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NO DRAWINGS

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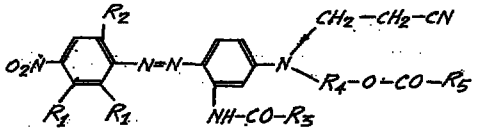
(72) Inventor WOLFGANG GROEBKE

(54) MONOAZO DYES OF LOW SOLUBILITY IN WATER, THEIR PRODUCTION AND USE

(71) We, SANDOZ LTD., of 4002 Basle/Switzerland, Lichtstrasse 35, a Body Corporate organised according to the laws of Switzerland, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is performed, to be particularly described in and by the following statement:—

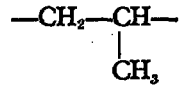
This invention relates to monoazo dyes of the 2 - chloro- or -bromo - 4 - nitro - 2' - alkylcarbonylamino - 4' - (N - cyanoalkyl - N - alkylcarbonyl - oxyalkyl) - amino - 1,1' - azobenzene series, which are highly suitable for dyeing and printing synthetic and semi-synthetic fibres consisting of high molecular-weight organic substances of hydrophobic character, and of textile materials made thereof.

The new monoazo dyes are of formula



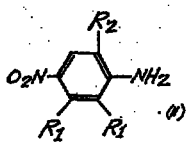
(I),

where R_1 in both instances represents hydrogen, or one R_1 represents chlorine or bromine and the other hydrogen,
R_2 represents chlorine or bromine,
R_3 lower alkyl,
R_4 a radical of formula $-CH_2CH_2-$ or



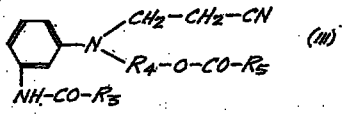
and R_5 lower alkyl, and where the alkyl radicals R_3 and R_5 may be substituted by chlorine, bromine, cyano or lower alkoxy. By "lower alkyl and alkoxy" are understood alkyl and alkoxy radicals having 1, 2, 3 or 4 carbon atoms.

The new monoazo dyes may be produced by diazotization of an amine of formula



(II)

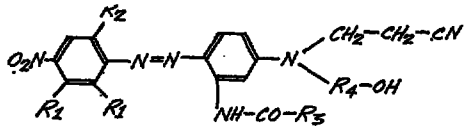
and coupling of the resulting diazonium compound with a compound of formula



(III)

The diazotizing and coupling reactions may be carried out by the known methods. The coupling reaction generally takes place in acid medium at temperatures in the range of −10°C to +35°C, or more particularly between 0°C and +5°C. It is often of advantage to conduct coupling in a medium buffered with, e.g. sodium acetate.

The compounds of formula (I) can alternatively be produced by acylation of a compound of formula



(IV)

with an acid of formula

$$R_5—COOH \qquad (V)$$

or a functional derivative of an acid of this type, e.g. the anhydride, chloride or bromide, using the generally known methods.

It is advisable to convert the new dyes thus formed into dyeing preparations before their use, by means of the normal methods for this purpose, such as grinding in the presence

[Price 25p]

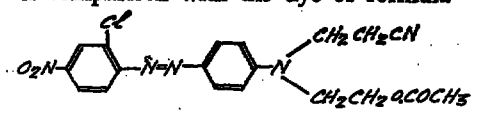
of dispersing agents and/or fillers. If desired the resulting mixtures can be vacuum or injection dried to give powder preparations. After the addition of a suitable volume of water they can be applied by exhaustion dyeing, pad dyeing or printing process at long or short liquor ratio.

From aqueous dispersion the dyes build up excellently on fibres and textiles consisting of synthetic or semi-synthetic high molecular-weight organic compounds of hydrophobic character. They are specially suitable for dyeing and printing polyester, cellulose diacetate, cellulose triacetate and polyamide fibres, and textiles of these fibres. They may also be used to dye polyolefin fibres.

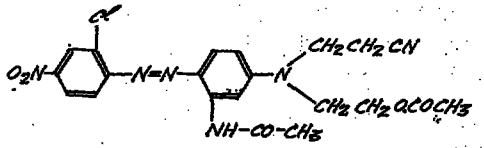
The known dyeing and printing processes are employed, for example that described in French Patent 1,445,371. The dyeings and prints obtained have very good all round fastness; in particular they are outstandingly fast to thermofixation, sublimation, pleating, solvents, lubricants, dry cleaning, rubbing, ozone and gas fumes. The wet fastness properties, e.g. water, sea water, perspiration and washing fastness, are also very good.

