Effect of using ink containing polyacrylate and silicone surfactant on the inkjet printing of quantum dot films

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ABSTRACT
For device printing, it is important for uniform films to be printed because the morphology of the film affects the performance of the device in inkjet printing. In this study, to prepare stable and well-dispersed ink, polyacrylate and silicone surfactant BD-3033H were added to ink. Then a uniform quantum dot (QD) film was obtained by optimizing the polyacrylate and silicone surfactant contents of the ink as well as the substrate temperature. The change of the polyacrylate structure from a curly chain structure to a three-dimensional network structure blocked the outward flow of QDs. Silicone surfactant BD-3033H caused Marangoni flow and made the QDs flow inward. When the substrate temperature was increased, the solvent evaporation rate was accelerated, the contact line was pinned, and the outward flow was enhanced, which changed the film structure from convex to flat. Finally, when the polyacrylate content was 12 wt%, the silicone surfactant BD-3033H content was 0.10 wt%, the substrate temperature was 40°C, and the coffee ring effect was eliminated. A uniform QD film was printed, providing a technical guarantee for the fabrication of QD devices in the future.

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1. Introduction
Quantum dots (QDs) are low-dimensional materials with excellent optical properties, including high color purity, stability, and tunable emission wavelength [1–4]. As an important class of photovoltaic materials [5], QDs can be fabricated into thin films. Inkjet printing is a direct patterned deposition process for thin films [6]. It has many advantages, including the facts that it does not come in contact with the substrate, the deposition volume and material compatibility can be precisely controlled [7], and material saving [8,9] and flexible substrate selection are possible [10].

In inkjet printing, the droplets sprayed on the substrate often show a ring structure after drying, which is called ‘coffee ring’ effect. In 1997, Deegan [11] explained the formation mechanism of the coffee ring. When a droplet is sprayed on the substrate, it spreads on the substrate, then the droplet is dried to form a film. During the process of film formation, the solvent evaporation rate is faster at the edge than in the center. Thus, the droplet has a pinned three-phase contact line (TCL). As the solvent at the edge evaporates quickly, the solvent in the center is added to the edge. Capillary flow then occurs. The outward capillary flow transports the solutes from the center to the edge, forming a coffee ring. One way of eliminating the coffee ring is using a dual-solvent ink system or surfactant to create an inward Marangoni flow and to achieve a uniform film. The inward Marangoni flow transports the solutes from the edge to the center. It produces a flat film profile. Jiang et al. [12] printed coffee-ring-free arrays on a modified substrate through a mixed-solvent QD ink system of cyclohexane and o-dichlorobenzene. In addition, red QLED has a 9700 cd/m² maximum brightness. Sander Kommeren et al. [13] successfully printed a uniform PEDOT:PSS layer on a hydrophobic P3HT/PCBM photoactive layer by adding FS3100 to the printed ink.

Another way of eliminating the coffee ring is to weaken the outward capillary flow by increasing the viscosity of the solution. This weakens the flow of the solute from the center to the edge, and also forms a flat film profile. Wang et al. [14] used a single solvent to print uniform polymer films by adjusting the viscosity of the solution. Yu et al. [15] printed polymer films without a coffee ring by adding high-viscosity cyclohexylbenzene to ink. In addition, Augustine et al. [16] controlled the slip of the TCL through electric wetting, thus eliminating the coffee ring effect. Tao et al. [17] first verified the...
critical role of solvent evaporation on the resolution of inkjet printing and a uniform profile was obtained by drying the microenvironment to adjust the air pressure. All the above methods, however, use only the dual-solvent ink system, surfactants, or viscosity regulators. These methods produce only one-way flow regulation. Furthermore, their use makes the experiment more difficult due to one-way flow regulation. In addition, when using only a dual-solvent ink system, the ratio of the two solvents has to be optimized carefully, and many combinations of the dual solvent need to be tested, which increases the cost of the experiment and makes the experiment more complex.

Using a novel ink system [18] can be another promising way. In this study, based on two-way flow regulation, a new ink system containing polyacrylate and silicone surfactant was obtained. To prepare a stable and well-dispersed ink, polyacrylate and silicone surfactant BD-3033H were added to ink. A uniform QD film was printed by optimizing the polyacrylate and silicone surfactant contents of the ink, as well as the substrate temperature. The polyacrylate and silicone surfactant BD-3033H contents as well as the substrate temperature were systematically studied, and it was revealed that increasing the polyacrylate content can hinder the outward flow of QDs. A silicone surfactant causing Marangoni flow makes QDs flow inward. In addition, increasing the substrate temperature accelerates the solvent evaporation rate, then pins contact line and strengthens the outward flow.

