Preparation of Encapsulated Disperse Dye Dispersion for Polyester Inkjet Printing Ink

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ABSTRACT: Encapsulated C.I. disperse dye blue 60 (disperse dye) was prepared with poly(styrene-maleic acid) via phase separation method, followed by the preparation of dispersion and inkjet printing ink. The results showed that the encapsulated disperse dye dispersion had a small particle size, narrow particle size distribution, and excellent stability to centrifugal force, freeze-thaw treatment, polyols and alkali and its rheological behavior belong to Newtonian fluid. The ink that prepared using this dispersion satisfied with the requirement of Mimaki JV4-180 and showed an excellent printing performance at room temperature. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 121: 1616–1621, 2011

Key words: encapsulated; C.I. disperse dye blue 60; inkjet printing ink; phase separation method; dispersion

INTRODUCTION

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

Disperse dye as the main colorant for polyester, had high performance in hue, brilliant, and color strength.[2] However, just as similar to the pigment, disperse dye also existed in aggregation or coagulation and was hard to be dispersed in aqueous media.[3,4] To improve the stability of disperse dye dispersion and to reduce its particle size, many methods have been done by the researchers such as spray and drying method,[5,6] emulsion or mini-emulsion polymerization method,[7–9] interfacial polymerization method,[10–13] direct dispersing with aid of dispersant.[14–16] Although there were so many methods for the modification of disperse dye, how to prepare the dispersion with high stability, small particle size, and narrow particle size distribution were still an obstacle for its wide application in inkjet printing technology.

Phase separation method had already been applied to fabricate the core-shell pigment composite materials.[17] It is proved that the stability of dispersion could be greatly improved when amphiliphic copolymer was used as shell materials.[18] However, this method was rarely used for the modification of disperse dye.

To improve the quality of disperse dye ink for inkjet printing, we used C.I. disperse dye blue 60 as core and poly(styrene-maleic acid) as shell materials and prepared the encapsulated disperse dye by phase separation method. The properties of the dispersion and the ink that was prepared using these materials were also investigated.

EXPERIMENTAL

Materials and methods

C.I. disperse dye blue 60 (disperse dye, its chemical structure was shown in Chart 1, press cake, water content 47%, Yabang Dyestuff, Changzhou, China) was dried before using. Poly(styrene-maleic acid) (molar ratio of styrene to maleic acid was about 0.56, M_n = 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, Shanghai, China). All the distilled water was used in the experimental.
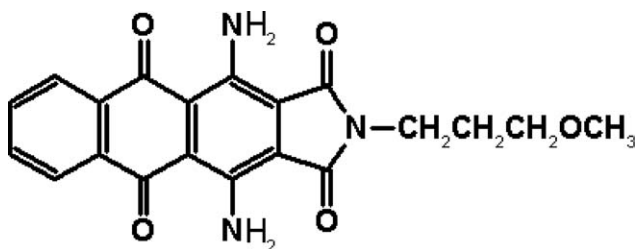
Chart 1 Chemical structure of C.I. disperse dye blue 60.

Preparation of encapsulated disperse dye dispersion

Ten grams of poly(styrene-maleic acid) was dissolved in 190 g acetone, and then, 50 g disperse dye was added into the above solution. The mixture was stirred for 30 min and then transferred to an Ultra Turrax IKA T18 Basic (IKA Instruments, Staufen, German) and dispersed at the rate of 25,000 r/min for 2 h, after that, 250 g ethanol was dropped into the above mixture. Finally, 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 the encapsulated disperse dye.

Ten grams of encapsulated disperse dye and 0.5 g of Tween-80 were dispersed into 89.5 g distilled water. The pH value of the dispersion was adjusted to 9 using 0.1 mol/L sodium solution. The dispersion was dispersed with a Branson Sonifier W450 Digital for 30 min at 90% amplitude to get the encapsulated disperse dye dispersion.

Preparation of comparative disperse dye dispersion

Nine grams of poly(styrene-maleic acid) was dissolved into 203.5 g distilled water. After the pH value of the solution was adjusted to 9 using 0.1 mol/L sodium solution, 37.5 g disperse dye was added. The mixture was treated with a Branson Sonifier W450 Digital for some time until the particle size did not change any more, and then the comparative disperse dye dispersion was prepared.

