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Inorganic Electro-Luminescence Created by Nobble Hybrid-Printing-Process

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ABSTRACT

The developments of flexible conductive patterns have been a significant attraction for electrical devices. Extensive research has been conducted in order to develop print patterns for flexible circuits; however, traditional technologies pose a plethora of problems from an economic standpoint. To solve these problems, a noble hybrid-printing-process (NHPP) equipped with inkjet printing of separating photo-curable polymer ink and polishing carbon group powders were proposed. An ordinary desktop inkjet printer was selected to fabricate conductive patterns. The nobility of this printing process is due to the interfacial reactions between the polymer ink and conductive powder: Carbon Black sustains a point to point interfacial reaction while Carbon Nanotubes (CNT) and Graphite sustain a face to face interfacial reaction.

Ultra-violet light-emitting diode (UV LED) polymer (40cP) and a 4watt UV LED lamp (365nm) were prepared for printing and curing of patterns. Conductive carbon group powders such as CNT, carbon black, and graphite were coated onto flexible patterns by polishing and the implementation of Inorganic Electro-Luminescence (IOEL) process. The study proved that printability was affected by changing the viscosity of the UV LED Polymer. The viscosity of the inks were adjusted from 6.6cP to 10.0cP by adding methanol to optimize the range of viscosity for more accurate pattern formation. It is observed that good quality of patterns can form on a flexible PET substrate when the ink's viscosity is in a range of 7.5-8.0cP and the width of the pattern is larger than 200 µm.

UV LED ink was printed on PET film at 1 cycle and at 20 cycles. The thickness of each sample measured 15.11µm and 136.23µm, respectively. The coated samples with conductive materials had a conductivity range between 2.40×10^{-1} to 4.46×10^{-1} S/cm measured at room temperature using NHPP. The IOEL samples had a conductivity of 3.154 x 10 S/cm, which enhanced its properties of electrical conductivity.

Keywords: Inkjet Printer, NHPP, UV LED Polymer, CNT, Viscosity, Flexible Conductive Patterns, IOEL

INTRODUCTION

Conductive patterns on flexible substrates have received significant attention in the past years. It was applied to a wide range of application fields such as electronic circuits, displays, and sensors [1-5]. Most of all, conductive lines have been formed via conventional electronic systems. Traditional technology and photolithography, are both, expensive and time consuming contributing to a substantial amount of environmental problems [5-6]. Limits to select substrates and printable materials are also a problem; this is because photolithography has been used in intricate procedures and expensive dispensers [7-8]. Therefore, fabrications of simple and productive techniques have been required to make conductive patterns in recent years.

Currently, inkjet printing is one of the most promising non-contact printing techniques because its process is simple in manufacturing and is cost efficient when printing inks on flexible substrates [9-10]. This technology is a non-contact printing method using a pattern on-demand system that experiences minimal loss and modification of the material. An ordinary desktop inkjet printer is able to handle pico-liter volumes of liquid when it prints precise patterns [11]. For these reasons, it has been widely used and applied to the fields of arts, ceramics, electrical devices, organic semiconductors, thermoelectric composite structures, and biopolymers [12-16].

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Inkjet printing techniques do experience problems that need to be rectified. These problems result from multiple types of materials. Various types of conductive materials such as molten metals, conductive polymers, metal precursors, metallic nanoparticle suspension, and metallic compounds are used as inks in inkjet processes. [17-21]. In these cases, a post-treatment process such as sintering is required to solidify the metal patterns from liquid form of conductive inks [22-23]. Another disadvantage of the inkjet printing process is the contraction of patterns and substrates caused by the sintering process [24-25]. High concentration of metal nanoparticles are needed to increase electrical conductivity, however, this causes the printing nozzles to clog via metal nanoparticles. Clogging of metal nanoparticles inhibits dispensing during inkjet printing which causes lower printability and accuracy. Further, printing should be repeated for more than 150 cycles with a low concentration of metal nanoparticles to compensate for low ink conductivity. [26].

