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Design and Fabrication of Infusion Pump to Control the Flow Rate of Solution for Synthesis of Zinc Oxide Nanomaterial

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Abstract

Currently, synthesis of nanomaterials requires different types of glassware like burettes and pipettes to control the flow rate of precursors during chemical reactions. Contemporary methods are found to be time consuming and inefficient to deliver the precise volume of precursor solution at a particular flow rate into the reaction mixture. To make precise delivery of fluids at a fixed flow rate, the design and development of a customized infusion pump is reported in this work. This includes the design of the system, 3D-printing of the parts, assembly of the spare parts and automation of the infusion pump using microcontrollers. Further, the synthesis of zinc oxide nanomaterial by conventional methodology and using the designed prototype has been compared and reported.

Keywords: Additive manufacturing, Automation, Embedded system, Infusion pump, Nanomaterial synthesis

1. INTRODUCTION

Synthesis of nanomaterials requires many measuring equipment, analytical equipment, glassware, and instruments. The reagents used in the reactions require precise measuring tools and glassware. These tools must deliver reagents into the reaction mixture at a constant rate in order to achieve the stable uniform product. This is specifically important in case of controlled experimental setups. For instance, CuO and ZnO nanomaterials have been synthesized in TSSA (Teflon-lined Stainless Steel Autoclave) under the controlled environment [1, 2]. Chemical reduction and sonication together used as a physiochemical process for the synthesis of tin oxide nanoparticles was reported for gas sensing layer [3]. Measuring instruments like pipette and other analytical instruments precisely measure the quantity of reagents required for the experiments. In most of these instruments flow rate of the fluid is not accurate. To overcome this issue and to synthesize uniform-sized nanoparticles, fabrication of a novel infusion pump is reported in this paper. The main goal is to maintain the flow rate [4] of the precursor solutions in order to synthesize uniformly sized nanoparticles. The design is inspired from infusion pumps, which are used in neonatal intensive care units for newborn babies and critical patients and lab-on-a-chip microfluidic system [5-7]. These pumps are generally used for providing nutrients and medicine in the liquid form. This allows minimum damage to blood vessels owing to the pressure associated with traditional methods like intravenous and syringe based delivery mechanism [8].

The work reported here includes the design of the physical structure of the infusion pump. The components are designed using Fusion 360 and fabricated by the 3D printer. The flow rate of the fluids is controlled using stepper motors. The complete operation of the pump is controlled by using ATmega328P (Arduino Nano) microcontroller and Easy Driver [9].

1.1 Infusion Pump

Infusion pumps are medical devices which facilitates the controlled delivery of fluids [5, 6], including nutrients and medications into the patient body [8]. Infusion pump can accurately and precisely deliver the precursor in the reaction mixture to get uniformity in size and shape of the nanomaterials. The schematic diagram of the infusion pump has shown in Figure 1. The Infusion pump consists of a syringe for delivery of fluids, microcontroller and stepper motor to control the piston movement of the syringe and LCD display. 3D printed customized infusion pump reduced the manufacturing cost of the industrial infusion pump [10,11].

1.2 3D Printing Technology

The 3D printing technology is additive manufacturing technology used to develop the rapid prototype. It helps in minimizing the material to be used, processing time and cost of manufacturing product [11,12]. Currently, additive manufacturing technology i.e., 3D printing has been used often to produce assorted tools to be used in laboratories and manufacturing processes. 3D printing can create precise 3D objects using layer by layer process called Fused Deposition Modelling (FDM). In the future, it may replace traditional subtractive manufacturing processes like grinding, bending, forging, molding, cutting, gluing, welding and assembling. The 3D printing make uses CAD/CAM software to design the object and slicing software like cura, slicer etc. can slice the object into the cross-sectional layers and convert the file into a Stereo-lithographic file (.STL). The software like SolidWorks, Opens SCAD, FreeCAD, 3DS MAX, AutoCAD, Autodesk Maya, Fusion 360, Blender, SketchUp, etc. are available to be used in 3D designing [11,13].

