# O 25. HOMEMADE DIP COATING MACHINE FOR THIN FILMS

Zeynep Cansu Ayturan[1*], Yasin Akın Ayturan[2], Emre Dalkılıç[1], Şükrü Dursun[1]

[1]*Konya Technical University, Department of Environmental Engineering, Engineering and Natural Science Faculty, Konya, Turkey*
[2]*KTO Karatay University, Department of Electrical and Computer Engineering, Graduate School of Natural and Applied Science, Konya, Turkey*

*E-mail: zcayturan@ktun.edu.tr, akin@ayturan.com, emredalkilic95@gmail.com, sdursun@ktun.edu.tr*

**ABSTRACT:** In recent years, the use of thin films for several purposes has been increased rapidly. The performance of thin film coated on the material is related to the sensitivity of electronic dip coating devices. Especially, the differences in production techniques and production conditions reveal many features in thin films. With the development of technology, new production technologies and techniques that may be alternative to each other in the production of thin films have been emerged and developed. Although the sol-gel dipping method does not have a very old history, it has been an important technique for gaining new properties to glass and ceramics.
In this study, the production, software, design and sensitivities of the device used in dip coating technique, which is one of the sol - gel coating techniques, were taken as the main issues. A new device has been developed to coat glass sheets of 50 cm long and produce substantially transparent conductive layers. In this new device, glass carrier arm is used to dipping them into a solution at a certain speed, waiting certain time period and removing them at the same speed in order to coat the surface with the colloidal suspensions formed by the solid particles in the prepared liquid named as sol. The device has been designed in laboratory environment, the software has been developed for arranging speed and waiting time period with manual and Bluetooth control. The performance of homemade dip coating device was evaluated according to optic and atomic force microscopy images and thin film thickness determined with special equation. According to the results, the film thickness of coated samples was almost 7 micrometres and the surface of the films was observed smooth with cracks.

*Keywords: Dip coating, dip coater, thin film, sol-gel*

## 1. INTRODUCTION

Surface coating; is the process of obtaining a new surface layer by coating the surface of the main material of a metal or alloy known in a chemical composition different from the main material (URL 1; Muğla, 2010). Coating technologies developed to provide a new and more functional characterization beyond the existing properties of the base material contribute not only to architecture but also to many areas. It is applied to prevent wear, tear, impact, corrosion and scouring, as well as to improve decor, optical, thermal, electrical, mechanical and chemical properties (URL 1; Özel, 2013).

### 1.1. Sol-Gel Process

The concept of "sol-gel" is formed by the abbreviation of solution-gelling. Thanks to sol-gel technology, which provides a versatile approach in the synthesis of inorganic polymers and organic-inorganic hybrid materials, homogeneous inorganic oxide materials with desired properties can be obtained at room temperatures without the need for high temperatures required for conversion to inorganic glasses (Karasu, 2018). General steps of sol-gel method; hydrolysis of the precursor, alcohol or water cohesion of sol-gel active species, gelation, aging, drying, high temperature process (Özler, 2007; Akıncı, 1995). Coating procedure mainly applied in the gelation step of sol-gel method and dip coating is one of the mostly applied method for thin film preparation. Dip coating has been extensively utilized for research purposes owing to the positive features of the method such as convenient and facile approach. In Figure 1 the sol-gel method basic nano material production steps were given.

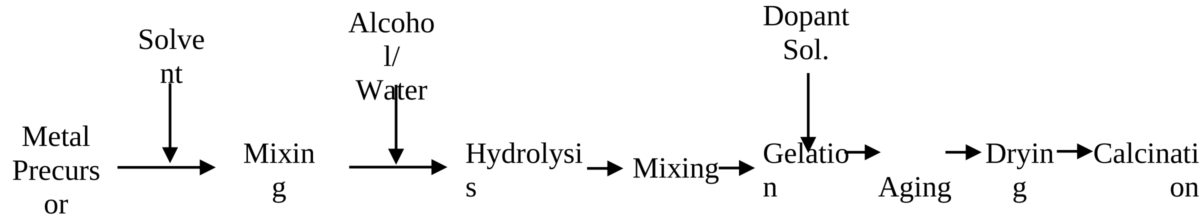


**Figure 1.** Sol-gel basic steps for nanomaterial production

### 1.2. Dip Coating

Dip coating method is the most important and widely used method among the coating methods to be made by using so-gel solution. The method is a film coating process which is carried out by dipping the prepared solution into a container and dipping the material to be coated in a solution without being subjected to vibrations at a certain and constant speed (Evcin, 2006; Evcin, 2016).

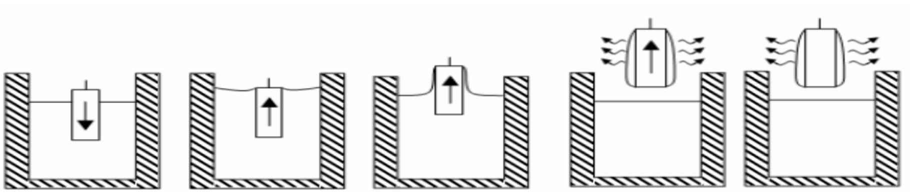

**Figure 2.** Dip coating process stages

Dip coating occurs in five stages. These are immersion, startup, deposition, drainage and evaporation. In immersion stage the substrate is dipped into the coating solution at a constant speed. The substrate remains in the solution for a designated time (30 seconds are recommended.), and then it is withdrawn with a same speed during startup. In deposition stage while the substrate is being pulled out, the thin film coating starts to be deposited on it. The thickness of the coating is directly dependent on the speed by which the substrate is being pulled out. The slower pull, the thinner the coating layer. In drainage step, excess liquid is drained from the substrate surface. Finally, solvent starts to evaporate from the surface of the substrate to form a thin film during evaporation. If the solvent is volatile, this step might happen in step 3 (Kakaei et al., 2019; Aegerter & Mennig, 2004). Figure 2 shows these steps respectively.

