Low-Temperature Carrier Dyeing of Poly(vinyl chloride) Fibers with Disperse Dyes

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ABSTRACT: The effects of various carriers on the color yield of poly(vinyl chloride) (PVC) filaments in a disperse dyeing system were investigated. The dyeability of disperse dyes on PVC fiber was improved using appropriate carriers. The enhancement of dye uptake imparted by carriers coincided with the reduction in glass-transition temperature, which in turn coincided approximately with the similarity of the inorganic/organic ratio values between PVC fibers and carriers. The results clearly demonstrate that hydrophobic carriers function as diluents of PVC fiber, facilitating dye diffusion by increasing segmental mobility of the fiber. The dyed PVC filaments with disperse dyes showed reasonable levels of build-up and color fastness properties in the carrier-assisted dyeing. © 2003 Wiley Periodicals, Inc. J Appl Polym Sci 90: 3896–3904, 2003

Key words: carriers; poly(vinyl chloride) (PVC); dyes/pigments; fibers; inorganic/organic ratio (IOR)

INTRODUCTION

Poly(vinyl chloride) (PVC) was invented in 1838, but its commercial exploitation had to await the discovery of suitable solvents and spinning techniques.[1] PVC was particularly appealing, being available in large quantity at relatively low cost. A mixture of carbon disulfide and acetone was found to be a suitable solvent for dry-spinning normal vinyl chloride homopolymer and this product was developed post-1945, especially in France and Germany by the Rhodiaceta companies (as "Rhovyl," "Thermovyl," and associated products) and in Japan. Reasonably strong hydrophobic fibers of attractive handle and appearance were obtainable but textile development was restricted by thermal shrinkage and poor dyeability at moderate temperatures. Shrinkage could be minimized by postheat relaxation of the stretched fiber at the expense of tensile properties but this approach did not provide an entry into mass markets. The principal fiber attributes were flame resistance and good chemical resistance, which formed the basis of some industrial and technical applications. In the apparel and household textile field, some uses were found where the low flammability and/or shrinkage potential could be put to use (e.g., simulated fur fabrics).[2]

More recently produced heavy decitex yarn prepared from a copolymer of vinylchloride and chlorinated vinylchloride has been used in wigs, for which low flammability and human hair–like character such as insulating property and subdued luster attributed to its irregular cross section have made it particularly suitable. However, PVC fibers are not dyeable by the conventional exhaust or padding processes because it does not have any particular dyeing sites such as ionic groups: vat dyes and ionic dyes (e.g., acid dye, metal complex dye, and chrome dye) show low affinity for PVC fibers.[3] Disperse dyes show better dyeing properties than ionic dyes, but still have limited dyeability in the dyeing temperature below 75°C. Also, in spite of relatively low crystallinity (about 10%) compared to that of poly(ethylene terephthalate) (PET), a high-temperature dyeing method with disperse dyes cannot be applied to PVC because of substantial shrinkage and deformation on heating. This constraint of poor dyeability of PVC led to exploration of an alternative process—mass coloration—which is defined as a method of coloring a manmade fiber by incorporation of the colorant before the filament is formed. Mass coloration has several advantages: qualitative, ecological, and economic. The economic advantage of mass coloration may be attained mainly by large production of commercial articles of the same color. However, this fact limits the use of PVC mass coloration in textile articles, the marketing of which requires a quick response to fashionable shades.
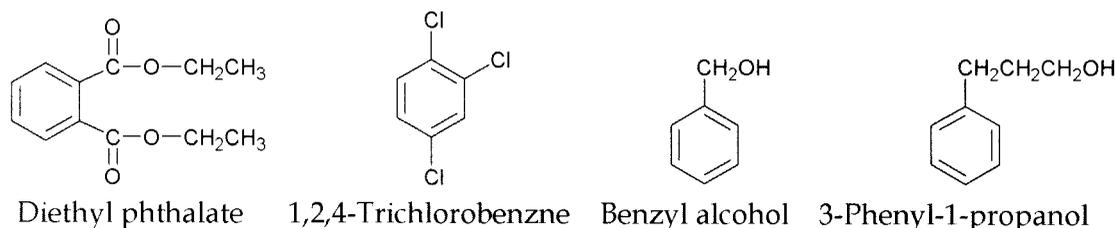
Figure 1 Molecular structure of various carriers used in this study.

Recently, one coauthor (I.S. Kim) reported on the low-temperature disperse dyeing of PVC with carrier.[4] In this study, we investigated the effect of various carriers on the color yield and the reduction of the glass-transition temperature (T_g) of PVC filaments to clarify the function of carriers in a disperse dyeing system.

