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A systematic approach to tune the rheological behavior of perovskite quantum dot inks for inkjet printing applications

M. Zimmermann*[*]*, M. Hölzle*[*]*, M. Oszajca*[+]*, F. Krieg*[+]*, N. Lüchinger*[+]*, K. Albrecht*[*]*, C. Nef*[*]*; *[*]* OST - University of Applied Sciences, Buchs, Switzerland;* [+] *Avantama AG, Stäfa, Switzerland

Abstract

Modern displays utilize color conversion layers to convert blue backlight into colored sub-pixels. Perovskite quantum dots (PQDs) are a very efficient material for this conversion and are currently applied as one continuous film combined with a color filter layer. Inkjet printing could enable the application of PQDs directly into colored sub-pixels, eliminating the filter layer and improving efficiency of the display by design. In this work, PQDs were modified for application by inkjet. UV-curable resin and PQD dispersion were modified to reduce viscoelasticity down into inkjetable range, which was characterized using the TriPAV high frequency rheometer. Printability of PQDs was further shown by dropwatching and manufacturing an inkjet-printed pixel-array demonstrator. The highly loaded ink is UV-curable and solvent-free. It can be applied by inkjet into precise sub-pixel arrays with a pixel thickness of 10μm in one pass. At that thickness, color conversion efficiency and optical density of the PQD sub-pixels fulfill specifications needed for application in modern displays.

Introduction

Perovskite quantum dots (PQDs) have rapidly emerged in the past decade as promising luminescent materials for next-generation displays [1]. In a backlit micro-LED display, the PQDs absorb the light of a blue LED backlight and re-emit green or red light. Figure 1 shows a model architecture of an RGB pixel. The blue LEDs are covered with banks filled with either a transparent scattering layer for the blue sub-pixel or a pixelated color converter (PCC) made out of a green or red emitting PQD/polymer nanocomposite. The role of the PCC is to absorb the blue light and convert it into the desired color with high efficiency, whereas the black matrix, of so-called banks, prevent cross talk between individual PCCs [2]. The industry goal a PCC layers with a high optical density (OD) and a low pixel thickness. Using high pixel thickness leads to more light absorption in the banks and thus to a lower extraction efficiency.

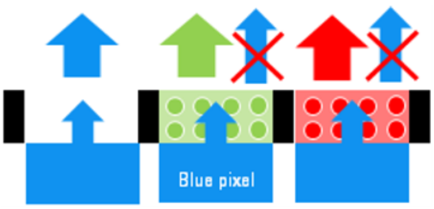

Figure 1. *Model architecture of a micro-LED RGB pixel using color conversion*

PQDs can reach photoluminescent quantum yield (PLQY) near unity, meaning almost every absorbed blue photon becomes an emitted lower-energy photon [3], making them very efficient. Thus the color conversion in PQDs is a purely photoluminescent process – no electrical charge injection is needed. Thanks to large absorption coefficients, in the order of $10^4 - 10^5$ cm^{-1}, the absorption of the blue backlight is very efficient, allowing them to work efficiently even in thin layers [4]. The absorption of a blue photon creates an electron-hole pair (exciton). High exciton binding energies and low defect densities favor radiative recombination, where non-radiative losses are minimized thanks to the defect-tolerant lattice [4]. The emission wavelength of the PQDs can span the entire visible spectrum and is controlled through their composition and size and has a narrow bandwidth of approx. 12-45 nm [5].

PQDs are often combined with polymers to create nanocomposites which makes them processable [6]. These nanocomposites might additionally include scatter particles, which are used to increase the path length that light takes through the film further enhancing the efficiency [2]. Stabilizing the PQD particles in the polymer matrix is often done using surface functionalization. However, modifying particle surface can change their color conversion properties [7].

Despite remarkable progress in device efficiency and color purity, widescale industrial adoption of PQD-based display technology still hinges on solving key challenges, most notably long-term operational stability and scalable, cost-effective manufacturing [5, 8]. Among the various strategies for device fabrication, inkjet printing has been identified as a critical technology route for accurate patterning of PQD emitters [9], due to its advantages as being mask-less, non-contact and scalable [5, 10, 11, 12]. Furthermore, it is superior in terms of material savings. Unlike spin coating – the most widely adopted procedure to obtain a PQD/polymer functional layer – inkjet printing is an additive process that deposits material only where it is needed. This drastically reduces waste of active materials and lowers production costs [8, 13].

