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(54) NON-AQUEOUS LAPPING COMPOSITION AND METHOD USING SAME

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(57) ABSTRACT

Lapping compositions which do not comprise water are disclosed, wherein those lapping compositions comprise a non-aqueous fluid, and wherein the lapping compositions are useful during a process to shape the surface of a substrate, wherein that process includes contacting a target surface of the substrate with one or more abrasives while also contacting that target surface with the lapping composition.

NON-AQUEOUS LAPPING COMPOSITION AND METHOD USING SAME

CROSS REFERENCE TO RELATED APPLICATIONS

[0001] This Application claims priority from a United States Provisional Application having Ser. No. 60/694,027 filed Jun. 23, 2005.

FIELD OF THE INVENTION

[0002] The invention relates to a composition which does not comprise water, wherein that composition is useful in grinding, cutting, and/or polishing the surface of ceramic and/or metallic objects.

BACKGROUND OF THE INVENTION

[0003] Lapping, grinding, cutting, and polishing, collectively referred to herein as "lapping," are key manufacturing technologies for shaping various ceramic and metallic materials. Lapping provides a mechanism to shape the surface of a substrate.

[0004] Lapping is often performed using a versatile, high-precision polishing machine using a scrolled cast iron plate. The machine generally includes a reciprocating roller bar mechanism to hold the sample in position while allowing for constant plate conditioning during the preparation process. In some applications, the lapping comprises a medium grit (10 to 15 micron) silicon carbide powder, suspended in a lapping composition. Other abrasive materials include medium grit aluminum oxide, boron carbide, and the like. By "lapping composition," Applicant means a fluid used during a process to shape the surface ota substrate, where that process includes contacting a target surface of the substrate with one or more abrasives while also contacting that target surface with the lapping composition.

[0005] The choice of abrasive depends on the type of material-a very aggressive abrasive, such as diamond will cause a deeper damage layer at the surface. Damage penetration can be reduced by decreasing the load on the sample and the plate speed as the final thickness is approached.

[0006] After lapping, the sample can be polished using chemo-mechanical suspensions of, for example, colloidal silica (0.125 micron) or aluminum oxide (0.3 micron). The slurry suspensions comprising one or inore abrasives suspended in Applicant's lapping composition are pumped continuously over the plate.

[0007] As a general matter, the lapping process using Applicant's lapping composition comprises the steps of providing a substrate, where that substrate comprises a target surface, where that target surface may comprise a metal surface, a ceramic surface, a polymeric material, such as for example polycarbonate, or combinations thereof; and the like; providing one or more abrasives; providing Applicant's nonaqueous lapping composition; mounting the substrate in a lapping machine comprising a moveable lapping/polishing surface. wherein in certain embodiments the one or more abrasives may be disposed on the lapping/polishing surface; providing Applicants' lapping composition, contacting the target surface with Applicant's lapping compositions where in certain embodiments the one or more abrasives may be disposed in Applicant's lapping composition; and moving the lapping/polishing surface against the target surface.

[0008] A number of factors influence the quality of surfaces shaped by these methods including the size and composition of the particular abrasive grit employed as well as the amount of pressure applied to the surface. Fluids are often also cascaded upon the part during these operations. Among other functions, such fluids remove heat during machining. In addition, such fluids lubricate the abrasive/surface contact area. Such fluids also remove swarf from the part. As those skilled in the art will appreciate, swarf comprises metallic/ceramic filings or shavings removed by a cutting tool.

[0009] Rapid swarf removal is desirable in order to efficiently shape the part as well as reproducibly produce smooth, high quality surfaces. Hence it is desirable that the machining fluid exhibits a significant affinity for the surface of the material being shaped.

[0010] In certain embodiments, such an affinity includes chemisorption upon the ceramic or metallic material; such that its swarf is wetted, dispersed within, and ultimately washed away by the cascading machining fluid.

[0011] T he invention will be described herein as embodied in a lapping composition useful in the manufacture of read/write heads for computer hard disks. The following description of Applicant's composition, and uses thereof, is not meant, however, to limit Applicant's invention to the manufacture of such hard disks, or to the manufacture of data storage devices in general, as the invention herein can be applied to surface preparation of metals, ceramics, and the like. In addition, Applicant's compositions are useful in a myriad of applications, including without limitation polishing and/or chemical mechanical polishing of various substrates, manufacture of semiconductor devices, and the like.

[0012] As one example, the manufacture of read/write heads for computer hard disks, and the manufacture of the hard disks, includes one or more lapping processes. As those skilled in the art will appreciate, such computer hard disks comprise rotatable disks comprising one or more magnetic materials. Information is written to, and read from, such magnetic disks using a read/write head.

[0013] As those skilled in the art will appreciate, computer hard disks are rotatably disposed in a disk drive unit such that the magnetic disk rotates rapidly as information is written thereto and/or read therefrom. In order to prevent the read/write head from damaging the disk surface when that disk is rapidly rotating, it is critical that the read/write head be planarized so that it exhibits minimal surface roughness.

[0014] There is an on-going need to maximize the storage density in such computer hard disks. In order to increase that storage density, the fly height between the rotating disk and the read write head is continually decreasing. As a result, increased demands are being placed upon surface quality of both the read—write head and the hard disk.

[0015] One such read/write head is often referred to as a Giant Magnetoresistive (GMR) Head. Such GMR Heads comprise a metallized ceramic material. GMR Heads typically comprise TiC—Alumina ceramic, sometimes called "AlTiC," that has been metallized with a variety of ferrous and non-ferrous alloy layers. Prior art GMR Head lapping methods utilize fluids comprising a diamond abrasive dispersed in a mixture of water, various alcohols, and other additives. In order to prevent corrosion of its GMR Head metal layers which would adversely effect the Head's electromagnetic properties and operational performance within the computer hard drive.

[0016] These prior art GMR Head lapping methods are inefficient, exhibiting low AlTiC and metallized layer material removal rates (MRR). A low MRR is undesirable from a manufacturing standpoint because the MRR often comprises a rate-limiting step in the production of read/write heads. In addition, such prior art lapping fluids are highly alkaline, i.e. pH>0. Moreover, these prior art lapping compositions are not biodegradable. The alkalinity in combination with the non-biodegradability of these prior art compositions results in significant handling and disposal costs and concerns.

