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DECOLORIZED DETERGENTS AND METHOD OF MANUFACTURE

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This invention relates to the manufacture of substantially color free detergents, and particularly to a process of bleaching color impurities contained in the sulfation and sulfonation acidic reaction mixture and simultaneously neutralizing the acid reaction mixture by treatment with a combined neutralizing and bleaching solution containing an alkali metal chlorite salt.

In the conventional manufacture of sulfated and sulfonated detergents, high molecular weight starting compounds of the character of alcohols, esters, aromatic and olefine hydrocarbons, fatty acid or other aliphatic, aromatic or mixed radical compounds are sulfated, or sulfonated, or both, by treatment with a strong mineral sulfuric acid or an equivalent powerful sulfating or sulfonating derivative thereof, such as chlorosulfonic acid, oleum, sulfur trioxide or the like. Sulfates, sulfonates and mixtures of the two are thereby formed from the high molecular weight organic substances in proportions depending upon various factors including the character of the organic substance, the strength of the sulfating or sulfonating agent, and the conditions of the reaction. As the treatment of the organic compounds with such strong acid compounds is a vigorous one, certain side reaction products, in addition to the sulfation and sulfonation products, are formed probably as the result both of the strong dehydrating action of the sulfation and sulfonation agents and their activity as catalysts for polymerization and condensation reactions. A considerable portion of these side reaction products are impurities constituted as color bodies which discolor and reduce the marketability and the utility for some purposes of the sulfated and sulfonated detergents, which without them would be substantially color free. A large portion of these color bodies are insoluble in pure water water but are solubilized by the action of the detergent in aqueous solution so that in effect they are water soluble in the environment of such sulfated and sulfonated detergents.

In appearance mono sulfuric acid ester, for example, as produced directly by reaction of strong sulfating acid upon lauryl alcohol, is a strongly acid dark brown, oily, fuming liquid. After neutralization, as with an aqueous solution of sodium hydroxide, the product, if used directly as a liquid detergent, has a yellow-brown to dark brown color; if it is subjected to a drying process the resulting powder or flake is unsightly and dirty gray or brown in color.

Synthetic detergents of the sulfated and sulfonated types are widely sold for a variety of uses in which such discoloration is most undesirable. In the household market, for laundering, dish washing, and the like, acceptability of the detergent in competition with natural soap products is enhanced by a light or colorless shade, which the consumer has come to associate with the concept of purity. In the cosmetic field, shampoos of sulfated or sulfonated products, merchandised either as clear liquids intended to be colorless, or as colored clear liquids or as opaque liquids or creams with or without artificial coloring, require substantial freedom from color contaminants for sales appeal. In the medical and pharmaceutical fields, where such detergents are obtaining wide favor, purity in appearance as well as in composition is an important attribute.

Prior efforts to solve the problem of discoloration have not been successful. It is not possible, so far as we are aware, to completely prevent color formation during the sulfation and sulfonation processes. Furthermore, adequate extraction of the color bodies from the detergent by the use of solvents is not economical or really effective because of the emulsifying action of the detergent on the solvent, among other reasons; extraction by adsorption, such as with activated carbon, encounters the problems of extraction of the detergent itself and of inadequate removal of color bodies. Nor has bleaching been effective prior to the present invention. No bleaching agent active in the neutralized product will remove enough of the color to be practicably adequate; prior attempts to treat the sulfuric acid ester or the sulfonic acid with bleaches of various kinds have made the problems worse because the generation of intense heat coupled with the release of oxygen or the catalytic effect of the oxidizing agent in the presence of the strong acid results in substantial hydrolysis of sulfuric acid esters and polymerization and condensation of organic bodies which appears to induce the formation of the very side reaction color body products which the bleaching treatment seeks to remedy. Thus prior to this invention there has been no method available to successfully remove color bodies from detergent substances of the sulfate or sulfonate type in an economical and practical manner.

