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METHOD OF DYEING HAIR

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1 Claim. (Cl. 167—88)

This invention relates to improvements in the dyeing of hair and more particularly to the application of non-toxic preparations to dyeing and tinting of hair, which preparations produce high rubbing fastness of the dye on the hair and render useful for hair dyeing purposes a large class of non-toxic dyes heretofore unavailable for these purposes. This invention is an improvement upon the invention of my copending application Serial No. 195,539, filed March 12, 1938, which issued July 23, 1940, as Patent No. 2,208,594, wherein is disclosed a process of dyeing human hair which comprises the application of a pre-treatment material to the hair after which the dye is applied thereto.

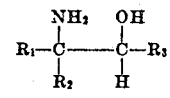
I have now discovered new pretreatment materials the use of which renders much more satisfactory the results obtained from applying non-toxic dyes to human hair. These pretreatment materials comprise salts of cation active materials which themselves possess a desired degree of cation activity. More specifically these materials comprise salts of reaction products of higher fatty acids with nitrogen containing lower aliphatic or alicyclic compounds having at least two basic reacting groups.

Considerable advantages over prior art hair dyeing processes and over the processes of my above mentioned copending application are effected by the use of these new fixative materials in that non-toxic dyes may be applied to the hair and are considerably faster to rubbing than dyeings obtained in heretofore used processes and further in that the fixative salts themselves have a beneficial effect on the hair and, being completely soluble in water in the concentrations used, are easily prepared for application and easily applied.

The fixative salts of my invention may be prepared by reacting higher fatty acids with the nitrogen containing compounds at temperatures sufficient to insure that true condensations take place as evidenced by the removal of water or alcohol, and then by treating this reaction mixture with the proper acid to produce the desired salt. As starting materials to synthesize the cation active fixative salts I may use acids of predominantly fatty character having at least 6 carbon atoms. These acids may be condensed with amino compounds of predominantly basic character having at least 2 basic reacting groups such as amino or hydroxyl groups and preferably of lower molecular weight than the fatty portion of the compound. The condensation product thus obtained may be neutralized with any convenient acid.

As examples of fatty acids which may be used I may mention caproic, caprylic, capric, lauric, myristic, palmitic, cetic, oleic, stearic, ricinoleic, linoleic, arachidic, behenic, lignoceric, palmitoleic, linolenic, eleostearic, clupandonic acids and mixtures of these acids and others such as cocoanut fatty acids which contain substantial quantities of lauric, myristic, palmitic and oleic acids. These acids obtained from cotton seed, corn, peanut, sun flower and similar vegetable sources may also be used.

Some of the amino compounds which may be used according to this invention may be represented by the following general formula:



in which R_1, R_2, and R_3 may be H, alkyl or aklylol and the sum of the carbon atoms in R_1 and R_2 is at least one. Specific examples of such compounds are 2-amino 2-methyl 1,3 propanediol and the higher substituted homologues of the same compound such as the 2-amino 2-ethyl 1,3 propanediol and the propyl and isopropyl compound, tris (hydroxy methyl) amino methane, 2-amino 2-methyl 1-propanol and the ethyl propyl, and iso-propanol substituted compounds, 2-amino 1 propanol, iso-propanol and butanol, 2-amino 1,3 propanediol, 3-amino 3-methyl 2-butanol, 4-amino 5-methyl 3-hexanol, 5-amino 4-octanol. It will be observed that these compounds are alcohols containing primary amino groups. Of the polyamines I have found preferable the aliphatic polyamines such as ethylene, propylene, butylene, amylene polyamines and their alkylated derivatives, as for example the dimethyl and diethyl derivatives. The diamines, triamines, tetramines and pentamines of this series have been found to be useful; I have found particularly useful diethylene triamine and triethylene tetramine. I may also use such nitrogenous compounds as pyridine, ethanol amines including the mono-, di- and triethanol amines and various substituted derivatives of these compounds.

Some examples of convenient acids which may be used, in salt formation are formic, acetic, lactic, tartaric, butyric, phthalic, maleic, malic, sulfuric, hydrochloric, hydrobromic, succinic, propionic, phosphoric and others. Of these acids I have found especially useful acetic and hydrochloric acids.

The process of preparing these pretreatment materials includes the steps of reacting the amino compound with the fatty acid at temperatures at which the condensation reaction will run at a reasonable speed and applying directly to the reaction mixture on completion of the reaction, a suitable amount of acid to cause the pH of the mixture to be reduced to 6 to 6.5. As examples of such reactions I offer the following, though it may be understood that these exam-

ples are not meant to limit the scope of the invention but rather merely to illustrate one embodiment thereof.

Example 1.—In a reaction kettle are charged 210 lbs. of cocoanut fatty acids and 109 lbs. of 2-amino 2-methyl 1-propanol. The reaction mixture is kept at a temperature of 175° C. for about six hours, at which time the reaction is complete. Glacial acetic acid is now added to the reaction mixture until a test shows clear solubility in water at a pH of 6 to 6.3. This acetate product is soluble in water and is used in from 1% to 3% strength in water solution for direct application to the hair. With less effective results but with some useful properties obtaining in the reaction product may be substituted any of the fatty acids mentioned above in this example.

