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(54) Wax encapsulated bleach particles and method for making same.

(57) Bleach particles encapsulated in a single coat of paraffin wax, the wax having melting point of 40 to 50 °C and needle penetration value of 10 to 60 mm at 25 °C. The coat comprises 35 to 55% by weight of the particle and may be from 100 to 1,000 microns thick. The coat prolongs the time in which chlorine bleach particles encapsulated therewith may remain active in aqueous environments.

The encapsulated particle is made by spraying molten wax on to the particles in a fluidized bed. Liquid or powder cleaning compositions, particularly automatic dishwashing liquid detergents, may incorporate 0.1-15% of the single coat wax-encapsulated bleach particles.

WAX-ENCAPSULATED BLEACH PARTICLES AND METHOD FOR MAKING SAME

Field of the Invention

This invention concerns paraffin wax-encapsulated bleach particles which remain stable for use in liquid and granular cleaning products, and a method for encapsulating the bleach.

BACKGROUND TO THE INVENTION

Bleach particles have been coated with a variety of materials. In U.S. 3,908,045 (Alterman et al.), bleach particles were coated with fatty acids, polyvinyl alcohol or polyethylene glycols. U.S. 4,078,099, 4,126,717, and 4,136,052 (Mazzola) coated bleach particles with a mixture of 35-89% by weight of fatty acid and 1-16% by weight of microcrystalline wax, the wax having melting point of 51-99° C. Other coating materials have included polymer latex, U.S. 4,759,956 (Amer et al.); polycarboxylate materials, U.S. 4,762,637 (Aronson et al.); polyethylene waxes of melting point 50-65° C, EP 132,184 (Scotte); and various waxes, U.S. 4,421,669 (Brichard). The wax coat in Brichard constitutes 0. 01-10% of the weight of the bleach to be coated.

One drawback of conventionally coated bleach particles is the instability of the bleach in liquid aqueous cleaning compositions: water or other components of the composition which are incompatible with bleach interact with the bleach during storage. The result is that little bleach activity remains as a cleaning agent.

Attempts have been made to increase the stability of encapsulated bleach particles by applying a second coat. Thus, Alterman et al. taught optionally applying a second coat of soap to the encapsulated bleach. And U.S. 4,657,784 (Olson) taught doubly coating the bleach core in an inner coat of paraffin or microcrystalline waxes having melting points of 40-94° C and a second coat of material having a melting point higher than the inner coat such as sodium carbonate. Encapsulating bleach in an inner coat of fatty acid or waxes and an outer coat of water-soluble cellulose ether has also been taught, European Patent Application 307,587 (Olson). Second coats are said to improve stability of the bleach capsules, because fissures or gaps in the first coat may allow materials to contact and react with the bleach.

These second coats are costly to apply and, while they raise the bleach stability somewhat, do not guarantee that bleach will be available as a cleaning agent after storage.

Bleach has been encapsulated by a variety of methods. U.S. 3,847,830 (Williams et al.) describes several methods for enveloping normally unstable peroxygen compounds in water-dispersible coatings including paraffin waxes. A coating material is "water-dispersible" if, within 30 minutes of adding 2 g of enveloped peroxygen to 1 liter of water at 15° C, at least 75% of the peroxygen is released. Three of the methods of Williams et al. require the enveloping agent to be molten prior to spraying on to the peroxygen particles in a fluidized bed. Two other methods involve dissolving the enveloping agent in an organic solvent and either spraying the resultant solution on to the particles or immersing them in the bulk solution to achieve coating. Disadvantages of these two methods are the expense of organic solvents and, more importantly, the associated environmental pollution problems.

U.S. 3,856,699 (Miyano et al.) describes a process of dispersing core particles under heating into a waxy material, cooling the resultant dispersion and crushing this into a powder. Thereafter, the powdered waxy material is agitated in an aqueous medium at a temperature higher than the melting point of the waxy material. Waxed core material is then passed into a non-agitated aqueous medium at a temperature lower than the melting point of the waxy material. Co-pending U.S. Patent Application Serial Number 202,853 (Kamel et al.) teaches the steps of dispersing active material in melted wax to form an active material/wax dispersion; adding the dispersion to-water dontaining at least one surfactant and emulsifying the active material/wax dispersion for no longer than 4 minutes therein to form capsules; cooling immediately thereafter said capsules; and retrieving the cooled capsules from the water to effect capsules of improved quality.

Bleach particles have also been directly sprayed with coating material in fluidized bed apparatuses, as in Brichard. Thus, in U.S. 3,908,045 fatty acid coating material was sprayed on to particles. And in U.S. 3,983,254 the spray height of the spray nozzle above the fluidized bed was said to be critical. In U.S. 4,078,099 a rotating drum device was used to apply coating material. Also in U.S. 4,759,956 polymeric latex was sprayed on to core materials (such as bleach) in a fluidized bed operated in a "Wurster" mode.

OBJECTS OF THE INVENTION

One object of the invention is to provide a single-coat wax-encapsulated bleach particle which has improved stability to degradation by ambient humidity or aqueous liquid media.

Another object is providing wax-encapsulated bleach particles which have a smooth, uninterrupted coating with excellent surface integrity.

A further object is producing such encapsulated particles by a process which avoids improper coating and the resultant problems of poor bleach stability and particle agglomeration.

Another object is to provide an encapsulated bleach having a coat which melts or softens sufficiently to release bleach early in most automatic dishwashing wash cycles.

A still further object of the invention is to provide an encapsulation process which is free of organic solvents that lead to environmental pollution problems.

Another object of the invention is to provide a process which operates with a minimum of processing steps.

Yet another object of the invention is to provide a liquid or solid cleaning composition containing the aforementioned single-coat, wax-encapsulated bleach particle, which capsule imparts stable bleach activity without leaving waxy soil after washing. An even more specific object is to provide stable bleach activity to liquid dishwashing or other hard surface cleaner which also contain oxidation-sensitive components such as enzymes, perfumes, fabric softeners, structurants and surfactants.

These and other objects of the present invention will become apparent as further details are provided in the subsequent discussion and Examples.

SUMMARY OF THE INVENTION

In a first aspect, the invention comprises an encapsulated bleach particle suitable for use in household cleaning products. Bleach forms the core of these particles and constitutes from 45 to 65%, preferably 50-60%, by weight of the final particles (i.e., the core plus the coat). A single-wax coat on the particles comprises 35-55% by weight of the particle, or preferably 40-55% by weight, or most preferably 45-55% by weight, and is selected from one or more low-melting point paraffin waxes having melting points of from about 40°C to about 50°C and penetration values of 10-60 mm at 25°C. The single-wax coat having thickness of 100 to 1,000 microns thick is applied to the bleach particles. Preferably, the coat thickness is from 200 to 750 microns and most preferably from 300 to 600 microns.

In a second aspect, the invention comprises a process of making the encapsulated bleach particles. This process comprises the steps of spraying molten paraffin wax having low melting point, i.e. melting point of from about 40°C to about 50°C, on to uncoated bleach particles in a fluidized bed. The bed temperature may be from 5°C up to about 50°C less than the melting point of the wax. The atomisation temperature of the molten wax being applied to the particles is at least from 5 to 40°C greater than the melting point of the wax. A single-wax coat having thickness of 100 to 1,000 microns thick is applied to the bleach particles. The rate of application of the wax coat is from 10 to 40 grams per minute per kilogram of bleach particles in the fluidized bed.

The fluidized bed may be operated in the top spray or Wurster spray mode. Where the top spray is used, an annealing step may advantageously follow the coating step in order to impart an uninterrupted surface and excel lent surface integrity to the coat. When the fluidized bed is operated in the Wurster spray mode, no annealing step is necessary.

In a third aspect, the invention comprises cleaning compositions which include 0.1 to 30% by weight of these encapsulated bleach particles. The compositions may further comprise 0.1-15% surfactant, 1-75% builder, and other components. These compositions leave little or no waxy soil on surfaces they clean.

BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 is a graph of the amount of wax coating which remains unemulsified through an automatic dishwashing cycle, as described in Example III.

Figure 2 is a graph of the spotting performance from autodish liquids containing bleach encapsulated with waxes of different melting points, as described in Example IV.

Figure 3 is a graph of chlorine released by bleach encapsulated with waxes of different melting points, as described in Example VII.

DETAILED DESCRIPTION OF THE INVENTION

The Encapsulated particle

The encapsulated bleach particle of the invention comprises 35-55% by weight of the particle of a single coat of paraffin wax and 45-65% by weight of a core of bleach suitable for use in household cleaning compositions. Preferably, the paraffin wax coating comprises 40-50% by weight of the particle and the core 50-60% by weight of the particle.