In accordance with our invention, a commercial, acid-activatable bleaching composition, namely, an alkali metal chlorite, is employed to remove the color from the color bodies by a novel treatment including specifically the steps of adding the alkali metal chlorite to the neutralizing alkaline solution prior to the admixture of the alkaline solution with the mono sulfuric acid ester and/or sulfonic acid reaction mixture (hereinafter referred to as "acid oil") and thereafter mixing the acid oil and alkali as will be described, to effect the neutralization. The resulting product is a substantially color free detergent with a marked absence of the color contamination which would characterize the same detergent made without the addition of the alkali metal chlorite to the alkaline neutralizating solution. Without these specific steps the desired bleaching is not accomplished. For example, if the bleaching were attempted by treatment of the neutralized detergent with the alkali metal chlorite, ineffective results would be obtained, as much of the color would remain despite the treatment. This is so even when the detergent solution is made acid—frequently for some unaccountable reason this produces more, rather than less, color in the solution and, in addition, causes extensive hydrolysis and breakdown of common sulfated detergents. Any attempt to remove the color contaminants from the acid oil itself by the addition to it of dry alkali metal chlorite would present a serious fire, if not explosion, hazard from the violent oxidation reaction, and furthermore, that reaction would result in the formation of still more color contaminants. It is only by the addition of alkali metal chlorite to the alkali neutralizing solution prior to neutralizing the acid oil that an adequately color-free detergent is attained.

It is known that alkali metal chlorites, of which sodium chlorite is typical, have effectiveness as bleaching agents both when used in acid solutions and to a lesser degree when activated by agents such as formaldehyde, persulfate, peroxide, etc. in neutral solutions and even in slightly alkaline solutions up to a pH of about 7.5. However, regardless of activating agents, sodium chlorite

is comparatively ineffective in alkaline solution of a pH of about 8 and is substantially inert if the solution is strongly alkaline. Nevertheless, we have found that it may effectively be employed to remove color in the manufacture of these detergents only by adding it directly to the strongly alkaline neutralizing solution having a pH well above 8 and which may have a pH of the order of about 14.

The following examples are typical of the practice under the present invention.

EXAMPLE I

To 1,000 grams of a fatty alcohol consisting of 60 percent lauryl alcohol, 25 percent myristyl alcohol, the balance consisting of a mixture of small amounts of decyl, cetyl and stearyl alcohols, was slowly added over a period of two hours, 560 grams of chlorosulphonic acid. The reaction was carried out in a glass flask suspended in a cold water bath, the flask being equipped with a stirrer, a dropping funnel and duct to carry away evolved gases. The temperature was maintained at about 30° C. The resulting monosulphuric acid ester of the fatty alcohols was divided into five 200-gram portions and neutralized as follows:

A. *Procedure according to present method*

A 200 gram portion was added slowly over about a ten minute period to a mixture of 34 grams of sodium hydroxide and one gram of technical grade sodium chlorite in 440 mls. of water. The pH remained above twelve until about 175 grams of acid ester had been added at which time it began to drop slowly and when the addition was completed the pH was slightly alkaline, about 8, and the temperature had risen to 55° C. A few ml. of concentrated hydrochloric acid were added to lower the pH to 6.0. Ten mls. of 22 percent sodium sulfite were then added to destroy the excess oxidizing agent and the pH of the solution was adjusted to 7 with sodium hydroxide.

B. *Comparative procedure of first neutralizing and then bleaching*

A second 200 gram portion of the acid oil was neutralized as in "A" without sodium chlorite in the alkali. When all of the sulphuric acid ester had been added, the pH was 7.6 and the temperature 55° C. After pH adjustment to 6.0, one gram of technical grade sodium chlorite was added and the solution was agitated for fifteen minutes after which 10 mls. of 22 percent sodium sulfite were added and the pH adjusted to 7.

C. *Comparative procedure of first neutralizing, then bleaching in a substantially acid medium*

A third 200 gram portion was neutralized in the same manner as in "B" except that the pH was adjusted to 4.0 before addition of the sodium chlorite. Ten mls. of 22 percent sodium sulfite were added to destroy the excess oxidizing agent after fifteen minutes of agitation and the pH was raised to 7.

D. *Comparative procedure of first neutralizing and then bleaching with bleaching salt and "activator"*

A fourth 200 gram portion was neutralized in the same manner as "B" except that 0.7 gram of 37 percent formaldehyde was added with the sodium chlorite.

E. *Control—neutralizing without bleaching*

A fifth 200 gram portion was neutralized in the same manner as "A," but without the use of sodium chlorite, the final pH being adjusted to 7.

