April 11, 1950 J. W. KENNEY, JR., ET AL. 2,503,744

METHOD AND AGENT FOR PREVENTING LINTING IN THE DRY CLEANING OF FABRICS

Filed Aug. 12, 1947

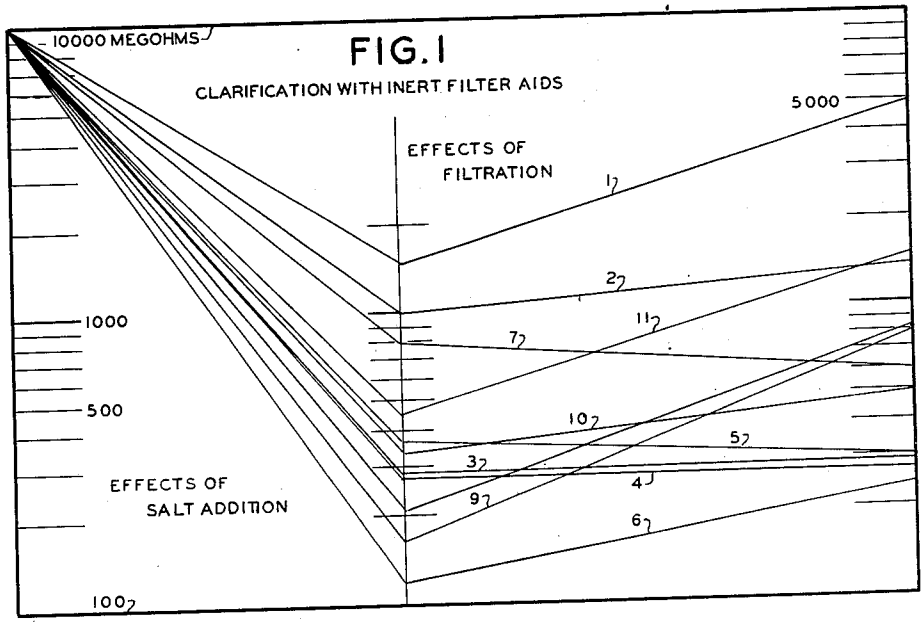


FIG. 1

CLARIFICATION WITH INERT FILTER AIDS

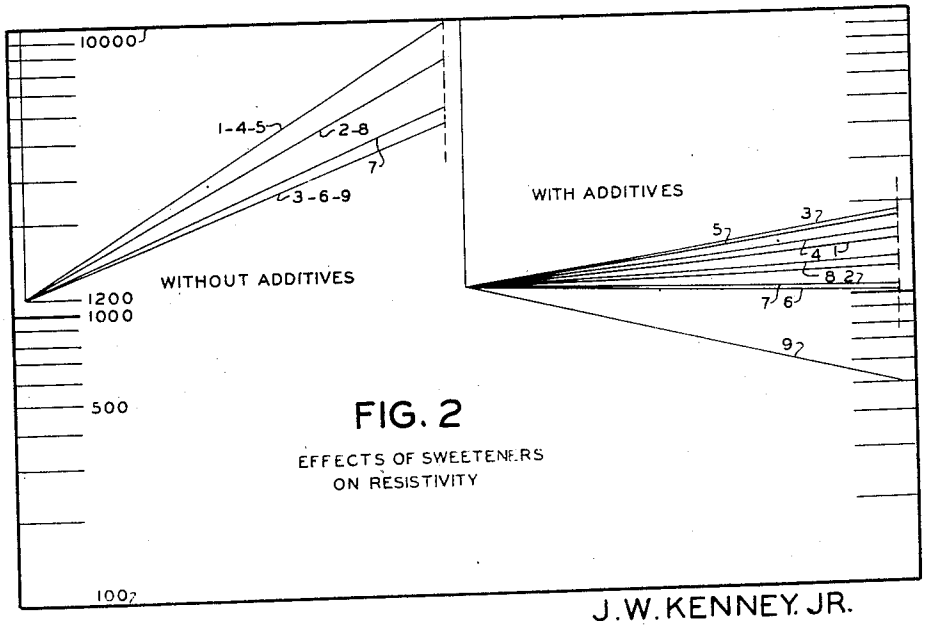


FIG. 2

EFFECTS OF SWEETENERS ON RESISTIVITY

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Patented Apr. 11, 1950 2,503,744