The bleach to be encapsulated in the paraffin wax coating may be a chlorine- or bromine-releasing agent or a peroxygen compound. Among suitable reactive chlorine-or bromine-oxidizing materials are heterocyclic N-bromo and N-chloro imides such as trichloroisocyanuric, tribromoisocyanuric, dibromoisocyanuric and dichloroisocyanuric acids, and salts thereof with water-solubilizing cations such as potassium and sodium. Hydantoin compounds such as 1,3-dichloro-5,5-dimethyl-hydantoin are also quite suitable.

Dry, particulate, water-soluble anhydrous inorganic salts are likewise suitable for use herein such as lithium, sodium or calcium hypochlorite and hypobromite. Chlorinated trisodium phosphate is another core material. Chloroisocyanurates are, however, the preferred bleaching agents. Potassium dichloroisocyanurate is sold by Monsanto company as ACL-59 ®. Sodium dichloroisocyanurates are also available from Monsanto as ACL-60®, and in the dihydrate form, from the Olin Corporation as Clearon CDB-56 ®, available in powder form (particle diameter of less than 150 microns); medium particle sise (about 50 to 400 microns); and coarse particle size (150-850 microns). Very large particles (850-1700 microns) are also found to be suitable for encapsulation.

Organic peroxy acids may be utilized as the bleach core. The peroxyacids usable in the present invention are solid and, preferably, substantially water-insoluble compounds. By "substantially water-insoluble" is meant herein a water-solubility of less than about 1% by weight at ambient temperature. In general, peroxyacids containing at least about 7 carbon atoms are sufficiently insoluble in water for use herein.

Typical monoperoxy acids useful herein include alkyl peroxy acids and aryl peroxyacids such as:

(i) peroxybenzoic acid and ring-substituted peroxybenzoic acids, e.g. peroxy-alpha-naphthoic acid;

(ii) aliphatic and substituted al-iphatic monoperoxy acids, e.g. peroxylauric acid and peroxystearic acid;

Typical diperoxy acids useful herein include alkyl diperoxy acids and aryldiperoxy acids, such as:

(iii) 1,12-diperoxydodecanedioic acid;

(iv) 1,9-diperoxyazelaic acid;

(v) diperoxybrassylic acid; diperoxysebacic acid and diperoxyisophthalic acid;

(vi) 2-decyldiperoxybutane-1,4-dioic acid.

Inorganic peroxygen-generating compounds may also be suitable as cores for the particles of the present invention. Examples of these materials are salts of monopersulphate, perborate monohydrate, perborate tetrahydrate, and percarbonate.

The coating materials which are suitable for encapsulating the bleach core particles are paraffin waxes which have low melting points, i.e. between about 35° C and about 50° C and penetration values of from 10 to 60 mm at 25° C.

This melting point range for the coat is desirable for several reasons. First, the minimum of 40° C generally exceeds any storage temperatures that are encountered by cleaning compositions. Thus, the wax coat will protect the bleach core throughout storage of the cleaning composition. The 50° C melting point cap for the wax coat was selected as providing a wax which will quickly melt or soften early in any automatic dishwashing wash cycle. Melting or softening sufficient to release bleach core will occur because operating temperatures in automatic dishwashers are usually between 40 and 70° C. Thus, the paraffin waxes of the invention will release bleach when the capsule is exposed to the warmed wash bath, but not before.

Moreover, melted paraffin waxes of the capsules of the invention will remain substantially molten at 40-50° C. Such molten wax is easily emulsified by surfactant elements in cleaning compositions. Consequently, such waxes will leave less undesirable waxy residue on items to be cleaned than waxes with higher melting points.

As a class, paraffin waxes have a melting point range of roughly 30 to 80° C and are constituted largely of normal alkanes with low levels of isoalkanes and cycloalkanes. Isoalkanes and cycloalkanes contribute to lack of order in solid wax structures and paraffin waxes are largely crystalline when solid. Thus, the wax coat should not include any paraffins having melting point substantially above 50° C, lest the higher melting point components remain solid throughout the wash cycle and form unsightly residues on surfaces to be cleaned.

The amount of solids in a wax at any given temperature may be determined by Differential Scanning Calorimetry. The more solids present in a wax at room temperature, the more suitable the wax is for the present invention: this is because such solids strengthen the wax coating, rendering the particle less vulnerable to ambient moisture or a liquid aqueous environment, whereas "oil" or liquid wax softens the wax and thereby provides poorer protection for the bleach particle. The wax solids content as measured by Differential Scanning Calorimetry for suitable paraffin waxes may range from 100 to 40%, optimally from 100 to 75%, at 40° C and from 0-15%, and preferably 0-5% at 5° C.

In contrast to paraffin waxes, micro-crystalline waxes have generally higher molecular weights and melting points. Thus, the melting point range for micro-crystalline waxes is from about 50 to about 100° C. Moreover, micro-crystalline waxes are more viscous in the molten state than paraffin waxes and softer than paraffin waxes when solid. Particles coated with micro-crystalline waxes would therefore have a poorer protective coating, and the wax coat which melts from such particles would be less likely to emulsify in cleaning compositions. Thus, micro-crystalline waxes are not considered within the operative scope of this invention.

The Penetration Test (ASTM D-1321) is the standard industrial test for hardness of waxes. This test measures the depth in tenths of a millimeter that a needle of a certain configuration under a given weight penetrates the surface of a wax at a given temperature. Paraffin waxes suitable for use in encapsulating the bleach particles have penetration values of from 10 to 60 mm at 25° C.

Commercially available paraffin waxes which are suitable for encapsulating bleach particles include Merck 7150 and 7151, ex E. Merck of Darmstadt, West Germany; Boler 1397 ex Boler (penetration value of 40 mm at 250° C); Ross fully refined paraffin wax 115/120 ex Frank D. Ross Co., Inc. (penetration value of 40-50 mm at 25° C); and Altafin "Okerin-2033" of Durachem Inc. (penetration value of 15-20 mm at 25° C). Most preferred is Boler 1397.

The Process of Encapsulating Bleach Particles

The process steps of encapsulating bleach particles comprise:

a) suspending bleach particles in fluid bed,

b) selecting one or more paraffin waxes to provide the coating, the waxes having melting point between about 40° C and 50° C, needle penetration value of 10-60 mm at 25° C,

c) heating the one or more paraffin waxes to a temperature sufficiently above the melting temperature to melt all the wax,

d) fluidizing the bed by passing warm air through the bleach particles, so as to maintain a bed temperature of at least from -5 to no more than 50° C, and

e) spraying the melted paraffin wax on to the fluidized bed at an atomization temperature of at least from 5 to 10° C above the melting temperature of the wax for a time sufficient to form a continuous, coherent paraffin wax coating of a thickness of from 10 to 3,000 microns on the bleach particles.

The amount of coating applied to the bleach core particles is typically from about 35 to 55%, preferably about 45 to 50% by weight of the total particle (i.e. core plus coating).

The coating is applied in a fluidized bed. There are several methods of operating a fluidized bed. In a common fluidized bed operation, air is introduced into the bed from below while the coating material is sprayed on to the fluidized material from above. The particles move randomly in the bed in this top spray operation.

Unless precautions are taken in applying molten coating materials in fluidized beds, the resulting material can be poorly coated or, alternatively, agglomerated together. These equally undesirable results follow from the temperature settings in operating the fluidized bed. For example, when the temperature of the bed is too far below that of the molten wax, the molten wax begins to solidify as soon as it enters the cool bed region. Thus, the wax loses some of its ability to adhere to the surface of the bleach particles, and the wax itself quickly solidifies. When this occurs, the fluidized bed is operating to spray dry the wax with little or no coating on the bleach. The poorly coated bleach particles consequently have little stability from ambient humidity or an aqueous liquid environment. Alternatively, when the bed temperature is too high, the wax which does contact bleach particles fails to cool and dry, and so remains soft and sticky. Consequently, bleach particles clump and agglomerate. It becomes difficult to control the size of the resulting clumps. These large clumps can be so large that they fail to melt in the washing cycle, and so remain as solid soil on materials to be cleaned. Thus, improper control of the fluidized bed temperatures can produce encapsulated bleach which fails to meet one of the objects of the invention.

Applicants have discovered that, even with the coatings of up to 1,000 micron thickness, proper control of the bed temperature and the atomization temperature in a fluidized bed avoids both agglomeration and insufficient coatings. Thus, when the bed temperature is from 20° C to no higher than the melting point of the wax, "spray drying" of the wax and agglomeration of coated bleach particles is reduced. Preferably, the bed temperature is to 35° C, and most preferably 25 to 32° C.

