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R. RIEGLER

2,330,922

MANUFACTURE OF SULPHONATED DETERGENTS

Filed Dec. 22, 1941

2 Sheets-Sheet 1

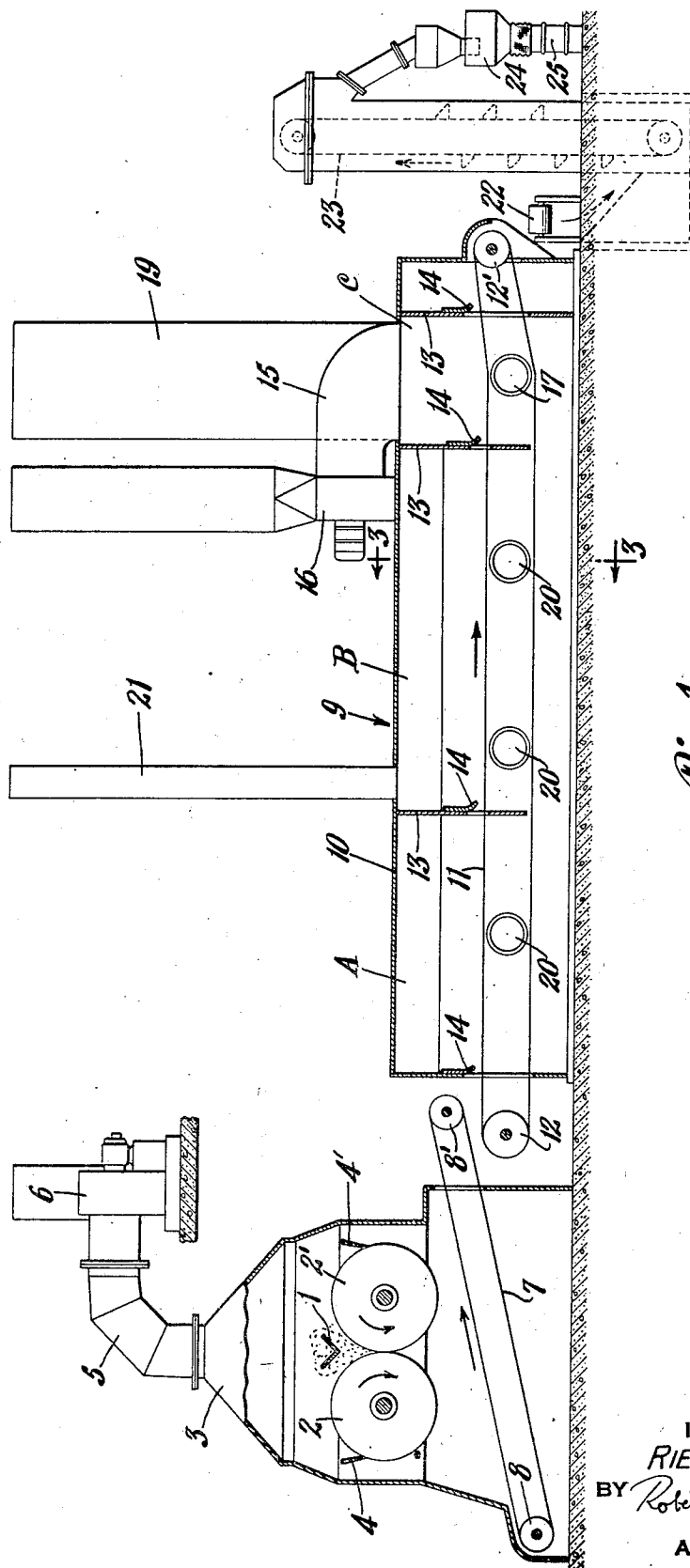


Fig:1

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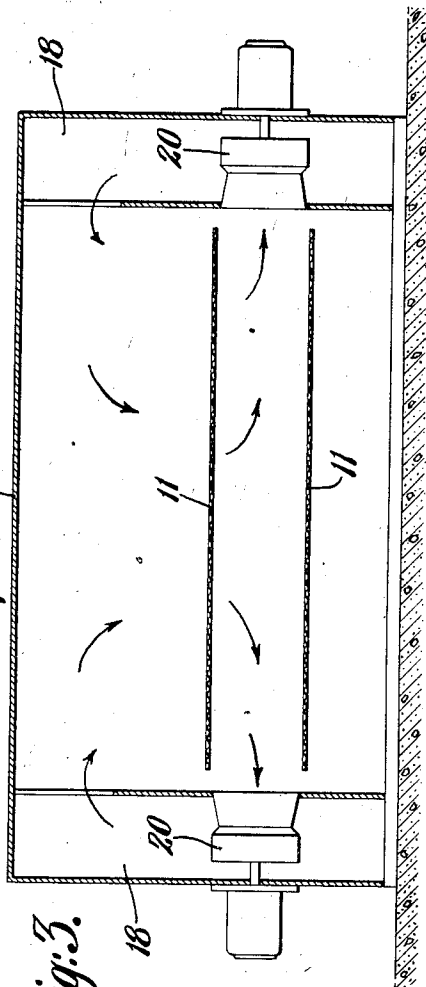
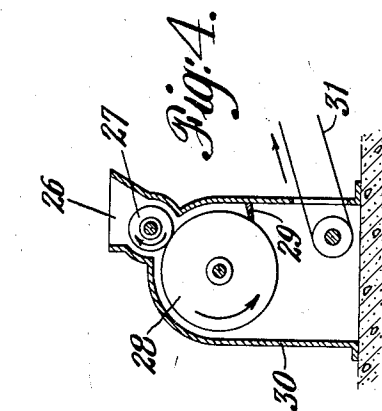
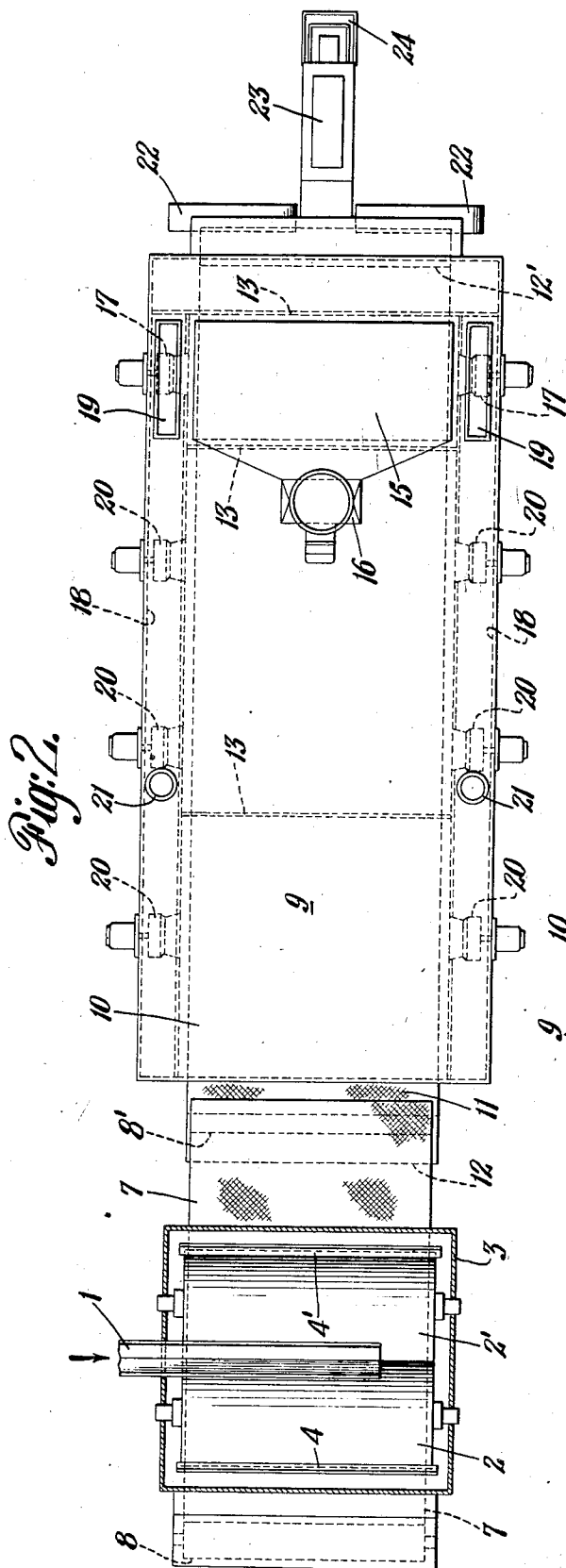
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2 Sheets-Sheet 2



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UNITED STATES PATENT OFFICE

2,330,922

MANUFACTURE OF SULPHONATED
DETERGENTSRiewen Riegler, Buffalo, N. Y., assignor to Allied
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Application December 22, 1941, Serial No. 424,067

9 Claims. (Cl. 18—47.5)

This invention relates to improvements in the manufacture of sulphonated detergents, more particularly to improvements in the manufacture of alkyl aryl sulphonates, the alkyl side chains of which are derived from petroleum distillates.

