Electroluminescent Colloidal Inks for Flexographic Roll-to-Roll Printing.

Christopher F. Huebner, Joseph B. Carroll, David D. Evanoff, Jr.,
Yurong Ying, Brian J. Stevenson, Justin R. Lawrence,
J. Michael Houchins, Alexandra L. Foguth,
Jay Sperry[†], and Stephen H. Foulger[*]

Center for Optical Materials Science and Engineering Technologies
School of Materials Science and Engineering
[†]Department of Graphic Communications
Clemson University, Clemson, SC 29634-0971, USA

[*]To whom correspondence should be addressed; e-mail: foulger@clemson.edu.

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1 Introduction.

The past two decades have seen an ongoing interest in developing all-organic light-emitting devices as alternatives to inorganic based systems. The inherent advantage in organic small molecule[1] and light-emitting polymer[2,3] systems, relative to inorganic systems that are fabricated through established lithography routes, is the potential that low-cost, large area electronics may be realized[4–6] when these materials are coupled with conventional ink-jet,[7] screen,[8] or roll-to-roll[9–12] printing technologies.

Currently, numerous academic and private industry researchers are working toward highly efficient white light emitting devices focusing primarily on information display applications,[13,14] with fewer groups showing interest in specialty applications that call for the emission of various colors within the visible spectrum,[15] for example, automotive lighting, decorative lighting, marketing (including point-of-purchase advertising and product packaging), and other novelty luminaire. To satisfy the performance criteria of these latter applications, the small molecules and π-conjugated polymers often exploited in OLEDs can require difficult and lengthy synthetic protocols and/or complex device fabrication techniques to create devices with distinct emissions over a broad range of wavelengths,[16–21] while inorganic quantum dot (QD) based light emitting devices (LED) are currently cost-prohibitive for widespread use.[22] An alternative to these systems lies in the creation of emissive materials that utilize an organic electroluminescent (EL) dye embedded in a hole and an electron transporting polymer host.[23] A thin film comprised of a single EL dye and hole & electron transporting host can exhibit excellent emission characteristics, but extending this device design to encompass other colors through the addition of various dyes to the host is not straightforward; a thin film containing several spatially-adjacent EL dyes can result in energy transfer processes which defeats the additivity approach.[24,25] The ability to sequester EL dye components in a host with no appreciable energy transfer offers the ability to create tailored emissions over a broad range of wavelengths by mixing red, green, and blue emitters.

In this effort, we present a simple route to producing electroluminescent colloidal particles that can be designed to produce a wide range of emission colors and that can be converted into printable inks. This approach exploits the concept that the particles in a colloidally-based OLED can be viewed as individual "particle-devices". In addition, due to the ubiquitous nature of aqueous-based colloids in printing inks utilized in current commercial printing technologies, the methodology presented herein is amenable to fabricating OLEDS through large scale & high-throughput printing techniques, for example, roll-to-roll manufacturing processes.[12] The development of low cost roll-to-roll printing processes to manufacture OLEDs has the potential to eliminate the manufacturing difficulties that currently exist with high performance organic electronics technologies and prevent their more widespread adoption; it is generally accepted that the few organic electronic products currently available to the consumer are constructed with conventional batch processes and are relatively high cost.