Applicants have further discovered that atomization temperature, or the temperature at which the wax is sprayed from a nozzle on to the fluidized bed, is advantageously held at from at least about 5 to 10° C above the melting temperature of the wax. When the top spray mode is used, the maximum atomization temperature is about 35° C greater than the wax melting point; above this temperature, too great a percentage of the particles agglomerate. When the Wurster mode is used to coat particles, the atomization temperature may be as high as 50° C and more above the wax melting point temperature. This is found to be a practicable atomization temperature, despite the expectation that partially coated particles with molten coats would stick to the spray nozzle. It is instead found that the air flow is strong enough to detach these partially coated particles. Alternatively, Applicants have found that the temperature of the molten wax may be maintained substantially above the wax melting point, e.g. from 50 to 100° C above the melting point. When this is the case, the atomization temperature is preferably near or even below the melting temperture of the wax, in order to lower the wax temperature sufficiently to solidify quickly on bleach particles in the fluidized bed.

There are many commercially available fluid bed apparatuses which are suitable for use in the process of the invention; among these are the GPCG-50 and GPCG-60 models of Glatt Air Techniques. These can coat 10 to 50 kg loads of bleach particles in from 0.5 to 3 hours. Table top encapsulation may be carried out in laboratory scale apparatuses as well, as for example in Granuglatt Model No. WSG-3, ex Glatt Air Techniques.

Applicants were surprised to discover that encapsulated particles made by the process of the invention have improved stability to ambient humidity when in powder cleaning products and to aqueous media when in liquid products. This increased stability results regardless of whether the bleach is encapsulated by top spray or Wurster modes in the fluidized bed. The increased stability is demonstrated in Examples V and VII.

An alternative to the top spray of molten coating material is the Wurster spray mode. This method is described in detail in U.S. Patent 3,253,944, which is hereby incorporated by reference. In general, fluidized beds are characterized by randomness of particle motion. Random motion is undesirable when coating particles because of the resultant slow coating rates. To overcome this problem, a cyclic flow pattern is established in the Wurster spray mode by controlled velocity differences. The Wurster mode involves use of a vertically disposed coating tower wherein particles are suspended in an upwardly flowing air stream entering the bottom of the tower. This air stream imparts controlled cyclic movement to the particles with a portion of the suspended bed flowing upwardly inside the tower and the other portion downwardly outside the tower. All of the coating material is directed into the high velocity air stream to provide coating of the particles moving upwardly in the tower. The fluid coating solidifies on the surface of the particles as the air stream lifts them away from the nozzle. The particles are carried to the top of the tower, from which point they fall to the base of the tower along a path outside the tower. At the base, the particles are drawn in through openings and re-directed upwardly in the air stream inside the tower. This cycle is repeated until the desired amount of coating has been deposited on the particles.

Given the steps of Wurster, it was believed that the Wurster mode would be inappropriate for encapsulating bleach particles in wax. The spray nozzle for Wurster is located at the bottom of the fluidized bed and sprays coating materials upwards. It was believed this configuration of the spray nozzle would lead to clogging of the spray noz zle when coated and agglomerated particles fell from the upward air spray into the noszle area. This risk seemed especially high because the nozzle temperature is generally above the melting point of the wax coating. However, Applicants have surprisingly discovered that use of the Wurster spray mode results in many benefits.

Thus, agglomeration of coated bleach particles may be further reduced when operating a fluid bed in the Wurster method. Whereas some 5-15% of the particles coated by top spray may agglomerate, and so be unusable, the level of agglomerated particles from the Wurster application of a fluidized bed does not usually exceed 5% of the particles.

Moreover, the coating time under Wurster can take half as long as top spray, or less, even with a substantially lower air flow rate, as demonstrated in Example 1 below. And although batch size is often smaller than in top spray, and the rate of spraying wax on to the core is not substantially higher in the Wurster mode, still the production rate of the encapsulated bleach particles may be as much as 2 to 3 times higher by the Wurster mode. This higher production rate may be maintained even when the air flow rate through the fluidized bed is lower than for the top spray mode. Thus, higher production rates with lower air flow rates in the Wurster mode produce particles with less agglomeration than the top spray mode.

Applicants have further discovered that performing an additional annealing step after coating the bleach particles in a top spray fluidized bed further improves the capsules. "Annealing" is the name given to a further heating of wax-encapsulated bleach particles at a temperature greater than room temperature but below the wax melting point. This heating step is performed with the bed being fluidized, i.e. with warm air flowing through it; however, no molten wax is being sprayed on to the particles during annealing. The annealing step renders the wax mobile enough that it fills in gaps and cracks in its surface, thus providing a better seal to the bleach within.

The temperature chosen for annealing is one which softens the wax without rendering it sticky. Typically, this temperature is from 5 to 15° C greater than the bed temperature during coating, and from 3 to 15° C less than the melting point of the wax coating. For example, when the wax has melting point of 46° C, the annealing temperature may be about 33-34° C. The bed temperature during spraying is only about 31-32° C, for above 32° C there is a good chance of the particles agglomerating: the high temperature of the molten wax, combined with an annealing temperature, would so soften the wax that particles would agglomerate in the fluidized bed. However, when no hot molten wax is being sprayed on to the particles, an annealing temperature alone in the bed is not warm enough to cause agglomeration.

Most preferably, annealing should be performed for a period between 10 minutes and 48 hours, optimally between about 1 and 24 hours. Mixing the capsules with an inert material, such as an amorphous silica, alumina or clay, prevents capsule sticking during the annealing process. Incorporation of the inorganic annealing adjunct allows use of higher temperatures during the annealing process, thus shortening the annealing period. Adjuncts may be used in an amount relative to the weight of the overall capsule in the ratio of 1:200 to 1:20, preferably 1:100.

A further advantage discovered by Applicants in using the Wurster spray mode is that no annealing step is needed. More accurately, self-annealing occurs automatically as part of the coating process when the Wurster mode is used. The hot molten wax droplet contacting the partly coated bleach particle causes the solid wax already on the particle to melt and to fill any cracks in the wax surface. Unlike the spray-coated particles in top spray mode, which fall into a crowded mass of other particles in the fluidized bed, the particles in the Wurster mode move out of the spray tower and fall through the less crowded space outside the tower. In this space, the particles have time to cool slowly. The smaller charge of bleach particles to be coated in Wurster also reduces the likelihood of contacting other particles while cooling. The result is annealing as part of the coating process.

The Cleaning Compositions incorporating the Encapsulated Particle

The wax-encapsulated bleach capsules of the invention may be incorporated into a variety of cleaning compositions. These compositions include fabric washing, fabric softening, automatic machine dishwashing, light duty dishwashing and hard surface cleaning powder and liquid compositions. Most of these compositions will contain from about 1-75% of a builder component and 0.001 to 5% of a perfume component. Certain of the foregoing type of products will also contain from about 0.01 to about 15% of a surfactant, preferably about 0.5% to about 10% by weight of the composition. Wax-encapsulated chlorine bleach is especially suitable for automatic dishwashing liquid or "gel" detergent products where capsules will be present in an amount of 0.1 to 15% by weight of the composition. Automatic dishwashing detergent powders and liquids will have the composition listed in Table I.

Table I

Automatic Dishwashing Detergent Compositions		
	Per cent by Weight	
Components	Formulation	Formulation
Builder	5-70	10-60
Nonionic Surfactant	1-15	0.01-3
Silicate	1-20	5-20
Filler	0-60	-
Bleaching Agent	0.1-20	0.1-20
Thickener	0-5	0-5
Perfume	0-5	0-5
Water	till 100	till 100

Gels differ from liquids in that gels are primarily structured by polymeric materials and contain only low levels of clay.

Detergent Builder Materials

The cleaning compositions of this invention can contain all manner of detergent builders commonly taught for use in automatic dishwashing or other cleaning compositions. The builders can include any of the conventional inorganic and organic water-soluble builder salts, or mixtures thereof, and may comprise from about 5 to about 90% by weight of the cleaning composition.

Typical of the well-known inorganic builders are the sodium and potassium salts of the following: pyrophosphate, tripolyphosphate, orthophosphate, carbonate, bicarbonate, sesquicarbonate and borate. Other non-phosphorus salts including crystalline and amorphous aluminosilicates may be used as well.

Particularly preferred builders can be selected from the group consisting of sodium tripolyphosphate, sodium carbonate, sodium bicarbonate and mixtures thereof. When present in these compositions, sodium tripolyphosphate concentrations will range from about 2% to about 40%, preferably from about 5% to about 30%. Sodium carbonate and bicarbonate, when present, can range from about 10% to about 50%, preferably from about 20% to about 40% by weight of the cleaning compositions. Potassium pyrophosphate is a preferred builder in gel formulations, where it may be used at from about 3 to about 30%, preferably from about 10 to about 20%.