Samples of each of the neutralized products were diluted with an equal amount of a 50 percent mixture of water and 99 percent isopropanol. The light absorbency of each solution was then determined at 4500 A. in a 1 cm. cell on a model #11 Coleman Junior spectrophotometer, with the following results:

Comparative results

Bleaching procedure:	Optical density
A	0.025
B	0.043
C	0.038
D	0.040
E	0.040

The portion treated in procedure A was by far the least colored of the five. Portion C, in which bleaching was done after neutralization, had 52 percent more color than A. In portions B and D, no improvement in color was obtained over the control sample portion E. The detergent prepared by procedure A having an optical density of .025 was substantially clear, transparent and colorless.

EXAMPLE II

To 1,000 grams of an alkyl benzene, obtained by alkylation of benzene with tetrapropylene, was added 1380 grams of 22 percent oleum over a period of 3½ hours, while the temperature was maintained between 10° and 15° C. The mixture was agitated for two hours before the addition of 260 grams of water, after which heat was applied and the mixture was agitated for one hour at 54° to 60° C. After standing overnight the bottom layer, consisting of dilute sulphuric acid along with a considerable amount of impurities, was discarded. The top layer consisting largely of dodecyl benzene sulphonic acid was divided into five 200-gram portions for further treatment:

A. *Procedure according to present method*

1.13 grams of analytical grade sodium chlorite was dissolved in an alkaline solution of 40 grams of sodium hydroxide in 530 ml. of water. The 200 gram portion of acid was slowly added thereto, the pH of the neutralizing solution remaining strongly alkaline until almost all of sulphonic acid had been added. The pH then dropped gradually to about pH 9 as the last of the sulphonic acid was added. A few ml. of concentrated hydrochloric acid was then added sufficient to bring the pH to 6.0, and then 10 ml. of 22 percent sodium sulfite was added to the mixture to destroy the excess oxidizing agent. A few drops of sodium hydroxide were then added to make the batch mildly alkaline again to a pH of 7.5.

B. *Comparative procedure of first neutralizing and bleaching and continuing the bleaching after neutralizing*

A second 200 gram neutralization was carried out in the same manner as with A except that the batch was agitated for one-half hour at pH 6.0 before adding the sodium sulfite solution.

C. *Comparative procedure of first neutralizing and then bleaching*

A third 200 gram sample neutralization was carried out by adding the acid oil slowly to a solution of 40 grams of NaOH in 530 ml. of water. The pH of the bath was adjusted to 6.0 and then 1.13 grams of sodium chlorite analytical grade was dissolved in the bath. The batch was agitated for one-half hour at pH 6.0 after which 10 mls. of 22 percent sodium sulfite solution was added and the batch made slightly alkaline to a pH of 7.5.

D. *Comparative procedure of first neutralizing and then bleaching in a substantially acid bath*

A fourth 200 gram batch was neutralized in the same manner as C except that the pH of the sodium alkyl aryl sulfonate solution was adjusted to 4.0 before addition of the sodium chlorite and the subsequent one-half hour of agitation.

E. *Control—neutralizing without bleaching*

A fifth 200 gram portion was run as a control sample

and the neutralizing was done in a similar solution of 40 grams of NaOH in 530 ml. of water without bleaching.

Comparative results

Color readings were then obtained on the resulting detergent solutions:

Sample	Optical Density	Percent Color Increase Over A
A	0.071	0
B	0.074	4.2
C	0.106	49
D	0.132	86
E	0.181	155

The results obtained illustrate not only the superiority of bleaching simultaneously with neutralizing as in sample A, but that the excess alkali of the neutralizing bath after neutralizing renders any excess bleaching salt in the bath inactive, i. e., the localized acidity from the acid oil effects the bleaching. Where the bath containing excess bleaching salt is rendered slightly acid, the continuing bleach causes more color to be formed as illustrated by B. The effect, however, of neutralizing first and then bleaching results in substantial discoloration as shown in C and D and, it will be apparent from D that in this case the discoloration is greater with greater acidity.

