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(71) Applicant (for all designated States except US): **THE PROCTER & GAMBLE COMPANY** [US/US]; One Procter & Gamble Plaza, Cincinnati, Ohio 45202 (US).

(72) Inventors; and

(75) Inventors/Applicants (for US only): **MCCHAIN, Robert, Joseph** [US/US]; 9901 McKelvey, Cincinnati, Ohio 45231 (US). **MCKIERNAN, Robin** [US/US]; 6793 Lexington Park Blvd., Mason, Ohio 45040 (US). **NEAL, Charles, William** [US/US]; 2350 Royal Manor Place, Fairfield, Ohio 45014 (US). **NWACHUKWU, Chisomaga, Ugochi** [US/US]; 322 West 4th Street, Cincinnati, Ohio 45202 (US). **SMITH, Steven, Daryl** [US/US]; 5238 Concord Mill Place, Fairfield, Ohio 45014 (US).

(74) Common Representative: **THE PROCTER & GAMBLE COMPANY**; c/o Timothy B. Guffey, Global

Patent Services, 299 East Sixth Street, Sycamore Building, 4th Floor, Cincinnati, Ohio 45202 (US).

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(54) Title: COMPOSITIONS COMPRISING SOIL ADSORPTION POLYMERS FOR REDUCING PARTICULATES IN THE AIR

(57) Abstract: Compositions comprising soil adsorption polymers for reducing particulates in the air are provided.



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## COMPOSITIONS COMPRISING SOIL ADSORPTION POLYMERS FOR REDUCING PARTICULATES IN THE AIR

### FIELD OF THE INVENTION

5           The present invention relates to compositions comprising soil adsorption polymers, and more particularly compositions having soil adsorption polymers for reducing particulates in the air.

### BACKGROUND OF THE INVENTION

10           Particulates are believed to have a significant effect on air quality and on the health of individuals, especially those susceptible to allergies. Particulates include household pollutants, dust particles, silica, lint, particulates containing allergens such as pet dander and dust mites. Particulates in the air are generally about 0.1 ums to 50 ums in size.

          Products for reducing particulates are well known and described in the patent literature. Many products use filtration and/or ionization technology to reduce particulates in the air, but this can be costly or cumbersome to use over sprayable products for controlling particulates. Such sprayable products are described in the patent literature and typically include ingredients that help precipitate particulates from the air or provide a barrier that covers particulates that land on surfaces. However, these sprayable products may be perceived as ineffective in removing particulates. For example, a precipitating ingredient may mechanically force particulates to a surface but smaller, lighter particulates that are precipitated can quickly re-circulate up into the air upon movement of air. Where a product includes dust controlling levels of a barrier forming ingredient, a sticky residue often times results on the surface. In some instances, this sticky residue can attract more dust.

25           For these reasons, there continues to be a need for improved compositions that reduce particulates in the air.

### SUMMARY OF THE INVENTION

          The present invention fulfills the need described above by providing compositions having novel polymers that exhibit improved soil adsorption properties compared to known polymers

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(e.g. Mirapol® and Lupsaol®) that exhibit soil adsorption properties as measured according to the Soil Adsorption Test Method described herein.

In one example, a composition for reducing particulates in the air comprises a soil adsorbing polymer comprising two or more monomeric units selected from the group consisting of:

- a) nonionic monomeric units;
- b) anionic monomeric units;
- c) cationic monomeric units;
- d) zwitterionic monomeric units; and
- e) mixtures thereof;

wherein said polymer exhibits a Soil Adsorption Value of at least 38 mg as measured according to the Soil Adsorption Test Method described herein.

In another example, a composition for reducing particulates in the air comprises:

a soil adsorbing polymer comprising two or more monomeric units selected from the group consisting of:

- a) nonionic monomeric units;
- b) anionic monomeric units;
- c) cationic monomeric units;
- d) zwitterionic monomeric units; and
- e) mixtures thereof;

wherein said polymer exhibits a Soil Adsorption Value of at least 38 mg as measured according to the Soil Adsorption Test Method described herein;

a surfactant selected from the group consisting of: nonionic surfactants, zwitterionic surfactants, amphoteric surfactants, and mixtures thereof;

a compressed gas propellant; and  
an aqueous carrier.

#### DETAILED DESCRIPTION OF THE INVENTION

“Number average molecular weight” as used herein means the number average molecular weight  $M_n$  as determined using gel permeation chromatography according to the protocol found in Colloids and Surfaces A. Physico Chemical & Engineering Aspects, Vol. 162, 2000, pg. 107-121.

“Weight average molecular weight” as used herein means the weight average molecular weight  $M_w$  as determined using gel permeation chromatography according to the protocol found in Colloids and Surfaces A. Physico Chemical & Engineering Aspects, Vol. 162, 2000, pg. 107-121.

5 “Polydispersity Index” PDI as used herein means the ratio of the weight average molecular weight to the number average molecular weight,  $M_w/M_n$ , as determined using gel permeation chromatography.

“Monomeric unit” as used herein is a constituent unit (sometimes referred to as a structural unit) of a polymer.

10 “Nonionic monomeric unit” as used herein means a monomeric unit that exhibits no net charge at a pH of 4.5 and/or is identified as a nonionic monomeric unit herein. A nonionic monomeric unit may be derived from a nonionic monomer.

“Nonionic monomer” as used herein means a monomer that exhibits no net charge at a pH of 4.5 and/or is identified as a nonionic monomer herein.

15 “Anionic monomeric unit” as used herein means a monomeric unit that exhibits a net negative charge at a pH of 4.5 and/or a pH of 6 and/or is identified as an anionic monomeric unit herein. An anionic monomeric unit may be derived from an anionic monomer. An anionic monomeric unit is generally associated with one or more cations such as cations of alkali metal or alkaline earth metal, for example sodium or cationic groups such as ammonium.

20 “Anionic monomer” as used herein means a monomer that exhibits a net negative charge at a pH of 4.5 and/or a pH of 6 and/or is identified as an anionic monomer herein. An anionic monomer is generally associated with one or more cations such as cations of alkali metal or alkaline earth metal, for example sodium or cationic groups such as ammonium.

25 “Cationic monomeric unit” as used herein means a monomeric unit that exhibits a net positive charge at a pH of 4.5 and/or is identified as a cationic monomeric unit herein. A cationic monomeric unit may be derived from a cationic monomer. A cationic monomeric unit is generally associated with one or more anions such as a chloride ion, a bromide ion, a sulfonate group and/or a methyl sulfate group.

30 “Cationic monomer” as used herein means a monomer that exhibits a net positive charge at a pH of 4.5 and/or is identified as a cationic monomer herein. A cationic monomer is generally associated with one or more anions such as a chloride ion, a bromide ion, a sulfonate group and/or a methyl sulfate group.

“Genuine malodor removal benefit” is defined as an analytically measurable malodor reduction. Thus, if the composition delivers a genuine malodor removal benefit, the composition will not function merely by using perfume to cover up or mask odors.

“Zwitterionic monomeric unit” as used herein means a monomeric unit that exhibits both a negative charge and a positive charge on the same monomeric unit at a pH of 4.5 and/or is identified as a zwitterionic monomeric unit herein. A zwitterionic monomeric unit may be derived from a zwitterionic monomer. A zwitterionic monomeric unit is generally associated with one or more cations such as cations of alkali metal or alkaline earth metal, for example sodium or cationic groups such as ammonium and one or more anions such as a chloride ion, a bromide ion, a sulfonate group and/or a methyl sulfate group.

“Zwitterionic monomer” as used herein means a monomer that exhibits both a negative charge and a positive charge on the same monomer at a pH of 4.5 and/or is identified as a zwitterionic monomeric unit herein. A zwitterionic monomer is generally associated with one or more cations such as cations of alkali metal or alkaline earth metal, for example sodium or cationic groups such as ammonium and one or more anions such as a chloride ion, a bromide ion, a sulfonate group and/or a methyl sulfate group.

“Basis Weight” as used herein is the weight per unit area of a sample reported in lbs/3000 ft<sup>2</sup> or g/m<sup>2</sup> and is measured according to the Basis Weight Test Method described herein.

For clarity purposes, the total “% wt” values do not exceed 100% wt.

The compositions of the present invention comprise soil adsorbing polymers for reducing particulates in the air. The reduction in particulates may be achieved by the adsorption of particulates in the air onto a physical surface (e.g. substrate) having the soil adsorbing polymer or by dispersing compositions having the soil adsorption polymers into the air thereby agglomerating particulates in the air.

The compositions of the present invention may have a viscosity of about 0.1 cps to about 8 cps, alternatively from about 1 to about 6 cps, alternatively about 1 to about 4 cps, alternatively about 2.5 to about 4 cps, alternatively about 3.5 cps when measured with a Brookfield Synchro-Lectric Viscometer (Model LVF) at 21°C with spindle 1 (60 RPM).

The pH of the compositions herein may be from about 1 to about 10, alternatively from about 1 to about 8, alternatively from about 3 to about 8, alternatively from about 4 to about 8, alternatively from about 4 to about 7. Accordingly, the compositions herein may further comprise an acid or base to adjust pH as appropriate.

A suitable acid for use herein is an organic and/or an inorganic acid. A preferred organic acid for use herein has a pKa of less than about 6. A suitable organic acid is selected from the group consisting of citric acid, lactic acid, glycolic acid, succinic acid, maleic acid, benzoic acid, glutaric acid and adipic acid and a mixture thereof. A suitable inorganic acid is selected from the group consisting hydrochloric acid, sulphuric acid, phosphoric acid and a mixture thereof. A typical level of such an acid, when present, is from about 0.01% to about 5.0%, alternatively from about 0.01% to about 3.0%, alternatively from about 0.01% to about 1.5 % alternatively about 0.1%, by weight of the composition.

In some embodiments, the compositions may be aqueous compositions comprising a compressed gas propellant. In some embodiments, the compositions may include a perfume that delivers a consistent perfume release profile (e.g. a perceivable perfume intensity which is delivered initially and a comparable intensity maintained for at least 10 minutes or longer). The compositions may also include a malodor counteractant that delivers a genuine malodor removal benefit.

There are numerous embodiments of the compositions described herein, all of which are intended to be non-limiting examples.

#### 1. Polymers

The compositions of the present invention comprise soil adsorbing polymers that may be present at a level of from about 0.001% to about 1%, alternatively from about 0.001% to about 0.5%, alternatively from about 0.001% to about 0.2%, alternatively from about 0.001% to about 0.1%, alternatively from about 0.001% to about 0.05%, alternatively about 0.001% to about 0.2%, alternatively about 0.01% to about 0.1%, alternatively about 0.01% to about 0.05%, by weight of the composition.

The soil adsorbing polymers of the present invention comprise two or more different types of monomeric units. As a result, the polymers of the present invention can be referred to as copolymers including terpolymers and higher rather than homopolymers, which consist of a single type of monomeric unit. The polymers of the present invention may be a terpolymer (3 different types of monomeric units). The polymers of the present invention may be a random copolymer. In one example, a polymer of the present invention is water-soluble and/or water-dispersible, which means that the polymer does not, over at least a certain pH and concentration range, form a two-phase composition in water at  $23^{\circ}\text{C} \pm 2.2^{\circ}\text{C}$  and a relative humidity of  $50\% \pm 10\%$ .

In one example, the polymers of the present invention exhibit a Number Average Molecular Weight of less than 2,000,000 g/mol and/or less than 1,750,000 g/mol and/or less than 1,700,000 g/mol and/or less than 1,500,000 g/mol and/or greater than 500,000 g/mol and/or greater than 900,000 g/mol. In another example, the polymers exhibit a Number Average  
5 Molecular Weight of from about 500,000 to 2,000,000 g/mol and/or from about 900,000 to 1,700,000 g/mol.

In another example, the polymers of the present invention exhibit a Soil Adsorption Value of at least 38 mg and/or at least 40 mg and/or at least 42 mg and/or at least 45 mg and/or at least 47 mg and/or at least 50 mg and/or at least 53 mg and/or at least 55 mg and/or at least 57 mg  
10 and/or at least 60 mg and/or at least 62 mg as measured according to the Soil Adsorption Test Method described herein.

In yet another example, the polymers of the present invention exhibit a charge density (at pH 4.5) of from about -0.1 meq/g and/or from about -0.05 meq/g and/or from about -0.02 meq/g and/or from about 0 meq/g and/or to about +0.1 meq/g and/or to about +0.09 meq/g and/or to  
15 about +0.08 meq/g and/or to about +0.06 meq/g and/or to about +0.05 meq/g and/or to about +0.02 meq/g as measured according to the Charge Density Test Method described herein. In still another example, the polymers of the present invention exhibit a charge density of from about -0.1 meq/g to about +0.1 meq/g and/or from -0.05 meq/g to about +0.1 meq/g and/or from about 0 to less than +0.1 meq/g and/or to less than +0.09 meq/g and/or to less than +0.08 meq/g and/or to  
20 less than +0.06 meq/g and/or to less than +0.05 meq/g as measured according to the Charge Density Test Method described herein.

In another example, the polymers exhibit a Polydispersity Index of less than 2.5 and/or of less than 2.0 and/or less than 1.7 and/or less than 1.5 and/or less than 1.3.

In one example, a polymer of the present invention comprises two or more monomeric  
25 units selected from the group consisting of: a. nonionic monomeric units; b. anionic monomeric units; c. cationic monomeric units; d. zwitterionic monomeric units; and e. mixtures thereof.

The polymers of the present invention may exhibit a Soil Adsorption Value of at least 38 mg as measured according to the Soil Adsorption Test Method described herein.

a. Nonionic Monomeric Units

30 The nonionic monomeric units may be selected from the group consisting of: nonionic hydrophilic monomeric units, nonionic hydrophobic monomeric units, and mixtures thereof.