EXPERIMENTAL

Materials

A commercial sample of 50 denier PVC filament (supplied by Woo-No Fiber Co., South Korea) was used. The filament was scoured in a weakly alkaline aqueous solution, rinsed using water, and dried at room temperature. Diethyl phthalate (DEP), 1,2,4-trichlorobenzene (TCB), benzyl alcohol (BZA), and 3-phenyl-1-propanol (PHP) were used as carriers (see Fig. 1 for their molecular structures). All carriers were laboratory-grade reagents. The disperse dye used in this work was C.I. Disperse Red 60, whose structure is depicted in Figure 2.

Dyeing

PVC filaments were dyed in a laboratory dyeing machine (Ahiba Nuiance, Datacolor International, Switzerland) at a liquor ratio of 30 : 1. A dye bath (60 mL) was prepared with the appropriate amount of a carrier and a disperse dye and adjusted to pH 4.5–5.0 using sodium acetate (1.2 g/L) and acetic acid. A PVC filament (2.0 g) was immersed in the dye bath and dyed at various temperatures to determine the optimum dyeing temperature. PVC filaments that had been dyed with disperse dyes were reduction cleared
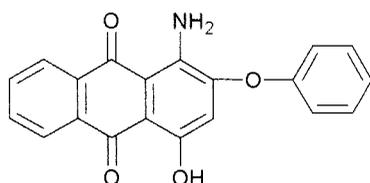
(Na$_2$S$_2$O$_4$ 2.0 g/L, NaOH 2.0 g/L, Sandozin NIE 2.0 g/L) for 15 min at 40°C. Dyestuff uptake on the PVC filament was determined by extracting the dye from the filaments with DMF at room temperature. The build-up property of disperse dye on PVC was also investigated by measuring K/S values [eq. (1)] of dyed filaments at various dye concentrations, defined as

$$K/S = \frac{(1 - R)^2}{2R} \qquad (1)$$

where R is the reflectance of an infinitely thick layer of material illuminated with light of a known wavelength, K is the absorption coefficient, and S is the scattering coefficient. The function K/S is directly proportional to the concentration of colorant in the substrate.[5]

Carrier treatment and T_g determination

PVC filaments were pretreated in the presence of various concentrations of different carriers and the T_g of the carrier-pretreated fiber was measured using a differential scanning calorimeter (DSC 2920, TA Instruments, New Castle, DE). For reasons of clarity, only the carrier (without dyestuff and surfactant) was treated in the investigation.

The carrier treatments were carried out for 60 min at 50°C in a laboratory dyeing machine. The treated filament was then removed, rinsed in distilled water, and dried in an oven, both at 40°C. After the treatment, the filament was dissolved in 20 mL of DMF at room temperature and the light absorbance of the solution was measured. The concentration of the carrier was calculated by reference to the optical density of the carrier at λ_{max} in the solvent.

Adsorption isotherm

The dye bath (60 mL) was prepared with 2.0 g/L of diethyl phthalate and various amounts of disperse dye (C.I. Disperse Red 60). PVC filaments were dyed for 60 min at 50°C in a laboratory dyeing machine at a liquor ratio of 1000 : 1. The dyeing isotherm curve was de-
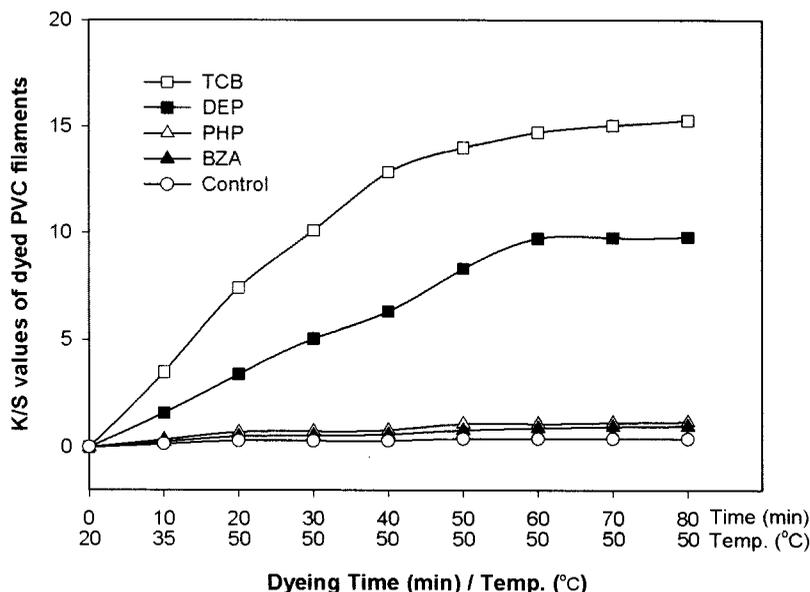

Figure 2 Molecular structure of the disperse dye (C.I. Disperse Red 60) used in this study.

