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Admixture for cement mixes. EP 0188471 A1

ABSTRACT translated from [French](#)

Aggregate containing mixtures of cement, for instance of cement milk, mortar and concrete used for construction or cement brick, cement block, stucco, and even ceramic tile. These blends include: a) a hydraulic cement; b) one or more flocculating agents selected from sodium alginate, soluble cellulose ether in water, polyacrylate, polyacrylamide, guar gum, gelatin, chitosan, dextrin and starches dialdehyde; c) one or more water-reducing agents selected from the condensates of naphthalene / formaldehyde sulfonated melamine / formaldehyde condensates sulfonated lignosulfonates, modified lignosulfonates, polyhydroxycarboxylic acids, salts of polyhydroxycarboxylic acids, glucosaccharides, linear or cyclic olefin copolymers having from 4 to 6 C and unsaturated dicarboxylic acids ethylenes; d) an aggregate and e) water.

DESCRIPTION (OCR text may contain errors) (text from [WO1986000291A1](#))

Improvements in or relating to organic compounds for cement mixes .

The invention relates to aggregate containing cement mixes (such as grouts, mortars and concrete) for use in construction and for cementing brick, cement block, stucco and even ceramic ti les.

According to the invention there is provided a cement mix comprising: a) hydraul ic cement; b) one or more flocculating agents, selected from sodium alginate, water soluble cellulose ether, polyacrylates polyacrylamides, guar gum, gelatin, chitosan, dextrin and dialdehyde starches; c) one or more water reducing agents selected from sulphonated naphthalene/formaldehyde condensates, sulphonated melamine/formaldehyde condensates, lignosulphonates, modified lignosulphonates, salts of polyhydroxy carboxylic acids, polyhydroxy carboxylic acids, glucosaccharides, copolymers of linear or cyclic C₄₋₆- olefins and unsaturated ethylenic dicarboxylic acids; d) aggregate; and e) water.

Preferred flocculating agents are selected from water soluble ethers, preferably hydroxypropyl -methyl -cellulose and sodium carboxymethyl cellulose and sodium alginate. Preferably when the water reducing agent is a copolymer of an unsaturated ethylenic dicarboxylic acid, the acid is itaconic acid.

Preferably when the flocculating agent is a polyacrylate it is sodium polyacrylate.

Preferably when the water reducing agents are polyhydroxy carboxylic acids these are C₆₋₈mono- or di-carboxylic acids, more preferably gluconic or glucoheptonic acids. Preferably the salts of carboxylic acids are those of the preferred and more preferred carboxylic acids. Most preferred carboxylic acid, salts are sodium gluconates and sodium glucoheptonates.

Preferred water reducing agents are selected from sulphonated naphthalene/formaldehyde condensates, sulphonated melamine/ formaldehyde condensates, sodium or calcium lignosulphonate and alkali-treated or ultra-filtered modified lignosulphonates.

Preferably the amount of flocculating agent present is 0.01 to 0.5 % by weight (based on the dry weight of cement present). The amount of flocculating agent

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CLAIMS (OCR text may contain errors) (text from [WO1986000291A1](#))

AMENDED CLAIMS

[received by the international Bureau on 27 December 1985 (27.12.85); original claims 1-14 replaced by new claims 1 -10 (2 pages)]

1. A cement mix comprising a) hydraulic cement b) hydroxypropyl methylcellulose c) a lignosulphonate or modified lignosulphonate d) aggregate and e) water.
2. A cement mix according to Claim 1 in which d) is fine aggregate and lime is additionally present.
3. A cement mix according to Claim 1 or Claim 2 containing additionally a compound selected from polyhydroxycarboxylic acids and salts thereof and polysaccharides.
4. A cement mix according to Claim 3 in which the additional component is a gluconate.
5. A mortar comprising a) a hydraulic cement b) hydroxypropyl methylcellulose c) a lignosulphonate or modified lignosulphonate and at least one compound selected from polyhydroxycarboxylic acids and salts thereof and polysaccharides. d) aggregate e) water and f) an anionic surfactant selected from olefin sulphonates and alkyl benzene sulphonates.
6. A mortar comprising a) a hydraulic cement b) hydroxypropyl methylcellulose c) sodium or calcium lignosulphonate and sodium gluconate d) aggregate e) water and f) a sodium alpha olefin sulphonate.
7. A mortar according to Claim 6 in which the amount of component b) is 0.02 to 0.07 %, of lignosulphonate is 0.10 to 0.20%, of sodium gluconate is 0.35 to 0.6%, of component d) is 200-800%, of component e) is 25-65% and of component f) is 0.001 to 0.008%, all percentages being by weight based on the dry weight of cement present.
8. A mortar according to any one of Claims 5 to 7 containing in addition up to 80% of lime by weight based on the dry weight of cement present.

