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Dyeing and Fastness Properties of Disperse Dyes on Poly(Lactic Acid) Fiber

Jantip Suesat^{1,2} and Potjanart Suwanruji³

¹Department of Textile Science, Faculty of Agro-Industry, Kasetsart University,

²Center of Advanced Studies for Agriculture and Food,

KU Institute for Advanced Studies, Kasetsart University

³Department of Chemistry, Faculty of Science, Kasetsart University,
Thailand

1. Introduction

Poly(lactic acid) or PLA is an aliphatic polyester being considered as a green material due to its natural-based origin and biodegradability properties. Lactic acid obtained from the fermentation of sugar and vegetables e.g. corn and cassava is used as a monomer for PLA polymerization. Production of PLA polymer can be achieved by 2 major synthesis routes viz., direct condensation polymerization of lactic acid and ring-opening polymerization of lactide, a cyclic dimer of lactic acid, yielding poly(l-lactic acid), poly(d-lactic acid) or poly(d,l-lactic acid) depending on lactic acid isomers employed. The chemical structure of PLA is shown in fig. 1. PLA possesses desired properties required for packaging materials. Major market share of PLA therefore falls in the packaging industry. At the same time, its interesting properties have drawn attention from the textiles industry. An attempt to use PLA as a textile fiber has been pursued with the aim of replacing poly(ethylene terephthalate), PET, fiber with this green polyester fiber. PLA fiber can be produced by both melt and solution spinning processes (Gupta et al., 2007) but the former is used more regularly due to the more eco-friendliness and ease of processing. Thermal degradation of the PLA polymer during melt spinning can be prevented by addition of a thermal stabilizer. The processing of PLA fiber/yarn is one of the important parameters in controlling the properties of PLA. PLA yarns which are formerly passed through different yarn processing possess different physical properties and morphological characteristics, which subsequently influence the accessibility of the chemicals into the fiber during textile wet processing for example, dyeing and finishing (Suesat et al., 2003).

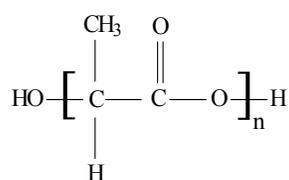


Fig. 1. Chemical structure of PLA

PLA fiber has superior elastic recovery and a slightly higher hydrophilicity as compared with PET. It also exhibits lower flammability and less smoke generation. One of the

important properties influencing dyeing properties of PLA is claimed to be the effect of its lower refractive index. It was informed by NatureWorks, Co., Ltd. that refractive indices of PLA and PET were 1.35-1.45 and 1.50, respectively while Yang & Huda claimed that they were 1.45 and 1.58 for PLA and PET, respectively (Yang & Huda, 2003). The lower refractive index of PLA causes a deeper shade of the disperse dyes obtained on PLA at the same applied dye concentration (Lunt & Bone, 2001). Thermal properties of PLA were reported to be similar to that of polypropylene. The glass transition temperature (T_g) of PLA is 55-65°C. The melting temperature (T_m) of PLA, containing the L- or D-isomeric form alone, is between 171-180°C whereas that of the stereocomplex analogue is 220°C (Perepelkin, 2002). The T_m of PLA is dependent on the molecular weight, thermal history, and isomeric composition of the polymer (Södergård & Stolt, 2002). The most typically used PLA for textile application is poly(*l*-lactic acid) or PLLA. PLA has a lower melting temperature than PET. Fig. 2. shows the DSC scans of the fabrics derived from PLA and PET fibers. The melting temperature of PLA is at 170°C while PET melts at 260°C. This allows PLA to be processed at a lower temperature, for example disperse dyeing of PLA is done at 110°C while PET is dyed at 130°C, heat setting of PLA is carried out at 130°C whilst PET is heat set at 180°C (Phillips et al., 2003). These lower thermal properties are a cause of sensitivity of PLA fabric to high temperatures employed in textile processing and the conditions being experienced during its service life. Exposure to high temperatures could harm the fiber. Therefore, precaution is taken for the textile products obtained from PLA fiber to avoid ironing at high temperatures which can cause fiber damage. Alternatively, PLA is recommended for knitted goods rather than wovens in order to avoid such problems.

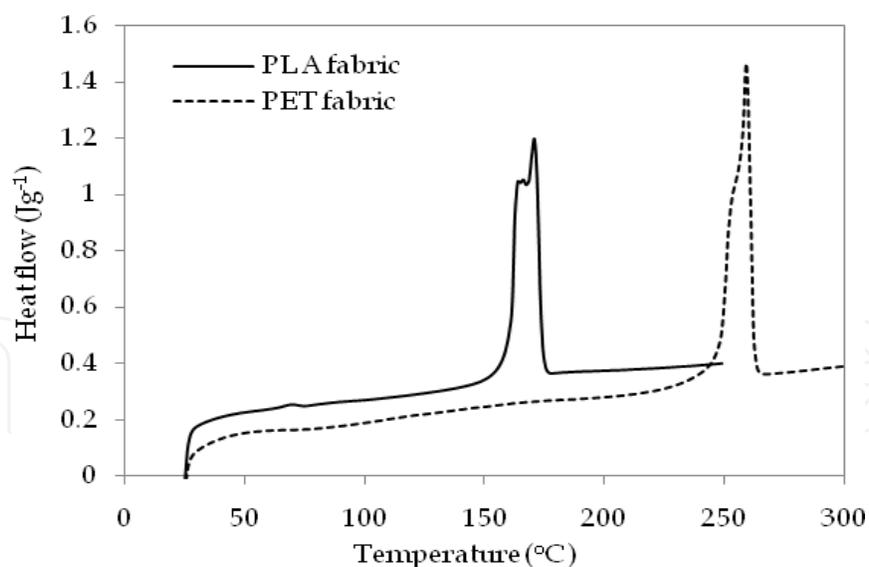


Fig. 2. DSC scans of the knitted fabrics derived from PLA and PET fibers

As PLA fiber is rather thermally sensitive, even the heat generated by scanning electron microscope (SEM) during a measurement performed at 15 kV could cause the fiber to melt and fuse together after being exposed to electron beam within a few seconds as seen in Fig. 3.a), while no damage was observed on PET fiber (Suesat, 2004). The same electron beam damage has also been found on the low melting point polymer such as polypropylene.

Jamshidi et al. claimed that PLA was relatively sensitive to thermal degradation, especially at a temperature higher than 190°C. It was explained that the degradation reactions involved cleavage of the ester bonds on the main chain of the polymer. In addition, the presence of low molecular weight compounds e.g. water, monomers, oligomers, and catalysts in the polymer seemed to influence the reduction of the molecular weight at high temperatures (Jamshidi et al., 1988).

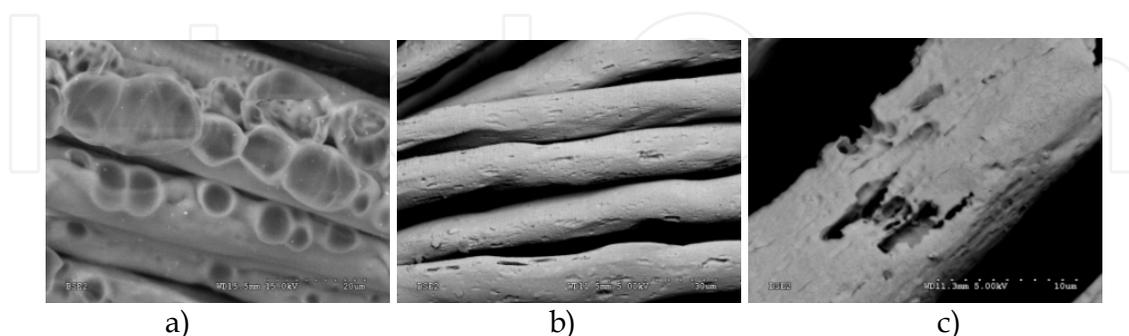


Fig. 3. SEM photographs of damage on PLA fiber caused by ; a) electron irradiation of SEM; b) and c) peroxide bleaching used for PLA/cotton blend

PLA is not only thermally sensitive but it is also sensitive to alkali. It can be destroyed more easily by alkaline hydrolysis than PET. Thus, it can be deteriorated by those using alkaline wet processing in textiles production. Under alkaline conditions, PLA can be damaged by an alkaline hydrolysis reaction. The fiber surface is eroded and its strength is impaired, especially at high temperatures. An example of the alkaline preparation process is peroxide bleaching used to whiten the cotton component in the PLA/cotton blend. The alkaline hydrolysis takes place and the fiber surface is eroded as depicted in Fig. 3.b) and c), resulting in a substantial reduction of the fiber strength (Phillips et al., 2004a). Therefore, the preparation, dyeing and finishing processes for PLA should be milder than those used for PET. It is suggested to process PLA fiber at lower alkalinity, processing temperature and time.