Figure 3 Dyeing rate of PVC filaments with C.I. Disperse Red 60 (3.0% owf) in the presence of various carriers (5.0 g/L) expressed in terms of K/S values.

termined from the amount of dye applied ($[D]_b$, g/L) and the dye uptake values ($[D]_f$, g/g), which were estimated by extraction and spectrophotometry, respectively.

Color fastness

PVC filament was dyed [1.0 and 3.0% owf (on the weight of fiber)], reduction cleared, and heat-set (100°C, 60 s) to test the color fastness. The color fastness was determined according to the International Organization for Standardization (ISO). The specific tests used were ISO 105 C01 (color fastness to washing), ISO 105 E04 (color fastness to perspiration), ISO 105 X12 (color fastness to rubbing), and ISO105 B02 (color fastness to light); fastness tests were designed to establish the resistance to a wide range of aqueous agencies, including distilled water and perspiration and various other situations (e.g., rubbing, daylight) relevant to different end uses of the goods. For example, the washing fastness test is directed toward simulation of the conditions likely to be encountered in normal use. A multifiber strip of identical size is stitched to the dyed sample and the composite specimen is agitated in a solution with a defined concentration of detergent and, where appropriate, other prescribed additives. Controlled agitation is maintained at the temperature required by the test conditions and the ratio of liquid volume to mass of fabric is also defined. At the end of the testing the sample is removed and rinsed, and the components separated and allowed to dry. A visual assessment is then made for the staining and change in color using gray scales. In

the case of light fastness, the specimen was faded to the equivalent effect on the pattern of a grade 3 contrast on the gray scales and the degree of contrast between the exposed and unexposed pattern was compared with that for the standards; the rating given is the number of the standard that exhibits the same degree of contrast.[5]

RESULTS AND DISCUSSION

Dyeing properties

The term "carrier" describes a type of accelerant particularly used in the dyeing or printing of hydrophobic fibers with disperse dyes.[6] Several theories have been proposed to explain carrier action. In essence, the various mechanisms can be considered to support one of two main theories of carrier action: (1) that the carrier has an effect on the disperse dye in the dye bath or (2) that the carrier has an effect on the fiber. At present, it is generally considered that carrier action is attributable to the latter effect.[7]

The K/S values of dyed PVC filament with carriers at various dyeing temperatures showed the expected increase in the K/S values with increase in temperature. However, above the temperature of 60°C, the deformation of PVC filaments attributed to shrinkage was observed. Therefore, considering both color yield and dimensional stability, 50°C was chosen for the carrier dyeing temperature of PVC filaments in this investigation. The dyeing behavior of PVC filaments at 50°C in the presence of various carriers as dyeing proceeds is shown in Figure 3, which clearly demon-
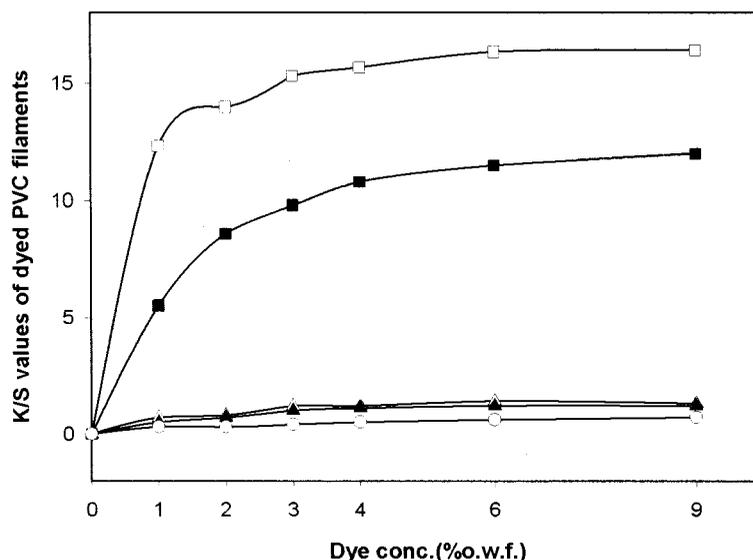
Figure 4 Build-up properties of C.I. Disperse Red 60 on PVC filaments in the presence of various carriers (dyed for 60 min at 50°C; see Fig. 2 for key).

strates the marked plasticizing action of TCB and DEP on the PVC filaments, as evidenced by the increased K/S values of the substrate compared with those of the dyed PVC filament without carriers, whereas BZA and PHP showed little effect on the dyeability of PVC filaments. In the case of TCB and DEP, the K/S values of dyed filaments increased as dyeing proceeded and finally reached saturation in about 40 min of dyeing time at 50°C.

