(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
20 October 2005 (20.10.2005)

PCT

(10) International Publication Number
WO 2005/097699 A1

(51) International Patent Classification[7]: C04B 28/02, 24/10, 16/00, 24/08

(21) International Application Number: PCT/AU2005/000466

(22) International Filing Date: 31 March 2005 (31.03.2005)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
2004901858 6 April 2004 (06.04.2004) AU

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(81) Designated States *(unless otherwise indicated, for every kind of national protection available)*: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SM, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW.

(84) Designated States *(unless otherwise indicated, for every kind of regional protection available)*: ARIPO (BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

Published:
— *with international search report*

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: EFFLORESCENCE REDUCING ADMIXTURES

(57) Abstract: An admixture that reduces efflorescence in concretes and mortars is disclosed. The admixture consists of a mixture of granulated blast furnace slag, finely divided fly ash, finely divided flour, a long chain fatty acid and an anionic dispersant. The admixture is typically used at 0.5 to 5% w/w cement or cementitious binding material in a concrete or mortar.

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EFFLORESCENCE REDUCING ADMIXTURES

Technical Field

This invention relates to concrete and mortar admixtures and in particular to admixtures for reducing the occurrence of efflorescence in concretes and mortars.

Background of the Invention

Concrete is a widely used construction material for buildings, roads and structural support members such as bridges and railway sleepers. Admixtures are additives that are added to the basic concrete composition when the concrete is prepared to modify various properties relating to the concrete while in the workable state, such as its liquid consistency as measured by its slump. Admixtures are also available that influence the workability characteristics and the set, hardening and strength gain times of concrete as well as enhancing the hardened properties of the concrete.

Efflorescence is a white crystalline bloom that may be uniform or patchy in appearance that sometimes appears on the surface of cast-in-place concrete, concrete products and mortar surfaces.

Hawley's "Condensed Chemical Dictionary", 12th Edition, Richard J. Lewis, defines efflorescence as "the loss of combined water molecules by a hydrate when exposed to air, resulting in partial decomposition indicated by presence of a powdery coating on the material". In the context of concrete, a similar phenomenon could also be referred to as the formation of laitance (cement scum) at the surface of concrete very soon after its placement. This is usually caused by bleeding of part of the mixing water which on hardening is weak, often dusty (often only about ½-1½ MPA in compressive strength) and pale, being lighter in colour than the concrete beneath it.

Weak friable surface laitance is a defect for hardened cast-in-place concrete, especially for floors and pavements. It causes a poor quality, dusty weak surface. Freedom from dusting, high strength, hardness and abrasion resistance is ideally required. The existence of laitance on existing hardened

concrete also prevents the adhesion and penetration of surface coatings, curing compounds, colouring stain/sealers, paving paints and other coatings. Furthermore, laitance "covers" and masks the true colour of mineral (metal) oxide pigment integrally ("through") coloured concrete, making it appear lighter/paler and giving the incorrect impression of fading of those pigments.

It would be desirable to have a concrete admixture that alleviated these problems in concrete and mortars.

Summary of the Invention

This invention provides in one form a concrete and mortar admixture comprising:

a finely divided pozzolan selected from the group consisting of ground granulated blast furnace slag and finely divided fly ash (10 - 70% w/w);

a finely divided flour (10 - 30% w/w);

a long chain fatty acid (5 - 20% w/w); and

an anionic dispersant (0.5 - 5% w/w).

Preferably, the group of pozzolans further consists of finely divided low density diatomaceous earth.

Preferably, the pozzolans are a combination of fine granulated blast furnace slag, finely divided fly ash and finely divided diatomaceous earth.

Preferably, the long chain fatty acid is a saturated fatty acid having C_{12} to C_{20} carbon chain length.

More preferably, the long chain fatty acid is stearic acid.

Preferably, the finely divided flour is cake or sponge flour.

Preferably, the anionic dispersant is a carboxylic acid salt or a sulfonic acid salt.

Preferably, the composition further comprises a microscopic air entraining admixture.

Preferably, the flour is a wheat flour.

In an alternative form the invention provides a method of reducing efflorescence in concretes and mortars by adding as an admixture to a concrete

or mortar mix 0.5 to 5% w/w cement or cementitious binding material, wherein the admixture comprises:

a finely divided pozzolan selected from the group consisting of granulated blast furnace slag and finely divided fly ash (10 - 70% w/w);

a finely divided flour (10 - 30% w/w);

a long chain fatty acid (5 - 20% w/w); and

an anionic dispersant (0.5 - 5% w/w).