Figure 1a presents a schematic of the colloidally-based organic light emitting device employed in this effort. In this construction, the anode of the device is a glass substrate which has a templated layer of indium tin oxide (ITO) on its surface. The glass/ITO surface is planarized with the addition of a ca. 150 nm layer of the transparent dielectric poly(butyl methacrylate) (PBMA) to the regions of the exposed glass surface. This dielectric layer is relatively soft at room temperature, exhibiting a glass transition temperature of ca. 35 °C, and assists in reducing field enhancements and corresponding electrical shorts at the edges of the ITO. Colloidal particles that are primarily composed of a hole-transporting polymer doped with an electron transporting small molecule and an EL dye (cf. Figure 1b), are spin-coated onto the anode to form the active layer of the device. The particles are formed through a mini-emulsion approach[26] and exhibit particle diameters which can, under varying synthetic conditions, range from 50 nm to 500 nm (cf. Figure 1b). Prior to their deposition on the anode, a poly(3,4-ethylenedioxythiophene):poly(styrenesulfonate) (PEDOT:PSS) dispersion is mixed into the colloids that acts as a binder for the colloids and assists in the reduction of field singularities around the particles that may result in regions of pin-hole formation. Finally, the cathode assembly is formed by the evaporation of a ca. 30 nm thick calcium layer onto the colloids, over which a thicker aluminum capping layer is evaporated. This device layout serves as a model system to establish the EL performance of the colloidal inks, while continuing efforts will focus on all in-line fabrication of printed devices.

Figure 1b presents the components of the emissive colloids. The colloids were composed of poly(9-vinylcarbazole) (PVK), 2-(4-biphenylyl)-5-(4-tert-butylphenyl)-1,3,4-oxadiazole (tBu-PBD), and an EL dye, either Coumarin 1 (B), Coumarin 6 (G), or Nile Red (R). The formation of the colloids utilized a simple and versatile approach referred to as a "mini-emulsion" process and has been exploited by Landfester and coworkers[26] for the formation of a range of colloidal systems, including a modified poly(*p*-phenylene) that was utilized to form an OLED.[27] One form of the mini-emulsion process involves the shearing of a surfactant stabilized hydrophobe in an aqueous environment. The hydrophobe can be composed of, for example, a water-insoluble polymer and/or small molecule dispersed in a organic solvent. Shearing the two-phase system results in the formation of particles which can span over the large range of 50 to 500 nm. A subsequent removal of the organic solvent through evaporation results in the formation of polymeric particles dispersed in an aqueous environment. The scanning electron micrograph (cf. Figure 1b) presents a typical batch of the mini-emulsion produced emissive colloids after an extensive dialysis to clean the particles. A typical batch of the colloids exhibited an average weight-based composition of ca. 63 % PVK and ca. 37 % tBu-PBD, with the dyes composing less than 0.5 %. Based on the micrograph in Figure 1b, it is clear that the colloids range in diameter from 20 nm up to 135 nm; the mean par-