The K/S values of the dyed PVC filaments at various pH values showed little difference, irrespective of dyeing pH, presumably because both PVC and disperse dyes have no ionic sites that are sensitive to the pH of the dye bath. In view of this result, it is proposed that a disperse dyes–PVC dyeing system is stable to pH conditions. However, in this study, dye baths were set at the pH of 4.5–5.0 in which disperse dyes are known to be most stable.

The build-up property of C.I. Disperse Red 60 on PVC filaments is shown in Figure 4. The color strength of dyed PVC filaments reached saturation at the dye concentration of 6.0% owf, which gave a K/S value of about 16.3 for TCB and 11.5 for DB, respectively. However, the PVC filaments dyed in the presence of BZA or PHP showed very weak color strength even at the dye concentration of 9.0% owf.

Attempts were made to evaluate the interaction of carrier with PVC with which carrier reduced the T_g of PVC. It is believed that carriers function by plasticization of the compact structure of fibers and this effect is reflected in the reduction of fiber T_g. When the T_g of the fiber was plotted against each carrier, the difference in carrier efficiency was considerable; DEP and TCB reduced the T_g of PVC by 15.9 and 16.4°C, respectively, whereas no effect was observed for BZA

and PHP. The T_g gives an indication of the temperature region over which the rate of diffusion of dye into the fiber becomes significant. Thus, any lowering of the T_g of the fiber in a dye bath will result in faster diffusion of dye, as previously shown in Figures 3 and 4. Clearly, hydrophobic carriers (DEP and TCB) are considerably more efficient than hydrophilic carriers (BZA and PHP) for T_g reduction. BZA and PHP showed little effect on the color yield, probably because they are not sufficiently adsorbed at the fiber because of their strong interaction with water. From the overall results, it is proposed that the uptake of the disperse dyes on PVC is also dependent on T_g reduction, as was observed in the case of PET and polyacrylonitrile (PAN), and that the availability of the dye sites increases with increasing segmental mobility.

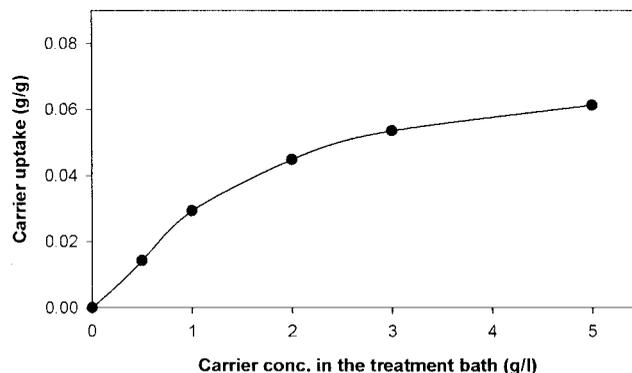Figure 5 shows the DEP uptake (g/g) on PVC filaments depending on the amounts of carrier applied

Figure 5 Correlation between the concentration in the treatment bath and DEP uptake on PVC filaments (diethyl phthalate was treated for 60 min at 50°C).
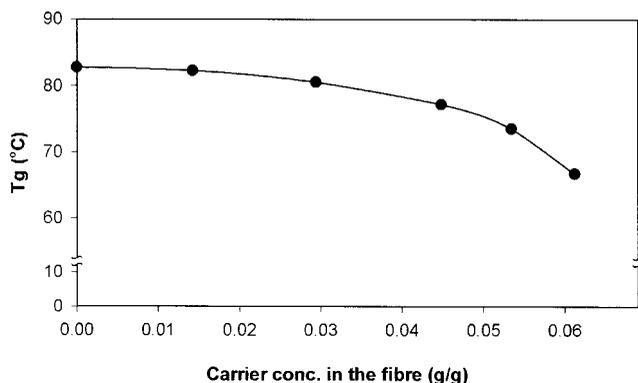
Figure 6 Effect of concentration of diethyl phthalate on the T_g of PVC filaments.