Non-limiting examples of nonionic hydrophilic monomeric units suitable for the present invention include nonionic hydrophilic monomeric units derived from nonionic hydrophilic monomers selected from the group consisting of: hydroxyalkyl esters of  $\alpha,\beta$ -ethylenically unsaturated acids, such as hydroxyethyl or hydroxypropyl acrylates and methacrylates, glyceryl monomethacrylate,  $\alpha,\beta$ -ethylenically unsaturated amides such as acrylamide, N,N-dimethylmethacrylamide, N-methylolacrylamide,  $\alpha,\beta$ -ethylenically unsaturated monomers bearing a water-soluble polyoxyalkylene segment of the poly(ethylene oxide) type, such as poly(ethylene oxide)  $\alpha$ -methacrylates (Bisomer S20W, S10W, etc., from Laporte) or  $\alpha,\omega$ -dimethacrylates, Sipomer BEM from Rhodia ( $\omega$ -behenyl polyoxyethylene methacrylate), Sipomer SEM-25 from Rhodia ( $\omega$ -tristyrylphenyl polyoxyethylene methacrylate),  $\alpha,\beta$ -ethylenically unsaturated monomers which are precursors of hydrophilic units or segments, such as vinyl acetate, which, once polymerized, can be hydrolyzed in order to give rise to vinyl alcohol units or polyvinyl alcohol segments, vinylpyrrolidones,  $\alpha,\beta$ -ethylenically unsaturated monomers of the ureido type, and in particular 2-imidazolidinone-ethyl methacrylamide (Sipomer WAM II from Rhodia), and mixtures thereof. In one example, the nonionic hydrophilic monomeric unit is derived from acrylamide.

Non-limiting examples of nonionic hydrophobic monomeric units suitable for the present invention include nonionic hydrophobic monomeric units derived from nonionic hydrophobic monomers selected from the group consisting of: vinylaromatic monomers such as styrene,  $\alpha$ -methylstyrene, vinyltoluene, vinyl halides or vinylidene halides, such as vinyl chloride, vinylidene chloride,  $C_1$ - $C_{12}$  alkylesters of  $\alpha,\beta$ -monoethylenically unsaturated acids such as methyl, ethyl or butyl acrylates and methacrylates, 2-ethylhexyl acrylate, vinyl esters or allyl esters of saturated carboxylic acids, such as vinyl or allyl acetates, propionates, versates, stearates,  $\alpha,\beta$ -monoethylenically unsaturated nitriles containing from 3 to 12 carbon atoms, such as acrylonitrile, methacrylonitrile,  $\alpha$ -olefins such as ethylene, conjugated dienes, such as butadiene, isoprene, chloroprene, and mixtures thereof.

#### b. Anionic Monomeric Units

Non-limiting examples of anionic monomeric units suitable for the present invention include anionic monomeric units derived from anionic monomers selected from the group consisting of: monomers having at least one carboxylic function, for instance  $\alpha,\beta$ -ethylenically unsaturated carboxylic acids or the corresponding anhydrides, such as acrylic, methacrylic or maleic acids or anhydrides, fumaric acid, itaconic acid, N-methacroylalanine, N-acryloylglycine,



and their water-soluble salts, monomers that are precursors of carboxylate functions, such as tert-butyl acrylate, which, after polymerization, give rise to carboxylic functions by hydrolysis, monomers having at least one sulfate or sulfonate function, such as 2-sulfooxyethyl methacrylate, vinylbenzene sulfonic acid, allyl sulfonic acid, 2-acrylamido-2-methylpropane sulfonic acid  
 5 (AMPS), sulfoethyl acrylate or methacrylate, sulfopropyl acrylate or methacrylate, and their water-soluble salts, monomers having at least one phosphonate or phosphate function, such as vinylphosphonic acid, etc., the esters of ethylenically unsaturated phosphates, such as the phosphates derived from hydroxyethyl methacrylate (Empicryl 6835 from Rhodia) and those derived from polyoxyalkylene methacrylates, and their water-soluble salts, and 2-carboxyethyl  
 10 acrylate (CEA), and mixtures thereof. In one example, the anionic monomeric unit is derived from an anionic monomer selected from the group consisting of: acrylic acid, AMPS, CEA, and mixtures thereof. In another example, the anionic monomeric unit is derived from acrylic acid.

#### c. Cationic Monomeric Units

Non-limiting examples of cationic monomeric units suitable for the present invention  
 15 include cationic monomeric units derived from cationic monomers selected from the group consisting of: N,N-(dialkylamino- $\omega$ -alkyl)amides of  $\alpha,\beta$ -monoethylenically unsaturated carboxylic acids, such as N,N-dimethylaminomethylacrylamide or -methacrylamide, 2-(N,N-dimethylamino)ethylacrylamide or -methacrylamide, 3-(N,N-dimethylamino)propylacrylamide or -methacrylamide, and 4-(N,N-dimethylamino)butylacrylamide or -methacrylamide,  $\alpha,\beta$ -  
 20 monoethylenically unsaturated amino esters such as 2-(dimethylamino)ethyl acrylate (DMAA), 2-(dimethylamino)ethyl methacrylate (DMAM), 3-(dimethylamino)propyl methacrylate, 2-(tert-butylamino)ethyl methacrylate, 2-(dipentylamino)ethyl methacrylate, and 2(diethylamino)ethyl methacrylate, vinylpyridines, vinylamine, vinylimidazolines, monomers that are precursors of amine functions such as N-vinylformamide, N-vinylacetamide, which give rise to primary amine  
 25 functions by simple acid or base hydrolysis, acryloyl- or acryloyloxyammonium monomers such as trimethylammonium propyl methacrylate chloride, trimethylammonium ethylacrylamide or -methacrylamide chloride or bromide, trimethylammonium butylacrylamide or -methacrylamide methyl sulfate, trimethylammonium propylmethacrylamide methyl sulfate, (3-methacrylamidopropyl)trimethylammonium chloride (MAPTAC), (3-methacrylamidopropyl)trimethylammonium methyl sulphate (MAPTA-MES), (3-acrylamidopropyl)trimethylammonium chloride (APTAC), methacryloyloxyethyl-trimethylammonium chloride or methyl sulfate, and acryloyloxyethyltrimethylammonium

chloride; 1-ethyl-2-vinylpyridinium or 1-ethyl-4-vinylpyridinium bromide, chloride or methyl sulfate; N,N-dialkyldiallylamine monomers such as N,N-dimethyldiallylammonium chloride (DADMAC); polyquaternary monomers such as dimethylaminopropylmethacrylamide chloride and N-(3-chloro-2-hydroxypropyl)trimethylammonium (DIQUAT or DQ) and 2-hydroxy-N<sup>1</sup>-(3-

5 (2((3- methacrylamidopropyl)dimethylamino)-acetamido)propyl)-N<sup>1</sup>, N<sup>1</sup>, N<sup>3</sup>, N<sup>3</sup>, N<sup>3</sup> - pentamethylpropane-1,3-diaminium chloride (TRIQUAT or TQ), and mixtures thereof. In one example, the cationic monomeric unit comprises a quaternary ammonium monomeric unit, for example a monoquaternary ammonium monomeric unit, a diquaternary ammonium monomeric unit and a triquaternary monomeric unit. In one example, the cationic monomeric unit is derived

10 from MAPTAC. In another example, the cationic monomeric unit is derived from DADMAC. In still another example, the cationic monomeric unit is derived from TQ.

In one example, the cationic monomeric units are derived from cationic monomers selected from the group consisting of: dimethylaminoethyl (meth)acrylate, dimethylaminopropyl (meth)acrylate, di-tert-butylaminoethyl (meth)acrylate, dimethylaminomethyl (meth)acrylamide,

15 dimethylaminopropyl (meth)acrylamide, ethylenimine, vinylamine, 2-vinylpyridine, 4-vinylpyridine and vinyl imidazole, and mixtures thereof.

In another example, the cationic monomeric units are derived from cationic monomers selected from the group consisting of: trimethylammonium ethyl (meth)acrylate bromide, chloride or methyl sulfate, trimethylammonium ethyl (meth)acrylate bromide, chloride or methyl

20 sulfate, trimethylammonium ethyl (meth)acrylate bromide, chloride or methyl sulfate, dimethylaminoethyl (meth)acrylate benzyl chloride, 4-benzoylbenzyl dimethylammoniummethyl (meth)acrylate bromide, chloride or methyl sulfate,, trimethylammonium ethyl (meth)acrylamido bromide, chloride, or methyl sulfate, trimethylammonium propyl (meth)acrylamido braomide, chloride, or methyl sulfate, vinyl benzyl trimethyl ammonium bromide, chloride or methyl

25 sulfate, diallyldimethyl ammonium chloride, , 1-ethyl-2-vinylpyridinium bromide, chloride or methyl sulfate, 4-vinylpyridinium bromide, chloride or methyl sulfate, and mixtures thereof.

#### d. Zwitterionic Monomeric Units

Non-limiting examples of zwitterionic monomeric units suitable for the present invention include zwitterionic monomeric units derived from zwitterionic monomers selected from the

30 group consisting of: sulfobetaine monomers, such as sulfopropyl dimethylammonium ethyl methacrylate (SPE from Raschig), sulfopropyldimethylammonium propylmethacrylamide (SPP from Raschig), and sulfopropyl-2-vinylpyridinium (SPV from Raschig), phosphobetaine

monomers, such as phosphatoethyl trimethylammonium ethyl methacrylate, carboxybetaine monomers, N-(carboxymethyl)-3-methacrylamido-N,N-dimethylpropan-1-aminium chloride (CZ), 3-((3-methacrylamidopropyl)dimethylammonio)propane-1-sulfonate (SZ). In one example, the zwitterionic monomeric unit is derived from CZ, SZ, and mixtures thereof.

5           In one example, a polymer of the present invention may comprise at least one monomeric unit selected from groups a (nonionic monomeric units) and b (anionic monomeric units) and at least one monomeric unit selected from groups c (cationic monomeric units) and d (zwitterionic monomeric units).

10           In one example, the polymer comprises at least 69.9% wt and/or at least 70% wt and/or at least 75% wt and/or at least 80% wt and/or at least 85% wt and/or at least 90% wt and/or at least 95% wt and/or at least 98% wt and/or at least 99% wt and/or at least 99.5% wt of a monomeric unit from group a. The balance of the polymer (no more than 30.1% wt and/or no more than 30% wt and/or no more than 25% wt and/or no more than 20% wt and/or no more than 15% wt and/or no more than 10% wt and/or no more than 5% wt and/or no more than 2% wt and/or no more than 1% wt and/or no more than 0.5% wt total) comprises one or more monomeric units selected from groups b, c, and d.

          In one example, the polymer comprises at least 0.1% wt and/or at least 1% and/or at least 5% wt and/or at least 7% wt and/or at least 10% wt and/or to about 25% wt and/or to about 20% wt and/or to about 15% wt of a monomeric unit from group b.

20           In one example, polymer comprises at least 0.1% wt and/or at least 0.3% wt and/or at least 1% and/or at least 5% wt and/or at least 7% wt and/or at least 10% wt and/or to about 75% wt and/or to about 70% wt and/or to about 65% wt and/or to about 55% wt and/or to about 40% wt and/or to about 30% wt and/or to about 25% wt and/or to about 20% wt and/or to about 15% wt of a monomeric unit from group c.

25           In one example, polymer comprises at least 0.1% wt and/or at least 0.3% wt and/or at least 1% and/or at least 5% wt and/or at least 7% wt and/or at least 10% wt and/or to about 75% wt and/or to about 70% wt and/or to about 65% wt and/or to about 55% wt and/or to about 40% wt and/or to about 30% wt and/or to about 25% wt and/or to about 20% wt and/or to about 15% wt of a monomeric unit from group d.

30           In another example, the polymer comprises no more than 30.1% wt of a monomeric unit selected from the group consisting of: group b, group c, group d, and mixtures thereof.

In one example, the polymer may comprise a monomeric unit from group a and a monomeric unit from group b.

In one example, the polymer may comprise a monomeric unit from group a and a monomeric unit from group c.

5 In another example, the polymer of the present invention may comprise a monomeric unit from group a and a monomeric unit from group d.

In still another example, the polymer of the present invention may comprise a monomeric unit from group b and a monomeric unit from group c.

10 In still another example, the polymer of the present invention may comprise a monomeric unit from group b and a monomeric unit from group d.

In still another example, the polymer of the present invention may comprise a monomeric unit from group c and a monomeric unit from group d.

In yet another example, the polymer of the present invention may comprise a monomeric unit from group a, a monomeric unit from group b, and a monomeric unit from group c.

15 In even another example, the polymer of the present invention may comprise a monomeric unit from group a, a monomeric unit from group b, and a monomeric unit from group d.

In yet another example, the polymer of the present invention may comprise a monomeric unit from group a, a monomeric unit from group c, and a monomeric unit from group d.

20 In another example, the polymer of the present invention may comprise a monomeric unit from group b, a monomeric unit from group c, and a monomeric unit from group d.

In even yet another example, the polymer of the present invention may comprise a monomeric unit from group a, a monomeric unit from group b, a monomeric unit from group c and a monomeric unit from group d.

25 In one example, when present in the polymer, the monomeric unit from group b and the monomeric unit from group c are present in the polymer at a molar ratio of from about 3:1 to 1:3 and/or from about 2:1 to 1:2 and/or from about 1.3:1 to 1:1.3 and/or about 1:1 or less or about 1:1 or more.

30 In another example, when present in the polymer, the monomeric unit from group b and the monomeric unit from group d are present in the polymer at a molar ratio of from about 3:1 to 1:3 and/or from about 2:1 to 1:2 and/or from about 1.3:1 to 1:1.3 and/or about 1:1 or less or about 1:1 or more.

In another example, when present in the polymer, the monomeric unit from group c and the monomeric unit from group d are present in the polymer at a molar ratio of from about 3:1 to 1:3 and/or from about 2:1 to 1:2 and/or from about 1.3:1 to 1:1.3 and/or about 1:1 or less or about 1:1 or more.

5        In still another example, the polymer comprises a monomeric unit from group a and a monomeric unit from group c. For example, the polymer may comprise an acrylamide monomeric unit and a quaternary ammonium monomeric unit. The quaternary monomeric unit may be selected from the group consisting of: monoquaternary ammonium monomeric units, diquaternary ammonium monomeric units, and triquaternary ammonium monomeric units. In one  
10        example, the polymer may comprise at least 69.9% wt of the monomeric unit from group a and no more than 30.1% wt of the monomeric unit from group c.

