

Rapid Analysis of the Quality of the Recording Coating on Ink Jet Printing in Preparation

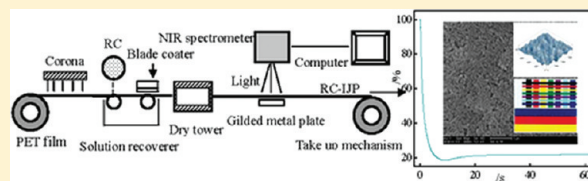
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 Supporting Information

ABSTRACT: Preparation of the recording coating on the ink jet printing (RC-IJP) is proposed. The microstructure of RC-IJP was analyzed by scanning electron microscopy and atomic force microscope. The surface infiltration process of RC-IJP was studied. Fourier transform near-infrared (FT-NIR) spectra showed the combination and overtone vibrations information of the hydrogen-containing groups in RC-IJP structure. NIR spectra combined with partial least-squares methods were proposed for the noncontact analysis of the surface infiltration time and weight per unit area in RC-IJP. Forty-five samples were selected for the calibration sets of the surface infiltration time and weight per unit area, respectively. Spectral pretreatment method was used to develop robust calibration models. After the spectral pretreatment was optimized, the determination coefficient (R^2) of the surface infiltration time and weight per unit area were 0.90 and 0.97, respectively. The analytical results showed that NIR spectra had significant potential for the analysis of the surface infiltration time and weight per unit area of RC-IJP.



1. INTRODUCTION

Ink-jet printing has been applied in organic,^{1–3} electronics,^{4–7} nanotechnology,^{8–10} and tissue engineering, etc. The recording coating on the ink-jet printing (RC-IJP) affects the printing quality. The surface infiltration time (water infiltrates onto the recording material surface) and the weight per unit area in the recording material are the important factors on evaluation of the RC-IJP quality. The surface infiltration time of RC-IJP reflects the dispersibility of the recording coating. The dispersibility of the recording coating is more uniform in terms of the short surface infiltration time. On the other hand, the hydroscopicity of the recording coating is poorer in terms of the long infiltration time. Excessive weight per unit area causes the split of the recording material, while the less weight per unit area influences the definition and permanent property of the printing image. In order to enhance the printing quality, the analysis of the quality indexes of RC-IJP is very important in preparation.

As a matter of fact, traditional method analyzes the surface infiltration time using a liquid osmosis analysis instrument, and the weight per unit area is used for the weight method by analytical balance. However, such a technique is actually destructive, usually time consuming, and produces waste. In addition, these methods are not able to measure two quality indexes of RC-IJP simultaneously. So far, study on the simultaneous analysis of two quality indexes of RC-IJP has never been reported.

Near-infrared (NIR) spectroscopy is an excellent alternative analytical method. It is a very rapid, accurate and nondestructive method for simultaneous measurements on different constituents in various products. Nowadays, NIR spectroscopy is

applied in a variety of fields including pharmaceuticals,^{11,12} food industry,^{13–15} and polymer industry for quality assurance.¹⁶ The aim of this study is to develop an analysis method for the simultaneous prediction of the quality indexes of RC-IJP using diffuse reflection NIR spectroscopy. Calibration models of the weight per unit area and the surface infiltration time are developed by partial least squares (PLS), which are used to analyze unknown samples. Furthermore, in this study we attempted to prepare the RC-IJP.

2. EXPERIMENTAL SECTION

2.1. Preparation of RC-IJP. The inorganic pigments-nano-scale silica with the average nanoparticle size of 12 nm and specific surface area of 170–230 m²/g was obtained from Degussa A200, Germany. In addition, the binder poly(vinyl alcohol) (PVA-1788), the substrate poly(ethylene terephthalate) (PET) and accessory ingredients were provided by Shanghai Chemical Co., Ltd., China.

First, 0.07 g of silicone dispersing agent (γ -aminopropyltriethoxysilane) is added into 20 g of distilled water with magnetic stirring for 10 min. Nitric acid is subsequently added until solution pH = 4. Nanoscale silica (3.5 g) is added in the above solution under a homogenizer (Fluko Equipment Shanghai Co., Ltd.) for 30 min.