[0017] In contrast, Applicant's lapping composition is derived from natural products and are biodegradable. In addition, Applicant's lapping composition poses minimal environmental concerns. Moreover, Applicant's lapping method using Applicant's lapping fluids has a high MRR rate. Applicant's lapping composition includes additives comprising a combination of hydroxyl and carboxylic acid/carboxylate functionalities.

SUMMARY OF THE INVENTION

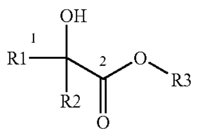
[0018] Applicant's invention includes a composition which does not comprise water, wherein that composition is useful for removing swarf from the surface of a ceramic or metallic object while that surface is being polished. In certain embodiments, Applicant's composition is formed by mixing in a non-aqueous fluid an acid selected from the group consisting of citric acid, glycolic acid, tartaric acid, and gallic acid, methyl gallate, ethyl gallate, propyl gallate, lauryl gallate, Guerbet gallate esters, mono, di, or trialkyl esters if citric acid, and a base selected from the group consisting of monoethanolamine, triethanolamine, diglycolamine, guanidine, choline, tetraalkylammonium hydroxide, 2-ethyl-1-hexyl amine, bis(2-ethyl-1-hexyl) amine, tris (2-ethyl-1hexyl) amine, fatty primary, secondary, or tertiary amines and potassium hydroxide.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

[0019] In certain embodiments, Applicant's lapping composition comprises one or more salts of hydroxy carboxylic acids disposed in a nonaqueous fluid. Applicant's nonaqueous fluid comprises one or more hydrocarbons, petroleum distillates, mineral oils, vegetable oils, esters, alcohols, amines, propylene glycol, ethylene glycol, one or more dipolar aprotic solvents, and mixtures thereof.

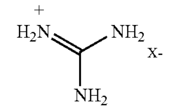
[0020] In certain embodiments, Applicant's lapping composition comprise salts of one or more alpha hydroxy acids (AHC). AHC comprise compounds having structure I, which comprises one or more hydroxyl groups attached to a first carbon atom, where that first carbon atom is covalently bonded to a second carbon atom comprising a carboxyl group.

I

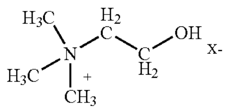


[0021] In certain embodiments, Applicant's lapping composition includes one or more of amine and/or alkali neutralized salts of citric, glycolic, tartaric acid, and/or gallic acid. In the citric acid embodiments, R1 is CH_2—CO_2—R4, and R2 is CH_2 —CO_2—R5. In the glycolic acid embodiments, R1 is methyl and R2 is hydrogen. In the tartaric acid embodiments, R1 is hydrogen and R2 is $C(H)(OH)CO_2$—R6. In these embodiments, R3, R4, R5, and R6, are each independently selected from the group consisting of hydrogen, an ammonium salt, such as tetramethylammonium hydroxide, an alkali metal ion, and alkaline earth metal ion, a guanidinium cation IV, a choline cation V, and the like.

IV



V



[0022] In certain embodiments, Applicant has discovered that lapping compositions comprising citric acid show enhanced lapping MRR performance. Table I, below, summarizes MRR using citric acid based lapping compositions. Lapping was conducted using a Crane Lapmaster Model 15 lapping machine comprising a 15 inch diameter diamond lapping film adhered to the lapping platen.

TABLE 1

MRR Lapping Performance of Various Citrate Salts

Formulation	Component	pH	Avg. MRR (g/min.)	MRR Relative to CONTROL
A	DGA Citrate Tribasic	8.94	0.00121	303%
B	DGA Citrate Dibasic	5.06	0.00138	345%
C	MEA Citrate Tribasic	8.5	0.00135	338%
D	MEA Citrate Dibasic	5.23		
E	MEA Citrate Tribasic/Na Polyaspartate Blend	8.8	0.00122	305%
F	Na Polyaspartate	8.1	0.00095	238%
G	Na Citrate Tribasic	8.2	0.00142	355%
H	TEA Citrate Tribasic	6.8	0.00101	253%
I	TEA Citrate Tribasic/Na Polyaspartate Blend	5.05	0.00127	318%
J	TMAH Citrate Dibasic	5.19	0.00137	343%
K	MEA Citrate Tribasic Poly Sodium Aspartate -co-diglycol Aspartamide	8.8	0.00147	368%

TABLE 1-continued

MRR Lapping Performance of Various Citrate Salts

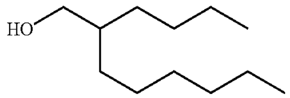
Formulation	Component	pH	Avg. MRR (g/min.)	MRR Relative to CONTROL
CONTROL	Control Conventional GMR Head Lapping Fluid	10	0.00040	—

"DGA" comprises Diglycolamine. "MEA" comprises monoethanolamine. "TEA" comprises triethanolamine. "TMAH" comprises Tetramethylammonium Hydroxide. Na Polyaspartate was Baypure DS 00 product supplied by Bayer Corp. (Pittsburgh, Pa.) having approximately 1500 Dalton molecular weight

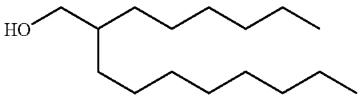
[0023] In certain embodiments, Applicant's non-aqueous lapping composition comprises a citrate molecule disposed in a mixture of a lipophilic alcohol and mineral oil. In certain embodiments, the lipophilic alcohol comprises a Guerbet alcohol. In certain embodiments, Applicant's non-aqueous lapping composition comprises a gallate Guerbet ester, formed for example and without limitation, by reacting gallic acid with a Guerbet alcohol.

[0024] Guerbet alcohols are the oldest and best-understood material in the class of compounds, first synthesized by Marcel Guerbet. The reaction sequence, which bears his name, is related to the Aldol Reaction and occurs at high temperatures under catalytic conditions. The product is an alcohol with twice the molecular weight of the starting alcohol minus a mole of water.

[0025] In certain embodiments, Applicant's non-aqueous lapping composition comprises 2-butyl-1-octanol and/or 2-hexyl-1-decanol. Alcohols 2-butyl-1-octanol and 2-hexyl-1-decanol were diluted with various amounts of mineral oil. The mass ratios of these solutions varied from 1:2 to 1:10 alcohol to mineral oil. These components were miscible without heating.