UNITED STATES PATENT OFFICE

2,503,744

METHOD AND AGENT FOR PREVENTING LINTING IN THE DRY CLEANING OF FABRICS

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Application August 12, 1947, Serial No. 768,166

12 Claims. (Cl. 252—89)

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This invention relates to the dry cleaning of fabrics and specifically to means by which the solvents used by dry cleaners may be rendered electrically conductive and maintained in that condition.

A purpose of the invention is to provide a filter-aid through the use of which a used solvent may be clarified for reuse and simultaneously rendered electrically conductive.

A purpose of the invention is to provide methods and agents through the use of which in the clarification of solvents the redeposition of soil and lint on the cleaned fabrics may be wholly prevented.

In the use of gasoline as a cleaners' solvent, once common practice, a serious risk of fire or explosion resulted from the accumulation of static charges on agitation of the solvent with the fabrics being cleaned, and many attempts were made to ground off and dissipate these charges. With the increasing use of Stoddard solvent and some of the chlorides of carbon to replace gasoline, this risk has substantially disappeared, Stoddard solvent having a flash test of 100° Fahr. or above while carbon tetrachloride and perchlorethylene are noninflammable.

The static charges which formerly engendered risk of explosion are also effective in causing deposition of soil and lint on the fabrics being cleaned, redeposition of suspensoids occurring as the charge increases during the period of the run. The redeposition of soil solids is detrimental to the appearance of white and light colored fabrics while the accumulation of lint on dark colored woolens, velvets and the like is still more detrimental to the quality of the finished work.

The mere grounding of the cleaning vessel does not suffice to prevent the accumulation of static because of the high dielectric strength of the various solvents, and many attempts have been made to render either the fabrics themselves or the solvent batch electrically conductive.

Thus, it is somewhat common practice to humidify the fabrics prior to their immersion in the cleaning bath, thus causing them to discharge their static on touching the wall of a grounded cleaning vessel. This method is moderately effective but has the obvious disadvantages that humidification is a tedious operation and one which, if carried too far, may result in damage to the fabrics.

The step most commonly taken is to add to the bath an alkali metal soap, usually sodium

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oleate, together with a free fatty acid, usually oleic acid—i. e., a superfatted soap. This method may be made effective for reducing the resistivity of the solvent to the level at which redeposition (commonly referred to as "linting") is prevented, and the added soap, under suitable conditions, functions also as an important aid to cleansing.

The use of alkali metal soaps as cleansing aids is well established, but when an attempt is made to render the solvent sufficiently conductive in this manner, serious difficulties are encountered. First, the quantity of soap required to produce conductivity is very large, and as the added soap is removed when the used solvent is reconditioned by filtering, this unduly large quantity must be added each time the solvent is filtered. This is an item of expense which is very nearly prohibitive.

Again, the alkali metal soaps are insoluble in the commercial solvents other than in the presence of enough free oleic acid to form a ternary system, and this system is unstable in the presence of material quantities of water, an emulsion being formed and the conductivity disappearing. Thus great care is required in the addition of the water which is necessary to render the soap effective as an aid to cleansing. And finally, in order to avoid the very undesirable presence of soap and free fatty acid in the finished work, it is necessary to omit these additives during the break and rinse stages, thus leaving the solvent highly resistive in the particular periods during which conductivity is most essential to the prevention of linting.

We have discovered that such of the oleates of the heavy metals as are oil-soluble, when added to the solvent in minute quantity and under suitable conditions, are capable of rendering the solvent conductive to such degree that linting is wholly prevented. We have also discovered that the quantity of heavy metal oleate required for this purpose is so small that it may be present in the solvent during the last stages of the wash, thus affording protection against linting throughout the operation. We have also discovered, and this is most surprising, that these heavy metal soaps, unlike the superfatted alkali metal soaps, are not removed from the solvent by the work nor by filtration with diatomaceous earth filter aids, and that the conductivity imparted by them persists through repeated uses and filtrations.