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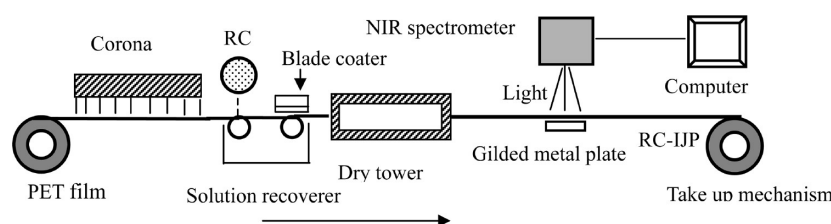


Figure 1. Schematic diagram of the NIR monitoring of the manufacturing process of RC-IJP.

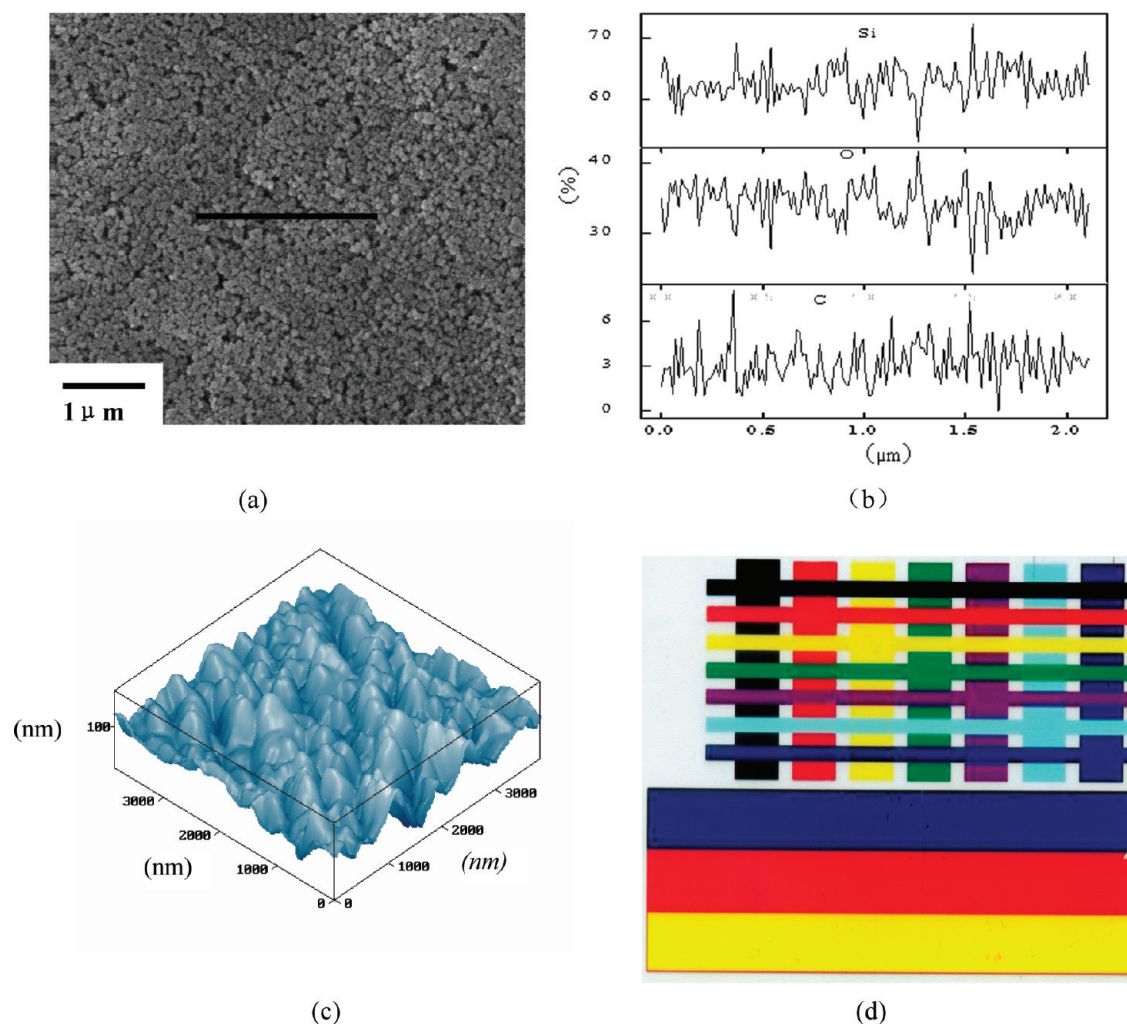


Figure 2. RC-IJP: (a) SEM, (b) distribution of the main element composition, (c) AFM, and (d) printing image imported by scanning instrument.

On the other hand, 1 g of PVA is dissolved in distilled water for 30 min, and then it is placed in a water bath at 90 °C for 40 min to obtain the PVA solution. The dispersive nanoscale silica solution is added in PVA solution by the homogenizer for 10 min, and then the mixed solution is placed in an oven with the temperature of 40 °C for 120 min. Consequently, by using the mentioned method, the recording coating is prepared.

The blade coater method used in the preparation of RC-IJP is schematically shown in Figure 1. In order to enhance the property of polarity on the PET film, the surface is disposed by corona, and then the recording coating is coated on PET film. The excessive solution is removed by the blade coater. In the next step of the process, in order to evaporate the solvent, the PET

film with recording coating is moved through a dry tower. Finally, RC-IJP is wrapped to the take up mechanism.

2.2. Instrument Analysis. The surface morphologies of RC-IJP are observed using a high-resolution scanning electron microscopy (SEM, Quanta 200F, USA). Samples are sputtered with gold in the aluminum piece. Subsequently, these sputtered samples are placed on the aluminum piece for SEM observation. The surface roughness of RC-IJP is examined by atomic force microscope (AFM, Solver-P47H, NT-MDT, Russia). NIR spectra are collected by a diffuse reflection FT-NIR spectrometer, (MATRIX, Bruker, Germany) equipped with an indium gallium arsenide (InGaAs) detector and a CaF₂ beamsplitter (details in the Supporting Information).