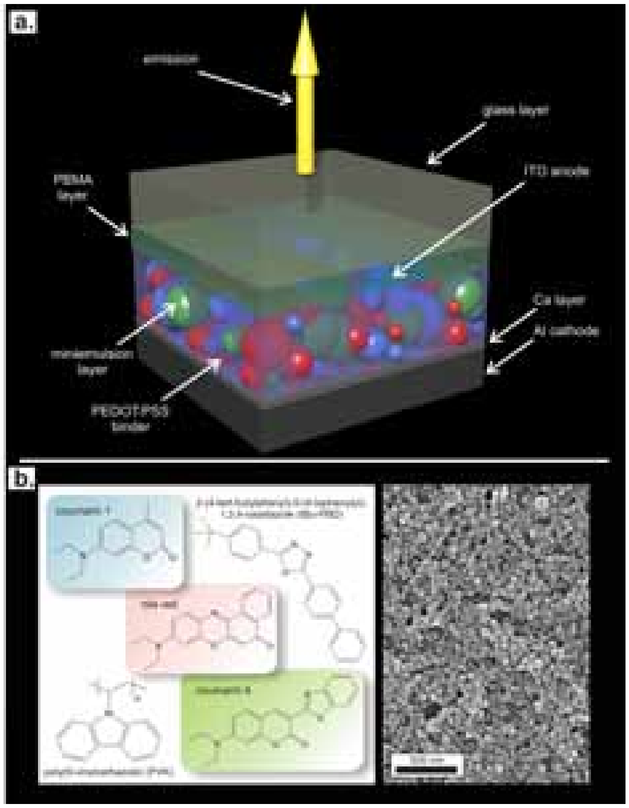

Figure 1: (**a.**) Schematic illustration depicting the construction of a single pixel colloidally-based organic light emitting device. A glass substrate with a templated layer of indium tin oxide serves as the anode of the device. In order to planarize the surface and prevent regions of field enhancement, a ca. 150 nm layer of poly(butyl methacrylate) is spin-coated onto the anode and removed from the ITO region. Emissive colloids, mixed with a binding poly(3,4-ethylenedioxythiophene):poly(styrenesulfonate) dispersion, are deposited onto this electrode assembly. The cathode is formed by the evaporation of a ca. 30 nm thick Ca layer onto the colloids, over which a thicker Al layer is evaporated. (**b.**) The emissive colloids were formed through a mini-emulsion[26] approach and were composed of poly(9-vinylcarbazole) (PVK), tBu-PBD, and an EL dye, such as Coumarin 1 (B), Coumarin 6 (G), or Nile Red (R). SEM micrograph of a typical batch of the dye-doped colloids.

ticle diameter was 51 nm, but unlike a more traditional emulsion polymerization,[28] the polydispersity was significant relative to the mean and resulted in a standard deviation of 15 nm.

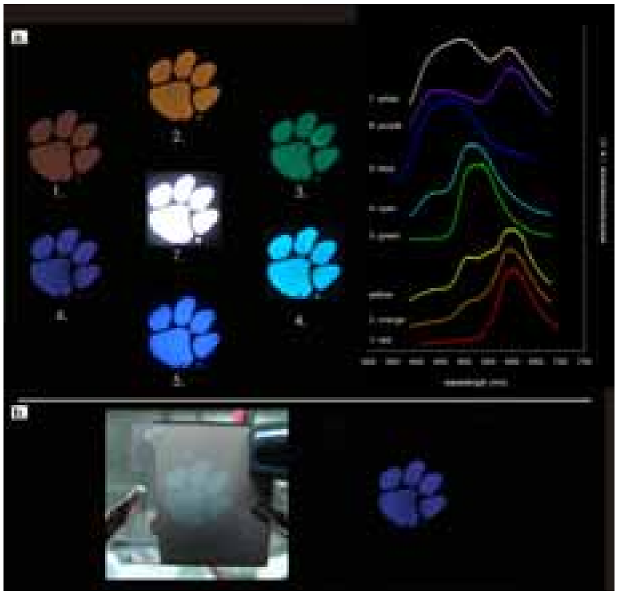

Figure 2: (**a.**) Optical images (**1**-**7**) of colloidally-based organic light emitting devices templated into a 12.7 mm wide tiger paw insignia with varying RGB dye ratios (left side of the figure) and the corresponding electroluminescence spectra (right side of the figure). In addition, the electroluminescence spectrum of a yellow emitting device is presented. (**b.**) Optical image of a colloidally-based organic light emitting device: prior to being biased (left image) and with an applied 9 volt bias (right image).

Figure 2a presents the optical images of a number of energized colloidally-based organic light emitting devices templated into a 12.7 mm wide tiger paw insignia. The devices were fabricated with varying ratios of the dye-doped colloids to produce a wide spectrum of colors. The corresponding EL spectra of the devices are presented in the right side of the figure, with an additional EL spectrum of a yellow emitting device. The excitation of the dyes in a PVK:tBu-PBD host is accomplished through at least two proposed mechanisms: (1) energy transfer or (2) carrier trapping. Energy transfer can take place through a Coulombic interaction (Förster) or electron exchange (Dexter) mechanism and occurs when an excited donor (exciton) located in either the PVK, tBu-PBD, or a PVK:tBu-PBD complex transfers energy over to a ground state dye (acceptor). To be an effective means of energy transfer, both the Förster and Dexter mechanisms have a number of conditions that must be satisfied, but two major considerations include the spatial separation between the donor & acceptor and the spectral characteristics of the donor's emission and the acceptor's absorption. A Förster-type transfer can be potentially effective up to ca. 10 nm, while a Dexter-type transfer, though highly dependent on the electronic configuration of the donor-acceptor, is roughly limited to distances under 2 nm.[29] In addition, both modes of energy transfer require a high level of spectral overlap of the donor's emission and the acceptor's absorption. The second mechanism of exciting a dye relies on charge carrier trapping on the dye, with a subsequent recombination.[30] Both means of exciting a dye in a PVK:tBu-PBD host are potentially operative in a EL configuration, while the reduced probability of generating free carriers in a PL study implies that a trapping mechanism will not contribute to dye emission in this configuration.[31]

