Studies on the Possibility of Recycling Microencapsulated Disperse Dye-Bath Effluents

Jun-ling Ji,[1,2] Xu-Jie Yang,[1] Lu-De Lu,[1] Xin Wang[1]

[1]*Material Chemistry Lab., Nanjing University of Science and Technology, Nanjing, Jiangsu 210094, China*
[2]*Chemical Engineering Department, Jiangsu Polytechnic University, Changzhou, Jiangsu 213164, China*

Received 11 April 2007; accepted 20 December 2007
DOI 10.1002/app.30215
Published online 15 May 2009 in Wiley InterScience (www.interscience.wiley.com).

ABSTRACT: The volume of water consumed by human beings has increased dramatically in recent years while water supply has remained constant; both demographic growth and the expansion of industrial activity require more water consumption. The textile industry is undoubtedly one of the most pressured industries that need water intensively. Efficient use of water tends to be a crucial subject for the dyeing industry. In this study, melamine resin microcapsules containing pure disperse dyes were prepared by in situ polymerization. The microcapsules were characterized on the basis of structure, morphologies, mean particle size, and size distribution. The dyeing behaviors of microencapsulated disperse dyes (MDDs) were evaluated on polyester fabrics in the absence of auxiliaries. Its effluent can be reused several times after being simply filtered and can be used as solvent for PET fabric scouring. The treated fabrics exhibited satisfactory levelness and fastness properties. MDDs can be used in dyeing PET, without using surfactants, and the effluents can be recycled and reused. © 2009 Wiley Periodicals, Inc. J Appl Polym Sci 113: 3774–3781, 2009

Key words: textile industry; disperse dye; microencapsulated; effluent; recycle

INTRODUCTION

As shown by recent scientific research,[1,2] 20–230 m_3 of water is required to produce 1 kg of textile fabric. Dyeing effluents containing high concentrations of organic and inorganic chemicals are characterized by very high COD value and strong color.[3,4] Dye wastewater discharged from the textile industry can be extremely deleterious to the environment if improperly disposed.[5] Conventional physicochemical and biological treatment methods are ineffective and expensive for color removal.[6–9] The increasing cost of water, energy and restrictions imposed on wastewater disposal by changing environmental regulations dictate the needs for wastewater recycle in the industry on a large scale.

Vandevivere et al. and Skelly reviewed the efficiency of the recycling process in textile wet processing and found that reuse of the treated dye baths saves chemicals and water.[10] Vreese et al. reported that colored wastewater from direct, reactive, and disperse dyeing processes was suitable for reuse if it is treated by nanofiltration or ozone.[3,10] Although numerous reports regarding the water recycle issue have been published earlier, not many of them can ensure color difference and levelness of dyeing at an acceptable level, which is directly linked to the feasibility of industrial application.

In the case of traditional disperse dyes, surfactants also play an important role in enhancing the solubility of dyes by the formation of a disperse system.[11] Hence, large volume of surfactants and the stability of dye molecules in the water are responsible for the high COD/BOD values and a heavy color of dyeing effluents; avoiding the use of surfactants would effectively abate the pollution problem.

In previous works, polyurea microcapsules of disperse dye have been tested as clean dyeing on PET in the absence of auxiliaries.[12] As shown in MDD (Fig. 1), the shell of MDD is equivalent to a semitransparent membrane which, under certain conditions, allows water to get in and dissolve dye molecules. The dissolved dye then penetrates through the membrane slowly. MDD dyeing process consists of six steps: diffusion of water into microcapsules; dissolution of dye in microcapsules; diffusion of dye in the solution; adsorption at the fiber surface; diffusion of dye into the fiber; and fixation of the dye to the fiber. It is required that the controlled releasing action of microcapsule dye plays the role of a dispersing and leveling agent. Being thoroughly auxiliary-free, the MDD dyeing method costs much less in reducing pollution than the traditional method.

In the present work, a series of MDDs in different colors were prepared for dyeing polyester fabrics and the recycling of MDD bath after simple filtration

Correspondence to: X. Wang (wangx@mail.njust.edu.cn).