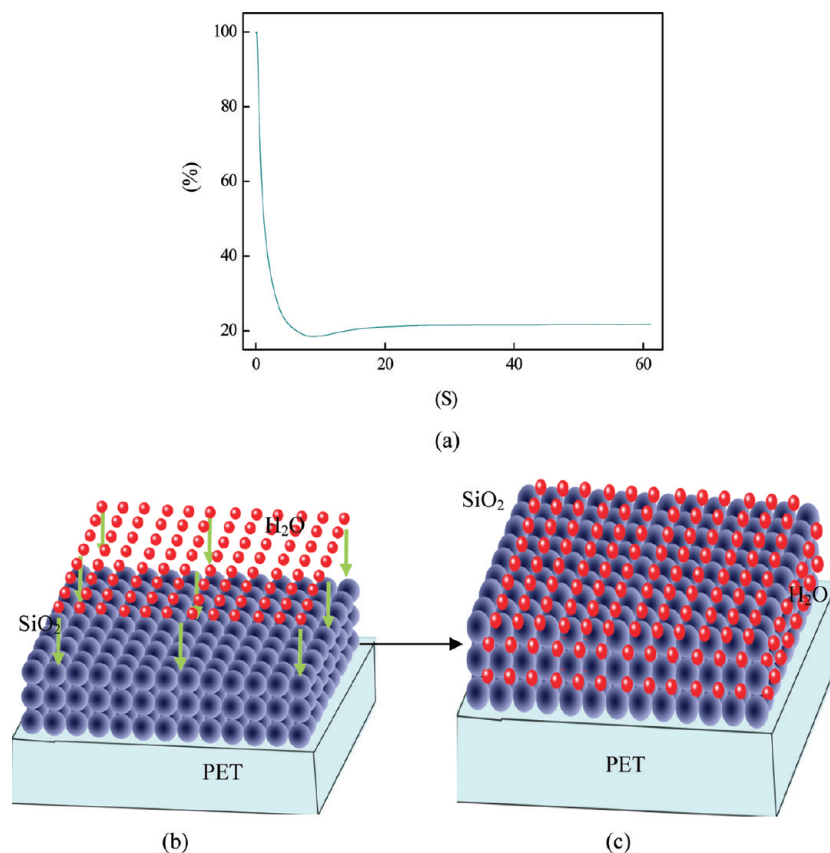


Figure 3. (a) Infiltration analysis of RC-IJP. (b) Schematic of the infiltration process: water infiltrates the surface RC-IJP and (c) water fully infiltrates the RC-IJP.

2.3. Reference Analysis. Forty-five samples of RC-IJP (7.5×4.5 cm) are used according to the various weight and physical aspects in the preparation of RC-IJP. Reference methods are utilized to determine the quality indexes of RC-IJP. The surface infiltration time which is analyzed by liquid infiltration instrument (Emtec EST12 Co., Ltd., Germany) is the time at which water infiltrates on the recording coating surface under ultrasound (details in the Supporting Information). Weight per unit area is analyzed by the dissolution method. The test sample of initial weight W_1 (precision 0.001 g) is placed in water until recording coating is dissolved completely and the weight is marked W_2 using the following equation:

$$\text{Gr} = (W_1 - W_2)/S \quad (1)$$

where Gr is the weight per unit area and S the area of RC-IJP (m^2).

3. RESULTS AND DISCUSSION

3.1. Structural Analysis RC-IJP. The microstructure of RC-IJP is analyzed by SEM. It can be observed from Figure 2a that the dispersibility of the recording coating is uniform. The excellent dispersibility is beneficial to infiltration of the ink. The distribution of the main element composition (C, O, and Si) on the recording coating surface is shown by energy-dispersive spectrum (EDS) in Figure 2b. The contents of C, O, and Si do not change obviously from EDS. The analytical result implies that dispersibility of each composition is uniform in the

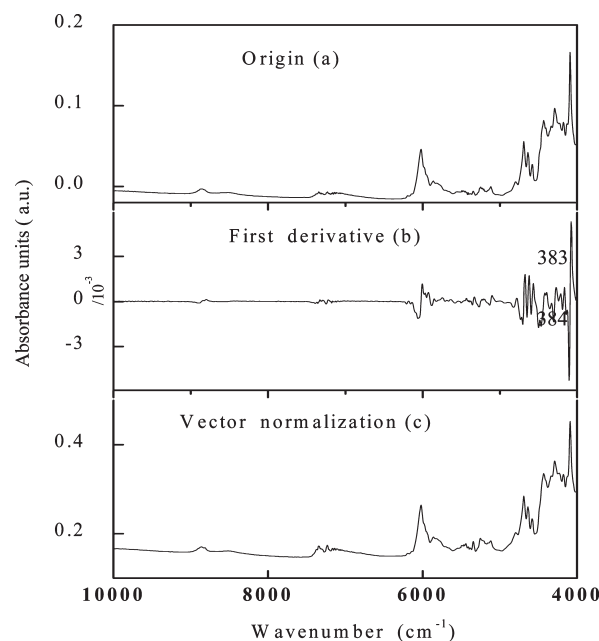


Figure 4. NIR diffuse reflectance spectral of the several pretreatments: (a) origin, (b) vector normalization, and (c) first derivative.

recording coating. AFM images of recording coating surface are provided in Figure 2c. From Figure 2c, it can also be observed that the recording coating surface has roughness, which is

Table 1. Statistical Results of the Developing Models

quality indexes	range	preprocessing routines	spectral range (cm^{-1})	PLS factors	R^2	RMSEC
surface impregnation time	0.049–1.05 (S)	vector normalization	4000–6100 7100–9000	8	0.90	0.151
weight per unit area	0.73–1.76 (g/m^2)	first derivative	4000–8000	5	0.98	0.062

beneficial to sorption of the ink. The dispersibility of the roughness is uniform from Figure 2c; this recording coating avoids the ink diffusion. Moreover, as it can be seen in Figure 2d that the color of the printing image is clear by RC-IJP.