The primary RGB colors utilized in this work for creating additional colors are presented in Figure 2a(1, 3, & 5). The EL spectral characteristics of the red (2a-1) and green (2a-3) colloidally-based OLED are very similar to the PL spectra of the individual dyes dilutely dispersed in cholorform, while the corresponding PL response of the dye-doped colloids results in the appearance of an additional peak centered at 425 nm (cf. Figure 4a). Recent PL studies of PVK:tBu-PBD films indicate that the PL emission peak centered at 425 nm (cf. Figure 4b) will red-shift to 440 nm under an EL configuration and has been ascribed to the formation of PVK:tBu-PBD exciplex.[32,33] The absence of this peak in the EL spectra of the R&G-derived devices suggests a complete energy transfer from the PVK:tBu-PBD host to these dyes. In contrast to the R&G systems, the B-derived device exhibits an EL spectral response that is similar to the PL response of the blue-doped colloids, as indicated in Figures 2a-5 & 4a, but is significantly different to the PL response of the dilutely dispersed blue dye. Both the EL and PL response of the blue-doped colloids exhibit an onset to emission that is at ca. 350 nm, which is at least 40 nm less than the onset for PL of the dilutely dispersed blue dye. This can be attributed to the poor spectral overlap in dye absorption and host emission; the dilutely dispersed Coumarin 1 (B) dye exhibits an absorption peak centered at ca. 370 nm, while the emission of the PVK:tBu-PBD exiplex (cf. Figure 4b) has a maximum of 425 nm. The limited energy transfer possible from the host to the dye results in the appearance of the PVK:tBu-PBD emission characteristics in the EL response of the blue dye-doped colloid. In addition, there is a small shoulder visible within the EL response of the blue dye-doped colloid that is centered at 610 nm (cf. Figure 2a-5); this weak contribution has been seen in EL studies of PVK and has been tentatively assigned to PVK electromers due to its absence in PL studies of PVK.[32]

These devices exhibited luminance values ranging from under 10 cd/m^2 for the blue and red-doped colloidal devices to 30 cd/m^2 for the green-doped colloidal device. Figure 3 presents a typical current-voltage plot for the colloidally-based OLEDs templated into 4 x 3 mm rectangular pixels. The curve represents the average current characteristics of five samples composed of varying RGB dye-doped colloid ratios, while the error bars represent the upper and lower currents exhibited during a specific run. The inclusion of the PEDOT:PSS does not appear to act as a short circuit for the devices by evidence of a lack of an Ohmic response in the current-voltage curves, suggesting that the PEDOT:PSS is unable to percolate across the electrodes at the concentration utilized. Although the luminous output for these system is not optimal relative other current "color-tailored" OLEDS[15,23] or QD-based devices,[22] the charge density characteristics correlate well with commercially available light emitting devices that compete in the same niche markets that call for specifically tailored emissions across the visible spectrum. For example, typical inorganic LED devices used in automotive lighting applications operate at a forward bias of 12 V with a current draw of 20-30 mA, similar to the performance of the colloidally-based OLEDs (cf. Figure 3). In addition, the calculated current density at 10 V forward bias is ca. 100 mA/cm^2; the low device turn-on voltages of the colloidally-based system, coupled with low current requirements and reasonable operating voltages, is comparable to inorganic alternatives such as QD-based devices, which are fabricated at a significantly higher material costs and require both a distinct hole and electron injection layer that necessitate lengthy and expensive fabrication methods.