used is preferably dependent on its molecular weight. Where the molecular weight is at the higher end of the range (e.g. a molecular weight of approximately 140,000) the more preferred amount of flocculating agent used is 0.01 to 0.135, most preferably 0.03 to 0.1 % (based on the dry weight of cement present). Where the molecular weight is at the lower end of the range (e.g. a molecular weight of approximately 15-20,000) more preferably the amount of flocculating agent used is 0.10 to 0.4 % most preferably 0.1 to 0.3 % (based on the dry weight of cement present).

The amount of water reducing agent varies depending on the efficiency of the water reducing agent and the amount needed to compensate for loss in fluidity properties due to the flocculating agent. Preferably the amount of water reducing agent present is 0.1 to 2 % , more preferably 0.2 to 1.3 % , most preferably 0.2 to 1.0 % by weight (based on the dry weight of cement present). Preferably aggregate includes one or more of the following natural silica sand, manufactured or processed sand, blast furnace slag, masonry sand, crushed stone, gravel and any other coarse aggregate.

Preferably the amount of aggregate present is 200 to 800 % by weight (based on the dry weight of cement present). Where the aggregate is fine aggregate the more preferred amount is 200 to

600 % and where the aggregate is fine and coarse aggregate the more preferred range is 400 to 800 %. In concrete generally at least 40 % of the aggregate is fine aggregate.

Preferably the amount of water present is 25 to 65 % by weight (based on the dry weight of cement present). More preferably the amount of water present is 30 to 60 %, most preferably 35 to 55 %.

In cement mixes according to the invention the hydraulic cement preferably comprises 10 to 30 %, more preferably 15 to 25 % of the total weight of cement mix.

Hydraulic cement preferably includes portland cements, blended cements, high alumina cements and pozzolaic cements. Portland cement comprises tricalcium silicate (alite), dicalcium silicate (belite), tricalcium aluminate and tetracalcium alumina ferrite. Further minor components include sodium and potassium oxides and magnesium oxide. Calcium sulphate may also be ground into the cement. Blended cements are those containing cement and finely divided blast furnace slag and/or fly ash.

The flocculating agent and water reducing agent can be worked simultaneously or singly at any stage into the cement mixture and before or after any other cement admixtures have been added.

Further, according to the invention, there is provided a method for preparing a cement mix including aggregate and water comprising adding to the cement mix one or more flocculating agents selected from sodium alginate, water soluble cellulose ethers, polyacrylates, polyacrylamides, guar gum, gelatin, chitosan, dextrin and dialdehyde starches and one or more water reducing agents selected from sulphonated naphthalene/formaldehyde condensates, sulphonated melamine/formaldehyde condensates, lignosulphonates, modified lignosulphonates salts of polyhydroxy carboxylic acids, polyhydroxy carboxylic acids, glucosaccharides, copolymers of linear and cyclic C₄₋₆-olefins and unsaturated ethylenic dicarboxylic acids.

Loss in fluidity in concrete is measured by slump loss. Slump is a measure of how much a given height of a conical cement specimen will drop from its original height when no longer constrained by the mould of a slump cone. A slump cone is standardized at 30.6 cms. Therefore, a cement mix with a slump of 20 cms means that the mix has settled to about 1/3 of its original height. Slump loss is a measure of how much slump diminishes on standing for a given time.

Loss in fluidity for mortars and grouts may be measured by the known vibration drop table test for spreading loss which is analogous to the slump test.