Journal of Applied Polymer Science, Vol. 113, 3774–3781 (2009)

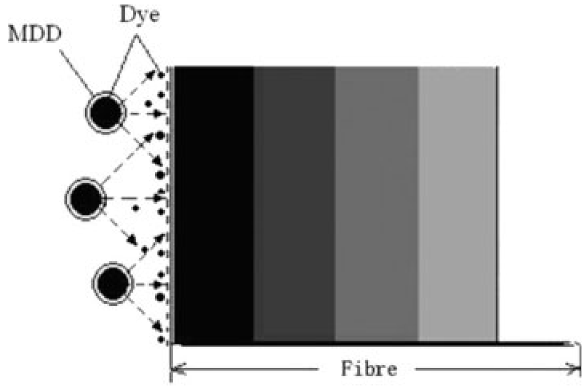

Figure 1 Model of diffusion of MDD on fiber.

was assessed. Experiments using the recycled effluent in dyeing and scouring operations were carried out and the spectral reflectance value and relative levelness index value of each pair of samples were collected. These results were compared to that using distilled water. Based on the color reproduction ability, it has been found that effluents generated by MDD after simple filtration can be reused several times without any noticeable change in color, and the levelness of the fabric shade produced in the recycling of effluents is either excellent or good in all the cases.

The simplified Figure 2 presents the challenges of this study.

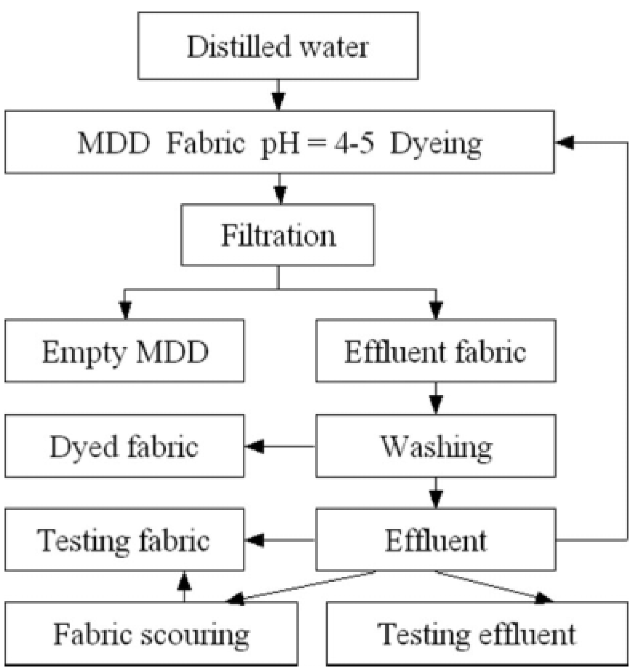

Figure 2 Auxiliary-free dyeing process with disperse dye microcapsule.

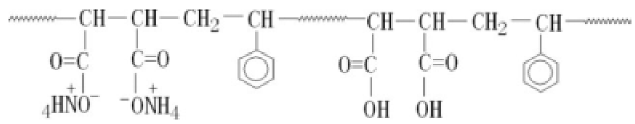

Figure 3 Polyelectrolyte MS.

EXPERIMENT

Materials

The materials used in the experiment are as follows:

- Melamine prepolymer (hexamethylol- and trimethylol-malamine) as wall material, from Shanghai Chemical Factory (China/Shanghai).
- Polyelectrolyte maleic anhydride and styrene copolymer (MS) (Fig. 3) as emulsifier, scouring agent (China/Jiangsu).
- C. I. Disperse Blue 291 (Fig. 4), C. I. Disperse Violet 93 (Fig. 5), and C. I. Disperse Orange 288 (Fig. 6) as core material (the disperse dye contains no additives), from Yaband Chemical Engineering Co. (China/Jiangsu).
- Substrate: 100% polyester, plain 69.0 gm^{-2} 50 D*50 D/72*72, from Wuxi Huayuan Textile Co. (China/Jiangsu).
- Acetic acid, ammonia, sodium carbonate, *N*,*N*-dimethylformamide (DMF), laboratory reagent grade.

Microcapsule preparation

C. I. Disperse Blue 291 (or C. I. Disperse Violet 93 or C. I. Disperse Orange 288) (10 g) in 100 mL of 0.5% w/w MS aqueous solution was stirred and mixed by high-speed emulsifier (10,000 rpm) for 30 min, and the pH of the mixture was adjusted to 4 by the dropwise addition of 10% v/v acetic acid solution. The system was transferred immediately into a reactor stirred at 260 rpm. Then trimethylol-melamine (75% w/w) (9 g) was added and was constantly stirred for 1 h at 30°C. Subsequently, the reaction system was heated up to 65°C and maintained for 90 min to form a covering of Disperse Blue 291 with a single-layered shell. After being cooled to 30°C, hexamethylol-melamine (55% w/w) (6 g) was also added dropwise into the reaction system while

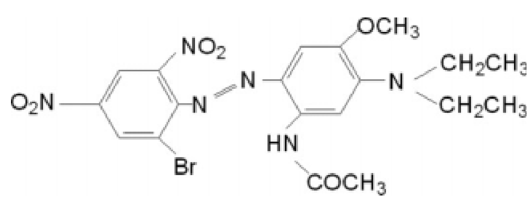

Figure 4 C. I. Disperse Blue 291.