(g/L). The carrier uptake increased slowly with the increase of the amount of carrier applied and it seems to reach saturation at the concentration of 5.0 g/L. Figure 6 shows the effect of concentration of DEP on the T_g of PVC filaments. The T_g decreased with increasing carrier concentration in the fiber. This observation is in agreement with observations obtained by Thorton (plasticization of Acrilan filament by phenol)[8] and by Fujino and Walter (decrease in T_g of polyester fiber with increasing concentration of plasticizers within the fiber).[9] Because plasticization is reflected in the reduction of fiber T_g, which in turn is related to the concentration of the plasticizer present, the observed reduction in the T_g of PVC must be related to the concentration of the carrier adsorbed by the fiber. Thus it can be concluded that the carrier acts as a diluent of the substrate and the extent of carrier action (i.e., plasticization) increases with increasing concentration of carrier in the fiber.

The solubility parameter concept, initially developed to predict the ease of mixing of nonpolar liquids, has been extended to embrace polar solvents and polymers, as well as the mixing of fibers with dyes and carriers. In the context of the present study, the solubility parameter essentially indicates the potential of interaction between a polymer and a carrier. Two components will be miscible if their solubility parameters coincide. The application of this theory in the context of fibers has been discussed by Ingamells.[10] Table I presents the various solubility parameters of PVC and four carriers. The solubility parameters were calculated from the solubility parameter component group contributions and molar volume component group contributions using eqns. (2)–(6) (method of Hoftyzer and Van Krevelen).[11]

$$\delta_d = \frac{\sum F_{di}}{V} \tag{2}$$

$$\delta_p = \frac{\sqrt{\sum F_{pi}^2}}{V} \tag{3}$$

$$\delta_h = \sqrt{\frac{\sum E_{hi}}{V}} \tag{4}$$

$$\delta_a^2 = \delta_p^2 + \delta_h^2 \tag{5}$$

$$\delta_t^2 = \delta_a^2 + \delta_d^2 \tag{6}$$

where V is the molar volume; F_{di} is the group contribution to the dispersion component of the molar attraction constant; F_{pi} is the group contribution to the polar component of the molar attraction constant; E_{hi} is the group contribution of hydrogen bonding component of the cohesive energy; and δ_t, δ_d, δ_a, δ_p, and δ_h are the total, dispersion, association, polar, and hydrogen bonding solubility parameters, respectively.

From the solubility parameter concept, any possible dependency between the solubility parameters of the carriers and T_g reduction or color yield in PVC filaments should be apparent from the values quoted in Table I. In fact, when the solubility parameters of PVC and the four carriers used in this work were compared with their color yield expressed in terms of K/S values brought about by each carrier, no correlation could be found (Table I and Figs. 7–9): the δ_t value of DEP was considerably lower than that of PVC and the other carriers used (Fig. 9). According to the theory proposed by Ingamells et al., DEP should show the least color yield, whereas it showed better dyeability than PHP and BZA, which have solubility parameter values closer to that of PVC.

Lemons et al.[12] proposed that PET–carrier interactions occur primarily by means of dispersion forces; similarly, Derbyshire and Peters[13] suggested that carriers were adsorbed onto PET primarily by virtue of nonpolar forces operating between the aromatic regions in both carrier and fiber, although with carriers, such as phenol, that carry polar substituents, polar forces may also contribute to adsorption. Support for this view was furnished by the findings of Ingamells et al.[14–16] that plasticization of PET fibers is dominated by dispersion forces with polar forces playing a small part in the process. Brown and Peters[16,17] observed no detectable shifts in the infrared absorption spectra of PET, which had been treated in various chloroben-

TABLE I
Three-Dimensional Solubility Parameters of PVC and Carriers (J/cm³)^(1/2)

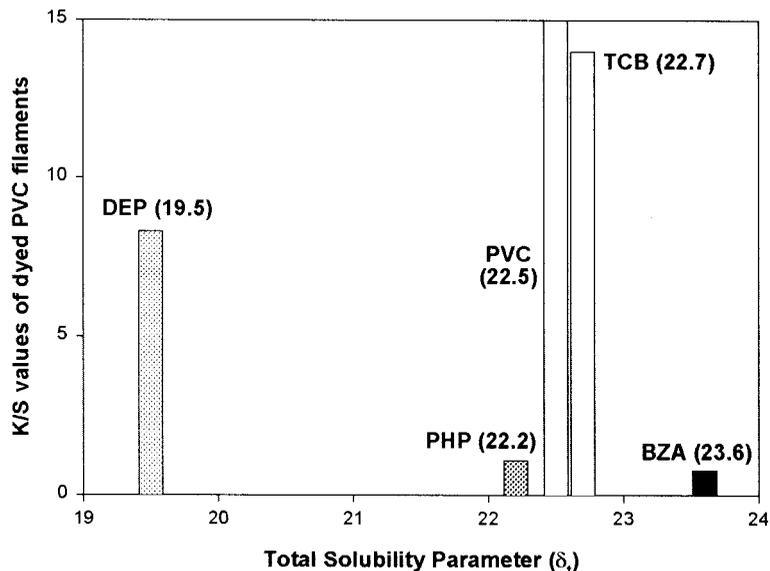|	δ_t	δ_a	δ_d	δ_p	δ_h
PVC	22.5	11.7	19.2	9.2	7.2
1,2,4-Trichlorobenzne	22.7	8.3	21.1	7.7	3.1
Diethyl phthalate	19.5	8.6	17.3	1.8	8.4
Benzyl alcohol	23.6	14.7	18.4	4.9	13.9
3-Phenyl-1-propanol	22.2	12.8	18.1	3.8	12.2

