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ALKYL-SUBSTITUTED AROMATIC SULPHONIC ACIDS

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The present invention relates to improvements in moistening and permeating media, and in the process of manufacturing the same.

Broadly, the present invention relates to the use of sulphonated alkylated aromatic compounds and their metallic salts, preferably the inorganic water-soluble salts, wherein said aliphatic group is obtainable from a halogenated gasoline or kerosene fraction and preferably a gasoline or kerosene fraction having a distilling range of substantially 160° C. to 245° C. More particularly, the present invention relates to wetting agents, permeating agents and the like comprising sulphonated alkylated aromatic compounds and preferably aromatic hydrocarbons, as for example benzene, toluene, naphthalene, diphenyl, chlor diphenyl, mono chlorobenzene, dichlorbenzene and the like, wherein said aliphatic group contains more than nine and less than fourteen carbon atoms.

The products of the present invention are particularly adaptable for and find use in dyeing of fabrics and textiles wherein penetration of the dye is facilitated. Furthermore, the products of the present invention are of particular value in the kier boiling of cotton fabrics with dilute aqueous caustic solutions. The said compounds are also capable of being used to advantage in the paper and treating industry wherein it is desirable to improve the wetting effect. They may be used as penetrants in acid solutions which are used for carbonizing vegetable matter in wool. They may be employed as assistants in flax retting and as assistants in fulling and felting processes. They find use in sizing preparations in combination with the usual materials such as starches, gelatine and their equivalents. The products of the present invention may be conveniently employed as wetting, penetrating and like agents in bleaching liquors. They may be added to lye liquors for mercerizing cotton goods. They may be employed to improve the absorption capability of fibrous materials when such materials are subjected to treatments for finishing, softening, impregnating, waterproofing and mildew proofing. They may also be employed in lustering and delustering fabrics and in silk degumming.

The compounds of the present invention also may be used in the leather industry as wetting agents in deliming, soaking, tanning and dyeing baths.

These compositions may also be employed in conjunction with insecticidal compositions for enhancing the spreading and penetrating action thereof.

These compositions may also be employed as wetting agents in the ceramic industry and particularly may be employed in the manufacture of sewer pipe, brick and refractories.

As embodiments of the broader aspects of the invention kerosene was distilled and fractions collected having the distilling ranges of 140° to 162° C., 162° to 182° C., 182° to 204.5° C., 204.5° to 224.5° C., 224.5° to 244° C., 244° to 260° C., and 260° to 280° C. The fractions so obtained correspond to the boiling points of nonane, decane, undecane, dodecane, tridecane, tetradecane and pentadecane respectively. The distillates so obtained were halogenated by means of a suitable halogen, for example chlorine, the halide so obtained condensed with an aromatic compound, for example benzene, toluene, naphthalene, diphenyl, chlor diphenyl, mono chlorbenzene, o-dichlor benzene and the like and the product so obtained sulphonated by means of a suitable sulphonation agent, and the sulphonic acid isolated preferably as the alkali metal salt.

As illustrative of the above process substantially three molecular proportions of hydrocarbon distilling between 204.5° and 224.5° C. are substantially one-third chlorinated on a molar basis. The product so obtained is treated with substantially 6.4 molecular proportions of benzene in the presence of aluminum chloride and a Friedel-Crafts synthesis carried out, after which the unreacted benzene and unchlorinated hydrocarbon are removed preferably by fractionation from the alkyl benzene. Substantially 50 parts by weight of the alkyl benzene so obtained and substantially 10 parts by weight of 95% sulphuric acid are placed in a suitable reactor, and substantially 65 parts by weight of 25% oleum added slowly thereto, preferably with cooling. After the reaction is completed, water is added thereto to effect separation of the major portion of the spent acid, which is drawn off, and the upper layer comprising the alkyl benzene sulphonic acid is preferably converted to the desired metal salt.

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Other methods of preparing the preferred class of compounds may, of course, be employed as is obvious to those skilled in the art. Thus, it is obvious that other sulphonating agents, as for example chlor-sulphonic acid may be employed.

The metallic salts, as for example the sodium salts of the alkylated and sulphonated benzenes employing the kerosene fractions given above have all been prepared in a manner analogous to that hereinbefore described and on testing by the method of Carl Z. Draves as outlined in the 1931 and 1932 Year-books of the American Association of Textile Chemists and Colorists, found to possess desirable wetting properties in neutral, acid and alkaline media.

Briefly the Draves wetting test consists in measuring the time of immersion of a 5 gram skein of unbleached cotton made up of 35/2 cotton yarn, folded so as to be immersed in the liquid. This skein is attached to a 1.5 gram sinker which is itself attached to a heavy weight by means of a fine thread so that the distance between them is one inch. When the skein becomes thoroughly wetted, the small sinker causes it to fall to the bottom of the container, the time being accurately noted. The test is made in a tall cylinder.