2. Dyeing PLA fiber with disperse dyes

Although PLA fibers exhibit many attributes similar to other synthetic fibers, they are a new category that requires modified dyeing and finishing techniques to maximize their benefits. The dyeing properties of PLA have been investigated, especially in comparison with PET fiber. The dyeing of 100% PLA fiber has been intensively studied (Scheyer & Chiweshe, 1999; Nakamura et al., 2001; Phillips et al., 2003, 2004a, 2004b, 2004c). Owing to its relatively hydrophobic nature like PET, PLA can normally be dyed with disperse dyes. The optimum dyeing conditions for dyeing PLA are 110°C for 30 mins under an acidic pH (pH 5) (Fig. 4.) whereas PET dyeing is normally carried out at 20°C higher (130°C) under a more acidic condition (pH 4) (Phillips et al., 2004b). Disperse dyes which show good dyeing properties on PET do not always provide good dyeability on PLA. According to the study of DyStar (2004), the disperse dyes recommended for dyeing PLA fiber are the medium-energy azo dyes which exhibit a superior degree of exhaustion as compared with other dye types. The disperse dyes based on benzodifuranone structure are not recommended due to their low uptake and poor build-up on PLA, therefore, a heavy depth of shade seems to be commercially infeasible (Phillips et al., 2003).

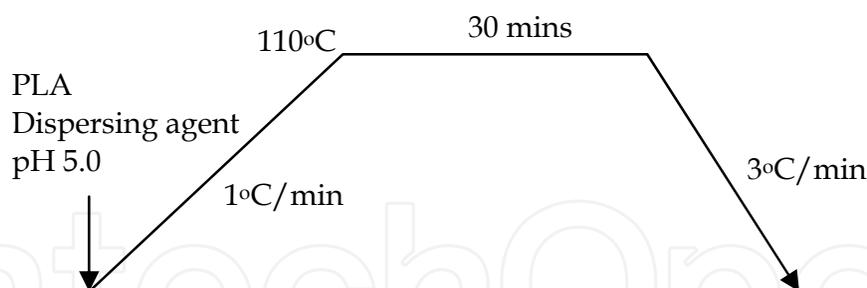


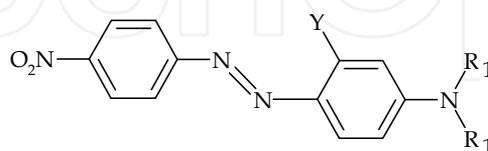
Fig. 4. Typical dyeing profile of PLA with disperse dye

Dyeing of PLA blended fibers has also been given a great attention as PLA blended with other fibers has been developed in order to improve some inferior properties of PLA and to gain a new type of fiber with better characteristics. One important blend is PLA/cotton. Due to a high price of PLA fiber, the blend of PLA/cotton was primarily produced so as to provide more economical PLA-based products with better desired properties. This PLA/cotton blend brought about a subsequent dyeing issue to be figured out. As PLA is alkaline sensitive, it should be noted that the cotton dyeing condition should not be harmful to PLA fiber. Reactive dyeing of cotton involves the use of the alkaline condition for dye fixation, so this should be carefully controlled. Nevertheless, under acidic and neutral conditions, PLA was reported to be severely damaged when it was treated for a longer time at higher temperatures, with neutral conditions exhibiting a more severe effect in deteriorating PLA strength. The hydrolysis reaction taking place in such conditions was reported to occur in a bulk erosion manner whereby hydrolysis degradation of the polymer occurred simultaneously all over the fiber cross section. The hydrolysis mechanism of PLA was said to be strongly pH dependent and it was claimed to undergo bulk erosion under acidic and neutral conditions whereas under concentrated alkaline media, it was dominated by surface erosion (Yuan et al., 2002, 2003; Burkensroda et al., 2002). The effect of preparation and dyeing processes on the strength of PLA contained in the PLA/cotton blend was investigated and it was illustrated that these processes did weaken the PLA fiber but its retained strength after processing was in a commercially acceptable level. However, dyeing PLA/cotton using 1-bath, all-in process with Kayacelon React dyes under neutral conditions at 110°C did ruin the strength of PLA beyond acceptability (Phillips et al., 2004a, 2004c). This experience suggests that the use of higher temperatures or longer times of dyeing can cause degradation of the polymer, an observation confirmed by Kameoka et al. (1997) who claimed that the temperature, time and pH of dyeing resulted in a significant reduction in the molecular weight of the polymer.

2.1 Dyeing and build-up properties of disperse dyes on PLA fiber

Concentrations of disperse dye applied on PLA and PET fibers with the aim to achieve the same level of visual color yield on both fibers were examined and the values found indicates a less amount of dye required for PLA. Table 1 depicts the amount of disperse dyes used for dyeing PLA compared with those used to apply on PET. As seen that in order to gain the same K/S level of 10, a lower amount of % dye applied is required on PLA. One explanation can be made from the lower refractive index of PLA as compared with PET, rendering a deeper shade observed visually. Another important explanation is applied by the findings

from the solvatochromism study of disperse dyes mentioned in our previous work (Suesat et al., 2011). The light absorption capacity of the dye is influenced by polarity of the polymer medium. It was found that the azo disperse dyes exhibited a higher molar extinction coefficient (ϵ_{\max}) when dyed on PLA as compared with PET. Therefore, when the dyes are applied on the two fibers at the same concentration, a more intense color will be observed on PLA, in other words, less dye is needed on PLA so as to gain the same visual color yield compared with PET.

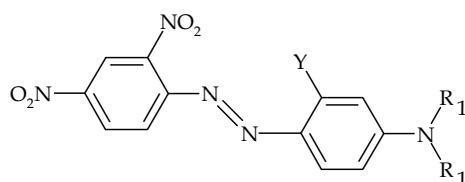


| Dye | Y group | R ₁ group | Concentration applied (%owf) | |
|-----|-----------------|--|------------------------------|--------|
| | | | on PET | on PLA |
| D1 | H | C ₂ H ₅ | 0.16 | 0.12 |
| D2 | H | C ₂ H ₄ OCOCH ₃ | 0.26 | 0.17 |
| D3 | CH ₃ | C ₂ H ₅ | 0.16 | 0.10 |
| D4 | CH ₃ | C ₂ H ₄ OH | 0.31 | 0.24 |
| D5 | CH ₃ | C ₂ H ₄ OCOCH ₃ | 0.25 | 0.20 |

Table 1. Concentration of dyes applied to achieve the visual color yield (K/S) of 10 on PLA and PET fibers

The dyeing properties viz. % exhaustion, K/S values and λ_{\max} of selected azo disperse dyes are shown in Table 2. A marginally higher degree of dye exhaustion was observed on PLA for this series of azo disperse dyes. At about the same %dye exhaustion, a higher visual color yield (K/S values) obtained on PLA. The difference in visual color yield observed on the PLA and PET can be considered from two important parameters, namely degree of exhaustion and the tinctorial strength (i.e. ϵ_{\max}) of the dyes. High visual color yield on the fiber is expected to be obtained when the dyes render a high exhaustion percentage on the fiber. However, if the dye used is tinctorially weak, the deeper shade would not be able to be obtained. Therefore, high visual color yield achieved on polyester fiber involves the use of the disperse dyes which are tinctorially strong and well exhaust on the fiber. Yang & Huda studied the exhaustion of 10 disperse dyes on PLA and PET fabrics and found that, although the degree of dye exhaustion of all the disperse dyes on PLA was lower. The color yield of dyed PLA was higher than that of PET because of the lower reflectance of PLA (Yang & Huda, 2003). The λ_{\max} of the dyes depicted in Table 2. was also shifted to a shorter wavelength on PLA. The higher K/S values and shorter λ_{\max} of the dyes on PLA is explained by the effect of the polymer on the spectroscopic properties of the dyes which will be discussed in detail in the next section.