EXAMPLE III

To 1500 grams of technical glyceryl monolaurate was added 1050 grams of 98% H_2SO_4 over a period of two hours, while the temperature gradually rose from 38° to 65° C. The reaction mixture was then agitated for an additional 20 minutes and then three 300 gram portions were neutralized in the following procedures:

A. *Procedure according to present method*

2.4 grams of technical grade sodium chlorite was added to an alkaline solution of 50 grams of sodium hydroxide in 310 ml. of water. The monosulphuric acid ester of glyceryl monolaurate 300 gram sample was slowly added to the neutralizing and bleaching bath over a period of 10 minutes. After addition of the acid ester the pH of the mixture was 6.0. 10 mls. of 22 percent sodium sulfite were then added to destroy excess oxidizing agent.

B. *Comparative procedure of first neutralizing and then bleaching*

The second 300 gram portion of sulphuric acid ester was neutralized in an alkali neutralizing bath of 50 grams of NaOH in 310 mls. of water, containing no sodium chlorite. To the neutralized product adjusted to a pH of 6.0 was added 2.4 grams of sodium chlorite and after 15 minutes agitation 20 mls. of 22 percent sodium sulfite was added to destroy excess oxidizing agent.

C. *Control—neutralizing without bleaching*

A third 300 gram portion of sulphuric acid ester was neutralized without bleaching by adding to a bath of 50 grams of NaOH in 310 mls. of water.

Comparative results

Color readings were then obtained as follows:

Sample	Optical Density	Percent Color Increase Over A
A	0.145	0
B	0.456	215
C	0.559	285

In the practice of our invention it is highly desirable that the acid oil be added slowly with agitation to the solution of alkali and chlorite. We believe that in so doing, the sodium chlorite, although homogeneously dissolved in the strong alkali solution, is acid-activated to liberate chlorine dioxide and to effect the bleaching of the color contaminants in the acid oil. It is our theory that as the slow admixture takes place, the acid oil is broken up into small droplets or globules by the slow addition and/or agitation; that around each of these droplets of acid oil there exists a thin layer of acid solution in which the sodium chlorite, added with the alkali, performs its bleaching function; and that the acid chlorite bleaching process is performed in this apparently strongly alkaline environment of the caustic bath only by localized acidification of solution containing both sodium chlorite and the color contaminants to be bleached. Until the acid oil is added to the alkaline neutralizing solution, the sodium chlorite is maintained stably alkaline by the alkali present. By the addition of the acid oil slowly to the larger body of the alkaline solution containing the acid-activatable sodium chlorite, the rate of heat evolution of the strongly exothermic reaction between the sodium chlorite and the acid is minimized apparently by localizing the bleaching action to the thin layer of acidic solution around the individual acid oil droplets.

The temperature of the entire quantity of acid oil remains well below that at which color-body-producing reactions take place.

The bleaching agent, alkali metal chlorite, is unique. In its alkaline solution environment it is stable and has no continuing bleaching effect on the detergent after neutralization in the alkaline environment; the entire bleaching action takes place at the point of neutralizing admixture and ceases when the product becomes alkaline. Moreover, this bleaching agent operates, when acidized, to release chlorine dioxide which is a gas, leaving no contaminating residue except the alkali metal ion in the detergent. The commonest of these alkali metal chlorites is the sodium salt, but the potassium or lithium chlorites work as well. The quantity of chlorine dioxide liberated by these alkali metal chlorites, upon being activated with acid, varies directly with the concentration of the chlorite and the temperature of the bath and inversely with the pH. A small quantity of alkali metal chlorite bleaching composition will suffice, within the range of .05% to 2% or 3% and preferably about 0.1% to 1.0% of the neutralizing solution.

The alkaline component of the neutralizing solution may be of any soluble alkaline material of sufficiently alkaline character to react rapidly with the sulfated and sulfonated acid oil. Any water soluble strongly alkaline substance may be used such, for example, as the water soluble oxides, hydroxides, and carbonates of any of the alkali metals, alkali earth metals, or of ammonia. Thus, as alkali metal, sodium, potassium, or lithium may be used as their oxides, hydroxides, or carbonates; any alkali earth metal, such as calcium, barium, or strontium may be used as their water soluble hydroxides; or ammonium hydroxide.