At present, many polymeric filament materials have been used for printing by considering their properties enlisted in **Table 1.**

Table 1: Commonly used materials in 3D printing [11,12]

S. No.	Material	Extruded temperature (°C)	Bed temperature (°C)	Advantage	Disadvantage	Application
1	Nylon	220 to 270	70 to 90	Strong, Durable, and Versatile, Translucent surface	Sensitive with moisture	Hinges, Functionally strong parts
2	Polypropylene (PP)	220 to 250	85 to 100	Flexible and Not chemically reactive	Very hygroscopic, enclose environment	Flexible hinges, Medical devices, and Chemistry equipment.
3	PETG	230 to 250	75 to 90	Strength is much higher than PLA, durable, flexible and Heat resistant	Not biodegradable,	Mechanical parts and Snap fit enclosures
4	Polycarbonate (PC)	260 to 310	80 to 120	Strong and very resistant to impact, Heat resistant, when cool bend without cracking.	Malleable	Bullet-proof glass
5	PVA	160 to 170	40 to 45	Biodegradable, Recyclable, Non toxic., Water soluble,	Expensive Deterioration due to air Moisture, Special storage necessary	Used to print support materials,
6	PLA	170 to 220	45 to 60	Biodegradable plastics, Nontoxic, No heated print bed necessary, High print speed and resolution, Less warping or shrinking issues.	Slow cooling down, Low heat resistance, Easier to break then ABS, Needs thicker walls then ABS.	Cosmetic prints, Prototypes, Toys, Packaging applications
7	ABS	215 to 250	80 to 110	Smooth finish, Heat resistant, Solidifies quickly, Durable, flexible and Difficult to break, Ideal for mechanical parts.	Petroleum-based, Non biodegradable, Heated print bed necessary, Non biodegradable, Heated print bed necessary, Deterioration through sunlight, toxic.	Interlocking parts like Gears, parts exposed to UV and heat like a car cup holder, or prototyping

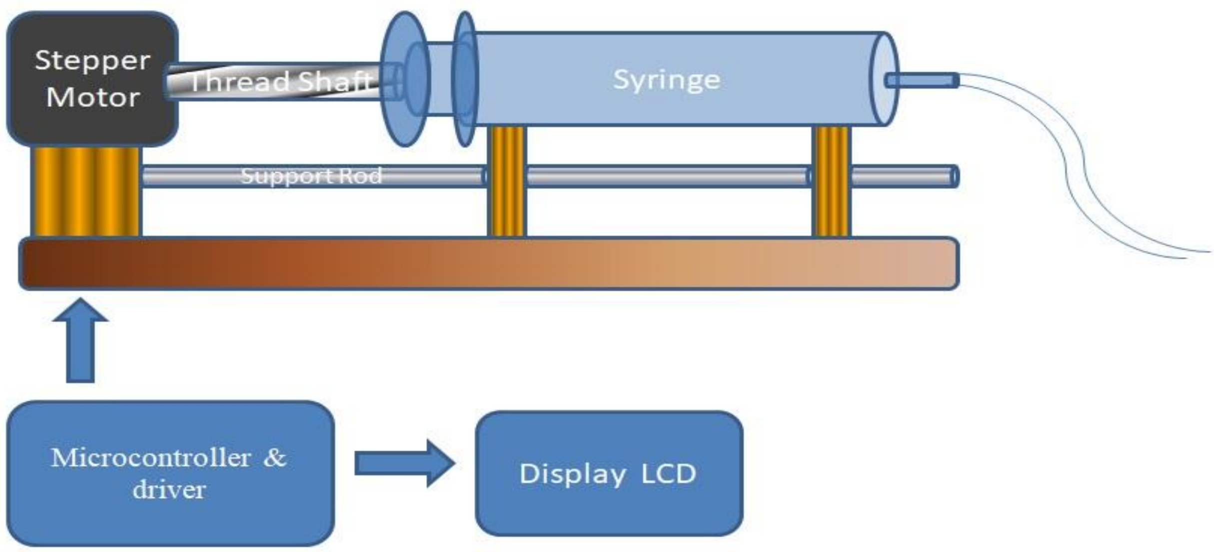


Figure 1: Schematic representation of the infusion pump

2. MATERIALS AND METHODS

2.1 Design and 3D Printing of Parts of Infusion Pump

The proposed model was designed in open source cloud-based Autodesk Fusion360 tool which allows the user to have easy access for functions and fabrication of objects. Fusion360 is based on solid modelling technique, which allows the user to simulate the design, analysis, manufacturing and rapid prototyping of 3D objects [11,12]. Designed files have been imported as a Stereo Lithographic file (.STL), which slices the surface of the designed object into the number of cross-sectional layers for fabrication. The filament materials used to print the components are Polylactic acid (PLA) and Acrylonitrile Butadiene Styrene (ABS) [13].