2-butyl 1-octanol
(Jarcol I-12)



2-hexyl-1-decanol
(Jarcol I-16)

TABLE 2

Sample #	2-Butyl-1-octanol (g)	Eq. of Mineral Oil	Mineral Oil(g)
1	0.41	2	0.82
2	0.34	2.5	0.85
3	0.25	3	0.75
4	0.27	3.5	0.95
5	0.30	4	1.2
6	0.24	5	1.2
7	0.22	6	1.32
8	0.21	7	1.47
9	0.21	8.5	1.79
10	0.39	10	3.9

[0026]

TABLE 3

Sample #	2-Hexyl-1-decanol (g)	Eq. of Mineral Oil	Mineral Oil (g)
1	0.47	2	0.94
2	0.38	2.5	0.95
3	0.37	3	1.11
4	0.36	3.5	1.26
5	0.35	4	1.4
6	0.27	5	1.35
7	0.22	6	1.32
8	0.24	7	1.68
9	0.23	8	1.84
10	0.39	10	3.9

[0027] These solutions were then diluted with approximately 5 mL of iso-propyl alcohol ("IPA"). These solutions were shaken and allowed to sit overnight. Visual inspection reveals that all of the 2-Hexyl-1-decanol/Mineral Oil solutions were miscible in IPA. The 2-Buty-1octanol/Mineral Oil solutions have an upper limit of 1:8. hereafter, Application's lapping composition was formed by adding trioctyl dodecyl citrate.

[0028] In certain embodiments, Applicant's non-aqueous lapping fluid comprises a gallate molecule disposed in a non-aqueous fluid. Table 4 provides a list of such gallate-based non-aqueous lapping compositions.

TABLE 4

Gallate and Amine Miscibility Experiments in Lipophilic Alcohols

Base	M. wt.	Amount	2-Butyl-1-octanol	2-Hexyl-1-decanol
Lauryl gallate = 250 mg = 0.74 mmol*				
TEA	101.2	0.07	soluble	soluble
DBU	152.24	0.11	soluble	soluble

[0029] Lauryl (C12) gallate, in the presence of triethyl amine, was miscible in both 2-Butyl-1-octanol and 2-Hexyl-1-decanol. Lauryl gallate, in the presence of triethanol amine and DBU, was miscible in 2-Butyl-1-octanol, but only somewhat soluble in 2-Hexyl-1-decanol.

[0030] Poly Sodium Aspartate-co-diglycol Aspartamide was prepared via aminolysis ring opening reaction of succinimide repeat units within 3000 Dalton Polysuccinimide (PSI) (Bayer Polymers Pittsburgh, Pa.) dissolved in DMSO solvent. Compositions having <30 wt. % 3000 Dalton MW PSI solute are desirable due to apparent PSI solubility and viscosity limitations. Diglycolamine was added dropwise to the PSI/DMSO composition at room temperature accompanied by rapid stirring to produce a dark read colored mixture. Suitable lapping fluid aspartamide copolymers should be prepared via reaction of between 0 and 75 mol. % of PSI succinimide repeat units with diglycolamine. The remaining, unreacted SI repeat units in the aspartamide copolymer are hydrolyzed via reaction with NaOH in 1:1 molar stoichiometry producing the desired Poly Sodium Aspartate-co-Diglycol Aspartamide copolymer.

[0031] Formulations A through K, inclusive, and the Control, had identical citrate Molality within a non-aqueous fluid, namely 0.214 Moles Citrate/Kg fluid. Formulations A through K, inclusive, were prepared via mixing of anhydrous citric acid in a non- aqueous fluid followed by dropwise addition of amines accompanied by rapid stirring until a product having desired stoichiometry was obtained.

[0032] During lapping, AHC compounds are believed to strongly chemisorb onto the AlTiC GMR Head specimen surfaces. As the ceramic material becomes abraded, the adsorbed AHC compounds impart a significant anionic charge to the swarf particles.

[0033] Significant interparticle electrostatic repulsion facilitates rapid removal of the swarf from the specimen during lapping.

[0034] In order to minimize corrosion of the GMR Head metallic layers during lapping, it is desirable that the lapping fluid have an alkaline pH. Under alkaline conditions the surface of AlTiC acquires a net negative charge which would normally inhibit the adsorption of these anionic carboxylate compounds. The presence of hydroxyl groups on the citrate anion, however, promotes adsorption even under alkaline conditions. Such adsorption may result from the formation of highly stable cyclic ligands bound to surface metal cations or via strong hydrogen bonding interactions between the hydroxyl groups and the ceramic surfaces.

[0035] Formulations F and K showed enhanced MRRs during AlTiC Head Lapping These rate enhancements likely result, in part, from the combination of pendent carboxylate and amide functional groups in the respective polymeric materials. The amide groups may promote polymer adsorption on the AlTiC surfaces via hydrogen bonding interactions.

[0036] A synergistic MRR effect is observed when lapping AlTiC with a Formulations E, I, and K. These Formulations include both citrate and polyaspartate species. Higher MRRs are observed by using formulations comprising a combination of these components compared to fluids containing either individual component alone. Applicant has discovered that preferred weight ratios of the polyaspartic (or aspartamide copolymer) to citrate salt range from 0.0 to about 1. In certain embodiments, Applicant's lapping formulation comprises a weight ratios of the polyaspartic (or aspartamide copolymer) to citrate salt range from about 0.1 to about 0.5.

[0037] Tables 5 and 6 recites formulations comprising polyaspartate or aspartamide copolymer, in combination with a citrate blend.

TABLE 5

Polyaspartamide Copolymer/Citrate Lapping Fluid Composition

Component	Concentration (Wt. %)
Non-aqueous fluid	90.7
MEA Citrate Tribasic	7.3
Poly Sodium Aspartate -co- diglycol Aspartamide (33 mol. % polymer repeat units amidated)	2.0

[0038]

TABLE 6

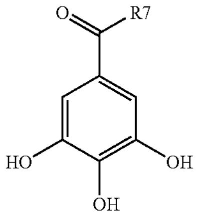
Polyaspartate/Citrate Lapping Fluid Composition

Component	Concentration (Wt. %)
Non-aqueous fluid	69.43
Triethanolamine Citrate Tribasic	25.9
Sodium Polyaspartate (Baypure DS 100)	4.67

[0039] In certain embodiments, Applicant's lapping composition comprises one or more species selected from the genus of compounds commonly known as flavonoids. Such flavonoids can be obtained from naturally-occurring materials, including without limitation green tea, brown tea, coffee, grapes, and the like. For example, grape extract is also rich in polyphenolic flavonoids. As those skilled in the art will appreciate, such flavonoids comprise compounds having one or more benzene ring moieties with two or more hydroxyl groups attached to those aromatic ring structures.

