



One-bath One-stage Dyeing Process of Polyamide/polyester Blend Fabric Using Carbocyclic Vat Dyes



Abdalla A. Mousa^{1*}, Yehya A. Youssef¹, Khalid M. Abd El Moteleb² and Ashraf F. Wasfy²

¹Textile Research Division, National Research Centre, 33 El Bohouth St, Dokki, Giza, 12622, Egypt.

²Chemistry Department, Faculty of Science, Benha University, Benha, 13518, Egypt.

THE DYEING behavior of selected both carbocyclic and heterocyclic vat dyes on polyamide PA and polyester PET fabrics have been examined. Factors affecting dyeability such as sodium hydrosulphite concentration, dyeing temperature and dye concentration were also studied. It was found that color strength (K/S) values of the carbocyclic vat dyes on PA and PET are high, which confirms the usefulness of these dyes for dyeing PA and PET fabrics in a neutral medium (acid leuco) at the specific temperature 120°C with very good fastness properties. However, K/S values obtained using heterocyclic vat dyes are very low compared with those obtained using carbocyclic vat dyes. The results indicate the possibility of using carbocyclic vat dyes for the one-bath and one-stage dyeing of PA/PET blend fabric with very good fastness properties.

Keywords: Blends dyeing, one-bath dyeing process, vat dyeing, vat dyeing of polyamide/polyester blend fabric

Introduction

Dyeing of synthetic fabrics with vat dyes has been studied by a number of researchers. Several researchers attempted to dye PET fabrics with vat dyes or indigo to seek a part of the market share [1-6]. Sugawara studied vapor phase dyeing on PET fabrics [6], which requires very high temperatures up to 220–250 °C. Other authors proposed a leuco vat acid dyeing method, in which organic acids were added to the dyeing bath to get the pH below 7 [1]. However, this dyeing method required additions of organic acids, which increased the cost of treating wastewater. Only a few studies [7–13] showed that PA, PET and other synthetic fibers could be dyed with selected vat dyes using the conventional (sodium hydrosulphite/sodium hydroxide) reduction system. Vat dyes are considered to be one of the most industrially important class of dyes for cotton fabric owing to

their excellent light and wet fastness properties. However, vat dyes are not normally used on synthetic fabrics due to its characteristically low substantivity towards synthetic fabrics and the generally pale shades that arise from their limited diffusion within the synthetic fabrics. PA/cotton blends fabrics have been dyed with vat dyes and pale shades on the blend fabric are reported [14]. To the best of our knowledge, no work has been published concerning the application of vat dyes to PA/PET blend fabric. In the present work, carbocyclic and heterocyclic vat dyes are applied to PA, PET and PA/PET blend fabric through an acid leuco exhaust dyeing method. Also this work will describe the effect of sodium hydrosulphite concentration, dyeing temperature and dye concentration on the K/S values and color coordinates of dyed PET, PA and PA/PET blend fabric.

*Corresponding author, email: mousanrc@gmail.com. Tel: 00201111627085

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Experimental

Fabrics

Woven 100% polyamide fabric and 100% polyester fabric were supplied by El-Mahalla El-Kobra Company, Egypt. A blended woven PA/PET blend fabric in a blend ratio 20:80 was made in our laboratory. All fabrics treated before dyeing with an aqueous solution containing 2 g/l nonionic detergent (Sera Wash M-RK, DyStar, Egypt) at liquor to goods ratio of 50:1 and at 80 °C for 30 min, and then rinsed and allowed to dry in the open air.

Chemicals, Auxiliaries and Vat dyes

Anionic dispersing agent (Avolan® IS, Tanatex Chemicals B. V.) and non-ionic detergent (Sera Wash M-RK, DyStar) were used as supplied. All other reagents used were of standard laboratory grade. Commercial vat dyes comprising three carbocyclic based dyes of CI Vat Yellow 33 (VY33), CI Vat Violet 1 (VV1) and CI Vat Blue 20 (VB20), and three heterocyclic based dyes of CI Vat Yellow 2 (VY2), CI Vat Red 10 (VR10) and CI Vat Blue 14 (VB14) were used as received. The CI generic name and chemical structures of both carbocyclic and heterocyclic vat dyes are illustrated in Tables 1 and 2.

Vat dyeing of PET, PA and PA/PET blend fabrics

The vat dyeing procedure of PA, PET and PA/PET blend fabrics with different vat dyes including carbocyclic vat dyes and heterocyclic vat dyes was performed at 1-4% omf depth of shade in the presence of 5-20 g/l sodium hydrosulphite, 2 g/l sodium hydroxide and 10g/l dispersing agent (Avolan® IS) at a liquor ratio 40:1. The dyebath pH was adjusted to 5.5 using acetic acid. Under such conditions, the reduced anionic vatted dye form is converted into its more hydrophobic nonionic acid form that allows the dye molecules to be more substantive to the hydrophobic PA, PET and PA/PET blend fabrics. The vat dyeing was then performed by raising the temperature of the dyebath from 40 °C to 110-120 °C at a rate of 2.5 °C/min, holding at this temperature for 60 min and then cooling the bath to 60 °C at a rate of 3 °C/min. The dyed samples were then rinsed in distilled water for 5 min followed by dye oxidation using 2 g/l hydrogen peroxide of 35 wt% at pH 4.5, 70 °C at a liquor ratio 20:1 for 15-20 min. The dyed, oxidized fabrics were thoroughly rinsed with water and then subjected to reduction clearing. The reduction clearing step for all samples was carried out at 60-70 °C for 10 min in a solution containing 3 g/l, sodium