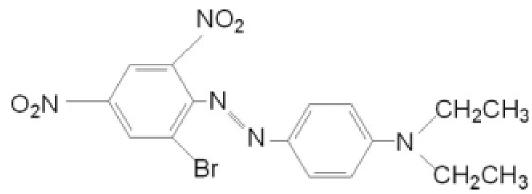

Figure 5 C. I. Disperse Violet 93.

maintaining agitation at 30°C. After continued agitation for 30 min, the system was then heated up to 75°C and was maintained at that temperature for 2.5 h. The double-shelled dye microcapsules were thus formed. To adjust the pH of the solution to 7, the microcapsules were then separated by vacuum filtration and washed with 10% v/v ethanol to remove disperse dye on the surface, and were dried under reduced pressure at room temperature for 24 h. Three microencapsulated dispersed dyes were named as MDD blue 291, MDD Violet 93, and MDD Orange 288, respectively.

Characterization

MDDs were then analyzed by scanning electron microscopy (SEM). SEM was performed using a JSM-5600LV (JEOL Co., Japan). Microcapsules were sprinkled onto a sample piece, sputter-coated with gold and examined under the microscope.

Transmission electron microscopy (TEM) measurements were performed on JSM-2100 (JEOL Co., Japan) microscope operating at 200 kV, by depositing a drop of CuO dispersion onto 300 mesh Cu grids coated with carbon layer.

Mean particle size and size distribution of microcapsules were determined using Mastersizer 2000 Particle Size Analyzer (Malvern, UK). The test with a few drops of suspension was carried out after dispersion by sonicator for 10 min.

UV-visible spectrophotometer experiments were conducted on a 752N instrument (Lengguang, China) at the maximal absorption wavelength of the dye. The dry melamine resin microencapsulated disperse dye powder was then analyzed in UV-visible to determine the amount of dyes entrapped in the microcapsules. The dye loading of the microcapsules was determined spectrophotometrically from a calibration curve of dye absorption at the maximal absorption wavelength after dissolving the microcapsule in 100 mL DMF at room temperature. The percentage of the content of dye was obtained.

Pretreatment scouring

All pretreatment were performed in a Rapid lab dyeing machine, using sealed stainless steel dye pots, LR = 20 : 1. The fabrics were initially scoured for 30 min at 85–90°C in a solution containing 2 gl^{-1} sodium carbonate and 2 gl^{-1} scouring agent, then were thoroughly rinsed with warm water (50°C) and finally samples were dried in a vacuum oven.

Auxiliary-free dyeing of MDD

Dyeings were carried out in an IR dyer (Newave, Taiwan): 5 g pieces of scoured fabric were dyed at a liquor ratio of 20 : 1.

The dyebaths were prepared with the MDD (10%, omf), adjusted pH value. Dyeing was performed at 40°C. Temperature in the dyebath was raised at a rate of 1°C/min until 130°C. Maintained at this temperature for 40 min, the dyebath was then rapidly cooled to 60°C, and the fabric was taken out afterward. The dye liquor was filtrated and poured into the dyepot again. Then, the fabric was placed into the dyepot, the temperature was raised to 80°C and held for 10 min, and then cooled down. The fabric was taken out and allowed to dry at room temperature.

The microencapsulated disperse dye black (10%, omf) is mixture of MDD Blue 291 (accounting for 3.36%), MDD Violet 93 (accounting for 2.13%), MDD Orange 288 (accounting for 4.51%).