[0040] In certain embodiments, Applicant's lapping composition includes vanillin and/or vanillin derivatives made via reaction of vanillin's aldehyde moiety. Vanillin has been found to inhibit corrosion. In addition, the aldehyde group comprises functionality that allows vanillin to be grafted onto a polymer backbone via reaction with amines, i.e. formation of a Schiff Base, or reaction with a urea, or a reaction with other phenolic group moieties. As those skilled in the art will appreciate, Vanillin is nontoxic, has, a pleasant fragrance, and is available from both natural and synthetic sources. As, those skilled in the art will further appreciate, Vanillin is structurally similar to catechol having two adjacent oxygen-containing groups attached on an aromatic ring, where one of those groups comprises a methyl ether.

[0041] In certain embodiments, Applicant's lapping composition further includes compounds comprising hydroxyl substituted benzoic acids. In certain embodirnents, Applicant's lapping composition is formed using gallic acid, Compound II where R7 is OH. In certain embodiments, Applicant's lapping composition includes Compound II wherein R7 is selected from the group consisting of OH, O—R16, N(R17)(R18), and the like, wherein R16 is selected from alkyl, alkali metal cation, alkaline earth cation, cation IV, cation V, and the like, and wherein R17 and R18 are selected from hydrogen, alkyl, aryl, phenyl, and the like.

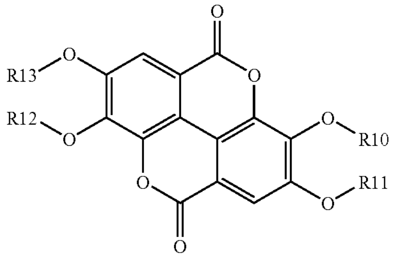


II

[**0042**] In certain of these embodiments, gallic acid is dispersed in a non-aqueous fluid in the amounts recited in Table I. Due to the highly acidic nature of gallic acid, Applicant neutralizes the gallic acid mixture using a variety of amines, alkali hydroxides, choline derivatives, guanidine derivatives, or TMAH prior to its use in lapping formulations. Suitable bases include but are not limited to alkanolamines, alkylamines, choline, guanidine derivatives, and the like. Alkaline lapping compositions are desirable to minimize corrosion which may occur during lapping of metallic surfaces. The reaction stoichiometry required to neutralize Applicant's gallic acid embodiments is a function of the pKa of the particular base used.

[**0043**] As a general matter, at least one equivalent of base is used for each equivalent of gallic acid. As a further general matter, Applicant has found it desirable to have lapping fluid compositions comprising a pH greater than 8. As those skilled in the art will appreciate, such a resultant pH requires excess equivalents of base with respect to the gallic acid used.

[**0044**] It is known in the chemical arts that Gallic acid dimerizes to form Ellagic acid derivatives, Compound III, where R10, R11, R12, and R13, are selected from the group consisting of ammonium salts, alkali metal ions, alkaline earth ions, guanidinium ions, choline cations, and the like.



III

[**0045**] In certain embodiments, Applicant's composition comprises polyphenolic compounds other gallic acid/gallates/gallamides. Such polyphenolic compounds include tannins; soluble black tea extracts; soluble green tea extract, such as for example Sunphenon 100S from Taiyo Kagaku Co., Ltd.); quebracho bark extract used for leather tanning; tannic acid; humic acid; catechol; pyrogallol, d, 1-Dopamine; d, 1-DOPA derivatives; N-Acetyl Dopamine; and the like.

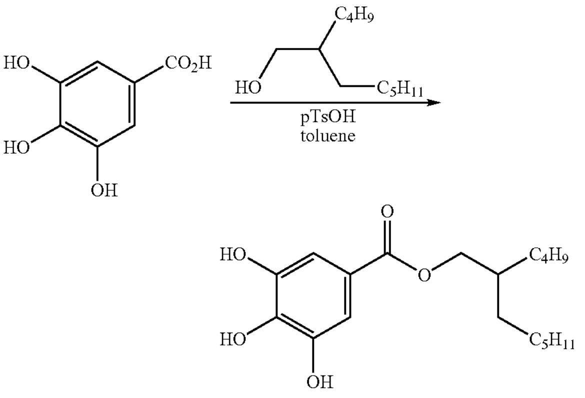
[**0046**] In certain embodiments, Applicant's composition comprises Catechin derivatives such as those found in tea extract, including but not limited to catechin, epicatechin, gallocatechin, epigallocatechin, epicatechin gallate, gallocatechin gallate, epigallocatechin gallate,

[**0047**] In certain embodiments, Applicant's composition includes Caffeoyl Derivatives, such as for example including esters, amides, and salts of caffeic acid. In certain embodiments, Applicant's composition includes for example Procyanidin derivatives, Theaflavins, Thearubins, Phenolic Flavanol derivatives, Galloyl Glucose derivatives, Rugosin D, Enterochelin, Chlorogenic acid and its derivatives, Tyrosine derivatives, Alkyl Catechol derivatives including but not limited to tert-Butyl Catechol, and combinations thereof.

[**0048**] In certain embodiments, Applicant's composition includes Phenol Ether derivatives including but not limited to guaiacol, veratrole, 2-methoxy phenol, manedelic acid, and the like, and combinations thereof. In certain embodiments, Applicant's composition includes one or more Orthoquinone derivatives.

[**0049**] Gallic acid, Ellagic acid, and related polyphenolic derivatives, exhibit low toxicity, and can be readily obtained via the hydrolysis of tannic acid, a natural product derived from wood by products. As those skilled in the art will appreciate, Tannic acid is essentially composed of a mixture of polysaccharides having a high concentration of gallic acid esterified along the saccharide polymer chains.

[**0050**] In certain embodiments, Applicant's non-aqueous lapping composition comprises the ester VI formed from gallic acid and 2-butyl 1-octanol, wherein that ester was formed in 74 percent yield.