Several strategies for inkjet printing of PQDs emerged during the last years. Printing of a perovskite precursor solution on to a polymeric substrate leads to in situ crystallization of PQDs, resulting in perovskite nanocomposites [3, 9, 14, 15]. A disadvantage of this strategy lies in controlling the complex crystallization kinetics [8, 14, 15], making it unfavorable for industrialization. Another printing strategy uses solvent based PQD inks, showing viscosity and surface tension well aligned with inkjet printing [11, 16, 17]. However, dispersion stability, solvent evaporation and coffee stain effect may be challenging [8].

Ideally, PQD inks would be solvent free, consisting only of the PQDs and scatter particles embedded in the resin of the nanocomposite. First, the evaporation rate of the resin during printing has to be controlled, to avoid the formation of the coffee stain effect [15]. Second, in a production process, the whole bank volume should be filled in a single printing step with a low shrinkage after curing, which can not be reached when evaporation is present. Furthermore, the ink should be UV-curable, as this facilitates hanling compared to thermal curing, where the temperature has to be strictly controld during transport and storage. Table 1 summarizes the physical properties making the ink ideal to be used in an inkjet production line.

Ideal properties of inkjet ink

Viscosity	4-12 mPas at jetting temp.
Surface tension	25-38 mN/m
Good dispersion stability	accelerated aging test passed
High degree of filling	>15 wt%

Herein, we show a systematic approach to tune the rheological behavior of PQD/polymer inks for the production of PCC layers via inkjet printing, focusing on green inks. As our approach is generic, it can be easily transferred to red PCC inks or other applications as well.

Ink formulation, characterization and printing

Existing PQD formulations served as a starting point. However, the viscosity of these formulations was too high for inkjet applications. Thus, step (1) was to reduce the viscosity by modifying particles and changing the base resin, while keeping surface tension and viscoelasticity in range. As modifying PQD particles can also change their optical properties, step (2) was to fine-tune the optical properties of the new formulation. Step (3) was to ensure particle stability and chemical compatibility between printhead and ink. The jettability of the ink was validated in step (4) by dropwatching and sample printing and finally (5) by manufacturing a demonstrator.

High frequency rheology with TriPAV

The TriPAV by TriJet ltd. is a high-frequency squeeze-flow rheometer, originally developed by Prof. Pechhold at the University of Ulm, and capable of measuring complex rheology at up to 10kHz [18]. A sample is placed between two plates and exited by a piezo actuator, as shown in Figure 2. The response of the sample is captured with another piezo, allowing to calculate the complex rheology.

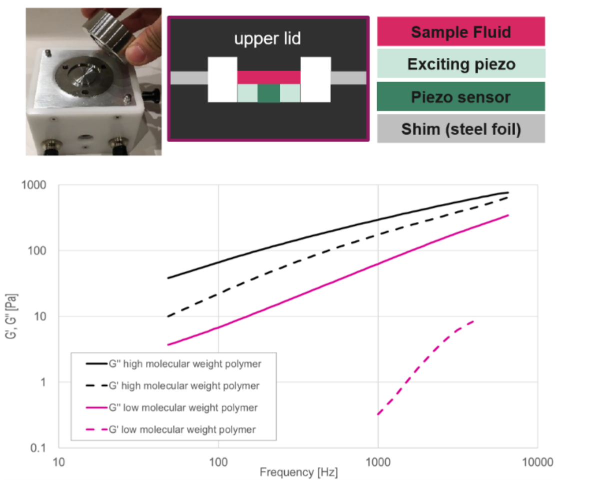

Figure 2. *Schematic drawing of a TriPAV device and an example measurement showing G' and G" for a low and a high molecular weight polymer*

TriPAV measurements do not only measure the viscosity η of a sample, but also the viscoelastic behaviour under vibratory conditions, where the dynamic modulus G^* can be expressed as $G^* = G' + iG''$, with G' being the elastic modulus and G'' being the viscous modulus. This allows to further investigate the behaviour of an ink at high frequencies, important for jettability [18]. In suspensions, viscous properties dominate. However, the presence of G' is a measure for the interaction between the molecules or particles [19]. By analyzing the slope of G', the nature of the elasticity can be evaluated [20].