3.2. Infiltration Analysis of RC-IJP. Figure 3a shows the infiltration time of recording coating surface. In Figure 3a, x and y axes show the infiltration time of the sample and the energy of ultrasound, respectively. Figure 3a shows that water infiltrates on recording coating surface at 0.12 s (S) and at this moment, RC-IJP is receiving the full energy (100%) under ultrasound. If weight per unit area is invariant, the dispersibility of the recording coating is more uniform in terms of the short surface infiltration time. As shown in Figure 3a, while the RC-IJP is receiving the lowest energy by ultrasound at 18.48%, the infiltration time of water is 8.9 s, and then the energy rises mildly to be stabilized. The process mentioned above is schematically shown in Figure 3b,c. Figure 3b shows that water infiltrates on the surface of RC-IJP, while Figure 3c shows that water infiltrates fully on RC-IJP by ultrasound. The hydroscopicity of the recording coating is poorer in terms of the long infiltration time; this recording coating induces the poor image of the inkjet printing for water-based ink.

3.3. NIR Spectrum of RC-IJP. NIR spectrum of RC-IJP is shown in Figure 4a. As shown in Figure 4a, the methyl groups (CH_3-) give combination bands at 4080 and 4295 cm^{-1} . In addition, ethenyl ($\text{CH}_2=\text{CH}-$) demonstrates two combination bands at 4425 and 4694 cm^{-1} , and an overtone band at 6034 cm^{-1} . On the other hand, OH gives stretching combination and overtone bands at 5250 and 7260 cm^{-1} , respectively. The analysis described above can represent adequately the required information of the RC-IJP structure in NIR spectra.

In order to eliminate variations in offset and overlapped peaks of NIR spectra, to ensure a good correlation between the spectral data and the concentration values, first-derivative and vector normalization spectral pretreatments are applied and presented in Figure 4b,c.

3.4. Developing Models. The samples included in the calibration set should encompass the maximum possible variability and span the whole range of values of the property to be measured. Table 1 shows the information about reference data for the surface infiltration time and weight per unit area. The ranges of two quality indexed RC-IJP are summarized in Table 1. Forty-five samples are developed in the calibration model of the surface infiltration time. The maximum and minimum values are 1.05 and 0.049 s, respectively. Calibration models are developed by PLS, and full cross validation is applied to optimize the calibration model. It is worth mentioning that in the various calibration models based on diverse spectral pretreatments (in the collection of near-infrared (NIR) spectra, there could be the baseline drift, noise, scattering effects, and overlapped peaks; these errors can disturb the robustness and reliability of multivariate calibration models; the spectral pretreatment methods are used in developing the calibration model), a number of PLS factors and spanning variable wavenumber ranges are tested. It is observed from Table 1 that the determination coefficient (R^2) and root-mean-square error of calibration (RMSEC) are 0.90 and 0.151 by first derivative (first derivative is applied in