So far as we have been able to determine, all of the heavy metals which form oil-soluble ole-

ates are functional for this purpose to some degree, though their relative effectiveness and utility vary over a wide range. For practical purposes the choice of a metal to form the oleate lies between six of the group II metals (magnesium, calcium, zinc, strontium, cadmium and barium), three of the group VIII metals (iron, nickel and cobalt), and copper. The utilities of certain of these metals are limited to special cases: of zinc, barium and copper by the toxicity of the oleate, and of iron, nickel, cobalt and copper by the relatively dark colors of the oleates. A trace of a toxic oleate in rugs, woolen drapes and the like might well be advantageous as moth protection, while a dark color would be harmless in the cleansing of dark or black fabrics. The quantity of the oleate retained by the work after centrifuging would be almost disappearingly small, considering the extremely low concentration of the oleate in the rinse solvent. On the score of low cost combined with high effectiveness our preference is for the oleates of magnesium, strontium, barium and calcium.

The stearates of these metals have not been found useful for this purpose and, so far as we are at present aware, only the oleates are functional to a useful degree.

The invention may take two forms, in one of which the oleate as such is added to the solvent while in the other it is formed in the batch by the reaction of a salt of the heavy metal with an alkili metal soap previously added to or present in the solvent. In either case the agent is introduced into the solvent in intermixture with a diatomaceous earth filter aid, this step being essential to practical operativeness for reasons which will appear hereinafter.

In the first form of the invention, the preferred oleate is dispersed throughout the filter aid powder in a finely comminuted condition, in small proportions of the general order of 0.5% to 1.0% by weight, these figures being suggestive only and not critical. As these oleates are found in commerce they are in the form of waxy or rubbery grains or masses, very slowly soluble in the solvent and extremely difficult to reduce to powdered form. We have therefore devised a method, later described, by which these soaps may be formed by direct reaction between oleic acid and the oxide or hydroxide of the metal, in the absence of water, the resulting product being friable and readily reduced to finely powdered form by grinding.

The filter aid containing the heavy metal oleate may be added to the batch of solvent at the beginning of the run, or it may be formed into a precoat on a filter leaf and the solvent circulated through it. In either case the oleate, being in a fine state of subdivision and intimately dispersed throughout the earth, passes rapidly into solution and the solvent is rendered conductive immediately. The quantity of the oleate required to maintain conductivity is of the general order of 0.005% by weight in the case of magnesium oleate or slightly more in the use of the other oleates.

The solvent so treated does not lose its conductivity by repeated filtrations through diatomaceous earth filter aids, through conductivity will diminish as the concentration decreases with the addition of make-up untreated solvent and may be reduced or destroyed by the use of highly adsorbent "sweetening" powders, a subject which will be considered separately. The treated solvent is not affected by the presence or absence of alkali metal soaps which, if used, should be added in such quantity only as is required to obtain the maximum cleansing effect.

In the form of the invention in which the heavy metal oleate is produced in situ it is necessary to have present in the solvent a quantity of an alkali metal oleate sufficient to produce the desired quantity of heavy metal oleate by double decomposition, together with a sufficient quantity of water to produce ionization. The alkali metal soap merely provides a source of fatty acid radicals for reaction with the heavy metal salt and the quantity requisite for this purpose is extremely small, a mere fraction of the quantity ordinarily added to the solvent as a cleansing aid. The presence of a quantity of alkali metal soap greater than that required for the reaction with the heavy metal salt does not interfere, nor does it affect conductivity in either direction. In practice, the soap added will usually be superfatted, but the free oleic acid of such a soap is not appreciably reactive with the heavy metal salt, and free oleic acid may not be substituted for alkali metal soap other than in instances in which a hydroxide of an alkaline-earth metal is substituted for the heavy metal salt.