It has been found that alkyl aryl sulphonates, the alkyl side chains of which contain from 7 to 30 carbon atoms and preferably from 12 to 16 carbon atoms, have surface active properties which make them highly useful as wetting and emulsifying agents and particularly as detergents. A simple and economical method for preparing such compounds involves forming a condensation product of an aromatic compound with a polycomponent non-aromatic hydrocarbon mixture the constituents of which contain the desired number of carbon atoms, sulphonating the mixture of alkyl aryl compounds thus formed, neutralizing the sulphonated product and drying the neutralized solution. Thus, for example, a preferred method for preparing such compounds involves chlorinating a polycomponent non-aromatic hydrocarbon mixture, e. g. a petroleum distillate such as kerosene, condensing the chlorinated mixture with a mononuclear aromatic hydrocarbon such as benzene, sulphonating the mixture of alkyl aryl compounds thus formed, neutralizing and drying. Such compounds may also be prepared by condensing an olefin fraction containing the desired number of carbon atoms with an aromatic hydrocarbon, sulphonating the alkyl aryl compounds formed, neutralizing and drying. Extensive practice of these methods has shown, however, that the alkyl aryl sulphonates produced usually contain appreciable quantities of unsulphonated organic impurities, principally unsulphonated aliphatic hydrocarbons, which impurities impart unpleasant odors to the final compositions and cause the compositions to become discolored and to develop objectionable odors during storage.

As pointed out in the copending application of Lawrence H. Flett Serial No. 403,457, filed July 21, 1941, alkyl aryl sulphonate compositions containing an exceedingly small amount of unsulphonated organic impurities and having excellent detergent characteristics may be prepared by halogenating a polycomponent non-aromatic hydrocarbon mixture containing straight and/or branched chain alkanes having an average carbon content between 7 and 30, preferably between 12 and 16, carbon atoms per molecule in a manner such that between about 10% and about 50% of the mixture remains unhalogen-

ated, condensing this halogenated mixture with an aromatic compound, sulphonating the mixture of alkyl aryl compounds produced, neutralizing the sulphonic acids and drying the neutralized solution by exposing a large surface area of the solution to relatively high temperatures for a relatively short period of time, e. g. by drum or spray drying the solution. It has been found, however, alkyl aryl sulphonates prepared in accordance with the above Flett application possess a faint but nevertheless definite odor which tends to limit their usefulness in highly specialized fields such as the manufacture of dentifrices and the laundering of fine textiles.

Undesirable odors associated with alkyl aryl sulphonate compositions of the type above described may be removed therefrom in accordance with the process described in the copending application of Lawrence H. Flett Serial No. 350,145, filed August 2, 1940, which process involves treating a solution of the alkyl aryl sulphonate with a highly porous solid adsorbent such as activated carbon, separating the treated solution from the adsorbent and recovering the sulphonate. This method of removing the undesirable odor from such compositions has been found to be highly satisfactory. It is evident, however, that this odor removal step inevitably adds to the cost of the final product. Hence, in some cases it would be highly desirable from an economic standpoint to dispense with the adsorbent treatment in the preparation of substantially odorless products.

It is an object of this invention to provide an improved process for the preparation of a detergent product in the form of substantially uniform and odorless flakes or particles.

It is a more specific object of this invention to provide an improved method for preparing substantially odorless alkyl aryl sulphonates derived from polycomponent non-aromatic hydrocarbon mixtures.

I have made the surprising discovery that substantially odorless alkyl aryl sulphonates, the alkyl side chains of which are derived from polycomponent non-aromatic hydrocarbon mixtures having an average carbon content between 7 and 30, preferably between 12 and 16, carbon atoms per molecule, may be prepared in a simple and economical manner by forming an aqueous slurry of the crude alkyl aryl sulphonate composition, said slurry containing between about 20% and about 50% solids (suspended and dissolved), continuously feeding the slurry onto a heated surface such as a rotating drum to form a continuous, coherent film having a maximum thick-

ness of about 0.03 inch, preferably between 0.007 and 0.015 inch, and having a residual volatile content of from about 5% to about 12%, continuously moving the film into contact with hot circulating gas to complete the drying and removal of volatile impurities, and then discharging the dried product, preferably after contact with a cool circulating gas, which product, particularly when cool, automatically breaks up into particles or flakes. A preferred embodiment of my invention involves chlorinating a petroleum distillate of the kerosene type containing hydrocarbons having an average carbon content of 12 to 16 carbon atoms per molecule, condensing the chlorinated petroleum distillate with a mononuclear aromatic compound, stripping the mixture of alkyl aryl compounds thus produced of low-boiling constituents, sulphonating the stripped alkyl aryl compounds, permitting the sulphonation mass to stratify and removing therefrom the top layer of unsulphonated material, neutralizing the sulphonic acid with an aqueous solution of a strong alkali, forming an aqueous slurry of the sulphonate containing between about 20% and about 50% solids, and drying the slurry in the manner above described. I have found it particularly important to produce a continuous, coherent film on the heated surface having a maximum thickness of less than about 0.03 inch, since by operating in this manner a product is obtained which at the completion of the drying disintegrates readily into odorless flakes or particles suitable for all detergent uses. The production of films of the proper thickness depends upon correlation of several factors such as the concentration of the slurry fed to the preliminary drying unit, the rate of feed of the slurry, the type of preliminary drying unit employed, the size of the drying surface and the temperature thereof, and, if a drum drier is employed as is preferred, the rate of rotation of the drum or drums and the point of removal of the partially dried film from the rotating surface; by suitably correlating these variables a film of the desired thickness may readily be produced. Furthermore, for the production of dry products which disintegrate into flakes or particles suitable for detergent use the temperature of the hot circulating gas and the time of contact of the partially dried film with the gas are preferably adjusted so that a product having a residual volatile content of about 0.3% is obtained.