Another interesting property is the build-up characteristics of the dyes on PLA. The build-up properties of disperse dye on PLA has been studied in comparison with those of PET fiber. Fig. 5. and 6. show build-up curves of the selected azo disperse dyes on PLA and PET, respectively. All disperse dyes built up differently on the two fibers.



| Dye | Y group | R ₁ group | Exhaustion (%) | | K/S | | λ_{\max} (nm) | |
|-----|-----------------|--|----------------|--------|--------|--------|-----------------------|--------|
| | | | On PLA | On PET | On PLA | On PET | On PLA | On PET |
| 1 | H | C ₂ H ₅ | 99.30 | 96.43 | 17.67 | 11.94 | 530 | 550 |
| 2 | H | C ₂ H ₄ OCOCH ₃ | 98.30 | 91.55 | 9.82 | 7.97 | 490 | 520 |
| 3 | CH ₃ | C ₂ H ₅ | 99.12 | 98.48 | 15.41 | 12.46 | 540 | 550 |
| 4 | CH ₃ | C ₂ H ₄ OCOCH ₃ | 99.10 | 98.27 | 9.78 | 8.93 | 510 | 520 |

Table 2. Dyeing properties of azo disperse dyes on polyester fibers when applied at 0.2%owf

On PLA, these dyes exhibited a larger variation in build-up characteristics although their basic molecular structures were the same. At the same time, a less variation in build-up characteristics was found in the case of PET fiber. These results showed that the same set of azo disperse dyes performed differently on PLA as compared with PET. A change in the substituted group on the same basic structure of these azo disperse dyes did affect their build-up properties on PLA whereas this influence of the substituted groups was lower on PET. This property variation observed may be a result of the poorer fiber-dye interaction in the case of PLA.

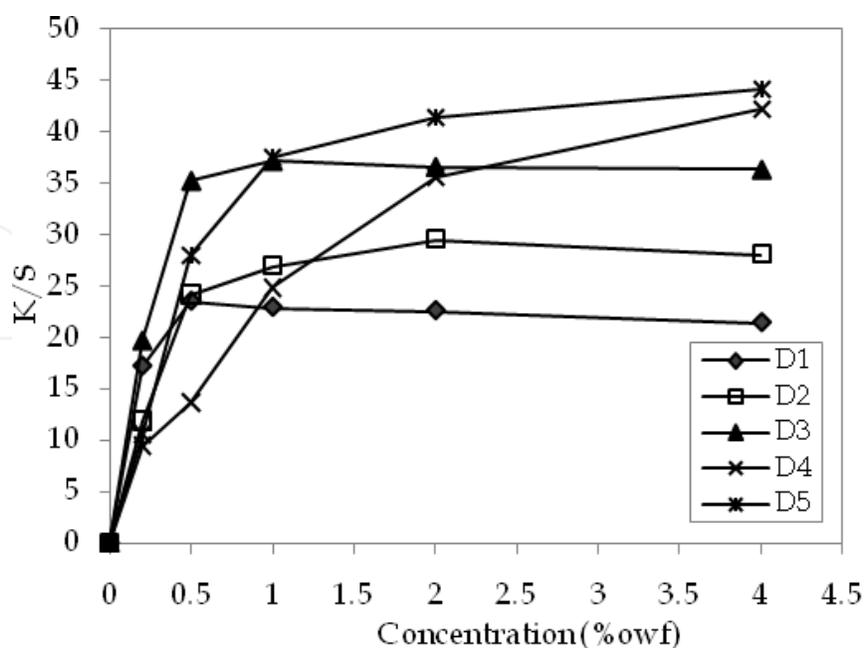


Fig. 5. Build-up curves of azo disperse dyes on PLA fiber (Structures of the dyes are in Table 1.)

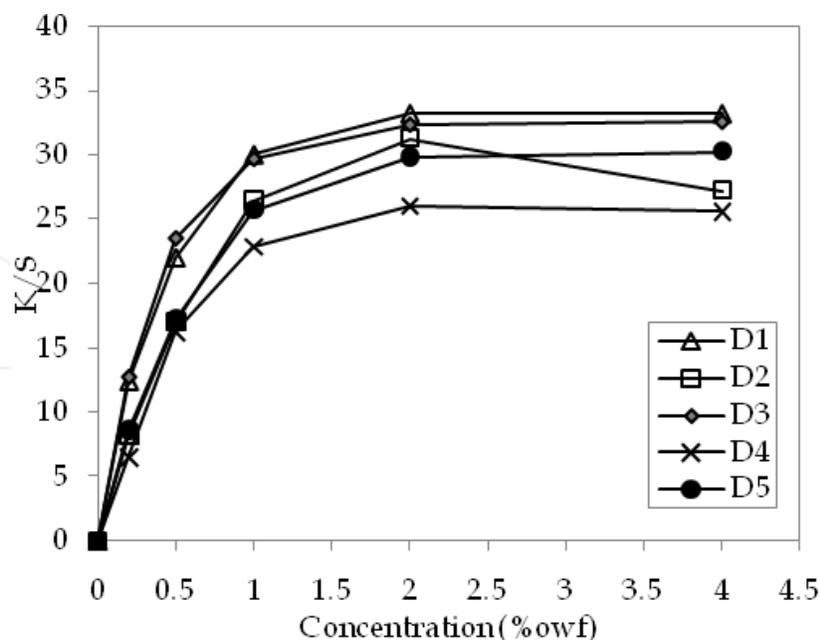


Fig. 6. Build-up curves of azo disperse dyes on PET fiber (Structures of the dyes are in Table 1.)

2.2 Study on the spectroscopic properties of disperse dyes on PLA in comparison with those on PET

PLA and PET polymers have different reflectance properties. As mentioned in the previous section that the different refractive index of the fibers affected the shade of dye obtained on the fibers (Lunt & Bone, 2001). The lower refractive index of PLA enhances a deeper shade as compared with PET. When the dye is applied at the same depth on the two fibers, the brighter shade is observed on PLA. Several studies reported that shades of the disperse dyes observed on PLA differed from those obtained on PET, the orange and red dyes appeared yellower whereas the yellow and blue dyes became greener on PLA (Choi & Seo, 2006; Avinc, 2007). Nakamura et al. studied the absorbance of C.I. Disperse Red 60 on PLA fabric. The results exhibited that the λ_{\max} of the dye on PLA occurred at a shorter wavelength (Hypsochromic shift) as compared with that on PET (Nakamura et al., 2001). These works confirm the difference in spectroscopic properties of disperse dyes on these two polyester fibers.

In order to gain a clear understanding on the influence of the polymer on the spectroscopic properties of the dyes, a study has been conducted. The polymer (fiber) is considered as a medium (solvent) having the dye molecules dissolving in. PLA and PET fibers have different molecular characteristics, for example molecular size/structure, polarity, dipole moment, etc. These different molecular properties could influence spectroscopic properties of the dyes that stay in the polymer media. Suesat et al. reported that different spectroscopic properties of azo disperse dyes on these two polyesters could be explained in the same way as a solvatochromic effect. The organic solvents, ethyl acetate and methyl benzoate, having similar molecular structure were selected as representatives of PLA and PET, respectively, for this investigation. The chemical structures of ethyl acetate and methyl benzoate are shown in Table 3. compared with

those of PLA and PET, respectively. Refractive indices of ethyl acetate and methyl benzoate are 1.372 and 1.517, respectively (wypych, 2001) being closely similar to those of PLA (1.35-1.45) and PET (1.54). As a chemical structure resemblance of ethyl acetate and methyl benzoate with the two polyesters, they could be used to dissolve disperse dyes and imitate the environment of the dyes in PLA and PET and their influence on the dye could then be monitored (Suesat et al., 2011).