2. Experiment section

2.1. Materials

Red-QD CdSe@ZnS liquid was purchased from Tianjin Xingguang Yinghua Co., Ltd. Toluene was purchased from Sinopharm Chemical Reagent Co., Ltd. O-dichlorobenzene (oDCB) was obtained from Shanghai Macklin Biochemical Co., Ltd. A silicone surfactant (BD-3033H) was supplied by Hangzhou Bald Advanced Materials Co., Ltd. Polyacrylate was purchased from Guangdong Poly Optoelectronics Co., Ltd. Finally, PC substrates were obtained from Dongguan Qiyi Plastic Material Co., Ltd.

2.2. Preparation of the inks

Red QDs were dissolved in a mixed solvent of toluene and oDCB at 10 mg/ml to make QD inks. The volume ratio of toluene to oDCB was 8:2. Polyacrylate had higher viscosity and was dissolved in toluene. The mass was rotated at a 400 rpm speed, and was stirred at 30°C for 10 minutes to ensure that the polyacrylate would be fully dissolved. Then BD-3033H was added to the ink, followed by polyacrylate. The final ink was ultrasonically oscillated for 10 minutes and was then made to stand for 72 hours. The QD inks were used for inkjet printing with a MicroFab JETLAB II piezoelectric printer equipped with a 30-μm-diameter nozzle.

2.3. Measurements

The inks’ viscosities were measured at room temperature using a viscometer (Brookfield, DV2T, USA). The surface tension measurements were carried out at room temperature using an automatic surface tension meter (Hengping, BZY-1, Shanghai). The two-dimensional (2D) morphologies of the QD films were observed with an optical microscope (Olympus, BX51M, Japan), and the three-dimensional (3D) morphologies were investigated with a 3D microscope (Olympus OLS4100, Japan). The thicknesses of the QD films were measured using a step profiler (Bruker, Dektak-XT, Germany).

3. Results and discussion

3.1. Stability of the inks

First and foremost, inkjet printing ink should have good stability. In this study, ink without polyacrylate and silicone surfactant (ink 1) and ink with polyacrylate and silicone surfactant (ink 2) were prepared. Figure 1 shows the states of the two kinds of ink after placing for different days. On the third day, there was solute precipitation at the bottom of the ink 1 bottle, but no precipitation was found at the bottom of the ink 2 bottle, and the solution was clear. Figure 2 shows the action process of the two kinds of ink. For ink 2, the addition of surfactant reduces the surface tension of the solution and made the surface energy of the solvent match the surface energy of the solute [19]. The chain structure of polyacrylate enlarges the distance between the QDs in a solution, which can prevent the QDs from agglomerating, and can improve the dispersion and stability of the ink.

3.2. Effects of the printing parameters

Satellite dots often appear in the process of inkjet printing, which greatly affects the quality of printing and the performance of devices. By optimizing the printing parameters, the satellite droplets can be eliminated, which is helpful for the subsequent experiments. Figure 3 shows the inkjet printing pulse waveform. Within a certain range, with the increase of the dwell voltage, the electrostatic pressure at the nozzle and the volume and...
Figure 1. States of two kinds of ink after placing for different days: (a) ink 1 without polyacrylate and silicone surfactant; and (b) ink 2 with polyacrylate and silicone surfactant.

Figure 2. Action process of two kinds of ink: (a) ink without polyacrylate and silicone surfactant; and (b) ink with polyacrylate and silicone surfactant.

Figure 3. The diameter of droplets also increase. As the dwell voltage becomes bigger, the voltage difference between the two ends of the droplet also becomes bigger, and the speed increases. As shown in Figure 4, with the increase of the idle voltage, the satellite droplets gradually converged with the main droplets, forming a single droplet when the idle voltage was 4 and 6 V. With the increase of the idle voltage, the voltage difference between the two ends of the droplet decreased, which resulted in falling droplet velocity, thus avoiding high voltage and causing a residual wave with a sufficient amplitude to cause the satellite droplet to be ejected. As shown in Figure 5, when the dwell voltage was 46 and 45 V, the satellite droplets were gradually eliminated. Another way of eliminating the satellite droplets is to adjust the backpressure. As shown in Figure 6, when the backpressure was increased, the main droplet eventually coincided with the satellite droplet when the backpressure was $-18$ and $-20$ V.