Organic detergent builders can also be used in the present invention. They are generally sodium and potassium salts of the following: citrate, nitrilotriacetates, phytates, polyphosphonates, oxydisuccinates, oxydiacetates, carboxymethyloxy succinates, tetracarboxylates, starch, oxidized heteropolymeric polysaccharides, and polymeric polycarboxylates such as polyacrylates of molecular weight of from about 5,000 to about 200,000. Polyacetal carboxylates such as those described in U.S. Patents Nos. 4,144,226 and 4,146,495 may also be used.

Sodium citrate is an especially preferred builder. When present, it is preferably available from about 1% to about 35% of the total weight of the detergent composition.

The foregoing detergent builders are meant to illustrate but not limit the types of builder that can be employed in the present invention.

Surfactants

Surfactants may be incorporated in the household cleaning product incorporating the encapsulated bleach particles. Useful surfactants include anionic, nonionic, cationic, amphoteric, zwitterionic types and mixtures of these surface-active agents. Such surfactants are well known in the detergent art and are described at length in "Surface Active Agents and Detergents", Vol. II, by Schwartz, Perry & Birch, Interscience Publishers, Inc. 1959, herein incorporated by reference.

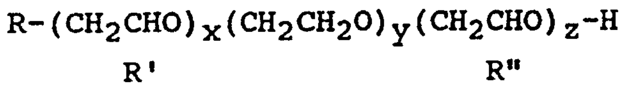
Anionic synthetic detergents can be broadly described as surface active compounds with one or more negatively charged functional groups. Soaps are included within this category. A soap is a C_8-C_{22} alkyl fatty acid salt of an alkali metal, alkaline earth metal, ammonium, alkyl-substituted ammonium or alkanol ammonium salt. Sodium salts of tallow and coconut fatty acids and mixtures thereof are most common. Another important class of anionic compounds are the water-soluble salts, particularly the alkali metal salts, of organic sulphur reaction products having in their molecular structure an alkyl radical containing from about 8 to 22 carbon atoms and a radical selected from the group consisting of sulphonic and sulphuric acid ester radicals. Organic sulphur-based anionic surfactants include the salts of C_{10}-C_{16} alkylbenzene sulphonates, C_{10}-C_{22} alkane sulphonates, C_{10}-C_{22} alkyl ether sulphates, C_{10}-C_{22} alkyl sulphates, C_4-C_{10} dialkylsulphhosuccinates, C_{10}-C_{22} acyl isethionates, alkyl diphenyloxide sulphonates, alkyl napthalene sulphonates, and 2-acetamido hexadecane sulphonates. Two suitable commercially available anionic surfactants are mono- and di- C_8-C_{14} alkyl diphenyl oxide mono- and/or di-sulphates sold under trade names Dowfax 2A-1 ® and Dowfax 3B-2 ® by Dow Chemical Co. Organic phosphate-based anionic surfactants include organic phosphate esters such as complex mono- or di-ester phosphates of hydroxyl-terminated alkoxide condensates, or salts thereof. Included in the organic phosphate esters are phosphate ester derivatives of polyoxyalkylated alkylaryl phosphate esters, of ethoxylated linear alcohols and ethoxylates of phenol. A surfactant which is particularly suitable for combination in cleaning compositions with the wax-encapsulated bleach is Emphos CS-1361 ®, believed to be complex mono- or di-ester of ethoxylated nonyl phenols ex Witco Chemical Co., Inc. Also included are nonionic alkoxylates having a sodium alkylene carboxylate moiety linked to a terminal hydroxyl group of the nonionic through an ether bond. Counter-ions to the salts of all the foregoing may be those of alkali metal, alkaline earth metal, ammonium, alkanolammonium and alkylammonium types.

Nonionic surfactants can be broadly defined as compounds produced by the condensation of alkylene oxide groups with an organic hydrophobic material which may be aliphatic or alkyl aromatic in nature. The length of the hydrophilic or polyoxyalkylene radical, which is condensed with any particular hydrophobic group, can be readily adjusted to yield a water-soluble compound having the desired degree of balance between hydrophilic and hydrophobic elements. Illustrative, but not limiting examples, of various suitable nonionic surfactant types are:

(a) polyoxyethylene or polyoxypropylene condensates of aliphatic carboxylic acids, either linear- or branched-chain and unsaturated or saturated, containing from about 8 to about 18 carbon atoms in the aliphatic chain and incorporating from 5 to about 50 ethylene oxide and/or propylene oxide units. Suitable carboxylic acids include "coconut" fatty acids (derived from coconut oil) which contain an average of about 12 carbon atoms, "tallow" fatty acids (derived from tallow-class fats) which contain an average of about 18 carbon atoms, palmitic acid, myristic acid, stearic acid and lauric acid.

(b) polyoxyethylene or polyoxypropylene condensates of aliphatic alcohols, either linear- or branched-chain and unsaturated or saturated, containing from about 6 to about 24 carbon atoms and incorporating from about 5 to about 50 ethylene oxide and/or propylene oxide units. Suitable alcohols include "coconut" fatty alcohol, "tallow" fatty alcohol, lauryl alcohol, myristyl alcohol and oleyl alcohol. Particularly preferred nonionic surfactant compounds in this category are the "Neodol"-type products, a registered trademark of the Shell Chemical Company.

Also included within this category are nonionic surfactants having the formula:



$$R-(CH_2\underset{R'}{C}HO)_x(CH_2CH_2O)_y(CH_2\underset{R''}{C}HO)_z-H$$

wherein R is a linear alkyl hydrocarbon having an average of 6 to 10 carbon atoms, R' and R'' are each linear alkyl hydrocarbons of about 1 to 4 carbon atoms, x is an integer from 1 to 6, y is an integer from 4 to and 2 is an integer from 4 to 25. A particularly preferred example of this category is Poly-Tergent SLF-18, a registered trademark of the Olin Corporation, New Haven, Conn. Poly-Tergent SLF-18 has a composition of the above formula where R is a C_6-C_{10} linear alkyl mixture, R' and R'' are methyl, x averages 3, y averages 12 and z averages 16. Also suitable are alkylated nonionics as are described in U.S. Patent 4,877,544 (Gabriel et al.), incorporated herein by reference.

(c) polyoxyethylene or polyoxypropylene condensates of alxyl phenols, either linear- or branched-chain and unsaturated or saturated, containing from about 6 to 12 carbon atoms and incorporating from about 5 to about 25 moles of ethylene oxide and/or propylene oxide.

(d) polyoxyethylene derivatives of sorbitan mono-1 di-, and tri-fatty acid esters wherein the fatty acid component has between 12 and 24 carbon atoms. The preferred polyoxyethylene derivatives are of sorbitan monolaurate, sorbitan trilaurate, sorbitan monopalmitate, sorbitan tripalmitate, sorbitan monostearate, sorbitan monoisostearate, sorbitan tristearate, sorbitan monooleate, and sorbitan trioleate. The polyoxyethylene chains may contain between about 4 and 30 ethylene oxide units, preferably about 20. The sorbitan ester derivatives contain 1, 2 or 3 polyoxyethylene chains dependent upon whether they are mono-, di or tri-acid esters.

(e) polyoxyethylene-polyoxypropylene block copolymers having the formula:

$HO(CH_2CH_2)_aCH(CH_3)CH_2O)_b(CH_2CH_2O)_cH$

wherein a, b and c are integers reflecting the respective polyethylene oxide and polypropylene oxide blocks of said polymer The polyoxyethylene component of the block polymer constitutes at least about 40% of the block polymer. The material preferably has a molecular weight of between about 2,000 and 10,000, more preferably from about 3,000 to about 6,000. These materials are well known in the art. They are available under the trademark "Pluronics", a product of BASF-Wyandotte Corporation.

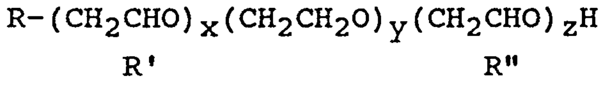
Amphoteric synthetic detergents can be broadly described as derivatives of aliphatic and tertiary amines, in which the aliphatic radical may be straight chain or branched and wherein one of the aliphatic substituents contains from about 8 to about 18 carbons and one contains an anionic water-solubilizing group, i.e. carboxy, sulpho, sulphato, phosphato or phosphono. Examples of compounds falling within this definition are sodium 3-dodecylamino propionate and sodium 2-dodecylamino propane sulphonate.

Zwitterionic synthetic detergents can be broadly described as derivatives of aliphatic quaternary ammonium, phosphonium and sulphonium compounds in which the aliphatic radical may be straight-chained or branched, and wherein one of the aliphatic substituents contains from about 8 to about 18 carbon atoms and one contains an anionic water-solubilising group, e.g. carboxy, sulpho, sulphato, phosphato or phosphono. These compounds are frequently referred to as betaines. Besides alkyl betaines, alkyl amino and alkyl amido betaines are encompassed within this invention. Cocoamido-propyl dimethyl betaine is a particularly useful surfactant.