The alkyl aryl sulphonate products obtained in accordance with my invention have been found to be substantially odorless. This is most surprising since it had previously been thought odoriferous impurities would remain in such sulphonates even on prolonged drying and could be removed only by an adsorbent treatment. Hence, these products may be employed in the manufacture of detergents suitable for the most specialized uses without imparting even a faintly discernible odor thereto. Furthermore, my invention permits reduction of the moisture content of the sulphonates to an extremely low figure, e. g. about 0.3% moisture, thereby permitting the production of products which show practically no tendency to lump or cake even upon long standing. From an economical standpoint my invention affords a method of preparing substantially odorless alkyl aryl sulphonate compositions without resorting to the uneconomical step of subjecting the sulphonate to an adsorbent treatment.

The alkyl aryl suphonate compositions which

may be treated in accordance with my invention may be prepared from any polycomponent non-aromatic hydrocarbon mixture, the constituents of which contain the desired number of carbon atoms, in any suitable manner known to the art. The term "polycomponent non-aromatic hydrocarbon mixture" is employed throughout the specification and claims to include hydrocarbon mixtures such as petroleum, petroleum distillates and otherwise refined petroleum fractions containing straight and/or branched chain alkanes, or mixtures of olefins; such mixtures may also contain cycloalkanes and aryl hydrocarbons, the amount of the latter, however, being less than 20% of the mixture. Thus, for example, the polycomponent non-aromatic hydrocarbon mixture employed in accordance with the preferred method of preparation, i. e. the method involving chlorinating a polycomponent non-aromatic hydrocarbon mixture comprising chiefly straight and/or branched chain alkanes and condensing the chlorinated mixture with an aromatic compound, may be any petroleum distillate containing straight and/or branched chain alkanes having an average carbon content of 7 to 30 carbon atoms per molecule, such as, for example, a petroleum distillate, the lower boiling point of which is not below 80° C. at atmospheric pressure and the upper boiling point of which is not more than about 350° C. at 25 mm. absolute pressure; preferably kerosene fractions from Pennsylvania or Mount Pleasant, Michigan, petroleum having an average carbon content of 12 to 16 carbon atoms per molecule and at least 80% thereof boiling within the range of 180°-300° C., and more particularly purified petroleum distillates at least 80% of which boil between 200° and 260° C. are employed. For convenience, alkyl side chains derived from petroleum distillates of the kerosene type are referred to as "keryl side chains." Mixtures of olefins which have approximately the same average carbon content as the petroleum distillates described above may also be employed as the polycomponent non-aromatic hydrocarbon mixture; such olefinic mixtures may be directly condensed with the aromatic compound to produce the desired mixture of alkyl aryl compounds.

In carrying out the preferred method of preparing alkyl aryl sulphonate compositions, a polycomponent non-aromatic hydrocarbon mixture derived from petroleum and containing straight and/or branched chain alkanes having an average carbon content of from 12 to 16 carbon atoms per molecule may be halogenated in any suitable manner. Preferably, a petroleum distillate of the kerosene type, from which any aryl hydrocarbons have been largely removed, is chlorinated so as to produce a chlorinated mixture of keryl mono- and poly-chlorides containing between about 10% and about 50% unchlorinated hydrocarbons. This chlorination may be most readily accomplished by bubbling chlorine gas through the distillate until a mixture containing an amount of organically combined chlorine substantially corresponding with about 75% to about 175%, and not more than 200%, theoretical monochlorination of the petroleum distillate, preferably between about 100% and about 140% theoretical monochlorination, is produced; the term "theoretical monochlorination" means the hydrocarbon mixture contains an amount of organically combined chlorine corresponding to about 1 atom of chlorine per molecule of hydrocarbon. The temperature at which chlorination

is carried out may be between 0° and about 130° C., preferably between 60° and 125° C.; catalysts, such, for example, as phosphorus trichloride, iodine, sunlight, etc., may be employed.