3.3. Polyacrylate content

After the optimization of the printing parameters, the film was printed. It is known that polyacrylate, as a kind of high-molecular compound, has high viscoelasticity and can inhibit the movement of QDs. $D$ is defined as the diameter of the printed film. To express the coffee ring effect degree, the coffee ring factor, $H$, was introduced, as follows [20]:

$$H = \frac{H_{\text{max}}}{H_{\text{min}}}$$  

where $H_{\text{max}}$ is the maximum thickness of the film, and $H_{\text{min}}$ is the minimum thickness of the film. When $H$ is equal to 1, the uniformity of the film is the best, and the coffee ring effect is completely overcome. The uniformity of the film is worse with the increase of $H$.

When the substrate temperature was $35^\circ$C, the silicone surfactant BD-3033H content was 0.05 wt%, adding 4, 8, 12, 16, and 20 wt% polyacrylate in the ink, respectively. Table 1 summarizes the properties of the inks.

The morphology characteristics of printed QD films with different polyacrylate contents are shown in Figure 7. The $H$ and $D$ values with different polyacrylate contents are shown in Table 2. It can be seen that the $H$ value decreases with the increase of polyacrylate content. The size of the driving force of the outward capillary flow can
Figure 3. (a) Inkjet printing pulse wave. (b) Curve of the volume value and the dwell voltage. (c) Curve of the diameter value and the dwell voltage. (d) Curve of the velocity value and the dwell voltage.

Figure 4. [(a–d)] Droplets with 0, 2, 4, and 6 V idle voltages.

Figure 5. [(a–d)] Droplets with 48, 47, 46, and 45 V dwell voltages.

Figure 6. [(a–d)] Droplets with −14, −16, −18, and −20 V backpressure.
Table 1. Related characteristics of inks with different polyacrylate contents.

| Polyacrylate content (wt%) | Viscosity (cP) | Surface tension (mN/m) |
|----------------------------|----------------|------------------------|
| 4                          | 0.96           | 27.3                   |
| 8                          | 1.41           | 27.3                   |
| 12                         | 1.89           | 26.9                   |
| 16                         | 2.60           | 27.2                   |
| 20                         | 3.01           | 27.1                   |

be shown as equation [21].

\[
Q_{ca} \sim \frac{\gamma H^4}{\mu r^3} \left(1 + \frac{\Delta \gamma}{\gamma}\right),
\]

(2)

where \(Q_{ca}\) is the driving force of the outward capillary flow, \(\gamma\) is the surface tension of the solution, \(H\) is the height of the droplet, \(r\) is the radius of the droplet, \(\mu\) is the viscosity of the solution, and \(\Delta \gamma\) is the difference in surface tension between the edge and center of the droplet. Therefore, with the increase of polyacrylate content, the solution viscosity gradually increases, resulting in a weaker outward capillary flow, which weakens the flow of the solute from the center to the edge. Eventually, the uniformity of the film gradually improves, as shown in Figure 7(f), (g), and (h). An extremely high viscosity will not improve the morphology of the films, as shown in Figure 7(i) and (j).

3.4. Silicone surfactant content

For controlling the outward flow of QDs, the uniformity of the film is greatly improved when the polyacrylate content is 12%, but the film thickness chart shows that the coffee ring structure is not eliminated at this time, so it was decided in this study that the inward flow was increased. The method that was used was to add surfactant in the ink. When surfactant was added, with the evaporation of the solution, the concentration of the surfactants increased locally, which caused strong gradients in the surface tension, and enhanced the Marangoni flow. The silicone surfactant BD-3033H was used in the experiment. When the substrate temperature was 35°C, the polyacrylate content was 12 wt%, adding 0.05, 0.10, and 0.15 wt% silicone surfactant BD-3033H in the ink, respectively. Table 3 summarizes the properties of the inks. It can be seen that the surface tension of the solution decreased with the increase of silicone surfactant BD-3033H content, but the decrease range was very small. This may be because [22] the surface tension of the solution itself was very low and the range of surface tension drop was limited.

