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(54) **Fabric conditioners.**

(57) Dilute dispersions of cationic fabric conditioners in water undergo creaming in the presence of dissolved electrolyte to form concentrated, readily dispersible creams having a spherulitic structure and typically containing from 8 to 50% by weight of fabric conditioner and 1 to 15% by weight of electrolyte.

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Description

'FABRIC CONDITIONER'

Fabric conditioners are widely used to soften fabrics after washing in anionic detergents. As used herein the term refers to cationic organic compounds such as amine salts or quaternary nitrogen compounds, all of which have two long chain (e.g. C_{15-22}) alkyl and/or alkenyl groups, and especially two C_{16-18} alkyl and/or alkenyl groups (e.g. tallowyl, oleyl, palmityl and/or stearyl groups).

Among the types of fabric conditioners which have been used are included quaternary ammonium salts and, in particular, distearyl dimethyl ammonium chloride, amido imidazoline salts, quaternised amido imidazolines, amido amine salts quaternised amido amines and ethoxylated amido amines. The amido imidazoline salts and quaternised amido imidazolines may be derived from amido amines by cyclisation. For convenience therefore, amido amine salts, amido imidazoline salts and their quaternised and/or alkoxylated derivatives are referred to collectively herein as "amido amine derivatives". Although they are only very sparingly soluble in water, fabric softeners can be fairly easily dispersed to form dilute aqueous dispersions with concentrations of up to about 20%. However a major problem encountered in the handling and formulation of fabric conditioners is the high viscosity of even the more dilute of such aqueous dispersions.

This is a particular problem for the manufacturer who supplies cationic fabric conditioners in bulk to the formulator, since it is difficult to obtain concentrated dispersions of fabric conditioners in a readily pourable and stable form. The manufacturer is thus concerned to reduce the viscosity of the bulk products, and at the same time to maintain and, if possible increase the relative concentration, in order to reduce the cost and inconvenience of transporting and storing large volumes of water.

The problem for the formulator is more complicated. There is an obvious need for a compromise between increased cost per unit volume and greater difficulty in pouring, on the one hand and increased effectiveness, per unit volume, on the other as the concentration is increased. However, consideration must also be given to the prejudice of the consumer who believes that more viscous products are necessarily more concentrated and therefore more effective, and may purchase a more viscous, but more dilute product despite its being less cost effective and harder to pour.

Control of viscosity, both in concentrated products, and on dilution, is therefore of crucial importance in making and formulating fabric conditioners.

The formulator normally sells compositions containing 3 to 7% fabric conditioner which represents the maximum concentration at which the formulation is sufficiently pourable, although higher concentrations e.g. up to about 15% can be achieved by adding trace amounts, usually of the order 100 to 1,500ppm, of calcium ion, e.g. as calcium chloride, which tends to reduce the viscosity. Concentrations greater than about 20% can only be achieved by using water soluble organic solvents. Manufacturers who need to achieve concentrations of greater than 50%, preferably 75 to 90%, for efficient distribution to the formulators, have therefore had to make substantially anhydrous concentrates in organic solvents such as lower alcohols. In practice the solvent most commonly used is isopropanol, which is expensive, flammable, odourous and may produce undesirable effects in the final formulation, such as variation of dispersion viscocity on ageing. Isopropanol has, however, generally been considered essential to obtain a pourable and concentrated composition.

The concentrate containing the water miscible solvents can be dispersed by stirring in hot water and blended with fatty alcohols, non-ionic surfactants, colouring, perfumes and traces of soluble calcium salts such as calcium chloride, in order to obtain a pourable formulation acceptable to the consumer.

The difficulty of handling concentrates, the need to use hot water and the disadvantages of solvent, have all led manufacturers for many years to look for alternative ways of preparing concentrates, but hitherto without success. It is possible to alleviate some of the problems by using an unsaturated or partly unsaturated feedstock such as oleyl or soft tallow to provide the long chain alkyl/alkenyl group of the fabric conditioner and products have been prepared using feedstocks of a high iodine value (ie high degree of unsaturation).

Unfortunately it has been found that the higher the iodine value of the feedstock, the poorer is the fabric softening effect, and the more prone is the product to oxidation leading to discolouration and malodour.