Ink formation

Inkjet printing ink was prepared with encapsulated disperse dye dispersion. The formulation in a weight basic was given as follows: encapsulated disperse dye dispersion 20%, glycerol 13%, ethylene glycol mono-methyl ether 12%, Tween-80 1.5%, and distilled water 53.5%. The above components were mixed under stirring until a homogeneous dispersion was obtained. The ink was prepared after filtered through a 0.5-μm pore filtering sieve and then loaded on inkjet printing machine (Mimaki JV4-180, Pizeo-electric inkjet printer, Shinagawa Tokyo, Japan).

Characterization

Properties of encapsulated disperse dye

One drop of the sample, which was diluted by distilled water for 1000 times, was placed on a 400-mesh carbon-coated copper grid and then dried in the air. The morphologies of the sample were observed with a transmission electron microscope (TEM, JEM-100SX, Akishima, Japan). Thermogravimetric analyses (TGA) were performed by Diamond TG/DTA Analyzer (Perkin-Elmer instruments, Shanghai, China), with a heating rate of 10°C/min under the air atmosphere. The samples were diluted to 1000 times by distilled water. Zeta potential of the particle against the temperature was measured by Nano-ZS90 (Malvern Instruments, Worcestershire, England).

Properties of encapsulated disperse dye dispersion

The encapsulated disperse dye dispersion was diluted to 1000 times, and then its particle size distribution was measured by Nano-ZS90. The dispersion was equilibrated at 25°C for 10 min, and its apparent viscosity against shear rate was performed with Brookfield DV-III (Brookfield Company, MA).

The maximum absorbance of the dispersion at 630 nm was conformed using a UV/Vis spectrophotometer (UV-2000 and UV-2100 model, UNICO Instruments, Shanghai, China). The absorbance of the dispersion at 630 nm was measured, when it was centrifuged at a desired 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 another 12 h. The freeze-thaw stability was evaluated by comparing the changes of particle size distribution before and after freeze-thaw treatment.

Thirteen grams of glycerol and 12 g of ethylene glycol mono-methyl ether were added into 75 g encapsulated disperse dye dispersion and stirred for 30 min, and then the particle size distribution of the dispersion was measured by Nano-ZS90. The pH value of the dispersion was adjusted to 10 using 0.1 mol/L NaOH solution, and the particle size distribution at different pH value was measured by Nano-ZS90.