The morphology characteristics of printed QD films with different silicone surfactant contents are shown in Figure 8. The \(H\) and \(D\) values with different silicone surfactant contents are shown in Table 4. From Table 4, it can be seen that the \(H\) value was relatively small when the

Table 2. H and D values with different silicone surfactant contents.

| Silicone surfactant content (wt%) | Viscosity (cP) | Surface tension (mN/m) |
|----------------------------------|----------------|------------------------|
| 0.05                             | 1.82           | 27.3                   |
| 0.10                             | 1.83           | 26.7                   |
| 0.15                             | 1.88           | 26.1                   |

Table 3. Related characteristics of inks with different silicone surfactant contents.

Figure 7. Printed 2D topographies and film thicknesses with different polyacrylate contents: [(a), (b), (c), (d), (e)] printed 2D topographies at 4, 8, 12, 16, and 20 wt% polyacrylate contents; and [(f), (g), (h), (i), (j)] printed film thicknesses at 4, 8, 12, 16, and 20 wt% polyacrylate contents.
Figure 8. Printed 2D topographies and film thicknesses with different surfactant contents: [(a), (b), (c)] printed 2D topographies at 0.05, 0.10, and 0.15 wt% surfactant contents; and [(d), (e), (f)] printed film thicknesses at 0.05, 0.10, and 0.15 wt% surfactant contents.

Table 4. $H$ and $D$ values with different silicone surfactant contents.

| Silicone surfactant content (wt%) | $H_{\text{max}}$ (nm) | $H_{\text{min}}$ (nm) | $H$ | $D$ (μm) |
|----------------------------------|-----------------------|-----------------------|-----|---------|
| 0.05                             | 352.41                | 305.69                | 1.15| 100.08  |
| 0.10                             | 453.3                 | 374.86                | 1.21| 102.33  |
| 0.15                             | 322.11                | 287.35                | 1.12| 96.84   |

silicone surfactant content was 0.10 and 0.15 wt%. Further observation showed that when the surfactant was 0.10 wt%, the film no longer had a coffee ring structure but a convex structure, as shown in Figure 8(e). This was because when surfactant was added, the concentration of the silicone surfactant at the edge became higher than in the center, resulting in an inward Marangoni flow, which transported the too many solutes from the edge to the center. The size of the Marangoni flow can be obtained using equation [21].

$$|\nabla \gamma| = \left| \frac{\partial \gamma}{\partial c_{\text{sur}}} \right| |\nabla c_{\text{sur}}|,$$  

where $|\nabla \gamma|$ is the Marangoni force, and $|\nabla c_{\text{sur}}|$ is the local gradient of the surfactant concentration. For the same surfactant, $\left| \frac{\partial \gamma}{\partial c_{\text{sur}}} \right|$ is a fixed value. Therefore, the magnitude of the Marangoni force depends on the local concentration gradient of the surfactant [23]. With the increase of the surfactant concentration, the inward Marangoni flow increases. The inward Marangoni flow then transports the solutes from the edge to the center, thus improving the film’s uniformity. As shown in

Figure 9. Printed 2D topographies and film thicknesses with different surfactant contents: [(a), (b), (c), (d)] printed 2D topographies at 0.11, 0.12, 0.13, and 0.14 wt% surfactant contents; and [(e), (f), (g), (h)] printed film thicknesses at 0.11, 0.12, 0.13, and 0.14 wt% surfactant contents.
Table 5. $H$ value with 0.11, 0.12, 0.13, and 0.14 wt% silicone surfactant.

| Silicone surfactant content (wt%) | $H_{\text{max}}$ (nm) | $H_{\text{min}}$ (nm) | $H$  |
|----------------------------------|------------------------|------------------------|------|
| 0.11                             | 271.63                 | 219.68                 | 1.23 |
| 0.12                             | 312.57                 | 265.48                 | 1.18 |
| 0.13                             | 235.91                 | 164.95                 | 1.43 |
| 0.14                             | 371.64                 | 308.81                 | 1.20 |

Table 6. $H$ and $D$ values at different substrate temperatures.

| Substrate temperature (°C) | $H_{\text{max}}$ (nm) | $H_{\text{min}}$ (nm) | $H$  | $D$ (μm) |
|----------------------------|------------------------|------------------------|------|----------|
| 35                         | 453.30                 | 352.41                 | 1.21 | 102.33   |
| 40                         | 497.79                 | 474.27                 | 1.05 | 93.34    |

Figure 10. Drying process of ink containing polyacrylate and surfactant.