The light fastness and the reserve of cotton and wool are other salient features. The dyeings are highly stable to pre-cure and post-cure permanent press finishing and to soil release finishes, readily dischargeable, and stable to boiling and reduction at temperatures up to about 220°C and especially in the range up to 150°C. They also show good stability to acids and alkalis. These stability properties are not adversely affected by the liquor ratio or by the presence of dyeing accelerants.

In comparison with the dye of formula



which is described in Example 6 of French Patent 1,211,477 and Example 15 of British Patent 819,664, the next comparable dye of the present invention, which has the formula



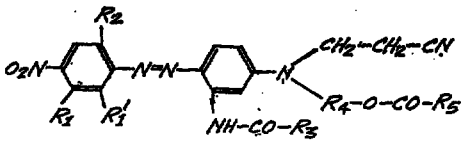
(Example 1) is unexpectedly faster to light and thermofixation.

In the following Examples the parts are by weight and the temperatures are given in degrees centigrade.

Example 1

173 parts of 1 - amino - 2 - chloro - 4 - nitrobenzene are stirred into 250 parts of concentrated hydrochloric acid and 350 parts of ice-water and diazotized at 0—5° with a solution of 69 parts of sodium nitrite in 250 parts of water. The diazonium salt solution is combined with a solution of 300 parts of glacial acetic acid and 289 parts of 1(N-β-cyanoethyl - N - acetoxyethyl) - amino - 3 - acetyl - aminobenzene and 25 parts of urea at 0—5°. Sodium acetate solution is added to complete the coupling reaction at pH 4. the dye settles out and is filtered off with suction, washed with water and dried. It gives dyeings of red shade on polyester fibres which have excellent fastness properties.

In the following table further dyes are specified which can be produced in accordance with this invention and are of formula

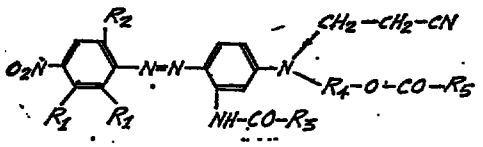


TABLE

Exple No.	R_1	R'_1	R_2	R_3	R_4	R_5	Shade on Polyester fibre
2	H	H	Cl	$-C_2H_5$	$-CH_2CH_2-$	$-C_2H_5$	red
3	H	H	Cl	$-CH_3$	do	do	do
4	H	Cl	Cl	do	do	do	red-brown
5	H	Cl	Cl	$-C_3H_7$	do	do	do
6	H	H	Br	$-CH_3$	do	do	red
7	Cl	H	Cl	do	do	do	do
8	H	Cl	Br	do	do	do	red-brown
9	H	Br	Br	do	do	$-C_2H_5$	do
10	H	Cl	Cl	$-C_4H_9$	do	$-C_4H_9$	do
11	Br	H	Br	$-CH_3$	do	$-C_2H_5$	red
12	H	H	Br	do	$-CH_2-CH(CH_3)-$	do	do
13	H	H	Cl	$-C_3H_7$	do	$-C_3H_7$	do
14	H	H	Cl	$-CH_3$	do	$-CH_3$	do
15	H	H	Cl	$-CH_2Br$	$-C_2H_4-$	do	do
16	H	H	Cl	$-CH_2CH_2Cl$	$-C_2H_4-$	do	do
17	H	H	Cl	$-CH_2CN$	do	do	do
18	H	H	Cl	$-CH_2OCH_3$	do	do	do
19	H	H	do	$-CH_3$	do	$-CH_2Cl$	do

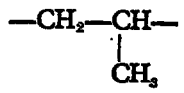
WHAT WE CLAIM IS:—

1. Monoazo dyes of formula



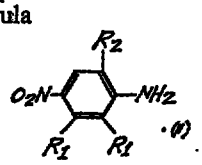
(I),

where R_1 in both instances stands for hydrogen or one R_1 stands for hydrogen and the other for chlorine or bromine,
R_2 for chlorine or bromine,
R_3 for lower alkyl,
R_4 for a radical of formula $-CH_2-CH_2-$ or



and R_5 for lower alkyl, and where the alkyl radicals R_3 and R_5 may be substituted by chlorine, bromine, cyano or lower alkoxy.