Characterization of the PQD ink

For optical density (OD) measurement the ink was placed between two glass plates with a separation of 10 µm followed by UV-curing. OD was then measured using an Ocean Optics Spectrometer at 450nm wavelength.

Surface tension was measured by pendant drop using a Krüss DSA30E drop shape analyzer. Results were averaged over at least 20 dispensed drops.

Sedimentation behavior was characterized with accelerated aging at 60°C over 4 weeks. Before the test, TriPAV rheology of the sample was measured as reference. After the test, samples were shaken thoroughly and then measured again with the TriPAV.

As a final method to validate printability of the formulated ink, the jetting behavior was observed with the dropwatcher setup. Drop speed can be deduced from the dropwatcher images by measuring the distance the front of a drop has travelled between two images. A high drop speed is needed to allow high printing speed, further it also increases placement accuracy on the substrate. In addition, the tail speed of the drop is measured. The difference between these two values is important for satellite reduction. If tail speed is higher than head speed, the total tail length will decrease over time, resulting in satellites merging with the main drop during flight as can be seen in Figure 3. However, if the tail speed is lower than the head speed, tail will get longer over time and will generate satellites that cannot merge with the main drop.

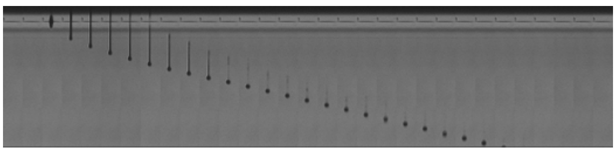

Figure 3. *Example of a drop ejection sequence of a PQD ink. In this case, tail speed is higher than head speed, resulting in the tail shrinking after detachment from the nozzle and subsequent merging of satellites.*

Validating inks for jetting tests

Introducing an ink into a printhead for testing not only takes a lot of time, but also poses a risk of damaging the head and equipment, especially with new and unproven ink formulations. Therefore our goal was to reduce printing tests by using a range of measurement methods to characterize printability of an ink as good as possible before introducing it to the printing system. This way, it was possible to use a set of fail/pass conditions to accept/reject an ink for printing tests, reducing the risk of equipment damage.

Viscosity is the most important property of any inkjet ink and printhead manufacturers provide a viscosity range that an ink has to fulfill to be printable by that printhead. The TriPAV measurements were used to ensure that ink viscosity was in this range. Furthermore, we used the dimensionless elasticity, which is defined as G'/G^*, to define a fail/pass criterion. Max dimensionless elasticity depends on the exact printing setup, but as a general guide, a maximum of 10% can be used [18].

A next criterion for printability is the surface tension. Most printhead manufacturers specify an acceptable range for surface tension. However, to reduce satellites it can be necessary to reduce the acceptable range even further, especially the lower limit of the surface tension.

Reducing sedimentation is crucial for reliable printing. Not only could sedimentation lead to clogging inside the printhead, but it can also lead to changes in ink properties resulting in print failure. Sedimentation can often not be avoided fully, due to the required size of scatter particles to achieve their purpose. In this case it is important that sedimented material can be redispersed easily, for example by shaking. This was tested for our inks by accelerated aging tests and comparing TriPAV rheology before and after. To pass the test, rheology changes should be negligible. Viscosity and elasticity are considered for this.

Inkjet printing and dropwatcher setup

For inkjet printing, a PixDro LP50 printer was used with a Dimatix SE-128 AA printhead. The printer itself can print onto planar substrates of various thicknesses of size up to A4 papers. For the evaluation of the printing process, printing was performed onto nano-porous photopaper.

The printhead used has a nominal drop volume of 30pl and a native resolution of 50dpi. Higher resolutions were achieved by multipass printing. This emulates printing in one pass. Actual one-pass printing could be achieved by stacking multiple printheads and/or using higher resolution heads.