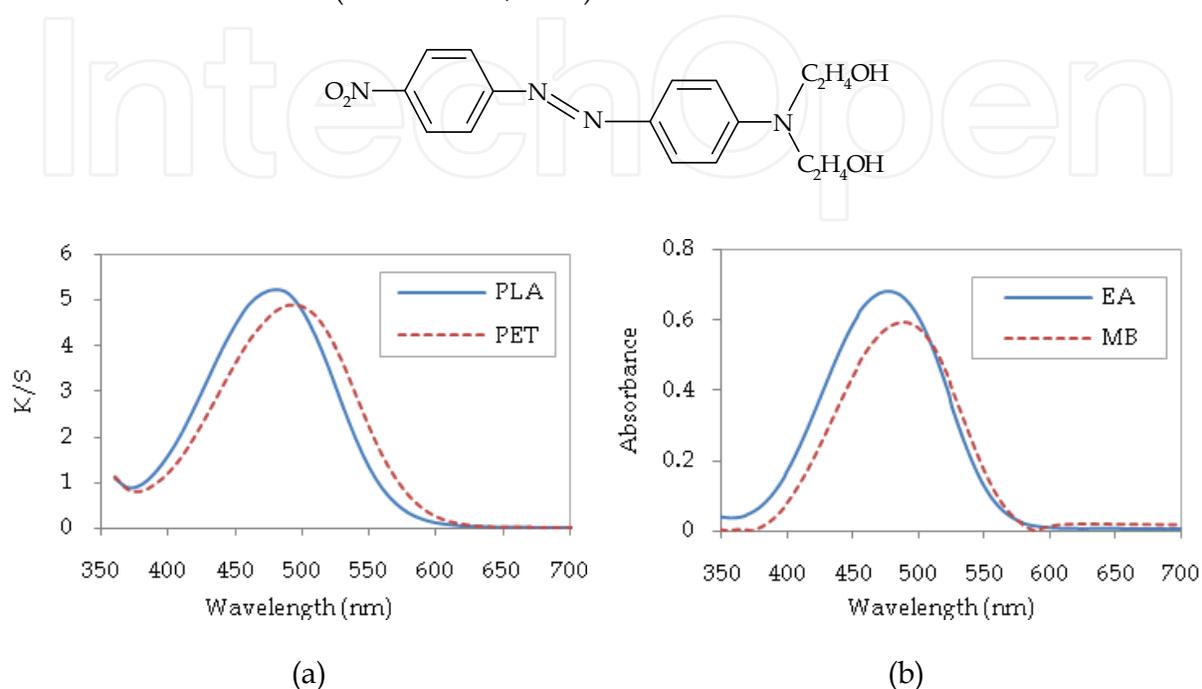


Fig. 7. The K/S spectra curves of disperse dye in a) PLA and PET; b) ethyl acetate (EA) and methyl benzoate (MB)

| Polymer structure | Solvent Structure |
|--|---|
| $\text{HO} - \left[\begin{array}{c} \text{CH}_3 \\ \\ \text{C} - \text{C}(=\text{O}) - \text{O} \\ \\ \text{H} \end{array} \right]_n - \text{H}$ <p>PLA</p> | $\text{CH}_3\text{CH}_2 - \text{O} - \text{C}(=\text{O}) - \text{CH}_3$ <p>Ethyl acetate</p> |
| $\text{H} - \text{O} - \text{C}(=\text{O}) - \text{C}_6\text{H}_4 - \text{C}(=\text{O}) - \text{O} - \left[-\text{CH}_2\text{CH}_2 - \text{O} - \text{C}(=\text{O}) - \text{C}_6\text{H}_4 - \text{C}(=\text{O}) - \text{O} - \right]_n - \text{CH}_2\text{CH}_2\text{OH}$ <p>PET</p> | $\text{H}_3\text{C} - \text{C}_6\text{H}_4 - \text{C}(=\text{O}) - \text{O} - \text{CH}_3$ <p>Methyl benzoate</p> |

Table 3. Chemical structures of ethyl acetate and methyl benzoate versus PLA and PET

The absorption spectral curves of the dyes in the representative solvents are illustrated as seen in Fig. 7.b). The same tendency was noticed when compared the absorbance curves of the dye in the solvents with the K/S curves of the same dye when applied on the polymers (Fig. 7.a)). The λ_{max} of the dyes exhibited a hypsochromic shift when dyed on PLA and dissolved in ethyl acetate. About a 10 nm difference was observed between λ_{max} of the dyes on the two fibers and in the two solvents. The shift of λ_{max} on K/S spectra of the dye on PLA

and PET, being dyed with the same dye, is a reason for differing in shade of the dyes obtained on the fibers. This change in spectroscopic properties of disperse dyes is affected by the difference in the interaction between the dye molecule and the polymer (solvent). Avinc (2007) mentioned that the difference in λ_{\max} values of azo disperse dyes when dyed onto PLA and PET was about 10 nm or higher whilst such a difference was lower in the case of anthraquinone disperse dyes.

When the dyes were dissolved in methyl benzoate and ethyl acetate, the color of the dye solutions was different. This is the effect of the solvatochromism. The solvatochromic effect happened as a consequence of polarity of the solvent used, influencing λ_{\max} to shift towards shorter or longer wavelengths depending on types of the interaction between the solvent and the dye molecule in its ground and excited states. For the dye molecules, their excited state is more polar than their ground state. When polar solvent interacts with the molecules in their excited state, it results in a bathochromic shift because the energy gap between their ground and excited states (HOMO-LUMO gap), is lowered (Bamfield, 2001). The polarity of ethyl acetate and methyl benzoate is 0.795 and 0.836, respectively (Wypych, 2001). Thus when a given dye was dissolved in ethyl acetate (less polar), poorer stabilization of the dye's excited electronic state brought about a higher energy gap between the ground and excited states of the dye molecules and a hypsochromic shift was observed, compared with methyl benzoate. The corresponding explanation could be used to describe the influence of the polyester polymers on the spectroscopic properties of the dye.

One more interesting point obtained from Fig. 7.a) and b). is that the magnitude of the spectral curves was higher in the case of PLA and ethyl acetate compared with those of PET and methyl benzoate. This indicates that not only shifting of the dye spectra, but the polymer/solvent also affects the molar absorptivity (ϵ_{\max}) of the dyes.

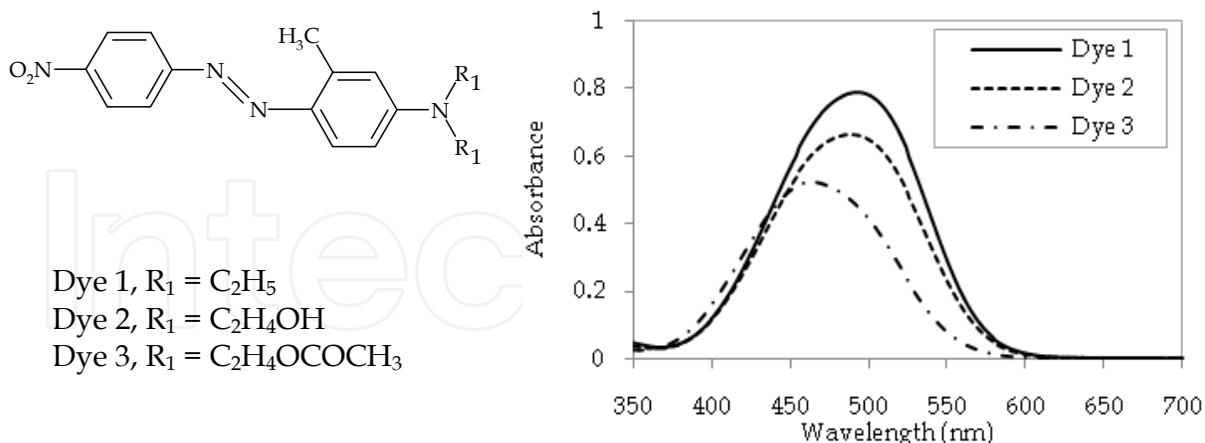
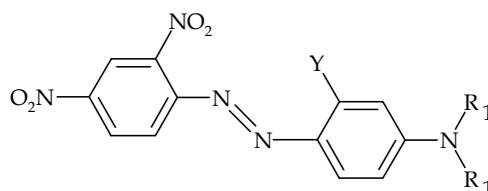


Fig. 8. Absorption spectra of the selected azo disperse dyes in ethyl acetate at concentration of 6.25 mg/l

The substituted group on the dye molecule is also influenced the absorption properties of the dyes. Fig. 8. shows the absorbance curves of the aminoazobenzene based disperse dyes in ethyl acetate. Shift in spectral curves and change in their magnitudes were observed when the substituted groups were changed on the dye molecules. The λ_{\max} and ϵ_{\max} values

varied depending on the degree of electron donating/withdrawing capacity of the substituted groups.

