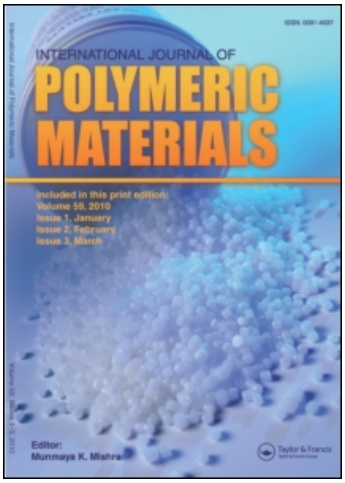

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Bactericidal Reactive Dyes: Use in Bactericidal Polymeric Fabrics

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It was shown that fibrous materials (FM) dyed with bactericidal monochlorotriazine reactive dyes containing hexachlorophene (HCF) possess a wide spectrum of bactericidal action. This effect depends on the dye functionality based on phenol OH-groups. With monofunctional dyes the effect is 3–8 times larger than observed with dyes containing disubstituted HCF residue.

KEY WORDS Bactericidal dyes, bactericidal fabrics.

INTRODUCTION

Obtaining FM possessing bactericidal properties is a real problem. It can be solved through the use of dyes which possess both bactericidal activity and dye reactivity relative to FM.[1] However, the know bactericidal dyes either have a very small bactericidal effect or insufficient coloristic properties.[2]

In this connection the use of reactive dyes allowing to link the bactericidal agent with FM is of considerable interest. The bactericidal reactive dyes (BRD) can be obtained on the basis of the initial dichlorotriazine dyes (IDD), which contain two labile Cl-atoms, one of which can be substituted with the bactericidal agent residue. The unsubstituted Cl atom is free to form the covalent bond with FM.

The investigations on the synthesis of BRD were directed to obtain dyes possessing higher bactericidal activity and good stability of the dyeings. The bactericidal dyes can find wide application for imparting bactericidal properties to stockings and socks, shoe cloth, filters and clothes for medical personnel.

EXPERIMENTAL

The fibrous materials. The bleached cotton cloth and cloth from polycaproamide (nylon 6) were used as a cellulosic FM and polyamide FM, respectively.

The initial dyes. As IDD the dichlorotriazine dyes: C. I. Reactive blue 4 (I), C. I. Reactive red 1 (II), C. I. Reactive yellow 4 (III) and Cr-complex (1:2) C. I. Reactive orange 1 (IV) were used.

The bactericidal agent. Hexachlorophene (2,2-dihydroxy-3,3,5,5,6,6-hexa-

chlorodiphenylmethane), the product of the reaction of the condensation 2,4,5-trichlorophenol with formaldehyde, was used as a bactericidal agent. Formaldehyde contains two reactive phenol OH-groups, allowing HCF to enter via a nucleophilic substitution reaction with IDD.

The medico-biological investigations. The bactericidal activity was determined both for the obtained BRD and the FM treated by them. For BRD the method of serial dilutions was used, allowing for determination of the minimal suppressing concentration (MSC) relative to a number of test-cultures: Staphilococcus aureus 209, Escherichia Coli K-12 and Proteus vulgaris.[3]

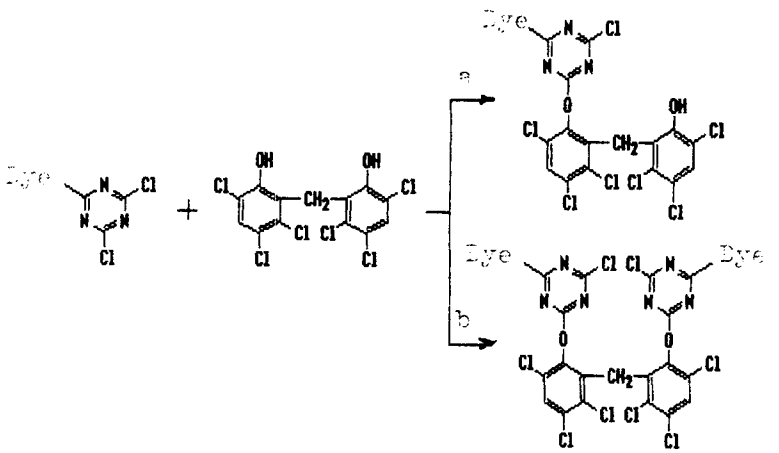
The fungicidal activity was determined by a method of serial dilutions in man-made nourishing medium Saburo (pH = 6.8–7.0) relative to Trichophyton red, Tpichoderma viride and Candida albicans.[2]

The dyeing of cellulosic and polycaproamide FM. Cellulosic FM was dyed by impregnating FM in water solution containing BRD in concentration 10–30 g/l, urea—100–250 g/l, Na_2CO_3—25 g/l at a liquor ratio 5:1–7:1 and temperature 20°C. Impregnated FM was squeezed, dried and fixed by the steaming or the heat setting. The samples of dyed FM were washed in a solution of detergent at a concentration of 1 g/l at 50°C for 30 min.