The printer includes a dropwatching unit that uses a stroboscopic illumination synchronized with drop ejection to capture the drop breakup with a camera. The camera image is calibrated to enable measurements from the images. This way, drop volume and speed can be measured. Drop head speed was compared to drop tail speed, which indicates whether a print is satellite-free or not. Timed sequences as in Figure 3 have been captured by capturing drop ejection at equally spaced intervals and arranging the images side by side resulting in an image showing the full drop ejection. The first printing tests were done onto nano-porous photopaper, where it is easy to evaluate dot placement accuracy and the presence of satellites.

Further, the printer also comes with an alignment system to align the printed image with the substrate. This is done via a camera mounted next to the printhead, that can measure the offset of alignment markers between the substrate and the printed image. The offset is used to correct the position and rotation of the image. This was used to print the PQD ink accurately into the banks of the demonstrator.

Manufacturing of PQD pixels demonstrator

Banks were manufactured by standard photolithography using a black photoresist. The photoresist was spin-coated onto a glass substrate with resist thickness $> 10\mu m$ and subsequently exposed with a mask and developed to create an array of square holes with 100µm edge length. The PQD ink was inkjet-printed into these holes and cured. Finally, the demonstrator was covered with a glass. The demonstrator was mounted into a housing including a blue backlight to illuminate the PQD pixels.

Resulting ink formulation process

Ink formulation started with ligand-stabilized PQDs, dispersed in a resin mixture of different acrylate-monomers. Initial viscosity tuning was achieved by switching to monomers with a lower base viscosity.

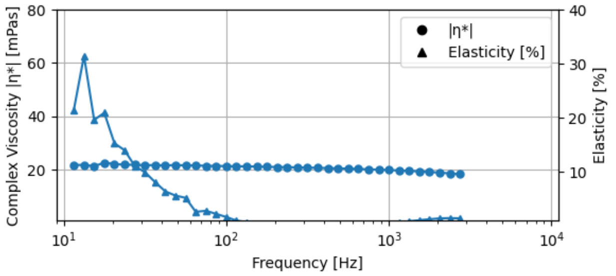

Figure 4. TriPAV measurement of ink 1 showing insufficient particle stability. The elasticity in the low frequencies indicates particle agglomerations.

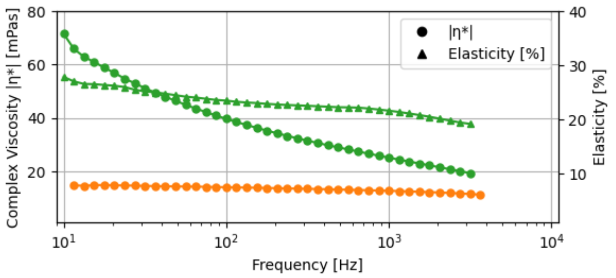

Figure 5. TriPAV measurements of two PQD inks, ink 2 in green and ink 3 in orange, with particles modified with different ligands. They show significant differences in elasticity and viscosity. Ink 2 shows no elasticity.

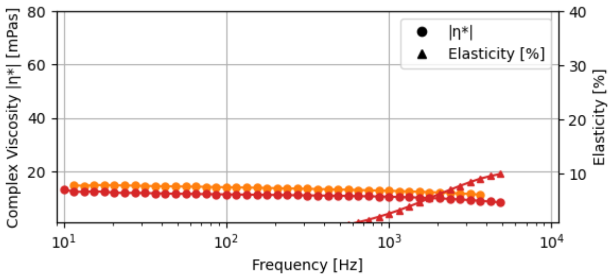

Figure 6. TriPAV measurement of two PQD inks, ink 3 in orange and ink 4 in red. Ink 4 was modified for higher surface tension, which as a side effect added some elasticity.

Once the viscosity of the ink was modified to lay within jettable range, the viscoelastic properties of the ink were investigated using TriPAV measurements. These measurements allow to detect particle instabilities, as shown in Figure 4. Herein, elasticity in the low frequency range can be observed. The elasticity comes from particles agglomerating due to insufficient particle stabilization. For this reason, this ink did not qualify for printing test, due to the risk of agglomerations inside the printhead leading to clogging and head failure.