hydroxide, 3 g/l sodium hydrosulphite and 2 g/l nonionic detergent (Sera Wash M-RK) at a liquor ratio 40:1, then the samples were finally rinsed thoroughly with water and air dried.

Equipment

Dyeing was carried out on a Pyroteclaboratory dyeing machine with an infra-red heating system. The colorimetric data of dyed PET, PA and PA/PET blend fabrics after washing were determined using an UltraScan PRO spectrophotometer with a D65 illuminant and 10° standard observer.

Fastness testing

The colorfastness of dyed PET, PA and PA/PET blend fabrics after washing-off using 2 g/l nonionic detergent at 80 °C for 15 min, were tested in accordance with ISO standard methods. The wash fastness test was assessed in accordance with the standard method ISO 105-C06 B2S [15] (4g/l of ECE detergent, 1 g/l of sodium perborate, 25 steel balls) at 50 °C for 30 min and at a liquor ratio of 50:1. Fastness to acidic and alkaline perspiration was determined with a perspirometer set at specific pressure, temperature and time in accordance with ISO 105-E04 [16]. Any change in color (ΔE) of the specimens and color staining of the adjacent cotton (SC) and polyester (SP) multifibre was then assessed with the corresponding ISO grey scales for color change and staining. Light fastness was also assessed using a Xenon arc lamp test in accordance with ISO 105-B02 [17].

Results and Discussion

It is important to note that this study including dyeing of PA, PET and PA/PET blend fabrics using both carbocyclic and heterocyclic vat dyes. Effect of sodium hydrosulphite concentrations, dyeing temperature and dye concentrations on the K/S values and color coordinates of the dyed fabrics are reported.

Effect of sodium hydrosulphite concentration

Figures 1 and 2 summarize the results of the K/S values of the carbocyclic vat dyes applied on PET and PA fabrics respectively. Carbocyclic vat dyes applied on both fabrics at different sodium hydrosulphite concentrations (5-20g/L) and at 4% dye concentration and 120 °C dyeing temperature. The results assessed for the acid leuco exhaust dyeing indicate that carbocyclic vat dyes showed higher K/S values on PA fabrics compared with those obtained on PET fabrics at all sodium hydrosulphite concentrations. Also, it

TABLE 1. Commercial names and chemical structures of carbocyclic vat dyes

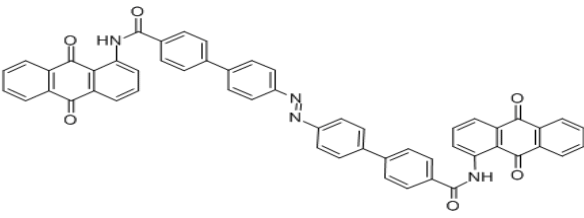
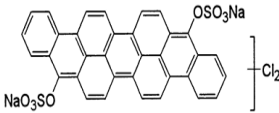
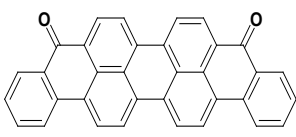
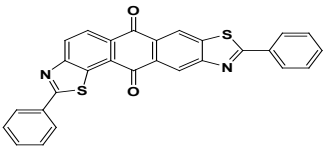
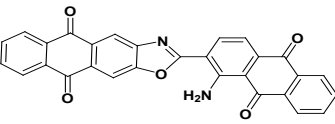
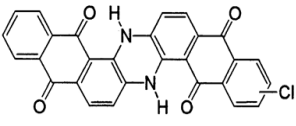
CI Generic name	Commercial name	Dye structure
CI Vat Yellow 33 VY33	Vat Yellow 3G	
CI Vat Violet 1 VV1	Vat Violet RR	
CI Vat Blue 20 VB 20	Indanthrene Dark Blue BOA	

TABLE 2. Commercial names and chemical structures of heterocyclic vat dyes

CI Generic name	Commercial name	Dye structure
CI Vat Yellow 2 VY2	Vat Yellow GCN	
CI Vat Red 10 VR10	Indanthrene Red FBB	
CI Vat Blue 14 VB14	Vat Brilliant Blue 5RLC	