Example 2.—141 lbs. of oleic acid and 58 lbs. of 2-amino 2-methyl 1,3 propanediol are heated to 200° C. in a distilling kettle. The temperature is allowed to go gradually to 215° C. over a space of an hour at which time the condensation is complete. Glacial acetic acid is now added until the product has a pH in water solution of 6 to 6.5. A 1% to 3% solution of this product may be applied to the hair as a fixative in hair dyeing.

Example 3.—210 lbs. of cocoanut fatty acids and 152 lbs. of triethylene tetramine are charged into a still and the temperature is raised to 175° C. and then gradually over a space of about 4½ hours to 185° C. At this time the distillate amounts to 19 lbs. and the acid number of the mass has reduced to about 3. The mass is cooled to a temperature of about 70° C. and glacial acetic acid is added, about 43% of the weight of the mass, roughly 145 to 150 lbs. being necessary to reduce the pH to the desired range about 6 to 6.5. A 1% to 3% solution of this acetate in water is used as a fixative in hair dyeing. A molecularly equivalent amount of hydrochloric or other acid or acid salt may be used in place of acetic acid in these examples.

Example 4.—142 lbs. of stearic acid is mixed with 80 lbs. of triethylene tetramine and heated in a still to 180° C. The temperature is gradually raised over a space of 4½ hours to 245° C. at which time the distillate has amounted to 12 lbs. and the acid number of the mass in the kettle has dropped to 1. This product, a crumbly nearly white wax, is now treated with glacial acetic acid until a test shows clear solubility in water at a pH of 6 to 6.3. A 1% to 3% solution of this acetate is used as a fixative in dyeing hair.

Example 5.—210 lbs. of cocoanut fatty acids is mixed with 113 lbs. diethylene triamine and heated in a still. The temperature is raised to 150° C. then gradually to 190° C. taking 7½ hours. The distillate amounts to 17 lbs. The acid number of the mass in the kettle is then 1.5 and the condensation is finished. The product is nearly white soft wax. The acetate is prepared as in the above examples. A 1 to 3% solution in water is used as a fixative for the dye on the hair.

In some cases it has also been found desirable, as for example where the basic compound selected has more than 2 basic reacting groups, to cause the salt formation to run only partially to completion. Such compounds are often characterized by higher solubility in water which renders their use in professional beauty shops or in the home a matter of great convenience and ease. Furthermore it has been found that a desired color may often be more conveniently obtained with the use of such compounds and the rubbing fastness of the dye on the hair be somewhat enhanced approximately in ratio to the extent to which the salt formation has taken place.

The method of procedure in dyeing hair is as follows: A 2% aqueous solution of the acetate of the condensation product of cocoanut fatty acid with 2-amino 2-methyl 1-propanol is applied to the hair at room temperature. This solution is shampooed through the hair and allowed to remain for 15 minutes. The hair is then rinsed once and a 1 to 3% solution of naphthol blue-black thickened with gum tragacanth is applied and allowed to remain for twenty minutes. The hair may then be rinsed with water until no color is seen in the wash water, whereupon the process is complete and the hair is dyed a solid black color.

Practically any desired color may be applied to the hair and any desired non-toxic dye may be used. By employing such dyes I have been able to produce a complete range of colors which are natural in tint and being non-toxic are non-injurious to the hair and scalp. Examples of dyes which give suitable shades are:

Dye	Color index
Durol Black B	307
Direct Fast Brown M	420
Alizarine Brown 2R	167
Direct Fast Orange GC	621

I do not, however, intend to limit my invention to any particular dyes as it will be understood that a great variety of non-toxic dyes are at my disposal; for example, polyhydroxylated anthraquinones, sulfonated and carboxylated compounds and compounds containing sufficient hydroxyl groups to render them of predominantly acid character, may be employed. By referring to the dyes above-mentioned and others of generally similar character as non-toxic, it is to be understood that the dyes used are to be of such character, that, when applied to the hair in accordance with the directions herein given, they are not injurious to the hair or scalp of the average individual. Of course, particular individuals may be allergic to one or another particular dye or class of dyes. This does not, however, make it improper to characterize such a dye as non-toxic. As compared with the highly toxic metallic salts and dyes of the phenylene diamine type which have heretofore been the only classes of dyes generally available for hair dyeing, the acid and direct dyes which I am able to use by my new process, are non-injurious and substantially non-toxic.

Wherever the term "neutral salt" is used in the claim, it is understood to refer to salts which are substantially neutral for all practical purposes in application to hair.

I claim:

The method of dyeing hair which comprises the steps of rendering the hair receptive to non-toxic organic dyes by applying thereto a salt of a condensation product of a higher fatty acid and an alcohol containing a primary amino group, and subsequently applying a water soluble non-toxic organic dye containing negative salt forming groups.

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