The Influence of Long-Chain Alkyl Groups on the Colour, Dyeing and Fastness Properties of 2-(and 1-) Phenylazo-1-(and 2-) Naphthol Acid Dyes

Y. C. Chao & C. W. Yeh

Department of Textile Industries, National Taipei, Institute of Technology, Taipei, Taiwan

ABSTRACT

The synthesis of a series of acid dyes obtained by diazotization of p-alkyl-anilines and coupling with 1-(and 2-) naphthol sulphonic acid derivatives is described. The effects of the alkyl groups on the colour, dyeing, and fastness properties of the dyes are discussed. The dyes coloured polyamide fibres well. It is concluded that substantivity, water-repellency, and wet-fastness of the dyes on polyamide fibres increase with increase in the chain length of the alkyl groups, provided that the dyes dissolve completely in the dyebath.

1 INTRODUCTION

Acid dyes with dodecyl or dodecyloxy groups (Carbolan dyes) were developed by ICI and are characterized by high milling fastness, a property conferred on the molecule by the long-chain alkyl group.[1] Dewitt and Shroff[2] found that longer-chain alkyl-substituted dyes give better water-repellency than shorter-chain substituted dyes. They also found that long-chain alkyl groups decrease the substantivity of dyes on cotton and wool fibres through steric hindrance.

Hydrophilicity, molecular size, and electron-density parameters of the dyes, which are changed by the introduction of a long-chain alkyl group into the dye molecule, affect the colour, dyeing and fastness properties of dyes on polyamide fibres, the extent depending on the structure

285

of the dyes, but little detailed information with respect to this is available. The synthesis of a series of acid dyes obtained by diazotization of p-alkylanilines and coupling with H-acid, NW-acid, M-acid, and R-acid, respectively, is reported, together with a study of the effect of a long-chain alkyl group on the properties of the resulting acid dyes.

2 EXPERIMENTAL

2.1 2-(4'-alkyl)phenylazo-8-amino-1-naphthol-3,6-disulphonic acids (I)

H-acid (11·17 g, 0·031 mole) was dissolved in water and the pH raised to 10·0 by adding 20% aq. Na_2CO_3. Ice was added to lower the temperature to 0°C and 0·032 mole of benzenediazonium chloride (prepared from 3·0 g aniline) was then added at such a rate that the temperature remained below 5°C. The pH of the coupling medium was maintained near 10·0 by dropwise addition of 20% aq. Na_2Co_3. After stirring for about 3 h and then leaving the medium to stand overnight, the precipitate was filtered and washed with hot water. The product was dried in vacuum at 50°C for 8 h to give 11·62 g (yield 76·2%) of 2-phenylazo-8-amino-1-naphthol-3,6-disulphonic acid (dye I.1) as a purple solid (m.p. above 400°C).

Dyes I.2–I.5 were synthesized by the above procedure, except that aniline was replaced by the appropriate p-alkylaniline. Relevant data on yield and m.p. are shown in Table 1.

2.2 Other dyes (series II–IV)

The above procedures (Section 2.1) were repeated, except that H-acid was replaced by NW-acid (Dye II), M-acid (Dye III), and R-acid (Dye IV) (see Table 1).

2.3 General

All dyes were purified by TLC on Kieselgel 60 (Merck). Electronic spectra were recorded on a Shimadzu UV 240 from dye solutions in dimethylformamide (DMF) or dimethylsulfoxide (DMSO) (Table 2). Structure and purity were established by IR (Hitachi 260-50) and elemental analysis (Perkin–Elmer 240C). Since most dyes were not completely soluble in water, dyeing of nylon 6 was carried out from a solution of 19 parts water and one part DMF (liquor ratio 50:1) at 100°C for 1 h; the samples were then cooled to room temperature and washed with an aqueous solution of detergent (2 g/liter) at 60°C for 20 min.