Figure 7 Relationship between δ_t of carriers and dye uptake.

zenes, and concluded that only van der Waals forces were operative.

However, in this investigation on carrier–PVC interaction, Table I and Figures 7–9 clearly show the complete absence of correlation between the dyeability of PVC fibers and the similarity of solubility parameters (δ_t, δ_a, and δ_d) of PVC and four carriers. Because no clear correlation was observed in the case of PVC fiber, an alternative explanation of the findings is necessary.

Although relatively little work has focused on the nature of the forces of interaction that contribute toward carrier–fiber substantivity, it seems reasonable

to suggest that the substantivity of carriers toward the hydrophobic fibers accrues from the various forces of interaction that have been proposed to account for the substantivity of disperse dyes toward such fibers. From Figures 3 and 4, it is noteworthy that hydrophobic carriers showed better efficiency in T_g reduction and improvement of dyeability than hydrophilic carriers. Actually, it has been demonstrated that hydrophobic carriers are more effective on PET fibers than their hydrophilic counterparts[18] and that the more hydrophobic a carrier, the larger is its partition coefficient on PET[19] and PAN[20] fibers. The distribution of the carrier between the liquor and the fiber plays a
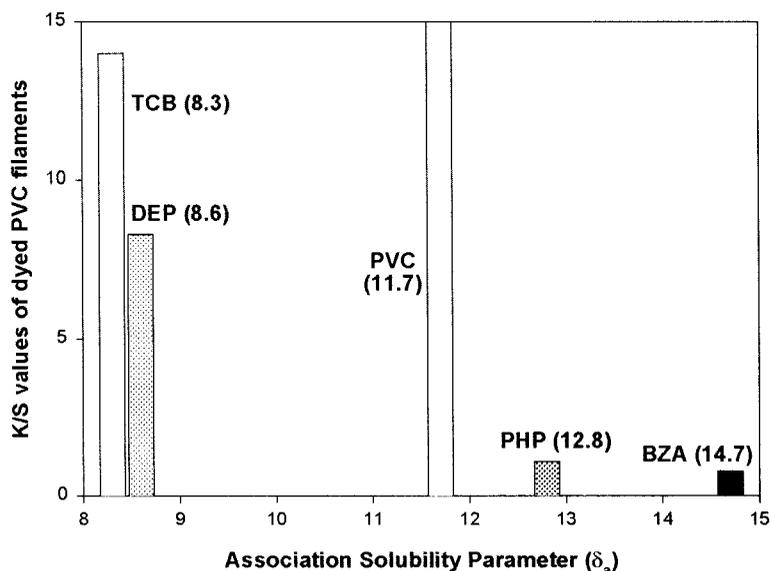

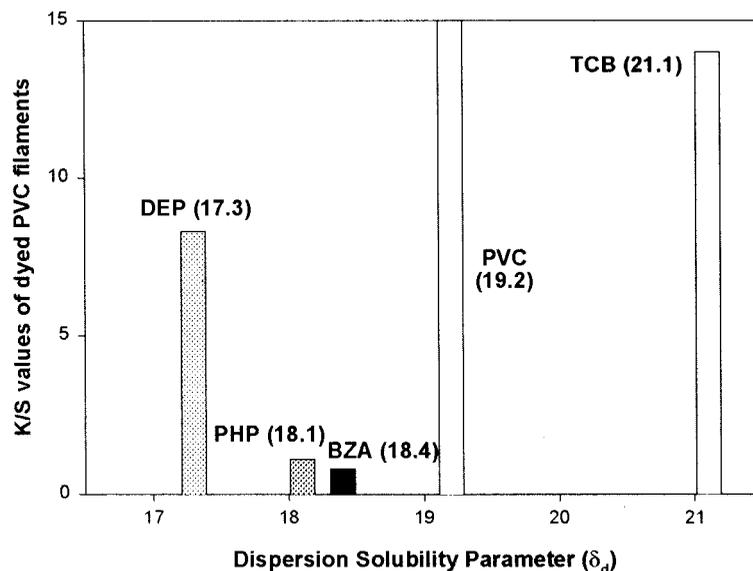**Figure 8** Relationship between δ_a of carriers and dye uptake.

