

Implementation of an IoT-based Dip Coating System for The Fabrication of ZnO Thin Film

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Abstract

An Internet of Things (IoT) based dip coating system was developed in this research to produce a uniform thin film. The dip-coater consisted of an IoT-based microcontroller (ESP32), a Nema 17 stepper motor, and certain low-cost recyclable elements. The range discovered for dipping and withdrawal speed was 1 to 100 mm/min without disturbances caused by vibrations. The total expenditure for the development of the dip coater based on IoT was below one hundred dollars. Thin films of ZnO (Zinc Oxide) were successfully deposited on glass substrates. Different dip coater performances were investigated and compared with the designed IoT-based dip coater. The dipping process was performed 5 times with the same concentrations of 1M to achieve the desired film thickness. X-ray diffraction (XRD) analysis revealed that ZnO possesses the hexagonal Wurtzite crystal structure. As for the inspection by SEM (Scanning Electron Microscope), it was observed that uniform layers were obtained. The chemical properties were also analyzed by EDS (Energy-dispersive X-ray Spectroscopy). Over 80% optical transmittance was demonstrated by the thin films in the wavelength ranging from 380 to 800 nm. For scientist's incapable of purchasing expensive equipment that accomplishes the same goal and delivers the same outcomes, this inexpensive equipment supports scientific advancement. In the nanoscale range, the production of thin films with uniform thickness revealed the effectiveness of the dip-coater.

Keywords: Thin Film, IoT, Dip Coater, Sol-Gel, ZnO, ESP32, Low-Cost

1. Introduction

Dip coating is a simple method for coating a substrate in a laboratory. A dip coater is utilized for the preparation of thin film. They have been utilized across a broad range of applications [1]. Numerous industries, including electronics, optics, and photocatalysis, employ the thin films made using the dip-coating process [2]. It has been reported that the thickness of ZnO films that are formed from the dip coating process is greatly impacted by both the dipping and withdrawing speeds. To maintain a uniform film thickness, it is necessary for these rates to remain constant [3]. Nevertheless, in conventional sol-gel procedures [4-6], the dipping and withdrawing processes are carried out manually, causing difficulties in maintaining a uniform and smooth dipping and withdrawing rate. An effective approach to this difficulty is implementing an automated dipping and withdrawing system [7,8].

In many laboratories, dip-coaters are an especially popular method of preparing thin films because they produce smooth, uniform layers. A dip coater may be used to produce thin coatings with the proper thickness, uniformity, and repeatability [9]. Dip coater can be employed for applying homogeneous and consistent coatings to a substrate [10]. The commercially available dip coating system is needed for producing thin films of higher quality [11]. The cost of accurate dip coaters is very high. The market's most competitive dip coaters cost lies about \$3000 [12]. To avoid paying such expensive costs, a number of entities have developed their own dip coaters. For instance, Castillo-Vilcatoma et al. used salvaged components to construct a dip coater for less than \$100. Their device achieved a dipping speed range of 0.1–6 mm/s by using an Arduino board and software. The machine's wooden frame shortens this dip coater's lifetime and durability [13]. Hossain et al. developed a dip coater with a stepper motor that contains a single substrate holder.

Multiple substrates can be deposited at a time by incorporating multiple substrate holders [14]. To prevent the substrate from dust, Dunlap et al. created a dip coating machine with an acrylic shell and touch screen display. The machine has three preprogrammed speed settings: slow (half mm/s), medium (one mm/s), and rapid (five mm/s). A large range of speed variation is required for versatile operation [10]. Yepuri et al. constructed a dip-coater employing a linear actuator that ensures minimal vibration during deposition and a fixed speed of 3 mm/s [15]. The fixed limited speed is not desirable for versatile operation.

There are a wide variety of dip coater designs available on the market. Most of the time, they

come equipped with a stepper motor, which enables a movement that is both highly accurate and controllable. A digital control panel that is designed to enable customizable path distance depending on the size of the sample, the ability to regulate the speed for both the dipping and withdrawal rate of the substrate holder, as well as control over the total quantity of cycles done are among the factors that contribute to the most successful sales [10].

Utilizing IoT-based microcontroller boards, like ESP32, to create inexpensive laboratory apparatus has garnered significant interest [16]. Arduino often comes with inexpensive costs for scientific instruments and is available under permissive licenses, allowing for free redistribution and modification by everyone [17]. Comparing Arduino to other equipment, it is easier to program and manipulate [13].

In light of the aforementioned factors, the primary emphasis of this work is to design and implement a low-cost, IoT-based dip-coating system containing multiple substrate holders that was made with an ESP32 microcontroller and a Nema 17 stepper motor that had

been recycled from a printing device. Since dip coating is a longer process, hence IoT features are included in the dip coating system to real-time monitoring the process through a smartphone app. User can perform their own other tasks when the dip coating is running. After completing the coating, the system will provide a message to the smartphone app user and this is our future goal. The app can store programs for future use. Hence, the user does not need to input the same operational information for repeated deposition. It is difficult to input operational information manually by matrix keypad whereas through a smartphone app, users can experience a very easy and comfortable interface in order to input operational information. Finally, ZnO film was produced onto bare glass substrates to generate high-quality thin films, confirming the performance of our homemade IoT-based dip coating system and its use in this research. The benefits of the manufactured dip coating system used in this study are its low-cost, user-friendly interface and its easy modification of the thin film characteristics and thickness.