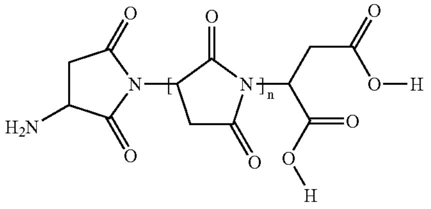


VI

[**0051**] Treating gallate ester VI either triethyl amine or triethanol amine, a dark tan solution was observed. Diluting the gallate ester VI/-triethyl amine (or triethanol amine) solution in 2-Butyl-1-octanol, then treated with Mineral Oil, a homogenous solution was observed. This solution had a light tan complexion.

[**0052**] In certain embodiments, Applicant's lapping composition comprises polymeric materials. In certain of these polymeric materials embodiments, Applicant's polymeric materials include a plurality of pendent gallic acid moieties and/or gallic acid derivatives, such as without limitation gallamide moieties. In certain embodiments, these polymeric materials are formed by reacting a polymer which includes a plurality of polysuccinimide repeat units with an amino-terminated gallamide.

[0053] In certain embodiments, Applicant's polymeric material which includes a plurality of gallamide moieties is formed from polysuccinimide XI, wherein n is in the range from 5 to 30. In certain embodiments, polysuccinimide XI has a number average molecular weight of about 3,000 Daltons.

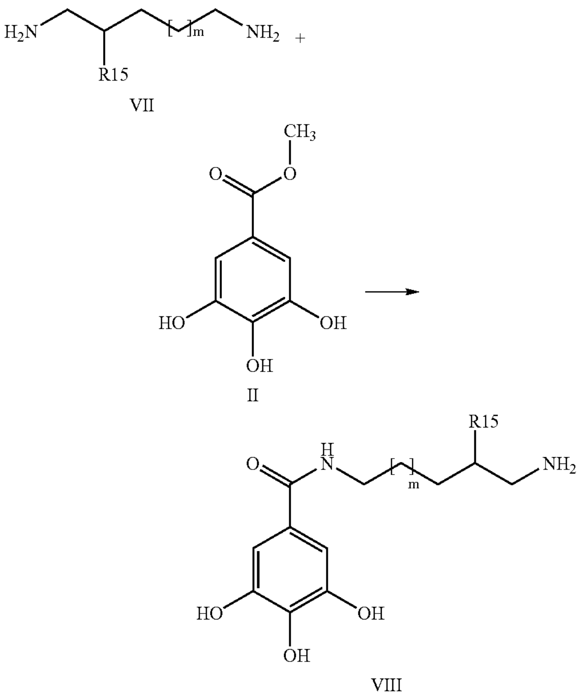


XI

[0054] In certain embodiments, methyl gallate, Compound II wherein R7 is O—Me, is reacted with a diamine to give an amino-terminated gallamide. Such diamines include, without limitation, diamino-terminated hydrocarbons, diamino-terminated polyethers, diamino-terminated polyamides, and the like, In other embodiments, methyl gallate is reacted with an amino alcohol to give a hydroxyl-terminated gallamide.

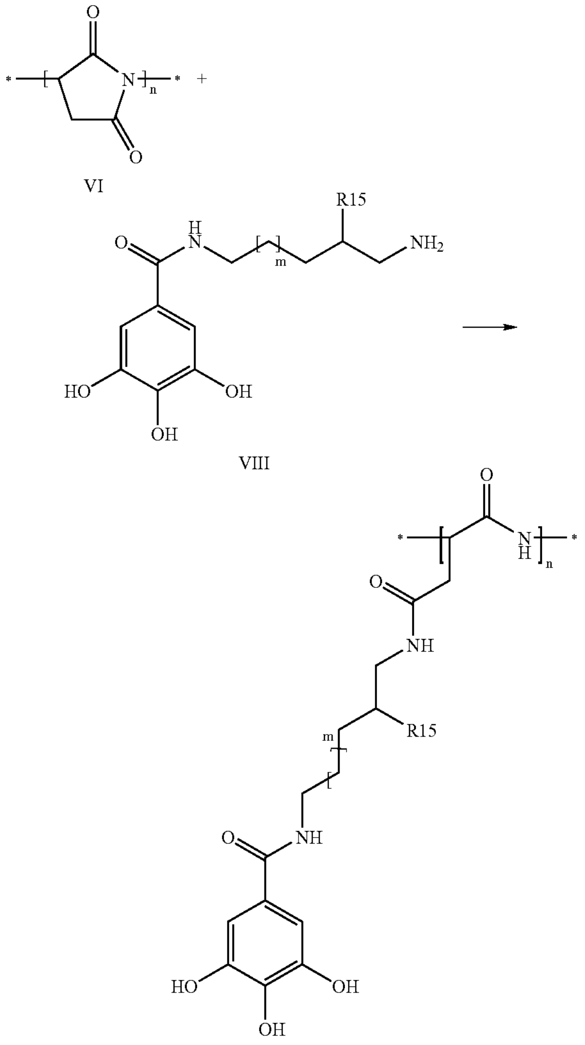
[0055] For example, in certain embodiments methyl gallate is reacted with diamine VII to give amino-terminated gallamide VIII wherein R15 is selected from the group consisting of hydrogen, alkyl, oxyalkyl, aryl, phenyl, and oxyphenyl, and wherein (m) is greater than or equal to 1 and less than or equal to about 10.

Reaction (1)



Amino-terminated gallamide VIII is then reacted with polysuccinimide VI to form derivatized polyaspartamide IX

Reaction (2)



IX

EXAMPLE 1

Synthesis of Compound VIII

[0056] About 6.60 g (0.036 mole) of Methyl-3,4,5-trihydroxybenzoate was dissolved in about 46.4 g of Dimethyl Sulfoxide (DMSO) by gently heating the mixture at 45° C. for about 20 minutes with stirring. As those skilled in the art will appreciate, DMSO is a polar, aprotic solvent having low toxicity and good biodegradability. Other polar, aprotic solvents may be used in place of, or in combination with, DMSO. Such polar, aprotic solvents include, for example, N-Methyl Pyrrolidone, Sulfolane, Tetrahydrofuran, or Dimethyl Sulphone.

[0057] The resulting DMSO solution was then added to a 250 ml round bottom flask which had been previously flushed with inert nitrogen gas. About 10.368 g (0.08922 mole) of 2-methyl-1,5-pentanediamine, Compound VII, wherein R15 is methyl and (m) is 1, is sold in commerce by DuPont under the tradename Dytek A, were then added to that DMSO solution, and the resulting reaction mixture was heated under vacuum at about 160° C. for about 8 hours. Diamine 2-methyl-1,5-pentanediamine was selected on the basis of its nucelopilicity combined with its high pKal value of 11.2.