2.2 Fabrication of Infusion Pump

All the 3D printed parts were assembled on shaft and motor. The nozzle, barrel, and plunger of the syringe were fixed with 3D printed parts of the infusion pump. The 3D printed customized infusion pump can be used efficiently to replace conventional synthesis methods of nanomaterials, reduces manual errors, time, manpower etc.

2.3 Interface of Embedded Control System with 3D Printed Parts of Infusion Pump

Embedded Control system has been designed to automate the infusion pump by controlling the movement of the syringe plunger. The ATmega328P (Arduino Nano) microcontroller and Easy Driver was used to control the speed of the stepper motor.

2.4 Synthesis and Characterization of ZnO Nanomaterials by Burette and Customized 3-D Printed Infusion Pump method

2.4.1 Materials

Zinc Chloride ($ZnCl_2$, purity ≤ 98%) and Sodium Hydroxide (NaOH, purity ≤ 98%) Sisco Research Laboratories Ltd-Mumbai. Chemicals were used without further modifications.

2.4.2 Method

ZnO nanomaterials were synthesized by chemo-thermal method through both the instruments in atmospheric pressure at constant temperature. Initially, 1:2 proportions of metal salt (Zinc Chloride- $ZnCl_2$) and reducing agent (Sodium Hydroxide- NaOH) was dissolved separately in 25 ml of DI water. The $ZnCl_2$ solution was stirring continuously at 120 ºC on the magnetic stirrer for 3 hours. NaOH was added dropwise using burette and infusion pump into the $ZnCl_2$ solution in different experimental set-ups. The white color precipitate was obtained by centrifugation of solution at 10000 rpm for 15 minutes. The pellet was washed twice with DI water and dried at 60 ºC for 6 hours. Further, the powder was kept for calcination at 400 ºC for 4 hours and resultant material used for characterization.

$$ZnCl_2 + 2NaOH \rightarrow Zn(OH)_2 + 2NaCl$$

$$Zn(OH)_2 \rightarrow ZnO + H_2O$$

3. RESULT AND DISCUSSION

3.1 Design and Fabrication of Infusion Pump

The parts of the customized infusion pump were designed by Fusion 360 tool and printed on PRUSA i3 (Figure 2). Infusion pump assembly contains six 3D printed components, two steel shafts, a piston shaft, syringe and stepper motor with the embedded system. A syringe has a nozzle, barrel, and plunger.

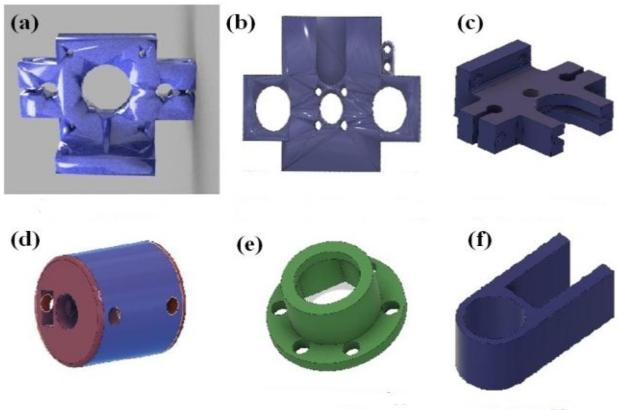


Figure 2: 3D printed infusion pump parts (a) Motor holder, (b) Syringe plunger holder, (c) syringe holder, (d) shaft coupling coupler, (e) shaft connector coupling and (f) switch holder

gets attached to 3D printed objects where, motor holder holds stepper motor, shaft connected coupling attached with stepper motor shaft which then connected to piston shaft. Syringe plunger holder and shaft connector coupler together hold piston shaft. Syringe barrel holder holds barrel at the front end whereas, the syringe plunger is mounted on syringe plunger holder at the backend. Syringe plunger holder rotates with the stepper motor. Steel shaft was used for mechanical support and it provides balance to the system. Switch holder plays a role to stop the syringe plunger when it moves backward. Switch holder and a stepper motor connected with the embedded system. (Figure 3)

3.2 Device Interface

Arduino Nano has a Mini-B USB cable port for serial communication, which allows to interface with the computer system. The Easy Driver designed with A3967 IC which drives a bipolar- full, half, quarter, eighth step modes and 4, 6 and 8-wire stepper motors. It can either work with 3.3 V or 5 V. It has the capability to drive bipolar stepper motors between 150 mA to 700 mA per phase [9]. Stepper motor was controlled by Easy driver IC as programmed in the microcontroller. The flow rate of the fluid is displayed on the LCD. The amount of the fluid and time are the two user input that process the program to control the speed of the stepper motor. Reset button in the system can be used to stop the process whenever required. The controller has been programmed for the particular flow rate of the precursor and it also has to the programmable facility for others material concentration and viscosity of a precursor. The experimental setup has shown in **Figure** 4.