One advantage of a method according to the invention is that fluid loss can effectively be reduced and fluidity is maintained at roughly the same level for a long period of time. In the case of concrete it has been found that there is practically no slump loss for approximately 1 hour after combined addition of the flocculating agent and the water reducing agent. Further, there is no need to increase the amount of water present to compensate for slump loss as was necessary in the past. (This increase caused the physical properties of the concrete to be impaired). Further, the physical properties of the concrete will be improved by the addition of the water reducing agent and the flocculating agent, for instance the resistance of concrete to segregation will be increased and the rheological characteristics and pumpability will be increased.

In the past there has been a tendency to avoid adding a high molecular flocculating agent to a cement mix, especially concrete, since fluidity would generally be greatly reduced. Further, where a water reducing agent alone has been added

9. A method for preparing a mortar according to Claim 5 comprising adding to a mix of hydraulic cement, aggregate and water the following admixture a) hydroxypropyl methylcellulose b) a lignosulphonate or modified lignosulphonate and at least one compound selected from polyhydroxy carboxylic acids and salts thereof and polysaccharides and c) an anionic surfactant selected from olefin sulphonates and alkyl benzene sulphonates.

10. A method according to Claim 9 comprising adding to a mix of hydraulic cement aggregate and water the following admixture: a) 0.02 to 0.07% of hydroxypropyl methylcellulose b) 0.10 to 0.20% of sodium or calcium lignosulphonate and 0.35 to 0.6% of sodium gluconate and c) 0.001 to 0.008% of sodium alpha olefin sulphonate, all percentages being by weight based on the dry weight of cement present.

although the fluidity of the cement mix is increased immediately after addition, it has rapidly deteriorated.

Still further according to the invention there is provided a mortar comprising: a) a hydraulic cement b) a water soluble cellulose ether c) at least one water reducing agent selected from sulphonated naphthalene/formaldehyde condensates, sulphonated me1amine/formaldehyde condensates, lignosulphonates, modified lignosulphonates, copolymers of linear or cyclic C₄₋₆ olefins and unsaturated ethylenic dicarboxylic acids,

(preferably lignosulphonates)and at least one water reducing agent selected from polyhydroxy carboxylic acids and salts thereof and polysaccharides (preferably gluconates). d) water e) aggregate; and f) an anionic surfactant selected from olefin sulphonates and alkyl benzene sulphonates.

Preferably in a mortar according to the invention lime is present.

Yet still further according to the invention there is provided a method for preparing a mortar comprising adding to a mix of hydraulic cement, aggregate and water the following admixture: a) a water soluble cellulose ether;

b) at least one water reducing agent selected from sulphonated naphthalene/formaldehyde condensates, sulphonated melamine/formaldehyde condensates, lignosulphonates, modified lignosulphonat,es, copolymers of linear or cyclic C₄₋₆ olefins and unsaturated ethylenic dicarboxylic acids,

(preferably lignosulphonates)and at least one water reducing agent selected from polyhydroxy carboxylic acid and salts thereof and polysaccharides (preferably gluconates); and c) an anionic surfactant selected fromolefin sulphonates and alkyl benzene silphonates. In a mortar according to the invention the amount of water soluble cellulose ether present is 0.02 to 0.07 % by weight (based on dry weight of cement present).

Preferably in a mortar according to the invention the amount of lignosulphonate present is 0.10 to 0.20 % by weight (based on the dry weight cement present).

Preferably, in a mortar according to the invention the amount of gluconate present is 0.35 to 0.6 % by weight (based on the dry weight of cement).

Preferably in a mortar according to the invention the amount of anionic surfactant is 0.001 to 0.008 % by weight (based on dry weight of cement present).

In a mortar according to the invention the lignosulphonate is preferably sodium lignosulphonate.

In a mortar according to the invention the water soluble cellulose ether is preferably hydroxypropylmethyl cellulose.

In a mortar according to the invention gluconate is preferably sodium gluconate.