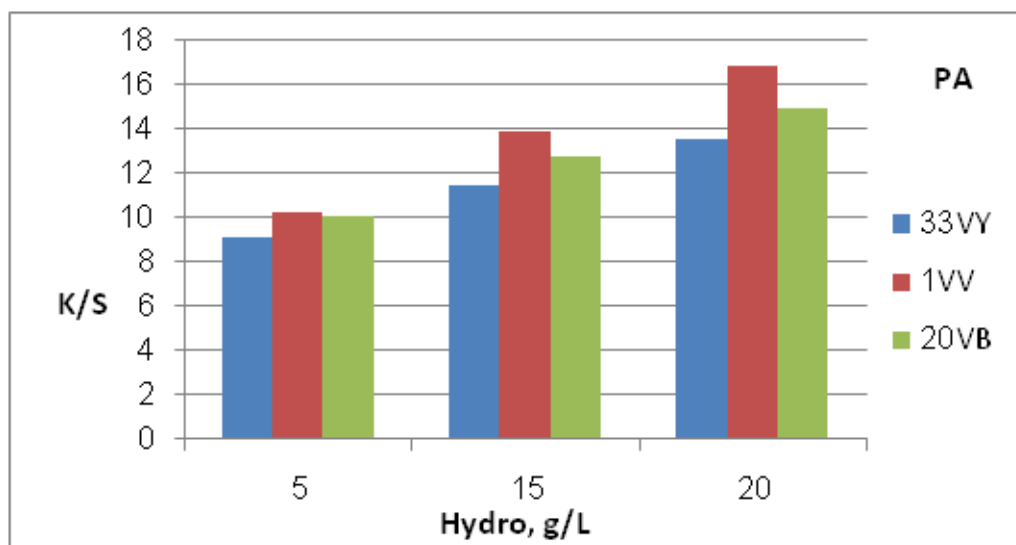


Fig. 1. K/S values of carbocyclic vat dyes on PA fabric at different sodium hydrosulphite concentrations and at 4% dye concentration and 120 °C dyeing temperature for 60 min.

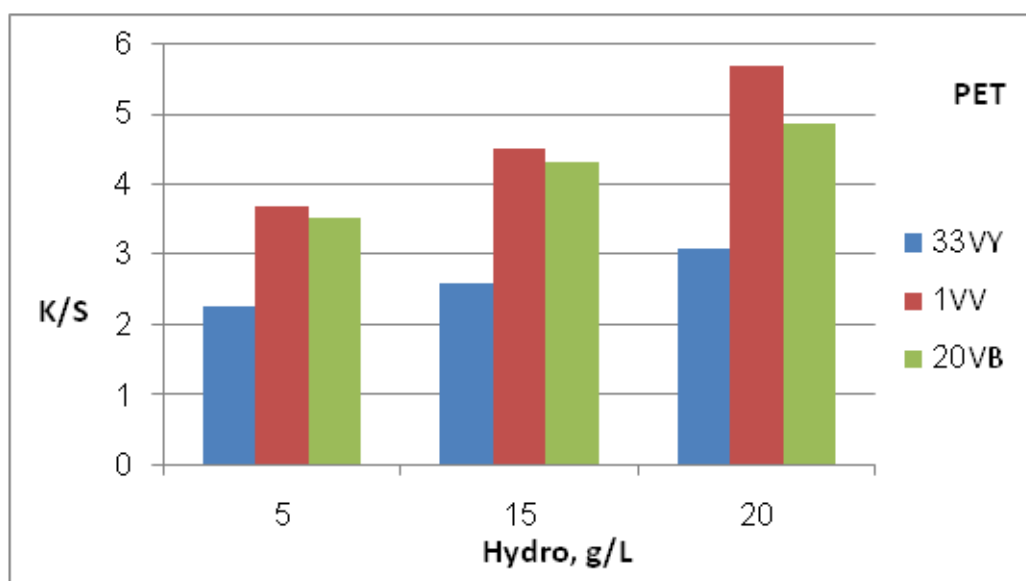


Fig. 2. K/S values of carbocyclic vat dyes on PET fabric at different sodium hydrosulphite concentrations and at 4% dye concentration and 120 °C dyeing temperature for 60 min.

is clear that an increase in sodium hydrosulphite concentrations from 5 to 20g/L increased the K/S values of both dyeings, which can be explained in terms of the higher sodium hydrosulphite concentrations resulted in greater dye reduction and/or penetration of the acid leucocarbocyclic vat dyes in both PET and PA fabrics.

In the context of the color coordinates (Tables 3 and 4) recorded for both dyed PET and PA fabrics. From both it should be noticed also show that, lightness (L^*), hue (h) and chroma (C^*) of both PET and PA dyeings using carbocyclic vat dyes becoming dark and deep at 20g/L sodium hydrosulphite concentration compared with those obtained at 15g/L sodium hydrosulphite concentration.