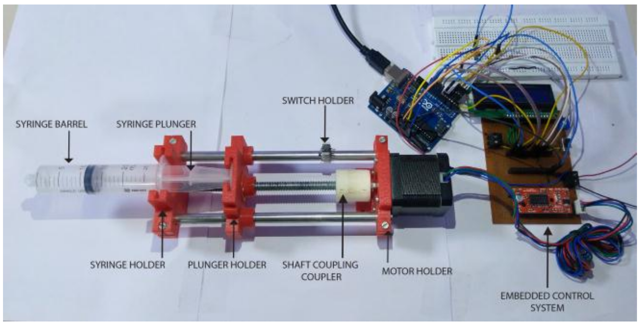


Figure 3: 3D printed infusion pump

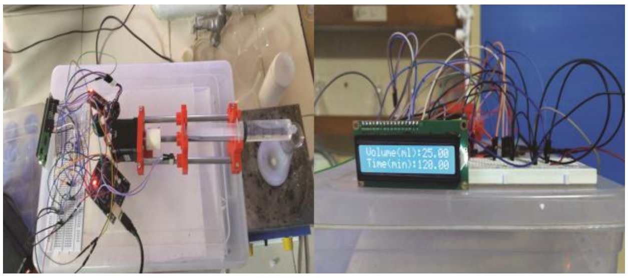


Figure 4: Experimental setup for synthesis of ZnO

3.3 Characterization of ZnO nanomaterials

3.3.1. UV-Vis Spectroscopy

The absorption spectra of synthesized ZnO nanomaterial by infusion pump and burette setup have shown in Figure 5. The maximum absorption (λ max) of ZnO at 300 nm and 307 nm was recorded for samples made by infusion pump and burette setup, respectively. This wide absorption of UV visible spectroscopy results for ZnO NPs was reported in the literature [14, 15]. This result indicates that the optical properties of both the samples were same.

3.3.2. XRD Analysis

The synthesized ZnO powder using the infusion pump and burette setup was characterized by X-ray Diffraction (XRD) to identify the phase composition and crystalline structure. The peaks given in Figure 6 represent the hexagonal wurtzite crystalline structure of ZnO nanomaterials. The planes in Figure 6 (1 0 0), (0 0 2), (1 0 1), (1 0 2), (1 1 0), (1 0 3), (2 0 0), (1 1 2), (2 0 1), (0 0 4), (2 0 2) are compared and confirmed with the standard XRD database available in materials project ID No: mp-2133 and other reported research [14-18].

