2,206,535

# UNITED STATES PATENT OFFICE

## 2,206,535

## STRIPPING DYED TEXTILE

Herbert August Lubs, Wilmington, Del., assignor to E. I. du Pont de Nemours & Company, Wilmington, Del., a corporation of Delaware

No Drawing. Application May 19, 1939, Serial No. 274,496

#### 4 Claims. (Cl. 8—102)

This invention relates to a process for stripping dyed textile fiber, both of vegetable and of animal origin, the present application being a continuation-in-part of my copending application Ser. No. 173,848, filed November 10, 1937 (Patent

No. 2,164,930, issued July 4, 1939).

It is an object of this invention to provide a novel process of stripping which may be applied either to vegetable fiber, for instance cotton, or to

- 10 animal fiber such as wool and silk, without unduly tendering the fiber. It is a further object of my invention to provide a novel stripping process which may be used effectively on materials dved with vat direct, acid, or other azoic colors.
- 15 dyed with vat, direct, acid, or other azoic colors. Other and further important objects of this invention will appear as the description proceeds. Stripping has been defined by some authorities as the removal or lightening of the shade or tone
  20 of a previously applied dye from textile material,
- 20 other wise than by extraction with solvents. This process is generally used in the arts either for the purpose of correcting an overdyed shade or for obtaining a lighter colored bottom for the redye-
- 25 ing of another color. It is also used largely in the recovery of wool from waste for the purpose of producing shoddy or for stripping of rags for paper manufacture. The methods employed heretofore involved the use of aqueous baths of vari-
- 30 ous stripping agents, according to the material treated or nature of dye to be removed. Among the various reagents employed heretofore have been ammonium hydroxide, soda ash, soap, potassium bichromate with sulfuric acid, sodium hy35 drosulfite with or without acetic acid, stannous
- chloride with hydrochloric acid, and potassium permanganate with sulfuric acid. Now according to my present invention, I ac-

complish stripping effectively by the use of formamidine-sulfinic acid preferably from an alkaline

- aqueous bath. Formamidine-sulfinic acid is a compound obtainable by the oxidation of thiourea with hydrogen peroxide. It was first prepared and described by Barnett in Jour. Chem. Soc.,
- **45** vol. 97, at pages 63-65 (1910). A commercially practical method for its production has recently been published by Emeric Havas in U. S. Patent No. 2,150,921.

The mode of procedure according to my novel 50 process involves simply treatment of the dyed fabric in a hot aqueous solution of formamidinesulfinic acid of about 0.1 to 1% strength, and enough alkali to give the bath an alkaline reaction. Wetting or penetrating agents may be added if desired. The quaternary ammonium com-

pounds mentioned in U. S. Patents Nos. 2,003,928 and 2,019,124 are particularly recommended.

Without limiting my invention to any particular procedure, the following examples are given to illustrate my preferred mode of operation. **5** Parts mentioned are by weight.

#### Example 1

50 parts of cotton, linen, rayon or mixtures of these, in the form of cloth or yarn, previously 1 colored with vat dyes are treated in 1000 parts of water containing:

Cetyl - trimethyl - ammonium bromide,

with or without oleic acid (See U. S. P.	Pa	$\mathbf{rts}$	¢
Nos. 2,003,928 and 2,019,124)	0.25	to 5	4
Caustic soda	1.5	to 3	
Formamidine-sulfinic acid	1 -	to 5	

The above materials may be added to the bath either before or after entry of the textile mate- 20 rial. In either event, the bath is heated to a temperature of 120 to 140° F. at the beginning of the treatment, and is then gradually raised in temperature to at least 200° F., and preferably 212° F., the treatment being continued with suitable agitation of the material for from 30 to 60 minutes. The source of heat is then removed, and cold water is run in, permitting the excess to flow away through any suitable arrangement, until all of the original liquor has been displaced. This water is then drained and 1000 parts of a  $\frac{1}{2}^{\circ}$  Tw. solution of sodium hypochlorite is added, and the material is treated in it at a temperature between 65 and 80° F. for 20 to 30 minutes. This is followed by the usual rinsing in cold water, or treatment in cold solutions of sodium thiosulfate or of other suitable substances to remove the residual hypochlorite. This treatment will remove some or all of the color, depending upon the dye or dyes and their quantity that the material originally contained.