[0058] During the reaction, methanol was removed from the reaction mixture as it was formed as the product from the aminolysis reaction occurring between the gallate ester and Diamine 2-methyl-1,5-pentanediamine. A clear, slightly reddish colored solution comprising about 26.6 weight percent compound VIII was obtained.

EXAMPLE 2

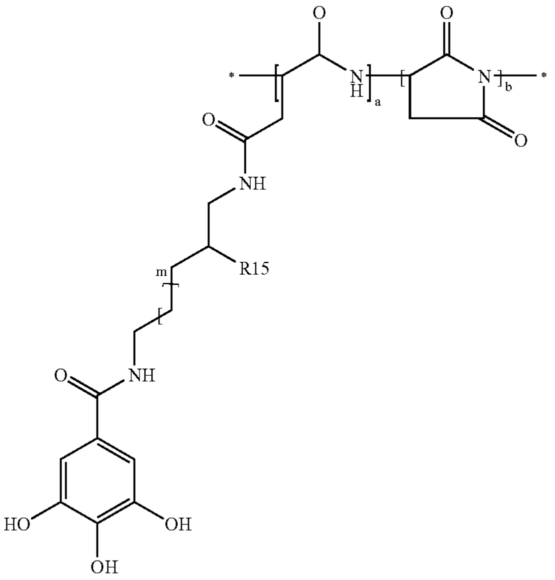
Polyaspartamide IX/X Preparation

[0059] About 7.82 g of 3,000 MW Polysuccinimide, polymer VI, sold in commerce by Bayer Chemicals Corp. Pittsburgh, Pa. under the tradename Baypure, was dissolved in about 33.0 g DMSO at 55° C. This yielded a solution comprising about 19.1 weight percent polymer VI

[0060] About 19.72 g of a DMSO solution of compound VIII, from Example 1, was diluted with about 8,61 g additional DMSO. This DMSO solution was rapidly mixed with the previously prepared DMSO/polymer VI solution.

[0061] In certain embodiments, a stoichiometric excess of Compound VII was added in an amount such that all the succinimide moieties were ring-opened. In other embodiments, less than a stoichiometric amount of VII was used, thereby giving co-polymer X having both succinimide linkages and substituted aspartamide linkages.

X



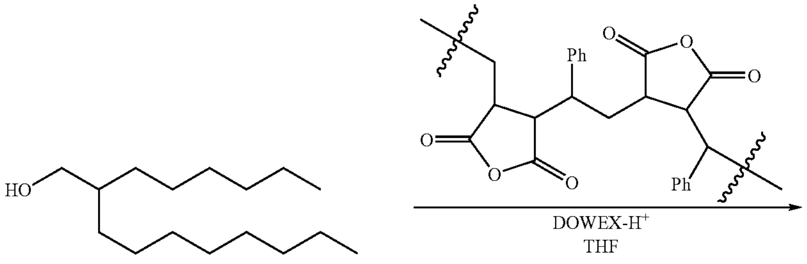
[0062] For example, in one embodiment about one-fourth of the succinimide repeat units in PSI VI were ring-opened using Reaction 2 to give co-polymer X wherein (a) is about 0.25(b), and wherein (a+b) is in the range of 4 to about 5000. The structure shown for polymer X should not be interpreted to mean that polymer X is necessarily a block copolymer. Rather in certain embodiments, the (a) repeat units are randomly dispersed among the (b) repeat units.

[0063] An exotherm was noticed upon mixing accompanied by a darkening of the solution such that it became sanguine in appearance. The solution was allowed to stand at room temperature for 30 minutes resulting in a clear, homogeneous, sanguine colored solution.

[0064] Applicant has discovered that grafting a plurality of gallamide groups onto a polyaspartic acid/aspartamide backbone gives a gallamide polymer, where Applicants' solvent system does not phase separate or precipitate upon standing. In contrast, the corresponding mixtures of potassium gallate having similar molar concentration of gallate moieties darkens and phase separates as particulates upon standing. As noted above, gallic acid is known to undergo oxidative coupling.

[0065] Significantly, Applicant has found that attaching pendant gallamide groups to a polymer backbone allows the product to stay in solution longer. In addition, Applicant has found that using a polymer comprising pendent gallate/gallamide groups rather than a plurality of lower molecular weight gallate anions has the added advantage of forming electrosteric stabilized dispersions. Individual gallate anions comprise small discrete charges and impart electrostatic charges upon the swarf as it is removed during the polishing operation. Gallamide functionalized polymers, on the other hand also electostatically charge the particles but by virtue of them being attached to a backbone enables the adsorbed polymer chain to form a brush like structure upon the swarf, further hindering particulate agglomeration.

[0066] In certain embodiments, Applicant's non-aqueous lapping composition comprises 2-Butyl-1-octanol grafted onto a styrene maleic anhydride (S MA) co-polymer. Treatment of 2-Butyl-1-octanol with SMA in the presence of DOWEX resin in refluxing THF provided the desired ester.