We have now discovered a novel physical state of aqueous cationic fabric conditioners which enables them to be obtained as mobile creams of high concentration. We have discovered that when electrolytes are included in certain critical concentrations in aqueous dispersions of cationic fabric conditioners a characteristic spherulitic structure is obtained, which typically undergoes spontaneous creaming. The cream is a mobile concentrated aqueous dispersion with good fabric softening properties, which is rapidly dispersible in cold water by stirring or shaking. It is possible to use alkyl feedstocks with low iodine values without sacrificing mobility. The rediluted creams can be obtained in a stable form of satisfactory viscosity. Creams having a similar structure and composition to those obtained by allowing a dilute system to cream can also be prepared directly by mixing an electrolyte solution with the concentrate, until the desired cream concentration is achieved. However the properties of products so prepared are usually inferior to those obtained by actual creaming of a diluted solution. They are, nevertheless, within the scope of our invention and may be preferred in some cases due to their ease of preparation. In general we have found that creams of our invention can be identified by a characteristic spherulitic appearance under the optical microscope. The spherulites show crosses when viewed between cross polarisers. The creams also exhibit characteristic neutron scattering paterns, with peaks at around 50 and 25 Angstroms.

According to one embodiment our invention therefore provides a composition comprising: water and at least one cationic fabric conditioner having two C_{15-22} alkyl or alkenyl groups dispersed in the water, in the presence of dissolved electrolyte in a concentration sufficient (i) to provide an optically anisotropic spherulitic composition and/or, (ii) to cause creaming of a more dilute dispersion of said fabric conditioner.

According to a second embodiment our invention provides a cream formed by the inclusion of sufficient of a water soluble electrolyte in an aqueous dispersion of a cationic fabric conditioner, to cause creaming of the dispersed phase, and separating the cream from remainder of the liquid.

According to a third embodiment our invention provides a composition comprising water, a dissolved electrolyte in a concentration greater than 1% by weight of the composition, and a cationic fabric conditioner comprising at least 40% by weight, based on the total active weight, of an amido amine derivative having two C_{15-22} alkyl or alkenyl groups dispersed in the aqueous electrolyte as optically anisotropic spherulites in a concentration between 18 and 50% by weight.

According to a further embodiment our invention provides a method of manufacture of a fabric conditioner cream which comprises forming a dispersion in water of at least one creamable cationic fabric conditioner, in the presence of a water soluble electrolyte in an amount sufficient to cause the fabric conditioner to cream, and separating the cream from the remainder of the aqueous dispersion. Preferably the creaming is accelerated by centrifuging. The dispersion is conveniently formed by adding a concentrate of the fabric conditioner in a water miscible, organic solvent to an aqueous electrolyte.

Alternatively, our invention provides a method of preparing a fabric softener cream which comprises mixing together an aqueous electrolyte and a solution of from 50 to 80% by weight of a cationic fabric conditioner comprising at least 40% by weight, based on the total active weight, of an amido amine derivative having two C_{15-22} alkyl or alkenyl groups in a water miscible organic solvent to provide a final electrolyte concentration of from 1 to 10% by weight of the composition and a fabric conditioner concentration from 18% to 40% active matter by weight of the composition.

According to our invention we prefer to use quaternised imidazoline fabric conditioners having two long chain, e.g. C 15-20, alkyl and/or alkenyl groups such as ditallowyl or distearyl imidazolinium methosulphate. Unquaternised imidazolinium salts, having two C15-20 alkyl groups are also useful as are mono or di-alkylated diamidoammonium salts having two C1-4 alkyl groups and two C15-20 alkyl groups. Examples include bis (tallowamidoethyl) ammonium, or bis (stearylamidoethyl) ammonium, mono or di-methosulphates.

Generally speaking tetra alkyl ammonium fabric conditioners such as dimethyl ditallowyl ammonium chloride are difficult to cream, tending to form coagulated masses which are difficult to disperse, unless the initial active concentration of the dilute dispersion, and the electrolyte concentration, are relatively dilute, e.g. from 4 to 8% by weight and from 0.3% to 0.5% respectively. The creamed concentrates of such quaternary ammonium salts are correspondingly dilute, compared to amidoamine derivative creams, and therefore of less commercial value. Mixtures of amido amine derivatives with other quaternary fabric conditioners in which the former component is present in concentrations of about 40% or more of the total active weight will generally form acceptable creams according to the invention.