Detailed Description of the Invention

The individual ingredients for the admixture of the present invention are well known and do not require detailed description. A "pozzolan" is defined as "a siliceous or siliceous and aluminous mineral material which in itself possesses little or no cementing property, but will in a finely divided form and in the presence of moisture chemically react with calcium hydroxide at ordinary temperatures to form compounds possessing cementitious properties." This definition of pozzolans is taken from "Pozzolanic and Cementitious Materials" by Malhotra and Mehta (Gordon and Breach Publishers, 1996). The particle size of the pozzolans are such that the majority by weight of the particles pass through a 300μm sieve. Generally better results are obtained as the particle size is reduced. This is particularly the case with the cake flour. The admixture of the present invention is prepared by dry blending suitable raw materials.

This improved formulation of the admixture is to be marketed by Ability Building Chemicals of 133-135 Northern Road, West Heidelberg, Victoria, Australia, a trading arm of Australian Oxides Pty Ltd, under the brand name EFFLOREIN MARK 2. It will replace and supersede an earlier formulation of EFFLOREIN which while offering improved properties in its features and benefits, is generally not as efficacious as the formulation of the present invention.

The admixture is an easily dispersible, free-flowing, off-white coloured powder. It is used as an admixture for addition to virtually all types and grades of pre-mixed (ready mixed) concrete. It is also suitable for Portland

cement-based and sand-lime low water content "semi-dry mix" no slump concrete product mixes such as those used to manufacture hand or machine-made masonry bricks/blocks/pavers as well as for concrete roofing tile mixes. It may be added to factory compounded cement-based dry mixes. These include most types of factory blended dry mortar and grout mixes including masonry mortar, cement rendering mortar, external mortar plastering mixes, "bagging" wall finishes, and cementitious "deco-pave" decorative pavement finishes. In addition to controlling the occurrence of efflorescence, the admixture water-proofs concrete, brightens the effect of colouring pigments if used and, in pre-mixed or site-mixed concrete, substantially reduces or inhibits bleeding.

It is also an outstandingly effective water-proofing and water-repelling admixture as well as a general conditioner and improver for all cement and/or lime based mixes. The product may be used as a high performance "clear" water-proofer for mortar and concrete as well as a "clear" masonry mortar dampcourse admixture.

The admixture when added into all Portland cement-based mixes results in greater impermeability and water shedding ability. These beneficial characteristics are applicable to hardened cast-in-place concrete, concrete products ("semi-dry" mix or "wet-mix" machine made types) as well as resin dispersions, typically acrylic, modified mortar and grout materials/products. These characteristics are usually obtained without reducing the necessary bond required for bricks, blocks, pavers and other concrete products used with laying, jointing or bedding mortars and grouts.

In machine made "semi-dry-mix" concrete masonry block and paver manufacture, the admixture usually results in the same, unchanged degree of apparent consistency as the relevant unset concrete/mortar mix designs but additionally imparts beneficial thixotropy, extrudability and mix rheology. The same degree of natural average "slick" "wipe" and/or surface appearance of "semi-dry-mix" concrete masonry, block, brick paver and concrete roofing tile product mixes is retained - unless modified by another admixture.

Higher "green" strength of set but not yet hardened concrete products as well as higher earlier strength is achieved, thus reducing breakages and wastage and often (if the concrete temperature is adequate) allowing a shorter time before de-moulding. With "wet-mix" pre-mixed "slump" concrete mixes, the water content can usually be reduced by around 10-15% for the same consistency. Therefore, the required strength grade of concrete may be made at the pre-mixed concrete supply plant at a 40mm slump instead of the typical 80mm and, after the mixing in the EFFLOREIN MARK 2, a slump close to 80mm is achieved and, if necessary, later adjusted to the required level.

The admixture may also be used at lower than normal dose rates to obtain beneficial synergistic complimentary effects for eliminating efflorescence (as well as for providing extra water-proofing) in concretes or mortars to which acrylic polymer emulsion or spray dried powder based admixture products such as Ability Building Chemicals' liquid ADMIX 2000 and ABIL-STRENGTH powder bond increasing and flexibilising admixtures, and permeability reducing liquid admixtures such as liquid ammonium stearate have been added.