As further specific embodiments of the present invention toluene, xylene, naphthalene, diphenyl, chlor diphenyl, mono chlorbenzene and o-dichlor benzene have been reacted with the halogenated kerosene distillates exemplified above, for example the alkyl halide prepared from the kerosene distillate having a distilling range of 162° to 182° C. and the alkylated aromatic hydrocarbon after sulphonating found on testing by the Draves wetting test to function as desirable and valuable wetting agents in neutral, acid and alkaline media. Further, diphenyl and chlordiphenyl have been reacted with the alkyl halide prepared from a kerosene distillate having a distilling range of 182° to 204.5° C. and the alkylated compound after sulphonating found to possess desirable wetting action.

As a preferred embodiment of the present invention aliphatic hydrocarbons have been prepared by careful refractionation of cuts obtained from commercial kerosene. The octane, nonane, decane, undecane, dodecane, tridecane, tetradecane, pentadecane and hexadecane fractions so prepared were halogenated preferably by means of chlorine, and the halide so formed condensed with an aromatic hydrocarbon for example benzene in the presence of aluminum chloride as a catalyst and the alkyl benzene sulphonated in a manner analogous to that employed above in the preparation of an alkylated sulphonated aromatic compound employing halogenated kerosene cuts as the alkylating agent.

Heretofore, certain sulphonated alkylated aromatic hydrocarbons have been employed as wetting, penetrating and capillary agents. According to the present invention, however, it has been discovered that the most efficient wetting properties are present in those sulphonated alkylated aromatic compounds which contain more than nine and less than fourteen carbon atoms in the side chain. This is demonstrated and will be evident from the following data wherein the wetting times of the sodium salts of sulphonated alkylated benzenes containing eight, nine, ten, eleven, twelve, thirteen, fourteen, fifteen, sixteen and twenty-three carbon atoms in the side chain are compared. The wetting times were determined by the Draves method at 25° C.

TABLE I

Tests in distilled water

Grams wetting agent per liter	5.0	2.5	1.25
Wetting agent	Time of wetting in seconds		
Octyl benzene sodium sulphonate	12.7	Over 180	
Nonyl benzene sodium sulphonate	4.6	12.5	168
Decyl benzene sodium sulphonate	5.0	6.5	12.9
Undecyl benzene sodium sulphonate	4.2	5.9	12.0
Dodecyl benzene sodium sulphonate	5.0	6.5	11.7
Tridecyl benzene sodium sulphonate	5.1	6.5	13.2
Tetradecyl benzene sodium sulphonate	12.4	13.6	32.4
Pentadecyl benzene sodium sulphonate	32.6	60.9	91.2
Hexadecyl benzene sodium sulphonate	92.0	255.0	
Tricosyl benzene sodium sulphonate	126 min.	180 min.	186 min.

TABLE II

Tests in 2% sulphuric acid solution

Grams wetting agent per liter	5.0	2.5	1.25
Wetting agent	Time of wetting in seconds		
Octyl benzene sodium sulphonate	12.7	Over 180	
Nonyl benzene sodium sulphonate	Instant	5.2	30.9
Decyl benzene sodium sulphonate	Instant	3.5	11.7
Undecyl benzene sodium sulphonate	Instant	4.5	9.8
Dodecyl benzene sodium sulphonate	Instant	5.7	11.9
Tridecyl benzene sodium sulphonate	5.5	7.7	14.8
Tetradecyl benzene sodium sulphonate	11.9	16.7	37.2
Pentadecyl benzene sodium sulphonate	35.2	55.6	84.6
Hexadecyl benzene sodium sulphonate	126	165	300
Tricosyl benzene sodium sulphonate	68 min.	85 min.	22 min.

It is thus clearly shown that the use of wetting agents comprising salts of sulphonated alkylated aromatic compounds containing more than nine and less than fourteen carbon atoms in the side chain comprises an important advance in the art.

What is claimed is:

1. The process of preparing a wetting agent which comprises substantially monochlorinating a kerosene fraction distilling between substantially 160° C. and 245° C., and consisting of paraffinic hydrocarbons, the major proportion of which is selected from the group consisting of decane, undecane, dodecane, and tridecane, employing the mixture of alkyl chlorides so produced as an alkylation medium by condensing therewith by means of a Friedel-Crafts synthesis an aromatic hydrocarbon of the benzene series and sulphonating the condensation products to form the mono-sulphonic acids thereof.

2. The process of preparing a wetting agent which comprises substantially monochlorinating a kerosene fraction distilling between substantially 160° C. and 245° C. and consisting substantially of paraffinic hydrocarbons and containing more than nine and less than fourteen carbon atoms, condensing therewith by means of a Friedel-Crafts synthesis an aromatic hydrocarbon of the benzene series, sulphonating the condensation products to form a mixture of alkylated benzene mono-sulphonic acids, and converting the sulphonic acids to the alkali metal salts.