      In still another example, the polymer comprises a monomeric unit from group a and a monomeric unit from group b. For example, the polymer may comprise an acrylamide monomeric unit and an acrylic acid monomeric unit. In one example, the polymer may comprise  
15        at least 69.9% wt of the monomeric unit from group a and no more than 30.1% wt of the monomeric unit from group b.

      In yet another example, the polymer comprises a monomeric unit from group b and a monomeric unit from group c. For example, the polymer may comprise an anionic monomeric unit derived from an anionic monomer selected from the group consisting of: acrylic acid,  
20        methacrylic acid, 2-acrylamido-2-methylpropane sulfonic acid, carboxyethyl acrylate, and mixtures thereof and a quaternary ammonium monomeric unit. The quaternary ammonium monomeric unit may be derived from a quaternary monomer selected from the group consisting of: monoquaternary ammonium monomeric units, diquaternary ammonium monomeric units, triquaternary ammonium monomeric units, and mixtures thereof. In one example, the polymer  
25        comprises an anionic monomeric unit derived from acrylic acid and a quaternary ammonium monomeric unit derived from MAPTAC. In one example, the polymer may comprise no more than 25% wt of the monomeric unit from group b and no more than 75% wt of the monomeric unit from group c.

      In even yet another example, the polymer comprises a monomeric unit from group a and a  
30        monomeric unit from group b and a monomer unit from group c. For example, the polymer may comprise an acrylamide monomeric unit, and an anionic monomeric unit derived from an anionic monomer selected from the group consisting of: acrylic acid, methacrylic acid, 2-acrylamido-2-

methylpropane sulfonic acid, carboxyethyl acrylate, and mixtures thereof and a quaternary ammonium monomeric unit. The quaternary ammonium monomeric unit may be derived from a quaternary monomer selected from the group consisting of: monoquaternary ammonium monomeric units, diquaternary ammonium monomeric units, triquaternary ammonium monomeric units, and mixtures thereof. In one example, the polymer comprises a nonionic monomeric unit derived from acrylamide, an anionic monomeric unit derived from acrylic acid, and a cationic monomeric unit derived from MAPTAC. In another example, the polymer comprises a nonionic monomeric unit derived from acrylamide, an anionic monomeric unit derived from acrylic acid, and a cationic monomeric unit derived from DADMAC. In still another example, the polymer comprises a nonionic monomeric unit derived from acrylamide, an anionic monomeric unit derived from acrylic acid, and a cationic monomeric unit derived from TQ. In another example, the polymer comprises a nonionic monomeric unit derived from acrylamide, an anionic monomeric unit derived from CEA, and a cationic monomeric unit derived from MAPTAC. In still another example, the polymer comprises a nonionic monomeric unit derived from acrylamide, an anionic monomeric unit derived from AMPS, and a cationic monomeric unit derived from MAPTAC. In one example, the polymer may comprise at least 69.9% wt of the monomeric unit from group a and no more than 30.1% wt combined of the monomeric units from groups b and c. In another example, the polymer may comprise from about 70% to about 99.5% wt of the monomeric unit from group a, from 0.1% to about 30% wt of the monomeric unit from group b, and from about 0.1% to about 30% wt of the monomeric unit from group c. In still another example, the polymer may comprise from about 70% to about 99.5% wt of the monomeric unit from group a and from about 0.5% to 30% wt combined of the monomeric units from groups b and c.

In even still yet another example, the polymer comprises a monomeric unit from group a and a monomeric unit from group c and a monomer unit from group d. For example, the polymer may comprise an acrylamide monomeric unit, a quaternary ammonium monomeric unit, and a zwitterionic monomeric unit selected from the group consisting of: CZ, SZ, and mixtures thereof. The quaternary ammonium monomeric unit may be derived from a quaternary monomer selected from the group consisting of: monoquaternary ammonium monomeric units, diquaternary ammonium monomeric units, triquaternary ammonium monomeric units, and mixtures thereof. In one example, the polymer comprises a nonionic monomeric unit derived from acrylamide, a cationic monomeric unit derived from MAPTAC, and a zwitterionic

monomeric unit derived from CZ. In another example, the polymer comprises a nonionic monomeric unit derived from acrylamide, a cationic monomeric unit derived from MAPTAC, and a zwitterionic monomeric unit derived from SZ. In one example, the polymer may comprise at least 69.9% wt of the monomeric unit from group a and no more than 30.1% wt combined of the monomeric units from groups c and d. In another example, the polymer may comprise from about 70% to about 99.5% wt of the monomeric unit from group a, from 0.1% to about 30% wt of the monomeric unit from group c, and from about 0.1% to about 30% wt of the monomeric unit from group d. In still another example, the polymer may comprise from about 70% to about 99.5% wt of the monomeric unit from group a and from about 0.5% to 30% wt combined of the monomeric units from groups c and d.

In even yet another example, the polymer comprises a monomeric unit from group a and a monomeric unit from group b and a monomer unit from group d. For example, the polymer may comprise an acrylamide monomeric unit, and an anionic monomeric unit derived from an anionic monomer selected from the group consisting of: acrylic acid, methacrylic acid, 2-acrylamido-2-methylpropane sulfonic acid, carboxyethyl acrylate, and mixtures thereof and a zwitterionic monomeric unit selected from the group consisting of: CZ, SZ, and mixtures thereof. In one example, the polymer comprises a nonionic monomeric unit derived from acrylamide, an anionic monomeric unit derived from acrylic acid, and zwitterionic monomeric unit derived from CZ. In another example, the polymer comprises a nonionic monomeric unit derived from acrylamide, an anionic monomeric unit derived from acrylic acid, and a zwitterionic monomeric unit derived from SZ. In one example, the polymer may comprise at least 69.9% wt of the monomeric unit from group a and no more than 30.1% wt combined of the monomeric units from groups b and d. In another example, the polymer may comprise from about 70% to about 99.5% wt of the monomeric unit from group a, from 0.1% to about 30% wt of the monomeric unit from group b, and from about 0.1% to about 30% wt of the monomeric unit from group d. In still another example, the polymer may comprise from about 70% to about 99.5% wt of the monomeric unit from group a and from about 0.5% to 30% wt combined of the monomeric units from groups b and d.

In even yet another example, the polymer comprises a monomeric unit from group a and a monomeric unit from group d. For example, the polymer may comprise an acrylamide monomeric unit, and a zwitterionic monomeric unit selected from the group consisting of: CZ, SZ, and mixtures thereof. In one example, the polymer comprises a nonionic monomeric unit

derived from acrylamide and zwitterionic monomeric unit derived from CZ. In another example, the polymer comprises a nonionic monomeric unit derived from acrylamide and a zwitterionic monomeric unit derived from SZ. In one example, the polymer may comprise at least 69.9% wt of the monomeric unit from group a and no more than 30.1% wt of the monomeric unit from group d. In another example, the polymer may comprise from about 70% to about 99.5% wt of the monomeric unit from group a, from 0.5% to about 30% wt of the monomeric unit from group d.

In one example, the polymer of the present invention comprises a nonionic hydrophilic monomeric unit. Non-limiting examples of suitable hydrophilic monomeric units are derived from nonionic hydrophilic monomers selected from the group consisting of: hydroxyalkyl esters of  $\alpha,\beta$ -ethylenically unsaturated acids,  $\alpha,\beta$ -ethylenically unsaturated amides,  $\alpha,\beta$ -ethylenically unsaturated monoalkyl amides,  $\alpha,\beta$ -ethylenically unsaturated dialkyl amides,  $\alpha,\beta$ -ethylenically unsaturated monomers bearing a water-soluble polyoxyalkylene segment of the poly(ethylene oxide) type,  $\alpha,\beta$ -ethylenically unsaturated monomers which are precursors of hydrophilic units or segments, vinylpyrrolidones,  $\alpha,\beta$ -ethylenically unsaturated monomers of the ureido type, and mixtures thereof. In one example, the nonionic hydrophilic monomeric unit is derived from acrylamide.

In another example, the polymer of the present invention comprises a nonionic hydrophobic monomeric unit. Non-limiting examples of suitable nonionic hydrophobic monomeric units are derived from nonionic hydrophobic monomers selected from the group consisting of: vinylaromatic monomers, vinyl halides, vinylidene halides,  $C_1$ - $C_{12}$  alkylesters of  $\alpha,\beta$ -monoethylenically unsaturated acids, vinyl esters of saturated carboxylic acids, allyl esters of saturated carboxylic acids,  $\alpha,\beta$ -monoethylenically unsaturated nitriles containing from 3 to 12 carbon atoms,  $\alpha$ -olefins, conjugated dienes, and mixtures thereof.

In one example, the polymer comprises an anionic monomeric unit. Non-limiting examples of suitable anionic monomeric units are derived from anionic monomers selected from the group consisting of: monomers having at least one carboxylic function, for instance  $\alpha,\beta$ -ethylenically unsaturated carboxylic acids or the corresponding anhydrides, monomers that are precursors of carboxylate functions, monomers having at least one sulfate or sulfonate function, monomers having at least one phosphonate or phosphate function, esters of ethylenically unsaturated phosphates, and mixtures thereof. In one example, the anionic monomeric unit is derived from an anionic monomer selected from the group consisting of: acrylic acid,



methacrylic acid, 2-acrylamido-2-methylpropane sulfonic acid, carboxyethyl acrylate, and mixtures thereof.

In one example, the polymer comprises a cationic monomeric unit. Non-limiting examples of suitable cationic monomeric units are derived from cationic monomers selected from the group consisting of: acryloyl- or acryloyloxyammonium monomers, 1-ethyl-2-vinylpyridinium or 1-ethyl-4-vinylpyridinium bromide, chloride or methyl sulfate, N,N-dialkyldiallylamine monomers, polyquaternary monomers, N,N-(dialkylamino- $\omega$ -alkyl)amides of  $\alpha,\beta$ -monoethylenically unsaturated carboxylic acids,  $\alpha,\beta$ -monoethylenically unsaturated amino esters, vinylpyridines, vinylamine, vinylimidazolines, monomers that are precursors of amine functions which give rise to primary amine functions by simple acid or base hydrolysis, and mixtures thereof. In one example, the cationic monomeric unit is derived from MAPTAC. In another example, the cationic monomeric unit is derived from DADMAC. In still another example, the cationic monomeric unit is derived from 2-hydroxy-N<sup>1</sup>-(3-(2((3-methacrylamidopropyl)dimethylamino)-acetamido)propyl)-N<sup>1</sup>, N<sup>1</sup>, N<sup>3</sup>, N<sup>3</sup>, N<sup>3</sup> - pentamethylpropane-1,3-diaminium chloride.

#### NON-LIMITING SYNTHESIS EXAMPLES

##### Sample Preparation

##### Initiator Solution Preparation

10ml of water is added to a flask along with 1 gram of 2,2'-azobis(2-methylpropionamidine) dihydrochloride (available from Wako Chemicals), herein called V-50. This solution is sparged with argon gas to remove oxygen.

##### Monomer Preparation

Synthesis of 2-Hydroxy-N<sup>1</sup>-(3-(2((3-Methacrylamidopropyl)Dimethylammonio)-Acetamido)Propyl)-N<sup>1</sup>,N<sup>1</sup>,N<sup>3</sup>,N<sup>3</sup>,N<sup>3</sup>-Pentamethylpropane-1,3-Diaminium Chloride (Herein Called TQ)

To a jacketed round bottom flask equipped with mechanical stirrer, gas inlet, condenser and thermometer is added 340.6 grams of dimethylamino propyl methacrylamide (DMPMA, available from Sigma-Aldrich), 238.8 grams of methyl chloroacetate (available from Sigma-Aldrich), 0.5 g 4-methoxy phenol (available from Sigma-Aldrich), and 423 grams of methanol (available from Sigma-Aldrich). The round bottom flask is heated at 70 °C for 5 hours. This reaction is cooled to room temperature and then 0.5 grams of 4-methoxy phenol (available from Sigma-Aldrich) and 225 grams of dimethylaminoipropylamine (available from Sigma-Aldrich) is

added evenly over a 2 hour period. After 2 hours the reaction is heated to 65 °C for 2 hours after which methanol is distilled out at 50 °C under vacuum. To this is added 690 grams of (3-chloro-2-hydroxypropyl)trimethylammonium chloride (available as a 60% aqueous solution from Sigma-Aldrich). The temperature is maintained at 65-70 °C for 2 hours. During these 2 hours  
5 methanol is stripped out and water is added to make a 55% solution in water based on weight. The reaction is continued in water at 65-70 °C for another hour to yield the TQ monomer.

Synthesis of 3-((3-Methacrylamidopropyl)Dimethylammonio)Propane-1-Sulfonate (Herein Called SZ)

Into a round bottom flask is added 26.4 grams of anhydrous acetonitrile (available from  
10 Sigma-Aldrich) and 15.5 grams of propane sultone (available from Sigma-Aldrich), and this is stirred for 30 minutes. After the 30 minutes, a solution of 25.6 grams of DMAPMA in 56.5 grams of acetonitrile is added. The mixture is stirred and warmed to 35 °C. A white precipitate quickly forms. Once the white precipitate takes up the bulk of the volume, the liquid is decanted. The solid is washed once with acetonitrile and again the liquid is removed by decanting. The  
15 solids are then washed in 2x volume diethyl ether. They are then filtered via funnel and washed with copious amounts (via filtration) of diethyl ether. The NMR structure is consistent with the structure of the target molecule SZ.

Synthesis of N-(Carboxymethyl)-3-Methacrylamido-N,N-Dimethylpropan-1-Aminium Chloride (Herein Called CZ)

To a round bottom flask is added 16.5 grams of methyl bromoacetate (available from  
20 Sigma-Aldrich), 74 grams of tetrahydrofuran (THF, available from Sigma-Aldrich), and 16.5 grams of DMAPMA. The solution is stirred for 16 hours at 25 °C, and then the stirring is discontinued. After settling, the top layer of THF is discarded. The lower layer is washed with 50 mL of hexanes (available from Sigma-Aldrich) twice and becomes a viscous material. The  
25 material is then dissolved in 15 mL of methanol (available from Sigma-Aldrich) and precipitated into 150 mL of diethyl ether (available from Sigma-Aldrich). The precipitate is washed several times with diethylether until it becomes a viscous semi-solid. It is then dried overnight under high vacuum at room temperature. A small portion is taken for NMR analysis. The remainder of the intermediate is placed in a glass desiccator containing calcium chloride until the next step.