Gold and silver inks have been used in inkjet processes due to its high electrical conductivity and oxidation stability [27-28]. However, the use of gold and silver inks results in high manufacturing cost [28-29]. For most of these metal inks, the conductivity decreases while the addition of a binder or surfactant increases. The machines used in the inkjet industry, such as, maskless mesoscale materials deposition (M3D), electro-static deposition (ESD), and jet-printing are also relatively expensive (>$700) compared to desktop inkjet printers (<$100). Higher price for inkjet printing machines, materials and high energy consumable processes should also be resolved for practical application processes for printed electronics.

The ink's viscosity and surface tension plays an important role in accuracy of the traditional inkjet printing processes [30]. Size and dispersion of metal nanoparticles are also crucial characteristics that prevent the metal particles from clogging the printing nozzles and creating aggregation [5, 31-33].

In order to solve the complications and maximize the economic benefit of the inkjet printing technology, this study is focused on the development of a new hybrid printing process and its materials. The application of using Inorganic Electro-Luminescence (IOEL) is the combination of UV LED inkjet printing while simultaneously curing after the patterned product has been created from the printing process. The use of IOEL is a more cost efficient way of processing and results in higher electrical conductivity due to its alternating layering method. Consequently, low prices of UV LED curable polymers was used as the ink as an alternative of the conventional high-conductive metal inks while a noble hybrid-printing process was developed to make conductive patterns more efficiently.

EXPERIMENTAL PROCEDURE

Materials and Equipment

In this study, an ordinary desktop inkjet printer (Epson XP-202) is a type of piezoelectric used to print conductive patterns. As shown in Figure 1(a), the XP-202 printer consists of 4 different printing heads (black, yellow, red, and blue), each printing head includes 42 nozzles with the exception of the black cartridge which includes 128 nozzles, respectively. The minimum volume of ink droplets that can be dispensed is 3pL (pico liter). Only the black cartridge is used to print the UV LED curable ink, and the remaining cartridges are filled with methanol. This is depicted in Figure 1(b). The reason, is that if there are empty cartridges, printing is not applicable. Figure 1(c) is a depiction of the ink cartridges used throughout the study paired with the UV LED Lamp.

Various types of inkjet processes include bubble-jet, electrostatic, acoustic, and piezoelectric methods. Piezoelectric print nozzle can dispense the viscous inks in the range of 5-12cP while the viscosity spectrum that can be printed by the other ink-jetting methods is 2-30cP. [17, 34]. In this study, UV LED curable polymer (CHOKWANG, Inc.) is used as an ink. The polymer has 40cP of viscosity and is cured only in 365nm wavelength of UV light. Thus, the viscosity of UV LED curable polymer was lowered by adding methanol (Fisher Chemical, ≥99.8 %) for its piezoelectric inkjet printing. The amount of UV LED curable polymer was adjusted from 60 to 70% in volume to observe the printability and accuracy of the UV LED polymer as a function of viscosity. A 4watt UV LED lamp emitting 365nm single wavelength was used to cure the printed patterns on a flexible substrate. Specifically, the UV LED lamp obtains 10 of 4-in-1 type LED chips which is described in Figure 1(d).