Figure 9 Relationship between δ_d of carriers and dye uptake.

decisive role in reduction of T_g and is dependent on the structure of the carrier.[21] Therefore, the IOR (inorganic/organic ratio) concept may shed light on the findings so far in this investigation.

The IOR concept was introduced by Fujita[22,23] and is capable of predicting an organic compound's general properties by means of a numerical value [eq. (7)[rsqb]:

$$IOR = \frac{Inorganity}{Organity} \qquad (7)$$

where "Inorganity" is determined by the radicals with inorganic character and "Organity" is determined by

the radicals with organic character. All organic compounds can be regarded as an extension of the simplest compound, methane, taken as the first ancestor, or the derivation of methane after its hydrogen atom(s) are substituted by other atom(s) or group(s). The IOR method assigns the value of 20 to each carbon atom representing elementary hydrocarbon (–CH₂–). Thus, the organity of a compound can be expressed by the multiplication of 20 and the number of carbon atoms. This method also assigns the value of 100 to the hydroxyl group in correspondence with the organitic value of a carbon atom. The value of other substituting groups and abnormal parts are then decided on the basis of the values of methylene and hydroxyl groups.
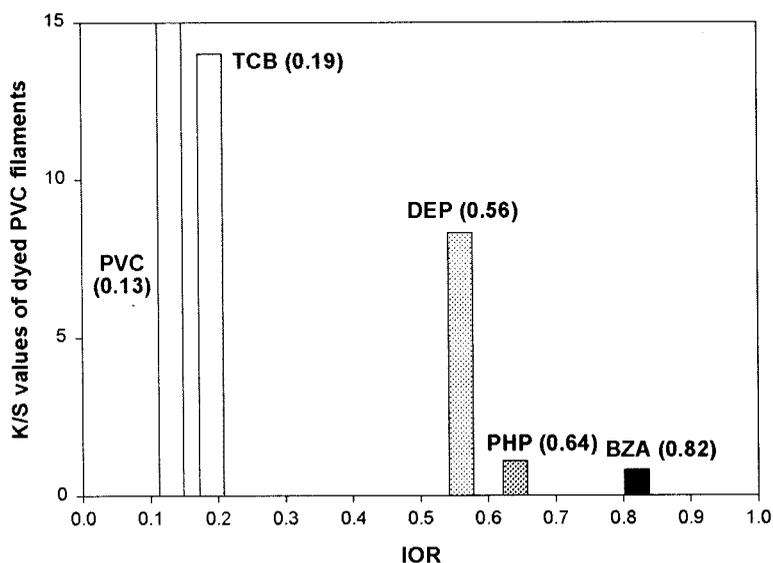

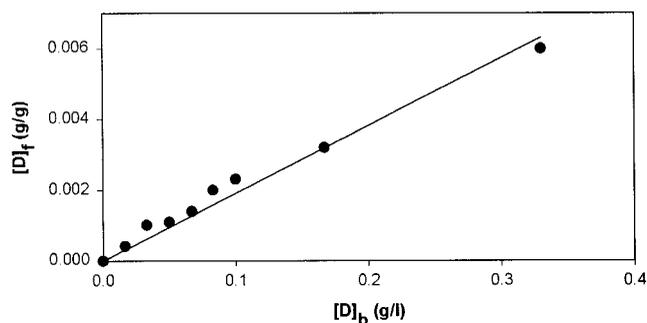**Figure 10** Relationship between IOR of carriers and dye uptake.

Figure 11 Adsorption isotherm of C.I. Disperse Red 60 on PVC filaments at 50°C.

This technique, which has been used to predict the suitability of a dye for a given fiber, could be used to estimate the suitability of a carrier for PVC fiber. Because the IOR values represent the hydrophilic/hydrophobic character of compounds, the closer the IOR values of different compounds, the closer are their hydrophilic/hydrophobic characters. Figure 10 shows that maximum plasticization and dyeability of PVC occurred when the IOR value of the carrier coincided with that of the fiber; the IOR values of TCB or DEP, which showed better dyeability, were closer to that of PVC than to that of either PHP or BZA. Because the IOR values of the carriers are similar to that of PVC, this indicates that the carrier and fibers have similar hydrophilic/hydrophobic characters and thus the carrier should exhibit substantivity toward PVC fibers. Consequently, in view of the results shown in Figures 7–10, it is proposed that the plasticization efficiency of the carrier is related to the similarity in the hydrophilic/hydrophobic characters rather than the solubility parameter between fibers and carriers in carrier–PVC fiber interaction.

