



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

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<p>(21) International Application Number: PCT/US94/07951 (22) International Filing Date: 14 July 1994 (14.07.94) (30) Priority Data: 08/099,086                      29 July 1993 (29.07.93)                      US  (71) Applicant: W.R. GRACE &amp; CO.-CONN. [US/US]; 1114 Avenue of the Americas, New York, NY 10036 (US).  (72) Inventors: ABDELRAZIG, Baha, Eldin, Ismail; 33 Shalcross Crescent, Hatfield, Hertfordshire AL10 9QH (GB). KINDT, Lawrence, Joseph; 181 Brass Eagle Drive, Sykesville, MD 21784 (US). MYERS, David, Francis; 10920 Rock Coast Road, Columbia, MD 21044 (US). MARTIN, Timothy, John; Loxwood Chase, Loxwood, Billingham, W Sussex RH14 0QW (GB).  (74) Agent: TROFFKIN, Howard, J.; W.R. Grace &amp; Co.-Conn., 7379 Route 32, Columbia, MD 21044 (US).</p>	<p>(81) Designated States: AT, AU, BB, BG, BR, BY, CA, CH, CN, CZ, DE, DK, ES, FI, GB, GE, HU, JP, KE, KG, KP, KR, KZ, LK, LT, LU, LV, MD, MG, MN, MW, NL, NO, NZ, PL, PT, RO, RU, SD, SE, SK, TJ, TT, UA, UZ, VN, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG).</p> <p><b>Published</b> <i>With international search report.</i></p>	
<p>(54) Title: EFFLORESCENCE INHIBITING CEMENT COMPOSITION</p> <p>(57) Abstract</p> <p>A hydraulic cement mixture and resultant cement composition formed therefrom which contains at least one C<sub>14-22</sub> alkyl alcohol in from 0.05 to 3 weight percent based on the cement. These products exhibit reduced efflorescence.</p>		

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**EFFLORESCENCE INHIBITING CEMENT COMPOSITION****BACKGROUND OF THE INVENTION**

The present invention is directed to the inhibiting of efflorescence on concrete. Specifically, the present invention is directed to a cement mixture and to cement compositions containing the mixture in which the cement mixture contains at least one fatty alcohol in certain specified amounts to inhibit efflorescence of the resultant structure.

In the construction industry, the term "efflorescence" refers to white deposits which appear on building materials of concrete and cement based products. Efflorescence is normally produced by processes known as lime bloom, lime weeping and to a lesser extent by crystallization of soluble salts.

Lime bloom is a phenomenon which is particularly noticeable on concrete and other products formed with Portland cement. It is a thin white deposit which appears either as white patches or as an over-all lightening in color. The latter effect is sometimes mistakenly interpreted as color fading or wash out.

The cause of lime bloom is related to the chemical composition of Portland cement. When water is added to cement, a series of chemical reactions take place which result in setting and hardening. One product of these reactions is lime in the form of calcium hydroxide which is slightly soluble in water and migrates through damp concrete to the surface where it reacts with atmospheric carbon dioxide to produce a surface deposit of calcium carbonate. This surface deposit is similar to a very thin coat of whitewash and gives rise to the undesired white patches or lightening of color mentioned previously.

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Lime weeping is closely related to lime bloom. Water moving across or through concrete dissolves the lime formed therein and deposits this lime as calcium carbonate at the structure's surface. In this instance, the calcium carbonate may not be deposited as a thin surface layer, but instead may build up to form thick encrustations.

For many years, the problem of lime bloom and lime weeping has been recognized and considerable effort has been undertaken in attempts to inhibit or prevent its occurrence. One method suggested is to reduce the fines (sand) contained in the cement compositions formulation. This results in a coarser and more porous surface structure which causes the calcium carbonate to form below the surface of the concrete structure. The unsightly appearance is thereby reduced. However, such cement composition formulation produce concretes of inferior strength and durability. Another suggested method is to apply water-repellant materials, such as silicones or polymers, to the surface of the formed structure. However, such treatments are costly and labor intensive. In addition, they tend to be readily removed by weathering.

It is highly desired to provide a cement capable of inhibiting efflorescence to occur on the surface of cement-based structures.

#### SUMMARY OF INVENTION

The present invention is directed to a cement mixture and resultant cement composition capable of inhibiting the formation of efflorescence on structures formed therewith. The cement mixture comprises hydraulic cement having from 0.05 to 3 weight percent of at least

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one C<sub>14-22</sub> alkyl alcohol. The present invention is further directed to cement compositions composed of hydraulic cement, sand, aggregate (optional) and water which includes at least one C<sub>14-</sub>-C<sub>22</sub> alkyl alcohol in from 0.05 to 3 weight percent based on the weight of hydraulic cement therein.