After the wax capsule has melted, it remains molten or re-solidifies depending on the temperature of the washing medium. Whether in molten or solid state, however, the wax may deposit on the surface of pieces being washed as a soil and impart a spotted, streaked or filmy appearance to those pieces. Wax may also build up on the surfaces in which cleaning is being performed, i.e. in sinks, bathtubs and automatic dishwashing machines.

This soiling by the wax coating may be reduced by incorporating one or more surfactants in the cleaning composition. Applicants have discovered that certain surfactants are much better at preventing wax residue than others. These surfactants are polytergent SLF-18 ^{R} ex Olin Corporation, and Emphos CS-1361 ex Witco Chemical; and Dowfax 2A-1 ^{R} and Dowfax 3B-2 ^{R}.

Thus, a preferred embodiment of the cleaning composition comprises 0.1-15% by weight of wax-encapsulated bleach as described above; 1-75% builder; and 0.1-15% surfactant selected from the group consisting of polyoxyalkylated alkylaryl phosphate esters; mono- and di-C_8-C_{14} alkyl diphenyl oxide mono and/or di-sulphates; and nonionic surfactants of the formula



$$R-(\underset{R'}{CH_2CHO})_x(CH_2CH_2O)_y(\underset{R''}{CH_2CHO})_zH$$

where R is a C_6-C_{10} linear alkyl mixture, R' and R'' are methyl, x averages 3, y averages 12 and z averages 16.

Silicate

The compositions of this invention may contain sodium or potassium silicate at a level of from about 1 to about 40% by weight of the cleaning composition. This material is employed as a cleaning ingredient, source of alkalinity, metal corrosion inhibitor and protector of glaze on china tableware. Especially effective is sodium silicate having a ratio of SiO_2:Na_2O of from about 1.0 to about 3.3, preferably from about 2 to about 3.2. Some of the silicate may be in solid form.

Filler

An inert particulate filler material which is water-soluble may also be present in cleaning compositions in powder form. This material should not precipitate calcium or magnesium ions at the filler use level. Suitable for this purpose are organic or inorganic compounds. Organic fillers include sucrose esters and urea. Representative inorganic fillers include sodium sulphate, sodium chloride and potassium chloride. A preferred filler is sodium sulphate. Its concentration may range from 0% to 60%, preferably about 10% to 20% by weight of the cleaning composition.

Thickeners and Stabilizers

Thickeners are often desirable for liquid cleaning compositions. Thixotropic thickeners such as smectite clays including montmorillonite (bentonite), hectorite, saponite, and the like may be used to impart viscosity to liquid cleaning compositions. Silica, silica gel, and aluminosilicate may also be used as thickeners. Salts of polyacrylic acid (of molecular weight of from about 300,000 up to 6 million), including polymers which are cross-linked may also be used, alone or in combination with other thickeners. Use of clay thickeners for automatic dishwashing compositions is disclosed, for example, in U.S. Patent Nos. 4,431,559; 4,511,487; 4,740,327; 4,752,409. Use of salts of polymeric carboxylic acids is disclosed, for example, in UK Patent Application GB 2,164,350A. Commercially available bentonite clays include Korthix H and VWH ex Combustion Engineering, Inc.; Polargel T ex American Colloid Co.; and Gelwhite clays (particularly Celwhite GP and H) ex English China Clay Co. Polargel T is preferred as imparting a more intense white appearance to the composition than other clays.

For liquid formulations with a "gel" appearance and rheologyl particularly if a clear gel is desired, a chlorine-stable polymeric thickener is particularly useful. U.S. Patent No. 4,260,528 discloses natural gums and resins for use in clear autodish detergents, which are not chlorine-stable. Acrylic acid polymers that are cross-linked, manufactured by B.F. Goodrich and sold under the trade name "Carbopol", have been found to be effective for production of clear gels, and Carbopor 940, having a molecular weight of about 4,000,000 is particularly preferred for maintaining high viscosity with excellent chlorine stability over extended periods. Further suitable chlorine-stable polymeric thickeners are described in U.S. Patent Application Serial Number 157,425, filed February 17, 1988 by Elliott et al. This application is incorporated by reference herein.

The amount of thickener employed in the composition is from 0 to 5%, preferably 1-3%.

Stabilizers and/or co-structurants such as long-chain calcium and sodium soaps and C_{12} to C_{18} sulphates are detailed in U.S. Patents Nos. 3,956,158 and 4,271,030 and the use of other metal salts of long-chain soaps is detailed in U.S. Patent No. 4,752,409. The amount of stabilizer which may be used in the liquid cleaning compositions is from about 0.01 to about 5% by weight of the composition, preferably 0.1-2%. Such stabilizers are optional in gel formulations. Co-structurants which are found especially suitable for gels include trivalent metal ions at 0.01-4% of the composition and/or water-soluble structuring chelants at 1-60%. These co-structurants are more fully described in the co-pending U.S. Patent Application Serial Number 139,492, by Corring et al., filed December 30, 1987, which application is hereby incorporated by reference.

Defoamer

Liquid and "gel" formulations of 'the cleaning composition comprising surfactant may further include a defoamer. Suitable defoamers include mono- and di-stearyl acid phosphate, silicone oil and mineral oil. Even if the cleaning composition has only defoaming surfactant, the defoamer assists to minimize foam which food soils can generate. The composition may include 0.2 to 2% by weight of defoamer, orpreferably 0.05-1.0%.

Minor amounts of various other components may be present in the cleaning composition. These include flow control agents (in granular forms), soil-suspending agents, anti-redeposition agents, anti-tarnish agents, enzymes (e.g. protease, amylase and/or lipase at 0.05-2% by weight, preferably 0.5-1.5%) and other functional additives and perfume. The pH of the cleaning composition may be adjusted by addition of strong acid or base.

The following Examples will more fully illustrate the embodiments of the invention. All parts, percentages and proportions referred to herein and in the appended claims are by weight unless otherwise indicated.

EXAMPLE I

Two batches of wax-encapsulated bleach particles are produced with lower-melting point waxes in the Glatt WSG-5 fluid bed. Batch A is coated with a mixture of Boler 941/Altafin 125 paraffin waxes in a 79.54/20.46 ratio. Batch B is coated with 100% Bolar 941. The following conditions are used to coat the Clearon CDB-56 bleach particles.

Batch A	
Fuidized Bed Apparatus	Glatt WSG-5
Spray Mode	Top spray
Nozzle	Middle Port W 11" extension
Nozzle tip diameter	1.2 mm
Volume	22 liter
Bed Weight	11 lbs.
Air Flow Rate	400-450 cfm
Inlet Air Temperature	27-32 °C
Bed Temperature	28-32 °C
Coating Rate	52 g/min
Coating Temperature	75-80 °C
Atomization Air Pressure	5 Bar
Atomization Air Temperature	80-90 °C
Batch Time	148 mins

Batches made with the top spray set up normally have 15-20% loss as agglomerated material. The 11 pounds (5 kg) of Clearon CDB-56 bleach particles are coated in Batch A with 6 kg of a mixture of

79.54/20.46 of Boler 941 and Altafin 125 paraffin. The resulting encapsulated bleach particles have excellent stability in autodish liquid.

Batch B is coated with 100% Boler 1397 wax applied in a fluidized bed at the following settings:

Batch B	
Spray Mode	Wurster
Unit	Glatt GPCG-5
Partition Height	1.0"
Nozzle tip diameter	1.2 mm
Volume	10.5 liter
Bed Weight	17.5 lbs.
Air Flow Rate	200-270 cfm
Inlet Air Temperature	18-24° C
Bed Temperature	30-31° C
Coating Rate	72 g/min
Coating Temperature	75-80° C
Atomization Air Pressure	1.5 Bar
Atomization Air Temperature	80-90° C
Batch Time	70 mins

The encapsulated CDB-56 of Batch B has excellent stability in autodidish liquid at 40° C and pH of 12.3.

EXAMPLE II

The solubility of coating compositions made from micro-crystalline wax and fatty acid in alkaline media may be contrasted to that of coating compositions made from one paraffin wax having melting point between 40 and 50° C. Four different coating compositions are made from a microcrystalline wax with a pair of fatty acids in the proportions appearing below. Two different paraffin waxes are selected for comparison. The four fatty acid/wax and the two waxes are identified as coating compositions 1 through 6 below.

Equal amounts (0.27 g) of each of coating compositions 1 through 6 are placed in separate beakers, which already contain 2.87 liters of a 0.02% aqueous solution of Emphos CS-1361. The contents of each beaker are heated to 49° C, maintained at this temperature with stirring for 45 minutes, then cooled to room temperature and poured through a USA standard metal sieve with size 48 mesh (300 microns).