Any water-soluble salt of one of the metals above named may be used in admixture with the diatomactous earth filter aid, though the nitrates and chromates, while fully functional, might be undesirable because of the possible detrimental effect of traces of the corresponding alkali metal salts which might remain in the finished work. By preference, we use the chlorides and such of the sulfates and hydroxides of the named metals as are sufficiently water-soluble for reaction. These substances are referred to collectively herein as water-soluble inorganic compounds of the metals named.

The quantity of the salt added to the filter aid is not critical but will ordinarily be of the order of 0.1% to 1.5% by weight, varying somewhat with the combining weight of the metal. The salt or hydroxide, as the case may be, is reduced to a finely powdered condition and intimately blended with the powdered filter aid. The blend is applied to the solvent in either manner above described, i. e., by addition to the batch of solvent or by formation of a filter precoat through which the solvent is passed. The reaction between heavy metal salt and alkali metal soap goes to completion with some rapidity and as fast as the heavy metal soap is formed the solvent becomes conductive.

The figures in Table 1 below illustrate the effectiveness of several of these salts in reducing the resistivity of a solvent containing a superfatted alkali metal soap and in maintaining the reduced resistivity through a step of filtration to brightness with an inert filter aid.

In these experiments one batch of 1000 ml. of fresh solvent having a resistivity of 10,000 megohms was treated with 1.3 grams of a commercial dry cleaners' soap, then agitated for 20 minutes with 10 grams of diatomaceous earth filter aid and finally filtered to brightness through paper. Ten additional batches were treated each with a mixture of 1.3 grams soap and 2.6 grams water, agitated for 20 minutes with 10 grams of the same filter aid with which in nine cases 0.0004 gram molecule of the various salts named in the table had been blended, and finally filtered to brightness. The resistivity in megohms instrument reading was determined on each batch, both before and after filtration.

TABLE 1

Test No.		Resistivity in Megohms	
		Before Filtration	After Filtration
1	Soap only	1,500	5,000
2	Soap and water	10,000	10,000
3	Soap + water + $MgCl_2$	280	290
4	Soap + Water + $MgSO_4.7H_2O$	275	270
5	Soap + water + $MgSO_4$, Anhydrous	360	300
6	Soap + water + $CdCl_2$	120	260
7	Soap + Water + $BaCl_2$	800	600
8	Soap + water + $SrCl_2$	210	850
9	Soap + water + $CaCl_2$	165	800
10	Soap + water + $ZnSO_4$	330	500
11	Soap + water + $Al_2(SO_4)_3$	450	1,200

In Fig. 1 of the attached drawings, in which these results are shown graphically, the point at the upper left corner of the chart is the original resistivity of the solvent; the low point is the resistivity to which it is brought by the added agents without filtration, and the right hand point is the resistivity after filtration, the upward slope of the line indicating the stability of the imparted conductivity to filtration.

A resistivity not exceeding 1750 megohms (as measured on the instrument used) if constantly maintained, is sufficient to avoid linting and soil redeposition. Soap alone (test 1) in the quantity used, did not meet the test after filtration; soap and water (test 2) in proportions commonly used in cleaning room practice did not meet the test. The added salts, with the exception of aluminum sulfate, brought the resistivity to a considerably lower figure and held it at a lower level through the step of filtration, affording protection against the advent of water in such quantity as would destroy the conductivity produced by the superfatted soap and also affording room for further reduction of even the very minute quantities of agents used.

All of the resistivities mentioned herein are observed figures with the instrument and cell used and may be converted into specific resistivity in megohms per centimetre cube by dividing the observed figure by 0.00702.

As above stated, permanent conductivity is attained only when the filter aid used is inert, i. e., substantially nonadsorptive. When filter aids having decolorizing properties are used, the heavy metal oleate may be selectively adsorbed and thus removed from the solvent, the desired conductivity being reduced or lost.

It is possible, however, by either of two modifications of the above procedure, to maintain such degree of conductivity as to avoid linting and at the same time to maintain the purity of the solvent by the use of adsorptive sweetening powders.