TABLE 1
Synthesis and Characterisation Data of Dyes

Dye	1° arom.amine(3·0 g)	Coupler	m.p.(°C)	Mass (yield)	Appearance of precipitate
I.2	*p*-(n-butyl)-aniline (0·02 mole)	H-acid (7·31 g, 0·02 mole)	above 400°C	8·645 g (82·1%)	Purple solid
I.3	*p*-(n-octyl)-aniline (0·0146 mole)	H-acid (5·312 g, 0·0146 mole)	above 400°C	7·238 g (85·4%)	Purple solid
I.4	*p*-(n-dodecyl)-aniline (0·0114 mole)	H-acid (4·172 g, 0·0114 mole)	above 400°C	5·882 g (80·6%)	Purple solid
I.5	*p*-(n-hexadecyl)-aniline (0·0094 mole)	H-acid (3·435 g, 0·0094 mole)	above 400°C	5·116 g (78·24%)	Purple solid
II.1	aniline(0·032 mole)	NW-acid (7·872 g, 0·032 mole)	above 400°C	8·750 g (78·34%)	Orange solid
II.2	*p*-(n-butyl)-aniline (0·02 mole)	NW-acid (4·953 g, 0·02 mole)	263–264°C	6·870 g (84·32%)	Orange solid
II.3	*p*-(n-octyl)-aniline (0·0146 mole)	NW-acid (3·600 g, 0·0146 mole)	348–349°C	6·080 g (90·2%)	Orange solid
II.4	*p*-(n-dodecyl)-aniline(0·0114 mole)	NW-acid (2·827 g, 0·0114 mole)	306–307°C	5·248 g (88·32%)	Orange solid
II.5	*p*-(n-hexadecyl-aniline (0·0094 mole)	NW-acid (2·328 g, 0·0094 mole)	126–127°C	4·360 g (80·43%)	Orange solid
III.1	aniline (0·032 mole)	M-acid (8·352 g, 0·032 mole)	254–246°C	9·830 g (84·16%)	Brown solid
III.2	*p*-(n-butyl)-aniline (0·020 mole)	M-acid (5·255 g 0·020 mole)	above 400°C	7·086 g (83·6%)	Brown solid

TABLE 1—*contd.*

Dye	1° arom.amine(3·0 g)	Coupler	m.p.(°C)	Mass (yield)	Appearance of precipitate
III.3	p-(n-octyl)-aniline (0·0146 mole)	M-acid (3·819 g, 0·0146 mole)	above 400°C	5·540 g (79·34%)	Brown solid
III.4	p-(n-dodecyl)-aniline (0·0114 mole)	M-acid (3·000 g, 0·0114 mole)	280–282°C	5·371 g (87·68%)	Brown solid
III.5	p-(n-hexadecyl)-aniline (0·0094 mole)	M-acid (2·470 g, 0·0094 mole)	245–246°C	4·760 g 85·41 %)	Brown solid
IV.1	aniline(0·032 mole)	R-acid (11·226 g, 0·032 mole)	320–321°C	11·63 g (80·41%)	Red solid
IV.2	p-(n-butyl)-aniline (0·020 mole)	R-acid (7·006 g, 0·020 mole)	above 400°C	9·453 g (92·43%)	Red solid
IV.3	p-(n-octyl)-aniline (0·0146 mole)	R-acid (5·093 g, 0·0146 mole)	above 400°C	7·230 g (87·6%)	Red solid
IV.4	p-(n-dodecyl)-aniline (0·0114 mole)	R-acid 4·000 g, 0·0114 mole)	above 400°C	5·874 g (82·4%)	Red solid
IV.5	p-(n-hexadecyl)-aniline (0·0094 mole)	R-acid (3·293 g 0·0094 mole)	above 400°C	5·460 g (85·5%)	Red solid

The dye-uptake was evaluated by extracting the dye from a known weight of dyed material with dimethylformamide and determining the absorbance of the solution (Shimadzu UV 240). Dye-uptake was calculated from the calibration curve of absorbance against concentration of dye.

The wash-fastness was tested according to CNS 1494-A3, a similar test to AATCC 61-1989-1A, except that the test conditions were replaced by 100 ml of liquor (5 g/liter detergent and 2 g/litre anhydrous Na_2CO_3) at 60°C ± 2°C for 30 min.