The simple construction of these devices allowed for the rapid screening of various dye-doped colloidal ratios to explore their combined emission characteristics. Figure 2a(2, yellow, 4, 6, & 7) presents a number of templated tiger paw insignias with their corresponding EL spectra. In a typical thin film device, the simple mixing of dyes at molecular lengths scales will result in the transfer of energy from shorter wavelength to higher wavelength emission, while the sequestering of the dyes in individual particles allows for their mixing with the retention of their individual emission characteristics due to a lack of energy transfer between particles. Figure 2b presents an optical image of a colloidally-based organic light emitting device templated into a tiger paw insignia, before and after it has been energized. The design of the active area is clearly visible on the unbaised device (left image) and was achieved through a templated photoresist. The energized device (right image) exhibits a purple emission when biased at 9 volts and was achieved through the color mixing of colloids comprising red and blue emitting dyes. Figure 2a(7) presents a device which exhibits a broad spectral emission and a "white" appearance; the ratio of RGB dye-doped colloids utilized for this device was intially estimated through a PL study.

Figure 5 presents the 1931 International Commission on Illumination (CIE) chromaticity diagram with the color characteristics of the devices presented in Figure 2a plotted at their specific CIE *x,y*-color coordinates. All the colors that can be formed by mixing three color sources together are found inside the triangle formed by the source points on the chromaticity diagram, while white appears approximately

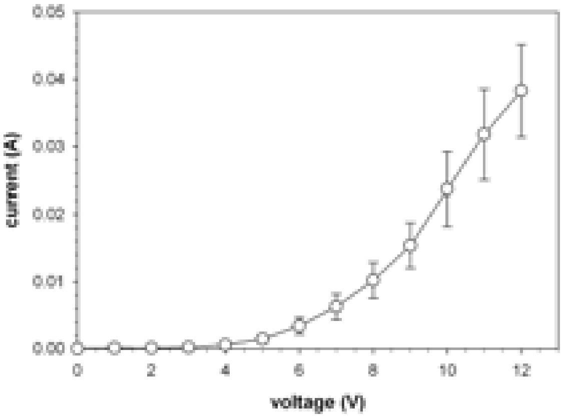

Figure 3: Current-voltage characteristics for colloidally-based organic light emitting devices templated into 4 x 3 mm rectangular pixels. The curve represents the average current characteristics of five samples and the error bars represent the upper and lower currents exhibited during a specific run.

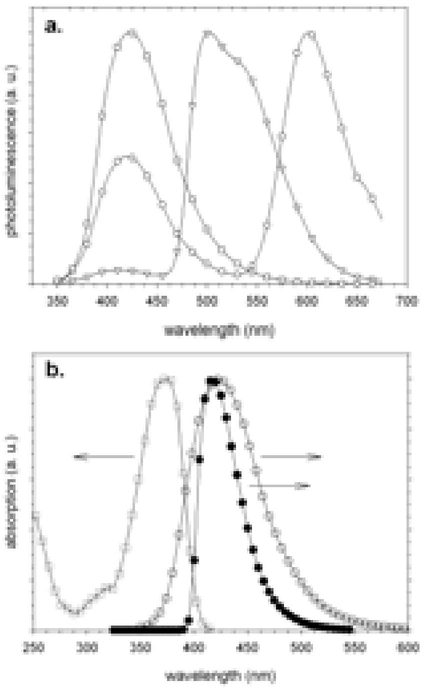

Figure 4: (**a.**) The photoluminescence spectra of the three RGB dye-doped colloids at an excitation wavelength of 343 nm: red (◯); green (▽); and blue (□). (**b.**) The absorption (□) & photoluminescence (excitation at 375 nm; ◯) spectra of the Coumarin 1 (B) dye in chloroform. The photoluminescence spectra (excitation at 343 nm; ●) of a colloid composed of only PVK and tBu-PBD in a ca. 63/37 weight ratio. All curves have been scaled for clarity.