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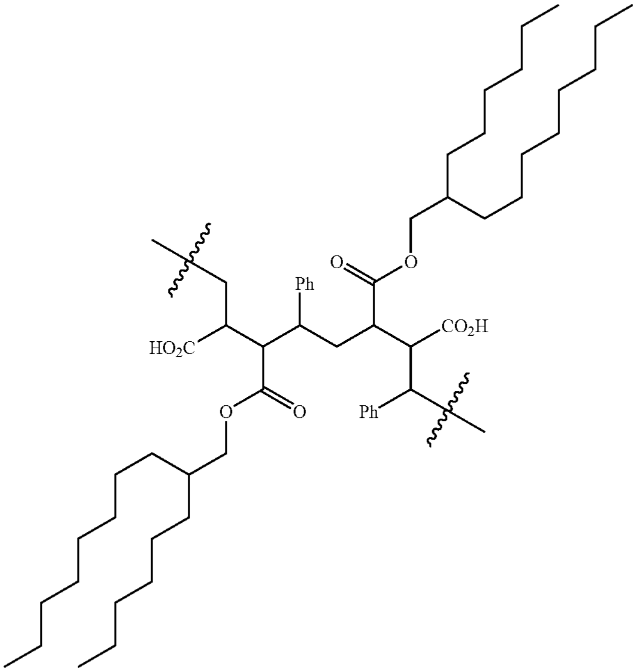
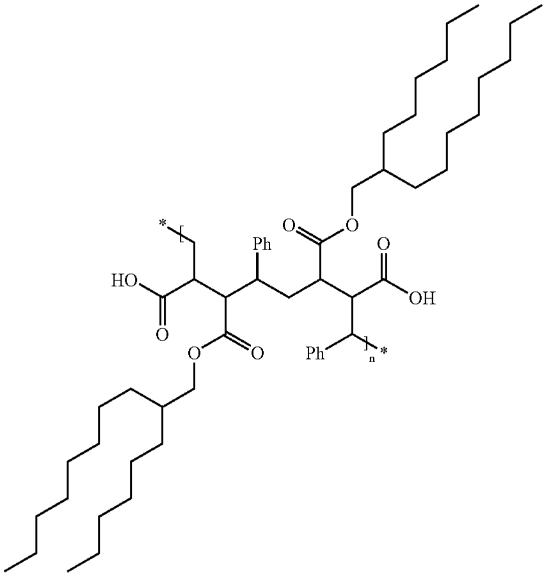


[0067] The structure of the ester product was established using IR spectroscopy. The top spectrum is of SMA copolymer. The bottom spectrum is of the product. The disappearance of the maleic anhydride carbonyl peak at 1770 cm^{-1} and the presence of a peak at 1743 cm^{-1} suggest the presence of an ester group. The weak stretches in the 3400-3600 cm^{-1} region suggests O—H bonds of carboxylic acid, not alcohol, moieties.

[0068] In certain embodiments, Applicant's non-aqueous comprises compound XI disposed in a non-aqueous fluid, wherein n is between 1 and about 5000.

XI



[0069] While the preferred embodiments of the present invention have been illustrated in detail, it should be apparent that modifications and adaptations to those embodiments may occur to one skilled in the art without departing from the scope of the present invention.

I claim:

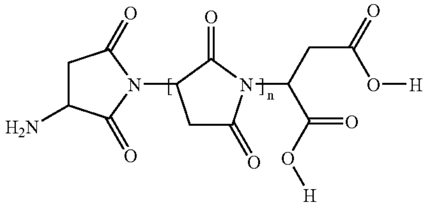
1. A lapping composition which does not comprise water, formed by mixing in a non-aqueous fluid an acid selected from the group consisting of citric acid, glycolic acid, tartaric acid, and gallic acid, and a base selected from the group consisting of monoethanolamine, triethanolamine, diglycolamine, guanidine, choline, tetraalkylammonium hydroxide, 2-ethyl-1-hexyl amine, bis(2-ethyl-1-hexyl) amine, tris (2-ethyl-1hexyl) amine, and potassium hydroxide.

2. The composition of claim 1, further comprising poly sodium aspartate.

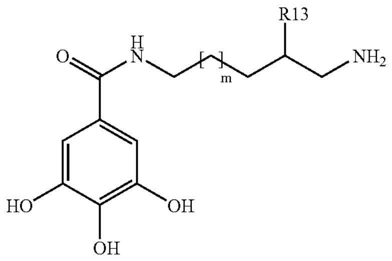
3. The composition of claim 1, further comprising poly sodium aspartate-co-diglycol aspartamide.

4. A lapping composition which does not comprise water, formed by adding to gallic acid in a non-aqueous fluid a base selected from the group consisting of monoethanolamine, triethanolamine, diglycolamine, guanidine, choline, tetraalkylammonium hydroxide, 2-ethyl-1-hexyl amine, bis(2-ethyl-1-hexyl) amine, and tris (2-ethyl-1hexyl) amine.

5. A lapping composition which does not comprise water formed by adding to a polysuccinimide having structure disposed in a non-aqueous fluid



a gallamide having the structure

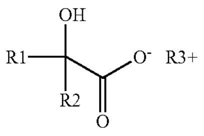


wherein n is between 5 and 30 and wherein m is greater than or equal to 1 and less than or equal to about 10, and wherein R13 is selected from the group consisting of hydrogen, alkyl, oxyalkyl, phenyl, and oxyphenyl.

6. The lapping composition of claim 5, wherein said polysuccinimide has a molecular weight of about 3,000 Daltons.

7. The lapping composition of claim 5, formed by adding about 0.25(n) equivalents of said gallamide to said polysuccinimide in DMSO.

8. A lapping composition which does not comprise water, comprising a salt having the structure

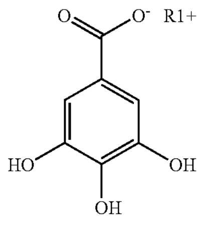


wherein R1 is selected from the group consisting of hydrogen, methyl, and CH_2—$CO_2^-R4^+$, and wherein R2 is selected from the group consisting of hydrogen, CH_2—$CO_2^-R5^+$, and C(H)(OH)—CO2$^-$R6$^+$, and wherein R3$^+$, R4$^+$, R5$^+$, and R6$^+$, are each selected from the group consisting of an alkali metal cation, an alkaline earth cation, a diglycolammonium cation, a monoethanolammonium cation, a triethanolammonium cation, a tetramethylammonium cation, a guanidinium cation, and a choline cation.

9. The composition of claim 8, further comprising poly sodium aspartate.

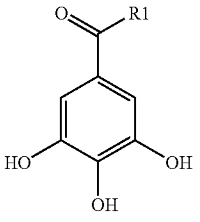
10. The composition of claim 9, further comprising poly sodium aspartate-co-diglycol aspartamide.