In a mortar according to the invention the olefin sulphonate is preferably a sodium alpha olefin sulphonate. More preferably the olefin sulphonate is a C₁₂₋₁₈ olefin sulphonate. By the term α olefin sulphonate is meant a compound having a double bond at the end of the molecule i.e. at the α position and such olefins are usually obtained by cracking a petroleum fraction or by polymerisation of ethylene.

Preferably the amount of lime when present is from greater than 0 to 80 % by weight (based on dry weight of cement present).

Preferably the amount of aggregate present is 200 to 800 % by weight based on the dry weight of cement present.

Preferably the amount of water present is 25 to 65 % by weight based on the dry weight of cement present.

The above described mortars may be premixed mortars which may be delivered and stored at construction sites for as long as 72 hours after the initial mix has been added. The mortars according to the invention have good storage ability and in particular retard the hydration of the mortar during mixing, transport and storage whilst not interfering with the properties needed to utilise the mortar as a cement binding mix. The mortars of the invention remain usable for as long as 72 hours without the need to retemper or extensively remix. The mortars of the invention have excellent adhesive properties to brick or block, adsorption of water from the mix onto the porous surface of building units is uniform, air content is stable and strength needed to support loads placed on it is adequate.

The invention will be illustrated by the following Examples. Examples 1 to 6

The materials used for these tests were the following:

- (a) Cement: Blend of 3 brands of ordinary portland cement
- (b) Fine Aggregate: Oi River Basin pit sand

(c) Coarse Aggregate: Ohme crushed stone, max. size = 20 mm

(d) Flocculating Agent

(l) Sodium alginate (hereafter AG-Na)

Five kinds with differing molecular weights were used. L₂ has a molecular weight of less than 15,000

L has a molecular weight of about 15,000

M has a molecular weight of about 70,000

H has a molecular weight of about 140,000 and

H₂ has a molecular weight of more than 140,000.

(l l) Sodium carboxymethyl cel l ulose (hereafter CMC) having a molecular weight of about 20,000 a highly etherized product of low viscosity type was used; met

(e) Water-reducing Agents:

(l) Sulfonated naphthalene/formaldehyde condensate sodium salt (hereafter NSF) having a molecular weight of about 2,000.

(ll) Sulfonated melamine/formaldehyde condensate sodium salt (hereafter MSF) having a molecular weight of about 2,000.

Mixing of concrete in these tests was done with a -100-liter tilting mixer. The total weight of aggregate used in Examples 1 to 35 is 1900 kg per m³ of total mix.

Examples 1 to 6

The mixture ratios of these examnles are given in Table 1 below A cement mix containing cement, aggregate, a water reducing agent and a flocculating agent (when present) is made up in the proportions given in Table 1 below. The symbols given in the Table are as follows:

W/C is the water to cement ratio;

S/a is the ratio of fine aggregate to coarse aggregate;

W is the amount of water present;

C is the amount of cement;

NSF is sulphonated naphthalene/formaldehyde concentrate sodium salt and

AG-Na-M is sodium alginate having a molecular weight of about 70,000



Table 2 shows the slump levels for each of the mixtures of Examples 1 to 6 of Table 1. TABLE 2



Example No. 6 is a mix using NSF alone aiming for a slump of 21 cm immediately after mixing, and this shows a large slump loss. The mix of Example No. 2 is the mix of Example No. 6 to which a high molecular flocculating agent (AG-Na-M) is added with NSF having increased dosage (0.2 percentage) to compensate for reduction in slump immediately after mixing due to addition of the flocculating agent. The cement of this combined use has a slump immediately after mixing of identical level with Example No. 6, but it is seen that subsequent slump loss is minimal. Examples Nos. 3 and 4 are cases where the NSF dosage is constant and the dosage of AG-Na-M is increased. The slump immediately after is smaller with increased dosage of AG-Na-M, but slump loss is also reduced. Example No. 5 is a mix where the dosage of NSF is made larger than Example No. 4. Similarly to Example No. 2, the slump loss was extremely small. Example No. 1 was a mix with AG-Na-M omitted. Segregation occurred and so it was impractical as a concrete mix. In essence, the slump loss reduction effects in Examples Nos. 2 to 5 have been brought about by the combined use of NSF and AG-Na-M, and it is seen that they do not result simply from increase in the dosage of NSF. Examples 7 to 14

Concrete mixes are made up as in Examples 1 to 6, in which however, the mix proportions are as given in Table 3 and the time- dependent changes in slump in Table 4. The symbols an these Tables are as defined above.

