Preparation of Nanoscale Waterborne Disperse Dye Dispersion by Phase Separation Technique for Inkjet Printing Ink

Shaohai Fu, Lei Ding, Anli Tian, Xia Zhang, and Chaoxia Wang, Key Laboratory for Eco-Textile of Ministry of Education, Jiangnan University, 1800 Lihu Road, Wuxi 214122, Jiangsu, P. R. China

Abstract

C.I. disperse dye blue 60 was encapsulated with poly(styrene-maleic acid) by phase separation method and followed by the preparation of encapsulated disperse dye dispersion. Experimental results showed that sodium hydroxide and ammonia provided the dispersion the smaller particle size as compared with other additives. An optimal process was attained when mass ratio of NaOH to poly(styrene-maleic acid) was about 0.5, mass ratio of SDBS or OP-10 to encapsulated disperse dye was about 15% and dispersed with bead mill or ultrasonic machine. The encapsulated C.I. disperse dye blue 60 dispersion had narrow particle size distribution and excellent stability to centrifugal force, freeze-thaw treatmen. The rheological behavior of the dispersion was close to Newtonian fluid.

INTRODUCTION

Inkjet printing is one of the fastest growing textile printing technologies, in addition to other advantages, it is more eco-friendly, requires low water and energy consumption, and has no or minimal residue dye water in comparison with conventional printing technologies [1, 2].

Disperse dye has become the main colorants for polyester in inkjet printing technology due to its high performance in hue, brilliant and color strength [2]. However, just as similar to the pigment, the disperse dye always exists in aggregation or coagulation and were hard to be dispersed in aqueous media [3]. In order to prepare the disperse dye dispersion for formulation into inkjet printing ink, considerable works have been done by some researchers. For example, Daubach et.al modified the disperse dye using spray drying method [4]. Kim et.al, Choi et.al and Chang et.al prepared the core-shell disperse dye composite using emulsion polymerization [5-7]. Lee et.al, Chrysavgi et.al and Shen et.al obtained stable dispersion with small particle size when the disperse dye was dispersed with aid of dispersant [8-10]. Although there are so many methods for disperse dye modification, how to prepare the disperse dye dispersion with high stability, small particle size and narrow particle size distribution were still an obstacle for wide application of disperse dyes in inkjet printing technology.

Phase separation is effectively method to prepare the core-shell composite. This technique has already been applied to fabricate core-shell pigment materials [11]. It has proved that the stability of the dispersion can be greatly improved when amphiliphic copolymer was used as shell materials [12]. However, according to my knowledge, this method was rarely applied for disperse dye modification.

In order to improve the quality of disperse dye dispersion and inkjet printing ink, in this paper, we used C.I. disperse dye blue 60 as core and poly(styrene-maleic acid) as shell materials, and prepared the core-shell composite by phase separation method. Further the properties of the dispersion and inkjet printing ink which prepared using these materials were investigated.

EXPERIMENTAL

Materials and method

C.I. disperse dye blue 60 （disperse dye, press cake, water content 47%, its chemical structure was shown in Chart 1, Yabang Dyestuff Co., Ltd, Changzhou, China）was dried before using. Poly (styrene-maleic acid) (molar ratio of styrene to maleic acid was about 0.56, Mn=9000, Nanocolorants and Digital Printing R&D Centre of Jiangnan University, Wuxi, China). NaOH, Acetone, Glycerol, Ethylene glycol mono-methyl ether and Tween-80（analytical grade，Lingfeng Chemical Reagent Co., Ltd., Shanghai, China）. All the distilled water was used in the experimental.

Encapsulated disperse dye by phase separation method

10g poly(styrene-maleic acid) was dissolved in 190g acetone, and then 50g C.I. disperse dye blue 60 was dispersed into the above solution. The mixture was stirred for 30min and then transferred to Ultra Turrax IKA T18 Basic (IKA Instruments, Ltd, Staufen, and German) and dispersed for 2h at rate of 25000r/min, and then 250g ethanol was dropwisely added into the above mixture. The mixture was centrifuged to get some slurry. The slurry was washed with ethanol for three times and then dried to obtain the powder of encapsulated disperse dye.