For instance we have creamed mixtures of amido imidazolines with ditallowyl, or distearyl, mon-or di-(lower, e.g. $C_1$to $_4$, alkyl) ammonium salts, such as distearyl dimethyl ammonium chloride, or with ditallowyl or distearyl benzalkonium or pyridinium salts or with alkoxylated (e.g. ethoxylated) amido amines or imidazolines, such as a bis (tallowamidoethyl) mono (1 to 8 mole) polyoxyethylammonium salt or quaternised ammonium salt.

For all the foregoing fabric conditioners the counter ion is usually chloride, sulphate, phosphate methosulphate, ethosulphate, or an organic ion such as formate, acetate, citrate, tartrate, or lactate.

The C15-22 groups (usually predominantly C16 and/or C18 groups) are preferrably straight chain or less preferrably branched chain alkyl groups, which may contain a proportion, preferably minor, of alkenyl groups such as oleyl groups. For maximum softening we prefer to use hard tallow or other alkyl groups having an iodine number less than 10, preferrably less than 5. However for ease of manufacture the partially unsaturated feedstocks are more usually selected.

Preferably, according to our invention, 1 to 20% e.g. 2 to 18%, typically 3 to 16% of cationic fabric conditioner, based on the total weight of the dispersion, is dispersed in water. The water preferably has an elevated temperature between 40° C and boiling e.g. 50 to 80° C. The fabric conditioner is preferably added as a concentrate in a water miscible organic solvent e.g. a mono-or dihydric alcohol or ether alcohol such as methanol, ethanol, propanol, isopropanol, ethylene glycol, ethylene glycol mono methylether, diethylene glycol or diethylene glycol mono methyl ether. The concentrate preferably has a concentration of greater than 50% e.g. 75 to 95% preferably 90% of the fabric conditioner based on the weight of the concentrate. Generally the higher the concentration, the better, consistent with adequate mobility to permit dispersion in, or mixing with, the aqueous electrolyte.

The initial concentration of the fabric conditioner in the dispersion is important to obtain satisfactory creaming and in determining the maximum achievable concentration.

Typically, where the fabric conditioner comprises an amidoamine derivative we prefer that the initially prepared aqueous dispersion has a concentration of from 5 to 15%. the exact concentration will depend on the particular fabric conditioner and the desired final concentration. If the initial concentration is above a critical maximum (typically about 17% in the case of amido amine derivatives, or 8% in the case of tetra alkyl amonium salts) spontaneous creaming may not occur.

However the maximum achievable concentration of the cream is usually about double the initial concentration.

Accordingly we prefer to employ initial concentrations close to the maximum e.g. 10 to 15% by weight, in the case of amido amine derivatives or 6 to 8% in the case of tetra alkyl ammonium salts.

Before or after addition of the fabric conditioner concentrate an electrolyte is dissolved in the water. The electrolyte may be a water soluble, non-flocculating, inorganic, alkali metal or ammonium salt which salts surfactants out of solution, such as lithium, sodium, potassium or ammonium chloride, sulphate, nitrate, acid phosphate, bromide, iodide, phosphonate, bicarbonate, acid silicate, borate, sulphite or phosphite, a water soluble alkaline earth metal salt such as calcium chloride or bicarbonate, or a water soluble alkali metal organic salt which salts surfactants out of solution such as sodium formate or acetate. The electrolyte should preferably not be strongly alkaline. Preferably the addition of the electrolyte to the aqueous dispersion results in pH of less than 10, more preferably less than 9, e.g. less than 8.5. Alkaline electrolytes such as sodium carbonate, sodium tripolyphosphate, trisodium phosphate or alkaline silicate tend to flocculate the fabric conditioner.

The concentration of the electrolyte required to promote creaming depends on the particular conditioner. In the case of imidazolinium fabric conditioners the concentraton is preferably between 3 and 15% by weight of the dispersion, more preferably 5 to 12% by weight e.g. 6 to 10%. In the case of tetra alkyl ammonium fabric conditioners electrolyte concentrations of from 0.3 to 0.5, eg 0.4% are preferred.

Creaming occurs after a few hours standing and proceeds rapidly until the concentration has risen to approximately double the initial concentration e.g. usually about 25 to 40% of the fabric conditioner by weight of the cream.