Table 4. shows the numerical data of the dye properties influenced by the solvents and the polymers. The difference in λ_{\max} suggested a different color of the dyes' solutions in the solvents, with a hypsochromic shift being obtained in ethyl acetate. Nakamura et al. found that the maximum absorption of C.I. Disperse Red 60 on PLA appeared at a shorter wavelength than on PET (Nakamura et al., 2001). Apart from the shifting of λ_{\max} , the molar extinction coefficients, ϵ_{\max} , were also found to be influenced by the solvents as their surrounding environment, the values being higher in ethyl acetate compared with those in methyl benzoate. Thus, the dye tended to be stronger and brighter in ethyl acetate than in methyl benzoate. This infers that a stronger shade of the dyes is expected on PLA than on PET. The brightness of the dyes is represented by half-band width ($\Delta\lambda_{1/2}$) of the dye spectra. The lower $\Delta\lambda_{1/2}$ values indicate a brighter shade. A brighter shade of the dyes was discovered on PLA, this corresponding to the dye properties observed in the solvents.



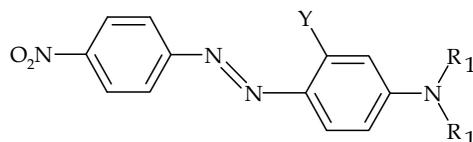
| Dye | Y group | R ₁ group | Properties in organic solvents | | | | Properties in polyesters | | | |
|-----|-----------------|--|--------------------------------|-----------------|--|-----------------|------------------------------|-----|-------|-------|
| | | | λ_{\max} (nm)* | | ϵ_{\max} (l mol ⁻¹ cm ⁻¹)* | | $\Delta\lambda_{1/2}$ (nm)** | | K/S** | |
| | | | Ethyl acetate | Methyl benzoate | Ethyl acetate | Methyl benzoate | PLA | PET | PLA | PET |
| 1 | H | C ₂ H ₅ | 522 | 538 | 40,600 | 37,400 | 104 | 114 | 17.67 | 11.94 |
| 2 | H | C ₂ H ₄ OCOCH ₃ | 496 | 509 | 33,100 | 30,100 | 111 | 118 | 9.82 | 7.97 |
| 3 | CH ₃ | C ₂ H ₅ | 534 | 552 | 39,300 | 37,500 | 110 | 114 | 15.41 | 12.46 |
| 4 | CH ₃ | C ₂ H ₄ OCOCH ₃ | 509 | 522 | 35,800 | 32,100 | 114 | 118 | 9.78 | 8.93 |

*measured on the dye solutions.

**measured on the dyed fabrics with % exhaustion of higher than 90% when dyed at 0.2% owf.

Table 4. The spectroscopic properties of azo disperse dyes measured in the solvents and polyester fiber

The color properties of the disperse dyes on PLA and PET are compared in Table 5. The dyed polyester fabrics with the same level of color yield (K/S=10) showed a differed shade, depicted by the L*, a*, b*, C* and H* values. The L* values representing lightness of shade were higher on PLA, indicating a lighter shade as compared with those on PET. A yellower shade was noticed on PLA with higher b* values whereas the C* (chroma) and H* (hue) were also higher on PLA. This points out that the shade obtained on PLA differs from that on PET. It was reported that the shade difference was observed on PLA and PET when the two fibers were dyed at the same level of visual depth (Suesat et al., 2011). In addition, a brighter shade obtained on PLA was found (Choi & Seo, 2006; Blackburn, 2005).



| Dye | Y group | R ₁ group | Fiber | L* | a* | b* | C* | H* |
|-----|-----------------|--|-------|---------|---------|---------|---------|---------|
| D1 | H | C ₂ H ₅ | PET | 49.1 | 50.68 | 25.56 | 56.76 | 26.77 |
| | | | PLA | 55.71 | 51.32 | 45.73 | 68.74 | 43.01 |
| | | | Diff. | 6.61 L | 0.64 R | 20.17 Y | 11.98 B | 16.24 Y |
| D2 | H | C ₂ H ₄ OH | PET | 54.52 | 46.44 | 34.19 | 57.66 | 36.36 |
| | | | PLA | 56.67 | 42.5 | 44.22 | 61.32 | 46.49 |
| | | | Diff. | 2.15 L | -3.94 G | 10.03 Y | 3.66 B | 10.13 Y |
| D3 | H | C ₂ H ₄ OCOCH ₃ | PET | 56.67 | 42.5 | 44.22 | 61.32 | 46.49 |
| | | | PLA | 66.55 | 41.82 | 65.32 | 77.56 | 60.12 |
| | | | Diff. | 10.07 L | -5.13 G | 13.94 Y | 7.96 B | 12.54 Y |
| D4 | CH ₃ | C ₂ H ₅ | PET | 43.29 | 48.74 | 12.69 | 50.37 | 14.6 |
| | | | PLA | 52.18 | 51.64 | 30.61 | 60.03 | 29.96 |
| | | | Diff. | 8.89 L | 2.90 R | 17.92 Y | 9.66 B | 15.36 Y |
| D5 | CH ₃ | C ₂ H ₄ OH | PET | 46.5 | 46.97 | 23.23 | 52.4 | 26.32 |
| | | | PLA | 52.3 | 48.34 | 35.66 | 60.07 | 36.2 |
| | | | Diff. | 5.80 L | 1.37 R | 12.43 Y | 7.67 B | 9.88 Y |
| D6 | CH ₃ | C ₂ H ₄ OCOCH ₃ | PET | 51.14 | 48.21 | 37.57 | 61.12 | 37.93 |
| | | | PLA | 58.97 | 47.53 | 53.83 | 71.81 | 50.2 |
| | | | Diff. | 7.83 L | -0.68 G | 16.26 Y | 10.69 B | 12.27 Y |

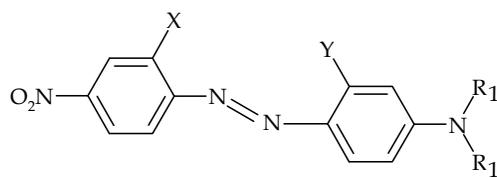
**Diff.= the values difference of PLA and PET

Table 5. Color properties of azo disperse dyes applied at visual color yield of 10 on PLA and PET

2.3 Parameters influencing dyeing properties of disperse dyes on PLA

The dyeing properties of disperse dyes on PLA have been studied by many researchers in order to gain an understanding as what the factors affecting dyeability of PLA are. Several papers involved the explanation of disperse dye sorption on polyester by solubility parameter concept (Gerber, 1978; Karst & Yang, 2005; Choi et al., 2010). Gerber stated that the azo disperse dye suited for dyeing polyester fiber should have a closely similar solubility parameter (δ) value to that of the fiber (Gerber, 1978). This concept was later used by Karst & Yang to correlate with disperse dye sorption on PLA fiber. They calculated the δ values of PLA, PET and disperse dyes by means of group contribution method. The δ values of PLA and PET were reported to be $20.2 (\text{J}\cdot\text{cm}^3)^{0.5}$ and $21.7 (\text{J}\cdot\text{cm}^3)^{0.5}$, respectively. The important point drawn from their work was that the dyes having the δ close to that of the PLA would exhibit a high exhaustion. From their study, the dyes with the δ values below $25.0 (\text{J}\cdot\text{cm}^3)^{0.5}$ had high sorption whereas those with δ values above $25.0 (\text{J}\cdot\text{cm}^3)^{0.5}$ exhibited lower than 70% sorption on PLA when dyed at 110°C

and the dyes with the δ values close to that of PLA were mainly azo dyes. In our work (Suesat et al., 2011), a correlation between the solubility parameter and the dye exhaustion was also established from a set of experimental data obtained from 22 azo disperse dyes on PLA and PET. The δ values were also calculated by means of group contribution methods mentioned in Karst & Yang (2005). The exhaustion of the dyes was acquired experimentally by dyeing at 0.2%owf on PLA and PET fabrics. The correlation between the δ values with the % exhaustion of the dye on PLA has been made as shown in equation (1). The δ values of each azo dye were used to calculate the dye exhaustion in equation (1) and the predicted dye exhaustion was obtained. The predicted values of dye exhaustion were plotted against the exhaustion observed from the experiment as seen in Fig. 10. The linear regression with the R^2 of 0.819 was obtained for the case of PLA whereas the correlation of the same set of azo disperse dyes on PET as shown in equation (2) had a lower R^2 of 0.764. The plot between the predicted and observed dye exhaustion on PET is shown in Fig. 11. The two equations indicate that the solubility parameter correlates well with the degree of exhaustion of azo disperse dye on the fibers. The dye having the solubility parameter close to that of the polymer would have high exhaustion. However, this establishment only experimented on the azo disperse dyes with the basic structure shown in Fig. 9. Application of the correlation for other type of disperse dyes may be impractical as reported by Karst et al. (2007).