Characterization of effluents of MDD

Characteristics of effluents generated in dyeing processes were assessed in terms of COD and color using standard methods for analysis of wastewater. The effect of recycling of the effluent from dyeing with microcapsule on fabric was analyzed using a Datacolor Spectraflash 600 (Plus CT, Switzerland) for the color measurements. The L^*, a^*, and b^* was based on the CIE.[13,14] In this system, the L^* value represents the dark-white, a^* represents the green-red and b^* represents the blue-yellow. The L^*, a^* and b^* values of the fabrics before dye adsorption were measured and taken as references. The color difference (CD) between scored or dyed and reference specimens were determined as shown in eq. (1):

$$\Delta E = \left[(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2\right]^{1/2} \qquad (1)$$

Dyed materials are generally accepted when the ΔE values are between 0 and 1.0 and the ΔL^* values are between –0.7 and 0.4. If the ΔE value is above

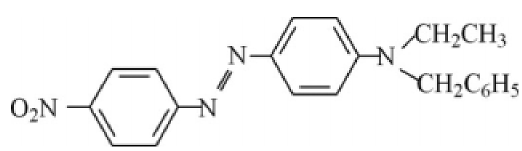

Figure 6 C. I. Disperse Orange 288.

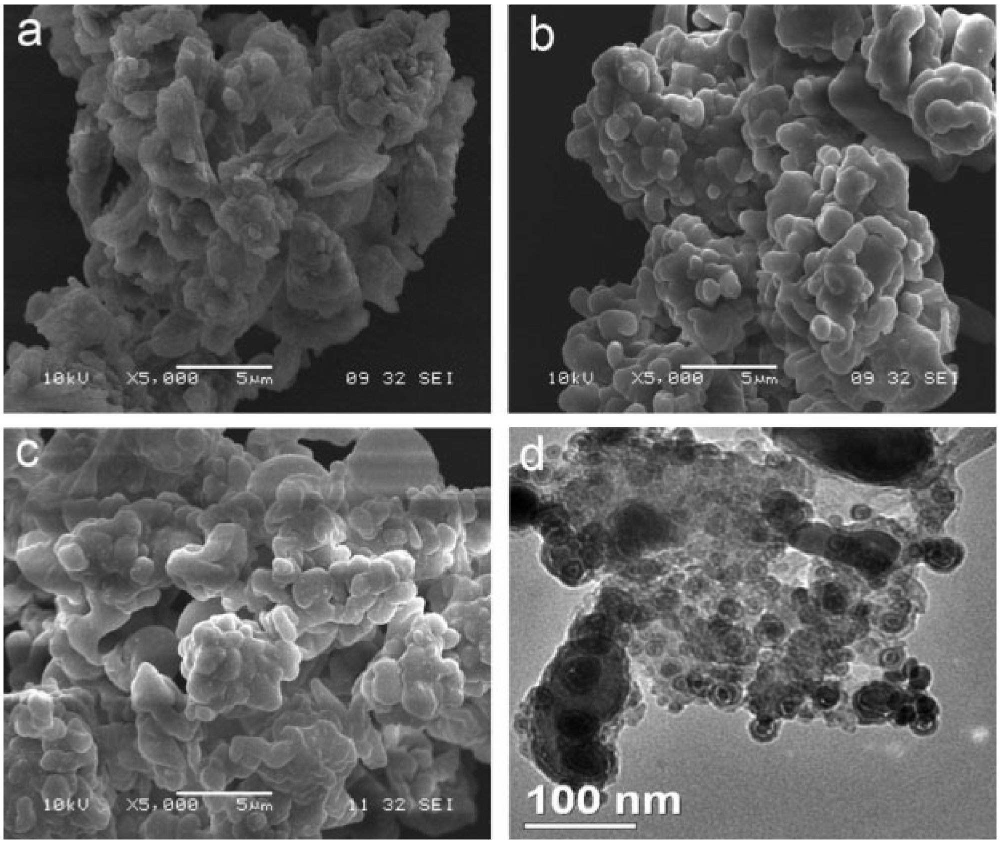

Figure 7 SEM photographs of MDD Blue 291, MDD Violet 93, MDD Orange 288, and TEM image of MDD Orange 288.

1.0, the color difference between sample and standard is very high and leads to rejection. The objective of this work was to determine the color reproducibility of dyeing in the effluent of MDD rather than to match the standard shade (dyeing in distilled water of MDD).

Color strength (*K/S*) on fabric

The relative color strength of dyed fabrics expressed as K/S value was determined as the reflectance technique by the Kubelka-Munk equation [eq. (2)].

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

R = Reflectance value in the maximum absorption wave length

K = Absorption coefficient

S = Scattering coefficient

Reflectance measurements were carried out using the same Datacolor spectrophotometer under illuminant D65 using the 10° standard observer. Each sample was folded twice to provide a total of four thicknesses and an average of five readings was taken at different positions.