Our invention may be employed to bleach impurities formed by strong acid treatment for sulfation or sulfonation of various organic substances followed by neutralization to form detergents. This type of detergent is well known in the art and the following examples are merely illustrative, viz., alkyl sulfates and sulfonates formed by treatment of higher fatty alcohols usually having from 8 to 20 carbon atoms. Such fatty alcohols are usually obtained by reduction of commercial fatty or fixed oils of animal, vegetable, fish or other marine origin of non-drying, semi-drying, or drying character. Typical commercial oils or coconut oil, palm oil, olive oil, tallow, stearin, castor oil. wool fat, lard oil, linseed oil, lanoline, etc. Typical examples are the sulfation and

sulfonation products of lauryl alcohol, cetyl alcohol, stearyl alcohol, ricinoleyl alcohol, linoleyl alcohol and oleyl alcohol. Similar products are obtained by the sulfation of so-called synthetic alcohols such as those prepared by catalytically combining a mixture of hydrogen and carbon monoxide, combining hydrogen and carbon monoxide with an olefinic hydrocarbon, by oxidation of hydrocarbons, by hydration of olefines, etc. Detergents of this type formed by reacting these alcohols with strong sulfating acids predominate in the sulfate form with lesser quantities of sulfonate, depending upon the degree of unsaturation of the alcohol.

A second type of detergent that may be bleached in its free acid form by the present method is that resulting from treatment of select heavy petroleum oil fractions with strong sulfuric acid or equivalent sulfonating agents. The mineral oil treated is usually a heavy oil fraction and the sulfonation products when neutralized are the well known mahogany sulfonates characterized by their dark color.

A third type of common sulfonated detergent compound is that formed by sulfonating aromatic hydrocarbons of the type of benzene or naphthalene usually containing 1 or more alkyl radicals attached to the ring usually of about 4 to 8 carbon atoms or longer chain length. The hydrocarbon may be directly sulfonated with the sulfonic acid group attaching to the ring or to the chain, or a sulfate group can be added to compounds of similar structure further containing a hydroxy radical.

Still another type of detergent which can be bleached by our process is formed by the sulfation or sulfonation of mono or diglycerides of fatty acids or other fatty esters which contain an unreacted hydroxyl group.

Other sulfation and sulfonation products, to form detergents by treating a high molecular weight compound with strong sulfuric acid or its equivalent in the art, may be treated to remove color imparting bodies according to this invention.

The following acid sulfate or sulfonic ester compounds are additional typical examples of compounds which may be substantially improved in color by the treatment herein described: the acid sulfation product of cetyl alcohol; the commercial mixture of alcohols obtained by reduction of the methyl esters of the fatty acids of palm oil and coconut oil; the acid sulfation oil of wool fat alcohols; acid 1,5 diisopropyl beta naphthalene sulfonate, dodecyl benzene sulfonic acid; the acid sulfate of di oleyl glyceride, and mono lauryl glyceride acid sulfate.

Various modifications will occur to those skilled in the art. The alkaline bath may be maintained quiescent while adding the acid oil thereto for neutralization dropwise or in thin streams at one or more points of the bath. Alternatively, since the neutralizing and bleaching effect is local at the point of entry of the acid oil into the alkaline bleaching solution, agitation may be applied to the alkaline bath while the acid oil is being added. Where very large bodies of acid oil are to be neutralized either the acid oil or the bath may be substantially cooled during the neutralization or both reagents may be cooled before interaction.

In another modification for continuous neutralization, the acid oil and the alkaline solution may be intermixed in a continuous or semi-continuous operation by conducting a thin stream of the acid oil through a pipe or duct into a relatively larger stream of the alkaline bleaching solution also passing through a pipe or duct.

Substantial variation is possible in the degree of concentration of the alkali, it being usually desirable to maintain the concentration of the alkali less than 50% preferably about 5 to 10%, or even less, to avoid overheating by generation of an excessive quantity of heat. The same result may be effected by application of continuous refrigeration during the reaction to allow wider variation in concentration of the alkali.

We claim:

1. The method of manufacturing bleached sulfated and sulfonated detergent substances comprising neutralizing the free acid form of the sulfated and sulfonated detergents by slowly adding with agitation said acid detergents to a sufficient quantity of a strongly alkaline neutralizing solution, having a pH well above 8, containing an alkali metal chlorite salt dissolved therein, to continuously maintain the reaction mixture alkaline at a pH above about 8 during said addition, until the addition of the acid material to the alkaline neutralizing solution is substantially completed.