One alternative is to alternate the treatment by which conductivity is produced with a purifying treatment with a sweetener, which may reduce or even destroy the conductivity and require that it be reestablished. In this procedure the solvent is maintained in a conductive condition in either manner above described until such time as it becomes overloaded with coloring matter or other adsorbable impurity. The impure solvent is then given a purifying treatment with an adsorbent powder, after which it is brought back to the desired conductivity (if then too low) by the addition of a filter aid carrying a heavy metal soap or by the addition of a filter aid carrying a heavy metal salt to a solvent containing an alkali metal soap. The same apparatus may be used for both treatments but it is desirable to apply

the purifying treatment to a batch of solvent not carrying any fabrics to be cleansed.

The other alternative is to add to the adsorptive filter aid or sweetener a small quantity of one of the heavy metal soaps and also a somewhat increased quantity of a heavy metal salt, using this agent on a solvent containing enough alkali metal oleate to produce double decomposition. As shown in Table 2 below, the effect of the double addition of soap and salt is to provide an immediate supply of the heavy metal soap for the initial production of conductivity and a supply of soap more slowly produced by reaction to replace that withdrawn by adsorption.

The results of these additions to the sweetening agent in preventing undue rise in resistivity during the period of a normal dry cleaning run are shown in Table 2 below. In these experiments a used solvent, having an initial resistivity of about 10,000 megohms and a resistivity in the condition taken of about 1,200 megohms (due to the presence of superfatted soap) was intimately blended with 1.3 gram soap and 2.6 gram water per 1000 millilitres. Successive batches of this low resistivity solvent were then strongly agitated for twenty-five minutes with one of the sweeteners described in the second column of the table, parallel batches being treated with the sweetening powder per se and with the same sweetener with the addition of 1% crystalline magesium sulfate and ¼% magnesium oleate, these quantities being weight percents of the total sweetener and the sweetener added being in each case 1% of the weight of the solvent. At the end of each treatment, and without filtration, the instrument resisivity of the solvent was again determined.

TABLE 2

Test No.	Composition of Sweetener	Resistivity After Treatment	
		Without Additives	With Additives
1	Filter Aid+Synthetic Adsorbent	10,000	1,600
2	Filter Aid+Synthetic Adsorbent+Carbon	7,500	1,300
3	Commercial Sweetener A	5,000	2,000
4	Synthetic Adsorbent A	10,000	1,700
5	Synthetic Adsorbent B	10,000	1,900
6	Filter Aid+Acid Treated Clay+Carbon	5,000	1,050
7	Filter Aid+Raw Adsorbent Clay+Carbon	4,500	1,100
8	Commercial Sweetener B	7,500	1,400
9	Filter Aid+Activated Clay	5,000	500

The comparisons are shown more effectively in Fig. 2 than by the figures in the table. It will be noted that in all cases the treatment with the unmodified sweetener brought the solvent to a resistivity far above that which is consistent with freedom from linting. On the other hand, the sweeteners modified by the addition of the salt and of a minute quantity of the oleate carried the solvent above the safe level in only two of the tests (3 and 5) while in four of the tests the resistivity was increased to only a minor degree (1, 2, 4 and 8) and in three it is reduced by the treatment with the sweetener (6, 7 and 9). Purifying treatments of this nature may be interspersed between runs in which an inert filter aid is used without interrupting the work of the cleaning plant.

Table 3 below illustrates the beneficial effect of the addition of a heavy metal salt to cleaning batches in which an alkali metal soap was used as a cleasing aid. These figures are the results

of plant scale runs in each of which the quantity of filter aid added to the batch was 2 pounds. In runs Nos. 1 to 6 inclusive the filter aid was diatomaceous earth containing 0.5% by weight of magnesium sulfate, crystalline. In runs 7 to 10 the filter aid was straight diatomaceous earth, and in runs 11 to 15 the filter aid was the diatomaceous earth used for making the agent used in runs 1 to 6 but without the addition of the salt. The observations of dusting and linting were made by trained observers.