Dye uptake

The dyed fabrics were completely washed with cold acetone and dried in a vacuum oven. The dried samples were weighed and then dyes were extracted using *N*,*N*-dimethylformamide at 120°C until the samples became colorless. The dye concentration in the extracts was measured using UV-visible

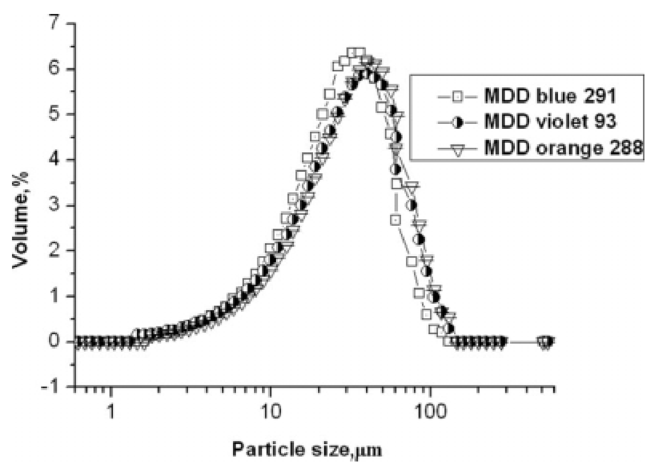

Figure 8 Size distribution curve for MDD.

TABLE I
Dyeing Characteristics of MDD Blue 291 on PET

Recycle	Dyed fabrics (λ_{max} = 630 nm)					Characteristics of effluent	
	ΔE	L^*	a^*	b^*	K/S	COD (mg·L^{-1})	Absorbency
Distilled water	–	–	–	–	15.11	48	0.020
First recycle	0.14	−0.14	0.03.	0.01	14.85	55	0.022
Second recycle	0.16	−0.14	0.07	0.02	15.19	59	0.025
Third recycle	0.24	−0.19	0.14	0.05	15.23	63	0.026
Fourth recycle	0.32	−0.03	0.10	0.30	14.90	68	0.028
Fifth recycle	0.31	−0.27	0.13	0.08	15.24	77	0.029
Sixth recycle	0.59	−0.50	0.28	0.14	15.46	86	0.031
Seven recycle	0.95	−0.56	0.24	0.73	15.36	95	0.032
Eight recycle	1.03	−0.69	0.21	0.74	15.47	101	0.035

spectrophotometer and the extent of dye adsorption was calculated; trials were repeated thrice.

The dye uptake was evaluated from eq. (3)

$$U(\%) = \frac{m_t}{m_o} \times 100 \quad (3)$$

where m_t is the amount of dye extracted from a dyed fabric at time t and m_o is the amount of dye in the dyebath.

Fastness test

The dyed samples were tested according to ISO standard methods. The specific tests were ISO 105 C06 (1994), color fastness to washing; ISO 105 B02 (1989), color fastness to light; and ISO 105 × 12 (1987), color fastness to rubbing.[15]

RESULTS AND DISCUSSION

Characterization of microencapsulated disperse dyes

The contents of C. I. Disperse Blue 291, C. I. Disperse violet 93 or C. I. Disperse Orange 288 were 49.83%, 49.85%, 49.86% in CDDs at a core/wall ratio 1 : 1.

The surface morphologies of MDDs are shown in Figure 7a–c; the microcapsules have a rough surface and non spherical appearance. The result of TEM [Fig. 7(d)] shows that MDDs are of multi-layers and their outer layers have a visible covering.

Fig. 8 presents the particle size distribution of the microcapsules with different disperse dyes. Mean size of microcapsules for MDD blue 291, violet 93, and Orange 288 were 32.70, 35.38, 36.53 μm, respectively. Moreover, the size distribution was similar and narrow.

Effect on color reproduction

The original dyeing was performed in distilled water and the dyed samples were used as the target shades for measuring repeatability. ΔE^* and ΔL^* values of the dyed sample produced in the present study are given in Table I. The L^*, a^*, b^* values of target and samples obtained from various recycling processes are given in Tables I–IV. It is observed from Tables I–IV that as the number of recycle processes increases, the ΔE values, COD, and absorbency of effluent increase. In MDD blue 291, the ΔE values increase from 0.44 in the first recycle to 1.07 in the eight recycle. COD values of effluent increase from 48 to 101 mg·L^{-1}, absorbency of effluent from 0.020 to 0.035.