3. A wetting agent composition comprising essentially a mixture of mono-sulphonated alkylated benzene compounds obtained by introducing

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chlorine into an excess of a kerosene fraction consisting substantially of paraffinic hydrocarbons, the major proportion of which is selected from the group consisting of decane, undecane, dodecane and tridecane, and having a distilling range of substantially 160° C. to 245° C., so as to produce a preponderant proportion of alkyl monochlorides, employing said alkyl monochlorides as an alkylation medium by condensing therewith a substantial molecular excess, based on the alkyl monochlorides present, of an aromatic hydrocarbon of the benzene series to obtain substantially monoalkylated benzene compounds, and sulphonating said monoalkylated benzene compounds.

4. A wetting agent composition comprising essentially a mixture of mono-sulphonated alkylated benzene compounds obtained by introducing chlorine into an excess of a kerosene fraction consisting substantially of paraffinic hydrocarbons, the major proportion of which is selected from the group consisting of decane, undecane, dodecane and tridecane, and having a distilling range of substantially 160° C. to 245° C., so as to produce a preponderant proportion of alkyl monochlorides, employing said alkyl monohalides as an alkylation medium by condensing therewith an aromatic hydrocarbon of the benzene series and sulphonating the alkylated benzene compounds.

5. A wetting agent composition comprising essentially a mixture of water-soluble inorganic salts of mono-sulphonated alkylated benzene hydrocarbons obtained by introducing chlorine into an excess of a paraffin base kerosene fraction consisting substantially of paraffinic hydrocarbons, the major proportion of which is selected from the group consisting of decane, undecane, dodecane and tridecane, and having a distilling range of substantially 160° C. to 245° C., so as to produce a preponderant proportion of alkyl monochlorides, employing said alkyl monochlorides as an alkylation medium by condensing therewith an aromatic hydrocarbon of the benzene series, sulphonating the alkylated benzene hydrocarbons and converting the sulphonic acids to the water-soluble inorganic salts.

6. A wetting agent composition comprising essentially a mixture of alkali metal salts of monosulphonated alkylated benzene hydrocarbons obtained by introducing chlorine into an excess of a paraffin base kerosene fraction consisting substantially of paraffinic hydrocarbons distilling between substantially 160° C. to 245° C., and containing more than nine and less than fourteen carbon atoms, so as to produce a preponderant proportion of alkyl monochlorides, employing said alkyl monochlorides as an alkylation medium by condensing therewith an aromatic hydrocarbon of the benzene series, and sulphonating the alkylated benzene hydrocarbons and converting the sulphonic acids to the alkali metal salts.

7. A wetting agent composition comprising essentially a mixture of alkali metal salts of monosulphonated alkylated benzenes obtained by introducing chlorine into an excess of a paraffin base kerosene fraction consisting substantially of paraffinic hydrocarbons distilling between substantially 160° C. and 245° C., and containing more than nine and less than fourteen carbon atoms, so as to produce a preponderant proportion of alkyl monochlorides, employing said alkyl monochlorides as an alkylation medium by condensing therewith benzenes, sulphonating the alkyl benzene and converting the sulphonic acids to the alkali metal salts.

8. A wetting agent composition comprising essentially an alkali metal salt of mono-sulphonated alkylated benzene hydrocarbons obtained by introducing chlorine into an excess of a paraffin base kerosene fraction distilling between substantially 162° C. and 182° C., and consisting substantially of paraffinic hydrocarbons, the major proportion of which is a decane, so as to produce a preponderant proportion of alkyl monochlorides, employing said alkyl monochlorides as an alkylation medium by condensing benzene therewith, sulphonating the alkylated benzenes and converting the sulphonic acids to the alkali metal salts.

9. A wetting agent composition comprising essentially an alkali metal salt of mono-sulphonated alkylated benzene hydrocarbons obtained by introducing chlorine into an excess of a paraffin base kerosene fraction distilling between substantially 182° C. and 204.5° C. and consisting substantially of paraffinic hydrocarbons, the major proportion of which is an undecane, so as to produce a preponderant proportion of alkyl monochlorides, employing said alkyl monochlorides as an alkylation medium by condensing benzene therewith, sulphonating the alkylated benzenes and converting the sulphonic acids to the alkali metal salts.

10. A wetting agent composition comprising essentially an alkali metal salt of mono-sulphonated alkylated benzene hydrocarbons obtained by introducing chlorine into an excess of a paraffin base kerosene fraction distilling between substantially 204.5° C. to 224.5° C., and consisting substantially of paraffinic hydrocarbons, the major proportion of which is a dodecane, so as to produce a preponderant proportion of alkyl monochlorides, employing said alkyl monochlorides as an alkylation medium by condensing benzene therewith, sulphonating the alkylated benzene hydrocarbons and converting the sulphonic acids to the alkali metal salts.

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