30 3.3 grams of the intermediate from above is dissolved in 10 mL of deionized water and run through a column consisting of 50 mL of Dowex Marathon A hydroxide exchange resin (available from VWR Scientific) in a glass column of 2.5 cm diameter at 2.7 mL/min. The

effluent is collected and 13 mL of 1N hydrochloric acid (available from Sigma-Aldrich) is added. The water is dried off under vacuum at room temperature. The sample is then dried overnight under high vacuum at room temperature. The material is removed from the vacuum and a small portion is taken for NMR analysis. 2.71 g of deionized water is added to the material to form the finished product CZ which is stored as a solution in water.

#### Polymer Preparation

Into a reaction vessel are added the monomers in the amounts listed for the examples in Table 1 below and 456 g of water. The monomers, acrylamide (herein called AAM), acrylic acid (herein called AA), diallyldimethylammonium chloride (herein called DADMAC), 2-carboxy ethyl acrylate (herein called CEA), 2-acrylamido-2-methylpropane sulfonic acid (herein called AMPS) and [3-(methacryloylamino)propyl] trimethylammonium chloride (herein called MAPTAC), are all available from Sigma Aldrich. MAPTAC is used as a 50% w/w solution. TQ, SZ and CZ are used as prepared above. The reaction vessel is sparged with nitrogen to remove oxygen from the system and a nitrogen atmosphere is maintained in the vessel. The reaction vessel and contents are heated to a temperature of 60 °C.

Once the contents have reached 60 °C, the initiator solution 1 mL of the V-50 as prepared above is added as a 10% solution (except for Example 1.17 which used 0.0562 g of V-50 neat). The reaction is kept at 60 °C for 48 hours.

Mirapol<sup>®</sup> HSC 300 was obtained from Rhodia S. A. (Paris, France).

The following tables set forth non-limiting examples of polymers of the present invention that were made.

| Table 1. Examples: Polymer Construction Data |            |           |               |               |           |            |             |           |           |
|--|------------|-----------|---------------|---------------|-----------|------------|-------------|-----------|-----------|
| Ex.  | AAM<br>(g) | AA<br>(g) | MAPTAC<br>(g) | DADMAC<br>(g) | TQ<br>(g) | CEA<br>(g) | AMPS<br>(g) | SZ<br>(g) | CZ<br>(g) |
| 1.1  | 21.60      | 0.00      | 2.40          | 0.00          | 0.00      | 0.00       | 0.00        | 0.00      | 0.00      |
| 1.2  | 21.60      | 0.31      | 2.09          | 0.00          | 0.00      | 0.00       | 0.00        | 0.00      | 0.00      |
| 1.3  | 21.60      | 0.60      | 1.81          | 0.00          | 0.00      | 0.00       | 0.00        | 0.00      | 0.00      |
| 1.4  | 21.60      | 1.20      | 1.21          | 0.00          | 0.00      | 0.00       | 0.00        | 0.00      | 0.00      |
| 1.5  | 21.60      | 1.80      | 0.61          | 0.00          | 0.00      | 0.00       | 0.00        | 0.00      | 0.00      |
| 1.6  | 21.59      | 2.40      | 0.00          | 0.00          | 0.00      | 0.00       | 0.00        | 0.00      | 0.00      |
| 1.7  | 0.00       | 6.00      | 18.00         | 0.00          | 0.00      | 0.00       | 0.00        | 0.00      | 0.00      |
| 1.8  | 2.41       | 5.40      | 16.20         | 0.00          | 0.00      | 0.00       | 0.00        | 0.00      | 0.00      |
| 1.9  | 7.20       | 4.20      | 12.60         | 0.00          | 0.00      | 0.00       | 0.00        | 0.00      | 0.00      |
| 1.10   | 12.00      | 3.00      | 9.00          | 0.00          | 0.00      | 0.00       | 0.00        | 0.00      | 0.00      |
| 1.11   | 16.79      | 1.81      | 5.42          | 0.00          | 0.00      | 0.00       | 0.00        | 0.00      | 0.00      |
| 1.12   | 19.22      | 1.20      | 3.60          | 0.00          | 0.00      | 0.00       | 0.00        | 0.00      | 0.00      |
| 1.13   | 20.41      | 0.90      | 2.70          | 0.00          | 0.00      | 0.00       | 0.00        | 0.00      | 0.00      |
| 1.14   | 21.61      | 0.60      | 1.80          | 0.00          | 0.00      | 0.00       | 0.00        | 0.00      | 0.00      |
| 1.15   | 22.81      | 0.31      | 0.92          | 0.00          | 0.00      | 0.00       | 0.00        | 0.00      | 0.00      |

|      |       |        |       |      |       |        |      |       |       |
|------|-------|--------|-------|------|-------|--------|------|-------|-------|
| 1.16 | 23.51 | 0.12   | 0.36  | 0.00 | 0.00  | 0.00   | 0.00 | 0.00  | 0.00  |
| 1.17 | 23.75 | 0.06   | 0.18  | 0.00 | 0.00  | 0.00   | 0.00 | 0.00  | 0.00  |
| 1.18 | 23.76 | 0.06   | 0.18  | 0.00 | 0.00  | 0.00   | 0.00 | 0.00  | 0.00  |
| 1.19 | 23.87 | 0.03   | 0.10  | 0.00 | 0.00  | 0.00   | 0.00 | 0.00  | 0.00  |
| 1.20 | 24.09 | 0.00   | 0.00  | 0.00 | 0.00  | 0.00   | 0.00 | 0.00  | 0.00  |
| 1.21 | 23.76 | 0.07   | 0.00  | 0.17 | 0.00  | 0.00   | 0.00 | 0.00  | 0.00  |
| 1.22 | 23.77 | 0.0285 | 0.00  | 0.00 | 0.212 | 0.00   | 0.00 | 0.00  | 0.00  |
| 1.23 | 23.76 | 0.00   | 0.145 | 0.00 | 0.00  | 0.0939 | 0.00 | 0.00  | 0.00  |
| 1.24 | 23.76 | 0.00   | 0.13  | 0.00 | 0.00  | 0.00   | 0.12 | 0.00  | 0.00  |
| 1.25 | 23.77 | 0.00   | 0.00  | 0.00 | 0.00  | 0.00   | 0.00 | 0.252 | 0.00  |
| 1.26 | 23.76 | 0.00   | 0.00  | 0.00 | 0.00  | 0.00   | 0.00 | 0.00  | 0.240 |
| 1.27 | 23.52 | 0.00   | 0.00  | 0.00 | 0.00  | 0.00   | 0.00 | 0.00  | 0.479 |
| 1.28 | 23.76 | 0.00   | 0.003 | 0.00 | 0.00  | 0.00   | 0.00 | 0.00  | 0.240 |
| 1.29 | 23.76 | 0.002  | 0.00  | 0.00 | 0.00  | 0.00   | 0.00 | 0.00  | 0.240 |

| Table 2. Examples: Polymer Solution Data |   |            |                      |                              |           |
|--|---|------------|----------------------|------------------------------|-----------|
| Ex.                                      | Mass Composition of Monomers                      | Solids (%) | Polymer Solution (g) | Polymer Solution + Water (g) | Conc. (%) |
| 2.1                                      | 90% AAM, 10% MAPTAC                               | 5.44       | 0.4253               | 115.68                       | 0.02      |
| 2.2                                      | 90% AAM, 1.3% AA, 8.7% MAPTAC                     | 5.41       | 0.3927               | 106.24                       | 0.02      |
| 2.3                                      | 90% AAM, 2.5% AA, 7.5% MAPTAC                     | 5.45       | 0.4013               | 109.34                       | 0.02      |
| 2.4                                      | 90% AAM, 5% AA, 5% MAPTAC                         | 5.43       | 0.3974               | 107.89                       | 0.02      |
| 2.5                                      | 90% AAM, 7.5% AA, 2.5% MAPTAC                     | 5.42       | 0.7522               | 203.84                       | 0.02      |
| 2.6                                      | 90% AAM, 10% AA                                   | 5.42       | 0.3985               | 108.00                       | 0.02      |
| 2.7                                      | 25% AA, 75% MAPTAC                                | 5.25       | 0.3823               | 100.36                       | 0.02      |
| 2.8                                      | 10% AAM, 22.5% AA, 67.5% MAPTAC                   | 5.24       | 0.3788               | 99.27                        | 0.02      |
| 2.9                                      | 30% AAM, 17.5% AA, 52.5% MAPTAC                   | 5.26       | 0.3979               | 104.64                       | 0.02      |
| 2.10                                     | 50% AAM, 12.5% AA, 37.5% MAPTAC                   | 5.36       | 0.3692               | 98.95                        | 0.02      |
| 2.11                                     | 69.9% AAM, 7.5% AA, 22.6% MAPTAC                  | 5.30       | 0.3810               | 100.97                       | 0.02      |
| 2.12                                     | 80% AAM, 5% AA, 15% MAPTAC                        | 5.31       | 0.3899               | 103.53                       | 0.02      |
| 2.13                                     | 85% AAM, 3.7% AA, 11.3% MAPTAC                    | 5.30       | 0.4403               | 116.69                       | 0.02      |
| 2.14                                     | 90% AAM, 2.5% AA, 7.5% MAPTAC                     | 5.26       | 0.3800               | 99.93                        | 0.02      |
| 2.15                                     | 94.9% AAM, 1.3% AA, 3.8% MAPTAC                   | 5.34       | 0.3982               | 106.34                       | 0.02      |
| 2.16                                     | 98% AAM, 0.5% AA, 1.5% MAPTAC                     | 2.54       | 0.7969               | 101.21                       | 0.02      |
| 2.17                                     | 99% AAM, 0.25% AA, 0.75% MAPTAC                   | 2.56       | 0.7944               | 101.68                       | 0.02      |
| 2.18                                     | 99% AAM, 0.25% AA, 0.75% MAPTAC                   | 5.32       | 0.3751               | 100.49                       | 0.02      |
| 2.19                                     | 99.5% AAM, 0.125% AA, 0.375% MAPTAC               | 2.57       | 0.7850               | 100.89                       | 0.02      |
| 2.20                                     | 100% AAM (Comparative Example)                    | 5.23       | 0.3979               | 104.02                       | 0.02      |
| 2.21                                     | 99% AAM, 0.3% AA, 0.7% DADMAC                     | 5.40       | 0.3876               | 104.70                       | 0.02      |
| 2.22                                     | 99% AAM, 0.12% AA, 0.88% TQ                       | 5.16       | 3.8100               | 980.46                       | 0.02      |
| 2.23                                     | 99.01% AAM, 0.39% CEA, 0.6% MAPTAC                | 5.27       | 0.3914               | 103.13                       | 0.02      |
| 2.24                                     | 99% AAM, 0.5% AMPS, 0.5% MAPTAC                   | 5.40       | 0.3823               | 103.22                       | 0.02      |
| 2.25                                     | 98.95% AAM, 1.05% SZ                              | 5.29       | 0.3791               | 100.25                       | 0.02      |
| 2.26                                     | 99% AAM, 1% CZ                                    | 5.28       | 0.4004               | 105.73                       | 0.02      |
| 2.27                                     | 98% AAM, 2% CZ                                    | 5.13       | 0.4055               | 104.15                       | 0.02      |
| 2.28                                     | 98.99% AAM, 0.01% MAPTAC, 1% CZ                   | 5.15       | 0.5177               | 133.36                       | 0.02      |
| 2.29                                     | 98.99% AAM, 0.01% AA, 1% CZ                       | 5.14       | 0.5941               | 152.90                       | 0.02      |
| 2.30                                     | Mirapol <sup>®</sup> HSC300 (Comparative Example) | 20.81      | 0.1378               | 143.38                       | 0.02      |
| 2.31                                     | Deionized Water (Control)                         |            |                      |                              |           |