Similarly in MDD Violet 93, the ΔE values increase from 0.45 in the first recycle to 1.55 in the fifth recycle. COD values increase from 50 to 103 mg·L^{-1}, absorbency from 0.023 to 0.055. In MDD Orange 288, the ΔE values increase from 0.35 in the first recycle to 1.25 in the fifth recycle. COD values increase from 51 to 121 mg·L^{-1}, absorbency from 0.021 to 0.069. In MDD Black, the ΔE values increase from 0.32 in the

TABLE II
Dyeing Characteristics of MDD Violet 93 on PET

Recycle	Dyed fabrics (λ_{max} = 570 nm)					Characteristics of effluent	
	ΔE	L^*	a^*	b^*	K/S	COD (mg·L^{-1})	Absorbency
Distilled water	–	–	–	–	24.27	50	0.023
First recycle	0.45	−0.07	−0.23	0.38	24.06	59	0.034
Second recycle	0.62	−0.15	−0.31	0.52	24.11	65	0.036
Third recycle	0.51	−0.29	−0.26	0.33	24.69	79	0.045
Fourth recycle	0.59	−0.37	−0.19	0.42	24.87	90	0.049
Fifth recycle	1.55	−1.37	−0.37	0.62	25.16	103	0.055

TABLE III
Dyeing Characteristics of MDD Orange 288 on PET

	Dyed fabrics (λ_{max} = 470nm)					Characteristics of effluent	
Recycle	ΔE	L^*	a^*	b^*	K/S	COD (mg·L^{-1})	Absorbency
Distilled water	–	–	–	–	21.26	51	0.021
First recycle	0.35	−0.04	−0.16	0.31	21.08	63	0.027
Second recycle	0.48	−0.23	−0.25	0.34	22.09	72	0.039
Third recycle	0.70	−0.15	−0.25	0.64	21.18	89	0.043
Fourth recycle	0.82	−0.23	−0.47	0.63	21.01	95	0.058
Fifth recycle	1.25	−0.48	−0.70	0.93	21.37	121	0.069

TABLE IV
Dyeing Characteristics of MDD Black on PET

	Dyed fabrics (λ_{max} = 590 nm)					Characteristics of effluent	
Recycle	ΔE	L^*	a^*	b^*	K/S	COD (mg·L^{-1})	Absorbency
Distilled water	–	–	–	–	18.90	52	0.022
First recycle	0.32	−0.21	0.01	−0.24	19.30	61	0.031
Second recycle	0.47	−0.37	0.10	−0.27	19.55	69	0.039
Third recycle	0.79	−0.73	0.00	−0.31	20.39	78	0.043
Fourth recycle	0.82	−0.70	0.03	−0.43	20.14	83	0.050
Fifth recycle	0.89	−0.80	0.05	−0.39	20.42	95	0.056
Sixth recycle	1.02	−0.93	0.12	−0.40	20.57	109	0.061

first recycle to 1.02 in the sixth recycle. COD values increase from 52 to 109 mg·L^{-1}, absorbency from 0.022 to 0.061. Dyed materials are generally accepted when the ΔE values are between 0 and 1.0. It can inferred from Tables I–IV that in MDD blue 291, Violet 93, Orange 288, Black, usage of recycled effluents can be carried out 7, 4, 4, 5 times, respectively. In the conventional disperse dye dyeing, COD