2. The method of manufacturing bleached sulfated and sulfonated detergent substances comprising neutralizing the free acid form of the sulfated and sulfonated detergents by slowly adding with agitation said acid detergents to a sufficient quantity of a strongly alkaline neutralizing solution, having a pH well above 8, containing sodium chlorite dissolved therein, to continuously maintain the reaction mixture alkaline at a pH above about 8 during said addition, until the addition of the acid material to the alkaline neutralizing solution is substantially completed.

3. The method of bleaching a mono sulfuric acid ester of a glyceride selected from the group consisting of mono and di glycerides of higher fatty acids comprising neutralizing the acid sulfated glyceride by slowly adding with agitation said acid ester to a sufficient quantity of a strongly alkaline neutralizing solution, having a pH well above 8, containing an alkali metal chlorite salt dissolved therein, to continuously maintain the reaction mixture alkaline at a pH above about 8 during said addition, until the addition of the acid material to the alkaline neutralizing solution is substantially completed.

4. The method of manufacturing a bleached alkyl aryl sulfonate detergent comprising neutralizing the free acid form of the sulfonated detergent by slowly adding with agitation said acid detergent to a sufficient quantity of a strongly alkaline neutralizing solution, having a pH well above 8, containing an alkali metal chlorite salt dissolved therein, to continuously maintain the reaction mixture alkaline at a pH above about 8 during said addition, until the addition of the acid material to the alkaline neutralizing solution is substantially completed.

5. The method of producing a bleached detergent of the mono sulfuric acid ester of a higher fatty alcohol having more than 8 carbon atoms comprising neutralizing the mono acid ester by slowly adding with agitation said acid ester to a sufficient quantity of a strongly alkaline neutralizing solution, having a pH well above 8, containing an alkali metal chlorite salt dissolved therein, to continuously maintain the reaction mixture alkaline at a pH above about 8 during said addition, until the addition of the acid material to the alkaline neutralizing solution is substantially completed.

6. The method of bleaching the mono sulfuric acid ester detergent formed by sulfating the higher fatty alcohols obtained by reduction of a fatty acid substance obtained from an oil selected from the group consisting of palm oil and coconut oil comprising neutralizing the acid sulfated alcohol by slowly adding with agitation said acid sulfated alcohol to a sufficient quantity of a strongly alkaline neutralizing solution, having a pH well above 8, containing an alkali metal chlorite salt dissolved therein, to continuously maintain the reaction mixture alkaline at a pH above about 8 during said addition, until the addition of the acid material to the alkaline neutralizing solution is substantially completed.

7. The method of bleaching the mono sulfuric acid ester formed by sulfating a commercial mixture of higher fatty alcohols predominating in lauryl alcohol comprising neutralizing the acid sulfated alcohol by slowly adding with agitation said acid sulfated alcohol to a sufficient quantity of a strongly alkaline neutralizing solution, having a pH well above 8, containing an alkali metal chlorite

salt dissolved therein, to continuously maintain the reaction mixture alkaline at a pH above about 8 during said addition, until the addition of the acid material to the alkaline neutralizing solution is substantially completed.

8. The method of bleaching and neutralizing an organic sulfonic acid comprising neutralizing the free acid form of the organic sulfonic acid by slowly adding said organic sulfonic acid with agitation to a sufficient quantity of the strongly alkaline neutralizing solution, having a pH well above 8, containing an alkali metal chlorite salt dissolved therein, to continuously maintain the reaction mixture alkaline at a pH above about 8 during said addition, until the addition of the acid material to the alkaline neutralizing solution is substantially completed.

9. The method of bleaching and neutralizing an acid sulfuric acid ester of an organic compound comprising neutralizing the free acid form of said sulfuric ester by slowly adding with agitation said acid sulfuric acid ester to a sufficient quantity of a strongly alkaline neutralizing solution, having a pH well above 8, containing an alkali metal chlorite salt dissolved therein, to continuously maintain the reaction mixture alkaline at a pH above about 8 during said addition, until the addition of the acid material to the alkaline neutralizing solution is substantially completed.

10. The method of manufacturing bleached sulfated and sulfonated detergent substances containing color imparting impurities comprising slowly adding with agitation said acid detergent to a sufficient quantity of a strongly alkaline neutralizing solution, having a pH well above 8, containing an alkali metal chlorite salt dissolved therein, to continuously maintain the reaction mixture alkaline at a pH above about 8 during said addition, until the addition of the acid material to the alkaline neutralizing solution is substantially completed.

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