11. A lapping composition which does not comprise water, comprising a salt having the structure



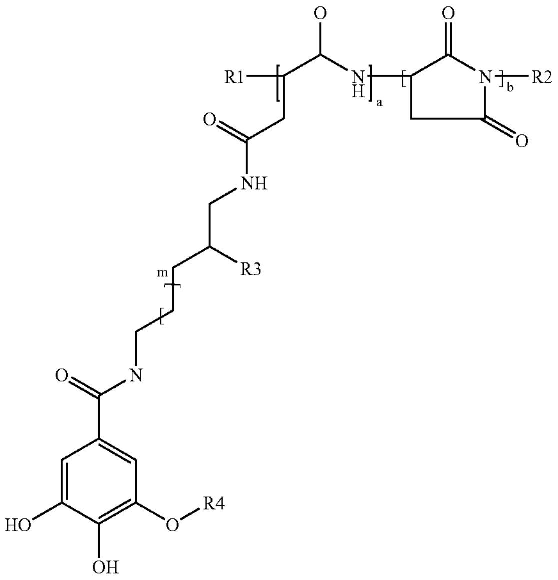
wherein R1+is selected from the group consisting of an alkali metal cation, an alkaline earth cation, a diglycolammonium cation, a monoethanolammonium cation, a triethanolammonium cation, a tetramethylammonium cation, a guanidinium cation, and a choline cation.

12. A lapping composition which does not comprise water, comprising a compound having the structure



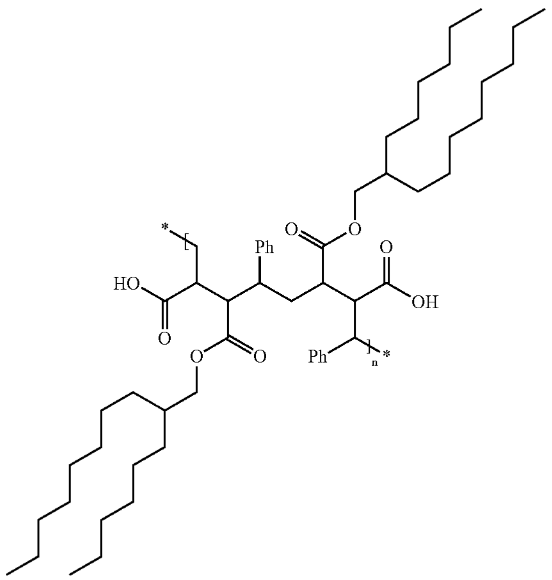
wherein R1 is selected from the group consisting of OH and N(R2)(R3), wherein R2 and R3 are each selected from the group consisting of alkyl and phenyl.

13. A lapping composition which does not comprise water, comprising a polymeric substituted gallamide having the structure



wherein R1 is selected from the group consisting of alkyl, aryl, succinimide, aspartic acid, and aspartate salt, and wherein R2 is selected from the group consisting of alkyl, aryl, succinimide, aspartic acid, and aspartate salt, and wherein (a+b) is between 4 and 5000 and wherein a is about (0.25)b, and wherein m is greater than or equal to 1 and less than or equal to about 10, and wherein R3 is selected from the group consisting of hydrogen, alkyl, oxyalkyl, phenyl, and oxyphenyl, and wherein R4 is selected from the group consisting of hydrogen and methyl.

14. A lapping composition which does not comprise water, comprising a polymeric material having the structure



wherein n is between 1 and 5000.

15. A method to shape the surface of a substrate, comprising the steps of:

providing a substrate, wherein said substrate comprises a target surface, wherein that target surface comprises a metal surface, a ceramic surface, or a combination thereof;

providing one or more abrasives;

providing a lapping composition which does not comprise water;

contacting said target surface with said one or more abrasives while contacting said target surface with said lapping composition is formed by mixing in a non-aqueous fluid an acid selected from the group consisting of citric acid, glycolic acid, taitaric acid, and gallic acid, and a base selected from the group consisting of monoethanolamine, triethanolamine, diglycolamine, guanidine, choline, tetraalkylammonium hydroxide, 2-ethyl-1-hexyl amine, bis(2-ethyl-1-hexyl) amine, tris (2-ethyl-1hexyl) amine, and potassium hydroxide.

16. A method to shape the surface of a substrate, comprising the steps of:

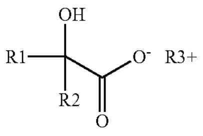
providing a substrate, wherein said substrate comprises a target surface, wherein that target surface comprises a metal surface, a ceramic surface, or a combination thereof;

providing one or more abrasives;

providing a lapping composition which does not comprise water;

contacting said target surface with said one or more abrasives while contacting said target surface with said lapping composition;

wherein said lapping composition comprises a salt having the structure



wherein R1 is selected from the group consisting of hydrogen, methyl, and CH_2—$CO_2^-R4^+$, and wherein R2 is selected from the group consisting of hydrogen, CH_2—$CO_2^-R5^+$, and $C(H)(OH)$—$CO2^-R6^+$, and wherein $R3^+$, $R4^+$, $R5^+$, and $R6^+$, are each selected from the group consisting of an alkali metal cation, an alkaline earth cation, a diglycolammonium cation, a monoethanolammonium cation, a triethanolammonium cation, a tetramethylammonium cation, a guanidinium cation, and a choline cation.

17. The lapping composition of claim 16, further comprising poly sodium aspartate.

18. The lapping composition of claim 16, further comprising poly sodium aspartate-co-diglycol aspartamide.

19. A method to shape the surface of a substrate, comprising the steps of:

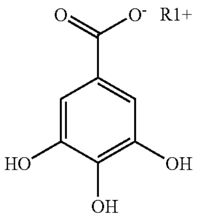
providing a substrate, wherein said substrate comprises a target surface, wherein that target surface comprises a metal surface, a ceramic surface, or a combination thereof;

providing one or more abrasives;

providing a lapping composition which does not comprise water;

contacting said target surface with said one or more abrasives while contacting said target surface with said lapping composition;

wherein said lapping composition comprises a salt having the structure



wherein R1+is selected from the group consisting of an alkali metal cation, an alkaline earth cation, a diglycolammonium cation, a monoethanolammonium cation, a triethanolammonium cation, a tetramethylammonium cation, a guanidinium cation, and a choline cation.

20. A method to shape the surface of a substrate, comprising the steps of:

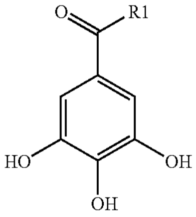
providing a substrate, wherein said substrate comprises a target surface, wherein that target surface comprises a metal surface, a ceramic surface, or a combination thereof;

providing one or more abrasives;

providing a lapping composition which does not comprise water;

contacting said target surface with said one or more abrasives while contacting said target surface with said lapping composition;

wherein said lapping composition comprises a compound having the structure



wherein R1 is selected from the group consisting of OH and N(R2)(R3), wherein R2 and R3 are each selected from the group consisting of alkyl and phenyl.

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