2. Materials and Methods

2.1. Designing and Development of an IoT Based Dip Coating System

The block diagram of IoT based automatic dip coater system is displayed in Figure 1. A DC power supply unit (Adapter-9V, 2A) rectifies a 230V AC supply into 9V and a buck converter converts this 9V DC to 5V DC, and this is subsequently distributed to various components of the system. However, the motor driver uses 9V DC directly from the adapter output. This

work uses a 2-phase, 3.6-volt bipolar stepper motor with a $1.8^\circ \pm 0.09^\circ$ step angle, which means that it completes a revolution in 200 steps. An IC known as the TMC2209 has been utilized as the motor driver.

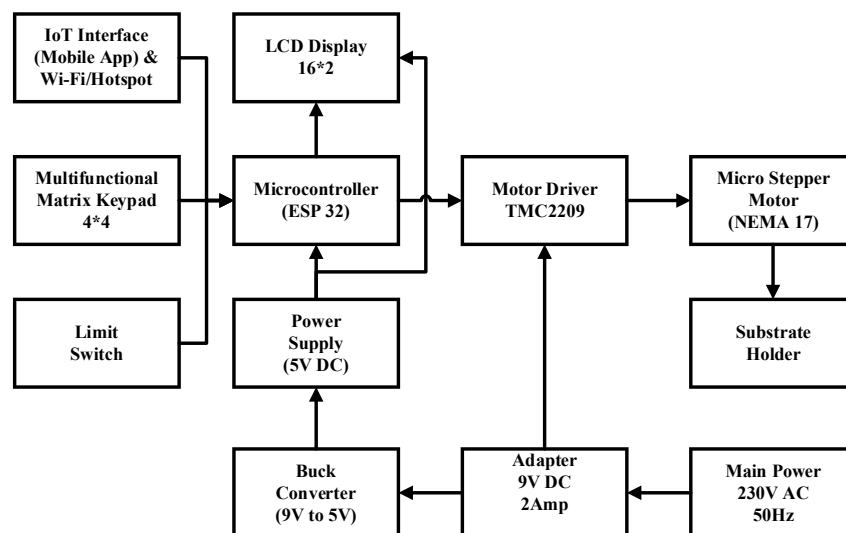


Figure 1: Operational Block Diagram of Iot-Based Sol-Gel System

The display device is a 16 by 2 LCD display and a multifunctional 4*4 matrix keypad has been used as the input device. Along with the previous features, a smartphone app interface unit can be connected through the Wi-Fi hotspot as an IoT device for inputting data to the microprocessor for desired operation. The motor driver then operates the stepper motor after receiving control signals from the microcontroller once the value has been entered. The stepper motor rotates in either an anticlockwise or clockwise direction, depending on the particular control signal. Consequently, the holder of the substrate also moves in the up and down directions, successfully accomplishing the action of dipping and withdrawing away from the solution.

Figure 2(a) and Figure 2(b) depict the home-made sol-gel device. The stepper motor's circular motion is converted into linear motion using a composite system of shaft coupler, motor shaft, threaded rod, ball bearing, smooth rod, anti-backlash T8 Lead screw nut, and a substrate holder. Every controlling function had been handled by an ESP 32 equipped with a microcontroller unit. Two screws fix multiple substrate holders to the middle part of the Aluminium strip. The front view of the substrate holder with a substrate is depicted in Figure 4(a), and the side view of the substrate holder with multiple substrates is shown in Figure 4(b). Two metal strips and two screws for holding the substrate that make up the substrate holder. The two substrate bases are separated by 0.5 cm, and they are both composed of Aluminium sheets.