The chlorinated hydrocarbon mixture thus obtained consists of mono- and poly-chlorinated hydrocarbons and unchlorinated hydrocarbons. This mixture is condensed with an aromatic compound such as benzene, toluene, naphthalene, phenol, phenetole, monochlorobenzene, etc., preferably a mononuclear aromatic compound, in the presence of a condensing agent of the Friedel-Crafts type such, for example, as anhydrous aluminum chloride, anhydrous zinc chloride, etc., aluminum chloride being preferably used for the condensation of aromatic hydrocarbons and zinc chloride for phenols. The amount of aromatic compound condensed with the chlorinated hydrocarbon mixture may vary somewhat, but preferably at least about 1.25 mols aromatic compound per mol of the halogenated fraction being condensed is employed and there is little to be gained by using more than 6 mols of the aromatic compound. The amount of condensing agent may vary over a wide range, and may be, for example, as low as 1% or as high as 20% of the weight of the non-aromatic hydrocarbons in alkylating form. The use of aluminum chloride in an amount corresponding to from about 5% to about 20% of the weight of the halogenated mixture, halogenated to an extent corresponding to between 75% and 175% theoretical monohalogenation, as disclosed and claimed in United States Patent 2,223,364 of December 3, 1940, has been found to improve the quality of the alkyl aryl sulphonate produced and, hence, the use of this improved method of condensation in carrying out the present invention is preferred. The temperature at which the condensation is carried out may also vary, but when carrying out the condensation with aluminum chloride as the catalyst in accordance with the preferred method, it should not be above 130° C. and preferably is between 20° and 80° C. At the completion of the condensation, the reaction mass may be permitted to stratify into an upper layer containing the greater part of the crude mixture of the alkyl aryl compounds as well as unchlorinated aliphatic hydrocarbons and a lower layer of darkly colored sludge; the upper layer is withdrawn, and, if desired, subjected to further treatment such as washing metallic compounds therefrom by treatment with water or dilute acid and, as is preferred, stripping the product of low-boiling unchlorinated hydrocarbons and low-boiling uncondensed aromatic hydrocarbons. This alkyl aryl substance may also be subjected to a fractionation to recover a narrow-boiling cut for sulphonation.

The alkyl aryl substance may then be sulphonated by any suitable sulphonation procedure in order to convert it into its corresponding sulphonic acid. Thus, for example, the sulphonating agent employed may be sulphuric acid of various strengths (66° Bé, 100%, 26% oleum, 65% oleum, etc.), chloresulphonic acid or sulphur trioxide; 100% sulphuric acid is preferably used. The temperature at which the sulphonation is carried out may vary between 0° and 140° C., preferably between about 50° and about 75° C.; the amount of sulphuric acid employed in terms of 100% sulphuric acid may range from 0.3 to 5 times or more the weight of the alkyl aryl compound being sulphonated. The extent of sul-

phonation may also vary, depending upon the material being sulphonated, the time of sulphonation and the use to which the sulphonate is to be put. After the sulphonation is complete the mass is preferably, but not necessarily, permitted to settle, whereby a layer of unsulphonated material slowly forms, provided the unsulphonated material is present in large excess and, after it has separated, may be removed, thus withdrawing from the sulphonic acid a large portion of the unsulphonated materials. The sulphonic acid may then be neutralized, preferably with an aqueous solution of a strong alkali, such as sodium hydroxide. However, other neutralizing agents may be used, for example, ammonia, an organic base, alkaline earth hydroxides, or alkali metal carbonates. The aqueous solution thus produced contains dissolved therein the desired sulphonate together with unsulphonated organic impurities equivalent to between about 10% and about 50% of the sulphonate. The concentration of this neutralized solution may then, in accordance with my invention, be adjusted so as to form a slurry containing between about 20% and about 50% solids, preferably about 40% solids, and the slurry then subjected to the drying steps of my invention.

In the accompanying drawings forming a part of this specification and showing for purposes of exemplification preferred forms of the drying apparatus employed in accordance with this invention,

Figure 1 is a side elevation partly in section of a preferred drying apparatus,

Figure 2 is a plan view,

Figure 3 is a cross-sectional view of the apparatus shown in Figure 1, taken along the line 3-3, and

Figure 4 illustrates another type of preliminary drying roll which may be employed.

As shown in Figure 1, a slurry feed trough 1 is disposed above a double drum drier assembly consisting of two steam-heated drums 2, 2' having a diameter of about 5 feet, encased in housing 3 and provided with adjustable notched knives 4, 4' which may be mounted to oscillate slowly in a lateral direction parallel to the face of the drum for a distance slightly greater than the width of a notch during each revolution of the drums; each knife 4, 4' has a plurality of notches of the same width therein, the number of notches being equal to the number of ribbons it is desired to produce. As shown in Figure 1, drums 2, 2' rotate in opposite directions toward one another. Housing 3 has attached thereto conduit 5 provided with fan 6 whereby vapors generated in the housing may be removed. Conveyor belt 7, which may be constructed of canvas, travels beneath drums 2, 2' on rollers 8, 8' to final drier unit 9.