TABLE 2

Spectroscopic Data

Dye	R	IR* (cm⁻¹/KBr)	Element analysis %: found (calculated)
I.1	H	2800—3500(b, phenol), 1500, 1400, (phenyl), 1180, 1030(SO₃⁻), 750(m.s.p)	C: 40·58(41·11), H: 3·17(2·35), N: 6·71(8·99)
I.2	n-C₄˙I₉	3450(b, phenol); 2960, 2930 2870(alkyl); 1490, 1390, 1600 (alkyl); 1290(N-phenyl); 1190, 1040(SO₃⁻), 820(P.d.p)	C: 46·68(45·88), H: 4·5(3·63), N: 6·85(8·03)
I.3	n-C₈H₁₇	3450(b, phenol), 2960, 2940, 2860(alkyl); 1610, 1580, 1420 (b) (phenyl) 1170(b), 1040 (SO₃⁻); 840(p.d.p)	C: 50·62(49·74), H: 6·38(4·66), N: 6·03(7·25)
I.4	n-C₁₂H₂₅	3450(b, phenol); 2970, 2930, 2860(alkyl)1600, 1460(b) (phenyl); 1180, 1040(SO₃⁻) 840(p.d.p)	C: 49·36(52·91), H: 6·29(5·51), N: 5·08(6·61)
I.5	n-C₁₆H₃₃	3450(b, phenol); 2960, 2925, 2850(alkyl); 1600, 1490, 1460(phenyl); 1190, 1030 (SO₃⁻)820(p.d.p)	C: 57·83(55·57), H: 7·14(6·22), N: 4·85(6·07)
II.1	H	3500(b), 1250(phenol); 1610, 1515, 1480, 1450(phenyl); 1205, 1060(SO₃⁻), 760, 690 (m.s.p)	C: 56·47(54·86), H: 4·30(3·14), N: 6·78(8·00)
II.2	n-C₄H₉	3500(b), 1240(phenol); 2950, 2930, 2870, (alkyl); 1600, 1560, 1500(phenyl); 1280(N-phenyl)1160, 1040(SO₃⁻); 820(p.d.p)	C: 58·88(59·10), H: 4·64(4·67), N: 6·54(6·89)
II.3	n-C₈H₁₇	3500(b), 1260(phenol); 2980, 2950, 2870(alkyl); 1620, 1540, 1500, 1460(phenyl) 1200, 1060(SO₃⁻)840(p.d.p)	C: 62·33(62·33), H: 5·23(5·84), N: 4·91(6·06)
II.4	n-C₁₂H₂₅	3500(b), 1240(phenol); 2970, 2950, 2870(alkyl)1600, 1500, 1480(phenyl); 1180, 1060 (SO₃⁻); 1280(N-phenyl); 780(p.d.p)	C: 66·87(64·86), H: 8·52(6·75), N: 5·46(5·40)

TABLE 2—*contd.*

Dye	R	IR* (cm^{-1}/KBr)	Element analysis %: found (calculated)
II.5	n-$C_{16}H_{33}$	3500(b), 1240(phenol); 2970, 2950, 2870(alkyl); 1600, 1500 1480(phenyl); 1180, 1060 (SO_3^-); 1280(*N*-phenyl); 760(*p*.d.p)	C:68·33(66·89), H:9·54(7·49), N:4·72(4·89),
III.1	H	3350(b, phenol); 1590, 1550, 1490, (phenyl); 1170, 1030 (SO_3^-), 750(m.s.p)	C:51·41(52·60), H:4·20(3·28), N:10·95(11·5)
III.2	n-C_4H_9	3500(b), 1250(phenol); 2970, 2940, 2870, (alkyl); 1610, 1510, 1480(phenyl); 1200, 1060(SO_3^-); 1280 (*N*-phenyl); 820(*p*.d.p)	C:56·62(57·0), H:5·60(4·80), N:8·40(9·90)
III.3	n-C_8H_{17}	3500(b), 1250(phenol); 2950, 2850, (alkyl); 1620, 1480, 1450, (phenyl); 1190, 1040 (SO_3^-); 825(*p*.d.p)	C:58·93(60.37), H:6·25(5·87), N:8·58(8·80)
III.4	n-$C_{12}H_{25}$	3500(b), 1240(phenol); 2890, 2870, (alkyl); 1610, 1450 (phenyl); 1190, 1035(SO_3^-) 830(*p*.d.p)	C:63·90(63·03), H:7·48(6·75), N:7·29(7·88)
III.5	n-$C_{16}H_{33}$	3450(b, phenol); 2950, 2870, (alkyl); 1620, 1480(phenyl); 1190, 1040(SO_3^-); 830(*p*.d.p)	C:64·57(65·19), H:7·87(7·47), N:7·01(7·13)
IV.1	H	3500(b), 1240(phenol); 1610, 1500, 1480(phenyl); 1190, 1040 (SO_3^-), 750(m.s.p)	C:40·39(42·47), H:3·46(1·99), N:4·47(6·19)
IV.2	n-C_4H_9	3500(b), 1250(phenol); 2970, 2950, 2870, (alkyl); 1610, 1500, 1450(phenyl); 1190, 1040(SO_3^-); 820(*p*.d.p)	C:47·56(47·09), H:5·65(3·54), N:4·38(5·51)
IV.3	n-C_8H_{17}	3470(b), 1230(phenol); 2950, 2890, (alkyl); 1620, 1510, 1490, (phenyl); 1210, 1040 (SO_3^-); 820(*p*.d.p)	C:52·88(51·06), H:5·82(4·43), N:6·39(4·96)
IV.4	n-$C_{12}H_{25}$	3450(b), 1250(phenol); 2950, 2870, (alkyl); 1620, 1480, 1450(phenyl); 1200, 1040 (SO_3^-); 820(*p*.d.p)	C:53·58(54·19), H:5·88(5·32), N:3·66(4·51)
IV.5	n-$C_{16}H_{33}$	3500(b), 1230(phenol); 2950, 2880(alkyl); 1620, 1480 (phenyl) 1200, 1040 (SO_3^-); 840(*p*.d.p)	C:56·27(56·80), H:6·37(6·06), N:3·19(4·14)