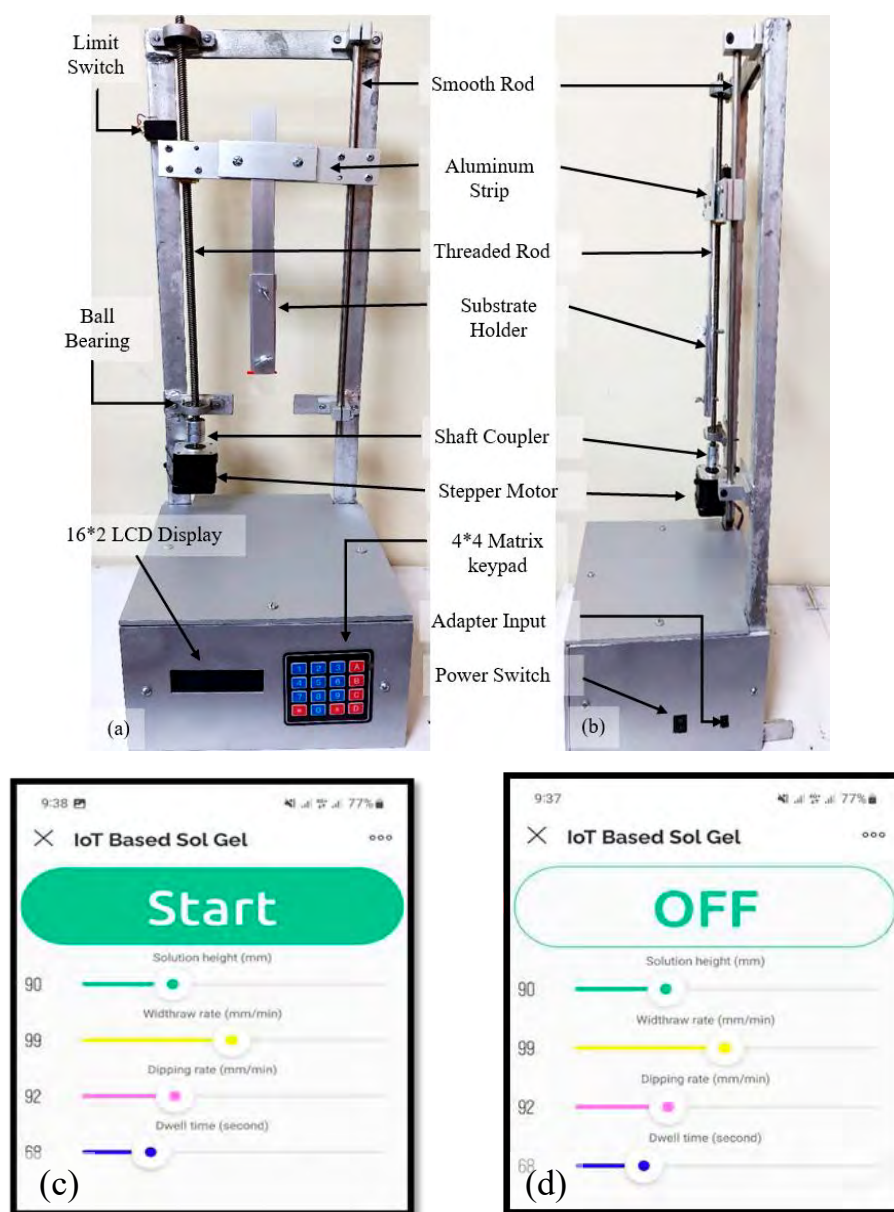


Figure 2: (a) Front-view, (b) Side-view of the IoT-based Sol-gel Device, and Smartphone App interface with (c) Start, and (d) Off control

Both two screw is used to lock the substrate to the first and second base by rotating it in a clockwise direction after the substrate has been positioned in the space between the two bases. Re-rotate the holding screw counterclockwise to extract the substrate from the base. The LCD display and matrix keypad are attached to the front side of the instrument box which is shown in Figure 3(a). On the other hand, the power switch and adapter input pin are attached to the right side of the instrument box which is also shown in Figure 3(b). The IoT-based app interface is shown in Figure 2(c) and Figure 2(d). All controlling functions such as dipping length, speed, dwell time, etc. can be inserted into the microcontroller through the app interface as well as on the device matrix keypad.

A beaker base is mechanically tied to the lower portion of the bar of iron which is about 4.5 inches above the surface of the ground and is fixed. Moreover, the beaker base is composed of an 8mm PVC sheet.

A small micro limit switch (1A, 125V) coupled to the ESP32 was utilized to determine the maximum height of the deposition route accessible for the arrangement made up of the iron bar support and the aluminium bar. The substrate holder is positioned at the top of the iron bar known as the home position when the dip-coater is switched on. The user may then specify the required settings after that.

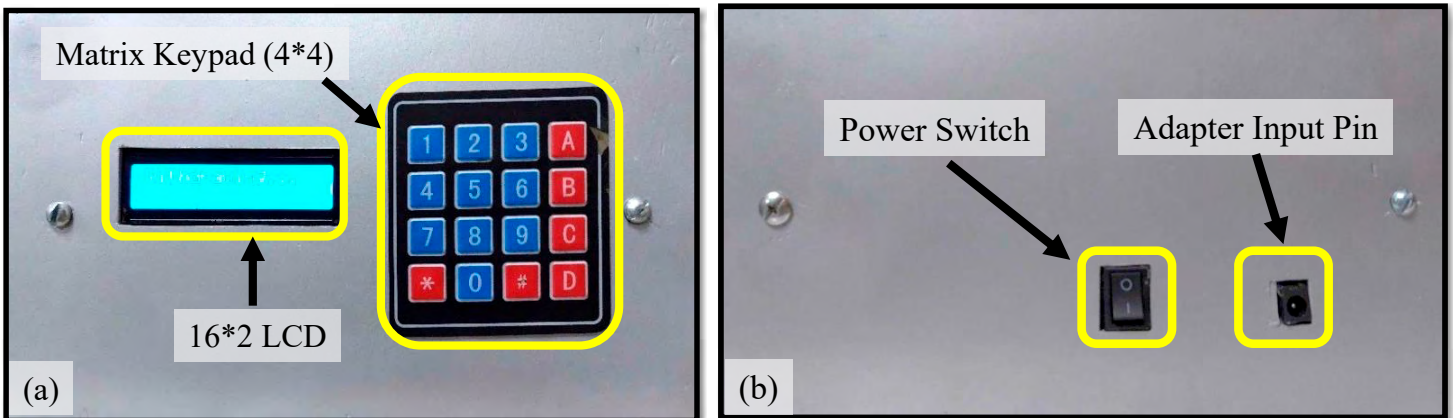


Figure 3: (a) Front Side And (b) Right Side View Of The Dip Coater's Instrument Box