On the other hand, the dyeing performance of heterocyclic vat dyes, VY2, VR10 and VB14, were also assessed on PET and PA fabric. These dyes secured very low K/S values compared to those of carbocyclic vat dyes. Figure 3 and Table 5 shows the results of K/S and color coordinates of dyed PET and PA fabrics using 4% dye concentration, 20g/L sodium hydrosulphite and at 120 °C dyeing temperature. The lower K/S values of heterocyclic vat dyes, particularly on PET fabric, can be attributed to the low substantivity caused by the presence of hetero atom in their respective acid leuco form structure. This phenomenon will be the subject of a future work. So, the heterocyclic vat dyes have been excluded and our study focused on factors that affect one-bath dyeing process of PA/PET blend fabric using only the carbocyclic vat dyes.

TABLE 3. Color coordinates of dyed PA fabric using 4% owf carbocyclic vat dyes at different sodium hydrosulphite concentration (15, 20g/l) and at 120°C for 60 min. 120°C

Carbocyclic vat dyes	Sod. Hydrosulphite (g/l)	L^*	a^*	b^*	C^*	h
VY33	15	71.18	5.36	40.50	40.80	82.46
	20	69.44	6.98	47.41	47.93	81.63
VV1	15	36.35	14.49	-18.57	23.56	307.96
	20	23.91	15.71	-23.81	28.52	303.42
VB20	15	32.54	3.38	-16.38	16.72	281.65
	20	26.21	2.98	-15.86	19.14	280.63

TABLE 4. Color coordinates of dyed PET fabric using 4% owf carbocyclic vat dyes at different sodium hydrosulphite concentration (15, 20g/l) and at 120°C for 60 min. 120°C

Carbocyclic vat dyes	Sod. Hydrosulphite (g/l)	L^*	a^*	b^*	C^*	h
VY33	15	81.75	2.83	32.82	30.12	87.84
	20	78.55	2.51	30.82	33.92	85.34
VV1	15	41.89	19.96	-16.24	22.44	318.34
	20	38.25	22.25	-22.23	31.45	315.04
VB20	15	42.59	10.91	-12.60	16.95	311.80
	20	40.81	10.60	-13.19	17.72	306.75

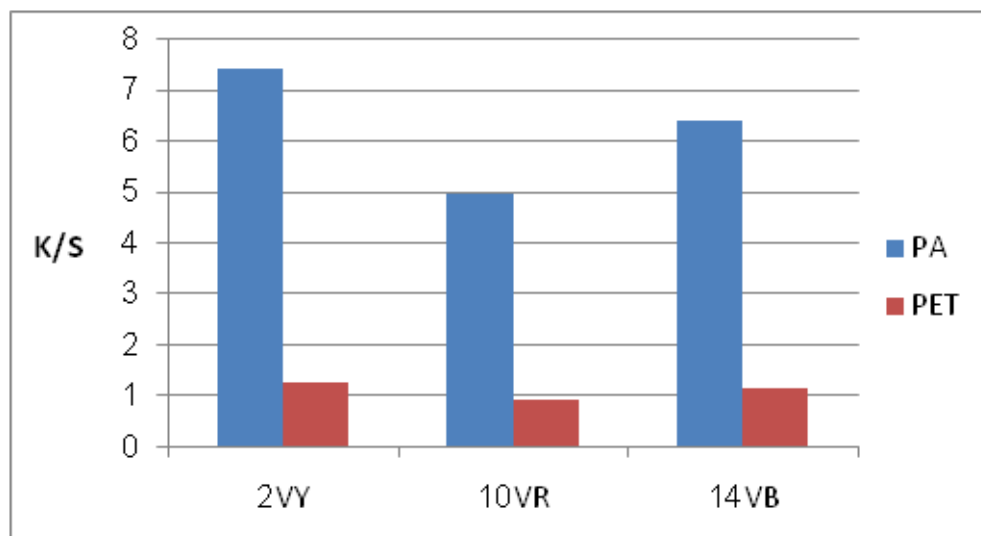


Fig. 3. K/S values of heterocyclic vat dyes on PET and PA fabrics at 20 g/L sodium hydrosulphite concentrations, 4% dye concentration and 120 °C dyeing temperature for 60 min.

TABLE 5. Color coordinates of dyed PET and PA fabrics using heterocyclic vat dyes at 4% owf, 20 g/l sodium hydrosulfite and 120°C dyeing temperature for 60 min

Heterocyclic Vat Dye	Fabric	L*	a*	b*	C*	h
VY2	PA	63.98	5.19	48.17	48.45	83.86
	PET	81.40	3.56	26.46	26.69	82.34
VR10	PA	48.93	37.79	9.23	38.90	13.72
	PET	71.64	30.02	5.27	30.48	9.95
VB14	PA	46.53	-5.15	-32.19	32.60	269.91
	PET	64.01	-1.55	-21.40	24.45	265.86

Effect of dyeing temperature

The study was extended to investigate the effect of dyeing temperature on the dyeing behaviour of carbocyclic vat dyes at 20 g/l sodium hydrosulfite. The results, illustrated in Fig. 4 and 5, indicate that the dyeing temperature had a pronounced effect on the dye uptake especially on PET fabric. The observed improvement of the color strength of each of the three carbocyclic vat dyes imparted by increasing the dyeing temperature from 100 to 120°C can be attributed to the relatively high substantivity of the acid leuco form which resembles as a disperse dye in terms of its affinity and adsorption characteristics. It is likely that the higher dyeing temperature is an outcome of greater dye diffusion of acid leuco form, which is subsequently converted into the insoluble parent vat dye in situ within the substrate by oxidation at the end of dyeing.