| Table 3. Test Results |   |           |       |                            |             |                                    |            |
|-----------------------|---|-----------|-------|----------------------------|-------------|------------------------------------|------------|
| Ex.                   | Mass Composition of Monomers                          | Mn        | PDI   | Soil Adsorption Value (mg) | St Dev (mg) | % Soil Retained <sub>avg</sub> (%) | St Dev (%) |
| 3.1                   | 90% AAM, 10% MAPTAC                                   | 1,211,000 | 1.240 | 41                         | 1           | 23                                 | 1          |
| 3.2                   | 90% AAM, 1.3% AA, 8.7% MAPTAC                         | 948,200   | 1.239 | 42                         | 6           | 24                                 | 3          |
| 3.3                   | 90% AAM, 2.5% AA, 7.5% MAPTAC                         | 852,500   | 1.351 | 47                         | 2           | 26                                 | 1          |
| 3.4                   | 90% AAM, 5% AA, 5% MAPTAC                             | 753,500   | 1.402 | 40                         | 3           | 23                                 | 2          |
| 3.5                   | 90% AAM, 7.5% AA, 2.5% MAPTAC                         | 970,300   | 1.271 | 43                         | 3           | 24                                 | 2          |
| 3.6                   | 90% AAM, 10% AA                                       | 1,021,000 | 1.222 | 46                         | 1           | 26                                 | 0          |
| 3.7                   | 25% AA, 75% MAPTAC                                    | 201,500   | 1.823 | 44                         | 3           | 24                                 | 2          |
| 3.8                   | 10% AAM, 22.5% AA, 67.5% MAPTAC (Comparative Example) | 226,400   | 1.712 | 32                         | 1           | 18                                 | 1          |
| 3.9                   | 30% AAM, 17.5% AA, 52.5% MAPTAC (Comparative Example) | 311,800   | 1.604 | 32                         | 2           | 18                                 | 1          |
| 3.10                  | 50% AAM, 12.5% AA, 37.5% MAPTAC (Comparative Example) | 583,800   | 1.406 | 34                         | 3           | 19                                 | 2          |
| 3.11                  | 69.9% AAM, 7.5% AA, 22.6% MAPTAC                      |           |       | 38                         | 1           | 21                                 | 1          |
| 3.12                  | 80% AAM, 5% AA, 15% MAPTAC                            | 821,000   | 1.269 | 40                         | 1           | 23                                 | 1          |
| 3.13                  | 85% AAM, 3.7% AA, 11.3% MAPTAC                        | 865,600   | 1.241 | 44                         | 3           | 25                                 | 2          |
| 3.14                  | 90% AAM, 2.5% AA, 7.5% MAPTAC                         |           |       | 45                         | 0           | 25                                 | 0          |
| 3.15                  | 94.9% AAM, 1.3% AA, 3.8% MAPTAC                       | 927,100   | 1.222 | 53                         | 3           | 30                                 | 1          |
| 3.16                  | 98% AAM, 0.5% AA, 1.5% MAPTAC                         |           |       | 55                         | 3           | 31                                 | 2          |
| 3.17                  | 99% AAM, 0.25% AA, 0.75% MAPTAC                       | 858,100   | 1.302 | 57                         | 3           | 32                                 | 2          |
| 3.18                  | 99% AAM, 0.25% AA, 0.75% MAPTAC                       | 814,200   | 1.293 | 57                         | 5           | 32                                 | 3          |
| 3.19                  | 99.5% AAM, 0.125% AA, 0.375% MAPTAC                   | 1,212,000 | 1.285 | 65                         | 3           | 36                                 | 2          |
| 3.20                  | 100% AAM (Comparative Example)                        | 1,116,600 | 1.204 | 40                         | 3           | 22                                 | 2          |
| 3.21                  | 99% AAM, 0.3% AA, 0.7% DADMAC                         | 520,400   | 1.432 | 53                         | 4           | 30                                 | 2          |
| 3.22                  | 99% AAM, 0.12% AA, 0.88% TQ                           | 1,050,000 | 1.165 | 54                         | 2           | 30                                 | 1          |
| 3.23                  | 99.01% AAM, 0.39% CEA, 0.6% MAPTAC                    | 791,200   | 1.219 | 61                         | 4           | 34                                 | 2          |
| 3.24                  | 99% AAM, 0.5% AMPS, 0.5% MAPTAC                       | 644,400   | 1.579 | 59                         | 2           | 33                                 | 1          |
| 3.25                  | 98.95% AAM, 1.05% SZ                                  | 542,800   | 1.566 | 54                         | 4           | 30                                 | 2          |
| 3.26                  | 99% AAM, 1% CZ  | 862,700   | 1.269 | 57                         | 3           | 32                                 | 1          |
| 3.27                  | 98% AAM, 2% CZ  |           |       | 62                         | 2           | 35                                 | 1          |
| 3.28                  | 98.99% AAM, 0.01% MAPTAC, 1% CZ                       |           |       | 60                         | 4           | 33                                 | 2          |
| 3.29                  | 98.99% AAM, 0.01% AA, 1% CZ                           |           |       | 60                         | 2           | 33                                 | 1          |
| 3.30                  | Mirapol <sup>®</sup> HSC300 (Comparative Example)     |           |       | 34                         | 3           | 19                                 | 1          |
| 3.31                  | Deionized Water                                       |           |       | 20                         | 4           | 11                                 | 2          |

## TEST METHODS

### Determination of Percent Solids

- 5 An empty weigh pan (VWR disposable aluminum crinkle dishes with tabs, VWR Catalog #25433-010; or equivalent pan) is weighed to within  $\pm 0.1$  mg (Weight<sub>Pan</sub>). An aliquot of polymer solution as prepared above,  $2.5 \pm 0.5$  grams, is placed into the pan and weighed to within  $\pm 0.1$  mg (Weight<sub>Pan + Polymer Solution</sub>). The pan and the polymer solution are placed in an 80 °C ventilated oven, uncovered for 12 hours. After cooling to room temperature, the pan and the

polymer solids are then weighed to within  $\pm 0.1$  mg ( $Weight_{Pan + Polymer\ Solid}$ ). The percent solids is calculated as follows:

$$PercentSolids(\%) = \left( \frac{Weight_{Pan+PolymerSolid} - Weight_{Pan}}{Weight_{Pan+PolymerSolution} - Weight_{Pan}} \right) * 100\%$$

#### 5 Preparation of 0.02% Polymer Solution

Using the amounts listed in Table 2, the polymer solutions prepared above are diluted to 0.02% with deionized water. A receiving vessel large enough to hold the diluted solution is tared. The desired amount of the original polymer solution is added to the receiving vessel and the weight (of the solution only) recorded to within  $\pm 1$  mg ( $Weight_{Polymer\ Solution}$ ). The polymer solution is then diluted to 0.02% with deionized water and the weight recorded to within  $\pm 0.01$  g ( $Weight_{Polymer\ Solution + Water}$ ). The diluted solutions are capped and allowed to sit for 24 hours with occasional agitation prior to use to ensure polymer dissolution. The concentration is calculated as follows:

$$Concentration(\%) = \frac{Weight_{PolymerSolution} * PercentSolids}{Weight_{PolymerSolution+Water}}$$

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#### Polymer Molecular Weight Determination

Polymer molecular mass is determined by GPC SEC/MALS. The HPLC is a Waters Alliance 2695 HPLC with an auto injector equipped with a bank of two linear  $\mu$ Styragel HT columns at room temperature. The flow rate is 1.0 mL/min and the mobile phase is dimethyl sulfoxide (DMSO) with 0.1% (weight/volume) LiBr. The detectors are Wyatt Dawn EOS Light scattering detector calibrated with toluene and normalized using 25K dextran in mobile phase and a Wyatt Optilab rEX refractive index detector at 30 °C.

Samples for analysis are prepared at a known concentration in the range of 1 to 5 mg/mL. Samples are filtered using 0.2  $\mu$ m polypropylene membrane filters. The injection volume is 100  $\mu$ L. The data are collected and analyzed using ASTRA 5.3.4.14. Values for  $dn/dc$  are calculated from the RI trace assuming 100% mass recovery. Number average molecular weight and polydispersity index are calculated and reported.

#### Soil Adsorption Test

A rectilinear 3.00 inch x 4.00 inch piece of a handsheet prepared and treated as set forth below is cut, if necessary, using a 3 inch x 4 inch die cutter to provide a sample portion having a

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basis weight of from  $19 \text{ g/m}^2$  to  $33 \text{ g/m}^2$  (sample portions outside this range are discarded). All specimens are obtained from a portion of the test material at least 0.5 inches from any edges. The handsheet is labeled with the specimen name using a ball-point pen or equivalent marker. After the handsheet has been conditioned in the conditioned room at  $70^\circ\text{F} \pm 2^\circ\text{F}$  and a relative humidity of  $50\% \pm 2\%$  for at least 2 hours (preferably overnight), the handsheet is weighed to within  $\pm 10 \text{ mg}$  ( $\text{Weight}_{\text{Substrate}}$ ) while still maintaining the conditioning conditions. The remainder of the work is done in a laboratory at a temperature of  $73^\circ\text{F} \pm 3.5^\circ\text{F}$  and a relative humidity  $< 70\%$ . The handsheet is then placed on a lattice ( $23.75'' \times 47.75''$  polystyrene light panel manufactured by Plaskolite, Inc., Columbus, Ohio, available from Home Depot as model #1425005A; or equivalent lattice). Each handsheet is then treated with a total of 3.8 mL (in 1-4 aliquots to avoid oversaturation if necessary) of the 0.02% diluted polymer solution prepared as described above. The 0.02% polymer solution is applied to the upper (treated) side of the handsheet only. At least 1.5 hours between aliquots is given to allow the handsheet to at least partially dry. After application of all the polymer solution, the handsheet are left to air dry for at least 4 hours on the lattice.

Once the handsheet is dry, the handsheet is folded in half with the treated side facing in so that the handsheet forms a  $1.5'' \times 4''$  testing strip. An accordion style (paper fan) folding technique is then used to fold the testing strip 5 times to produce a testing strip that contains 6 segments each about  $\frac{2}{3}''$  in width.

A Petri dish (VWR sterile Petri dish, Simport plastics, 60 mm x 15 mm, 28 mL volume, VWR Catalog #60872-306) is labeled with the handsheet name and weighed to within  $\pm 1 \text{ mg}$  ( $\text{Weight}_{\text{Dish}}$ ).

A capped centrifuge tube containing a model soil and water prepared according to the Soil Solution Preparation set forth below is then agitated/shaken to disperse the model soil in the water to form a soil dispersion. The centrifuge tube is then uncapped permitting the testing strip to be fully immersed into the soil dispersion so that the folds of the testing strip run parallel to the length of the centrifuge tube. The centrifuge tube is then immediately re-capped and shaken in a WS 180° shaker for  $60 \pm 1$  seconds. The WS 180° shaker (Glas-Col #099AWS18012) is set at 50% speed so that it inverts the specimen 160-170° every 1 second.

After shaking, the testing strip is carefully removed over a Petri dish using laboratory tweezers. Care must be taken to ensure that all of the soil dispersion is kept either in the original centrifuge tube or corresponding Petri dish. The soil dispersion is wrung from the testing strip

using a “wringing” motion and collected in the Petri dish ( $\geq 85\%$  of the soil dispersion should be collected). Once the soil dispersion has been removed from the testing strip, the testing strip is discarded. The remaining soil dispersion is poured from the centrifuge tube into the Petri dish after swirling the mixture to re-disperse the model soil into the water, thereby ensuring that no model soil is inadvertently left behind in the centrifuge tube. The Petri dish containing the soil dispersion is weighed to within  $\pm 1$  mg ( $\text{Weight}_{\text{Dish+Effluent}}$ ). The Petri dish is then placed into a vented laboratory drying oven at  $60^\circ\text{C}$  until the sample is dry, preferably overnight. Once the specimen is dry, the Petri dish is removed from the oven and allowed to cool to  $73^\circ\text{F} \pm 4^\circ\text{F}$ . The Petri dish is then re-weighed to within  $\pm 1$  mg ( $\text{Weight}_{\text{Dish+DriedSoil}}$ ).

Soil Solution Preparation - A centrifuge tube (VWR brand 50 mL superclear ultra high performance freestanding centrifuge tube with flat cap, VWR Catalog #82018-052; or equivalent tube) is labeled with the specimen name and weighed to within  $\pm 1$  mg ( $\text{Weight}_{\text{Vial + Cap}}$ ). Next  $0.1784 \text{ g} \pm 0.0005 \text{ g}$  of a model soil (Black Todd Clay available from Empirical Manufacturing Co., 7616 Reinhold Drive, Cincinnati, Ohio 45237-3208) is weighed ( $\text{Weight}_{\text{Added Soil}}$ ) and then placed into the centrifuge tube. Deionized water,  $25.0 \text{ mL} \pm 0.2 \text{ mL}$ , is added slowly to the centrifuge tube using a suitable dispenser. The deionized water is poured carefully into the centrifuge tube to avoid causing a plume of dust from the model soil. If a plume of dust occurs, the centrifuge tube is discarded and a new centrifuge tube is prepared. The centrifuge tube is then re-weighed to within  $\pm 1$  mg ( $\text{Weight}_{\text{Vial + Cap + Dispersion}}$ ).

Preparation of Handsheet - In order to test the soil adsorption properties of a material, such as a polymer, a handsheet is prepared as follows and is then used in the Soil Adsorption Test Method described above.

A handsheet is a hand made specimen of a fibrous structure. Handsheets are prepared at target basis weight of  $26.8 \text{ g/m}^2$ , but no less than  $19 \text{ g/m}^2$  and no more than  $33 \text{ g/m}^2$  using the following procedure.

a. Pulp Preparation - A pulp slurry of Northern Softwood Kraft (NSK) pulp is made as follows. Using an analytical balance capable of weighing to  $\pm 0.0002 \text{ g}$ , weigh out  $30 \text{ g}$  of NSK dry lap (pulp). Record the weight of the NSK dry lap. Record the percent bone-dry pulp or consistency for this pulp. Put  $500 \text{ mL}$  of  $23^\circ\text{C} \pm 2^\circ\text{C}$  of City of Cincinnati, Ohio Water (or equivalent having the following properties: Total Hardness =  $155 \text{ mg/L}$  as  $\text{CaCO}_3$ ; Calcium content =  $33.2 \text{ mg/L}$ ; Magnesium content =  $17.5 \text{ mg/L}$ ; Phosphate content =  $0.0462$ ) into a  $2000 \text{ mL}$  polypropylene beaker. Add the weighed NSK dry lap to the water in the beaker immediately



following the addition of the water to the beaker. After the NSK dry lap is completely wetted (about 50-60 seconds), remove the wetted NSK dry lap and manually tear into small pieces of wetted NSK dry lap, approximately 2 cm<sup>2</sup> or less pieces. Add the small pieces of wetted NSK dry lap back into the water in the beaker. Let the wetted NSK dry lap soak in the water for at least 1 hour, typically 1-2 hours. At the end of the soaking period, transfer the contents of the beaker (water and pulp) to a disintegrator tank of a pulp disintegrator commercially available from Testing Machines, Inc. under the tradename 73-18 Pulp Disintegrator or its equivalent. Follow the manufacturer's instructions for maintaining, calibrating, and cleaning the disintegrator, as needed. The disintegrator must meet TAPPI Standard T-205. Using more of the City of Cincinnati, Ohio water (or equivalent water as described above) delivered by a polyethylene wash bottle, wash and remove any remaining pulp adhering to the beaker into the disintegrator tank. Additional City of Cincinnati, Ohio water (or equivalent water as described above) is added to the disintegrator tank to result in a total of 1500 mL of total volume in the disintegrator tank.

Next, place the disintegrator tank containing the pulp and City of Cincinnati, Ohio water (or equivalent water as described above) (23°C ± 2°C) on the disintegrator's platform and position it under the shaft and impeller blade of the disintegrator. Clamp the disintegrator tank firmly in place on the disintegrator's platform. Lower the impeller blade into position and lock in place according to the manufacturer's instructions. Put the disintegrator tank's lid in place on the disintegrator tank. Set an interval timer with timed switch outlet for exactly 10 minutes. Turn the disintegrator on and start the timer with the alarm on the timer turned on such that the alarm sounds and the disintegrator turns off automatically after exactly 10 minutes of operation. Turn the alarm off. Use the pulp slurry (pulp plus City of Cincinnati, Ohio water (or equivalent water as described above)) in the disintegrator within an hour after the completion of the 10 minutes of operation. Do not let the pulp slurry stand idle for more than an hour before using it to make the handsheets.