Preparation of encapsulated disperse dye dispersion

10g encapsulated disperse dye, corresponding amount of surfactant and alkali was dispersed into 89.5g distilled water. The pH value of the dispersion was adjusted to 9 using 0.1mol/L sodium solution. The dispersion was treated with different method, and then the encapsulated disperse dye dispersion was obtained.

Preparation of comparative disperse dye dispersion

9g poly(styrene-maleic acid) was dissolved in 203.5g distilled water. After pH value of the solution was adjusted to 9.0 by adding 0.1mol/L sodium solution, and then 37.5g disperse dye was added. The comparative disperse dye dispersion was obtained after the mixture was treated by ultrasonic until the particle size didn’t change any more.

Transfer printing

The inks were prepared by prepared dispersion. The formulation in a weight basic was given as follows: the dispersion 20%, glycerol 21%, and ethylene glycol mono-methyl ether 9%, Tween-80 1% and distilled water 49%. The above components were mixed and filtered through a 0.5 μm pore filtering sieve. The ink loaded on inkjet printing machine (Mimaki JV4-180, Pizeo-electric inkjet printer, Shinagawa Tokyo, Japan) and printed on transfer printing paper. The polyester fabrics and printed paper was stacked together and then heated at different temperature for 30s.

Characterization

One drop of the dispersion was diluted by distilled water, and then placed on a 400-mesh carbon-coated copper grid and dried in the air. The morphologies of comparative disperse dye and encapsulated disperse dye were characterized with a transmission electron microscope (TEM, JEM-100SX, Japan). FTIR spectra of poly(styrene-maleic acid) (in KBr pellet), original disperse dye (in KBr pellet) and encapsulated disperse dye (in KBr pellet) were recorded on a Nicolet Nexus 560 FTIR spectrometer (Thermo Electron Corporation).

The particle size and its distribution of the dispersion which was diluted to 1000 times were measured by Nano-ZS90. The viscosity against shear rate of the dispersion was performed with Brookfield DV-III (Brookfield Company, Massachusetts, and America). The maximum absorbance at 630nm of the dispersion was measured when the dispersion was centrifuged at different speed for 30 min. The sample was sealed and stored at -5°C for 12 h and then put into an oven at 60°C for 12 h. The freeze-thaw stability is evaluated by comparing the particle size distribution before and after freeze-thaw treatment. *K/S* value of the printed fabrics were measured by colorimeter (Xrite-8400, America) under illuminant D65 using the 10 standard observer.

RESULT S AND DISCUSSION

Preparation of encapsulated disperse dye

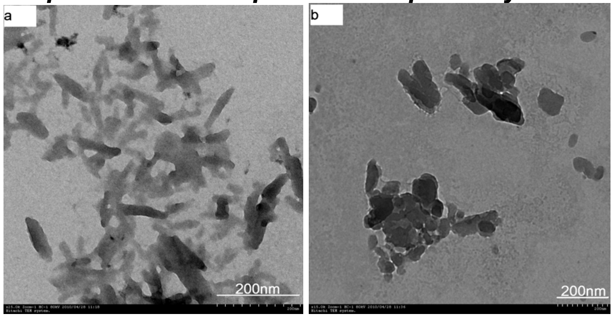

Figure 1 TEM imagines of the (a) original disperse dye and (b) encapsulated disperse dye

Figure 1 showed that the mean particle size of encapsulated disperse dye was about 100nm. Some particles were aggregated during the dispersion was dried. Comparing with the morphology of the two samples, we concluded that the disperse dye was encapsulated with poly(styrene-maleic acid) by phase separation method.

These results can be further proved by the FTIR curves of encapsulated disperse dye. Figure 2 indicated that all the vibration bands corresponding to poly(styrene-maleic acid) (C=O stretching vibration at 1726 cm^{-1}, the–O-H stretch at 3443cm^{-1}, C= C on benzene ring stretching vibration at 1454 cm^{-1}, 1495 cm^{-1} and 1601 cm^{-1}) are presented in the FTIR spectrum. Interestingly, all these absorbance peaks appeared in the FTIR spectrum of encapsulated disperse dye and do not appear in the FTIR spectrum of the original disperse dye, this resulted illustrated that some disperse dye was effectively encapsulated by poly(styrene-maleic acid).