TABLE 3

Plant test data

Load No.	Type Garments	Weight Load	Quarts Soap	Observed Dusting and Linting
		Pounds		
1	Mixed Woolens	200	8	None.
2	Dark Woolens	200	8	Do.
3	Men's Overcoats	200	4	Do.
4	Dark Trousers	200	8	Do.
5	Men's Suits	200	8	Do.
6	Velvet Drapes	65	2	Do.
7	Mixed Woolens	200	8	Linting.
8	Men's Suits	200	8	Do.
9	Men's Overcoats	200	4	Do.
10	Velvet Drapes	65	2	Heavy Linting.
11	Mixed Woolens	200	8	Linting.
12	Men's Suits	200	8	Do.
13	Men's Trousers	200	8	Do.
14	Ladies' Dark Coats	200	8	Do.
15	Men's Overcoats	200	4	Do.

The quantity of soap used in each of these runs was that customarily used for its effect in cleansing the fabrics and only a most minute part of it was consumed by combination with the magnesium salt.

The production of mixtures of filter aid with the heavy metal soaps of oleic acid is rendered very difficult by the unfortunate physical properties of these bodies. As found in commerce these substances are tough and gummy solids or very viscous semisolids, impossible to reduce to powdered form and therefore extremely difficult to disperse in small proportion through a mass of filter aid.

We have discovered that the heavy metal oleates may be produced by direct reaction between oleic acid and an oxide, hydroxide or, in some cases, a carbonate of the metal, and that the product of such reaction may be pulverized readily when an excess of the metallic compound is used and the reaction is completed by heating.

For example, in the following experiments, oleic acid was mixed at 25° C. for about 5 minutes with from 1.5 to 8 combining weights (referred to in the following table as "parts") of various basic magnesium compounds. After ob-

serving the consistency of the cold mix it was heated for from 1 to 2.5 hours at 110° C., until reaction appeared to be complete. The consistency of the heated batch was observed after cooling, and the products were then milled to a fine powder under identical conditions. Finally, 0.05 gram of each product was dissolved in 110 ml. of fresh Stoddard solvent and the observed resistivity of the solvent determined in megohms. The results of these experiments are set forth in Table 4 following:

TABLE 4

Reagents Oleic Acid +	Consistency Before Heating	Consistency After Heating	Millability	Megohms
1.5 parts CP MgO Trace H_2SO_4	Fairly Hard	Fairly Hard	Fair	550
2 parts CP MgO 1 part Stearic Acid	Hard	Very Hard	Very Good	210
2 parts CP MgO	do	Hard	Good	50
4 parts CP MgO	Very Hard	Very Hard	Very Good	168
2 parts CP MgO 2 parts $MgCO_3$	Thick Paste	do	do	75
4 parts Calc MgO	Fluid	Hard	Good	70
8 parts Calc MgO	do	Very Hard	Very Good	450
4 parts Activated Brucite	do	Fairly Hard	Fair	2,000
8 parts Activated Brucite	do	Hard	Good	130
4 parts Calc MgO 4 parts Dolomite	do	do	do	110
1 part CP MgO 3 parts Calc MgO	Fairly Hard	do	do	200
4 parts Heavy MgO	do	do	do	330
2 parts Mg $(OH)_2$	do	do	Fair	115

Properties of Oxides	CP MgO	Calc. MgO	Brucite
Particle Size, microns	0.13	0.54	2.0
Surface Area, sq. meters/gram	12.2	3.1	1.8
Per cent through 325 mesh	99.3	53.7	

The consistency of the cold mix is determined by the rapidity of the reaction, which in turn appears to be a factor of the particle size of the oxide. So long as the final product is hard and readily millable it is desirable to select reagents which give a soft or fluid cold mix, thus reducing the labor required for mixing. With the exception of the single test using four equivalents of activated brucite, in which reaction obviously was not complete, all of the products proved fully functional for the purpose of avoiding linting when mixed with filter aid in the manner above described.