**b. Proportioning of Pulp** - After the pulp slurry is prepared in the disintegrator tank as described above, the pulp slurry is then proportioned in a proportioner, such as a Noble and Wood Handsheet Forming Machine or a proportioner and handsheet forming machine, which is commercially available from Adirondack Machine Corporation as follows.

To a proportioner having a 19-21 L stainless steel tank, City of Cincinnati, Ohio water (or equivalent water as described above) is added to fill the tank to about half full (about 9-10 L).

The agitator of the proportioner is turned on and the speed of the agitator is adjusted to 23 rpm  $\pm$  2 rpm to provide good mixing once the pulp slurry is added. Good mixing can be determined by seeing that the pulp slurry is evenly mixing with the City of Cincinnati, Ohio water (or equivalent water as described above) that is added to the tank. Next, add the equivalent of 30 g of bone-dry  
5 pulp of the pulp slurry produced above to the tank. After addition of the pulp slurry to the tank, set the volume scale of the proportioner to the 19 L mark. Add additional City of Cincinnati, Ohio water (or equivalent water as described above) to make the liquid level approximately even with the top of the hook on the solution indicator pointer of the proportioner.

c. Forming Handsheet - A handsheet is made from the pulp slurry present in the  
10 proportioner, described above, as follows.

The handsheet is made using a 12" x 12" stainless steel sheet mold commercially available from Adirondack Machine Corporation. First, open the drain valve on the deckle box of the sheet mold and completely drain the deckle box. The deckle box needs to be clean and free of contaminants. Close the drain valve and open the deckle box. Turn on the water supply,  
15 City of Cincinnati, Ohio water (or equivalent water as described above) and allow the deckle box to overflow. Place a clean forming wire (84M 14" x 14" polyester monofilament plastic cloth, commercially available from Appleton Wire Co.), on the coarse deckle box wire so as not to entrap any air bubbles under the forming wire. If air bubbles persist, eliminate by rubbing the wire gently with hands before closing the deckle box. Air bubbles under the forming wire, if not  
20 removed, will cause holes in the handsheet and makes the handsheet unacceptable for use in the tests described herein.

After the forming wire has been thoroughly wetted by the water, close and lock the deckle box and allow the water to rise to 8 1/2" from the forming wire in the deckle box. A mark on the inside of the deckle box should be used to permanently indicate this volume. Add 2543 mL of  
25 the pulp slurry from the proportioner to the water in the deckle box using the proportioner sample container. Using the perforated metal deckle box plunger, distribute the pulp slurry uniformly by moving the plunger from near the top of the pulp slurry to the bottom of the pulp slurry within the deckle box and back for three complete up and down cycles. Do not touch the forming wire on the downward strokes. After the third cycle, bring the plunger up and pause for two seconds  
30 holding the plunger plate just beneath the pulp slurry surface (to eliminate wave action) and then withdraw slowly. Make sure that the pulp slurry is undisturbed in the deckle box.

Depress the switch to activate the timed opening of the drop valve of the deckle box. The drop valve will close automatically after the deckle box is completely drained. Most units completely drain in about 20-25 seconds. After the drop valve closes, open the deckle box and carefully remove the forming wire with fiber mat side up from the deckle box. Immediately

5 place the forming wire with fiber mat side up on a vacuum box's surface (a vacuum box table) having a surface at a vacuum slot (13" x 1/16" 90° flare) over which the forming wire with fiber mat passes. Keep the edge of the forming wire which is next to the operator in the same relative position during this transfer from the deckle box to the vacuum box table.

The vacuum box table's vacuum valves are set such that the low level of vacuum (pre-

10 vacuum) peaks at  $4.0 \pm 0.5$ " Hg and the high level vacuum peaks at  $10.0 \pm 0.5$ " Hg according to an Ashcroft Vacuum Gauge Model 1189, range 0-15" Hg commercially available from Ashcroft Inc.

Turn on the vacuum pump (a Nash H4 Pump with a draw of 106 cfm Motor-10 HP, 1745 rpm, 3 Ph, 60 Hz available from ECM Inc.) associated with the vacuum box table. Engage the

15 low level vacuum (pre-vacuum). Position the forming wire with the fiber mat side up on the vacuum box table so that the front edge of the forming wire (edge next to the operator) extends over the vacuum slot about  $\frac{1}{4}$ " –  $\frac{1}{2}$ ". Pull the forming wire with fiber mat across the vacuum slot in  $1 \pm 0.3$  seconds at a uniform rate. The vacuum gauge should peak at  $4.0 \pm 0.5$ " Hg. This step is referred to as the Pre-vacuum Step.

20 Next, turn the low level vacuum and open the high level side of the vacuum system. Place the knobby side up of a transfer wire (44M 16" x 14" polyester monofilament plastic cloth commercially available from Appleton Wire Co. with the knobby side, which is the sheet side, marked with an arrow indicating the machine direction) on the vacuum box table behind the vacuum slot. The transfer wire is placed on the vacuum box table such that the 16" length is

25 perpendicular to the vacuum slot. Carefully turn the forming wire with the fiber mat over keeping the edge of the forming wire, which has been next to the operator, in the same relative position. Gently place the forming wire with fiber mat onto the center of the transfer wire, forming a "sandwich" so that the front edge of the transfer wire (edge next to the operator) extends over the vacuum slot about  $\frac{1}{4}$ " –  $\frac{1}{2}$ ". The direction of travel of the fiber mat over the

30 vacuum slot must be identical to the direction of travel of the forming wire with fiber mat during the Pre-vacuum Step described above. The "sandwich" is pulled across the vacuum slot in  $1 \pm 0.3$  seconds at a uniform rate. The vacuum gauge should peak at  $10.0 \pm 0.5$ " Hg. This step,

which transfers the fiber mat from the forming wire to the transfer wire, is called the Transfer Vacuum Step.

Close the high level vacuum and turn off the entire vacuum system. By this time the fiber mat has become a handsheet. Next, place the “sandwich” on the vacuum box table. Separate the forming wire from the handsheet and the transfer wire by gently lifting one corner of the forming wire and removing it, leaving the handsheet attached to the transfer wire. Keep the edge of the fabric next to the operator in the same relative position as the handsheet as it was during the Transfer Vacuum Step. Make an arrow with an indelible pencil (a water color pencil commercially available from Dick Blick Art Supplies) on a corner of the handsheet to indicate the direction of travel across the vacuum slot. This identifies the handsheet’s machine direction.

Next, pass the transfer wire with the handsheet attached through an E-100 Drum Dryer commercially available from Adirondack Machine Corporation with the transfer wire next to the drum dryer and with the edge that was kept next to the operator going into the drum dryer last. Pass the transfer wire with the handsheet attached through the drum dryer a second time with the handsheet next to the drum dryer.

The handsheet is removed immediately after exiting the dryer drum the second time while it is still warm.

The handsheet formed must be at a target basis weight of 26.8 g/m<sup>2</sup>, but no less than 19 g/m<sup>2</sup> and no more than 33 g/m<sup>2</sup> suitable for testing. If the basis weight is less than 19 g/m<sup>2</sup> or greater than 33 g/m<sup>2</sup> then either the amount of pulp is too small or too large and the process needs to be adjusted accordingly to produce a handsheet with a target basis weight of 26.8 g/m<sup>2</sup>, but no less than 19 g/m<sup>2</sup> and no more than 33 g/m<sup>2</sup>.

#### Calculations

To calculate the amount of residual model soil (Mass<sub>Residual Soil</sub>) left in the Petri dish, the following equation is used:

$$Mass_{ResidualSoil} = Weight_{Dish+DriedSoil} - Weight_{Dish}$$

Residual model soil is reported in mg.

To calculate the amount of soil adsorbed (Soil Retained) in the specimen, the following calculation is used:

$$Soil\ Retained = Weight_{AddedSoil} - Mass_{ResidualSoil}$$

The amount of soil adsorbed is reported in mg.

To calculate the percent of soil retained (% Soil Retained), the following calculation is used:

$$\% \text{Soil Retained} = \left[ \frac{\text{Soil Retained}}{\text{Weight}_{\text{AddedSoil}}} \right] * 100\%$$

- The test is performed on four replicates and the average amount of soil adsorbed (also known as the Soil Adsorption Value) and the average percent of soil retained (%Soil Retained<sub>avg</sub>) are calculated for the material.

#### Charge Density Test Method

The charge density of a polymer, such as a soil adsorption polymer, can be determined by using a Mutek PCD-04 Particle Charge Detector available from BTG, or equivalent instrument.

- The following guidelines provided by BTG are used.

Start with a 0.1% solution (0.1 g polymer + 99.9 g deionized water) (sample). Depending on the titrant consumption increase or decrease polymer content if needed. Solution pH is adjusted prior to final dilution as charge density of many polymers and/or additives is dependent upon solution pH. A pH of 4.5 is used here.

1. Place 20 mL of sample in the PCD measuring cell and insert piston.
2. Put the measuring cell with piston and sample in the PCD, the electrodes are facing the rear. Slide the cell along the guide until it touches the rear.
3. Pull piston upwards and turn it counter-clock-wise to lock the piston in place.
4. Switch on the motor. The streaming potential is shown on the touch panel. Wait 2 minutes until the signal is stable.
5. Use an oppositely charged titrant (for example for a cationic sample having a positive streaming potential: use an anionic titrant). Titrants are available from BTG consisting of 0.001N PVSK or 0.001N PolyDADMAC.
6. An automatic titrator available from BTG is utilized. After selecting the proper titrant, set the titrator to rinse the tubing by dispensing 10 mL insuring that all air bubbles have been purged.
7. Place tubing tip below the surface of the sample and start titration. The automatic titrator is set to stop automatically when the potential reaches 0 mV.
8. Record consumption of titrant, ideally, the consumption of titrant should be 0.2 mL to 10 mL; otherwise decrease or increase polymer content.
9. Repeat titration of a second 20 mL aliquot of the polymer sample.

10. Calculate charge demand (solution) or charge demand (solids);

$$\text{Charge demand (eq/L)} = \frac{\text{V titrant used(L)} \times \text{Conc. of titrant in Normality (eq/L)}}{\text{Volume of sample titrated (L)}}$$

5

$$\text{Charge demand (eq/g)} = \frac{\text{V titrant used(L)} \times \text{Conc. of titrant in Normality (eq/L)}}{\text{Wt. solids of the sample or its active substance (g)}}$$

The charge demand (charge density) of a polymer is reported in meq/g units.

#### 10 Basis Weight Test Method

The rectilinear 3.00 inch x 4.00 inch piece of specimen cut as above in the soil adsorption test method is conditioned in a conditioned room at 70 °F ± 2 °F and a relative humidity of 50% ± 2% for at least 2 hours, typically overnight. The specimen is weighed to within ±10 mg ( $\text{Weight}_{\text{Substrate}}$ ) while still maintaining the conditioning conditions. The Basis Weight of the specimen is then calculated as follows:

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$$\text{BasisWeight(gsm)} = \left( \frac{\text{Weight}_{\text{Substrate}}(\text{g})}{3\text{inch} \times 4\text{inch}} \right) * \left( \frac{\text{inch}}{2.54\text{cm}} \right)^2 * \left( \frac{100\text{cm}}{\text{m}} \right)^2$$

#### 2. Buffers

The compositions of the present invention may include a buffer to prevent the soil adsorbing polymer from interacting other ingredients in the composition. The buffer may be present in an amount of from about 0.01% to about 5.0%, alternatively about 0.01% to about 2.0%, alternatively about 0.01% to about 2.0%, alternatively about 0.01% to about 0.2%, alternatively about 0.1.

20

A suitable buffer herein is a weak acid, an organic and/or inorganic salt. In one embodiment, the organic salt is selected from monovalent, divalent, or trivalent salts, or mixtures thereof such as sodium citrate, sodium chloride, sodium phosphate, potassium chloride, potassium phosphate.

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#### 3. Surfactants

The compositions of the present invention may comprise a surfactant. The surfactant may be present at a level of greater than about 0.001% to about 10%, by weight of the composition, alternatively from about 0.5% to about 3, alternatively about 0.7% to about 3%, alternatively about 1% to about 3%, alternatively from about 1% to about 2%, alternatively greater than 1%. The exact level of surfactants in the compositions depends on a number of factors including

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surfactant type, class and chain-length, surfactant contribution to viscosity, and desired level of polymer in the composition.

Suitable surfactants are those selected from the group consisting of nonionic surfactants, cationic surfactants, zwitterionic surfactants, amphoteric surfactants, and mixtures thereof.

5 Examples of suitable surfactants are described in McCutcheon's Vol. 1: Emulsifiers and Detergents, North American Ed., McCutcheon Division, MC Publishing Co., 2002.

In one embodiment, the composition comprises non-ionic surfactants. Non-limiting examples of suitable nonionic surfactants include alcohol alkoxylates, alkyl polysaccharides, amine oxides, block copolymers of ethylene oxide and propylene oxide, castor oil derivatives,  
10 fluoro surfactants, and silicon based surfactants. Other non-ionic surfactants that can be used include those derived from natural sources such as sugars and include C<sub>8</sub>-C<sub>16</sub> N-alkyl glucose amide surfactants.

Also suitable for use in the present invention are the fluorinated nonionic surfactants. One particularly suitable fluorinated nonionic surfactant is Fluorad F170 (3M Corporation, 3M  
15 Center, St. Paul, MN, USA). Fluorad F170 has the formula C<sub>8</sub>F<sub>17</sub>SO<sub>2</sub>N(CH<sub>2</sub>-CH<sub>3</sub>)(CH<sub>2</sub>CH<sub>2</sub>O)<sub>x</sub>. Also suitable for use in the present invention are silicon-based surfactants. One example of these types of surfactants is Silwet L7604 available from Dow Chemical (1691 N. Swede Road, Midland, Michigan, USA).

a. Solubilizers

20 In some embodiments, the compositions of the present invention may include a solubilizing surfactant to solubilize any excess hydrophobic organic materials, particularly any perfume materials, and also optional ingredients (e.g., insect repelling agent, antioxidant, etc.) which can be added to the composition, that are not readily soluble in the composition, to form a clear solution. A suitable solubilizing surfactant, is a no-foaming or low-foaming surfactant. In  
25 one embodiment, the composition contains hydrogenated castor oil. One suitable hydrogenated castor oil that may be used in the present composition is Basophor™, available from BASF.