Drier 9 comprises a housing 10 in which is disposed conveyor belt 11, which may be constructed of wire screen of 8 x 8 mesh to permit free passage of air therethrough, traveling horizontally through the drier on rollers 12, 12' and idlers suitably disposed (not shown). Housing 10 is divided into compartments A, B and C by means of insulated partitions 13. The ends of housing 10 and partitions 13 are provided with openings through which belt 11 may pass, these openings being partially closed by means of flexible curtains 14 fixed to housing 10 and partitions 13. Drier 9 is provided with an air intake 15 at the rear end thereof through which fresh atmos-

spheric air is forced by means of fan 16 into compartment C. The air thus introduced into compartment C is sucked through belt 11 by means of fans 17 disposed at the opposite sides of drier unit 9 and between the upper and lower runs of belt 11 as appears from Figures 1 and 2. A portion of the air thus introduced flows into chambers 18 which, as shown in Figures 2 and 3, are positioned at the opposite sides of the drier and in which are disposed steam pipes (not shown). The remainder of the air thus introduced is either directed to exhaust ducts 19 at both sides of drier unit 9 and there vented, or is recirculated through belt 11.

The air flowing into chambers 18 is heated therein by means of the steam pipes and then forced by means of fans 20 from chambers 18 over and through belt 11 in the compartments A and B of drier unit 9. Moisture-laden air is forced by means of fans 20 to exit from these compartments into exhaust ducts 21, through which it is discharged.

It will be understood suitable baffles (not shown) may be provided to give any desired flow of cold air through chamber C and hot air through chambers A and B.

The dried material passes from belt 11 at roller 12' to conveyor belts 22 traveling at right angles to belt 11. The material falling on belts 22 passes to elevator 23, and thence to hopper 24 where it is screened. The screened material falls into container 25.

It is to be understood the drum drier and the conveyor belts are driven by suitable mechanism not shown.

The double drum drier assembly shown in Figures 1 and 2 may be replaced by a single drum drier unit as shown in Figure 4 consisting of slurry feed hopper 26, an adjustable spreader roll 27, a steam-heated drum 28 and notched knife 29 encased in housing 30. Belt 31 disposed beneath knife 29 carries the material to drier unit 9, shown in Figures 1 and 2.

Drier unit 9 may, if desired, be modified by substituting for the single belt conveyor 11 a plurality of belt conveyors arranged in tiers, hot air being passed over the top conveyor belts and cool air over the bottom conveyor belt; the sulphonated detergent to be dried is passed from the drum drier unit to the top conveyor belt and descends through the drier unit to the bottom conveyor belt.

In carrying out the drying in the double drum drier apparatus shown, a slurry of the alkyl aryl sulphonate composition containing between about 20% and about 50% solids is preferably preheated to between about 55° and about 60° C. and then fed to the slurry feed hopper; the rate of feed of the slurry from the hopper to the drying drums may be about 2500 pounds per hour. The speed of rotation and temperature of the drums, the circumferential distance between the knives and the time of contact of the slurry with the drums are correlated so that a continuous, coherent film having a maximum thickness of 0.03 inch, and preferably between about 0.007 and 0.015 inch, is formed by the time the film reaches the notched knives; to accomplish this, when employing a double drum drier, as is preferred, each drum having a diameter of from 32 to 60 inches, the temperature of the drums may be between about 105° and about 160° C. and the speed of rotation between 3 and 15 R. P. M. Lateral movement of the notched knives serves

to remove accumulations of partially dried detergent on the face of the adjacent drum at positions thereon in front of the notches. The ribbons of partially dried detergent removed from the drums by the notched knives fall on conveyor belt 7, are carried to drier 9 and transferred to belt 11. On belt 11 they are first contacted in the chambers A and B with hot air at a temperature between about 120° and about 180° C. for about 3 to 4 minutes, whereby drying and removal of odoriferous impurities are completed; belt 11 then carries the warm dried material into chamber C, wherein it is contacted with cool air at a temperature between about 15° and about 35° C. for about 2 minutes. Thereafter the cooled material falls from belt 11 onto conveyor belts 22 to be carried by elevator 23 to screen hopper 24 and thence to fall as a cooled, flaked, substantially dry product into container 25.

The following examples are illustrative of my invention. Amounts are given in parts by weight and temperatures in degrees centigrade.

Example 1

Part 1.—3070 parts of a Pennsylvania petroleum distillate boiling over the range 186° to 291° and containing 1.3 parts of iodine dissolved therein were chlorinated by passing chlorine gas therethrough at about 55° until the specific gravity of the chlorinated mixture was 0.920 at 24°. The resulting product contained about 25% to 35% unchlorinated hydrocarbons.