The polycaproamide FM was dyed by conventional method.[4]

RESULTS AND DISCUSSION

The BRD was synthesized by nucleophilic substitution reaction of one labile Cl-atom in the IDD molecule with bactericidal agent HCF residue:

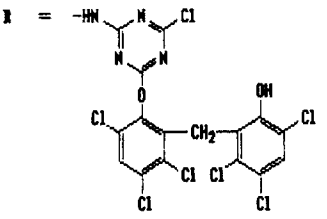


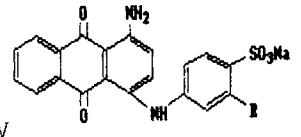
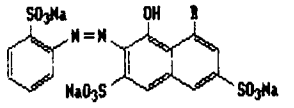
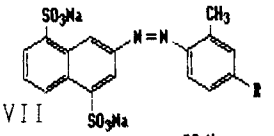
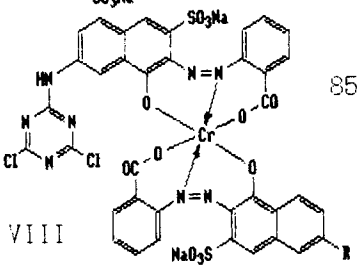
From this reaction scheme it was concluded that synthesis of BRD can be carried out in two directions: a) obtaining monofunctional and b) bifunctional reactive dye. The bifunctional BRD has been obtained and investigated earlier.[5]

The aim of our work was obtaining monofunctional BRD, in which a higher bactericidal activity was predicted because of increased mole fraction of HCF in

TABLE I

The structure and results of the analysis of obtained BRD



Structure of the BRD	Yield %	Rf	λ_{max} nm	C	H	S	N	Cl
 V	92	0,84	518	40,2 40,4	3,1 3,4	6,3 6,1	8,16 9,02	23,4 *a* 23,5 *b*
 VI	90	0,70	523	38,6 38,9	3,1 3,2	9,1 8,9	7,90 7,72	22,9 *a* 22,8 *b*
 VII	95	0,76	377	39,6 40,2	3,3 3,4	6,8 6,5	8,78 8,54	25,2 *a* 25,1 *b*
 VIII	85	0,66	558	40,2 40,1	1,6 1,5	3,9 4,1	10,90 10,80	20,6 *a* 20,4 *b*

Note *a*—observed; *b*—calculated (%).

the BRD molecule of monofunctional structure. The reaction between the components proceeds well at the stoichiometric proportion between them. The reaction between IDD and HCF may be conducted in an organic solvent medium such as dimethylformamide, which adequately dissolves both the IDD and HCF. It is preferable to conduct the reaction in an organic solvent since possible hydrolysis

TABLE II

The bactericidal activity of BRD

IDD	Functionality of BRD	MSC (g/l) to Staphiloc. aureus 209	Escherechia Coli K-12	Proteus vulgaris
	Mono-V	0,0026	0,17	0,62
I	Bi-	no active	no active	no active
	Mono-VI	0,0052	0,32	0,50
II	Bi-	0,0170	no active	no active
	Mono-VII	0,0126	0,23	0,50
III	Bi-	0,0170	no active	no active
IV	Mono-VIII	0,020	-	-

TABLE III

Comparative antifungal activity of BRD and IDD

	MSC, g/l							
Test-culture	I	V	II	VI	III	VII	IV	VIII
Trichophyton red	not active	1,8	not active	3,7	not active	0,11	1,8	0,03
Trichoderma viride	not active	3,7	not active	7,5	not active	0,22	3,7	0,22
Candida albicans	not active	15,0	not active	15,0	—	—	not active	7,5

of the IDD is completely eliminated, increasing the yield of the desired product. The reaction is conducted at 80–90°C over the course of 3–6 hours.[6] The monofunctional structure of BRD was established by a combination of different methods.[7]

The structure and results of analysis of the obtained BRD V–VIII are given in Table I.

The investigation of bactericidal activity of the obtained BRD was accomplished through comparison with their bifunctional analogs. In Table II the bactericidal activity of BRD are given.

TABLE IV

Coloristic and bactericidal properties of cotton FM dyed by BRD and IDD

Dye	Colorfastness, marks		Bactericidal activity (the zone of growth suppressing of the test-microbes), mm
	To washing	To rubbing	
I	4/5/5	5	no zone
V	4/4/4	4	6
II	4/5/5	5	no zone
VI	3/3/4	3	5–6
III	4/5/5	5	no zone
VII	4/4/4	4	6
IV	4/4/5	4	no zone
VIII	3/3/3	4	8–9

From these data we can conclude that bactericidal activity of the monofunctional BRD exceeds that for bifunctional BRD by 3–8 times. The increased bactericidal activity of the monofunctional BRD is also connected to the higher activity of monosubstituted HCF residue, containing a phenol OH-group.

The antifungal activity of the synthesized BRD relative to that for IDD was also investigated (see Table III).

From Table III it follows that IDD-IV (Cr-complex Reactive orange 1) has small but appreciable antifungal activity, associated with the presence of Cr(VI) in the structure of dye complex. The metallocomplexes in dye chromophore increase the fungicidal action of BRD.

The synthesized BRD are monochlorotriazinyl reactive dyes which can be used for dyeing natural and man-made fibers and articles made from them. The process of dyeing with BRD is similar to that used for conventional monochlorotriazine dyes. The BRD were applied to cellulose FM by a padding-mangle or discontinuous method and to polycaproamide FM by a bathwise method only.

The data of Table IV show that general coloristic properties of BRD are of the same order as for IDD.

The bactericidal socks successfully dyed by BRD were put through toxicological and clinical tests.[9,10] The bactericidal fabrics obtained through dyeing with BRD acquired high bactericidal activity, which was still retained after 20–30 washings.

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