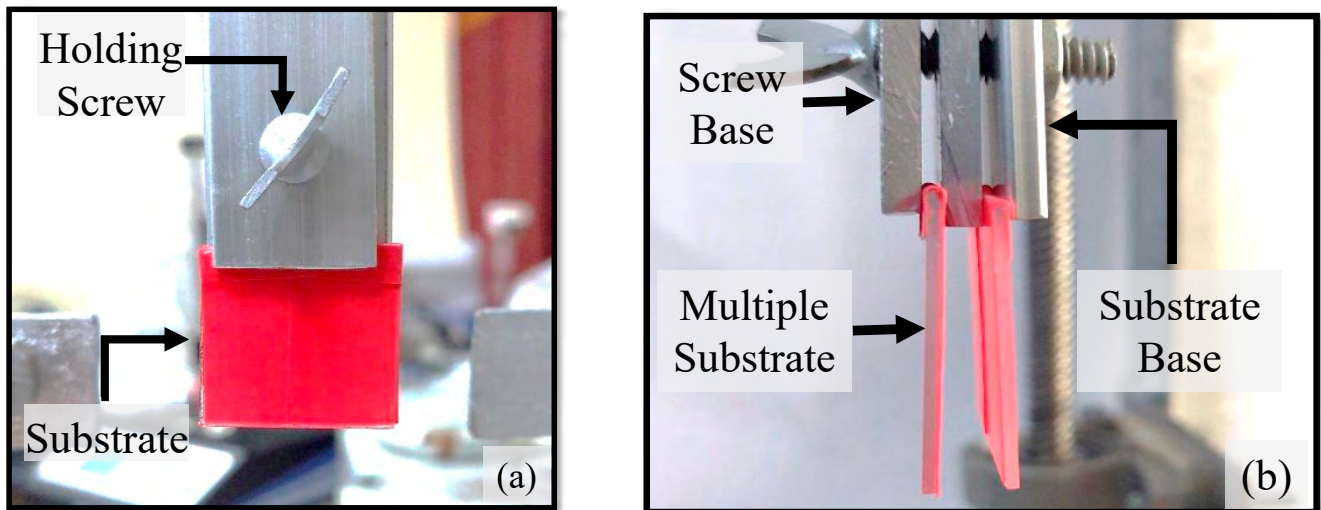


Figure 4: Close View Of The Substrate Holder (a) Front View (b) Side View

2.2. Electrical and Electronic Components

The electrical interconnections of equipment on Veroboard are shown in Figure 5(a). In our work, the dip-coater was developed using an electrical component consisting of an affordable recycled stepper motor from the 3D printer (Prusa i3 MK3/MK3S) shown in Figure 5(b) and controlled by an IoT-based electronic module composed of a microcontroller (ESP32-WROOM32) and a stepper motor driver (TMC2209). The Arduino IDE 2.3.3 was the software

that was used in our work. The simulation was carried out using an online IoT simulator (WOKWi) prior to installation. In addition, a Veroboard, a few electrical wires and connectors, a switch, a 4*4 matrix keypad, a Liquid Crystal Display, and an AC/DC adaptor (Maraj DC 9V, 2A) were used. Blynk IoT app is used as an IoT interface. The ESP32 microcontroller, TMC2209 motor driver, and buck converter are the main components that are kept inside the instrument box.

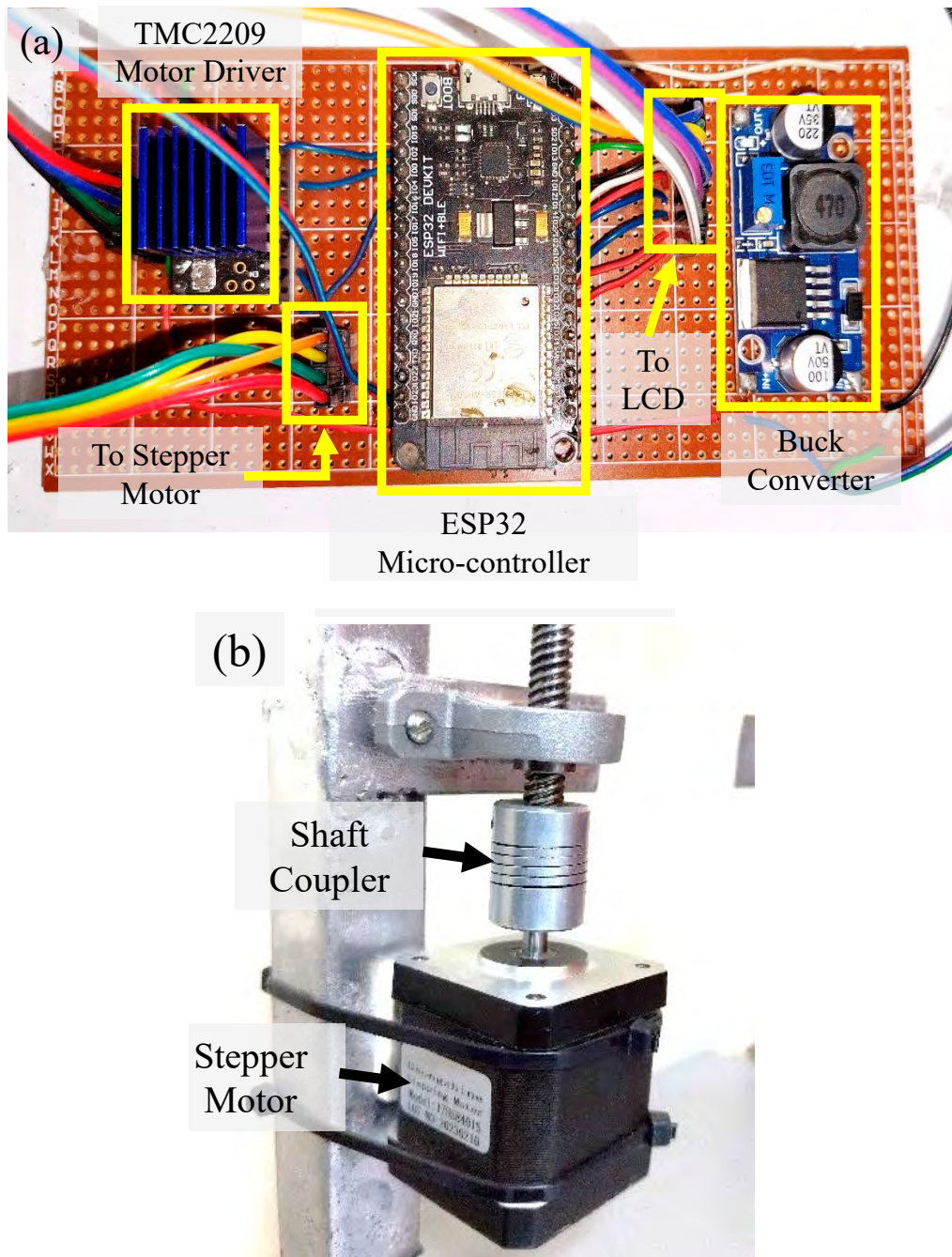


Figure 5: (a) Internal Circuit Connection Of Iot-Based Digital Dip Coating System, and (b) Nema 17 Stepper Motor.