* b: broad; m.s.p: monosubstituted phenyl; p.d.p: *para*-disubstituted phenyl.

The water-repellency was tested according to AATCC 39-1977. The light-fastness assessment was carried out as for a previous investigation.[3]

3 RESULTS AND DISCUSSION

3.1 Electronic spectra

Zollinger,[4] in studies of the mechanism of azo-coupling reactions by using H-NMR and UV/VIS, found that the benzenediazonium ion couples only ortho to the hydroxy group of Gamma-acid. Two-dimensional ^{1}H-, ^{13}C-, and ^{15}N-NMR spectra of azo dyes derived from J-, H-, and Gamma-acids were investigated by Lyčka and Jirman.[5] They concluded that the above-mentioned aminohydroxynaphthalenesulfonic acids undergo diazo coupling of benzenediazonium chloride ortho to the hydroxy groups under alkaline conditions. It is therefore assumed that the dyes of series I, II, and III exist in the 2-phenylazo-1-naphthol form, and those of series IV in the 1-phenylazo-2-naphthol form.

The azo–hydrazone tautomerism of hydroxyazo dyes was discovered in Zincke and Binderwalk[6] in 1884. Hydroxyazo dyes of the naphthalene series exist more in the hydrazone form than the azo form, taking into account bond energies and resonance stabilization energy. Generally, a more polar solvent favours the hydrazone form, whereas electron-donating groups in the diazo-component ring increase the proportion of the azo form.[6] In 2-phenylazo-1-naphthols and 1-phenylazo-2-naphthols, the predominance of the hydrazone form is such that intramolecular hydrogen-bonding is stronger in that form than in the azo form, and the effect of both solvents and substituents is much less marked than in other phenylazonaphthol systems.[7] Lyčka and Jirman[4] studied the structures of monoazo dyes derived from J-acid, H-acid, and Gamma-acid by using H-, ^{13}C-, and ^{15}N-NMR. They concluded that these dyes contained more than 80% of the hydrazone form at 300–360 K in DMSO-d_6. Thus, the dyes reported in this paper are presumed to exist exclusively in the hydrazone form.

Electronic-spectra data of the dyes are shown in Table 3. On comparing series III with series I, a bathochromic effect of 3–8 nm (III → I) is apparent. This may be due to the stabilizing effect of the peri-amino group on series I. The large difference in λ_{max} values between series III and series II suggests that strong reinforcement of electron density from the electron-donating amino group to the coupling-component ring gives a pronounced bathochromic effect (II → III, 28_31 nm) in hydroxyazonaphthalenes. Comparison of series II with series IV shows a signifi-