It is also necessary to consider the effect of the presence of functional groups (e.g., polar groups) on the plasticization of carriers: the polar groups of carriers, such as TCB, render the molecules more polar and favor their penetration in a polar polymer like PVC, given that a carrier–polymer interaction is similar to a polymer–polymer interaction. However, in nonhydrogen-donating media like PVC, alcohols (BZA and PHP) did not act as efficient plasticizers of PVC, probably because of the hydroxyl group that is capable of forming hydrogen bonds.[24]

In Figure 11 the adsorption isotherm of C.I. Disperse Red 60 on PVC filaments at 50°C is shown. A linear isotherm (i.e., a Nernst isotherm) was obtained for the adsorption of disperse dyes on PVC, which demonstrates that the adsorption of disperse dyes onto PVC fiber occurs by a mechanism identical to that of the adsorption of disperse dyes onto PET. The Nernst, or linear partition, adsorption isotherm takes the form

$$[D]_f = K[D]_s \qquad (8)$$

where K is a constant at constant temperature, $[D]_s$ is the concentration of dye in solution, and $[D]_f$ is the concentration of dye on the fiber.

A plot of $[D]_f$ against $[D]_s$ is linear according to the equation. This isotherm equation is a mathematical representation of the distribution law, which states that a dissolved substance, irrespective of its total amount, distributes itself between two layers or phases in a constant concentration ratio, at constant temperature.[25]

Color fastness

Tables II–IV show the results of color fastness to washing, perspiration, rubbing, and light, respectively. Overall color fastness ratings of dyed PVC filaments were generally moderately good to excellent; the ratings of color fastness to washing were in the range of 4 to 5, irrespective of the dye concentration applied. The ratings of color fastness to alkali perspiration and to acid perspiration were in the range of 4 to 5 for 1.0% owf of dye concentration and 3–4 to 5 for 3.0% owf of dye concentration, respectively. Color fastness to light

TABLE III
Color Fastness to Perspiration (ISO 105 E04)

Dye concentration (‰ wf)	Change	Staining					
		Wool	Acryl	PET	Nylon	Cotton	Acetate
1.0							
Acid	4/5	5	5	5	4	5	4
Alkali	4/5	5	5	5	4	5	4
3.0							
Acid	4/5	5	5	4/5	3/4	5	4/5
Alkali	4/5	5	5	4/5	4	5	4

TABLE II
Color Fastness to Washing (ISO 105 C01)

Dye concentration (‰ wf)	Change	Staining					
		Wool	Acryl	PET	Nylon	Cotton	Acetate
1.0	5	5	5	5	4/5	5	4
3.0	5	5	5	5	4/5	5	4

TABLE IV
Color Fastness to Light (ISO 105 B02) and Rubbing (ISO 105 X12)

Dye concentration (‰ wf)	Light fastness	Rubbing fastness	
		Dry	Wet
1.0	3/4	4/5	4/5
3.0	3/4	4/5	4

of dyed PVC filaments was 3–4 and color fastness to rubbing was in the range of 4 to 4–5.

CONCLUSIONS

The present work suggests that it is technically feasible to use carriers in PVC dyeing with disperse dyes to improve the dyeability of PVC fibers.

The presence of an appropriate carrier reduced the T_g of the fiber in terms of a diluent effect, which in turn increased color yield resulting from the enhanced segmental mobility of fibrous polymer chains. The extent to which carriers enhance the uptake of disperse dyes was proportional to the amount of carrier adsorbed by the fiber. Of the carriers applied in this investigation, only hydrophobic carriers such as diethyl phthalate and 1,2,4-trichlorobenzene showed good carrier effect. In line with this, it was found that the extent of carrier uptake and dyeability of PVC fiber was related to the IOR value, that is, hydrophilic/hydrophobic characters, rather than the solubility parameters of the carriers. Also, it is noteworthy that, in nonhydrogen donating media like PVC, alcohols (BZA and PHP) did not act as efficient plasticizers of PVC, probably because of the hydroxyl group that is capable of forming hydrogen bonds.

The absorption of disperse dyes onto PVC fibers was found to follow a Nernst mechanism that is identical to that of the adsorption of disperse dyes onto PET.

The dyed PVC filaments with disperse dyes showed reasonable levels of build-up and color fastness in the carrier-assisted dyeing.

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