3. Preparation of ZnO Thin Films

3.1. Precursor Solution Preparation

For experiments, analytical-grade chemicals have been employed without the need for additional purification. To prepare 1M solution, 10.98 g of Zinc Acetate Dihydrate $[Zn(CH_3COO)_2 \cdot 2H_2O]$ was dissolved in 50 ml of absolute ethanol. Using a magnetic stirrer, the solution was mixed thoroughly (at 50 °C) by a continuous string of 1 hour. When the solution was transformed into an emulsion a quantity of DEA (diethanolamine), $[NH(CH_2CH_2OH)_2]$ as a chelating agent was added to the emulsion. The molar ratio of DEA to zinc acetate was 1:1. After the string, the emulsion became transparent. After the aging of 24 hours, the sol solution was used for the deposition of thin film.

4. Thin Film deposition by Sol-gel System

Initially, Ethanol and De-ionized water (DI) were used to clean the bare glass substrate (25 mm x 25 mm). Clean glass substrates were coated with sol using the dip-coating technique

with a withdrawal rate of around 10 mm/min. To get the necessary film thickness, the previously described process was carried out 5 times. The films were then preheated for 15 minutes at 150 °C and then the resulting films were annealed at 500 °C for two hours while being heated at a rate of 20 °C per minute.

5. Characterization Techniques for Thin Films

This piece of writing deals with the preparation of three sets of ZnO thin films by previously developed homemade dip coater, commercial dip coater, and developed IoT-based dip coater to investigate the variations in their characteristics. Optical properties were measured within the wavelength range of 300 nm to 900 nm using a SHIMADZU UV-2600 visible spectrophotometer for all the prepared ZnO films [12,14]. The crystal structures of the ZnO films were analyzed using a SHIMADZU XRD-6100 X-ray diffractometer. Surface morphology (SEM) and chemical composition (EDX) were conducted on a JEOL, JCM 6000PLUS scanning electron microscope.

6. Result & Discussion

6.1. Optical & Crystalline Properties

The transmittance along with the absorbance curve of the produced thin films of ZnO for both devices is illustrated in Figure 6. Five layers of ZnO were applied on a glass substrate, and the absorbance and transmittance were carefully studied. It was found that within the visible spectrum (380 nm to 800 nm), approximately 80% of light is transmitted through the thin film with minimum absorption, suggesting a high level of transmittance. Alternatively, a remarkably low absorbance of less than 0.25 absorbance units was exhibited by the thin film, indicating minimal absorption of incident light due to its extremely thin nature.

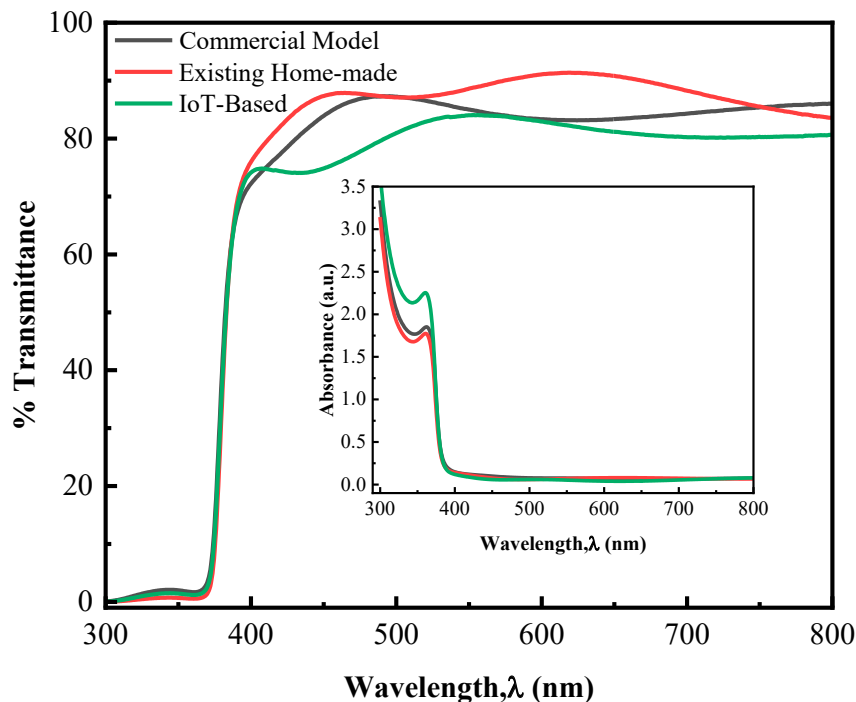


Figure 6: Transmittance Spectra Of ZnO Thin Film; (Inset) Absorbance Spectra Of ZnO Thin Film

The energy difference between the conduction and valence bands is represented by the optical band gaps, and is determined using the Tauc and Menth method [18].

$$(\alpha h\nu) = A(h\nu - E_g)^r \quad (2)$$

The symbols α , A , $h\nu$, and r stand for the coefficient of absorption, edge width parameter, the energy of the photon, and a value of

constant, correspondingly, with direct transitions, acceptable transition values are $r = \frac{1}{2}$. The bandgap values calculated from Figure 7 for the Commercial Dip Coater (3.22 eV), Existing Home-

made Dip Coater (3.24 eV), and IoT-Based Dip Coater (3.23 eV) show a close similarity, indicating consistent performance among the different dip coaters in preparing ZnO thin films.