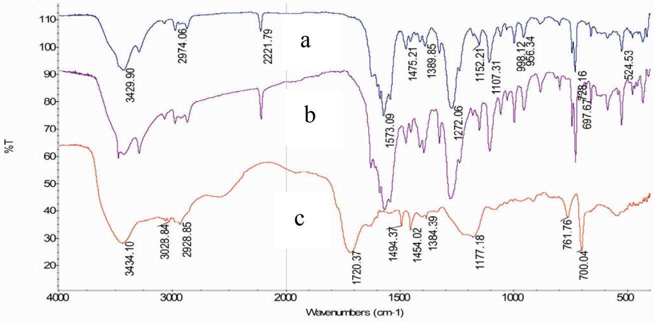

Figure 2 Image of infrared spectrum (a) original disperse dye; (b) encapsulated disperse dye; (c) poly(styrene-maleic acid)

Preparation of encapsulated disperse dye dispersion

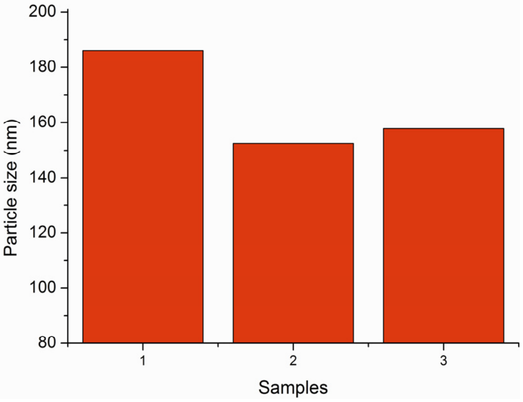

Figure 3 Effect of dispersing method on particle size of encapsulated disperse dye dispersion (1) mechanical stirring (2) bead mill (3) ultrasound.

Some process conditions, such as dispersing method, neutralization reagents and amount of surfactant can affect the particle size of the encapsulated disperse dye dispersion. Figure 3 showed the effect of dispersing method on the particle size of the encapsulated disperse dye dispersion. It can be seen that the encapsulated disperse dye also needed strong dispersing force, such as bead milling or ultrasonic treatment. These results indicated that the encapsulated disperse dye also aggregated in drying process when prepared by phase separation technique.

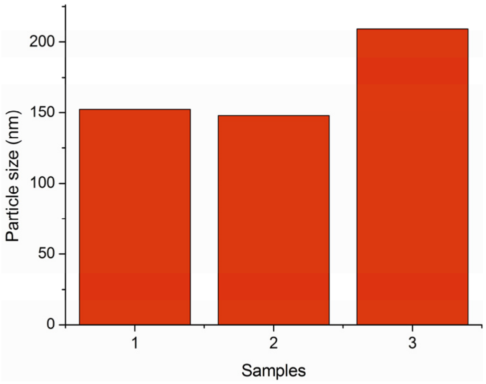

Figure 4 Effect of neutralization reagents on particle size of encapsulated disperse dye dispersion (1) sodium hydroxide (2) ammonia (3) triethanoamine.

Figure 4 showed that sodium hydroxide and ammonia can provide the smaller particle size to the encapsulated disperse dye dispersion than that of triethanoamine. Sodium hydroxide and ammonia could penetrate into solvated layer and then react with –COOH for their small molecular size, which resulted in increasing the amount of –COO$^-$ on encapsulated disperse dye surface. Therefore, the aggregated particles were easily separated for the repulsive forces, thus resulting in smaller particle size. Triethanolamine was a Lewis base which reacted with –COOH via isolated electrons on nitrogen atoms, its large molecular size prevents it penetrating into the solvated layer. As a result, -COOH groups on encapsulated disperse dye could not be neutralized enough. Thus the particles could not be separated under shear forces, which resulted in larger particle size.