TABLE 3
Color and TLC Data

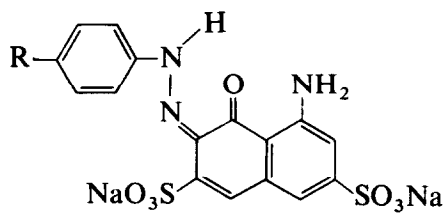| Dye	Rf* (TLC)	λ^{nm}_{max} (log ε) in DMF	λ^{nm}_{max} in DMSO
I.1	0·828	530 (4·26)	538
I.2	0·837	539 (4·10)	544
I.3	0·853	539 (4·09)	550
I.4	0·856	540 (4·30)	552
I.5	0·868	540 (4·07)	552
II.1	0·859	497 (3·41)	500
II.2	0·865	506 (4·19)	506
II.3	0·879	506 (4·12)	508
II.4	0·896	506 (3·70)	508
II.5	0·905	507 (4·12)	508
III.1	0·825	528 (4·14)	535
III.2	0·833	533 (3·81)	540
III.3	0·841	534 (4·06)	544
III.4	0·846	535 (4·32)	544
III.5	0·859	535 (4·38)	544
IV.1	0·720	484 (3·86)	486
IV.2	0·761	488 (3·92)	492
IV.3	0·820	488 (4·17)	492
IV.4	0·850	488 (3·64)	492
IV.5	0·870	488 (4·04)	492

* eluent: n-butanol: acetic acid: water = 4:1:5

cant hypsochromic effect of 13–19 nm (II → IV). This may be due to the electron-withdrawing effect of the sulphonic acid group. Introducing long-chain alkyl groups into the diazo component causes a less-pronounced bathochromic effect (3–9 nm) in each series, irrespective of the effect of chain length. This indicates that the reinforcement of electron density from the alkyl groups to the diazo component is weak.
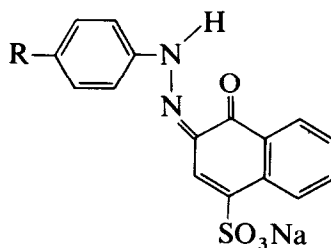
3.2 Dyeing and fastness properties

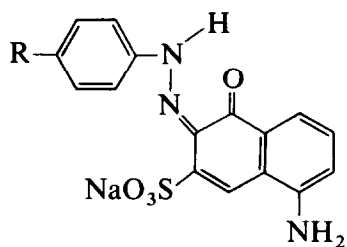All dyes in these four series gave level coloration on polyamide-fibre fabrics.

Comparing the solubility in the dyebath (at 100°C) of the dyes in series I (H-acid derivatives) with that of the corresponding dyes in series II (NW-acid derivatives), series III (M-acid derivatives) and series IV (R-acid derivatives) (Table 4), it is observed that H-acid was better than the other couplers. It can be seen from Table 5 that the dye-uptake of the three depths of dyes in series I is higher than that of the corresponding dyes in series II, III, and IV. This may be explained in terms of the solubility effect. Consideration of the dye-uptake of the three shades of
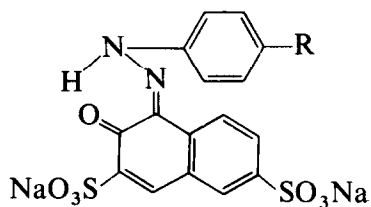
(I)

(II)

(III)

(IV)

TABLE 4
Solubility* of Dyes in Dyebath at 100°C

Dye \ o.w.f.	0·1%	0·5%	2·0%
I.1	1	1	1
I.2	1	1	1
I.3	1	1	1
I.4	1	1	1
I.5	1	1	1
II.1	1	1	1
II.2	1	1	1
II.3	1	1	1
II.4	1	1	2
II.5	1	2	3
III.1	1	1	1
III.2	1	1	1
III.3	1	1	2
III.4	2	2	3
III.5	3	3	4
IV.1	1	1	1
IV.2	1	1	1
IV.3	1	1	2
IV.4	1	2	3
IV.5	2	3	4

* 1: Dissolved completely, 2: Sight precipitate, 3: Appre-
ciable precipitate, 4: Apparent precipitate