Improvements of color strength on both fabrics at higher dyeing temperatures are also evidenced

by the reduction in lightness (L*) and the higher values of color saturation chroma (C*), as listed in Tables 6, 7.

Effect of dye concentration

The extent of the K/S values of PET and PA fabrics with different concentrations of carbocyclic vat dyes (1–4% owf) was investigated at 20g/L sodium hydrosulphite concentration and 120 °C dyeing temperature as shown in Figures 6 and 7, respectively. The results assessed for the acid leuco exhaust dyeing indicate that carbocyclic vat dyes showed higher K/S values on PA fabrics compared with those obtained on PET fabrics at all dye concentrations. Also the results for carbocyclic vat dyes studied indicate that the K/S values of carbocyclic vat dyes on PET and PA fabrics increases as the carbocyclic vat dye concentration increases. This emphasizes that the carbocyclic vat dye content plays an important role in increasing the K/S values on both PET and PA fabrics.

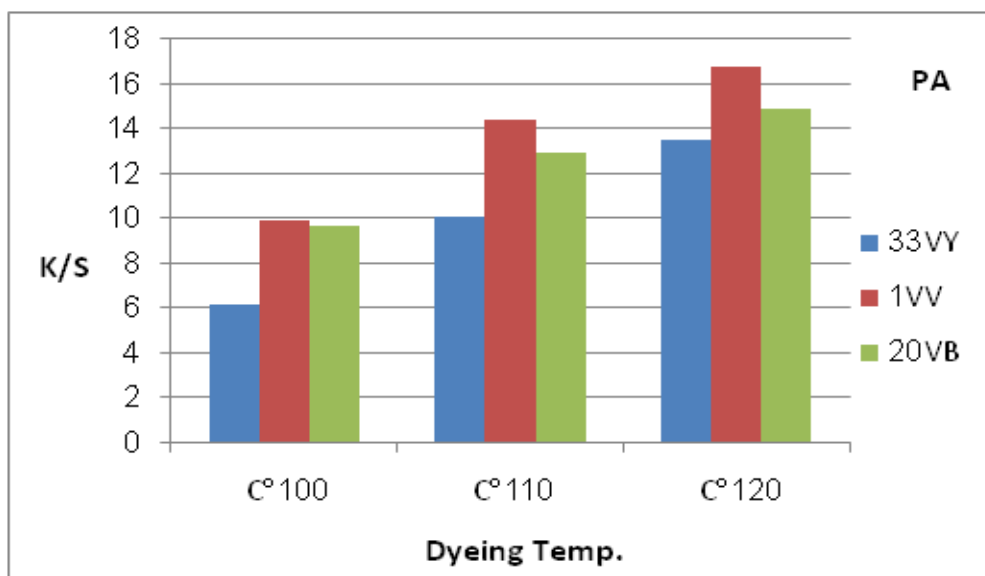


Fig. 4. Effect of dyeing temperature on K/S values of carbocyclic vat dyes on PA fabric at 20g/L sodium hydrosulphite concentrations, 4% dye concentration for 60 min.

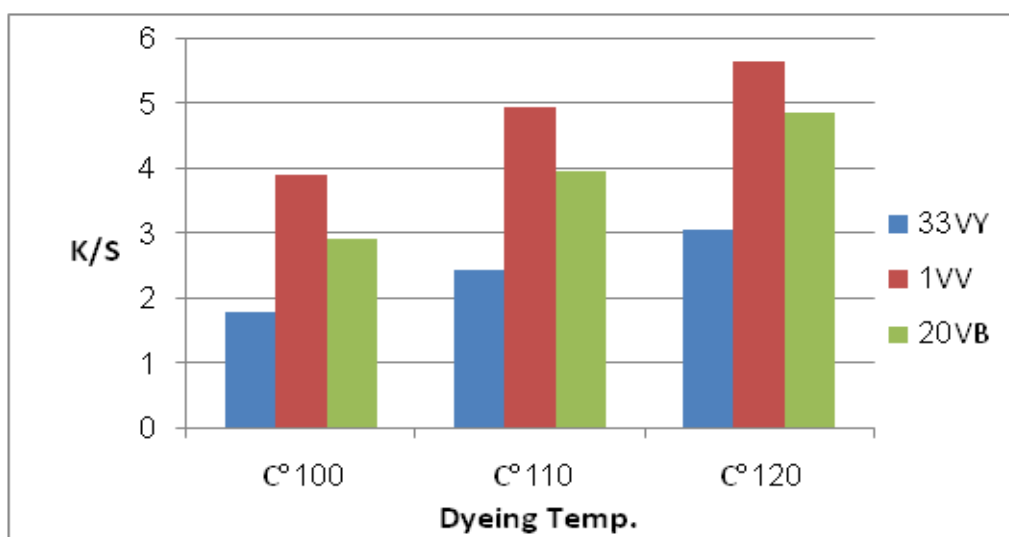


Fig. 5. Effect of dyeing temperature on K/S values of carbocyclic vat dyes on PET fabric at 20g/L sodium hydrosulphite concentrations, 4% dye concentration for 60 min.