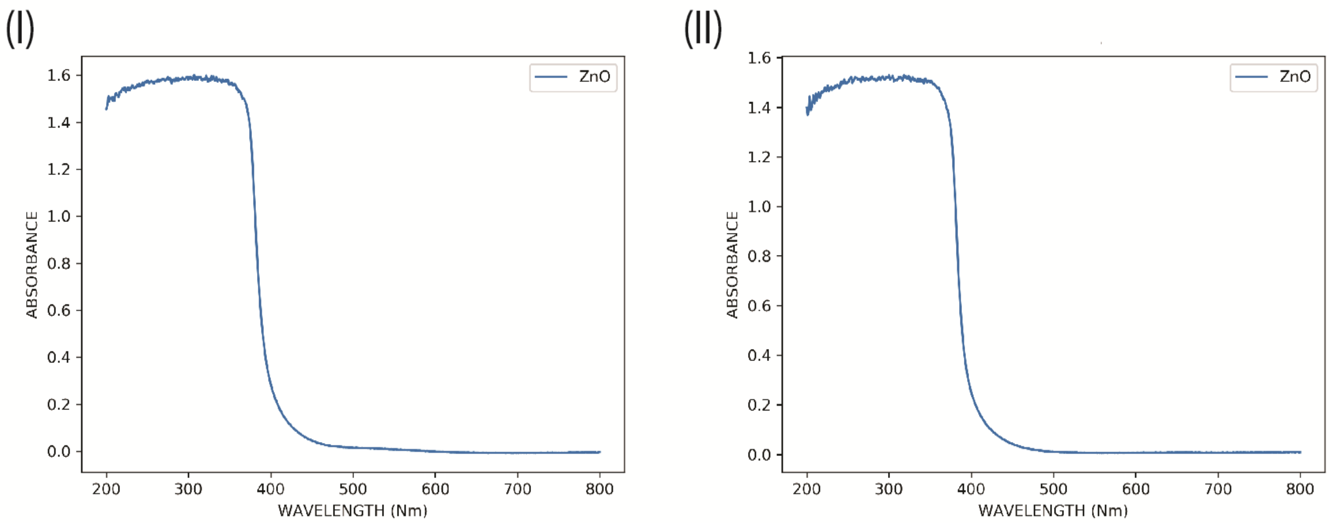


Figure 5: UV-Vis Spectra of ZnO nanomaterials synthesized using (I) Burette setup and (II) Infusion pump

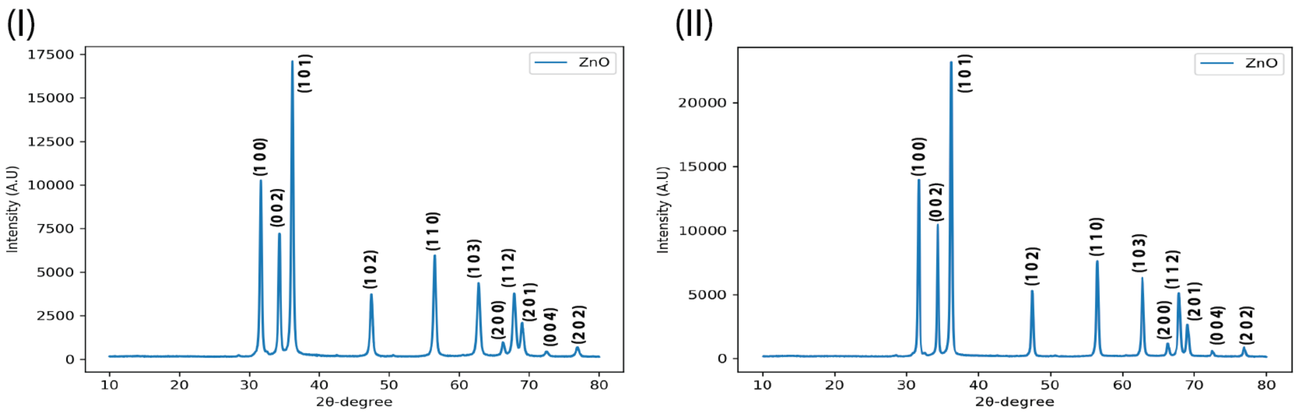


Figure 6: XRD of ZnO nanomaterials synthesized using (I) Burette setup and **(II)** Infusion pump

Sharp line and narrow width in Figure 6 signifies highly crystalline nature of the synthesized ZnO nanomaterial. The samples of the infusion pump and burette showed similar XRD results, however, the intensity was found to be high in the sample made by the infusion pump.

3.3.3. FESEM with EDS analysis

The morphological properties of ZnO nanomaterial have been studied using Field Emission Scanning Electron Microscope and Energy Dispersive Spectroscopy (EDS). FESEM images of the sample by burette setup showing non-uniform size distribution of spindle shape nanomaterials found in the range of 165 - 290 nm. Similarly, ZnO sample by infusion pump studied under FESEM showing uniform size distribution of pencil-like hexagonal rods found in the range of 250- 295 nm. N.U saidin et al., observed pencil like ZnO nanorods on Si -substrate with average size ~598 nm. [19]. Similar morphology of ZnO nanomaterial was achieved by young gan et al., however, the size of the nanomaterial obtained in the present work was four times smaller than the work reported [14]. Therefore, the proposed synthesis method using customized 3D- printed infusion pump for controlling the flow rate of reducing agent to obtain ZnO nanomaterials with specific size and shape and hence, can be used for scaling process. Synthesis method with and without controlled flow rate of reducing agent produced ZnO nanomaterials with different morphology like pencil-like nanorods (Figure 7 (a)) and spindle shape (Figure 7 (b)) with the difference in average size distribution and uniformity.