Usually this occurs within 24 hours. The cream is preferably separated from the clear aqueous phase in which a substantial proportion of the solvent and of the water soluble organic impurities remain, leaving a purified fabric conditioner. Preferably the cream has a fabric conditioner content of from 20 to 40% eg 25 to 35% by weight of the cream.

Creaming may continue at a reduced rate for several weeks, but the cream can easily be redispersed by gentle stirring or shaking.

Centrifuging for a short period at 20 to 1,000G preferably 40 to 800G e.g. 50 to 500G may be used to provide a more stable cream more rapidly with reduced tendencies to undergo further creaming. More vigourous centrifuging e.g. at 20,000G for 2 hours gives a thick, stable, relatively immobile paste which , however, is readily dispersible in water. Preferably the centrifugation is effected on material immediately after mixing. The centrifugation is particularly rapid at elevated temperatures eg 30 to 80°C preferably 50 to 70°C.

It is possible to prepare compositions, having substantially similar compositions to the cream, directly by mixing together the aqueous electrolyte and the concentrated solution of the fabric conditioner in water-miscible organic solvent, in a proportion adapted to provide the desired final concentration. The term "cream" as used herein includes such directly formed composition, where the context so permits. The product differs from that obtained by creaming of a more dilute product in that it normally contains more solvent, and is less homogeneous, more viscous and less readily dispersible. Normally the fabric conditioner is added to the electrolyte solution with stirring until the desired active concentration has been achieved.

The cream produced may be blended directly with perfumes, colourings, and/or other minor ingredients such as preservatives, stabilisers, clays such as bentonite and/or a small amount of the original fabric conditioner concentrate in solvent to stabilise it or enhance its effectiveness, and sold to the consumer as a high concentration product, or it may be dispersed in water, before or after addition of any such additives, to provide more conventional concentrations of the conditioner. The dispersions in water are generally non-creaming provided the electrolyte concentration is sufficiently reduced by dilution, e.g. below 3% by weight.

According to a preferred embodiment of our invention creamed fabric conditioners may be incorporated in liquid detergents, especially non-ionic based liquid detergents.

Preferably a softened liquid detergent according to our invention comprises: up to 30% by weight e.g. 5 to 25% by weight, preferably 8 to 20% by weight of surfactant, which is preferably at least predominantly non-ionic and/or amphoteric surfactant; and up to 15%, preferably 1 to 10%, by weight of active matter, based on the weight of the composition, of fabric conditioner cream.

The detergent may contain sufficient electrolyte to form a solids-supporting structure, e.g. up to 15% in solution, and preferably includes a builder in an amount up to 40% by weight e.g. 5 to 30% of zeolite, sodium tripolyphosphate, potassium pyrophosphate, N.T.A., E.D.T.A. or citrate.

The structure of our novel fabric conditioner cream can readily be identified with an optical microscope. Compositions of our invention show a predominantly spherulitic structure. When the spherulites are observed between crossed polarisers each spherulite shows evidence of a dark cross, similar to the so called "Maltese Cross" texture which is a characteristic of a liquid crystal 'G' phase. Electron microscopy suggests a substructure possibly entailing smaller spherulites agregated in concentric layers to form larger spherulites.

Neutron scattering studies on samples prepared in D_2O at 30% active concentration show two peaks, the first corresponding to a structured feature repeating with a periodicity of about 50 to 60 Angstrom and the second at about half the periodicity of the first e.g. 25 to 30 Angstrom. A conventional fabric conditioner e.g. containing only 100 ppm electrolyte gave peaks corresponding to double these periodicities e.g. 100-120 Angstrom and 50 to 60 Angstrom respectively.

The invention is illustrated by the following examples:

Example 1

An initial concentrate consisting of a 90% by weight solution of ditallowyl imidazolinium methosulphate in isopropanol was added to a solution of 5% by weight calcium chloride in water at 60°C and stirred for 15 minutes to give a 15% by weight active dispersion. After the dispersion had been allowed to stand for 7 days, a mobile cream was separated from a clear aqueous phase.

The cream gave highly effective softening performance compared with a conventional 5% dispersion of the imidazoline in water at equivalent active dosage.

The cream contained 42.3% by weight total solids (57.7% loss of weight on drying) and 0.9% by weight isopropanol.