Physical properties of inkjet printing ink

The viscosity was measured using Brookfield DV-III at 25°C with shear rate 30 s^{-1}. The surface tensions were measured using a ring method in Drop Shape Analysis System DSA100. The particle size was measured by Nano-ZS90. Freeze-thaw stability and the absorbance at 630 nm were measured according to above method. The printing performance was tested on Mimaki JV4-180. The clogging nozzle rate (B) was calculated according to eq. (1):
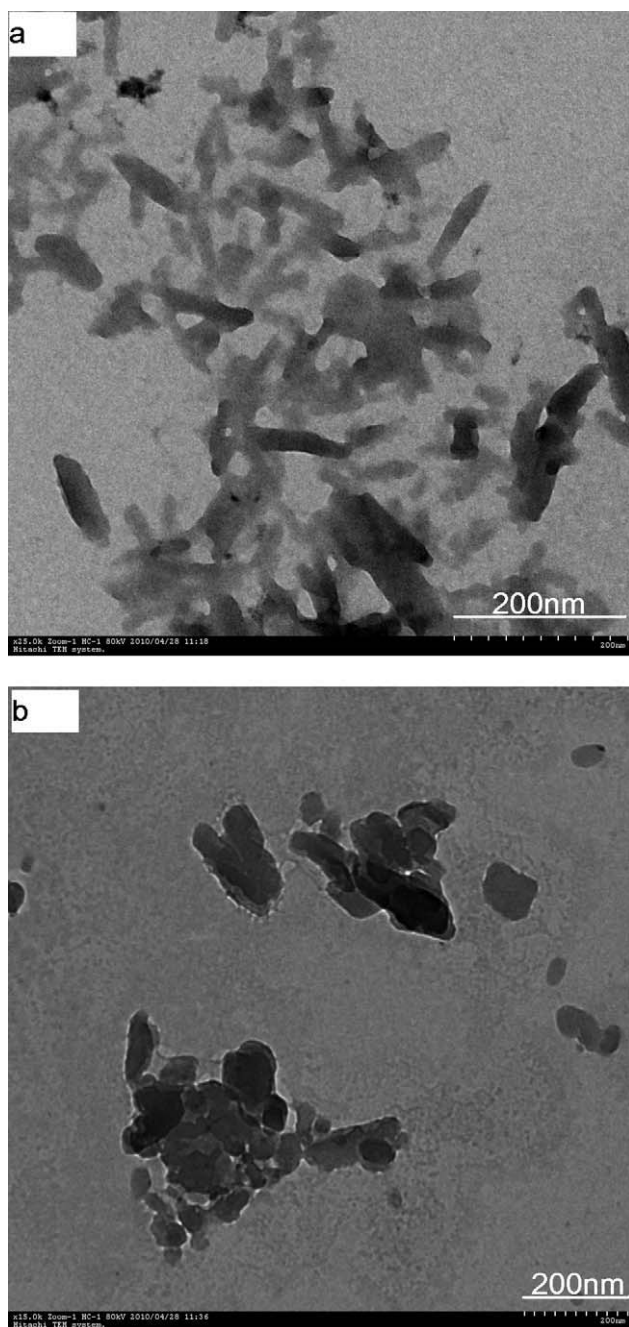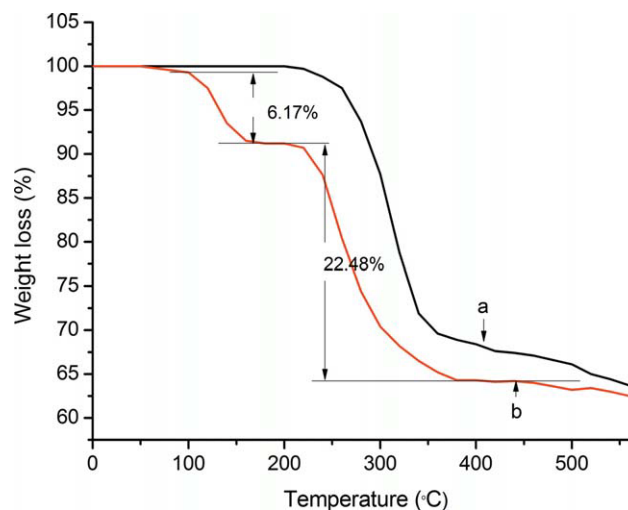
Figure 2 TGA imagines of the (a) original disperse dye and (b) encapsulated disperse dye. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

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

$$B = \frac{C_1}{S} \times 100\% \qquad (1)$$

where C_1 is the amount of clogged nozzle, and S is the sum of the nozzle on the print head. The cotton fabric (24×24 cm) was printed by Mimaki JV4-180.

RESULTS AND DISCUSSION

Properties of encapsulated disperse dye

Figure 1 showed that the mean particle size and the morphologies of encapsulated disperse dye were dif-

ferent from the original disperse dye, which indicated that some poly(styrene-maleic acid) was encapsulated onto disperse dye surface. These results could be further proved by the TGA curves. Figure 2 showed that only one large weight loss appeared at 220–550°C in original disperse dye, which was due to the sublimation of disperse dye. While in encapsulated disperse dye, two apparent weight loss peaks appeared, the initial weight loss appeared at 50–160°C was ascribed to the evaporation of physically adsorbed water. A subsequent weight loss appeared at 220–550°C was because of the decomposition of poly(styrene-maleic acid) and sublimation of disperse dye. These changes
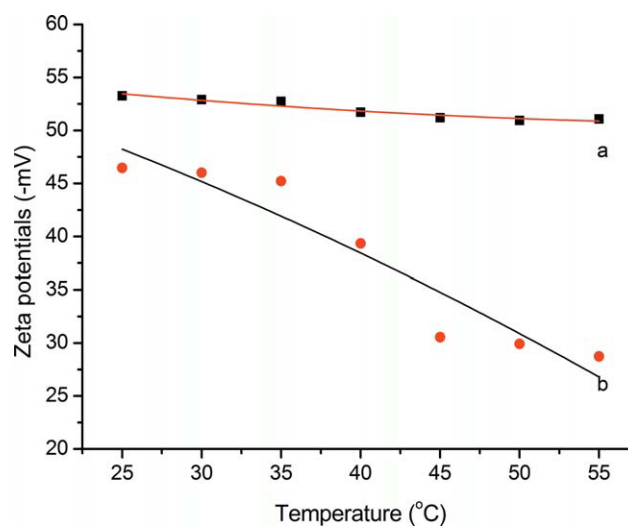