#### DETAILED DESCRIPTION

The present invention is directed to a hydraulic cement mixture and to cement compositions formed with such cement mixtures.

It has been unexpectedly found that, when at least one fatty alcohol having from 14 to 22 carbon atoms is combined with a hydraulic cement in the amounts and manners fully described herein below, resultant cement compositions formed into structural members exhibit a substantial reduction in efflorescence and of the patchy display thereof.

The compounds which have been unexpectedly found to provide inhibition to efflorescence, especially as caused by lime bloom and lime weeping, are fatty alcohols having 14 to 22 carbon atoms. Examples of such compounds are myristic alcohol, cetyl alcohol, stearyl alcohol and arachidic alcohol, docosanol and the like. The subject alcohols can be used singly or in combination. These fatty alcohols are solids at ambient temperatures and are readily available in commercial quantities. The preferred fatty alcohol is octadecanol (stearyl alcohol).

The subject fatty alcohols can be mixed with hydraulic cement in various manners. The fatty alcohols can be formed into an aqueous emulsion which can be distributed throughout the cement. These emulsions are composed of the subject fatty alcohol and water with a

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conventional emulsifying agent. The emulsifier can be selected from known cationic, anionic or nonionic emulsifiers. Examples of such emulsifiers include sorbitan monooleate, sorbitan monolaurate, polyoxyethylene monooleate, polyoxyethylene hexaoleate, polyoxyethylene esters of mixed fatty acids, polyoxyethylene tridecyl ether, polyoxyethylene sorbitan trioleate and monolaurate and the like. Further, the fatty alcohols found useful under the present invention can be formed into solutions or aqueous emulsions with lower alcohols, such as a C<sub>2</sub>-C<sub>6</sub> aliphatic alcohol as, for example, ethanol, isopropanol, n-butanol, t-butanol and the like, and mixtures thereof. These lower alcohols can also be used alone or in combination with the known emulsifying agents described above.

The aqueous emulsion should contain the subject fatty alcohol in concentrations of from about 0.5 to 5 weight percent. Low concentrations are preferred to enhance the stability of the emulsion admixture. Where higher concentrations are the preferred mode, one would include a lower alcohol. However, the concentration should not be of such a high value as to cause the aqueous emulsion to be too thick to readily disperse. The exact concentration will depend upon the particular fatty alcohol and emulsifier used and can be readily determined by the artisan. The alcoholic solutions may contain the fatty acid in from 0.5 to 50 weight percent.

The presently required fatty alcohols are readily transported as solids. Thus, they can be introduced into and made part of a dry mix of hydraulic cement and the fatty alcohols. Such improved cement can be formed into a substantially uniform mixture having from 0.05 to 3 (preferably 0.05 to 0.5) weight percent of fatty alcohol

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uniformly dispersed therein. For example, the solid fatty alcohol or mixture of such alcohols can be readily mixed with a hydraulic cement, such as portland cement to form a dry powder blend having the required 0.05 to 3  
5 (preferably 0.05 to 0.5) weight percent fatty alcohol based on the cement content. The final blend may contain other conventional materials, such as fillers and conventional cement admixtures and the like.

10 Alternately, the present fatty alcohol can be added (either in its solid state or as an aqueous emulsion to hydraulic cement compositions) either at the ready-mix batching plant or at the job site. It is important that the fatty alcohol be substantially uniformly dispersed in the unset cement composition. Upon curing such  
15 compositions have been found to inhibit efflorescence formation.

The fatty alcohols found useful by the present invention must be present in from 0.05 to 3 weight percent, preferably from 0.05 to 0.5 weight percent based  
20 on the cement of the resultant cement composition. Amounts greater than 3 weight percent is uneconomical and provides no additional benefit to the resultant cement composition structure.

25 The presently described fatty alcohol can be used to inhibit efflorescence with a variety of cement compositions such as mortars (cement, sand and water) and, most importantly, concretes (cement sand, gravel and water). Suitable hydraulic cements include ordinary portland cements (e.g. ASTM Type I), blended cements,  
30 special portland cements such as high early strength cements and the like which are conventionally used in forming structures where the occurrence of efflorescence is an undesired feature.

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The improved cement composition of the present invention is composed of a mortar (hydraulic cement, sand and water) or a concrete (hydraulic cement sand gravel or other large aggregate and water). The composition must contain at least one fatty alcohol ( $C_{14}$ - $C_{22}$  aliphatic alcohol) in from 0.05 to 3, preferably from 0.1 to 0.3 weight percent based on the hydraulic cement content of the composition. The composition may, in addition, contain conventional cement additives, such as water-reducing agents, as, for example, naphthalene sulfonate-formaldehyde condensates, lignin sulfonates and the like; set accelerators, as for example, sodium chloride, calcium chloride, calcium nitrite and the like; as well as other conventional additives for their indented purpose.