TABLE 6. Color coordinates of dyed PA fabric using carbocyclic vat dyes (4% owf), 20g/l sodium hydrosulphite at different dyeing temperatures (100, 120°C) for 60 min

Carbocyclic vat dyes	Temp., °C	L*	a*	b*	C*	h
VY33	100	72.55	3.13	28.07	28.25	83.64
	120	69.44	6.98	47.41	47.93	81.63
VV1	100	36.71	14.93	-24.03	28.29	303.85
	120	23.91	15.71	-23.81	28.52	303.42
VB20	100	28.44	5.05	-15.76	16.55	287.76
	120	26.21	2.98	-15.86	19.14	280.63

TABLE 7. Color coordinates of dyed PET fabric using carbocyclic vat dyes (4% owf), 20 g/l sodium hydrosulfite at different dyeing temperatures (100, 120°C) for 60 min

Carbocyclic vat dyes	Temp., °C	L*	a*	b*	C*	<i>h</i>
VY33	100	84.72	-1.76	35.76	30.81	92.81
	120	78.55	2.51	30.82	30.92	85.34
VV1	100	54.88	16.4 5	-15.31	22.48	317.06
	120	38.25	22.25	-22.23	31.45	315.04
VB20	100	43.36	12.30	-9.40	15.48	322.62
	120	40.81	10.60	-14.19	17.72	306.75

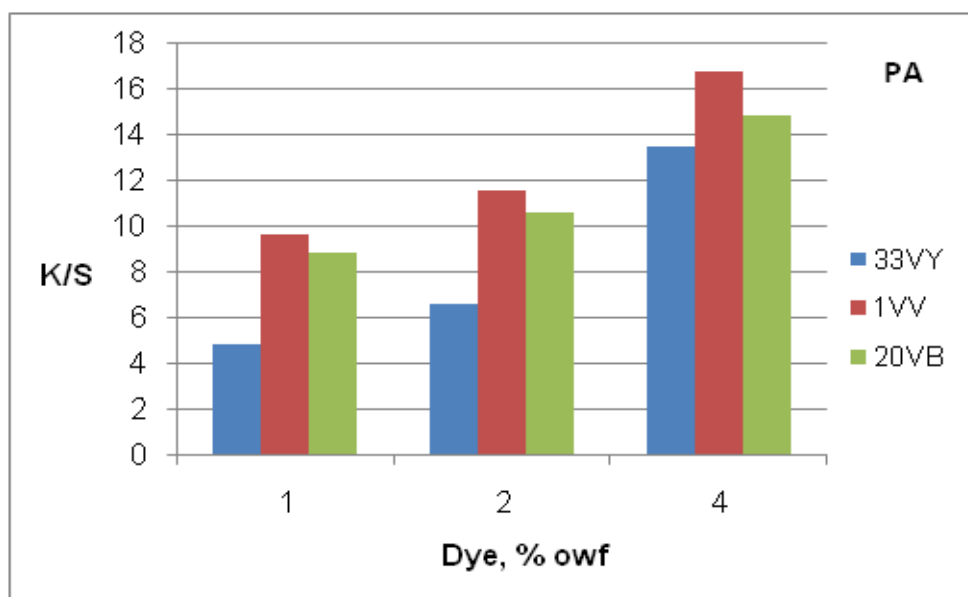


Fig. 6. K/S values at different dye concentrations, 20g/L sodium hydrosulphite concentrations and at 120 °C dyeing temperature for carbocyclic vat dyes on PA fabric.