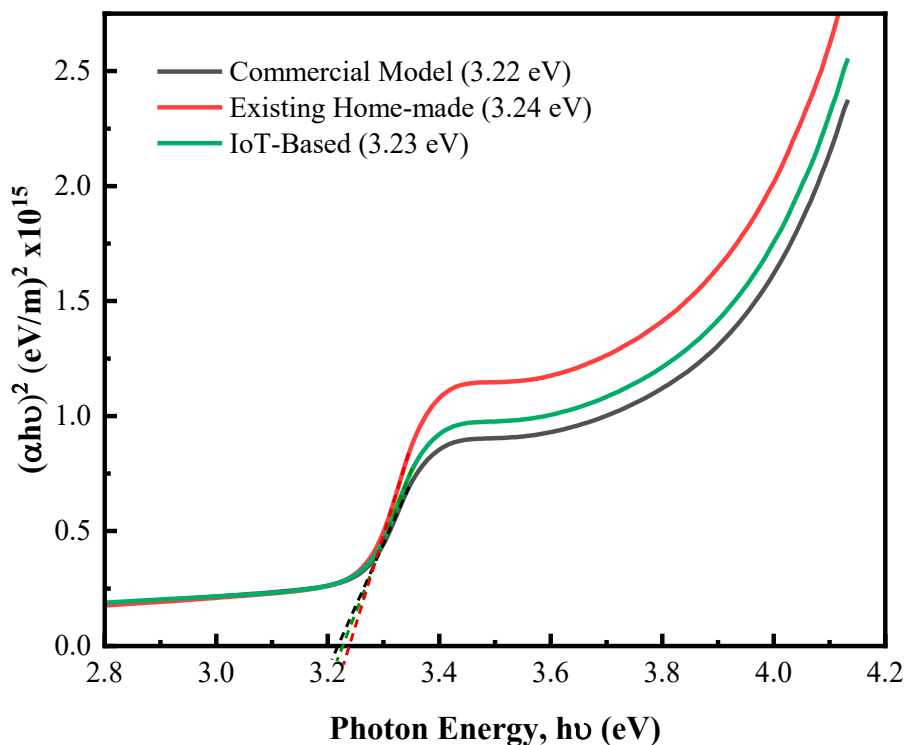


Figure 7: Bandgap of Prepared ZnO Thin Film Deposited By Various Dip Coaters

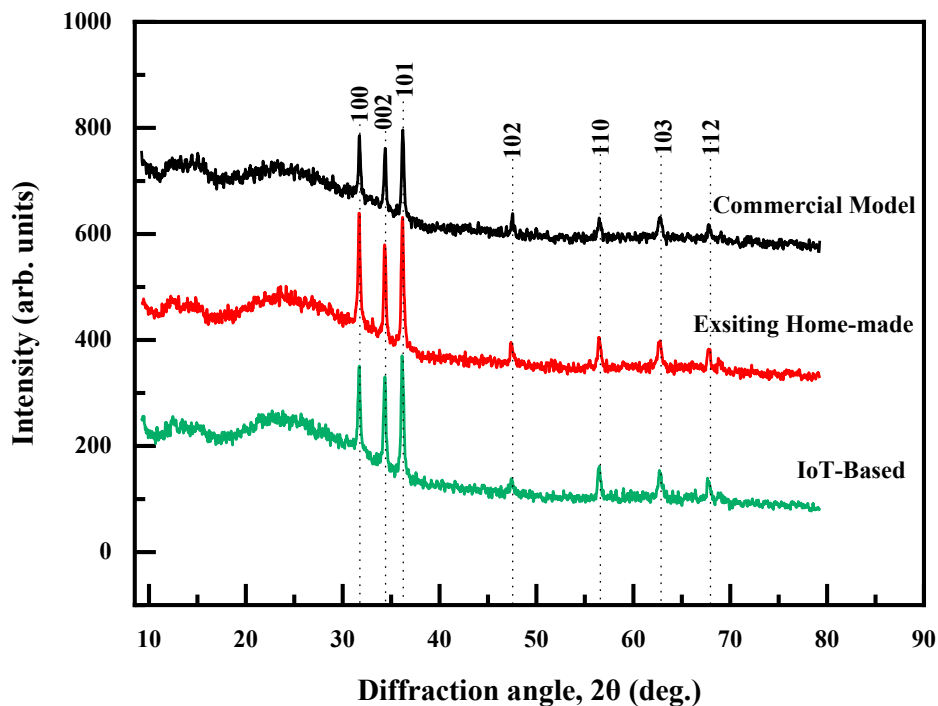


Figure 8: XRD Analysis of ZnO Thin Film Prepared By Various Dip Coaters

Figure 8 showcases the XRD patterns obtained from the deposition of ZnO (JCPDS card no. 36-1451) using different devices. The presence of sharp peaks in the patterns is indicative of a well-aligned wurtzite structure, which is characteristic of ZnO films. Specifically, the pronounced peaks corresponding to the (100), (002), and (101) orientations highlight the high level of crystallinity consistent with the wurtzite structure of ZnO. These results serve to validate the quality and structural soundness of the deposited ZnO thin films across a range of fabrication methods and devices. The Debye–Scherrer equation has been used to determine the particle's crystallite size, as follows-

$$d = \frac{0.94\lambda}{\beta \cos\theta} \quad (3)$$

The equation involves variables where d signifies crystallite size, λ represents the wavelength of X-ray radiation ($Cu - K\alpha = 0.15406 \text{ nm}$), θ denotes the angle of diffraction, and the full-width half maximum (FWHM) is β [18]. The crystallite sizes of ZnO nanostructures prepared using different dip coaters were determined as follows: 0.60 nm (Commercial dip coater), 0.564 nm (Existing homemade dip coater), and 0.4512 nm (IoT-based dip coater) by analyzing the wurtzite peak (101). Based on the

calculated data, it is evident that the crystal size of the ZnO nanostructures prepared using the IoT-based dip coater is smaller than that of the commercial model. This observation highlights the precision and effectiveness of the IoT model in producing nanostructures with finer crystallite sizes compared to commercial dip coater.