at the point (1/3, 1/3). Table 1 presents the dominate wavelength (λ_d) and color purity (p) of the devices presented in Figure 2a. The dominate wavelength for a color is defined as the wavelength on the edge of the diagram that lies on a line joining the white point and the *x,y*-position of any color, while the color purity is defined as the ratio of distances between (1.) the color point and the white point and (2.) the dominate wavelength and the white point. The primary colors will define the region of the chromaticity figure accessible by the additive color approach; achieving a saturated color in an OLED requires that the p values of the primary RGB colors used to generate colors within the chromaticity diagram be as saturated as possible with p values as close to 1. Monochromic or saturated colors have p values that approach 1, while a color will have a p value that approaches zero as the color becomes less vibrant or more "washed-out". In the current selection of dyes, the G and R-derived dye-doped devices exhibit superior color purities with values of 0.70 and 0.92, respectively, while the B-derived device exhibits a dominate wavelength of 484 nm and a color purity of 0.35. The lower p value is in part due to the limited overlap of the PVK:tBu-PBD emission with the absorption of the Coumarin 1 (B) dye, though through a judicious choice in dye selection, it is possible to optimize the overlap to achieve high color purities in the final colloidally-based organic light emitting devices.

color	CIE coordinates (x,y)	λ_d (nm)	p
red	0.56, 0.41	590	0.92
green	0.33, 0.57	553	0.70
blue	0.24, 0.28	484	0.35
cyan	0.28, 0.44	519	0.20
yellow	0.44, 0.44	578	0.66
orange	0.48, 0.43	583	0.73
purple	0.37, 0.33	611	0.11

Table 1: Chromaticity coordinates for colloidally-based organic light emitting devices presented in Figure 2a. Dominate wavelength (λ_d) and color purity (p) are based on white at CIE coordinates of (1/3, 1/3).

The dye doped colloids were formulated into a printable ink and printed using a flexographic roll-to-roll printer following techniques established previously for the printing of colloidal systems.[34] Figure 6a presents an optical image under ambient laboratory light of a printed zone on an ITO-coated PET sheet using a G-derived colloidal ink . The printed zone appears grey in color, but when the zone is excited with a 365 nm light source (cf. Figure 6b), the green PL characteristics of the dye-doped colloids is apparent. Current efforts within our group have begun to successfully extend this technology to commercial roll-to-roll printing of devices.[35]

The approach presented in this OLED effort de-

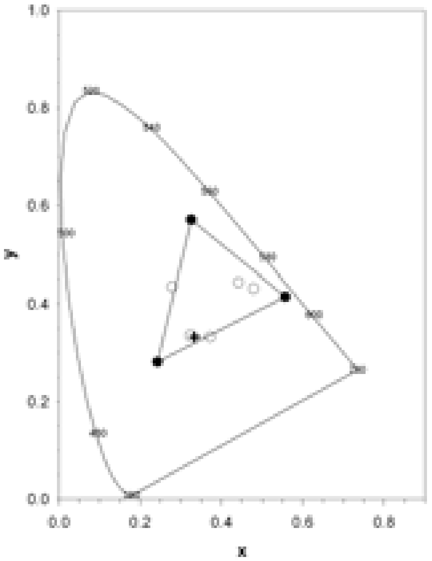

Figure 5: 1931 International Commission on Illumination (CIE) chromaticity diagram with the electroluminescence-derived color coordinates of the colloidally-based organic light emitting devices presented in Figure 2a. The CIE coordinates of the RGB primary color devices (●) define a triangle that delimitates the color region accessible with the current selection of electroluminescent dyes. These primary color dye-doped colloids were mixed in various ratios to create additional colors (○), while "white" (+) is presented at CIE (x, y)-coordinates of $\frac{1}{3}, \frac{1}{3}$.