Solid wax captured by the sieve is dried and weighed to determine the amount of wax which remained as solid residue after the heating with surfactant.

Table II.

Coating Compositions	
Coating Composition	(° C)
1. 3.6 % Multiwax W-145A 34.4% Capric acid* 62.0% Lauric acid*	40
2. 8.8 % Multiwax W-145A 39.8% Capric acid* 51.4% Lauric acid*	40
3. 3.6 % Multiwax W-145A 18.3 % Capric acid* 78.1 % Myristic acid*	50
4. 8.8 % Multiwax W-145A 19.0 % Capric acid* 72.2 % Myristic acid*	50
5. Boler Paraffin 1397	46
6. Ross fully refined paraffin 115/120	46

* Melting point of Capric acid = 31.2° C
Lauric acid = 43.9° C
Myristic acid = 54.1° C
Multiwax W-145A = 66-71° C

Table III

Wax Residue Amounts		
Coating Composition	Solid wax residue on filter paper (gm)	% of initial wax present as residue
1. 3.6 % Multiwax W-145A 34.4% Capric acid 62.0% Lauric acid	0.0906	33.56
2. 8.8 % Multiwax W-145A 39.8% Capric acid 51.4% Lauric acid	0.1395	51.70
3. 3.6 % Multiwax W-145A 18.3 % Capric acid 78.1 % Myristic acid	0.1632	60.44
4. 8.8 % Multiwax W-145A 19.0 % Capric acid 72.2 % Myristic acid	0.2678	99.19
5. Soler Paraffin 1397	0.0001	0.04
6. Ross fully refined paraffin 115/120	0.0092	3.41

The microcrystalline wax/fatty acid compositions leave slarge amounts of wax residues. Comparison of Samples 1 to 2 and 3 to 4 shows that less wax is deposited from coating compositions with lower levels of Multiwax W145-A. In contrast to coating compositions 1 through 4, the paraffin waxes having melting point from 40-50° C leave very little residue, and hence are much preferred as coatings for bleach particles.

EXAMPLE III

Bleach is encapsulated as in Example 1 but with coatings consisting of a wax melting at 72° C (30% Epolene C-16/70% Boler Paraffin 1426), 52° C (Altafin 125/130) or 46° C (Ross 115/120). The capsules coated with the high-melting waxes are coated in a fluidized bed as are the capsules of Batch A in Example I, except that for capsules coated with Epolene, the bed temperature is 60-65° C and for capsules coated with Altafin 125/130 the bed temperature is 40-45° C. The capsules coated with Ross 115/120 are prepared as are the capsules of Batch B in Example I. All three capsule batches are coated with a core:coat ratio of 47:53. Thus, in one gram of capsules, there should be 0.53 grams of wax.

1.88 grams of each type of capsule is placed in forty grams of an autodish liquid composition composed as follows:

Material	% Weight
45% KOH	1.10
Laponite clay	0.02
TKPP	4.00
Carbopol 941	1.00
STP	1.00
60% TKPP soln	25.00
D-silicate	17.00
K_2CO_3 (47% soln)	12.77
SLF-18	1.00
Colorant	0.5
Perfume	0.05
Water	36.56

The procedure for making this autodish gel formulation is as follows. Water is loaded into a vessel. The KOH is added with stirring for one minute, followed by the clay with further stirring for another 10 minutes. The blend of TKPP, STP and Carbopol 941 is then added over the next 12 minutes, followed by 30 minutes of stirring. The TKPP solution is then added and the mixture is stirred for 30 minutes. Then the D-silicate, K_2CO_3 and SLF-18 are each added separately, each one being followed by 5 minutes of stirring.

The autodish liquid composition containing the bleach capsules is in turn placed in the dispenser cup of a Kenmore automatic dishwashing machine. One 40 gram sample of autodish is placed in the dispensing cup of the dishwasher at a time and the machine is run through one complete cycle when empty. At the end of the wash cycle, the water draining from the machine is filtered through a U.S. standard metal sieve of 43 mesh into a bucket. The captured wax capsules or particles are dried and weighed. The results appear in the table below and Figure 1.

Coat Melting Point (° C)	Weight wax retained	% Total Wax retained
72	0.28 g	28%
52	0.17 g	17
46	0	0

EXAMPLE IV

The same three capsule types made in Example III are here tested in preventing spotting on glassware washed in an automatic dishwashing machine. Glass appearance tests are run in Bosch S-512 dishwashers at 140° F and using water of 120 ppm hardness.

In the test, two washing machines are loaded with ten clear glass plates and drinking glasses (all of which are clean and spotless). Forty grams of a fatty soil are then smeared on the interior of each washing machine door. The soil is formed by mixing four pounds of Imperial margarine with four packets (12.8 ounces each) of Carnation non-fat dry milk mixed together until smooth. Forty grams of the autodish liquid composition with one of the coated bleach capsules are then loaded into the washing machine cup dispenser. The glassware is then subjected to a short wash cycle. After the wash cycle, each glass is removed from the washer and evaluated for spotting according to the following scale:

Spotting Scale

0 = spotless
1 = few spots
2 = 1/3 glass spotted
3 = 2/3 glass spotted
4 = glass completely covered with spots

The summary of the spotting and filming evaluation results appear in Figure 2. The results show that lower melting point capsules melt easier and therefore release more bleach which in turn improves spotting scores.

EXAMPLE V

To compare the stability in alkaline media of bleach coated with paraffin wax of melting point 40-50 ° C to that of bleach coated with a mixture of microcrystalline wax and fatty acid, Clearon CDB-56 bleach particles ex Olin Corporation are coated with coating composition 3, 4, 5 or 6 described in Example II.

The capsules are made in a Granuglatt apparatus, model number WSG-3 at the following settings:

Spray Mode	Wurster
Initial Bed Charge	1,600 g
Inlet Air Temperature	16-20 ° C
Bed Temperature	ca. 18-22 ° C
Coating Rate	60-80 g/min
Coating Temperature	75-80 ° C
Atomization Air Pressure	1.5 Bar
Atomization Air Temperature	79-88 ° C
Batch Time	20-28 minutes

Then 1.8 grams of each capsule are dispersed evenly throughout the automatic dishwashing liquid of Example III. Thus, six autodish liquid compositions containing the capsules are formed and each is stored at 40 ° C.

Samples are set up in triplicate in 4 oz glass jars. Chlorine analysis is carried out after 1, 2, 7, 14, 28, 42, and 56 days.

5 ml aliquots are removed from each of the six autodish liquid samples and filtered through USA standard metal sieves, 48 mesh, to remove the capsules. The wax coating is dissolved from each capsule by gentle stirring in hexane for 20 minutes. The amount of active chlorine remaining is then measured by standard iodometric titration. The results are summarized in the following Table.

Table IV

Storage Stability Results of capsules stored in an Autodish Liquid, pH = 12.3, 40.0 C.			
Time	Percent available Chlorine Remaining		
(days)	Capsule 3	Capsule 4	Capsule 4'
0	100.0	100.0	100.0
1	45.8	36.3	46.2
2	14.2	9.6	8.9
7	--	--	--
Time	Percent available Chlorine Remaining		
(days)	Capsule 5	Capsule 6	
0	100.0	100.0	
1	--	--	
2	--	--	
7	100.0	100.0	
14	100.0	100.0	
28	100.0	83.2	
42	100.0	82.0	
56	100,0	47.6	

Capsules 3 and 4 have a melting point of 50° C and coating levels of 57 and 54 wt.% of the total capsule, respectively. Capsule 4' has the same composition as that of capsule 4 except that its coating level is higher, namely 66%. Capsules 5 and 6 have a coating level of 54%. The results show that fatty acid/microcrystalline wax coatings protect bleach poorly in an alkaline medium. Thus, these coating materials are not suitable for use in aqueous alkaline media. By contrast, when the coating is a paraffin wax of melting point from 40 to 50° C, the level of bleach preserved in an alkaline medium is excellent.

EXAMPLE VI

The stability of bleach encapsulated in single wax coatings of different thickness is measured as follows: a batch of clearon CDB 56 ^{R} is loaded into a fluidized bed and coated with enough Boler Paraffin 1397 so that the coat constitutes 35% of the encapsulated bleach (Batch A). A second batch (Batch B) of Clearon CDB 56 ^{R} is coated with sufficient Boler 1397 so that the coat is 55% of the particle. About one gram of each bleach encapsulate is then dispersed into 40 grams of the autodish composition of Example III and stored therein at 40° C.

Samples are set up in triplicate in 4 oz glass jars and stored at 40.2° C. Chlorine analysis is carried out at 1, 2, 4, and 6 weeks.