Figure 3 Zeta potentials of the disperse dye in (a) encapsulated disperse dye dispersion and (b) comparative disperse dye dispersion. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
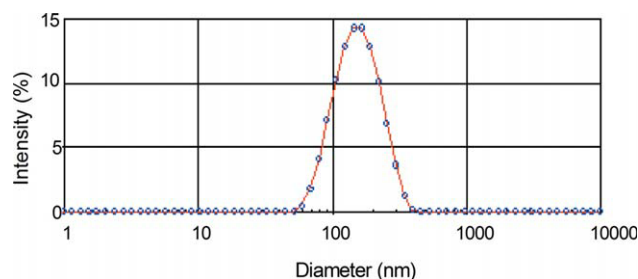
Figure 4 Particle size distribution of encapsulated disperse dye dispersion. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

indicated that the encapsulated disperse dye had a higher water content than the original disperse dye because the poly(styrene-maleic acid) contained $-COO^-$ that could impart better wettability to disperse dye.

Figure 3 illustrated that compared with the comparative disperse dye dispersion, the encapsulated disperse dye had higher zeta potentials and was relative constant to temperature. Zeta potentials could reflect the amount of dispersant that absorbed onto disperse dye surface, the lower absolute zeta potentials were, and the smaller amount of dispersant were.[19] In encapsulated disperse dye dispersion, the poly(styrene-maleic acid) was encapsulated onto disperse dye surface, desorption of poly(styrene-maleic acid) from disperse dye surface was small for its high attractive forces, thus led to a small changes of zeta potentials. But in comparative disperse dye dispersion, the poly(styrene-maleic acid) would be desorbed from the disperse dye surface for partly absorption, especially at high temperature, more amount of poly(styrene-maleic acid) were dissolved
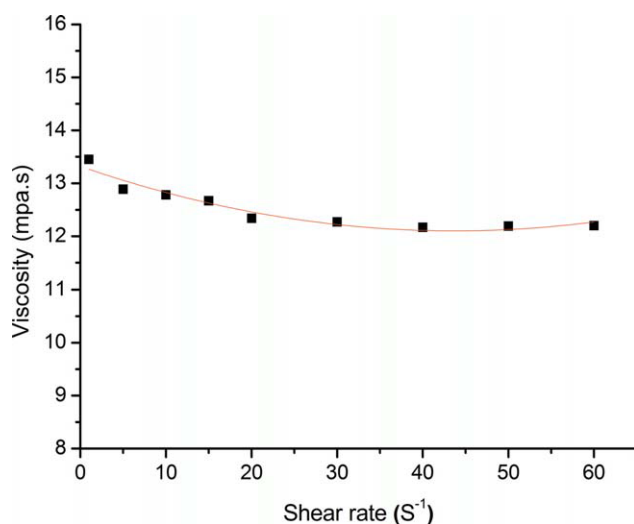

Figure 5 Effect of shear rate on viscosity of encapsulated disperse dye dispersion. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
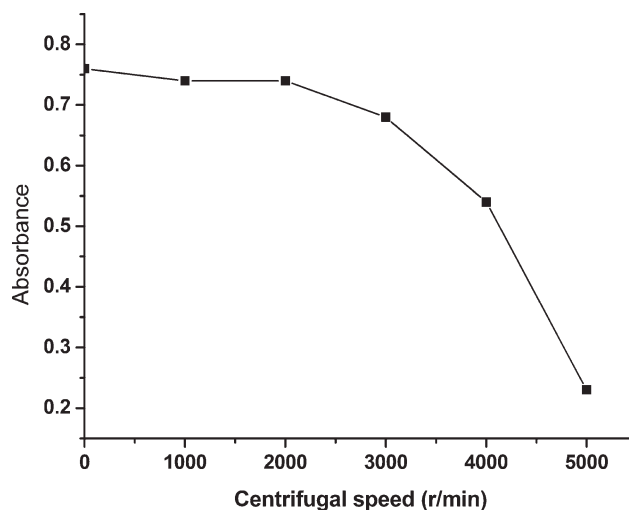

Figure 6 Effect of centrifugal stability on absorbance of encapsulated disperse dye dispersion.

into water, thus led to a large changes of zeta potentials.