Part 2.—1240 parts of a chlorinated kerosene produced as described in part 1 were added to a mixture of 100 parts of anhydrous aluminum chloride and 719 parts of benzene at a temperature between about 30° and about 45°; when all the chlorinated kerosene had been added the mixture was agitated at a temperature of 45° to 50° for one hour, was then permitted to settle, and the upper layer was distilled until the vapors had a temperature of 100° to 105°, the residue was then cooled to 14°, subjected to reduced pressures, and distillation continued until the vapors had a temperature of about 115° at 40 to 45 mm. pressure. 7 parts of flaked caustic soda were then added to the residue, distillation was resumed and a fraction distilling between 115° and 265° at 40 mm. absolute pressure was collected.

Part 3.—The distillate obtained as described in part 2 was washed with 133 parts of 100% sulphuric acid for about forty-five minutes, the mixture then permitted to stand for one-half hour and the acid layer withdrawn. The acid-washed oil was then cooled to about 10° and mixed with about 810 parts of 20% oleum, the temperature being maintained between 12° and 16° during addition of the acid. When all the acid had been added the mixture was agitated for one-half hour at 25°, was then permitted to stand for forty-five minutes, and the lower sulphonated oil layer drowned in 600 parts of ice water. The sulphonic acid thus obtained was neutralized with a strong aqueous sodium hydroxide solution. A solution of sodium keryl benzene sulphonate was thus obtained containing 25.5% solids.

This solution was concentrated so as to produce a slurry containing 40% solids. The slurry was then preheated to a temperature of about 55° and introduced into a feed trough disposed above a double drum drier; the drums of the double drum drier each had a diameter of 32 inches and were heated by means of steam at 20

to 30 pounds pressure and rotated in opposite directions toward one another at about 9 P. M. The slurry was fed into the valley between the drums and the partially dried mass was removed from the drums in the form of $\frac{1}{2}$ inch ribbons by means of knives notched every half inch and so disposed with relation to the drums that the product removed from the drums was in the form of a coherent film 0.007 inch thick and containing about 7% moisture; the notched knives were moved laterally during each revolution of the drums to remove accumulation of partially dried detergent. The ribbons were transferred by means of a conveyor belt into a drier unit, wherein they were contacted with hot air at a temperature of about 140° for about 3 minutes and then with cool air at a temperature of about 18° for about 2 minutes. The resulting product was substantially odorless and contained less than 0.3 moisture.

Example 2.—A solution of sodium keryl benzene sulphonate, prepared as described in Example 1, was concentrated so as to form a slurry containing about 40% solids. This slurry was then preheated to about 55° and fed to a feed trough disposed above a single drum drier having a diameter of 32 inches heated by means of steam at 20 to 30 pounds pressure and rotated at 9 P. M. The slurry was permitted to flow onto the drum, an adjustable spreader roll limiting the thickness of the film passed to the drum to about 0.007 inch. The partially dried product was removed from the drum in the form of $\frac{1}{2}$ inch ribbons by means of a notched knife disposed with relation to the drum so that the product removed was in the form of a coherent film containing 7% moisture; the notched knife was moved laterally during each revolution of the drum to remove accumulation of partially dried detergent. These ribbons were then further dried as described in Example 1 to form a product which was substantially odorless and contained less than 0.3% moisture.

While the above description has dealt with the preparation of substantially odorless alkyl aryl sulphonate compositions derived from polycarbon non-aromatic hydrocarbon mixtures, it is to be understood that the improved process of my invention may be applied to the removal of odoriferous volatile impurities from other sulphonated detergents containing these impurities; the term "sulphonated detergents" is used in the customary sense to include sulphated as well as sulphonated detergents. Thus, for example, sulphonated detergents derived from high molecular weight alcohols, such as lauryl sulphate and lauryl sulphaacetate detergents; sulphonated detergents derived from low molecular weight mineral oils, such as kerosene sulphonates and kerosene sulphates; and alkyl aryl sulphonates the alkyl side chains of which are derived from alcohols such as isopropyl alcohol, butyl alcohol, dodecyl alcohol and cetyl alcohol, e. g., di-isopropyl-sodium sulphonate and mono-butyl diphenyl sodium monosulphonate, may all be dried in accordance with the improved method of my invention.

From the above description it will be evident my invention provides an improved method for preparing substantially odorless sulphonated detergents in a simple and economical manner. Hence, this invention will undoubtedly be of great interest to those engaged in the production of such material.

Since certain changes in carrying out the above process and certain modifications in the products which embody the invention may be made without departing from its scope, it is intended that all matter contained in the above description shall be interpreted as illustrative and not in a limiting sense.