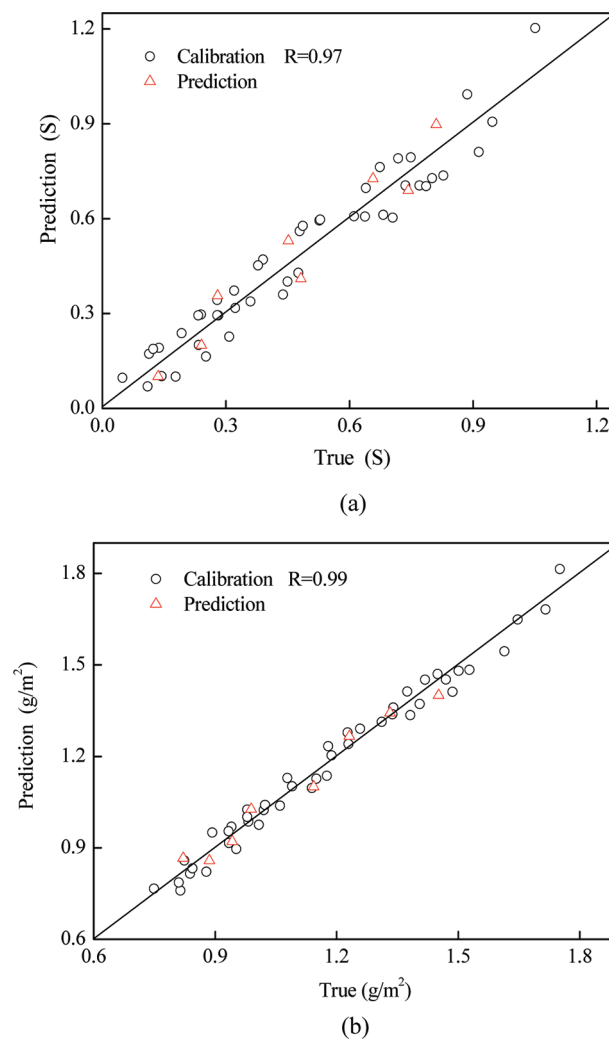


Figure 5. Correlation between predicted values and actual values: (a) weight per unit area and (b) surface infiltration time.

baseline correction and overlapping peaks), respectively. The spectral ranges give the best wavenumber 4000–6100 and 7100–9000 cm^{-1} , 8 PLS factors are used. (The number of PLS factors is the available information in the calibration model. If the chosen number of PLS factors is lacking, the information of the spectrum is lost. If the chosen number of PLS factors is excessive, other information is utilized such as measuring noise. The excessive number of PLS could cause the increase of prediction error of the calibration model.) The parameters of the developing models of the surface infiltration time and weight per unit area are shown in Table 1.

The plots of the actual values versus the predicted values by NIR for two calibration sets of the surface infiltration time and weight per unit area are shown in Figure 5a,b. The mentioned plots have regression coefficients of 0.97 and 0.99, respectively. Eight prediction samples were used for two quality indexes; the

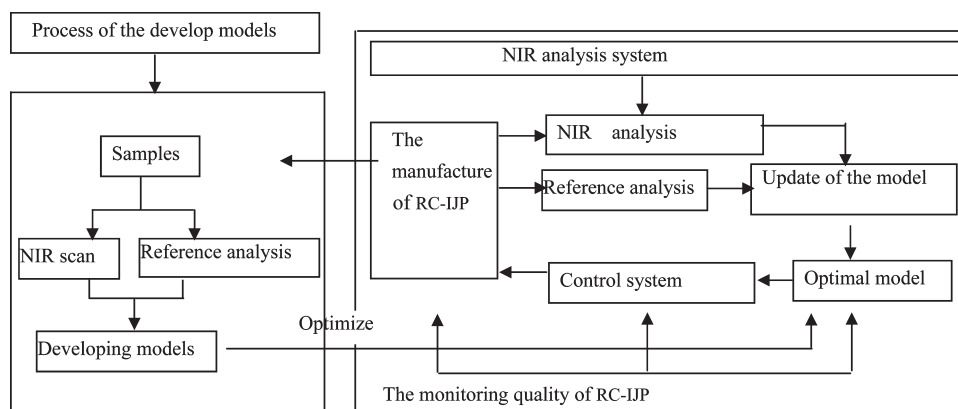


Figure 6. Sketch of the monitoring quality of RC-IJP.

plot of the actual values against NIR predicted values for the prediction sets is shown in Figure 5a,b also. From the above analysis, the NIR method is a good alternative technique for analyzing the surface infiltration time and weight per unit area in the preparation of RC-IJP.

If the processing parameters and conditions are changed, the model would be updated. For this purpose, the samples are selected from the production line and are added to the model. A sketch of the monitoring quality of RC-IJP is shown in Figure 6.