Example 2

A composition was prepared according to claim 1 but using D_2 O to dilute the initial concentrates and concentrating to 30% active concentration by adding 7% calcium chloride and centrifuging. The composition was submitted to neutron scattering which showed peaks at angles which correspond to regularly repeating structural features at a separation of 57 and 30 Angstrom respectively.

Comparative Example

A sample of a non-creamed fabric softener, prepared by diluting the initial concentrate directly to 30% with D_2O and including conventional levels of electrolyte, i.e. 1000ppm Ca Cl_2, showed peaks corresponding to periodic features with a spacing of 112 and 56 Angstroms respectively, the product showed no evidence under the optical microscope of spherulites, was immobile and very difficult to disperse in water at ambient temperature.

Example 3

A cream type formulation was prepared by adding the initial concentrate directly to aqueous Ca Cl_2 to provide the same active and electrolyte content as the product of example 1. The product was more viscous than Example 1, (see table 1) and was less readily dispersible. It was however substantially more mobile and more readily dispersible than the comparative example. The product had a slightly non-homogeneous "lumpy" appearance.

Example 4

Example 1 was repeated with various electrolytes in place of calcium chloride. The following electrolytes gave compositions which were clearly defined spherulitic compositions under the optical microscope, and which dispersed rapidly in water:-
$(NH_4)_2$ SO_4, K_2HPO_4, NaHCO3, $(NH_4)_2$ SO_3, Na_2 SO_3, K_2SO_4, LiCl, KH_2PO_4, (NH_4) Cl, KCl, Na_2 SO_4, NaCl, $NaHSO_4$, $KHSO_3$.

The following electrolytes gave compositions which showed evidence of lamellar structure in addition to spherulites, but which were relatively easily dispersed:-
KBr, Na Br, NaI, NH_4NO_3, $Na_2B_4O_7$

The following electrolytes gave compositions which showed evidence of flocculation, and which were more difficult to disperse:-
K_2CO_3, Na_2 CO_3 $Na_5P_3O_{10}$, $NaSiO_3$ (alkaline).

It was observed that the alkaline electrolytes, with a pH of 9 or greater tended to cause flocculation. When a flocculated product was prepared in D_2O and examined by neutron scattering peaks, corresponding to periodicities of 52 and 28 Angstrom were observed.

Example 5

A number of samples prepared following the method of example 1, all containing 30% fabric conditioner and 7.5% $CaCl_2$, together with the comparative example (made up in water instead of D_2O) were examined using a "Carrimed" controlled stress rheometer.

The results were analysed using the Herschel-Bulkley model for the Yield Point and the Bingham Body Model for the viscosity averaged over the range: 0 to 700 $sec.^{-1}$ shear and are shown in the table. The results were obtained at 20°C using a cone with a horizontal angle of 2° and base diameter of 4cm and a plate with a 56.6u gap.

Example 6

A fabric softener cream was prepared by the method of example 1 using bis(tallowylamidoethyl) amine hydrochloride as the fabric conditioner. The product was a mobile 30% active cream which dispersed readily in water and was spherulitic under the optical microscope.

Example 7

A 6% by weight aqueous dispersion of ditallowyldimethylammonium chloride and 0.4% $CaCl_2$ was spherulitic under the microscope, mobile, and readily dispersible in water. It underwent slow creaming but failed to give a clear separation. For comparison a 15% dispersion containing 7.5% $CaCl_2$ underwent rapid separation to provide a clear oil having a concentration of 30% which solidified on cooling, and gave an immobile waxy slab (see Table 1) which appeared as a fibrous floc under the microscope and dispersed in water with difficulty. The process was repeated with mixtures of the ditallowyl dimethylammonium chloride (DDAC) and ditallowylimidazoline methosulphate (DIM) in various proportions.

The products showed under the microscope as mixtures of spherulites and floc in substantially the same relative proportions as the proportion of DIM to DDAC.

Up to 50% of DDAC the mixtures were mobile and readily dispersible, (see Table 2), but above 70% DDAC they were immobile pastes.