Figure 8 represents the elemental analysis of ZnO nanomaterial synthesized using burette and infusion pump. The EDS results indicate the presence of Zinc and Oxygen element in synthesized powder without any impurities. Figure 9 (a) and (b) shows the elemental mapping of ZnO nanomaterials synthesized using burette and infusion pump respectively. The zinc and oxygen have shown in red and green dots, respectively, in ZnO nanomaterials (Figure 9 (a) (I) and (b) (I)). Further, Figure 9 (a) (II, III) and (b) (II, III)) reveals the distribution of oxygen and zinc, respectively, in the material.

4. CONCLUSION

The overall conclusion of present work can be drawn as controlled release of the reducing agent through an automated 3D printed infusion pump for the synthesis of ZnO nanomaterials has found more precise compared to manual burette method. Infusion pump provided pencil like hexagonal ZnO nanorods with uniform size distribution. This indicates the controlled flow rate of reducing agent plays a vital role to get the uniformity in the morphology of the nanomaterials. Further, the automated IoT based system of infusion pump can be used for scaling up various chemical and biochemical reactions for controlling the flow rate of precursors as per the demand.

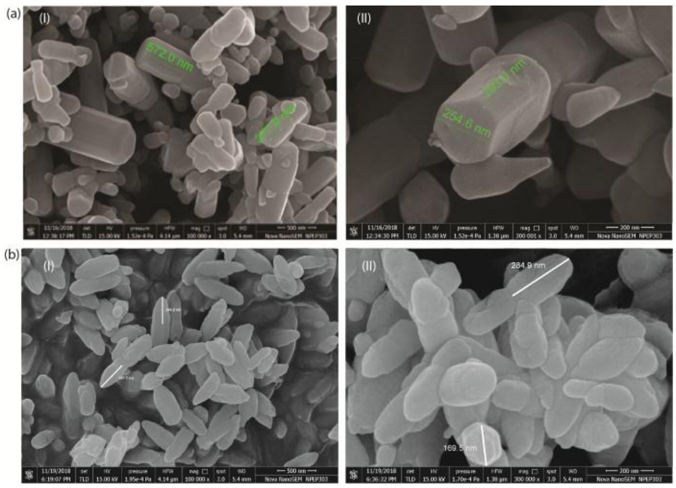


Figure 7: FESEM images of ZnO nanomaterials synthesized by using (a) 3D printed infusion pump; (I) lower and (II) higher magnification images of pencil like hexagonal ZnO nanorods found to have size in the range of 250 – 295 nm with uniform shape and size distribution. **(b)** Burette setup; (I) lower and (II) higher magnification images of spindle shaped ZnO nanostructures found to have size in the range of 165 – 290 nm with different size and shapes.