The standard sample is 7.5 cm × 4.5 cm which is analyzed by the reference method; however, the length–breadth of a roll of RC-IJP is 10 000 cm × 100 cm. If the quality indexes of the standard sample RC-IJP overflowed the prescriptive ranges, RC-IJP is a defective product and the recording coating should be prepared again which results in big extra costs. If the sample is analyzed by NIR noncontact method, the standard sample which has not been measured up would be removed from RC-IJP and the remainder could also be used. The above result indicates that the NIR method can also reduce the cost. It is worth mentioning that, in this study, the spectra of RC-IJP were collected once within less than 1 min without any sample destruction.

4. CONCLUSION

In this study, the surface infiltration time and weight per unit area of RC-IJP are analyzed. It was shown that, when the weight per unit area is invariant, the dispersibility of the recording coating is more uniform in terms of the short surface infiltration time. The surface infiltration time and weight per unit area are successfully performed based on NIR spectra combined with PLS methods. The results of this study show that NIR spectra are a reasonably accurate method. Furthermore, the results confirm that NIR spectra, in combination with the multivariate calibration method, are promising techniques to assess the quality of RC-IJP in the preparation.

■ ASSOCIATED CONTENT

Supporting Information. NIR spectroscopy and data analysis and liquid infiltration. This material is available free of charge via the Internet at <http://pubs.acs.org>.

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■ REFERENCES

- (1) de Gans, B. J.; Duineveld, P. C.; Schubert, U. S. *Adv. Mater.* **2004**, *16*, 203–213.
- (2) de Gans, B. J.; Xue, L.; Agarwal, U. S.; Schubert, U. S. *Macromol. Rapid Commun.* **2005**, *26*, 310–314.
- (3) Tekin, E.; Holder, K.; Kozodaev, E. D.; Schubert, U. S. *Adv. Funct. Mater.* **2007**, *17*, 23–28.
- (4) Reese, C.; Roberts, M.; Ling, M. M.; Bao, Z. *Mater. Today* **2004**, *7*, 20–27.
- (5) Sele, C. W.; Werne, T. V.; Friend, R. H.; Sirringhaus, H. *Adv. Mater.* **2005**, *17*, 997–1001.
- (6) Yoshioka, Y.; Jabbour, G. E. *Adv. Mater.* **2006**, *18*, 1307–1312.
- (7) Sirringhaus, H.; Kawase, T.; Friend, R. H.; Shimoda, T.; Inbasekaran, M.; Wu, W.; Woo, E. P. *Science* **2000**, *290*, 2123–2126.
- (8) Lee, K. J.; Jun, B. H.; Kim, T. H.; Joung, J. *Nanotechnology* **2006**, *17*, 2424–2428.
- (9) Chung, J.; Bieri, N. R.; Grigoropoulos, C. P.; Poulikakos, D. *Appl. Phys. A: Mater. Sci. Process.* **2004**, *79*, 1259–1261.
- (10) Chung, J.; Ko, S.; Bieri, N. R.; Grigoropoulos, C. P.; Poulikakos, D. *Appl. Phys. Lett.* **2004**, *84*, 801–803.
- (11) Sarraguca, M. C.; Lopes, J. A. *Anal. Chim. Acta* **2009**, *642*, 179–185.
- (12) Luypaert, J.; Massart, D. L.; Heyden, Y. V. *Talanta* **2007**, *72*, 865–883.
- (13) Pinto, L. A.; Galvao, R. K. H.; Araujo, M. C. U. *Anal. Chim. Acta* **2010**, *682*, 37–47.
- (14) Ferrer-Gallego, R.; Hernandez-Hierro, J. M.; Rivas-Gonzalo, J. C.; Escribano-Bailon, M. T. *Talanta* **2010**, *82*, 1778–1783.
- (15) Vigni, M. L.; Durante, C.; Foca, G.; Marchetti, A.; Ulrici, A.; Cocchi, M. *Anal. Chim. Acta* **2009**, *642*, 69–76.
- (16) Blanco, M.; Villaescusa, V. *Talanta* **2007**, *71*, 1333–1338.