At the time intervals shown in Table V below, 5 ml aliquots are removed from each autodish liquid sample. These are filtered through USA standard metal sieves, 48 mesh, to remove the capsules. The amount of active chlorine remaining is measured by standard iodometric titration. The results are summarized in the following Table.

Table V

Storage Stability in ADL Gel		
	% Original Chlorine Activity Remaining	
Time	Batch A (35% coat)	Batch B (55% coat)
1 week	100.0	100.0
2 seeks	95.4	100.0
4 weeks	72.5	83.2
6 weeks	63.0	82.0

Thus, it is shown that coats of greater thickness impart greater protection to bleach particles in aqueous media.

EXAMPLE VII

The capsules of Example III are incorporated into the autodish liquid composition of Example III. Forty grams of each composition are loaded into the dispenser cup of a Kenmore automatic dishwashing machine and the machine is operated through one wash cycle at 46° C. Every two minutes through the wash cycle, a 5 ml aliquot is removed from the wash liquor. The level of available chlorine released from the capsules is measured by standard iodometric titration. As the results show (Figure 3), the capsules having lower melting coats release bleach more quickly and more completely. Thus, these capsules demonstrate higher efficiency.

The foregoing description and Examples illustrate selected embodiments of the present invention. In light thereof, various modifications will be suggested to one skilled in the art, all of which are within the spirit and purview of this invention.

Claims

1. A method for forming a coherent, continuous coating around bleach particles suitable for use in cleaning compositions, the method comprising
   a) suspending bleach particles in a fluidized bed,
   b) selecting one or more paraffin waxes to provide the coating, the waxes having melting point between about 40° C and 50° C, needle penetration value of to 60 mm at 25° C,
   c) heating the one or more paraffin waxes to a temperature above its melting temperature sufficiently to melt all the wax,
   d) fluidizing the bed by passing air through the bleach particles, so as to maintain a bed temperature of at least 20° C to a temperature no higher than the wax melting point,
   e) spraying the melted paraffin wax on to the fluidized bed at a rate of 10 to 40 g/min per kilogram of bleach particles in the fluidized bed for a time sufficient to apply a coat 100 to 1,000 microns thick.
2. The method according to Claim 1 wherein the paraffin wax is sprayed into the fluid bed by the Wurster method comprising suspending the particles in an upwardly flowing air stream entering a bottom of the fluidized bed to impart controlled cyclic movement to the particles with a portion of the bed flowing upwardly.
3. The method according to Claim 1, further comprising annealing the coated particles at a temperature of from 5 to 15° C greater than the bed temperature during coating, and from 3 to 15° C less than the melting point of the wax coating for from 10 to 45 minutes after spraying is complete.
4. The method according to Claim 1 wherein the encapsulated particles have an average diameter ranging from 800 to 4,000 microns.
5. The method according to Claim 1 wherein the paraffin wax has a solids content of from 40 to 100% at 40° C and a solids content of from 0 to 15% at 50° C.
6. A wax-encapsulated bleach particle suitable for incorporation in cleaning compositions, the encapsulated particle comprising

a) 45-65% by weight of the final encapsulated particle of a solid particulate core of bleach,

b) 35-55% by weight of the final encapsulated particle of a waxy coating, the coating comprising one or more paraffin waxes having melting point of from about 40° C to about 50° C and needle penetration value of 10 to 60 mm at 25° C and being from 100 to 1,000 microns thick.

7. The encapsulated particle according to Claim 6 in which the coating thickness is 200-750 microns.

8. The encapsulated particle according to Claim 6 wherein the bleach is selected from the group consisting of chlorine-releasing agents, bromine-releasing agents, and organic and inorganic peroxygen-generating compounds.

9. The encapsulated particle according to Claim 8 wherein the bleach is chloroisocyanurate.

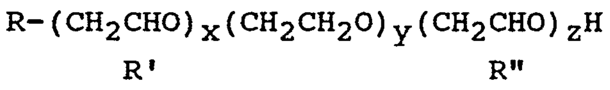
10. A liquid cleaning composition comprising

a) 0.1-15% by weight of the encapsulated bleach of Claim 6;
b) 1-75% by of weight builder;
c) 0.1-15% by weight of a surfactant; and
d) 15-95% water.

11. The cleaning composition according to Claim 10 wherein the composition is an automatic dishwashing liquid comprising

a) 10-60% builder,
b) 0.01-2% nonionic surfactant,
c) 5-20% silicate,
d) 0.1-15% of the encapsulated particle of Claim 6;
e) 0-5% clay; and
f) balance water.

12. The cleaning composition according to Claim 10 wherein the surfactant is selected from the group consisting of mono- and di-C_8-C_{14} alkyl diphenyl oxide mono- and/or di-sulphates, polyoxyalkylated alkylaryl phosphate esters, surfactants having the formula



$$R-(CH_2\underset{R'}{C}HO)_x(CH_2CH_2O)_y(CH_2\underset{R''}{C}HO)_zH$$

where R is C_6-C_{10} linear alkyl mixture, R′ and R″ are methyl, x averages 3, y averages 12 and z averages 16, and mixtures thereof.

Fig.1.

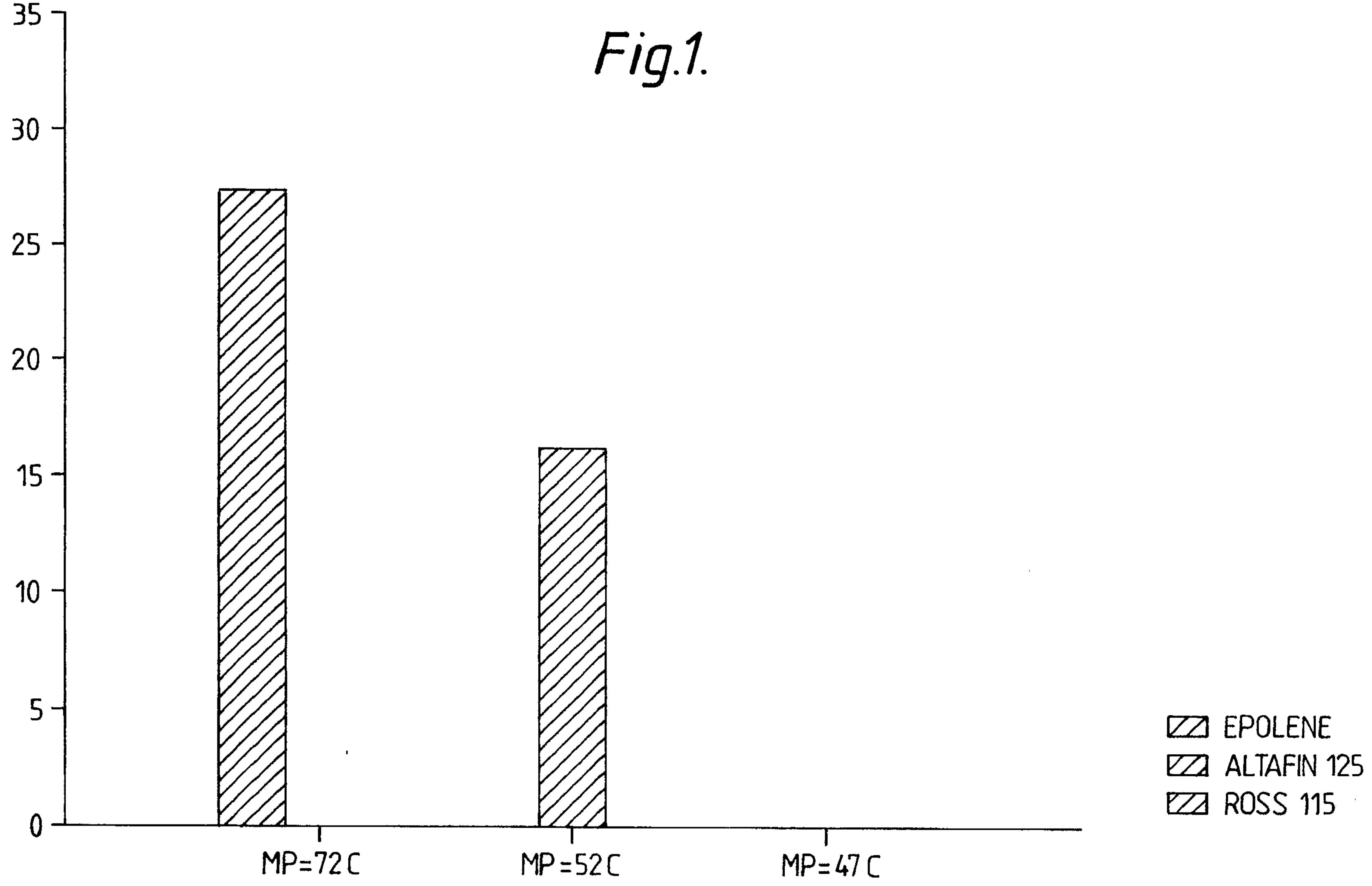

35
30
25
20
15
10
5
0
MP=72 C
MP=52 C
MP=47 C
EPOLENE
ALTAFIN 125
ROSS 115

Fig.2.