Figure 5 compares two PQD inks with different ligands for modifying particle surface. Ink 2 (green) shows high elasticity and increased viscosity. This is due to high particle interactions. For ink 3 (orange), the ligand was exchanged, which removed elasticity and lowered viscosity. Particle loading for both inks is identical.

Ink 2 was not accepted for printing tests due to its high viscosity, but ink 3 was. When printing ink 3 onto nano-porous photopaper, a good drop placement accuracy was observed, but a lot of satellites were visible. Reduction of voltage in the waveform lead to reduction of satellites. But this also lead to a reduction of the drop speed, decreasing the dot placement accuracy. Analyzing the drop ejection with the dropwatcher, it was found that tail speed after pinch-off was lower than head speed, which explains the satellites. A higher surface tension could help prevent that by pulling the tail faster towards the head.

Therefore, it was attempted to reduce satellites by modifying the surface tension. The base resin of ink 3 has a surface tension of 33mN/m. Its resin composition was specifically tuned to reach this value. However, the particle load lead to a reduced surface tension of 27mN/m of the ink itself, which resulted in the ink printing with a lot of satellites. For ink 4 (red), the ligands were adopted once more, resulting in an ink with a final surface tension of 30mN/m. Furthermore, this modification lead to an increase of elasticity in the high frequency response, as seen in Figure 6. The combination of higher surface tension and some elasticity resulted in an ink that printed without satellites. Figure 7 shows a photograph of inkjet printed logos on a paper substrate and a microscope image of a dot array printed onto the same paper. This shows, that even though ink surface tension is within specification for this printhead, for satellite-free printing an even higher surface tension was required in our case.

Figure 7. *Paper printout of PQD ink. a) shows a photograph printed PQD logos, b) shows a microscope image of a printed dot array.*

Finally, ink shelf life was investigated. Samples were stored at 60°C for 4 weeks, TriPAV measurements before and after show no change in rheology. Additionally, a sample was stored at room temperature for 1 year. Although this samples showed visible sedimentation after storage, a thorough shaking by hand was enough to redisperse the particles. TriPAV measurements did not show any significant change in rheology when compared to the fresh ink, indicating that particles redispersed well with no agglomerations left.

Final ink properties

The table below lists properties of the final ink developed during this work.

Properties of final ink formulation

Property	Value	Fail/Pass
OD at 10μm	1	> 1
Viscosity	11.5 mPas	8-12 mPas
max. Elasticity	9.7%	$<$ 10%
Surface tension	30 mN/m	28-35 mN/m
Dispersion stability	pass	aging test passed
Degree of filling	20 wt%	$>$ 15 wt%

The final ink was used to build a demonstrator, where the ink was printed into pre-formed banks. This demonstrator is shown in Figure 8. The ink was printed into 100x100µm banks of 10µm thickness. An array of 20x30mm of banks was filled with PQD by inkjet printing.

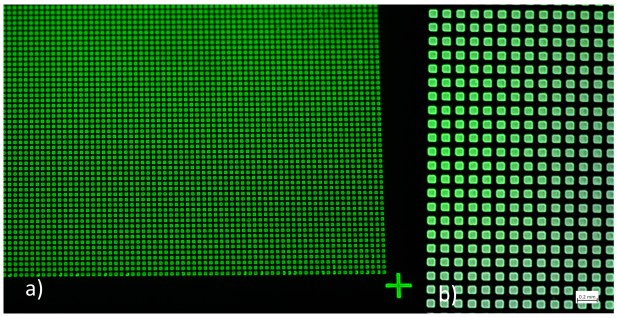

Figure 8. *Image of the final demonstrator manufactured with inkjet. a) shows a photograph of the pixel array including the alignment marks used for aligning the printing process with the substrate. b) shows a microscope image of the pixels.*

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Author Biography

Marco Hölzle recieved his BSc ins systems engineering with a major in photonics from OST - Eastern Switzerland University of Applied Sciences in 2025. He is a research assistant at the INstitute for Microtechnology and Photonics (IMP) at OST with a focus on ink development and chatacterization.

Manuel Zimmerman recieved his BSc FHO in systems engineering with a major in microtechnology from OST in 2022. He is currently working as process engineer in the field of inkjet printing at IMP OST and is pratricipating in the part-time Master of Science in Engineering (MSE) program.