Where X = H, Cl, CN and NO₂
 Y = H and CH₃
 R1 = C₂H₅, C₂H₄OH and C₂H₄OCOCH₃

Fig. 9. Structure of the azo disperse dyes used

$$\text{Exhaustion} = 145.282(\pm 5.344) - 2.314(\pm 0.225) \delta \quad (1)$$

$$R^2 = 0.819, n = 22, F \text{ change} = 90.328, s = 2.54$$

The explanation on molecular parameters affecting the dyeability of disperse dyes on polyester was also given by several researchers. Kim & Son described that the degree of disperse dye exhaustion was related to the solubility and hydrophobic/hydrophilic properties of the dyes which was, in turn, varied by the types of substituted group on the dye molecule. The hydrophobic/hydrophilic properties of the dyes could be considered from dipole moment values of the dyes. The dye having low dipole moment (low polarity) tended to be hydrophobic. This would result in their solubility in hydrophobic medium, like polyester fiber (Kin & Son, 2005). In addition, the dye-fiber interaction was reported by Karst et al. to play an important role on the disperse dyeing properties on PLA fiber. The substituted groups on the dye molecule that formed strong interaction with PLA were $-\text{N}(\text{C}_2\text{H}_4\text{OCOCH}_3)_2$, $-(\text{CO})_2\text{NC}_3\text{H}_6\text{OCH}_3$, $-\text{SO}_2\text{NHC}_6\text{H}_5$, $-\text{NO}_2$, $-\text{CN}(\text{NH})\text{C}_6\text{H}_4$ and $-\text{CH}(\text{CO})_2\text{C}_6\text{H}_4$, while those with $-\text{Br}$ and $-\text{Cl}$ were among the weakest. The dyes which interacted strongly with PLA would have more negative interaction energy with PLA, bringing a high sorption on the fiber (Karst et al., 2007).

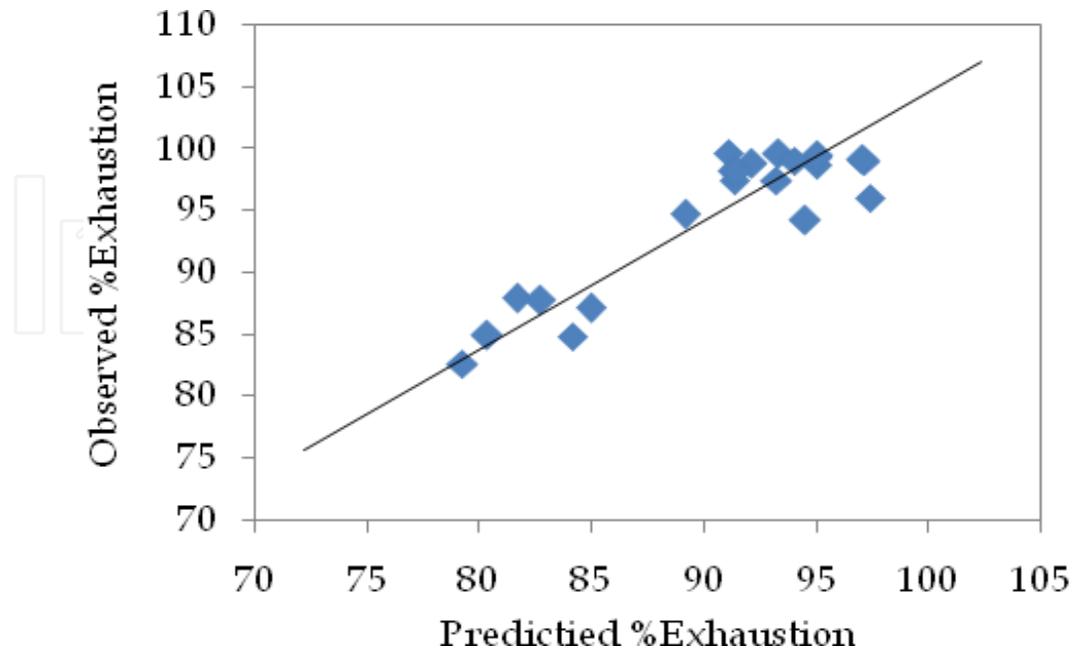


Fig. 10. Relationship between predicted and observed % exhaustion of the azo disperse dyes on PLA fabric

$$\text{Exhaustion} = 173.915(\pm 10.320) - 3.491(\pm 0.434) \delta \quad (2)$$

$$R^2 = 0.764, n = 22, F \text{ change} = 64.755, s = 4.91$$

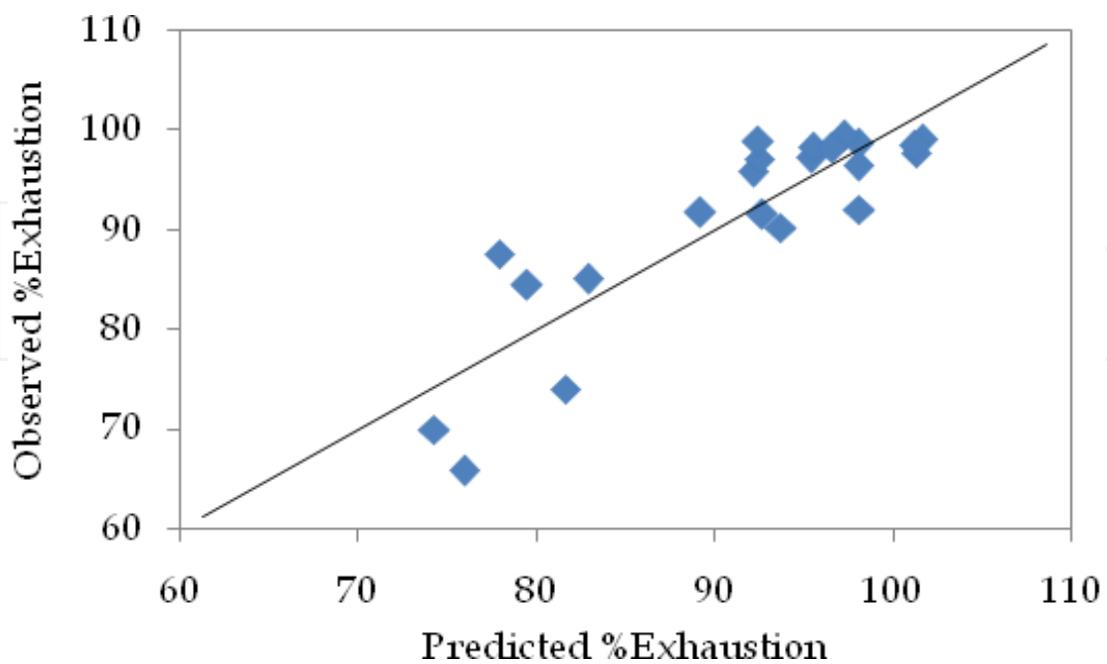
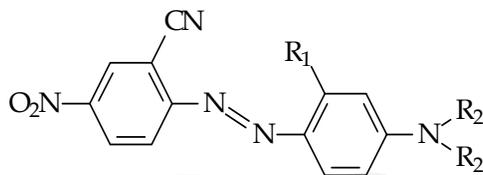


Fig. 11. Relationship between predicted and observed % exhaustion of the azo disperse dyes on PET fabric