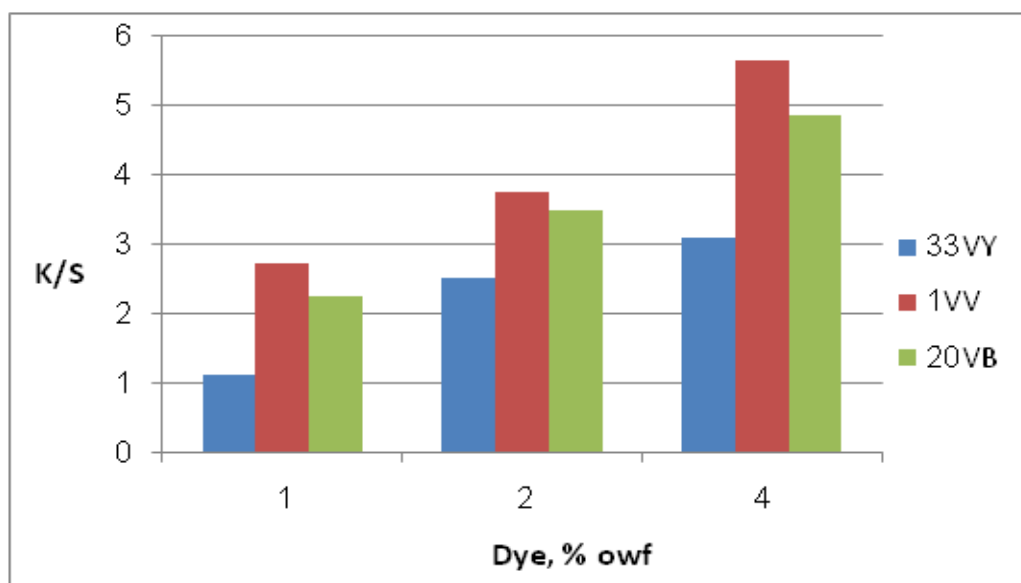


Fig. 7. K/S values at different dye concentrations, 20g/L sodium hydrosulphite concentrations and at 120 °C dyeing temperature for carbocyclic vat dyes on PET fabric.

In the context of the color coordinates (Tables 8 and 9) recorded for both dyed PET and PA fabrics. From both it should be noticed also that lightness (L^*), hue (h) and chroma (C^*) of both dyeings using carbocyclic vat dyes becoming dark and deep at 4% dye concentration compared those obtained at 1 and 2% dye concentration.

Vat dyeing of PA/PET blend fabric

By virtue of the importance of one-step one-stage dyeing process of blended fabrics, the present work was extended to investigate the dyeing behavior of the three carbocyclic vat dyes, listed in Table 1, when applied to a blended PA/PET fabric under the same dyeing conditions described above. Comparison of dyeing behavior and colorimetric coordinates of this series

towards a blended fabric in a blend ratio of 20:80 was illustrated in Figure 8 and Table 10. The K/S results of the dyed blend fabric were to be an expected result as it is found lower than those of 100% PA fabric. As the PA proportion reduces in the blend, the dye substantivity to the blend decreases, resulting in lower K/S of the blend fabric. In the case of colorimetric coordinates, listed in table 10, the color values, namely lightness L^* and chroma C^* of the blended fabric exhibited a little lower L^* values as well as higher C^* than those of PET fabric. It appears that these colorimetric parameters are almost affected by the PA component in the blend fabric. Other color values, namely a^* , b^* and h are in agreement with the expected substantivity of the applied dyes.

TABLE 8. Color coordinates of dyed PA fabric using carbocyclic vat dyes with different dye conc. (1, 2 and 4% owf), 20g/l sodium hydrosulfite and at 120°C for 60 min.

Carbocyclic vat dyes	Dye Conc., % owf	L^*	a^*	b^*	C^*	h
VY33	1	76.46	-0.90	24.43	24.45	92.11
	2	72.50	3.32	33.36	33.52	84.32
	4	69.44	6.98	47.41	47.93	81.63
VV1	1	43.61	19.81	-25.81	22.54	307.51
	2	35.25	18.56	-28.26	23.81	303.29
	4	23.91	15.71	-23.81	28.52	303.42
VB20	1	33.93	2.22	-25.31	13.93	275.01
	2	28.45	1.77	-19.82	14.02	277.96
	4	26.21	2.98	-15.86	19.14	280.63

TABLE 9. Color coordinates of dyed PET fabric using carbocyclic vat dyes with different dye conc. (1, 2 and 4% owf), 20g/l sodium hydrosulfite and at 120°C for 60 min. 120°C.

Carbocyclic vat dyes	Dye Conc., %owf	L^*	a^*	b^*	C^*	h
VY 33	1	88.34	-3.85	22.23	22.56	99.81
	2	85.71	-2.06	30.47	30.54	93.87
	4	78.55	2.51	30.82	30.92	85.34
VV1	1	71.78	18.43	-10.22	21.07	330.98
	2	60.47	23.79	-14.8	28.04	328.07
	4	38.25	22.25	-22.23	31.45	315.04
VB20	1	59.7	6.21	-12.19	16.4	292.24
	2	48.12	8.66	-13.28	16.42	306.03
	4	40.81	10.60	-14.19	17.72	306.75