Among the vat dyes that are completely or nearly completely removed by this treatment are

Anthraflavone GC (C. I. 1095)

Caledon Brown R (C. I. 1151)

Ponsol Blue GD (C. I. 1113)

- Caledon Olive (see C. I. Supplement page 30)
- 6,6-diethoxy-2,2-bisthionaphthen-indigo (C. I. 1217)

Among those less completely removed by this treatment are

Ponsol Dark Blue BOA (C. I. 1099) Ponsol Blue Green FFB (C. I. 1173) Ponsol Jade Green (C. I. 1101)

(

In a similar manner azoic dyes may be removed from cellulosic fiber, the procedure being the same as in the aforegoing example. Among the various azo dyes which are completely or nearly

completely removed by this treatment may be 5 mentioned

	5-nitro-2-amino-anisol→	*2,3-hydroxynaphthoic-		
		acid-anilide		
10	m-Nitro-p-toluidine→	o-toluidide of 2,3-hy-		
		droxynaphthoic acid		
	4-chlor-2-nitroaniline→	2-naphthylamide of 2,3-		
		hydroxynaphthoic		
	·	acid		
15	Alpha-naphthylamine→	m-nitroanilide of 2,3-		
		hydroxynaphthoic		
		acid		
$\rightarrow$ stands for "diazotized and coupled to."				

Among those less completely removed are 20

2,5-dichloroaniline $\rightarrow$  diacetoacetyl-o-tolidine 2,5-dichloroaniline→ di -(5-chlor-2,4-dimethoxyanilide) of terephthaloylbis-acetic acid

25 m-Nitro-p-anisidine→p-chloroanilide of 2-hydroxy-carbazol - 3 - car -

boxylic acid.

- As a general mode of procedure, the color may 30 be completely or partly removed from 50 parts of cotton, linen, rayon, silk and wool or combinations of these in the forms of cloth or yarn by treatment at 200 to 212° F. for 15 to 35 minutes
- in 1000 to 2000 parts of water containing 1.5 to 5 35 parts of formamidine sulfinic acid and sufficient ammonia, or sodium or potassium carbonate to cause the liquor to react distinctly alkaline. To prevent the animal fibers, silk and wool, from being deleteriously affected by the alkali, only
- 40 ammonia should be used, and in a quantity just sufficient to cause the liquor to react with Brilliant Yellow paper.

Formamidine sulfinic acid is particularly useful for stripping acid and direct dyestuffs from

45 wool or shoddy material. The product operates in a slightly alkaline bath at temperatures near the boil.

For the purpose of testing formamidine sulfinic acid for this purpose, I had the following tests 50 carried out:

#### Example 2

Samples of wool crepe were dyed with the following colors:

Per cent Pontamine Fast Yellow BBL (C. I. 814)\_\_\_\_\_ Pontacyl Fast Blue R (C. I. 208) \_\_\_\_ Pontacyl Fast Blue 5R Conc. (C. I. 289) \_\_\_\_ ~----2

Percentages are on weight of material.

60

55

The samples were then subjected to stripping

with various stripping baths according to the following compositions:

	<b>.</b>	Parts	
(a)	Sulfoxite S. Conc	0.05	
	Acetic acid	0.03	5
	Water	20.00	
(b)	Formamidine sulfinic acid	0.03	
	Ammonia (26%)	0.03	
	Water	20.00	
(C)	Formamidine sulfinic acid	0.00	10
	Ammonia (26%)	0.00	
	Water	0.00	
(A)		20.00	
(4)	Formamidine sumnic acid	0.03	
	Soda ash	0.06	
	Water	20.00	15
(e)	Formamidine sulfinic acid	0.06	
	Acetic acid	0.02	
	Water	90.00	
-		40.00	

In all the above compositions the figures are 20 based per unit part of textile material. In all the above tests the fabric was treated with the solution at 180° F. for ½ hour. It was found that formamidine-sulfinic acid

is superior to the Sulfoxite S type of stripper for 25 acid and direct colors, and reduces the fabric more nearly to a white. This effect was observed particularly in Tests (b), (c) and (d), as compared to Test (a).

It will be understood that the above examples 30 are merely illustrative, and that the details of my process are susceptible of wide variation and modification within the skill of those engaged in this art.

I claim:

1. The process of stripping the color from textile material which has been dyed with a color of the group consisting of vat and azoic colors, which comprises treating the fabric in an aqueous bath containing formamidine-sulfinic acid and alkali 40 at a temperature near the boiling point of the solution.

2. The process of stripping azoic colors from cellulosic fiber, which comprises treating the fiber, at a temperature between 180 and 212° F., 45 in an aqueous bath containing from 0.1 to 1% by weight of formamidine sulfinic acid and sufficient alkali to give the liquor an alkaline reaction.

3. The process of stripping azoic colors from wool fiber, which comprises treating the fiber, at 50a temperature between 180 and 212° F., in an aqueous bath containing from 0.1 to 1% of formamidine-sulfinic acid and just enough ammonia to give the liquor an alkaline reaction to Brilliant Yellow paper. 55

4. A process as in claim 1, the treatment being carried out in the further presence of a quaternary ammonium compound having an aliphatic radical which contains not less than 10 carbon atoms. 60

## HERBERT AUGUST LUBS.

35

2