The cement composition should contain sufficient water for hydration and to aid in mixing of the constituents into a substantially uniform material. The ratio of water to cement is normally from 0.3 to 0.6 and preferably from 0.35 to 0.5. When the presently required fatty alcohol is introduced into the composition as an aqueous emulsion, the water of the emulsion should be taken into account as part of the overall water of the composition.

The following examples are given for illustrative purposes only and are not meant to be a limitation on the claims appended hereto. All parts and percentages are by weight unless otherwise stated.

#### EXAMPLES

Emulsions of fatty alcohols were formed using a jacketed high speed mixer. In forming each emulsion, the mixer was filled with 149.0 parts water and heated to

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185°F. In each instance, a solid fatty alcohol (0.75 part) and a mixture of emulsifiers composed of a polyoxyethylene sorbitan monolaurate (0.15 part) and sorbitan monostearate (0.15 part) were added to the water. Once the fatty alcohol liquified, the mixture was mixed at high speed for two minutes. The resultant emulsion of each fatty alcohol was milky white and stable.

A series of mortar samples were prepared using a mixture of Type I portland cement and sand in a sand to cement weight ratio of 0.75. Water (for blank) or water plus an emulsion (prepared according to the above describe process) containing each fatty alcohol of octadecanol, eicosanol and docosanol was added to the dry sand/cement mix. The resultant water in each sample was present at a water to cement ratio of 0.40. The octadecanol ( $C_{18}$ ) was added at an amount of 0.05 weight percent octadecanol based on the portland cement of the formed mortar. The eicosanol ( $C_{20}$ ) and docosanol ( $C_{22}$ ) were each added at an amount of 0.1 percent based on the portland cement of the formed mortar.

The formed mortar compositions were mixed until substantially uniform and then cast into 10 inch by 16 inch panels. The panels were cured at 23°C and 40% relative humidity for 18 hours.

Four separate 4 inch by 6 inch areas of each panel's exposed surface was scanned using a NISCAN Spectra Scanner in combination with Signal Analytic's IPLAB Spectrum (2.2.0) scientific imaging processing software to determine the degree of efflorescence exhibited by each tested panel. The unit of measure was pixels with 200 pixels to the linear inch. The scale upon which the degree of efflorescence was measured was from 0 to 256,

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in which "0" value represents a pure white and "256" value represents a pure black with the intermediate values being degrees of grey. The lower the number the greater the degree of efflorescence.

5           The results of the test panels are given in Table I below.

TABLE I

	<u>Fatty Alcohol</u>	<u>Mean Value</u>	<u>Std. Deviation</u>
	None	106.6	19.4
10	Octadecanol	178.2	7.9
	Eicosanol	178.6	10.1
	Docosanol	157.3	11.4

15           The control panel (water only) had a mean gray scale reading of 106.6 with a mean standard deviation of 19.4 units. The test panels treated with octadecanol eicosanol and docosanol had much higher mean grey scale values of 178.2, 178.6 and 157.3, respectively. This clearly indicates that the treated samples were darker due to the dramatic decrease of efflorescence. Further,  
20           the mean standard deviation for the samples were 7.9, 10.1 and 11.4, respectively. This shows that the treatment provided a more uniform surface.

**WHAT IS CLAIMED:**

- 1           1. A dry cement having the capability to reduce efflorescence comprising a substantially uniform mixture of a hydraulic cement having from 0.01 to 3 weight percent of at least one C<sub>14</sub>-C<sub>22</sub> fatty alcohol.
- 5           2. The cement of Claim 1 wherein the fatty alcohol is present in from 0.05 to 0.5 weight percent of said dry cement.
- 10           3. The cement of Claim 1 wherein the fatty alcohol is selected from the group consisting of octadecanol, eicosanol and docosanol.
4. The cement of Claim 2 wherein the fatty alcohol is selected from the group consisting of octadecanol, eicosanol and docosanol.
- 15           5. An improved cement composition comprising hydraulic cement, sand, aggregate and water having substantially uniformly distributed therein from 0.01 to 3 weight percent of at least one C<sub>14</sub>-C<sub>22</sub> fatty alcohol based on dry weight of the cement present.
- 20           6. The composition of Claim 3 wherein the fatty alcohol is in from 0.05 to 0.5 weight percent based on the dry weight of cement present.
- 25           7. The composition of Claim 5 wherein the fatty alcohol is selected from the group consisting of octadecanol, eicosanol and docosanol.