TABLE V
Dye Uptake and Fastness of Dyed Fabric Using Recycling Waste Water

		Rubbing		Washing							
MDD	Recycle	Dry	Wet	W	PAN	PET	N	C	D	Light	Dye uptake
Blue 291	0	4/5	4	3/4	4/5	4	3/4	4/5	4	4	97.30
	1	4/5	4	3/4	4/5	4	3/4	4/5	4	4	97.42
	2	4/5	4	3/4	4/5	4	3/4	4/5	4	4	97.39
	3	4/5	4	3/4	4/5	4	3/4	4/5	4	4	97.46
	4	4/5	4	3/4	4/5	4	3/4	4/5	4	4	97.27
	5	4/5	4	3/4	4/5	4	3/4	4/5	4	4	97.33
	6	4/5	4	3/4	4/5	4	3/4	4/5	4	4	97.26
	7	4/5	4	3/4	4/5	4	3/4	4/5	4	4	97.24
Violet 93	0	4/5	4	3	4	3/4	3/4	3/4	2/3	4/5	97.23
	1	4/5	4	3	4	3/4	3/4	3/4	2/3	4/5	97.41
	2	4/5	4	3	4	3/4	3/4	3/4	2/3	4/5	97.35
	3	4/5	4	3	4	3/4	3/4	3/4	2/3	4/5	97.26
	4	4/5	4	3	4	3/4	3/4	3/4	2/3	4/5	97.15
Orange 288	0	4/5	4	3	4	4	3/4	3/4	2/3	4/5	97.29
	1	4/5	4	3	4	4	3/4	3/4	2/3	4/5	97.20
	2	4/5	4	3	4	4	3/4	3/4	2/3	4/5	97.18
	3	4/5	4	3	4	4	3/4	3/4	2/3	4/5	97.32
	4	4/5	4	3	4	4	3/4	3/4	2/3	4/5	97.25
Black	0	4/5	4	3/4	4/5	3/4	3/4	4	3	4	–
	1	4/5	4	3/4	4/5	3/4	3/4	4	3	4	–
	2	4/5	4	3/4	4/5	3/4	3/4	4	3	4	–
	3	4/5	4	3/4	4/5	3/4	3/4	4	3	4	–
	4	4/5	4	3/4	4/5	3/4	3/4	4	3	4	–
	5	4/5	4	3/4	4/5	3/4	3/4	4	3	4	–

W, wool; PAN, acrylic; PET, polyester; N, nylon; C, cotton; D, cellulose acetate.

values of wastewater are above 1000 mg·L^{-1}, color of dyeing wastewater is dark. In MDDs dyeing, COD values are much smaller of the effluent after simple filtration; wastewaters are nearly colorless.

The properties of samples, such as the fastness to rubbing, the fastness to washing and the fastness to light, were not related to dye solution being recycled. There was no difference between the dyeing processes with distilled water and the dyeing process with recycled effluent.

On the other hand, the dye uptake values given in Table V show that the use of MDD reaches as high as 97%, the remains of disperse dyes in microcapsules are less than 3%.

Effect in scouring using effluent of MDD

To assess the reuse of dyeing effluents, scouring experiments on polyester fabrics were carried out using various recycling effluents from different MDDs and distilled water for comparison. The color difference and the darkness or lightness of treated samples were evaluated.

Color difference of treated samples are generally accepted when the ΔE values are between 0 and 1.0 and the ΔL^* values are between −0.7 and 0.4. If the ΔL^* values are less than −0.7 the samples are darker in shade and if it is greater than 0.4 the samples are lighter in shade compared to that of the standard sample.

There was some color difference between the scouring carried out with distilled water and the scouring carried out with recycling effluent. The results are given in Figure 9. It is observed that as the number of recycle processes increases, the ΔE values increase. These values are acceptable if they remain less than 0.5.

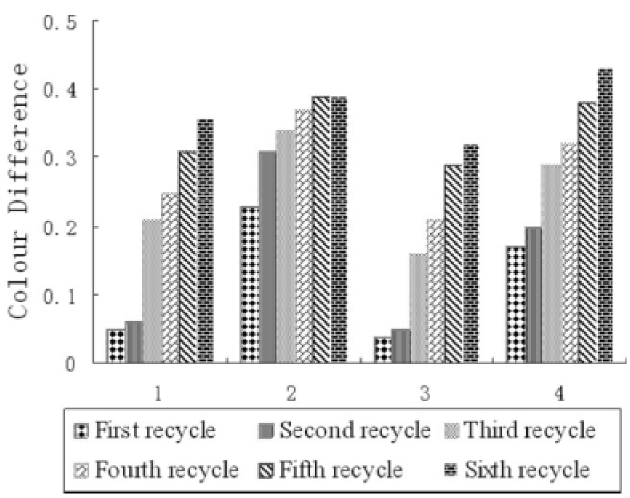

Figure 9 Color difference of samples obtained from various recycling processes.

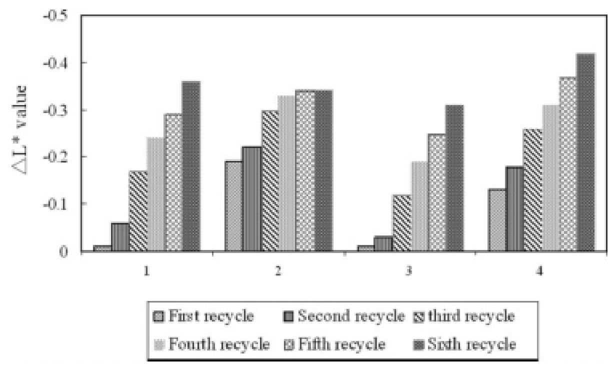

Figure 10 Lightness of samples obtained from various recycling processes.