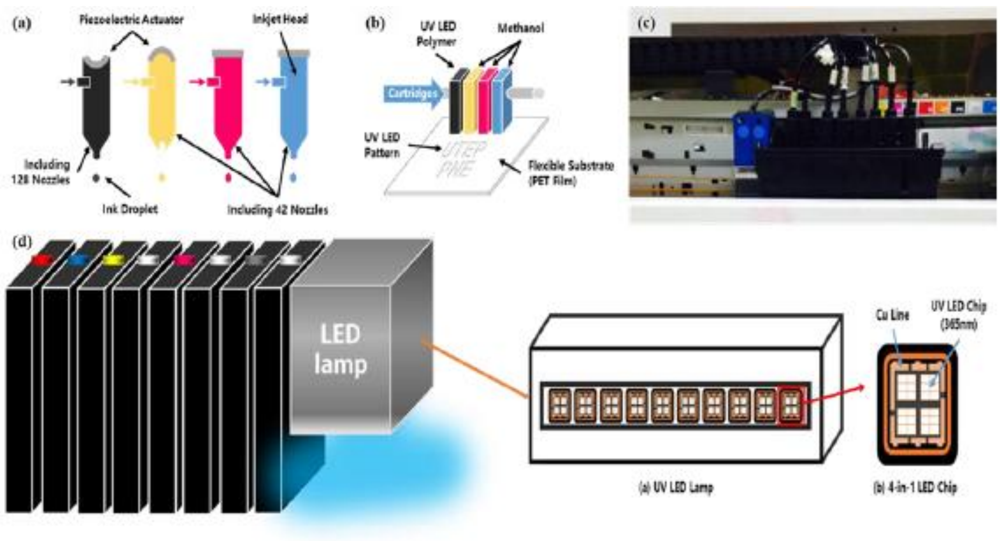


Figure1. *Figure 1. Schematic of piezoelectric inkjet printer and 4 watt UV LED lamp. (a) Piezoelectric inkjet heads; (b) inkjet printing of UV LED polymer; (c) photograph of Inkjet heads alongside with UV LED Lamp; (d) Structure of UV LED Lamp; (e) 4-in-1 UV LED Chip*

Noble Hybrid Printing Process

Detailed printing conditions are shown in Table 1. 10mL of UV LED curable ink and methanol were loaded in each cartridge. Microsoft PowerPoint software program was used to design the patterns. Fifteen patterns with varying widths were drawn to confirm the printability and accuracy of UV LED inkjet printing in terms of viscosity. The range of patterns was set up from 18µm to 3500µm, respectively. The various inks were dispensed on flexible substrates, the overhead projector (OHP) film which is a type of polyethylene phthalate at 25℃. Dispensed inks were cured for 15 seconds under LED UV light in 365nm wavelength to maintain the shape of the printed patterns and the distance between the film and lamp was 1cm. After that, 1mL of conductive materials were coated on the surface of rectangular shaped patterns without any dispensers in order to provide conductivity. Multi-wall carbon nanotubes (MWCNT), conductive carbon black, and graphite were used as conductive materials throughout this study. This hybrid printing process is a simpler and cheaper way to make electronic circuits when compared to traditional printed electronics techniques such as roll-to-roll (R2R), photomask, and direct writing.

Table1. *Specific Conditions of Noble Hybrid Printing Process*

Classification	Name of Materials or Machine	Characteristics
Printing Materials	UV LED Ink (UV Curable Polymer)	- Loading Amount: 60, 65, 67, 70 vol% - Loading Volume: 10ml
Substrate	OHP Film	- Transparent Polyethylene Phthalate (PET)
Device for Curing	UV LED Lamp	- Wavelength: 365nm singe wavelength - Energy Consumption: 4Watt
Printer	EPSON XP-202	- Piezoelectric Ink-jet Printer

Characterization of Printed Patterns

All of the measurements were performed at the room temperature. The viscosity of the UV LED polymer inks were analyzed by a viscometer (LVDV-E, BROOKFIELD). The optical properties, printability and accuracy of UV LED inkjet printing were determined using a video microscope (SOMETECH, ICS-305B). The conductivity of printed patterns was investigated with the help of 4-point probe (Loresta-GP MCP-T610, Mitsubishi Chemical). The surface structure of coated patterns were observed by a scanning electron microscope (Hitachi, TM-1000).

Inorganic Electro-Luminescence (IOEL) Process

Following the noble-hybrid printing process of creating a conductive pattern, the application of IOEL will be introduced in order to increase the magnitude of electrical conductivity. Using the silk-screen method, a layer of dielectric paste comprised of 70% $BaTiO_3$ powder and 30% binder is spread onto the substrate and heat treated at 120°C for 2 hours in a furnace. A fluorothermoplastic powder, manufactured by 3M, is made into a paste by mixing the powder and dielectric paste together. Using the same silk-screen method, a layer of fluorothermoplastic-dielectric paste is spread onto another surface of an ITO film and heat treated at 120°C for 2 hours. Once the heat treatment has been completed, the two layers of ITO films are compressed using a High Pressure Heat Press Machine$_{TM}$ for 60 seconds under a 38kg load to successfully make IOEL which increases electrical conductivity than solely using noble hybrid printing process.