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- 1           8. The composition of Claim 6 wherein the fatty alcohol is selected from the group consisting of octadecanol, eicosanol and docosanol.
- 5           9. The composition of Claim 5 which further contains a C<sub>2</sub>-C<sub>6</sub> aliphatic alcohol.
- 10          10. An improved cement composition comprising hydraulic cement, sand, and water having substantially uniformly distributed therein from 0.01 to 3 weight percent of at least one C<sub>14</sub>-C<sub>22</sub> fatty alcohol-based on the dry weight of cement present.
11. The composition of Claim 10 wherein the fatty alcohol is in from 0.05 to 0.5 weight percent based on the dry weight of cement present.
- 15          12. The composition of Claim 10 wherein the fatty alcohol is selected from the group consisting of octadecanol, eicosanol and docosanol.
13. The composition of Claim 11 wherein the fatty alcohol is selected from the group consisting of octadecanol, eicosanol and docosanol.
- 20          14. The composition of Claim 10 which further contains at least one C<sub>2</sub>-C<sub>6</sub> aliphatic alcohol.
- 25          15. A process for inhibiting efflorescence formation of the surface of hydraulic cement structural formations comprising forming a hydraulic cement composition of Claim 5; casting and curing the formed cement composition into a desired shape.

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1           16. A process for inhibiting efflorescence  
formation of the surface of hydraulic cement structural  
formations comprising forming a hydraulic cement  
composition of Claim 6; casting and curing the formed  
5 cement composition into a desired shape.

          17. A process for inhibiting efflorescence  
formation of the surface of hydraulic cement structural  
formations comprising forming a hydraulic cement  
composition of Claim 7; casting and curing the formed  
10 cement composition into a desired shape.

          18. A process for inhibiting efflorescence  
formation of the surface of hydraulic cement structural  
formations comprising forming a hydraulic cement  
composition of Claim 8; casting and curing the formed  
15 cement composition into a desired shape.

          19. A process for inhibiting efflorescence  
formation of the surface of hydraulic cement structural  
formations comprising forming a hydraulic cement  
composition of Claim 9; casting and curing the formed  
20 cement composition into a desired shape.

          20. A process for inhibiting efflorescence  
formation of the surface of hydraulic cement structural  
formations comprising forming a hydraulic cement  
composition of Claim 10; casting and curing the formed  
25 cement composition into a desired shape.

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1           21. A process for inhibiting efflorescence  
formation of the surface of hydraulic cement structural  
formations comprising forming a hydraulic cement  
composition of Claim 11; casting and curing the formed  
5 cement composition into a desired shape.

          22. A process for inhibiting efflorescence  
formation of the surface of hydraulic cement structural  
formations comprising forming a hydraulic cement  
composition of Claim 12; casting and curing the formed  
10 cement composition into a desired shape.

          23. A process for inhibiting efflorescence  
formation of the surface of hydraulic cement structural  
formations comprising forming a hydraulic cement  
composition of Claim 13; casting and curing the formed  
15 cement composition into a desired shape.

          24. A process for inhibiting efflorescence  
formation of the surface of hydraulic cement structural  
formations comprising forming a hydraulic cement  
composition of Claim 14; casting and curing the formed  
20 cement composition into a desired shape.

## INTERNATIONAL SEARCH REPORT

International Application No  
PCT/US 94/07951A. CLASSIFICATION OF SUBJECT MATTER  
IPC 6 C04B24/02

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)  
IPC 6 C04B

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 practical, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	CHEMICAL ABSTRACTS, vol. 106, no. 14, 6 April 1987, Columbus, Ohio, US; abstract no. 107046d, S. AZUMA 'Castable efflorescence-resistant compositions' page 314 ; see abstract & JP,A,61 242 961 (NICHIBEI ROZAI SEIZO CO. LTD.) 29 October 1986 ---	1,2,5,6, 15,16
X	US,A,3 486 916 (W.A. CORDON) 30 December 1969 see column 1, line 14 - line 61 see column 2, line 19 - line 68 see column 3, line 10 - line 34 see claims 1,2; examples 1,6 --- -/--	1-20

 Further documents are listed in the continuation of box C. Patent family members are listed in annex.

## \* Special categories of cited documents :

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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	FR,A,1 329 281 (K. BRANDT) 29 April 1963  see column 2, line 14 - column 3, line 34 see column 5, line 52 - column 6, line 2; claims 1,7  -----	1,3,10, 12

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/US 94/07951

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
JP-A-61242961	29-10-86	NONE	
US-A-3486916	30-12-69	NONE	
FR-A-1329281		NONE	