6.2. Surface Morphology & Surface Chemistry

Figure 9 displays SEM images of prepared ZnO films divided into two sections: the left side shows SEM images at 5 μm magnification, offering a macroscopic view of film morphology, while the right side presents higher magnification images (2 μm and 1 μm) for detailed examination of surface characteristics such as grain size and surface roughness.

The films deposited by various dip coaters, depicted in Figure 9, display a porous structure with visible pinholes, along with a uniform distribution over the substrate. Dip-coated films exhibit similar appearances, with comparable particle sizes. Hence, the SEM images suggest that the performance of the developed IoT-based dip coater is comparable to that of commercial dip coating equipment, as evidenced by similar film morphology and particle size distribution.

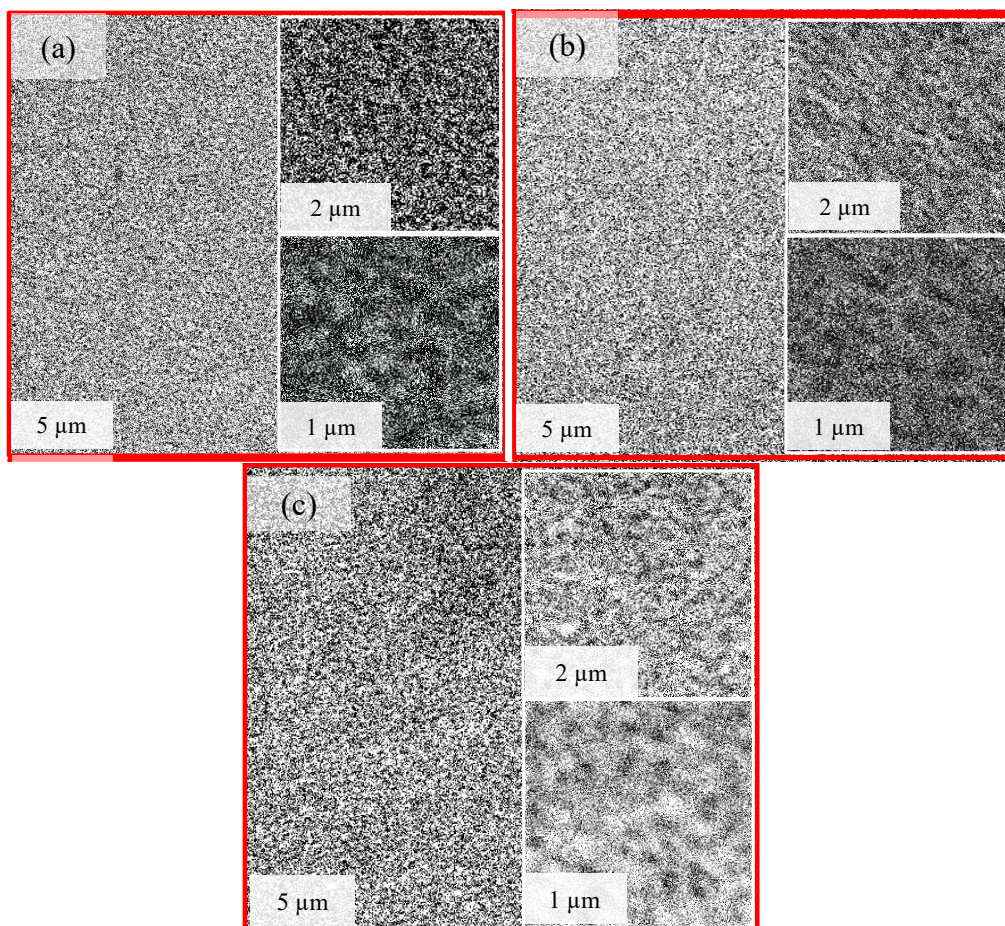


Figure 9: Sem Images of ZnO Thin Film Prepared By (a) Commercial Dip Coater, (b) Existing Homemade

Figure 10 presents a comprehensive display of the EDX spectra specifically obtained for ZnO across all of the devices. Clear Zn and O peaks in all spectra confirm ZnO phase formation on the substrates, while Si and Ca peaks indicate glass substrate presence [19]. Glass substrates typically consist of CaCO_3 (limestone), Na_2CO_3 (soda ash), SiO_2 (silica sand), and additives, as revealed by the EDX analysis. These findings provide straightforward evidence of successful ZnO thin film deposition on the glass substrates, with relevant peaks for Zn observed around 1, 8.6, and 9.6 keV in the EDX spectra. Additionally, Si and Ca signals originate from the glass substrate, attributed to the larger interaction volume of EDX compared to ZnO film thickness [20]. The EDX data of Zn and O for various dip coater are summarized in Table 1. Table 2 depicts the performance comparison table of various dip coating

system. In this table, bandgap, crystal size and surface morphology are compared. The performances of all the dip coating systems are nearly similar. The bandgap of commercial and IoT based dip coating system is exactly same. The surface morphology is also same for all the samples. So, it can be concluded that the performance of IoT based dip coating system is better than other system. The cost of IoT based system is also reasonable. Table 1 shows the cost comparison of various dip coating system. The cost of commercial dip coater is about \$3000 [12]. This price is very high for the researcher of developing country. The price of existing homemade dip coating system is only \$100. But it has vibration problems. On the other hand, the price of IoT based dip coating system is about \$100. All the problems of previous mentioned dip coating system have been solved in this system.