1 MDD Orange 288 2 MDD Violet 93 3 MDD blue 291 4 MDD black

The darkness or lightness of the color intensity on polyester fabrics was measured by comparing the values of ΔL^* (+ve means lightness whereas −ve means darkness, ΔL^* values are acceptable if they range from +0.5 to −0.5) against polyester fabric treated with distilled water. The ΔL^* values given in Figure 10 show that ΔL^* are –ve all, which indicates that polyester fabrics scoured with recycling effluent are darker than those with distilled water. In practice, ΔL^* values range from 0 to −0.5. So the recycling effluent of MDDS after simple filtration can be reused in scouring.

1. MDD Orange 288 2. MDDD Violet 93 3. MDD blue 291 4. MDD black

CONCLUSIONS

The process proposed by this study has the following advantages:

(1) The shell of MDD can be described as a semi-transparent membrane which, under certain condition, allows water to get in and dissolve dye molecules to penetrate through it slowly. No surfactants are needed. When PET fabric is in the dye bath, dye molecules are adsorbed onto and diffused into the fiber to realize the dyeing. The balances of the dyes in the bath are broken and dissolution and diffusion of dyes in the microcapsule continue based on Le Chatelier's Principle (eq. (4)).

$$\mathrm{Dye}_{\mathrm{solid}} \Leftrightarrow \mathrm{Dye}_{\mathrm{capsule}} \Leftrightarrow \mathrm{Dye}_{\mathrm{dissolved}} \quad (4)$$

The course of "dissolution ∼ diffusion ∼ adsorption ∼ penetration" continued until the required shade was obtained. The semi-transparent membrane serves as a dispersing and leveling agent of the conventional dyeing process.

(2) For MDD dyeing, recycling tests were carried out with effluents treated by simple filtration for dyeing and scouring. Acceptable change in color difference and ΔL^* were observed. At the same time, fastness performance of treated fabrics was similar to the control. MDDS can surely be used in dyeing PET without using surfactants, and the effluents can be recycled and reused.

This work was supported by the fund toward the provincial project in the area of pollution prevention of Jiangsu.

References

1. Savarino, P.; Parlati, S.; Buscaino, R.; Piccinini, P.; Degani, I.; Barni, E. Dyes Pigments 2004, 60, 223.
2. Akcakoca, E. P.; Ozguney, A. T.; Atav, R. Dyes Pigments 2007, 72, 23.
3. Allègra, C.; Moulin, P.; Maisseu, M.; Charbit, F. J Membr Sci 2006, 269, 15.
4. Xu, X.-R.; Li, H.-B.; Wang, W.-H.; Gu, J.-D. Chemosphere 2004, 57, 595.
5. Karim, M. M.; Das, A. K.; Lee, S. H. Anal Chim Acta 2006, 576, 37.
6. Alaton, I. A.; Insel, G.; Eremektar, G.; Babuna, F. G.; Orhon, D. Chemosphere 2006, 62, 1549.
7. Rao, V. V. B.; Mohan Rao, S. R. Chem Eng J 2006, 116, 77.
8. Hessel, C.; Allegre, C.; Maisseu, M.; Charbit, F.; Moulin, P. J Environ Manage 2006, 1.
9. Sirianuntapiboon, S.; Srisornsak, P. Bioresour Technol 2007, 98, 1057.
10. Senthilkumar, M.; Muthukumar, M. Dyes Pigments 2007, 72, 251.
11. Metwally, M. A.; Khalifa, M. E.; Amer, F. A. Dyes Pigments 2006, 1.
12. Yi, Z.; Jihong, F.; Shuilin, C. Color Technol 2005, 121, 76.
13. Lee, J. J.; Lee, W. J.; Choi, J. H.; Kim, J. P. Dyes Pigments 2005, 65, 75.
14. Mo, J. H.; Lee, Y. H.; Kim, J.; Jeong, J. Y.; Jegal, J. Dyes Pigments 2006, 1.
15. Burkinshaw, S. M.; Lagonika, K. Dyes Pigments 2006, 69, 185.