3. Color fastness properties of disperse dyes on PLA

3.1 Color fastness of dyed PLA vs PET

The color fastness of dyed PLA in comparison to dyed PET has been studied by many researchers. It has been claimed that the color fastness properties of disperse dyes on PLA were generally lower than those on PET. Yang and Huda studied the wash, rub and light fastness properties of PLA and PET dyed with ten disperse dyes from all three energy levels; low, medium and high-energy dyes. At the same 2%owf, PLA dyeings possessed 0.5 to 1.0 point lower wash fastness than those on the corresponding PET dyeings. The results indicated no significant influence of energy levels of the disperse dyes on the wash fastness grade. They also suggested that the wash fastness of PLA can be effectively improved in the range of 0.5 to 1.5 points by the appropriated reduction clearing process. Both dry and wet rub fastness of dyed PLA were either the same or up to 1.0 point lower than those of PET. Light fastness of disperse dyes on PLA and PET was very similar, even though the dye concentration on PLA was much lower than that on PET (Yang & Huda, 2003). The other factors, besides the concentration of dye, such as the interaction between the dye and fiber, the distribution of the dye in the fiber and the chemical structure of dye and fiber can also play important roles in light fastness (Yang & Huda, 2003; Kim & Park, 2001). The work of Choi and Seo is in agreement with Yang and Huda. They investigated wash and light fastness properties of PLA and PET fabrics dyed with eight disperse dyes at 4%owf. The PLA dyeing exhibited the same or 0.5 to 1.0 point lower wash fastness than its PET counterpart. Light fastness for seven of the eight dyes observed on PLA compared with PET showed no difference. There was no influence of the chemical classes of the dyes investigated, i.e. nitrodiphenylamine, monoazo and anthraquinone, on the light fastness. However, the differences in color (ΔE^*) upon light fastness testing were greater in the case of the PLA dyeing as compared with PET (Choi & Seo, 2006). Choi et al. investigated the wash and light fastness of PLA dyed with the commercial disperse dyes that were initially intended for the production of high light fastness polyester fabrics or for cellulose diacetate dyeing. The wash fastness results in terms of staining to acetate and nylon fabrics showed that the PLA dyeings were 1.0 up to 2.0 points lower than the PET counterparts. The significantly higher diffusivity of disperse dyes was allowed more in PLA than in PET. Therefore, the greater amount of dyes was able to migrate from the PLA fabric during wash fastness testing and stained heavier on the adjacent fabric, leading to lower wet fastness properties. The light fastness rating of PLA dyeings was 0.5 to 1.0 grey scale units lower than the corresponding PET dyeings because PLA allowed more UV transmittance than PET. Dorospers G/YEL KBGL was recommended as a good choice for dyeing PLA to meet the super light fastness requirement (Choi et al., 2007). Mogi et al. investigated the light fastness of monoazo and anthraquinone disperse dyes on PLA fabrics using a carbon arc light source. The results indicated that the photofading behaviors of disperse dyes increased with increasing the exposure time. Moreover, a deeper color fabric tended to give a higher light fastness than a paler color one which indicated that the light fastness depended on the concentration of dye inside the fabric (Mogi et al., 2001a, 2001b). Batch et al. reported that PLA dyed with C.I. Disperse Red 167:1, C.I. Disperse Blue 79 and Serilene Black RFS in super critical carbon dioxide gave good to very good wash and rub fastness. They also suggested the after-clearing process of dyed PLA in order to obtain high wet fastness properties (Batch et al., 2006). Our work with light fastness of monoazo dyes on PLA and PET fabrics, as shown in Table 6, also showed that the dyed PLA exhibited slightly lower light fastness properties, about 0.5 to 1.0 point, than the dyed PET.



| Dye | R ₁ | R ₂ | Light fastness | |
|-----|-----------------|--|----------------|-----|
| | | | PLA | PET |
| 1 | H | C ₂ H ₄ OH | 3 | 3-4 |
| 2 | H | C ₂ H ₄ OCOCH ₃ | 3-4 | 4-5 |
| 3 | CH ₃ | C ₂ H ₄ OH | 3 | 3-4 |
| 4 | CH ₃ | C ₂ H ₄ OCOCH ₃ | 4 | 4-5 |

Table 6. Light fastness of monoazo disperse dyes 1-4 on PLA and PET

Color fastness is also correlated with the chemical structures of disperse dyes on PLA. The monoazo disperse dyes having cyanoethyl or acetoxyethyl group in the coupling component provided high light fastness properties such as C.I. Disperse Red 50, C.I. Disperse Red 82, and C.I. Disperse Orange 30. Cyano and chloro groups had a positive effect on the light fastness, while methyl group showed a negative effect (Mogi et al., 2001a). Dyes 1 and 3 in Table 6. containing acetoxyethyl groups (R₂) also showed better light fastness properties. A methyl group (R₁) had no significant effect on the light fastness of the dye on PLA and PET fabrics. Phenoxy substituents on anthraquinone disperse dyes, such as C.I. Disperse Red 127 and C.I. Disperse Violet 26, showed high light fastness properties on PLA fabrics (Mogi et al., 2001b). Wash and light fastness properties of the substituted aminoanthraquinone disperse dyes on PLA were reported by He et al. The substituted amino groups had little effect on the wash fastness, but obviously influenced the light fastness. The results showed that all selected substituted amino groups gave good wash fastness ratings. Light fastness was higher with the introduction of aromatic amino or cyclo-fatty amino groups into the anthraquinone disperse dyes. In addition, the longer the aliphatic fatty amino group, the lower the light fastness on PLA was (He et al., 2009).

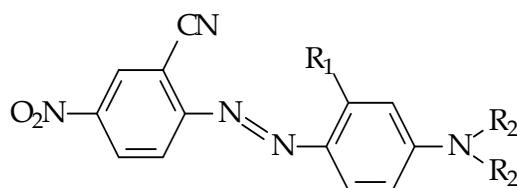
3.2 Thermal migration of disperse dyes on PLA

Thermal migration of disperse dyes is the movement of dye during post-heat treatment as a result of the breakage of the interaction between dye and fiber, leading to an accumulation of dye at the fiber surface and some limited sublimation of the dye to the bordering atmosphere. Even if the loose surface dyes were reduction cleared after dyeing, the heat treatment can promote the diffusion of the dye inside the fiber to fiber surface because of weak interactions between polyesters and disperse dyes which are mainly van der Waals forces and hydrogen bonding. A heat treatment for PLA fabrics is generally either to provide dimensional stability or in the application of a chemical finishing agent. The degree of thermal migration of a disperse dye is an important factor in determining the subsequent wet fastness properties of PLA fabrics (Avinc et al., 2009; Kim et al., 2004; Phillips et al., 2004b).

Phillips et al. reported that the wet fastness properties (wash and perspiration fastness) of dyed PLA fabric, dyed with three blue azo disperse dyes, were the same before heat treatment with almost no staining on the adjacent nylon. After the PLA fabric was heatset for 30 s at 130°C, both wash and perspiration fastness properties became lower as compared with the non-heatset counterpart. In order to determine the amount of dye migrated to the fiber surface, the heatset fabric was immersed in cold toluene and then the absorbance of the solution was measured at the assigned time. The amount of dye at the fiber surface was estimated from the extrapolated absorbance at time, $t=0$ min. The difference between the amounts of dyes at the fiber surface before and after heat treatment represented the amount of dye that had migrated to the fiber surface on heat treatment. In addition, the amount of dye lost from PLA fabric during heat treatment due to sublimation to the atmosphere was estimated from a monochlorobenzene extract containing both dye and PLA substrate. The calculation of the amount of lost dye via sublimation was also from the difference between the amount of dye on PLA fabric before and after heat treatment. As a result of heat treatment, the wet fastness properties reduced for all three disperse dyes studied. The amount of dye migrated to the PLA fiber surface was about 1.4%. The authors suggested that the high degree of thermal migration of the disperse dyes at the surface of PLA was because the absence of π - π bonding forces between the disperse dye and the polymer chain. They also concluded that the wet fastness of the heatset PLA fabric was not directly proportional to the amount of disperse dyes that has migrated to the fiber surface (Phillips et al., 2004b). Avinc et al. investigated the influence of different heat treatment conditions on the wet fastness properties viz. wash, alkaline perspiration and wet rub fastness of three red disperse dyes (Serilene Red 3B-LS, Foron Rubine S-2GFL and Dianix Deep Red SF) on PLA fabric. The wet fastness properties of PLA were reduced after subjecting the fabric to the heat treatment process due to thermal migration of dye on PLA (Avinc et al., 2009). The amount of dye thermally migrated on the PLA fabric finished with different softeners was examined in terms of their color fastness (Avinc, 2007). No clear relationship was found between the hydrophobic nature of the disperse dye and the ionicity character of the softener on the color fastness of finished PLA fabric. For example, softened PLA fabrics dyed with C.I. Disperse Red 167.1 showed greater thermal migration, resulting in lower color fastness than the corresponding PET fabrics. In contrast, softened PLA fabrics dyed with Dianix Deep Red SF exhibited less thermal migration, leading to higher color fastness than the corresponding PET fabrics. The application process also influenced the thermal migration of disperse dyes. Softened PLA fabrics from the exhaust process gave higher wet fastness up to 1.25 points more than the one from the padding process. The exhaust method allowed less thermally migrated dye onto the PLA surface than the pad method (Avinc et al., 2009). Dry heat fastness of Dyes 1-4 on PLA and PET fabrics with K/S = 10 was evaluated according to ISO 105-P01:2006(E)(150°C) as shown in Table 7. The dry heat fastness properties of the dyed PLA fabric exhibited 0.5 grey scale units being lower than those of the dyed PET fabric. The dye migrated to the fiber surface by thermal migration, leading to lower dry heat fastness properties.