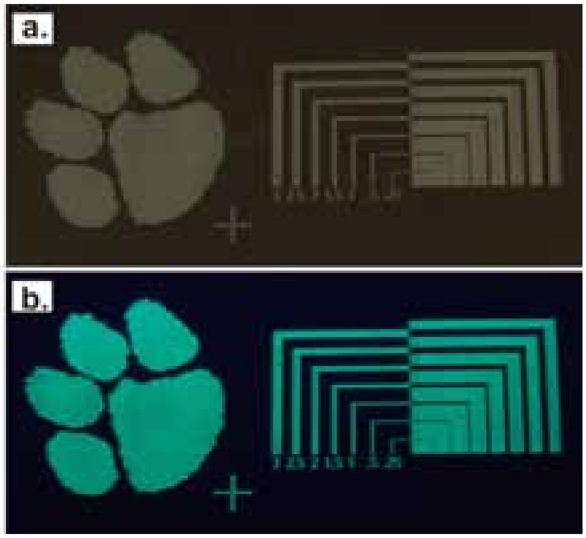

Figure 6: Optical image of flexographically printed image on ITO-coated poly(ethylene terephthalate) sheet using a G-derived colloidal ink. Printed zone illuminated with (**a.**) ambient laboratory light and (**b.**) 365 nm source. Line widths presented in Postscript points (1 Postcript point = 0.3528 mm).

fines a simple methodology by which materials designers can view their colloidal systems as individual "particle-devices". In essence, if one can efficiently transport charge to an individual colloidal particle, it is not unrealistic to assume that single particle devices could be built. In addition, this colloidally-based methodology for fabricating color-tunable OLEDs presented here is eminently amenable to the rapid printing of devices through commercial printing technologies.[12]

2 Methods.

General synthetic procedure for the emissive colloids & device active layer. In a typical synthetic attempt of the emissive colloids, a solution containing 1.43 ml of $CHCl_3$, 41.25 mg of poly(9-vinylcarbazole) (PVK), 33.75 mg of 2-phenyl-5-(4-biphenylyl)-1,3,4-oxadiazole (tBu-PBD), and an EL dye was formed. For the solution containing the red EL dye, 0.225 mg of Nile Red was added to the solution. Additionally, the green EL dye solution contained 0.45 mg of Coumarin 6, while the blue EL dye solution also contained 0.45 mg of Coumarin 1. These solutions were then added to an aqueous solution containing sodium dodecyl sulfate (SDS) (100 mg in 25 ml H_2O). This two-phase solution was then emulsified using a tip sonicator (VirTis Virsonic 600 Ultrasonic Cell Disruptor) for 4 minutes at a 12 W power setting. After sonication, the colloidal dispersions were placed onto a hot plate at ca. 60 °C and stirred for 12 hours to evaporate any remaining $CHCl_3$ present in the solution. The subsequent emissive colloids were cleaned through dialysis (Spectra/Por MWCO = 50000) to remove residual small molecules, dyes, or surfactant. The dialysis water was changed every ca. 12 hours with 18.2 $M\Omega{\cdot}cm$ deionized water until the conductivity of the dialysis bath measured less than 0.5 $\mu S/cm$ at the end of a 12 hour period.

Utilizing an additive primary color model, the red, green, and blue emitting colloids were mixed at various ratios to achieve color shades within the color space defined by the RGB dyes. The ratios of RGB colloids required for a specific color shade were estimated through the observed PL spectrum of the combined colloids. To create the "active layer" mixture that was finally spun-cast onto the device, the RGB colloidal mixture was combined with an electronic grade of poly(3,4-ethylenedioxythiophene):poly(styrenesulfonate) (PEDOT:PSS) (Baytron P Formulation VP CH 8000; H. C. Stark, Inc.; $\rho \approx 10^5 \Omega{\cdot}cm$) in a 4:1 (v/v) ratio.