2. A process for the production of a monoazo dye of formula (I) according to claim 1 which comprises the diazotization of an amine of formula



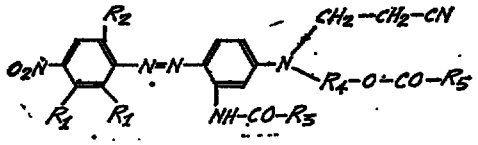
(II).

TABLE

Exple No.	R_1	R'_1	R_2	R_3	R_4	R_5	Shade on Polyester fibre
2	H	H	Cl	$-C_2H_5$	$-CH_2CH_2-$	$-C_2H_5$	red
3	H	H	Cl	$-CH_3$	do	do	do
4	H	Cl	Cl	do	do	do	red-brown
5	H	Cl	Cl	$-C_3H_7$	do	do	do
6	H	H	Br	$-CH_3$	do	do	red
7	Cl	H	Cl	do	do	do	do
8	H	Cl	Br	do	do	do	red-brown
9	H	Br	Br	do	do	$-C_2H_5$	do
10	H	Cl	Cl	$-C_4H_9$	do	$-C_4H_9$	do
11	Br	H	Br	$-CH_3$	do	$-C_2H_5$	red
12	H	H	Br	do	$-CH_2-CH(CH_3)-$	do	do
13	H	H	Cl	$-C_3H_7$	do	$-C_3H_7$	do
14	H	H	Cl	$-CH_3$	do	$-CH_3$	do
15	H	H	Cl	$-CH_2Br$	$-C_2H_4-$	do	do
16	H	H	Cl	$-CH_2CH_2Cl$	$-C_2H_4-$	do	do
17	H	H	Cl	$-CH_2CN$	do	do	do
18	H	H	Cl	$-CH_2OCH_3$	do	do	do
19	H	H	do	$-CH_3$	do	$-CH_2Cl$	do

WHAT WE CLAIM IS:—

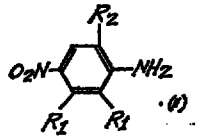
1. Monoazo dyes of formula



(I),

where R_1 in both instances stands for hydrogen or one R_1 stands for hydrogen and the other for chlorine or bromine,
R_2 for chlorine or bromine,
R_3 for lower alkyl,
R_4 for a radical of formula $-CH_2-CH_2-$ or $-CH_2-CH(CH_3)-$
and R_5 for lower alkyl, and where the alkyl radicals R_3 and R_5 may be substituted by chlorine, bromine, cyano or lower alkoxy.

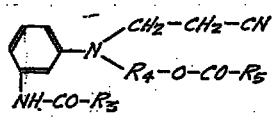
2. A process for the production of a monoazo dye of formula (I) according to claim 1 which comprises the diazotization of an amine of formula



(II)

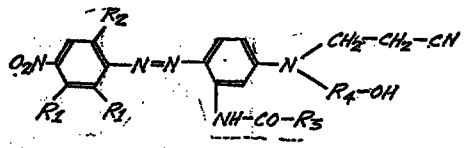
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and coupling of the diazonium compound with a compound of formula



(III)

3. A process for the production of a monoazo dye of formula (I) according to claim 1 which comprises the acylation of a compound of formula



(IV)

with an acid of formula

$$R_5—COOH \qquad (V)$$

or with a functional derivative thereof.

4. The particular monoazo dyes of formula (I) according to claim 1 herein described and exemplified in any one of Examples 1 to 19.

5. A process for the production of a monoazo dye of formula (I) according to claim 1 conducted substantially as herein described and exemplified with particular reference to any one of Examples 1 to 19.

6. Monoazo dyes of formula (I) according to claim 1 whenever prepared by a process according to any one of claims 2, 3 or 5.

7. A dyeing or printing process in which filaments, fibres or textiles made thereof, which consist of synthetic or semi-synthetic high molecular-weight organic compounds of hydrophobic character, are dyed or printed with a monoazo dye according to any one of claims 1, 4 and 6.

8. Filaments, fibres and textiles made thereof, which consist of synthetic or semi-synthetic high molecular weight organic compounds of hydrophobic character dyed or printed by a process according to claim 7.

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