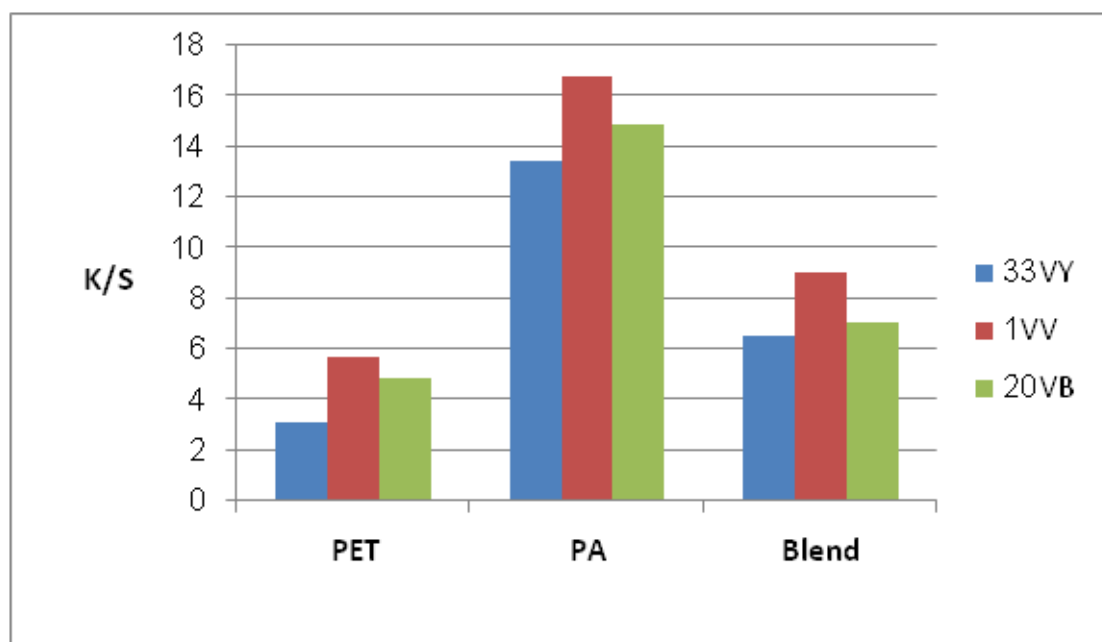


Fig. 8. K/S values of carbocyclic vat dyes on PET, PA and PA/PET blend fabrics at dye concentrations 4% owf, 20g/l sodium hydrosulfite and 120°C dyeing temperature for 60 min.

TABLE 10 Color coordinates of dyed PET, PA and PA/PET blend fabrics using carbocyclic vat dyes at dye concentrations 4% owf, 20g/l sodium hydrosulfite and 120°C dyeing temperature for 60 min.

Carbocyclic vat dyes	Dyed sample	L*	a*	b*	C*	h
VY 33	PA	69.44	6.98	47.41	47.93	81.63
	PET	78.55	2.51	30.82	30.92	85.34
	PA/PET blend	73.46	5.90	54.21	41.53	83.79
VV1	PA	23.91	15.71	-23.81	28.52	303.42
	PET	38.25	22.25	-22.23	31.45	315.04
	PA/PET blend	30.07	20.46	-22.62	30.81	314.13
VB20	PA	26.21	2.98	-15.86	19.14	280.63
	PET	40.81	10.60	-14.19	17.72	306.75
	PA/PET blend	34.67	6.47	-14.34	18.19	304.81

Fastness properties of vat dyed PA, PET and PA/PET blend fabric

The wash, perspiration and light fastness of PA, PET and PA/PET blend fabric dyed with the three carbocyclic vat dyes are listed in Table 11.

The wash and perspiration fastness ratings secured for vat dyes were very good to excellent for all dyed fabric. In addition, the light fastness of vat dyed samples was also exactly same as that of the PA, PET and PA/PET blend fabric.

TABLE 11 Fastness properties of PET, PA and PA/PET blend fabrics dyed with selected carbocyclic vat dyes

Dyed sample	Dye used	Washing Fastness*	Perspiration Fastness*								light	
			Acidic			Alkaline						
			Alt	SC	SP	Alt	SC	SP	Alt	SC		SP
PA	VY33	4	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	6
	VV1	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4	6-7
	VB20	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	3-4
PET	VY33	4	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	6
	VV1	4-5	4-5	4	4-5	4-5	4	4-5	4-5	4-5	4	6-7
	VB20	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4
PA/PET blend	VY33	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	6
	VV1	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	6
	VB20	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4	4

Conclusions

Dyeing behavior and color fastness properties of selected carbocyclic vat dyes on PET and PA fabrics have been investigated. It has been clear that the selected carbocyclic vat dyes possess the property of dyeing both PET and PA components of PA/PET blend fabrics at certain applied acid leuco exhaust dyeing conditions. The best appropriate conditions for the dyeing PET and PA fabrics were 20g/L sodium hydrosulphite concentrations and 120 °C dyeing temperature for 60 min. These carbocyclic vat dyes were found to dye the PA component more deeply than the PET component. The K/S values on PET and PA fabrics were good to very good and the color fastness properties were very good to excellent due to that, at the certain applied acid leuco exhaust dyeing conditions, the greater dye reduction and/or penetration of the acid leuco carbocyclic vat dyes in both PET and PA fabrics. Further study is still needed to get one-tone effect on PA/PET blend not by decreasing the K/S values on PA component, but by increasing the K/S values on PET component. Also it has been clear that the acid leuco heterocyclic vat dyes was un-

stable and return to the corresponding insoluble heterocyclic vat dye form, which would exhibit much lower substantivity towards both PET and PA fabrics, resulting in much lower K/S values. This phenomenon will be also the subject of a future work.

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