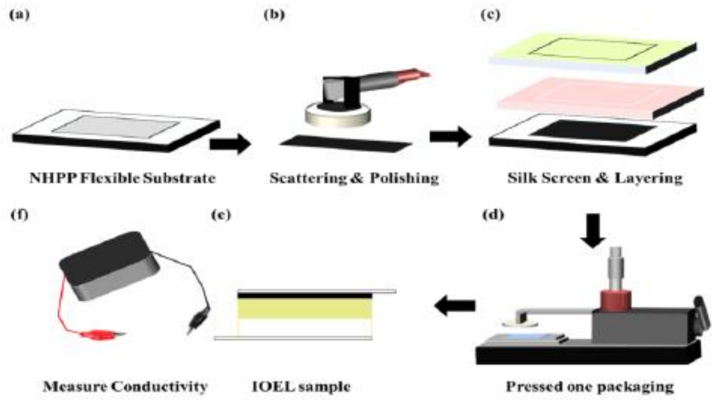


Figure2. *Schematic of IOEL process: (a) Flexible substrate with UV LED ink created by NHPP; (b) Scattering and Polishing of CNT, Carbon Black, and Graphite; (c) Silk Screen Layering Method for IOEL process; (d) Pressed packaging using Heat Press for IOEL; (e) IOEL final produt; (f) Measure Conductivity of final product*

RESULTS AND DISCUSSION

Printability and Accuracy of UV LED Polymer

Viscosity of inks affects to printability of inks and accuracy of final products. The use of methanol decreases the viscosity of UV LED polymer. Methanol is 1cPs at room temperature and viscosity drops as inclusion ratio increases. However, when Methanol is mixed with more than 40% of the volume ratio, it is conclusive that the UV LED polymer ink is not curable. Hence, adding amounts of Methanol was varied between 30 to 40 vol%. The quality of printed patterns were different with respects to change in viscosity of the UV LED polymers. The values of viscosity are depicted in table 2. The viscosity decreases by increasing methanol concentration.

Table2. *Viscosity of UV LED inks as change in concentration of UV LED polymer*

Content of LED UV Polymer (Vol%)	60	65	67	70
Viscosity (cP)	6.6 – 7.0	7.5 – 8.0	8.2 – 9.0	9.6 – 10

The width of printed patterns was measured with the help of a video microscope and the values were compared to the widths of target patterns in order to observe the accuracy of inkjet printer for the UV LED polymer as a function of polymer content or viscosity. The results are shown in figure 3. When the target width pattern is less than 200µm, the accuracy is decreased dramatically. On the other hand, accuracy improves as target width increases. This means that inkjet printing of UV LED polymers is valid when making a pattern larger than 200µm in width

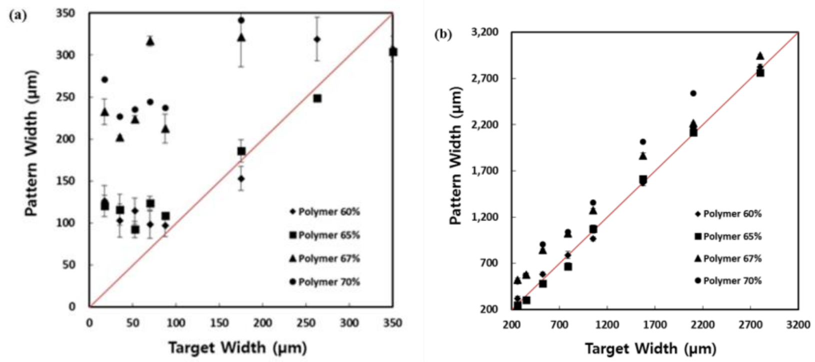


Figure3. *Minimum pattern width for accurate inkjet printing of UV LED polymer. Range of target width of patterns: (a) 0-200µm and (b) 200-300µm*

The accuracy of UV LED polymer printing is also dependent on the viscosity of the polymer. Figure 4 shows the effect of viscosity on accuracy of UV LED polymer printing by the inkjet process. As depicted in the Figure 4, when the viscosity of the ink is less than 8cP, printing accuracy is higher compared to inks having higher viscosity (>8cP). The accuracy decreases as viscosity of the inks is continuously increasing. This is relevant to surface tension of various inks with the nozzle. In other words, increase in surface tension of polymer improves continuous dispensing of inks.