TABLE 1

FABRIC CONDITIONER 30% ACTIVE CREAM	INITIAL YIELD NM^{-2} (H. BULKLEY)	DYNAMIC YIELD NM^{-2} (H. BULKLEY)	PLASTIC YIELD (BINGHAM)	PLASTIC VISCOCITY (BINGHAM)
ditallowylimidazoline methosulphate	4.77	4.044	19.66	0.184
ditallowylimidazoline acetate	10.44	10.08	18.56	0.1657
ditallowylimidazoline methosulphate + 1% bentonite	-	-	12.14	0.223
ditallowylimidazoline -methosulphate (Ex.3)	28.35	25.38	42.99	2.112
bis(tallowamidoethyl) ammonium methosulphate	19.68	18.88	25.70	0.6334
polyethylene oxy bis-tallow-amidoethyl) ammonium methosulphate	1.79	1.335	6.925	0.1666
ditallowyl(dimethyl-ammonium chloride	22.98	19.21	43.31	0.896
Comparative Example	-	-	71.91	4.67

TABLE 11

WT% DIM	WT% DDAC	INITIAL YIELD HERSCHEL BULKLEY	DYNAMIC YIELD HERSCHEL BULKLEY
100	0	4.774	4.044
90	10	12.53	10.04
80	20	17.9	14.23
50	50	22.98	18.51

Claims

1. A composition comprising: water; at least one cationic fabric conditioner having two C_{15-22} alkyl and/or alkenyl groups, dispersed in the water; and dissolved electrolyte in a concentration sufficient (i) to form said fabric conditioner into an optically anisotropic spherulitic composition, and/or (ii) to cause creaming if it were present at said concentration in a more dilute dispersion of said fabric conditioner.

2. A composition formed by dispersing at least one cationic fabric conditioner having two C_{15-22} alkyl and/or alkenyl groups in water in the presence of sufficient electrolyte to cause creaming thereof, and separating the cream so formed from the rest of the liquid.

3. A composition according to claim 2 wherein said separation is effected by centrifuging the dispersion.

4. A composition comprising: water; electrolyte dissolved in said water in a concentration greater than 1% by weight of said composition; and a cationic fabric conditioner comprising at least 40% by weight,

based on the total active weight, of an amidoamine derivative having two C_{15-22} alkyl and/or alkenyl groups, dispersed in the water as optically anisotropic spherulites.

5. A composition according to any foregoing claim wherein said fabric conditioner comprises at least 40% by weight of an amidoamine derivative, based on the total active weight, and is present in a concentration of from 18 to 50% by weight of said composition.

6. A composition according to claim 5 wherein the concentration of said fabric conditioner is from 20 to 40% by weight of said composition.

7. A compostion according to claim 6 wherein said fabric conditioner is present in a concentration of from 25 to 35% by weight of said composition.

8. A composition according to any foregoing claim wherein said fabric conditioner comprises at least 40% by weight of an amidoamine derivative, based on the total active weight, and said electrolyte is present in a concentration of from 3 to 15% by weight of the composition.

9. A composition according to claim 8 wherein said electrolyte is present in a concentration of from 5 to 12% by weight of said composition.

10. A method of preparing a creamed fabric conditioner which comprises forming a dispersion of a cationic fabric conditioner in water in the presence of sufficient electrolyte to effect creaming of the fabric conditioner, and separating the cream so formed.

11. A method according to claim 10 wherein said dispersion initally contains from 2 to 18% by weight, based on the total weight of the dispersion of a fabric conditioner comprising at least 40%, based on the total active weight, of an amidoamine derivative.

12. A method according to claim 11 wherein the electrolyte is present in a concentration of from 3 to 15% by weight of said dispersion.

13. A method according to any of claims 10 to 12 wherein said separation is effected by centrifuging.

14. A method of preparing a concentrated fabric conditioner which comprises mixing together: a cationic fabric conditioner comprising at least 40%, based on the total active weight, of an amidoamine derivative; water; and electrolyte; in proportions adapted to provided a concentration of from 18 to 50% by weight, based on the total weight of the composition, of said fabric conditioner and from 1 to 15% based on the total weight of the composition of said electrolyte.

15. A liquid detergent comprising water, from 5 to 25% by weight of nonionic and/or amphoteric surfactant and a composition according to any of claims 1 to 9, added in a proportion of from 1 to 10% expressed as weight of active matter based on the total weight of the detergent.

16. A composition according to claim 14 containing from 5 to 30% by weight of a builder.

17. A composition according to any of claims 1 to 10 substantially as described herein with reference to any one of the examples.