TABLE 3



TABLE 4



It is clear from the above table that the flocculating action becomes greater the larger the molecular weight of AG-Na, and consequently there is a tendency for the slump loss. to become smaller after mixture at the same dosage. In other words, there is a tendency for the sufficient dosage to be smaller the larger the molecular weight when slumps immediately after mixing are to be the same. Accordingly, there are appropriate combinations of dosages of NSF and AG-Na when slumps immediately after mixing are to be the same, and it can be seen that this combination varies depending on the molecular weight of AG-Na. Example No. 7 indicates that segregation occurs because of the use of NSF alone.

Examples 15 to 20

Cement mixes are made up similar to those of Examples 1 to 6 having the mix proportions given in Table 5 below. These mixes are superplasticized concrete with the combined addition of NSF and AG-Na-M. By superplasticized concrete is meant a concrete made by adding a superplasticizer (normally a high molecular weight water-reducing agent) to a concrete (called base concrete) consisting of cement, aggregate and water only.

The addition of superplasticizer is generally done after making the base concrete as described above (this is called the delayed addition system), but recently, a system for addition at the time of mixing the base concrete (this is called the simultaneous addition system) has been proposed. Although the simultaneous addition system has the advantage of simplification of the addition procedure, there is economic disadvantage that the dosage of superplasticizer becomes larger than with the delayed addition system, and the situation about slump loss itself still remains unimproved.

The proportions of the mix of concrete are given in Table 5 and the time-dependent changes in slump in Table 6. Superplasticization by the delayed addition system was done 15 minutes after completion of mixing of base concrete. TABLE No. 5



Note: 1) By base admixture is meant the admixture used when making the base concrete, which in this case was a commercial air-entraining water-reducing agent, Pozzoloth 70 (P-70). This is present in the base concrete.

2) The figures in the columns for "Base Admixture" and

"Superplasticizer" indicate the respective dosages (%) based on the dry weight of cement present.



Note: By "Immediately After Superplasticization" in Examples Nos. 15 to 20 is meant "immediately after discharge after completion of mixing in mixer."

Examples Nos. 15 and 17 indicate cases of conventional delayed addition and simultaneous addition systems using NSF as the superplasticizer, and slump losses after superplasticization are large in both cases. As is clear from Examples Nos. 16 and 18 the method of this invention is effectively applied to either the delayed addition system or the simultaneous addition system for superplasticized concrete.

Examples Nos. 19 and 20 are cases to confirm that the combined use of AG-Na-M irrespective of the fact that there is a large amount of superplasticizer (NSF) present produces favourable results.

Examples 21 to 24

In a similar fashion to the Examples 15 to 20 cement mixtures are made up having the proportions given in Table 7 below and the time-dependent changes in slump are given in Table 8.

TABLE No. 7



TABLE No. 8



Examples Nos. 21 to 23 are mixes having the amount of NSF dosage increased by 0.2 percentage points over the mix of No. 24 with CMC dosages being 0.1 to 0.3 percent, and it can be seen that the situation regarding slump loss is greatly improved by the combined addition. Examples 21 to 23 are mixes having a constant NSF. dosage with CMC dosage gradually increased, and it can be seen that slump immediately after mixing and slump loss is gradually reduced because of increased flocculating action due to increase in CMC dosage. However, this, tendency is less than with AG-Na.

Examples 25 to 31

Cement mixes similar to those of Examples 1 to 6 are made up of superplasticized concrete in case of using CMC as the high molecular flocculating agent. The mix proportions are given in Table No. 9 below, and time-dependent changes in slump in Table 10.

Comparisons are shown between delayed and simultaneous additions of the NSF and CMC to the base mix. TABLE No. 9



TABLE No. 10



As is clear from the above table, it can be seen that when CMC is used as the high molecular flocculating agent, it is also effective for application of the method of this invention to superplasticized concrete.