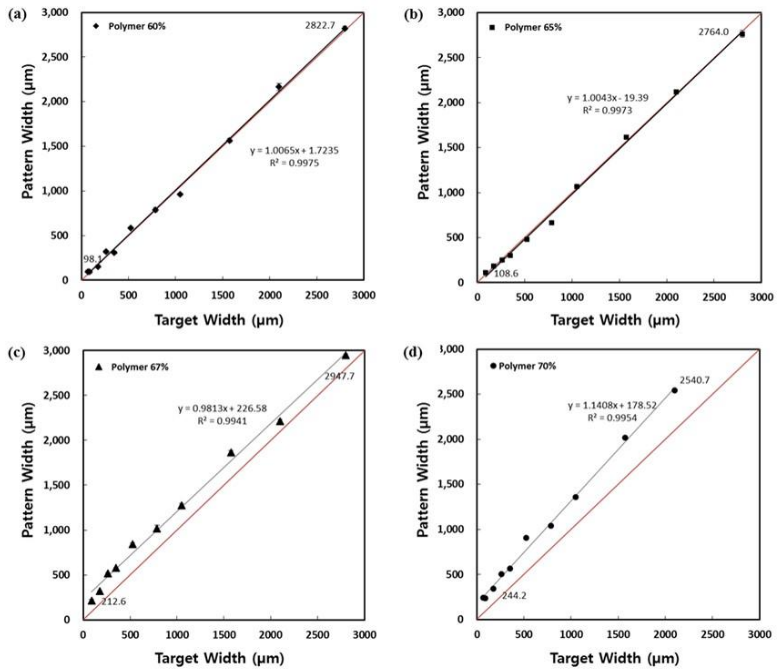


Figure4. *Accuracy of inkjet printing of UV LED polymer in terms of loading amount of polymer.*

Polymer content: (a) 60 vol%, (b) 65 vol%, (c) 67 vol%, and (d) 70 vol%

Effect of the Number of Printing Cycle on Thickness of Patterns

In this study, printing cycles were changed to observe the possibility of 3D printing of UV LED inkjet printing. The patterns were printed by 1 cycle and 20 cycle printing, respectively, using 65vol% of UV LED polymer. The study confirmed that the thickness of printed patterns is dependent on the number of printing cycles. For measurement, conductive printed patterns were cut along the printed sections and the thickness of the cross-sectional areas were measured by an optical video microscope in the absence of after-treatment (polishing). As shown in Figure 5, thickness patterns increased, however, were not proportional. Hence, the average thickness is used to represent overall thickness of the patterns. The average thickness of the patterns were 15.11μm and 136.23μm when printing is performed at cycles of 1 and 20, respectively. It is expected that those results can provide fundamental knowledge for 3D modeling using inkjet technology.

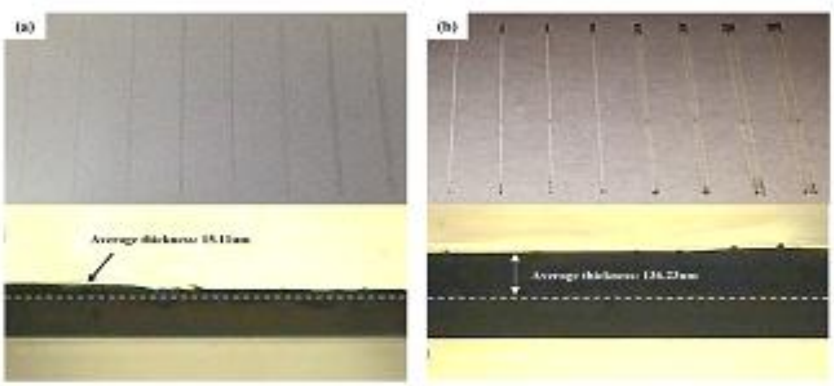


Figure5. *Thickness of printed patterns as a function of a number printing cycles: (a) 1 cycle and (b) 20 cycles*

Electrical Properties and Effects of IOEL Processes

Flexible circuits were produced by a noble-hybrid printing process. This process is categorized in four main processes: 1. inkjet printing, 2. curing, 3. polishing with conductive materials, and 4. Implementing IOEL. As shown in Table 3, the conductivity of the patterns varies between 3.11Ω-cm (ohm-cm) to 5.58Ω-cm (ohm-cm) due to changing the type of polished materials. SEM images were observed that the surface of the patterns do not have an important effect on conductivity. The results show that conductive patterns can be produced by an economical way without any time and energy consumable processes. More specific, the CNT provides better conductivity to the printed patterns when compared to carbon black and graphite. The conductive patterns were then used as an electric circuit for 3D printed electronic applications, for instance, a smartphone stand with an embedded speaker.