Our work on thermal migration of disperse dye on PLA fabric was carried out by using six commercial disperse dyes from all three different sublimation classes. The quantity of dye migrated to the fiber surface and dye lost by sublimation to the atmosphere after heat treatment at 130°C for 30 s was investigated according to the procedure in Phillips et al.

(2004b). Moreover, the wet fastness properties (wash, perspiration, and rub) were compared between the dyed PLA before and after heat treatment. Table 8. showed that the amount of dye migrated to the PLA surface as a result of the heat treatment being in a range of 0.04 to 3.04%. However, the amount of disperse dye that has migrated to the PLA fiber surface was not dependent on the sublimation class. Besides the molecular size of the dye, the other parameters such as the chemical structure, polarity and hydrophobicity of the dye can also affect the movement of disperse dye from the interior of the fiber to the surface. Loss of disperse dye due to sublimation was observed only from the low energy dyes, approximately 2%. Low-energy dyes tended to loss via sublimation easier than the other two higher energy classes. Table 9 illustrated the wet fastness properties of the dyed PLA after heat treatment which was slightly lower than the one before the treatment. The migrated dyes as a result of heat treatment led to lower wet fastness properties of dyed PLA fabric. Nevertheless, the wet fastness properties of dyed PLA fabric were not directly proportional to the amount of dye migrated to the fiber surface.



| Dye | R ₁ | R ₂ | Color change (PLA/PET) | Staining (PLA/PET) | |
|-----|-----------------|--|------------------------|--------------------|-----------|
| | | | | Nylon | Polyester |
| 1 | H | C ₂ H ₄ OH | 4-5/4-5 | 4-5/4-5 | 4-5/4-5 |
| 2 | H | C ₂ H ₄ OCOCH ₃ | 4-5/4-5 | 4-5/4-5 | 4-5/4-5 |
| 3 | CH ₃ | C ₂ H ₄ OH | 4-5/4-5 | 4/4-5 | 4-5/4-5 |
| 4 | CH ₃ | C ₂ H ₄ OCOCH ₃ | 4-5/4-5 | 4/4-5 | 4-5/4-5 |

Table 7. Heat fastness of monoazo disperse dyes 1-4 on PLA and PET

| Dye | Commercial name | Sublimation class | Dye migrated to surface (%) | Dye lost by sublimation (%) |
|-----|-------------------------------|-------------------|-----------------------------|-----------------------------|
| 1 | Dianix Orange E-3R | Low energy | 3.04 ±0.12 | 2.12±0.13 |
| 2 | Dianix Blue E-R 150% | Low energy | 0.37 ±0.07 | 1.66± 0.43 |
| 3 | Dianix Rubine SE-B | Medium energy | 0.44 ±0.10 | 0 |
| 4 | Dianix Yellow Brown XF | Medium energy | 0.04 ±0.003 | 0 |
| 5 | Dianix Yellow Brown S-2R 150% | High energy | 0.25 ±0.06 | 0 |
| 6 | Dianix Red S-BEL | High energy | 0.47 ±0.06 | 0 |

Table 8. The amounts of dye migrated to the fiber surface and lost by sublimation on heat treatment of PLA fabrics

| Dye | Wash* (C06/B2S, 50°C) | | Acid perspiration* (E04) | | Alkaline perspiration* (E04) | | Rub* (X12) | |
|-----|--------------------------|--------------|-----------------------------|--------------|---------------------------------|--------------|---------------|--------------|
| | PET | Nylon | PET | Nylon | PET | Nylon | Dry | Wet |
| 1 | 4 (4) | 2-3 (2-3) | 4-5 (4-5) | 4 (4) | 5 (4-5) | 4 (3-4) | 4-5 (4) | 4 (3-4) |
| 2 | 3 (3-4) | 2 (1-2) | 4-5 (4) | 4-5 (3) | 4-5 (4-5) | 4 (4) | 5 (4-5) | 4-5 (4) |
| 3 | 3-4 (3) | 3 (2-3) | 4-5 (4-5) | 4-5 (4-5) | 4-5 (4-5) | 4-5 (4-5) | 4-5 (4-5) | 4 (4) |
| 4 | 4 (3-4) | 4-5 (4) | 4-5 (4) | 4 (3) | 4-5 (4-5) | 4 (3-4) | 5 (5) | 4-5 (4-5) |
| 5 | 4 (3-4) | 4-5 (4) | 4 (3-4) | 3-4 (3) | 3-4 (3-4) | 3 (2-3) | 5 (5) | 4-5 (4-5) |
| 6 | 4 (2-3) | 4 (2-3) | 4-5 (4-5) | 4-5 (4) | 4-5 (4-5) | 4 (4) | 4-5 (4-5) | 4-5 (4-5) |

* Values in parentheses are those after heat treatment for 30 s at 130°C

Table 9. Wet fastness properties of disperse dyes on PLA before and after heat treatment

4. Conclusion

With promising properties, PLA has been intensively studied in order to gain its maximal benefit as a textile fiber for the textile industry. PLA fiber possesses many comparable properties to the conventional polyester, PET, but its thermal and alkaline sensitivities are inferior, therefore, milder processing conditions used in textile production are suggested for PLA. PLA can be dyed with disperse dyes to provide a stronger and brighter shade as compared with PET. The differences in dyeing properties of disperse dyes on PLA and PET are explained to be a result of different interactions between the polymer (fiber) and the dye molecule. The polymers (PLA and PET) are influential on the spectroscopic properties of the dyes. PLA provides less stabilization of the dye molecules in its excited states as compared with the case of PET, consequently, different shade and strength of the dyes are observed. The difference in dyeing properties of disperse dyes on PLA and PET was explained by several researchers that the key factor affecting this was the solubility parameter. The dye having a solubility parameter close to that of PLA, would exhibit high sorption on the fiber whereas those with solubility parameter being largely different from the fiber, would have low sorption. A new concept was proposed that there were other important factors, apart from solubility parameters, being influential on the dyeability of disperse dyes, for example hydrophilic/hydrophobic nature of the dye and the dye-fiber affinity. A study on color fastness of disperse dyes on PLA in comparison with PET indicates that fastness properties of the dyed PLA is slightly lower (about 0.5-1.0 unit of grey scale) than those on PET. A higher degree of thermal migration of the dye was found on PLA after subjected to heat

setting or chemical softening, resulting in a poorer wet fastness of the dyed PLA as compared with PET. A greater degree of thermal migration of the dye on PLA is explained to be due to higher dye diffusivity and lack of π - π bonding between the dye molecule and the polymer (fiber).

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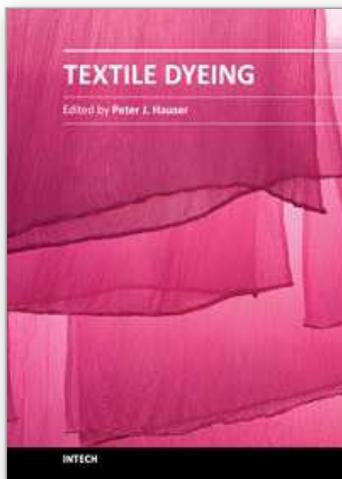
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University Campus STeP Ri
Slavka Krautzeka 83/A
51000 Rijeka, Croatia
Phone: +385 (51) 770 447
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Unit 405, Office Block, Hotel Equatorial Shanghai
No.65, Yan An Road (West), Shanghai, 200040, China
中国上海市延安西路65号上海国际贵都大饭店办公楼405单元
Phone: +86-21-62489820
Fax: +86-21-62489821

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