Fabrication procedure for colloidally-based organic light emitting devices. Small test-pixels were fabricated from a 12.5 mm on-edge square piece of float glass with an indium-tin-oxide (ITO) coating (Delta Technologies CG-50IN-5107) that was etched with a 4 x 12.5 mm rectangular pattern and cleaned through a solvent / sonication / plasma procedure. A poly(butyl methacrylate) (PBMA):$CHCl_3$ solution (25 mg/ml) was then spun cast onto the substrate to a thickness of ca. 150 nm and then removed from the ITO region with a $CHCl_3$ soaked cotton tipped applicator. In addition, a larger 25 mm on-edge square piece of ITO-coated float glass was etched and patterned with a positive photoresist (HPR 504; Arch Chemicals, Inc.) to form a tiger paw template; with these structures, no PBMA layer was required. The active layer mixture (i.e. emissive colloids with PEDOT:PSS) was then spun cast onto the substrate to a thickness of ca. 100 to 150 nm. A calcium electrode was then thermally evaporated onto the active layer and then capped with a layer of evaporated aluminum.

Characterization of colloids & devices. Thermal gravimetric analysis (TA Instruments TGA 2950) was carried out on the colloids under a nitrogen purge from 20 °C to 900 °C in a high-resolution mode, while their absorption spectra were collected with a Perkin

Elmer Lambda 900 UV/Vis/NIR spectrophotometer. In addition, the photoluminescence (PL) characteristics of the colloids were collected on an Horiba Jovin-Yvon Fluorolog FL-3-22/Tau-3 Lifetime spectrofluorometer over a wavelength range of 350-675 nm utilizing an excitation wavelength of 343 nm.

The electroluminescence characteristics of the colloidally-based organic light emitting devices were studied in a MBraun UniLab glove box under an argon atmosphere. A computer-controlled Keithley 228A Voltage/Current Source supplied a DC voltage to the ITO anode, while a Keithley 2001 Digital Multimeter recorded the current within the device. In addition, the EL spectra were obtained with a Horiba Jovin Yvon MicroHR monochromator with a Synapse CCD detector.

All data were taken at a temperature of 23 °C unless otherwise noted. All chemicals were purchased from either Sigma-Aldrich or Fisher Scientific unless otherwise noted.

Flexographic printing. The flexographic ink was formulated by taking 1.0 g of the aqueous dye-doped colloidal dispersion and adding 0.5% SDS (w/w) in order to decrease the surface tension of the dispersion. This colloidal solution (500 μl) was then added to a printing media (4400 μl) composed of 22.7% (v/v) PEDOT:PSS, 68.2% (v/v) poly(vinyl alcohol) (PVA), 9.1% (v/v) ethylene glycol, and 0.5% (v/w) SDS to arrive at the final printing ink.

The printing of the ink was done on a Flexiproof 100 Print Proofer (Testing Machines, Inc., Ronkonkoma, NY 11779 USA) operated at a feed rate of 25 m/min^{-1}. A digitally imaged DuPont DFH plate with a caliper of 0.17 cm was thermally developed using a DuPont FAST processor. For transfer of the formulated ink from the inking well to the flexographic plate, a 138lpi/11.7bcm anilox roller was used. Printing substrates were ITO-coated poly(ethylene terephthalate) (PET) films (CP Films, Martinsville, VA 24115 USA).

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35. Our initial attempts in printing colloidal-based OLEDs have focused on green-light emitting devices with the following construction: ITO-PET anode/printed electroluminescent ink/brush-applied colloidal silver cathode; these devices have yielded visible electroluminescence (not quantified).