Performance of encapsulated disperse dye dispersion

Figure 4 showed that the smallest particle size of encapsulated disperse dye dispersion was about 40 nm, the largest particle size was about 400 nm, and the mean particle size was about 154.7 nm, which was far smaller than the diameter of the nozzle.[20]

Figure 5 showed that encapsulated disperse dye dispersion exhibited a quite stable viscosity when the shear rate was in the range of 10–60 s^{-1}. In encapsulated disperse dye dispersion, the poly(styrene-maleic acid) were tightly encapsulated on disperse dye surface and its stretched chain was short. Therefore, there was no obvious fluctuation on viscosity as the shear rate increased.

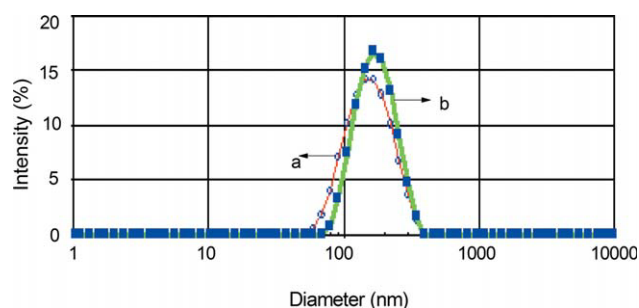Figure 6 indicated that the absorbance of the encapsulated disperse dye dispersion changed small

Figure 7 Particle size distribution of encapsulated disperse dye dispersion before and after freeze-thaw treatment; (a) before freeze-thaw treatment and (b) after freeze-thaw treatment. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
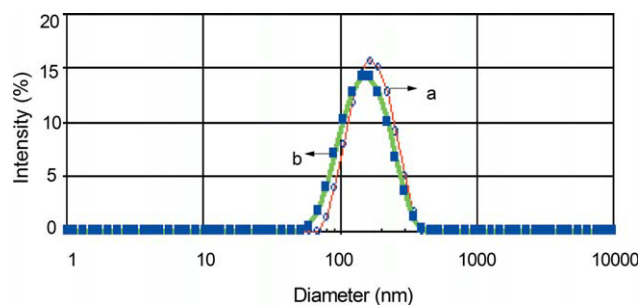
Figure 8 Effect of additives on particle size distribution of the encapsulated disperses dye dispersion; (a) encapsulated disperse dye dispersion and (b) encapsulated disperse dye dispersion after adding the additives. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

when centrifugal speed was lower than 3000 r/min. These results might be explained by stokes's equation, the higher centrifugal forces was, the faster settling velocity was.[19] In addition, at a low centrifugal speed, the settling velocity of the disperse dye might be offset by the Brownian motion, thus the absorbance changed small. When the centrifugal speed was higher enough, the settling velocity of some larger disperse dye particles might not be offset by the Brownian motion for high centrifugal forces, thus led to a greatly changes of absorbance. Figure 7 showed that the particle size distribution of the encapsulated disperse dye dispersion changed small after freeze-thaw treatment. In encapsulated disperse dye dispersion, the attractive force between poly(styrene-maleic acid) and disperse dye was large for encapsulation, and it was hard to be peeled off. From Figures 6 and 7, we may conclude that the encapsulated disperse dispersion had an excellent stability to storage.