I claim:

1. In the manufacture of sulphonated detergents, the improvement which comprises feeding a slurry of said sulphonated detergent containing between about 20% and about 50% solids onto a heated surface to effect a preliminary drying thereof and to form a continuous coherent film having a maximum thickness of not more than about 0.03 inch and a volatile content between about 5% and about 12%, removing said partially dried film from said heated surface, and contacting it with a hot circulating gas.
2. In the manufacture of alkyl aryl sulphonate compositions, the alkyl side chains of which are derived from polycarbon non-aromatic hydrocarbon mixtures, the improvement which comprises feeding an aqueous slurry of said alkyl aryl sulphonate composition, said slurry containing between about 20% and about 50% solids, onto a heated surface to effect preliminary drying thereof and to form a continuous coherent film having a thickness of not more than about 0.03 inch and a volatile content between about 5% and about 12%, removing said film from said heated surface, and contacting it with a hot circulating gas.
3. In the manufacture of alkyl aryl sulphonate compositions derived from polycarbon non-aromatic hydrocarbon mixtures of petroleum origin, the improvement which comprises feeding a slurry of said alkyl aryl sulphonate composition, said slurry containing between about 20% and about 50% solids, onto a drum drier to effect a preliminary drying thereof and to form a continuous coherent film having a maximum thickness of not more than about 0.03 inch and a volatile content between about 5% and about 12%, removing said film from the drum drier, and contacting it first with a hot circulating gas at a temperature between about 120° and about 180° C. and then with a cool circulating gas at a temperature between about 15° and about 35° C.
4. In the manufacture of alkyl aryl sulphonate compositions, the alkyl side chains of which are derived from a petroleum distillate of the kerosene type, the improvement which comprises feeding an aqueous slurry of a keryl aryl sulphonate composition, said slurry containing between about 20% and about 50% solids, onto a drum drier to effect preliminary drying thereof and to form a continuous coherent film having a maximum thickness of not more than about 0.03 inch and a volatile content between about 5% and about 12%, removing said film from said drum drier, and contacting it with a hot circulating gas at a temperature between about 120° and about 180° C. and then with a cool circulating gas at a temperature between about 15° and about 35° C.
5. In the manufacture of alkyl aryl sulphonate compositions, the alkyl side chains of which are derived from a petroleum distillate of the kerosene type, the improvement which comprises feeding an aqueous slurry of a keryl benzene sulphonate composition, said slurry containing between about 20% and about 50% solids, onto a drum drier to effect preliminary drying thereof and to form a continuous coherent film having

a maximum thickness of not more than about 0.03 inch and a volatile content between about 5% and about 12%, removing said film from said drum drier, and contacting it with a hot circulating gas at a temperature between about 120° and about 180° C. and then with a cool circulating gas at a temperature between about 15° and about 35° C.

6. In the manufacture of sulphonated detergents, the improvement which comprises feeding an aqueous slurry of a lauryl sulphate composition, said slurry containing between about 20% and about 50% solids, onto a drum drier to effect preliminary drying thereof and to form a continuous coherent film having a maximum thickness of not more than about 0.03 inch and a volatile content between about 5% and about 12%, removing said film from said drum drier, and contacting it with a hot circulating gas and then with a cool circulating gas.

7. In the manufacture of sulphonated detergents, the improvement which comprises feeding an aqueous slurry of a lauryl sulfo-acetate composition, said slurry containing between about 20% and about 50% solids, onto a drum drier to effect preliminary drying thereof and to form a continuous coherent film having a maximum thickness of not more than about 0.03 inch and a volatile content between about 5% and about 12%, removing said film from said drum drier, and contacting it with a hot circulating gas and then with a cool circulating gas.

8. In the manufacture of alkyl aryl sulphonate compositions derived from polycomponent non-aromatic hydrocarbon mixtures of petroleum

origin, the average carbon content of which is at least 12 and not more than 16 carbon atoms per molecule, the improvement which comprises feeding a slurry of said alkyl aryl sulphonate composition, said slurry containing between about 20% and about 50% solids, onto a drum drier to effect a preliminary drying thereof and to form a continuous coherent film having a maximum thickness of not more than about 0.03 inch and a volatile content between about 5% and about 12%, removing said film from the drum drier, and contacting it first with a hot circulating gas and then with a cool circulating gas.

9. In the manufacture of alkyl aryl sulphonate compositions, the alkyl side chains of which are derived from a petroleum distillate of the kerosene type, the improvement which comprises feeding an aqueous slurry of a keryl benzene sulphonate composition, said slurry containing between about 20% and about 50% solids, onto a drum drier maintained at a temperature between about 105° and about 160° C. so as to effect preliminary drying thereof and form a continuous coherent film having a maximum thickness of not more than about 0.03 inch and a volatile content between about 5% and about 12%, cutting said film into ribbons, removing said ribbons from the drum drier, and completing the drying by contacting said ribbons with hot circulating air at a temperature between about 120° and about 180° C. and then with cool circulating air at a temperature between about 15° and about 35° C.

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