Table3. *Conductivity and resistivity of patterns polished with carbon group materials*

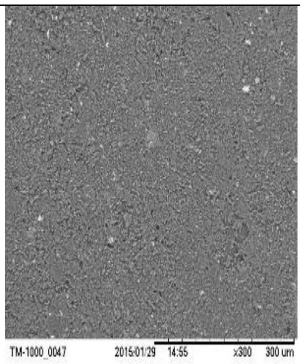
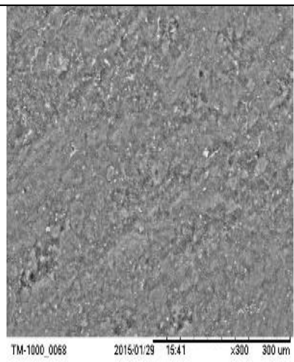
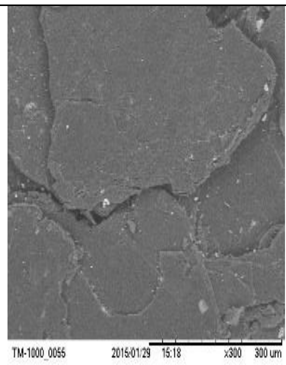
	Carbon Nano Tube	**Carbon Black**	**Graphite**
Resistivity (Ω-cm)	3.11	3.96	5.58
Conductivity (S/cm)	4.46×10^{-1}	3.00×10^{-1}	2.40×10^{-1}
SEM Images (x300)			

Figure 6 is a schematic of a cross sectional view of the different types of conductive carbon groups used throughout this experiment. Figure 6 (a) is a representation of using a Thermoplastic Polyurethane Carbon Nanotube (TPU/CNT) with a low viscosity UV LED polymer. The interface between the nanotubes is distorted because the UV LED polymer ink has encased the particle dividing the direct interface between them, thus, lowering conductivity. A PNE logo is projected using the TPU/CNT. Figure 6 (b) is a schematic of using Carbon Black powder with a low viscosity UV LED polymer ink. The interface between these particles is much higher, in that, the particles have a direct point to point interface which increases the electrical conductivity. Figure 6 (c) is a representation of the carbon nanotubes (CNT) spread over the UV LED polymer ink. The nanotubes have two types of interfacial reaction depending on the direction of all the nanotubes: the first case, they have a direct point to point interface which increases electrical conductivity, and the second case, they may have a face to face interfacial reaction where one nanotube may be lying on the surface of another which can decrease the electrical conductivity. Overall, the CNT have a majority of point to point interface which can be evaluated using the magnitude of conductivity. Using carbon black and CNT, a flexible circuit was able to be lit up with moderate to high conductivity ranges. Figure 6 (d) is a schematic of graphite being spread over the UVV LED polymer ink. Graphite is hexagonal shaped and shares a face to face interfacial reaction. Although this may seem that electrical conductivity would be increased, the fact that some plates are overlapping one another, the UV LED polymer ink can integrate itself throughout the matrix decreasing overall conductivity. A layering sequence was created by the silk screen method and an inverter was used on the IOEL samples to completely light up the flexible LED lamp.

It is evident that using an ordinary inkjet printer which does not separate the powder from the ink results in lower electrical conductivity due to the interfacial reaction between the ink and particles. The Noble Hybrid Printing Process is seen throughout this schematic, from Figure 6 (b-d), by separating the UV LED polymer ink and the carbon powders to increase electrical conductivity. This is done by printing the UV LED polymer ink first hand and embedding the carbon group powder(s) by the process of polishing, introducing a nobility in inkjet printing.