We claim as our invention:

1. The method of conditioning a dry cleaning solvent to avoid linting and dusting in the dry cleaning of fabrics which consists in contacting said solvent with a powdered diatomaceous earth filter aid containing from 0.25% to 5.0% by weight of an oleic acid soap of a metal selected from the group consisting of magnesium, calcium, zinc, strontium, cadmium and barium, and thereby causing said solvent to dissolve sufficient of said soap to reduce the resistivity of said solvent per centimetre cube to not over 250,000 megohms.

2. The method of conditioning a dry cleaning solvent to avoid linting and dusting in the dry cleaning of fabrics which comprises: dispersing an alkali-metal oleic acid soap in said solvent, and contacting the solvent containing said soap with a powdered diatomaceous earth filter aid containing from 0.1% to 1.5% by weight of a water-soluble inorganic compound of a metal selected from the group consisting of magnesium, calcium, zinc, strontium, cadmium and barium,

and thereby producing and dissolving in said solvent a sufficient quantity of the oleate of said metal to reduce the resistivity of said solvent per centimetre cube to not over 250,000 megohms.

3. The method of conditioning a dry cleaning solvent to avoid linting and dusting in the dry cleaning of fabrics which comprises: dispersing an alkali-metal soap of oleic acid in said solvent, and contacting the solvent containing said soap with an adsorptive filter aid consisting substantially of diatomaceous earth admixed with an adsorbent solid selected from the group consisting of the natural, acid treated and activated clays and the adsorbent carbons, said mixture further containing from 0.1% to 1.5% by weight of a water-soluble salt and from 0.25% to 5.0% by weight of an oleic acid soap, each of a metal selected from the group consisting of magnesium, calcium, zinc, strontium, cadmium and barium, thereby causing the solvent to dissolve sufficient of last said soap to reduce the resistivity of said solvent per centimetre cube to not over 250,000 meghoms and also providing a continuing supply of last said soap, by reaction between said salt and said alkali-metal soap, to replace the soap withdrawn from said solvent by said adsorptive filter aid.

4. A composition for clarifying by filtration a dry cleaners' solvent containing an alkali-metal soap in dispersion and for simultaneously reducing the electrical resistivity of said solvent to the point at which lint and dusting are avoided in the use thereof, consisting substantially of finely comminuted diatomaceous earth intermixed with at least one solid adsorbent selected from the group consisting of the natural, acid treated and activated clays and the adsorbent carbons, said mixture further containing from 0.25% to 5.0% by weight of an oleic acid soap of a metal and from 0.1% to 1.5% by weight of a water-soluble inorganic compound of a metal intimately dispersed therethrough, said metals being selected from the group consisting of magnesium, calcium, zinc, strontium, cadmium and barium.

5. A method substantially as recited in claim 1 in which said oleic acid soap is magnesium oleate.

6. A method substantially as recited in claim 2, in which said water-soluble compound is a compound of magnesium.

7. A method substantially as recited in claim 3, in which said metal is magnesium.

8. A composition for clarifying dry cleaners' solvent by filtration and for simultaneously reducing the electrical resistivity of said solvent to the point at which linting and dusting are avoided in the use thereof, consisting substantially of finely comminuted diatomaceous earth and a minor proportion, not substantially less than 0.25% nor more than 5.0% by weight, of an oleic acid soap of a heavy metal intimately dispersed therethrough, the metal of said soap being selected from the group consisting of magnesium, calcium, zinc, strontium, barium and cadmium.

9. A composition substantially as recited in claim 8, in which the metal of said soap is magnesium.

10. A composition for clarifying by filtration a dry cleaners' solvent containing an alkali metal oleate in dispersion and for simultaneously reducing the electrical resistivity of said solvent to the point at which linting and dusting are avoided in the use thereof, consisting substantially of finely comminuted diatomaceous earth and a minor proportion, not substantially less than 0.1% nor more than 1.5% by weight, of a water-soluble, inorganic compound of a metal selected from the group consisting of magnesium, calcium, zinc, strontium, barium and cadmium, said compound being intimately dispersed throughout said diatomaceous earth.

11. A composition substantially as recited in claim 10, in which said metal is magnesium.

12. A composition substantially as recited in claim 4, in which said metal is magnesium.

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