To formulate inkjet printing ink, some additives such as polyols, surfactant, alkali, deformers, and anti-bacterial should be added into the dispersion. In this article, we also investigated the effect of the main additives (glycerol and ethylene glycol monomethyl ether) and pH value on particle size distribu-
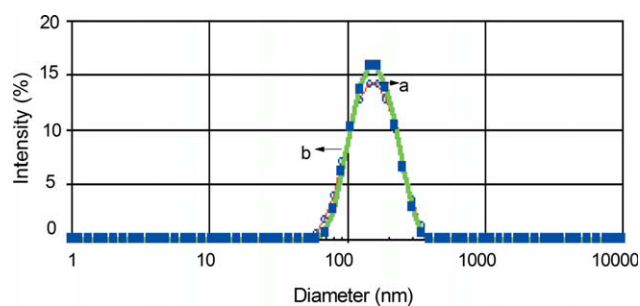

Figure 9 Effect of alkali on particle size distribution of the encapsulated disperses dye dispersion; (a) pH 8.5 and (b) pH 10. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

TABLE I
The Physical Properties of Encapsulated Disperse Dispersion and Inkjet Printing Ink

Samples	Particle size (nm)	Viscosity (mPa s)	Surface tension (m Nm^{-1})	pH value	Centrifugal stability (%)[a]
Encapsulated disperse dye dispersion	154.7	12.53	57.6	9	87.4
Inkjet printing ink	152.5	2.87	29.1	7.8	88.9
Inkjet printing ink[b]	155.3	2.84	29.3	7.6	91

[a] Centrifugal stability = $A_{30}/A_0 \times 100\%$, where A_0 was the absorbance (630 nm) of the dispersion before centrifuged, and A_{30} was the absorbance (630 nm) of the upper dispersion after centrifuged at 3000 r/min for 30 min.
[b] After freeze-thaw treatment.

tion of the encapsulated disperses dye dispersion, and the results were shown in Figures 8 and 9. It was seen that the particle size changed small after some additives and alkali were added.

Characteristics of inkjet printing ink

The physical properties of encapsulated disperse dispersion and inkjet printing ink were shown in Table I. The ink containing different kinds of dispersion and various ingredients must be carefully formulated to get the optimum properties for good quality inks.

Physical properties of inkjet printing ink, such as particle size, stability, viscosity, pH value, and surface tension, could influence on many aspects of the printing process including the choice of printing system, printing performance, and printed images. For example, surface tension and viscosity could greatly affect the wetting of nozzle surface, wetting width of
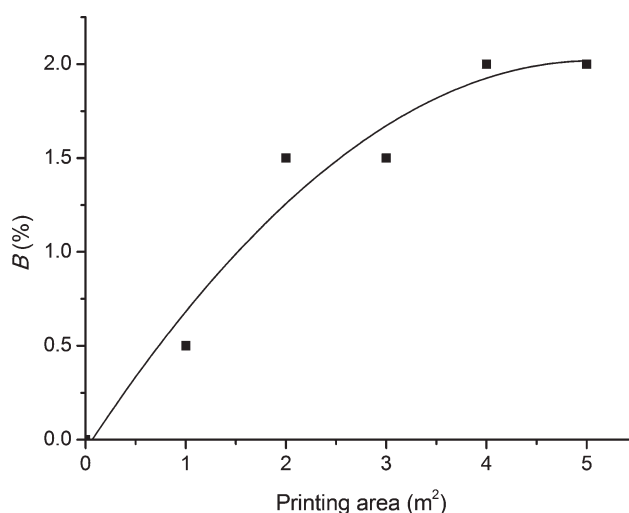

Figure 10 Effect of printing area on clogging nozzle rate.

printing substrates, penetration speed, and depth into substrates. Generally, a high surface tension and viscosity were desirable for a uniform and stable ink drop formation on the nozzle.[20] However, if the surface tension and viscosity were high enough, a poor printing performance would be gotten. Therefore, the surface tension and viscosity must adjust according to the requirement of the printing machine. Table I showed that the inkjet printing ink had an excellent stability, its viscosity, pH value, and surface tension were found to be 2.87 mPa s, 7.8, and 29.1 m Nm^{-1}, respectively. This ink was suitable for Mimaki JV4-180. Only 2% nozzles were blocked after printing 5 m^2 as shown in Figure 10.

CONCLUSIONS

The phase separation was an effective method to prepare the encapsulated C.I. disperse dye blue 60. The encapsulated disperse dye dispersion had small particle size, narrow particle size distribution, and excellent stability to additives, freeze-thaw treatment, and centrifugal forces. The properties of the ink that prepared by encapsulated disperse dye dispersion satisfied with the requirement of Mimaki JV4-180. It showed an excellent printing performance when tested at room temperature.

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