Examples 32 to 35

Cement mixes similar to those of Examples 1 to 6 have been up using MSF as the water-reducing agent and CMC as the high molecular weight flocculating agent. The mix proportions are given in Table No. 11 and the time-dependent changes in slumps in Table 12.

TABLE No. 11



TABLE No. 12



Example 35 is a concrete mix using MSF such that the initial slump is 20 cm. The slump loss of this concrete was large. Examples Nos. 33 and 34 are mixes with combined addition of MSF and CMC and slump losses were small. Example No. 32 is a mix only using MSF and in which segregation occurs.

All mortar proportions by volume for Examples No.s 36 to 40 are determined in accordance with ASTM Designation C 270-80-Standard Specification to Mortar for Unit Masonry Mortar Types (= Table 2).

Examples 36 to 38

A mortar mix is made up containing 898 g of Type 1 Portland cement, 100 g of masonry lime, 2840 g of masonry sand with a measured fineness modulus of 1.81 in half the required water for each example in a paddle type Hobart mixer.

An admixture comprising 0.52 % sodium gluconate, 0.175 % sodium lignosulphonate, 0.07 % hydroxypropylmethylcellulose and 0.008 % α -olefin sulphonate (all percentages are based on dry cement present in the combined mortar mix) is made up. The admixture is added to the mortar mix and additional water is added until the desired workability is achieved. The total amount of water present is about 540 g. The hydroxy propylmethyl cellulose has a molecular weight of about 120,000.

The water to cement ratios are used as given in Table 13 below. TABLE No. 13



Consistency (48 hours after mixing), pliable pliable pliable

The lignosulphonate used has a molecular weight of 2,000 (1) ASTM Designation C 185-80 Standard Test Method for Air Content of Hydraulic Cement Mortar.

(2) ASTM Designation C 102-00 Standard Test Method for Compressive Strength of Hydraulic Cement Mortars.

(3) ASTM Designation: C 472-79 Standard Methods for Physical Testing of Gypsum Plasters and Gypsum Concrete.

Examples 39 and 40

These examples comprise a mortar mix containing approximately 0.3 % water (based on the dry weight of cement), 898 g of Type 1 Portland Cement, 100 g of masonry lime and 2840 g of masonry sand with a measured fineness of 1.81 in half the required water in a paddle type Hobart mixer. The appropriate admixture for Examples 39 and 40 given in Table 13 below, is then added.

TABLE 14



all % are based on the amount of cement present.

The properties of these mixes are given in Table 15 below

TABLE 15 9



Examples 41 to 47

Cement mixes were made up analogously to the method of Examples 1 to 6 using lignosulphonate instead of NSF as the water reducing agent.

In these Examples the materials used were the following:

a) Cement: A mix of 3 brands of Normal Portland Cement from

Onoda, Asano and Sumitomo in equal quantities. b) Fine aggregate: Oi River Basin pit sand c) Coarse aggregate: Ohme crushed stone. d) Lignosulphonate: (LIG) George Pacific's LIG having a molecular weight of about 2,000. e) Flocculating agent: CMC with a molecular weight of about

20,000. f) Air entraining water reducing agent: Pozzolith 70

The amount of concrete made is 80 liters and the air temperature is 20°C. Air entraining agent us initially added in such an amount that the air content is about 5 %. The mixes and results for simultaneous addition are given in Table 16 and for delayed addition are given in Table 17.



NON-PATENT CITATIONS

Reference

1 * See references of [WO8600291A1](#)

* Cited by examiner

CLASSIFICATIONS

International Classification [C04B28/02](#)

Cooperative Classification [C04B28/02](#)

European Classification [C04B28/02](#)

LEGAL EVENTS

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Nov 24, 2007	PG25	Lapsed in a contracting state announced via postgrant inform. from nat. office to epo	Ref country code: IT Free format text: LAPSE BECAUSE OF FAILURE TO SUBMIT A TRANSLATION OF THE DESCRIPTION OR TO PAY THE FEE WITHIN THE PRESCRIBED TIME-LIMIT Effective date: 19891129