For concrete and mortars having average cement to aggregate ratios, (1:6 to 1:9 by weight a nominal 1½% ± ½% of the admixture by weight is usually used on the weight of the cementitious powder binder (pure Portland cement or Portland cement blended with supplementary cementitious materials such as cement/fly ash, cement/Diatomite D5, cement/ground granulated blast furnace slag, or a triple/quadruple blend of any of these). Higher dose weight percentages of the admixture may be required for lower cementitious material or pure cement to aggregate (C/A) ratios and the reverse for higher pure C/A ratios.

The above suggested starting point dose rate may need to be increased for concrete left to set and harden in low temperatures (particularly during the night with ambient temperatures falling below 14°C and therefore reducing the temperature of the concrete to impede its rate of hardening) and for concrete having lower total cementitious material or lower pure cement to

aggregate ratios and/or fine aggregate having a particle size distribution and (relatively) high surface area that results in concrete having excessive voids and excessive water demand.

Alternatively, the dose may be reduced for denser more impermeable concrete and concrete product mix designs based on higher total cementitious material or pure cement to aggregate ratios and/or fine aggregate combinations, that result in minimum voids and in particular, lower water/cement ratios for the same mix consistency.

The invention will be further described by reference to the following illustrative example in which all percentages are expressed as parts by weight.

Example 1

This example illustrates the preparation of an admixture according to the invention.

Ground Granulated Blast Furnace Slag (Surface Area: 400-450 m^2 per kg.)	27.600
Diatomite D30 (5-30μm)*	8.000
Cosmotron DPU-CA*	12.000
Cake Flour	20.100
Fly Ash (Classified)	18.590
Maxim-Air® Microscopic Air Entraining Admixture*	0.210
Palmac 70/18 Steric Acid Powder**	12.000
Aerosil 200 (Fumed Silica ex Degussa)	1.500
Total:	100.000

* Ability Building Chemicals Co.

** Acid Chem. International Sdn. BHD

The admixture was prepared by dry blending the above ingredients.

Example 2

This example shows the comparative testing of the admixture of Example 1.

Concrete was produced in the laboratory using an older basalt coarse aggregate, a washed natural concrete sand and GP* cement. The following properties were determined and/or calculated from the results obtained. The relevant Australian Standard test methods that were used are as follows:

Slump	AS 1012.3.1
Air Content	AS 1012.4.1
Plastic Density	AS 1012.5
Water Demand	
Bleed - Total and Rate	AS 1012.6
Set Time - Initial and Final	AS 1012.18
Water/Cement Ratio	
Compressive Strength - up to 56 days	AS 1012.9
Drying Shrinkage - to 13 weeks drying	AS 1012.13

* GP = Australian General Purpose cement.

In addition, for each batch of concrete, a "slab" was cast with the intention of later providing cored specimens for compressive strength, sorptivity, water absorption and volume of permeable voids (VPV). Note that VPV's were determined using the sorptivity and water absorption test specimens following completion of these tests.

Core compressive strength was determined to the provisions of AS 1012.14 except that the specimens were not conditioned. Sorptivity was determined, after trimming a few millimetres off the face of the core to minimise the potential for interference from finishing, etc., by placing the face

of the oven dried specimen in water and measuring the amount of water uptake with time. Sorptivity was calculated from the slope of the line obtained by plotting the increase in mass versus the square root of time. Water absorption was determined to the provisions of British Standard BS 1881: Part 122 and VPV to AS 1012.21, except that in both cases the testing was commenced at 91 days compared with the nominal 28 days required.

Three concrete mix conditions were examined:

Mix Type 1 - nominal 20MPa concrete with water reducing and air entraining admixtures at "normal" dose rates, i.e., the "control" concrete.

Mix Type 2 - same basic mix but with the admixture of Example 1 at a dose of 1.5% on the weight of cement and a typical industry water reducing admixture at a "normal" dose rate.

Mix Type 3 - same as the basic mix but with the admixture of Example 1 at 1.5% on the weight of cement and no other admixtures.

The results indicated that there was no significant difference in set times for the three mix conditions.