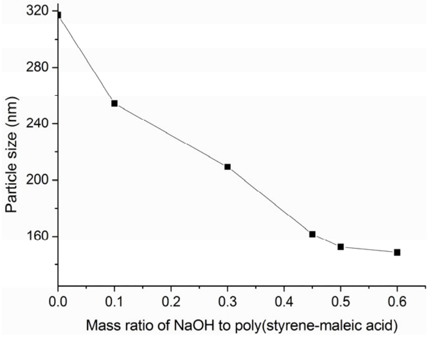

Figure 5 Effect of amount of NaOH on particle size of encapsulated disperse dye dispersion

The amount of sodium hydroxide used in preparation of encapsulated disperse dye dispersion was important as well. Figure 5 showed that the particle size of encapsulated disperse dye dispersion decreased with an increase of amount of NaOH, and reached the smallest when mass ratio of NaOH to poly(styrene-maleic acid) was higher than 0.5. The reason was that few negative charges produced when amount of sodium hydroxide was small, thus resulted in large particle size. On the other hand, the disperse dye hue would change when amount of NaOH was high enough.

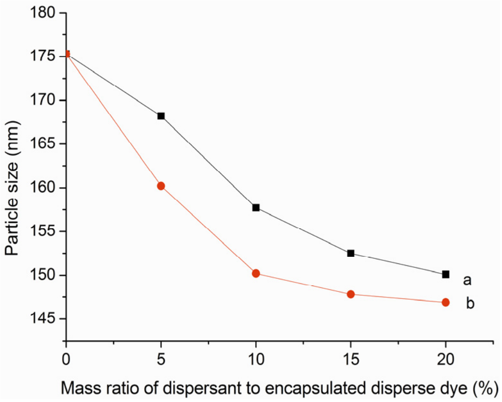

Figure 5 Effect of amount of low molecular dispersant on particle size of encapsulated disperse dye dispersion, (a) OP-10 (b) SDBS

The low molecular dispersant can also help to reduce the particle size of the encapsulated disperse dye dispersion. This result was shown in Figure 6. It can be seen that the particle size decreased with an increase of amount of low molecular dispersant. The low molecular dispersant can reduce the surface energy of the water, which resulted in high wetting speed and dispersing efficiency, thus led to small particle size.

Performance of encapsulated disperse dye dispersion

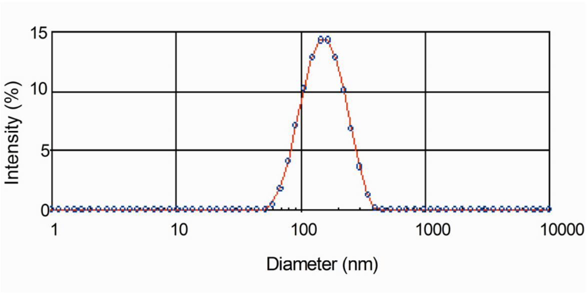

Figure 6 Particle size distribution of encapsulated disperse dye dispersion

Figure 6 showed the particle size distribution of encapsulated disperse dye dispersion. It can be seen that the smallest particle size was about 40nm and the largest particle size was about 400nm, the mean particle size was about 154.7 nm, which was far smaller than the diameter of the nozzle (40μm). These results indicated that the encapsulated disperse dye can be used to formulate inkjet printing ink.

Figure 7 showed that the encapsulated disperse dye dispersion exhibited a quite stable viscosity when the shear rate was in the range of 10 to 60 sec^{-1}. As mentioned above, the poly(styrene-maleic acid) were completely encapsulated on disperse dye surface. The stretched chain was short. Therefore, there was no obvious fluctuation on viscosity as shear rate increased. According to some reference, the rheological behavior of the dispersion that was suitable for formulation inkjet printing ink needs to be as close to Newtonian as possible.[13] Figure 5 showed that only slight changes in viscosity with increasing the shear rates of the encapsulated disperse dye dispersion, which indicated that it was suitable for preparation of inkjet printing inks.

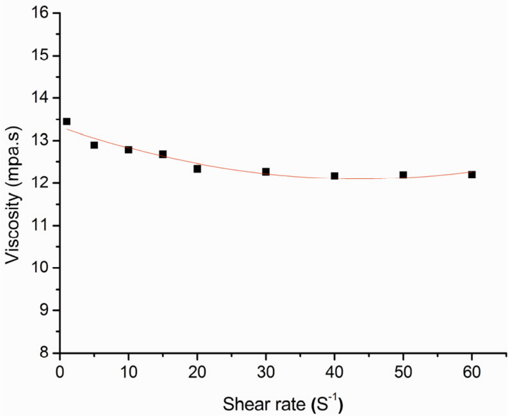

Figure 7 Effect of shear rate on viscosity of encapsulated disperse dye dispersion