Compositions containing anionic surfactants and/or detergent surfactants may generate chalky residue. In some embodiments, the composition is free of anionic surfactants and/or detergent surfactants.

30 b. Wetting Agents

In some embodiments, the compositions of the present invention may include a wetting agent that provides a low surface tension permitting the composition to spread readily and more

uniformly. It has been found that the aqueous composition, without such a wetting agent may not spread satisfactorily. The spreading of the composition also allows it to dry faster when the composition contacts a surface.

Nonlimiting examples of wetting agents include block copolymers of ethylene oxide and propylene oxide. Suitable block polyoxyethylene-polyoxypropylene polymeric surfactants include those based on ethylene glycol, propylene glycol, glycerol, trimethylolpropane and ethylenediamine as the initial reactive hydrogen compound. Polymeric compounds made from a sequential ethoxylation and propoxylation of initial compounds with a single reactive hydrogen atom, such as C<sub>12-18</sub> aliphatic alcohols, are not generally compatible with the cyclodextrin.

Certain of the block polymer surfactant compounds designated Pluronic<sup>®</sup> and Tetronic<sup>®</sup> by the BASF-Wyandotte Corp., Wyandotte, Michigan, are readily available.

Nonlimiting examples of wetting agents of this type are described in US 5,714,137 and include the Silwet<sup>®</sup> surfactants available from Momentive Performance Chemical, Albany, New York. Exemplary Silwet surfactants are as follows:

| Name   | Average MW |
|--------|------------|
| L-7608 | 600;       |
| L-7607 | 1,000;     |
| L-77   | 600;       |
| L-7605 | 6,000;     |
| L-7604 | 4,000;     |
| L-7600 | 4,000;     |
| L-7657 | 5,000;     |
| L-7602 | 3,000;     |

and mixtures thereof.

#### 4. Perfume Ingredients

The compositions of the present invention may comprise a perfume mixture. The perfume mixture may comprise perfume ingredients in an amount from about 0.01% to about 10%, alternatively from about 0.01% to about 5%, alternatively from about 0.01% to about 3%, alternatively about 2.5%, by weight of the composition.

Perfume ingredients often have different volatilities and odor detection thresholds. In general, a perfume ingredient's character and volatility may be described in terms of its boiling point ("BP") and its octanol/water partition coefficient (or "P"). The boiling point referred to



herein is measured under normal standard pressure of 760 mmHg. The boiling points of many perfume ingredients, at standard 760 mm Hg are given in, e.g., "Perfume and Flavor Chemicals (Aroma Chemicals)," written and published by Steffen Arctander, 1969.

The octanol/water partition coefficient of a perfume ingredient is the ratio between its equilibrium concentrations in octanol and in water. The partition coefficients of the perfume ingredients used in the compositions of the present invention may be more conveniently given in the form of their logarithm to the base 10, logP. The logP values of many perfume ingredients have been reported; see for example, the Pomona92 database, available from Daylight Chemical Information Systems, Inc. (Daylight CIS), Irvine, California. However, the logP values are most conveniently calculated by the "CLOGP" program, also available from Daylight CIS. This program also lists experimental logP values when they are available in the Pomona92 database. The "calculated logP" (ClogP) is determined by the fragment approach of Hansch and Leo ( cf., A. Leo, in Comprehensive Medicinal Chemistry, Vol. 4, C. Hansch, P. G. Sammens, J. B. Taylor and C. A. Ramsden, Eds., p. 295, Pergamon Press, 1990). The fragment approach is based on the chemical structure of each perfume ingredient, and takes into account the numbers and types of atoms, the atom connectivity, and chemical bonding. The ClogP values, which are the most reliable and widely used estimates for this physicochemical property, are alternatively used instead of the experimental logP values in the selection of perfume ingredients for the composition.

The perfume mixture may comprise perfume ingredients selected from one or more groups of ingredients. A first group of ingredients comprises perfume ingredients that have a boiling point of about 250 °C or less and ClogP of about 3 or less. Alternatively, the first perfume ingredients have a boiling point of 240°C or less, alternatively 235 °C or less, alternatively the first perfume ingredients have a ClogP value of less than 3.0, alternatively 2.5 or less. One or more ingredients from the first group of perfume ingredients can be present in any suitable amount in the perfume mixture. In certain embodiments, the first perfume ingredient is present at a level of at least 1.0% by weight of the perfume mixture, alternatively at least 3.5 %, alternatively at least 7.0 %, by weight of the perfume mixture.

A second group of perfume ingredients comprise perfume ingredients that have a boiling point of 250 °C or less and ClogP of 3.0 or more, alternatively the second perfume ingredients have a boiling point of 240 °C or less, alternatively 235 °C or less, alternatively the second perfume ingredients have a ClogP value of greater than 3.0, alternatively greater than 3.2. One or

more ingredients from the second group of perfume ingredients can be present in any suitable amount in the perfume mixture. In certain embodiments, the second perfume ingredient is present at a level of at least 1.0% by weight of the perfume mixture, alternatively at least 3.5 %, alternatively at least 7.0 %, by weight of the perfume mixture.

5           A third group of perfume ingredients comprises perfume ingredients that have a boiling point of 250 °C or more and ClogP of 3.0 or less, alternatively the third perfume ingredients have boiling point of 255°C or more, alternatively 260 °C or more. Alternatively, this additional perfume ingredient has a ClogP value of less than 3.0, alternatively 2.5 or less. One or more ingredients from the third group of perfume ingredients can be present in any suitable amount in  
10 the perfume mixture. In certain embodiments, the third perfume ingredient is present at a level of at least 10% by weight of the perfume mixture, alternatively at least 25%, alternatively greater than 40 %, alternatively greater than 50%, by weight of the perfume mixture.

          A fourth group of perfume ingredients comprises perfume ingredients that have a boiling point of 250 °C or more and ClogP of 3.0 or more, alternatively this additional perfume  
15 ingredient has boiling point of 255 °C or more, alternatively 260 °C or more, alternatively, the additional perfume ingredient has a ClogP value of greater than 3.0, even more alternatively greater than 3.2. One or more ingredients from the fourth group of perfume ingredients can be present in any suitable amount in the perfume mixture. In certain embodiments, the fourth perfume ingredient is present at a level of at least 10% by weight of the perfume mixture,  
20 alternatively at least 25%, alternatively greater than 40 %, alternatively greater than 50%, by weight of the perfume mixture.

          The perfume mixture may also comprise any suitable combination of perfume groups described above. For example, the perfume mixture may comprise at least 50% of perfume ingredients from groups 3 and 4, and the balance of the perfume mixture is from the first and/or  
25 second group of perfume ingredients.

          The perfume mixtures useful in the composition may include levels of perfume ingredients to achieve the odor detection threshold (ODT) while staying within odor detection range (ODR). The ODT is the minimum concentration of perfume ingredient which is consistently perceived to generate an olfactory response in an individual. As the concentration of  
30 perfume is increased, so does the odor intensity of the perfume and the olfactory response of the individual. This occurs until the concentration of the perfume reaches a maximum, at which point the odor intensity reaches a plateau beyond which there is no additional olfactory response

by the individual. This range of perfume concentration through which the individual consistently perceives an odor is known as the ODR.

In some circumstances, it may be desirable to exceed the ODR of at least some of the perfume ingredients. In one embodiment, at least one perfume ingredient is present at a level of 5 50% in excess of the ODR, alternatively 150% in excess of the ODR. For lingering scents, at least one perfume ingredient can be added at a level of more than 300% of the ODR.

ODTs are determined using a commercial gas chromatograph ("GC") equipped with flame ionization and a sniff-port. The gas chromatograph is calibrated to determine the exact volume of material injected by the syringe, the precise split ratio, and the hydrocarbon response 10 using a hydrocarbon standard of known concentration and chain-length distribution. The air flow rate is accurately measured and, assuming the duration of a human inhalation to last 12 seconds, the sampled volume is calculated. Since the precise concentration at the detector at any point in time is known, the mass per volume inhaled is known and concentration of the material can be calculated. To determine whether a material has a threshold below 50 parts per 15 billion (ppb), solutions are delivered to the sniff port at the back-calculated concentration. A panelist sniffs the GC effluent and identifies the retention time when odor is noticed. The average across all panelists determines the threshold of noticeability.

The necessary amount of analyte is injected onto the column to achieve a 50 ppb concentration at the detector. Typical GC parameters for determining ODTs are listed below. 20 The test is conducted according to the guidelines associated with the equipment.

Equipment:

GC: 5890 Series with FID detector (Agilent Technologies, Ind., Palo Alto, California, USA)

7673 Autosampler (Agilent Technologies, Ind., Palo Alto, California, USA)

25 Column: DB-1 (Agilent Technologies, Ind., Palo Alto, California, USA)

Length 30 meters ID 0.25 mm film thickness 1 micron (a polymer layer on the inner wall of the capillary tubing, which provide selective partitioning for separations to occur)

Method Parameters:

30 Split Injection: 17/1 split ratio

Autosampler: 1.13 microliters per injection

Column Flow: 1.10 mL/minute

Air Flow: 345 mL/minute

Inlet Temp. 245°C

Detector Temp. 285°C

Temperature Information

5 Initial Temperature: 50°C

Rate: 5C/minute

Final Temperature: 280°C

Final Time: 6 minutes

Leading assumptions: (i) 12 seconds per sniff

10 (ii) GC air adds to sample dilution

In certain embodiments, the composition may be dispensed from a dispenser providing larger droplets of composition (which have a smaller total surface area compared to a plurality of small droplets). This may reduce the speed with which the highly volatile top notes will volatilize. The droplets may not only release the perfume mixture when they are suspended in  
15 the air, they may also fall until they contact a surface (e.g., tables or countertops, furniture, and floors, carpets, etc.). The droplets that fall onto these surfaces can serve as reservoirs for the perfume mixture, releasing the perfume mixture after landing on such surfaces. In this manner, there can be a continual renewal of the scent originally perceived by the consumer, which is replenished by molecules released from the droplets over a period of time.

20 5. Malodor Counteractant

The compositions of the present invention may also comprise a malodor counteractant to deliver a genuine malodor removal benefit. The compositions may neutralize malodors via vapor phase technology, which is defined as malodor counteractants that mitigate malodors in the air via chemical reactions or neutralization. In such embodiments, the malodor counteractant may  
25 comprise one or more fabric-safe aliphatic aldehydes and/or one or more enones (ketones with unsaturated double bonds).

The following table illustrates the importance of proper selection of aldehydes and enones to avoid fabric yellowing.

| Aldehyde Solution Tested                        | Fadometer Test on treated Fabric<br>(0.75 grams of product are pipetted onto<br>a 4 inch X 4 inch (10 cm X 10 cm)<br>swatch which is then subjected to 5 hours<br>of exposure to simulated sunlight using a<br>SUNTEST CPS+ model Fadometer<br>supplied by Atlas, Chicago, Illinois,<br>USA. |
|---|--|
| Control- untreated fabric swatch                | No yellowing   |
| 1000 ppm amylic cinnamic aldehyde<br>(aromatic) | Yellowish brown  |
| 1000 ppm citronellal (aromatic)                 | Yellowish brown  |
| 1000ppm citral aldehyde (aliphatic)             | No yellowing   |
| 1000 ppm lauric aldehyde (aliphatic)            | No yellowing   |

Examples of suitable aliphatic aldehydes are R-COH where R is saturated C<sub>7</sub> to C<sub>22</sub> linear and/or branched with no more than two double bonds. Additional examples of aliphatic aldehydes are lylal, methyl dihydro jasmonate, ligustral, melonal, octyl aldehyde, citral, cymal, 5 nonyl aldehyde, bourgeonal, P. T. Bucinal, Decyl aldehydes, lauric aldehyde, and mixtures thereof.

The malodor counteractants that utilize vapor phase technology can be present in any suitable amount in a perfume mixture. In certain embodiments, the malodor counteractants may be present in an amount greater than or equal to about 1% and less than about 50% by weight of 10 the perfume mixture. In other embodiments, the malodor counteractants may be present in an amount greater than or equal to about 3% and less than about 30% by weight of the perfume mixture. In other embodiments, the malodor counteractants may be present in an amount greater than or equal to about 8% and less than about 15% by weight of the perfume mixture.

Malodor counteractants may also comprises cyclodextrins to neutralize the malodor when 15 the composition is a mist suspended in the air. Cyclodextrin forms complexes with different organic molecules to make them less volatile. In some embodiments, the compositions of the present invention may include solubilized, water-soluble, uncomplexed cyclodextrin. Cyclodextrin molecules are described in US 5,714,137, and US 5,942,217. Suitable levels of cyclodextrin are from about 0.01% to about 3%, alternatively from about 0.01% to about 2%,

alternatively from about 0.05% to about 1%, alternatively from about 0.05% to about 0.5%, by weight of the composition.

Some types of malodor counteractants function by sensory modification of those exposed to odors. There are at least two ways of modifying the sensory perception of odors. One way is to mask odors using perfume so that a person exposed to the odor smells the perfume more than the odor. The other way is to reduce the person's sensitivity to malodors. Ionones are compositions that are capable of reducing the sensitivity of a person's olfactory system to the presence of certain undesirable odors, such as sulfur odors caused by eggs, onions, garlic, and the like. Examples of suitable ionones are ionone alpha, ionone beta, ionone gamma methyl, and mixtures thereof.