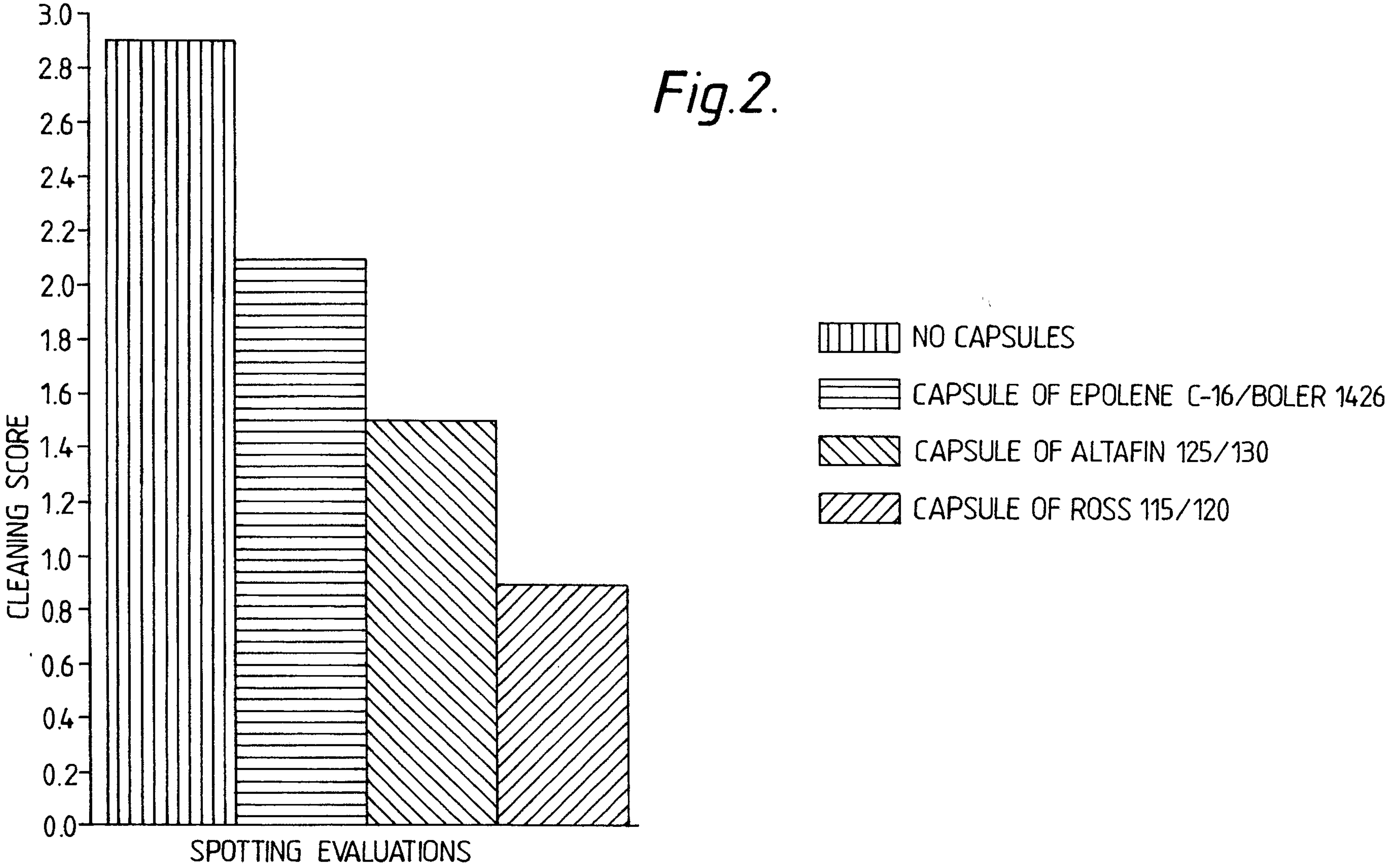
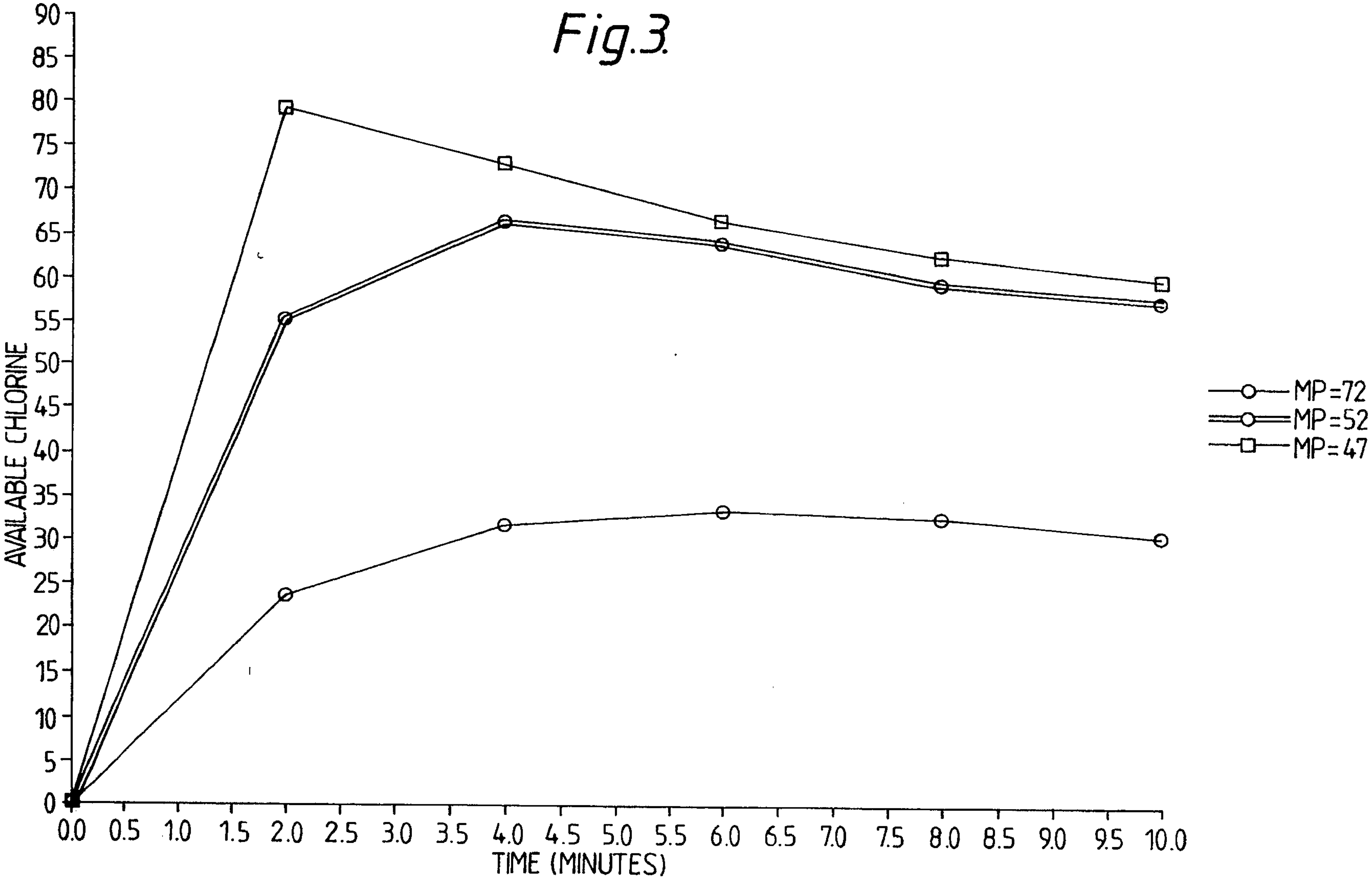

CLEANING SCORE
3.0
2.8
2.6
2.4
2.2
2.0
1.8
1.6
1.4
1.2
1.0
0.8
0.6
0.4
0.2
0.0
SPOTTING EVALUATIONS
▥ NO CAPSULES
▤ CAPSULE OF EPOLENE C-16/BOLER 1426
▧ CAPSULE OF ALTAFIN 125/130
▨ CAPSULE OF ROSS 115/120

Fig.3.


AVAILABLE CHLORINE
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5
9.0
9.5
10.0
TIME (MINUTES)
MP=72
MP=52
MP=47