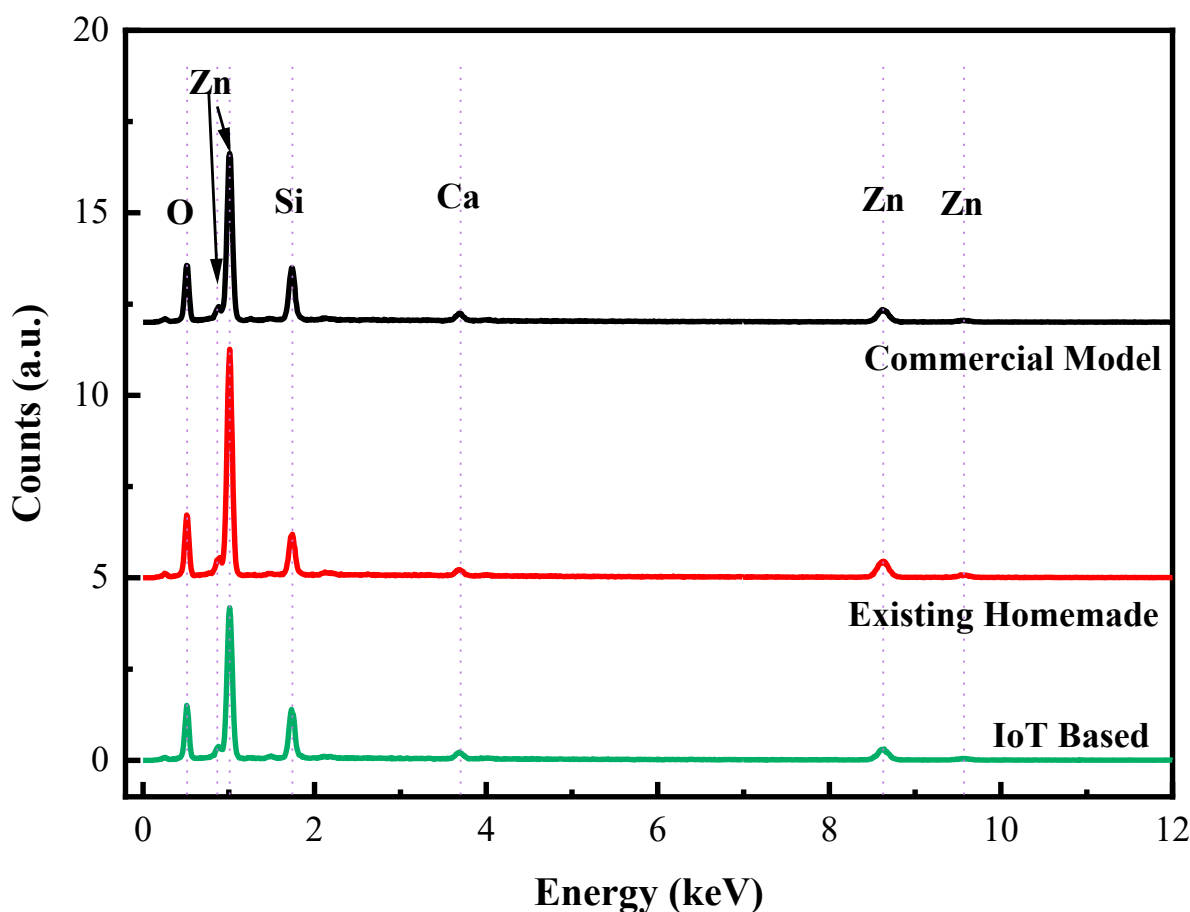


Figure 10: Edx Patterns of ZnO Thin Films Prepared By Various Dip Coaters

7. Conclusion

A cost-effective IoT-enabled dip coating system, priced at approximately 100 USD comparison as shown in Table 3, was successfully established. During testing, room temperature was used to produce ZnO thin films onto a glass substrate, with five coatings achieving nearly 80% transmittance and absorbance below 0.25 a.u., comparable to commercial systems. SEM analysis revealed similar porous structures and particle sizes between films from the IoT-based and commercial systems, showcasing

comparable performance illustrated on Table 2. The IoT-based dip coater outperformed homemade models by reducing mechanical vibration, managing multiple substrate holders simultaneously, and expanding dipping and withdrawing rates (1 mm/min to 100 mm/min). Additionally, it introduced data storage on its website and a durable metallic body. In summary, this dip coater offers an affordable, user-friendly solution for thin film preparation, benefiting scientific research.

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Devices	O	Zn	Si	Ca
Commercial Model	30.07	54.43	12.77	2.73
Existing Homemade	26.67	63.99	9.34	0
IoT Based	31.29	52.67	13.22	2.82

Table 1: Compositions (%Wt) in EDX Patterns

Parameters	Commercial Dip Coater [12]	Existing Homemade Dip Coater [14]	IoT-based Dip coater
Bandgap	3.22 eV	3.24 eV	3.23 eV
Crystal Size	0.60 nm	0.564 nm	0.4512 nm
Surface Morphology	Porous uniform	Porous uniform	Porous uniform

Table 2: Comparative Bandgap (from Tauc plot), Crystallite Size (from XRD), and Surface Morphology (from SEM) of Various Dip Coater.

Commercial Dip Coater [12]	Existing Homemade Dip Coater [14]	IoT-based Dip coater	
\$3000 (Model: Ossila L2006A-0290)	\$100	Mechanical components	\$80.87
		Electrical Components	\$17.20
		Total	\$98.07

Table 3: Comparative Cost Analysis of various Dip Coaters.

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