cellulose (CMC).

6. Additive according to one of the claims 1 to 5, wherein the additive consists essentially from 50 to 95 % by weight of one or more water-redispersible polymer powders a), and from 5 to 50 % by weight of one or more cellulose-ether powder b), based on the dry weight of the additive composition.

7. Additive according to one of the claims 1 to 6, wherein the additive is distributed in portions of 50 g to 10 kg.

8. Use of the additive in the preparation of a minerally setting aqueous mortar composition comprising one or more mineral binders, and one or more fillers, wherein 0,5 to 10 % by weight, based on the dry weight of the mineral binder, of the additive according to one of the claims 1 to 7 is added to the mortar composition.

9. Minerally setting aqueous mortar composition modified with the additive according to one of the claims 1 to 7, comprising 5 to 80 parts by weight of mineral binder, and from 20 to 95 parts by weight of filler, and water in an amount for reaching the desired consistency of the aqueous mortar.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/CN2015/084879

A. CLASSIFICATION OF SUBJECT MATTER

C04B 24/38(2006.01)i

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

C04B 24

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

VEN, ISI, CNKI, CNTXT: additive, mineral+, aqueous, mortar, water, redispersible, polymer, cellulose, ether, vinyl, acetate, ethylene, filler, binder

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	CN 101541706 A (WACKER CHEM AG) 23 September 2009 (2009-09-23) description, page 9, the last second paragraph 2 to page 10, paragraph 1	1-3, 6-9
Y	CN 101541706 A (WACKER CHEM AG) 23 September 2009 (2009-09-23) description, page 9, the last second paragraph 2 to page 10, paragraph 1	4-9
Y	CN 102295429 A (DOW CHEM CO) 28 December 2011 (2011-12-28) description, paragraph [0032]	4-9
A	CN 1599703 A (AKZO NOBEL NV) 23 March 2005 (2005-03-23) the whole document	1-9

☐ Further documents are listed in the continuation of Box C. ☑ See patent family annex.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier application or patent but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
10 September 2015	**22 September 2015**

Name and mailing address of the ISA/CN	Authorized officer
STATE INTELLECTUAL PROPERTY OFFICE OF THE P.R.CHINA **6, Xitucheng Rd., Jimen Bridge, Haidian District, Beijing 100088, China**	**LI,Lianlian**
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INTERNATIONAL SEARCH REPORT

International application No.
PCT/CN2015/084879

Box No. III Observations where unity of invention is lacking (Continuation of item 3 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

[1] Claim 1 additive for minerally setting aqueous mortar compositions;

[2] Claim 8 use of the additive according to one of the claims 1 to 7 in the preparation of a minerally setting aqueous mortar composition comprising one or more mineral binders;

[3] Claim 9 minerally setting aqueous mortar composition modified with the additive according to one of the claims 1 to 7.

[4] The same technical feature among claims 1, 8 and 9 is the additive defined in claim 1. It is found that the same or corresponding technical feature mentioned above does not make contribution to prior art, therefore, the any two between claims 1, 8 and 9 do not have same or corresponding special technical features, that is, the any two between claims 1 , 8 and 9 are not so linked as to form a single general inventive concept and do not have unity of invention as required by the PCT Rule 13.1.

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims.

2. ☑ As all searchable claims could be searched without effort justifying additional fees, this Authority did not invite payment of additional fees.

3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:

4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

☐ The additional search fees were accompanied by the applicant's protest and, where applicable, the payment of a protest fee.

☐ The additional search fees were accompanied by the applicant's protest but the applicable protest fee was not paid within the time limit specified in the invitation.

☐ No protest accompanied the payment of additional search fees.

INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.
PCT/CN2015/084879

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