TABLE 5
Dyeing and fastness Properties

o.w.f.	Dye-uptake (g/kg fibres)			Light-fastness			Wet-fastness (S.N/C.C)*			Water-repellency (2% o.w.f. in seconds)
Dye	0·1%	0·5%	2·0%	0·1%	0·5%	2·0%	0·1%	0·5%	2·0%	
I.1	0·83	3·47	12.10	3	3	3	5/4	4/2–3	3/2–3	35
I.2	0·85	3·75	12·46	3	3	3	5/4	4/3–4	4/3	45
I.3	0·87	3·84	12·87	3	3	3	5/4	4/3	4/3	53
I.4	0·88	3·93	13·54	3	3	4	5/5	4–5/4	4/3	88
I.5	0·89	4·04	13·79	3	3	3	5/5	4–5/3	4/3–4	117 (227**)
II.1	0·76	3·41	11·89	4	5	5	4/3–4	3–4/3	2–3/2	36
II.2	0·79	3·47	12·47	4	4	6	4/3–4	3–4/3	3/3	42
II.3	0·80	3·54	12·67	5	6	6	4/4	4/3–4	3/3	59
II.4	0·81	3·70	11·56	4	5	6	5/4	4–5/4	4/3–4	111
II.5	0·82	3·40	11·43	3	4	4	5/5	5/4–5	4–5/4	143 (282**)
III.1	0·74	3·32	11·34	3	3	3	4/3–4	3–4/3	3/2–3	38
III.2	0·76	3·41	12·47	3	3	5	4/4	3–4/3	3/3	44
III.3	0·78	3·50	11·16	3	4	5	5/4	4/3	3–4/3	61
III.4	0·70	3·21	10·74	3	4	5	5/4	4–5/3–4	4/3	128
111·5	0·68	3·10	10·61	3	3	4	5/4	5/4	5/4	156 (293**)
IV·1	0·75	3·39	11·47	4	4	4	5/3	4/2–3	4/2–3	35
IV·2	0·77	3·42	12·57	3	3	3	5/4	5/3	4/2–3	49
IV·3	0·79	3·51	11·31	3	3	4	5/4	5/3–4	4–5/4	55
IV·4	0·80	3·25	10·89	3	3	4	5/5	5/4–5	4–5/4	96
IV·5	0·72	3·12	10·42	4	5	6	5/5	5/5	5/5	129 (233**)

* S.N.: staining on nylon, C.C: colour change
** On 5% o.w.f.

dyes in series I shows that the substantivity on nylon 6 increases with increasing chain length of the alkyl groups. In series II, III and IV, the increase in substantivity can be correlated with the chain length and solubility factors (Tables 4 and 5), namely:

(a) 0·1% dyeings: II.5 > II.4 > II.3 > II.2 > II.1, III.3 > III.2 > III.1 > III.4 > III.5, IV.4 > IV.3 > IV.2 > IV.1 > IV.5

(b) 0·5% dyeings: II.4 > II.3 > II.2 > II.1 > II.5, III.3 > III.2 > III.1 > III.4 > III.5, IV.3 > IV.2 > IV.1 > IV.4 > IV.5

(c) 2·0% dyeings: II.3 > II.2 > II.1 > II.4 > II.5, III.2 > III.1 > III.3 > III.4 > III.5, IV.2 > IV.1 > IV.3 > IV.4 > IV.5

Since some dyes at 2·0% o.w.f. did not dissolve completely in the dye-bath but were completely soluble in a mixture of one part DMF and three parts water, dyeing of nylon 6 was also carried out in this medium (liquor ratio 50:1) at 100°C for 1 h. On the basis of uptake results (Table

TABLE 6
Uptake* of Dyes in Dyebath of Three Parts Water and
One Part DMF

Dye	0·5% o.w.f.	2·0% o.w.f.
II.1	3·04	10·68
II.2	3·16	10·85
II.3	3·25	10·98
II.4	3·30	11·14
II.5	3·37	11·26
III.1	3·13	11·05
III.2	3·34	11·98
III.3	3·41	12·30
III.4	3·43	12·47
III.5	3·51	12·58
IV.1	3·37	11·40
IV.2	3·42	12·42
IV.3	3·50	12·54
IV.4	3·51	12·67
IV.5	3·53	12·85

* g dyes/kg fibres

6), it can be concluded that the substantivity of dyes in series II, III, and
IV on nylon 6 increases with increasing chain length of the alkyl groups.

The wet-fastness of acid dyes decreases as the depth of dyeing increases.
This is readily understood, since the greater the amount of dye present,
the more likely it is that, during treatments sufficient to cause staining, it
will be detached from the material.[8] Hence it is not correct to discuss the
wet-fastness of dyes without consideration of the differences in substan-
tivity. It can be seen from Table 5 that the wet-fastness of dyes in every
series increases with increasing chain length of the alkyl groups, despite
the substantivity-increasing factor. On the basis of the results of water-
repellency of dyes in every series, and that of I.5, II.5, III.5, and IV.5 at
2% and 5% dyeings, the increase in water-repellency can be correlated
with the chain-length and dye-uptake factors. It can be seen from Table
4 that the light-fastness of dyes in series I and III is inferior to that of
dyes in series II and IV. This could be due to the lone electron pair on
the free-amino groups of the dyes in series I and III.

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