The water penetration test results, sorptivity, water absorption and VPV, in conjunction with the results obtained for drying shrinkage indicate that the concretes containing the admixture of Example 1 lose and absorb water at a slower rate than the concrete without the admixture. As a result, it would be expected that water movement, particularly in a wetting and drying situation, through a concrete containing the admixture of Example 1, would be reduced.

From the results it was concluded that the admixture of Example 1 when used in "nominal" 20MPa concrete mixes, produced concretes with superior properties to "conventional" 20MPa concrete as outlined in Mix Type 1, such as an increase in strength in excess of that expected from water reduction alone, with the exception of the bleeding rate.

Sorptivity and water absorption (BS 1881.22) test results indicated that the admixture enhanced the resistance of the concrete to water penetration and movement by capillary action.

Since modifications within the spirit and scope of the invention may be readily effected by persons skilled in the art, it is to be understood that the invention is not limited to the particular embodiment described, by way of example, hereinabove.

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The claims defining the invention are as follows:

1. A concrete and mortar admixture comprising:

a finely divided pozzolan selected from the group consisting of granulated blast furnace slag and finely divided fly ash (10 - 70% w/w);

a finely divided flour (10 - 30% w/w);

a long chain fatty acid (5 - 20% w/w); and

an anionic dispersant (0.5 - 5% w/w).

2. A concrete and mortar admixture as defined in claim 1 wherein the group of pozzolans further consists of finely divided low density diatomaceous earth.

3. A concrete and mortar admixture as defined in claim 2 wherein the pozzolans are a combination of fine granulated blast furnace slag, finely divided fly ash and finely divided diatomaceous earth.

4. A concrete and mortar admixture as defined in any one of claims 1 to 3 wherein the long chain fatty acid is a saturated fatty acid having C_{12} to C_{20} carbon chain length.

5. A concrete and mortar admixture as defined in claim 4 wherein the long chain fatty acid is stearic acid.

6. A concrete and mortar admixture as defined in any one of claims 1 to 5 wherein the finely divided flour is cake or sponge flour.

7. A concrete and mortar admixture as defined in any one of claims 1 to 6 wherein the anionic dispersant is a carboxylic acid salt or a sulfonic acid salt.

8. A concrete and mortar admixture as defined in any one of claims 1 to 7 wherein the composition further comprises a microscopic air entraining admixture.

9. A concrete and mortar admixture as defined in any one of claims 1 to 8 wherein the flour is a wheat flour.

10. A method of reducing efflorescence in concretes and mortars by adding as an admixture to a concrete or mortar mix 0.5 to 5% w/w cement or cementitious binding material, wherein the admixture comprises:

a finely divided pozzolan selected from the group consisting of granulated blast furnace slag and finely divided fly ash (10 - 70% w/w);

a finely divided flour (10 - 30% w/w);

a long chain fatty acid (5 - 20% w/w); and

an anionic dispersant (0.5 - 5% w/w).

INTERNATIONAL SEARCH REPORT

International application No.
PCT/AU2005/000466

A. CLASSIFICATION OF SUBJECT MATTER

Int. Cl. [7]: C04B 28/02, 24/10, 16/00, 24/08

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)

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)
DWPI, JAPIO: C04B and keywords flour, acid
ESPACE keywords: pozzolan, ash, slag, flour

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 5,736,600 A (KARKARE *et al.*) 7 April 1998 See whole document	
A	US 2002/0005149 A1 (KARKARE *et al.*) 17 January 2002 See whole document	
A	Patent Abstracts of Japan, JP 2001-328859 A (FUKIYA TADASHI) 27 Novenber 2001 See abstract	

☐ 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

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"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
5 May 2005	11 MAY 2005
Name and mailing address of the ISA/AU AUSTRALIAN PATENT OFFICE PO BOX 200, WODEN ACT 2606, AUSTRALIA E-mail address: pct@ipaustralia.gov.au Facsimile No. (02) 6285 3929	Authorized officer **DAVID GRIFFITHS** Telephone No : (02) 6283 2628

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.
PCT/AU2005/000466

This Annex lists the known "A" publication level patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent Document Cited in Search Report		Patent Family Member					
US	5736600	AU	39081/97	BR	9711182	CA	2262447
		EP	0920472	NZ	334020	WO	9805709
US	2002005149	US	2004122144				
JP	2001328859		NONE				

Due to data integration issues this family listing may not include 10 digit Australian applications filed since May 2001.

END OF ANNEX