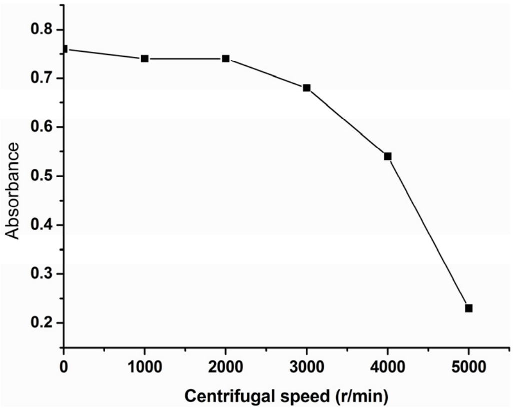

Figure 8 Effect of centrifugal stability on absorbance of encapsulated disperse dye dispersion

Figure 8 indicated that the absorbance of encapsulated disperse dye dispersion changed small when centrifugal speed was lower than 3000r/min. The sedimentation speed of the disperse dye particles would be offset by the Brownian motion at low centrifugal speed, thus the absorbance changed small. However, when centrifugal speed was higher than 3000r/min, some large particles would be deposited for high centrifugal forces, thus led to a large changing rate of absorbance.

Figure 9 showed the particle size distribution of encapsulated disperse dye dispersion before and after freeze-thaw treatment. It can be seen that the particle size distribution changed small after freeze-thaw treatment. The reason may be due to that in encapsulated disperse dye dispersion, the attractive force between poly(styrene-maleic acid) and disperse dye was large for completely encapsulation, and it was hard to be peeled off at freeze-thaw treatment temperature. From Figure 8 and Figure 9, we may conclude that the encapsulated disperse dispersion had an excellent stability to storage.

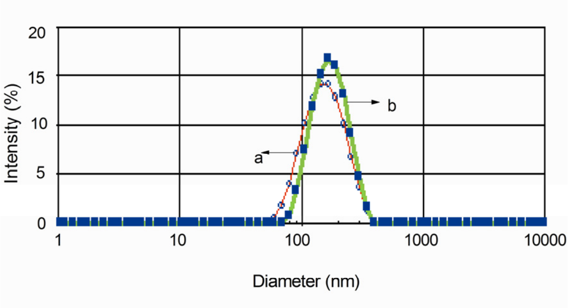

Figure 9 Particle size distribution of encapsulated disperse dye dispersion before and after freeze-thaw treatment

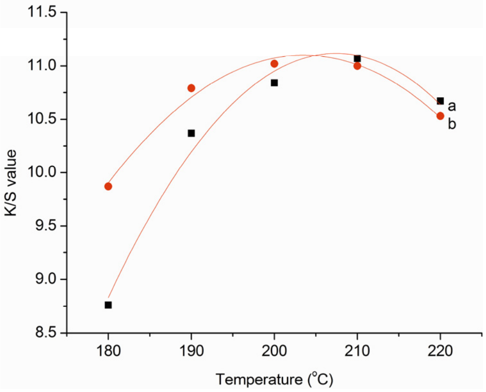

Figure 10 Effect of transfer temperature on K/S value of encapsulated disperse dye dispersion, (a) encapsulated disperse dye dispersion (b) comparative dispersion.

Figure 10 showed the transfer temperature on K/S value of encapsulated disperse dye dispersion. It can be seen that the sublimation of the disperse dye increased a little after encapsulated by poly(styrene-maleic acid). The reason may be due to that some energy was needed when the disperse dye penetrated from the polymer layer.

Conclusions

The phase separation method was an effective method to prepare the encapsulated C.I. disperse dye blue 60. The encapsulated C.I. disperse dye blue 60 dispersion had narrow particle size distribution and excellent stability to freeze-thaw treatment and centrifugal forces. The sublimation of the disperse dye increased a little after encapsulated by poly(styrene-maleic acid).

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Author Biography

Associate professor Shaohai Fu received his PhD in textiles from Jiangnan University (2006). Since then he has worked southern Yangtze University. His work has focused on the development of inkjet printing technology