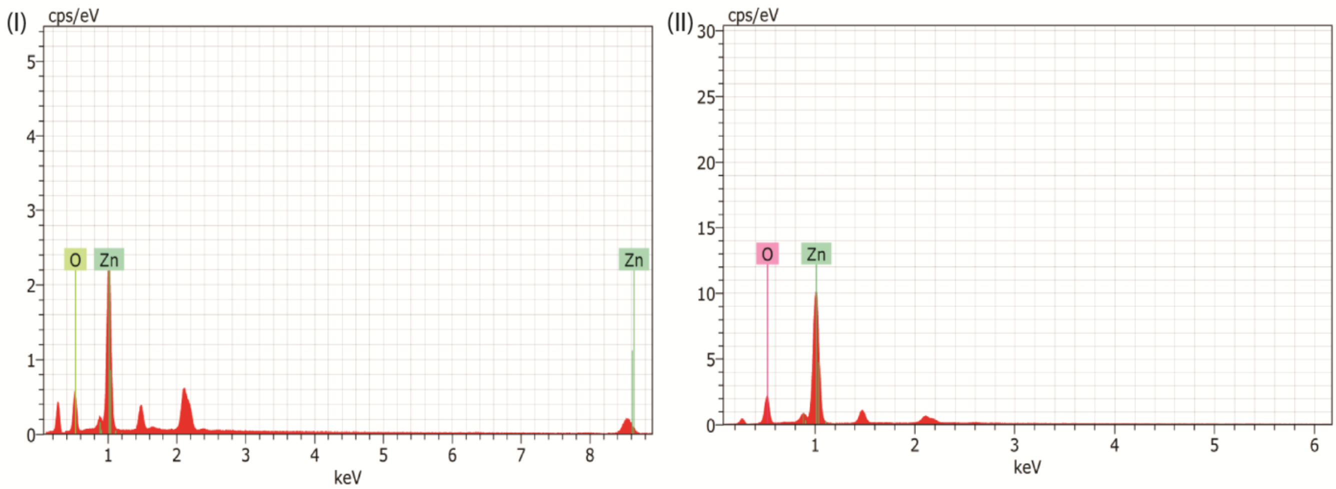
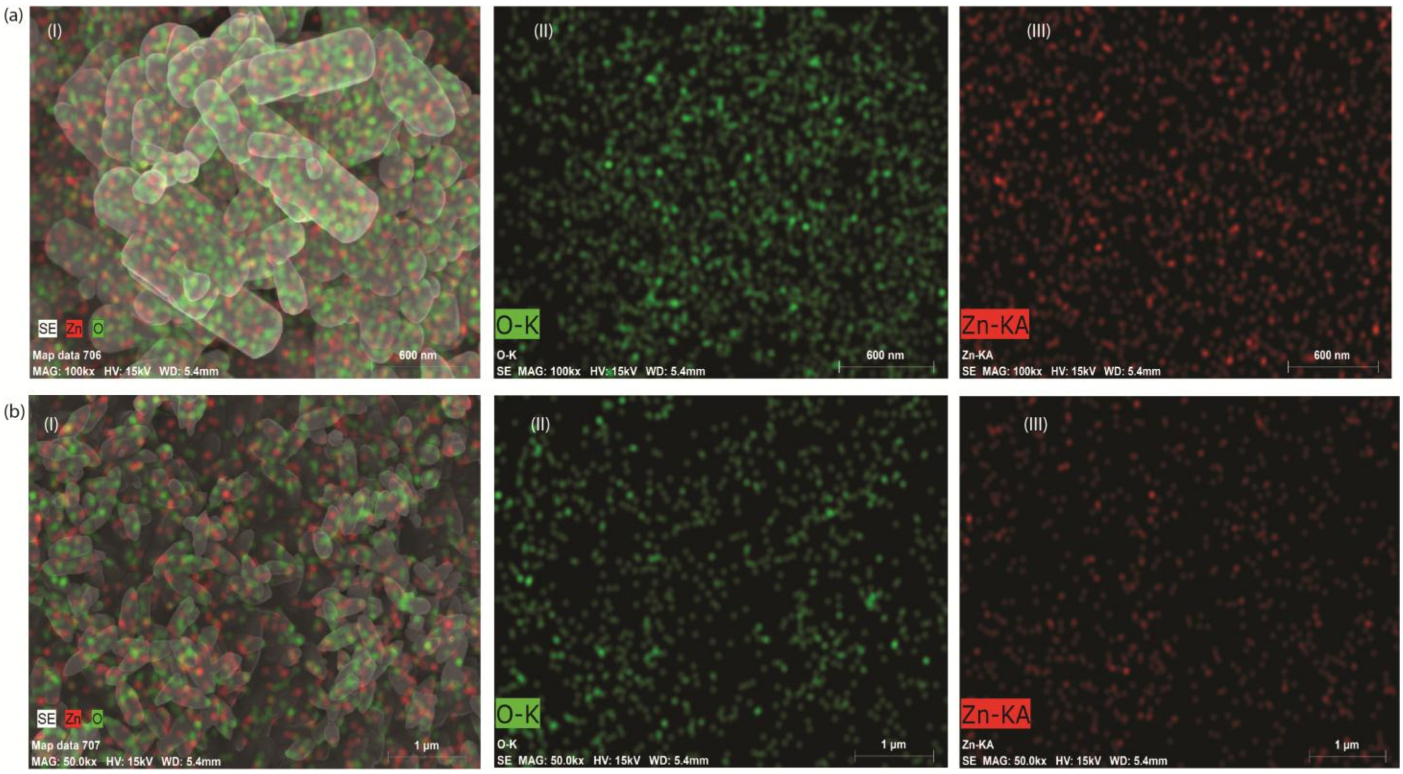


Figure 8: Elemental analysis by EDS of ZnO nanomaterials synthesized using (I) Burette setup and (II) 3D printed infusion pump



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