#### 6. Propellant

The compositions of the present invention may comprise a propellant for assisting with spraying the composition into the air. The compositions may comprise propellants that are primarily non-hydrocarbon propellants (that is, propellants that are comprised of more non-hydrocarbon propellants by volume than hydrocarbon propellants, that is, greater than or equal to about 50% of the volume of the propellant). In some embodiments, the propellant may be substantially free of hydrocarbons such as: isobutene, butane, isopropane, and dimethyl ether. In other embodiments, the propellant may be a hydrocarbon. In embodiments in which the composition uses a non-hydrocarbon propellant, such a propellant may include a compressed gas. Some compressed gases can be more environmentally-friendly than hydrocarbon propellants, which may make them more suitable for dust reducing compositions that also freshen the air. Suitable compressed gases include, but are not limited to compressed air, nitrogen, nitrous oxide, inert gases, carbon dioxide, etc., and mixtures thereof.

Suitable amounts of propellant in the composition are from about 20% to about 80%, alternatively about 30% to about 60%, alternatively about 30% to about 50%, by weight of the composition.

#### Spray Dispenser

The compositions of the present invention can be packaged in any suitable spray dispenser known in the art. One suitable dispenser is a plastic aerosol sprayer. The dispenser may be constructed of polyethylene such as a high density polyethylene; polypropylene; polyethyleneterephthalate ("PET"); vinyl acetate, rubber elastomer, and combinations thereof. In one embodiment, the spray dispenser is made of clear PET.

The spray dispenser may hold about 1 to about 300 grams of composition, alternatively about 275 grams, alternatively about 250 grams, alternatively about 150 grams of composition.

The spray dispenser may be capable of withstanding internal pressure in the range of about 50 p.s.i.g. to about 140 psig, alternatively about 80 to about 130 p.s.i.g..

5        Although compressed gas systems produce relatively larger particles than hydrocarbon systems and may provide superior particulate reduction and more desirable perfume release profile, these same particles can create wetness on the floor and other surfaces because they are heavier and fall to the ground. In one embodiment of the present invention, the total composition output and the spray droplet/particle size distribution are selected to support the particulate  
10        removal efficacy but avoid a surface wetness problem. Total output is determined by the flow rate of the composition as it is released from the spray dispenser. To achieve a spray profile that produces minimal surface wetness, it is desirable to have a low flow rate and small spray droplets. The flow rate may be less than 1.2 grams/second and the droplets will be small enough that when, dispensed at a height of 5 feet from the ground, less than 40% of the droplets fall to  
15        the ground.

A low flow rate can be achieved via the valve, the delivery tube and/or the nozzle but nozzle modifications have proven to be less susceptible to instances of clogging. Flow rate is determined by measuring the rate of composition expelled by a full container for the first 60 seconds of use. In one embodiment, the average flow rate of the composition being released  
20        from the spray dispenser is from about 0.0001 grams/second to about 2.0 grams/second. Alternatively, the average flow rate is from about 0.001 grams/second to about 1.5 grams/second, alternatively about 0.01 grams/second to about 1.5 grams/second, alternatively about 0.01 grams/second to about 1.3 grams/second, alternatively about 0.5 grams/second to about 1.3 grams/second, alternatively about 0.7 grams/second to about 1.3 grams/second. In an alternate  
25        embodiment, the average flow rate is from about 0.8 grams/second to about 1.3 grams/second.

The mean particle size of the spray droplets may be in the range of from about 10  $\mu\text{m}$  to about 100  $\mu\text{m}$ , alternatively from about 20  $\mu\text{m}$  to about 60  $\mu\text{m}$ . In one version of such an embodiment, at least some of the spray droplets are sufficiently small in size to be suspended in the air for at least about 10 minutes, and in some cases, for at least about 15 minutes, or at least  
30        about 30 minutes.

In one embodiment, the aerosol dispenser may be configured to spray the composition at an angle that is between an angle that is parallel to the base of the container and an angle that is

perpendicular thereto. In other embodiments, the desired size of spray droplets can be delivered by other types of devices that are capable of being set to provide a narrow range of droplet size. Such other devices include, but are not limited to: foggers, ultrasonic nebulizers, electrostatic sprayers, and spinning disk sprayers.

- 5           The compositions of the present invention can be made in any suitable manner. All of the ingredients can simply be mixed together. In certain embodiments, the acidic ingredients are combined with the solvent prior adding the soil adsorbing polymer. In another embodiment, it may be desirable to use the mixture of ingredients as a concentrated product (and to dispense such a concentrated product, such as by spraying). In other embodiments, the mixture of
- 10 ingredients can be diluted by adding the same to some suitable carrier and that composition can be dispensed in a similar manner.

## EXAMPLES

### Exemplary Formulas

- Table 4 includes non-limiting examples of particulate reducing compositions according to
- 15 the present invention.

Table 4

|                                 | I       | II      | III     | IV      | V       | VI      |
|---------------------------------|---------|---------|---------|---------|---------|---------|
| Ingredients                     | Wt.%    | Wt.%    | Wt.%    | Wt.%    | Wt.%    | Wt.%    |
| Hydroxypropyl beta-cyclodextrin | 0.2     | --      |         | --      | 0.3     | 0.1     |
| Soil Adsorbing Polymer          | 1.0     | 0.1     | 0.1     | -       | 0.1     | 0.05    |
| Diethylene glycol               | 0.25    | -       | -       | -       | -       | -       |
| Silwet L-7600                   | 0.1     | 0.2     | -       | 0.2     | 0.1     | 0.1     |
| Sodium Dioctyl Sulfosuccinate   | 0.2     | -       | 0.2     | 0.1     | 0.2     | 0.2     |
| Acid Salt                       | 0.1     | 0.1     | -       | 0.2     | 0.1     | -       |
| Ethanol                         | 3       | 5       | 5       | 3       | 5       | 5       |
| Hydrogenated castor oil         | 0.4     | 0.8     | 1.2     | 1.6     | 1.8     | 5       |
| Perfume Mixture                 | 0.6     | 0.8     | 0.4     | 0.2     | 1       | 0.1     |
| Organic Acid                    | 0.05    | 0.1     | -       | 0.1     | 0.05    | -       |
| Preservative                    | 0.015   | 0.015   | 0.015   | 0.015   | 0.015   | 0.015   |
| HCl or NaOH                     | to pH 5 | to pH 5 | to pH 5 | to pH 5 | to pH 7 | to pH 8 |
| Distilled water                 | Balance | Balance | Balance | Balance | Balance | Balance |



Dust Particle Reduction Test

To reduce particulates in air, in one embodiment, the time in which the composition contacts a particulate is less than about 30 seconds. To determine the profile of floating dust particles when treated with compositions according to the present invention, one may utilize the following test design which consists of:

- an enclosed environmental chamber 12.2 cubic feet in volume (39.25"W x 25."D x 21.5"H) equipped with a 4 inch 110 cfm fan;
- two additional fans are introduced for increased airflow that are 11.9 cm x 11.9 cm x 3.8 cm and 90 cfm;
- a sample probe placed inside the chamber connected by tubing with reduced electrostatics and particle adhesion;
- a Solair™ 3100laser particle counter is used;
- dust particles of known composition and particle size distribution;

All available channels should be selected on the particle counter for testing. Timing controls should be adjusted as necessary within the limits of the particle counter. Introduce a known amount of dust particles into the environmental chamber over time, as needed, for depletion of testing amount required. Continue sampling until desired equilibrium is reached. If treatment with aerosol is required, spray product into chamber and continue sampling until relevant time achieved.

It should be understood that every maximum numerical limitation given throughout this specification will include every lower numerical limitation, as if such lower numerical limitations were expressly written herein. Every minimum numerical limitation given throughout this specification will include every higher numerical limitation, as if such higher numerical limitations were expressly written herein. Every numerical range given throughout this specification will include every narrower numerical range that falls within such broader numerical range, as if such narrower numerical ranges were all expressly written herein.

The dimensions and values disclosed herein are not to be understood as being strictly limited to the exact numerical values recited. Instead, unless otherwise specified, each such dimension is intended to mean both the recited value and a functionally equivalent range surrounding that value. For example, a dimension disclosed as "40 mm" is intended to mean "about 40 mm."

Every document cited herein, including any cross referenced or related patent or application, is hereby incorporated herein by reference in its entirety unless expressly excluded or otherwise limited. The citation of any document is not an admission that it is prior art with respect to any invention disclosed or claimed herein or that it alone, or in any combination with  
5 any other reference or references, teaches, suggests or discloses any such invention. Further, to the extent that any meaning or definition of a term in this document conflicts with any meaning or definition of the same term in a document incorporated by reference, the meaning or definition assigned to that term in this document shall govern.

While particular embodiments of the present invention have been illustrated and  
10 described, it would be obvious to those skilled in the art that various other changes and modifications can be made without departing from the spirit and scope of the invention. It is therefore intended to cover in the appended claims all such changes and modifications that are within the scope of this invention.

## CLAIMS

What is claimed is:

1. A composition for reducing particulates in the air comprising a soil adsorbing polymer comprising two or more monomeric units selected from the group consisting of:

- a) nonionic monomeric units;
- b) anionic monomeric units;
- c) cationic monomeric units;
- d) zwitterionic monomeric units; and
- e) mixtures thereof;

wherein said polymer exhibits a Soil Adsorption Value of at least 38 mg as measured according to the Soil Adsorption Test Method described herein.

2. The composition of Claim 1 wherein said polymer is present in an amount from 0.001% to 1%, by weight of said composition.

3. The composition according to Claim 1 wherein said polymer exhibits a number average molecular weight of less than 2,000,000 g/mol.

4. The composition according to Claim 1 wherein said polymer exhibits a Soil Adsorption Value of at least 40 mg as measured according to the Soil Adsorption Value Test Method described herein.

5. The composition according to Claim 1 wherein said nonionic monomeric units comprise a nonionic hydrophilic monomeric unit selected from the group consisting of: hydroxyalkyl esters of  $\alpha,\beta$ -ethylenically unsaturated acids,  $\alpha,\beta$ -ethylenically unsaturated amides,  $\alpha,\beta$ -ethylenically unsaturated monoalkyl amides,  $\alpha,\beta$ -ethylenically unsaturated dialkyl amides,  $\alpha,\beta$ -ethylenically unsaturated monomers bearing a water-soluble polyoxyalkylene segment of the poly(ethylene oxide) type,  $\alpha,\beta$ -ethylenically unsaturated monomers which are precursors of hydrophilic units or segments, vinylpyrrolidones,  $\alpha,\beta$ -ethylenically unsaturated monomers of the ureido type, vinylaromatic monomers, vinyl halides, vinylidene halides, C<sub>1</sub>-C<sub>12</sub> alkylesters of  $\alpha,\beta$ -monoethylenically unsaturated acids, vinyl esters of saturated carboxylic acids, allyl esters of saturated carboxylic acids,  $\alpha,\beta$ -monoethylenically unsaturated nitriles containing from 3 to 12 carbon atoms,  $\alpha$ -olefins, conjugated dienes, and mixtures thereof.

6. The composition according to Claim 1 wherein said anionic monomeric units are derived from anionic monomers selected from the group consisting of: monomers having at least one carboxylic function, monomers that are precursors of carboxylate functions, monomers having at least one sulfate or sulfonate function, monomers having at least one phosphonate or phosphate function, esters of ethylenically unsaturated phosphates, and mixtures thereof.
7. The composition according to Claim 1 wherein said cationic monomeric units are derived from cationic monomers selected from the group consisting of: acryloyl- or acryloyloxyammonium monomers, 1-ethyl-2-vinylpyridinium or 1-ethyl-4-vinylpyridinium bromide, chloride or methyl sulfate, N,N-dialkyldiallylamine monomers, polyquaternary monomers, N,N-(dialkylamino- $\omega$ -alkyl)amides of  $\alpha,\beta$ -monoethylenically unsaturated carboxylic acids,  $\alpha,\beta$ -monoethylenically unsaturated amino esters, vinylpyridines, vinylamine, vinylimidazolines, monomers that are precursors of amine functions which give rise to primary amine functions by simple acid or base hydrolysis, and mixtures thereof.
8. The composition according to Claim 1 wherein said polymer comprises a monomeric unit selected from the group consisting of: at least 69.9% wt of a monomeric unit from group a; at least 0.1% wt of a monomeric unit from group b; at least 0.3% wt of a monomeric unit from group c; and at least 0.5% wt of a monomeric unit from group d.
9. The composition according to Claim 1 wherein said polymer comprises a monomeric unit from group a, a monomeric unit from group b, and a monomeric unit from group d.
10. The composition according to Claim 1 wherein said polymer exhibits a charge density of from -0.1 to +0.1 meq/g.
11. The composition according to Claim 1 wherein said polymer exhibits a Polydispersity Index of less than 2.5.
12. The composition of Claim 1 further comprising a perfume ingredient.
13. The composition of Claim 1 further comprising an aqueous carrier and a surfactant, said surfactant is selected from the group consisting of: nonionic surfactants, zwitterionic surfactants, amphoteric surfactants, and mixtures thereof.

14. The composition of Claim 13 wherein said surfactant is present in an amount from 1% to 3%, by weight of said composition.
15. The composition of Claim 1 further comprising a compressed gas propellant selected from the group consisting of compressed air, nitrogen, nitrous oxide, inert gases, carbon dioxide, and mixtures thereof.
16. The composition of Claim 1 further comprising a buffer.
17. The composition of Claim 1 wherein said composition comprises a viscosity of 0.1 to 8 cps.
18. The composition of Claim 1 further comprising a malodor counteractant selected from the group consisting of: cyclodextrin, carboxylic acids including mono, di, tri, and polyacrylic acids, and mixtures thereof.
19. The composition of Claim 1, wherein said composition is provided in a plastic container.
20. A composition for reducing particulates in the air comprising:
  - a soil adsorbing polymer comprising two or more monomeric units selected from the group consisting of:
    - a) nonionic monomeric units;
    - b) anionic monomeric units;
    - c) cationic monomeric units;
    - d) zwitterionic monomeric units; and
    - e) mixtures thereof;
  - wherein said polymer exhibits a Soil Adsorption Value of at least 38 mg as measured according to the Soil Adsorption Test Method described herein;
  - a surfactant selected from the group consisting of: nonionic surfactants, zwitterionic surfactants, amphoteric surfactants, and mixtures thereof;
  - a compressed gas propellant; and
  - an aqueous carrier.