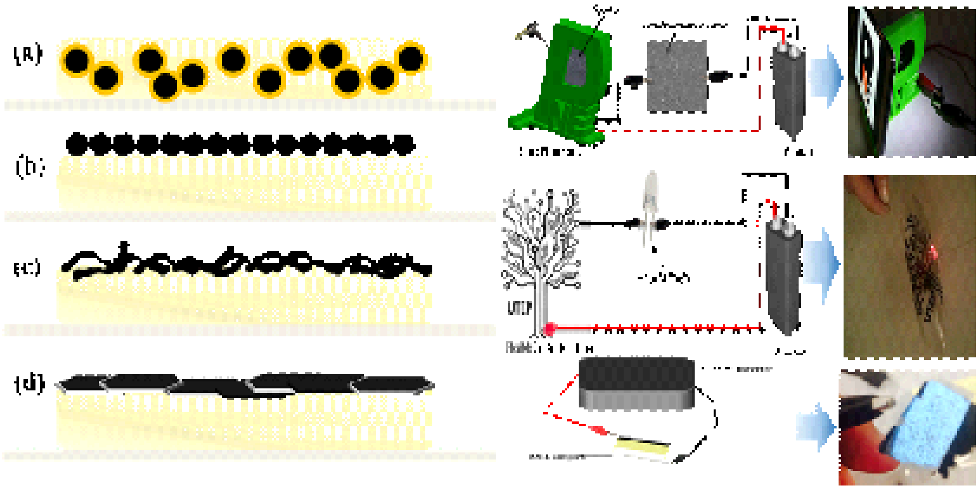


Figure6. *Schematic of Cross-Sectional view of the particles interaction and Application of Printed Circuits:*

(a) Schematic of TPU/CNT with UV LED polymer ink (left), PNE's smartphone stand with embedded speaker (right)

(b) Schematic of Carbon Black with UV LED polymer ink (left), Flexible circuit device with LED light (right)

(c) Schematic of CNT with UV LED polymer ink (left), Flexible circuit device with LED light (right)

(d) Schematic of Graphite with UV LED polymer ink (left), Flexible LED lamp (right)

CONCLUSION

A UV LED polymer and a 4watt LED UV lamp have been used to fabricate flexible conductive patterns by using an ordinary desktop inkjet printer by implementing a noble hybrid printing process. The NHPP separates the conductive powder from the UV LED polymer in the cartridge. It is observed that 7.5-8.0cP of ink is the most efficient viscosity to print UV LED polymer inks on a flexible PET substrate. The accuracy of inkjet printing was decreased remarkably when width patterns were less than 200µm. Inkjet printing of UV LED polymer is useful when drawing relatively larger patterns (width≥200µm).

The pattern thickness was controlled by increasing the number of printing cycles. When UV LED ink was printed with 1cycle versus 20cycles, the pattern thickness was 15.11µm versus 136.23µm, respectively. It is believed that UV LED inkjet printing can be used for 3D additive manufacturing methods. Despite its capability, further research should be performed to optimize printing parameters; the results are enough to suggest the possibility of 3D printing by use of inkjet printing with UV LED curable polymers and an ordinary inkjet printer.

It is also confirmed that flexible electronic circuits can be produced by inkjet printing of UV LED polymers and polishing of Carbon group materials, called to noble hybrid printing process (NHPP), at room temperature. The conductive patterns were produced using CNT, carbon black, and graphite which sustain an interfacial reaction such as: face to face, point to point, and face to face, respectively. These samples have an electrical conductivity range from 2.40×10^{-1} to 4.46×10^{-1} S/cm. By implementing the inorganic electro-luminescence process to create higher conductivity, the overall conductivity greatly improved to 3.154×10 S/cm.

An electrical device comprised of a speaker embedded smartphone stand and a transparent flexible LED circuit were produced without using complex processes such as sintering and etching. The processes which were performed in this study improved energy efficiency by using UV LED curable polymers under a 4watt LED UV lamp. In addition, the arrangement method of particles, with affinity to the ink by use of polishing significantly increased electrical conductivity because it prevents surfactants or binders from entering between the particles due to the interfacial reactions between the conductive particles. As a result, the noble-hybrid printing process implemented with inorganic electro-luminescence proposed in this research can be a more cost effective and time efficient method to manufacture flexible electronic circuits.

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