Figure 11. Printed 2D topographies and film thicknesses at different substrate temperatures: [(a), (b)] printed 2D topographies at 35°C and 40°C substrate temperatures; and [(c), (d)] printed film thicknesses at 35°C and 40°C substrate temperatures.

Figure 12. Printed SEM morphologies of different inks: (a) 8% polyacrylate ink; (a-1) edge contour pattern observed under 35,000× magnification from SEM; (b) 20% polyacrylate ink; (b-1) edge contour pattern observed under 30,000× magnification from SEM; (c) optimized ink; and (c-1) edge contour pattern observed under 5000× magnification from SEM.

With 0.15 wt% surfactant, a film will have a coffee ring structure, as shown in Figure 8(f), while with 0.10 wt% surfactant, a film will have a convex structure, as shown in Figure 8(e). Next, it was investigated if a flat film could be printed at a surfactant concentration between 0.10 and 0.15 wt%. The morphology characteristics of the printed QD film with 0.11, 0.12, 0.13, and 0.14 wt% silicone surfactant are shown in Figure 9. The $H$ value is shown in

Figure 8(f), however, an extremely high concentration will not improve the morphology of the films.
Table 5. It can be seen that the uniformity of the film did not improve at a surfactant concentration between 0.10 and 0.15 wt%.

The drying process of ink containing polyacrylate and surfactant is shown in Figure 10. Staudinger found out that macromolecule polymers generally have a chain structure [24], and that polyacrylate is a heat-curable resin that can be cured through crosslinking at a certain temperature. Thus, when a droplet is inkjet-printed on a substrate, the polymer chains in the polyacrylate are linked by covalent bonds to form a 3D network structure, which traps the solutes in the network structure. The surfactant then moves from the center to the edge of the droplets due to the capillary effect. The local concentration of the surfactant then increases, and an outside-in Marangoni flow is formed.

3.5. Substrate temperature

The substrate temperature affects the solvent evaporation rate, and then affects the printing morphology. Soltman et al. [25] printed films with different morphologies by changing the substrate temperature. Therefore, when the polyacrylate content is 12 wt% and the BD-3033H content is 0.10 wt%, the substrate temperature is 35°C and 40°C, respectively. The morphology characteristics of the printed QD film at different substrate temperatures are shown in Figure 11. The $H$ and $D$ values at different substrate temperatures are shown in Table 6. It can be seen that when the substrate temperature is 40°C, the film is uniform and $H = 1.05$, which indicates that the coffee ring effect has been successfully overcome. This is because with the increase in substrate temperature, three-phase contact wire pinning accelerates, and the compensation from the center to the edge increases [20], so the QD film structure is changed from a convex structure to a smooth structure.

3.6. QD array printing of the optimized ink

Figure 12 shows the printed SEM morphologies of the different inks. It can be seen in Figure 12(a) and (b) that there is a coffee ring pattern at the edge. In addition, QDs gather at the edges, as can be seen in the SEM images in Figure 12(a-1) and (b-1). In Figure 12(c), the QD film printed using the optimized ink is uniform, and the QDs are well distributed at the edge under 5000× magnification from the SEM image in Figure 12(c-1).

Figure 13 shows the 2D and 3D morphologies of the QD film printed using the optimized ink. It can be seen in Figure 13(a) that the printed QD film is uniform, and that the shape of the printed QDs is regular. The printed QD array showed great repeatability and stability. The printed point film with a good surface profile proves that QD devices can be fabricated in the future via inkjet printing.

4. Conclusion

When polyacrylate and silicone surfactant were added to quantum dot (QD) ink, a stable and well-dispersed ink was formed. Then a uniform QD film was obtained by optimizing the polyacrylate content and silicone surfactant in the ink, and the substrate temperature was further changed. The effects of the polyacrylate content, silicone surfactant BD-3033H, and substrate temperature on the morphologies of the printed QD films were investigated. It was concluded that polyacrylate could hinder the outward flow of QDs, and that the silicone surfactant BD-3033H could promote the inward Marangoni flow of QDs. With the increase in substrate temperature, the capillary flow rate increased. Finally, when the polyacrylate content was 12 wt%, the silicone surfactant BD-3033H content was 0.10 wt%, and the substrate temperature was 40°C. A uniform QD film without a ‘coffee ring’ structure was thus printed, which provides a technical guarantee for the fabrication of QD devices in the future.
Disclosure statement

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