After the coating process, Equation 1 is used to calculate the thickness of the coating. The thickness of the film formed after the coating process; the density and viscosity of the prepared solution, the speed of dip, surface tension and gravity may vary (Brinker,2013; Güngör & Güngör, 2015).

$$h = c.\frac{(\eta U)^{\frac{2}{3}}}{\gamma^{\frac{1}{6}}(\rho g)^{\frac{1}{2}}} \qquad \text{(Equation 1)}$$

(h: Thin film thickness, c: Constant (0.944), η: Viscosity, U: Dipping speed, γ: Surface tension, ρ: Density, g: Gravitational acceleration)

Because of the high prices of the dip coating devices, the lack of product in the market which is able to coat long materials, easy to apply procedure and the construction prices with cheaper than the market, developing a new dip coating device to coat glass sheets of 50 cm long and produce substantially transparent conductive layers was conducted in laboratory environment.

In this study, the design of the Dip Coating system based on the use of manual and bluetooth controlled step motor driver unit and the associated mobile unit will be explained. Owing to this system designed in the laboratory, thin films were coated on both sides by dipping the glass material into the prepared solution.

## 2. MATERIAL AND METHOD

There have been 4 steps followed to design dip coating device. These are provision of carrier arm, software development, construction of outline profile and construction of bottom platform.

### 2.1. Provision of Carrier Arm

Linear actuator of 500 mm long was provided as a carrier arm which is mainly designed for lifting and pushing movements of any objects. Actuator with the features given below was supplied:

- Operation Voltage: 12/24V
- Load Capacity: 6000N
- Speed: 2 mm/s for 12 V, 4mm/s for 24 V
- Stroke: 50 mm to 500 mm
- Operation Temperature: -20 to +40 ºC
- Limit Switch: Built-in, Factory Preset

### 2.2. Software Development

Arduino uno programing platform was used for controlling of actuator. In Figure 3 basic diagram of Arduino uno is given. Arduino is a physical programming platform consisting of an I / O card and a development environment that includes an application of Processing / Wiring. In the hardware of Arduino, one microcontroller such as ATmega328, ATmega256 and ATmega32u4 and other auxiliary parts for programming and connection with other circuits is placed. Each Arduino board has at least one 5 volt regulating integrated and a 16MHz crystal oscillator (some with ceramic resonator). Arduino cards do not need an external programmer for programming, because the microcontroller on the card is pre-written with a bootloader program (URL 2).

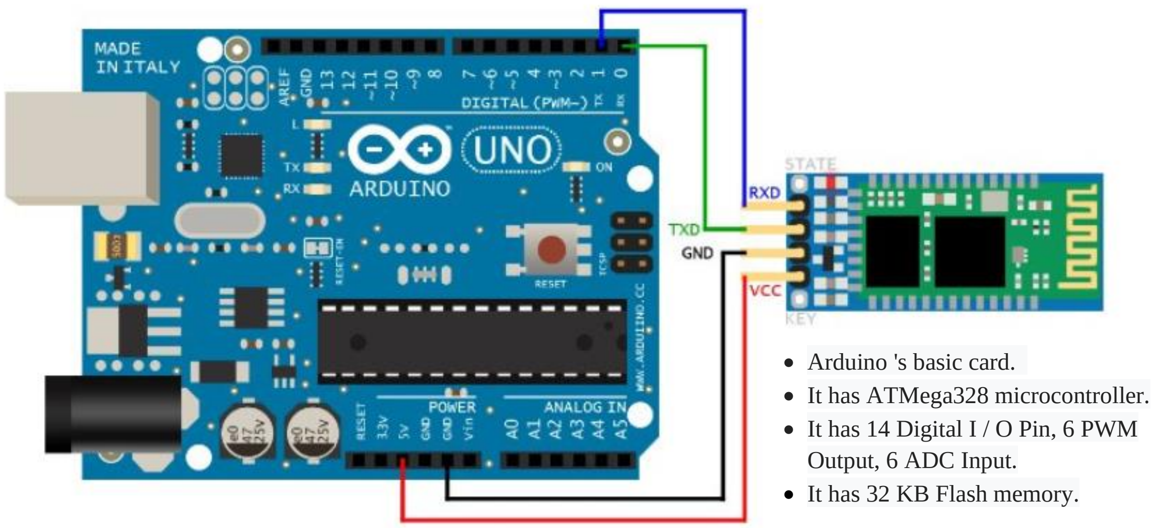


**Figure 3.** The schematic diagram of Arduino Uno (URL 2)

Arduino development environment (IDE), Arduino bootloader (Optiboot), Arduino libraries, AVRDude (Arduino remote microcontroller programming software) and compiler (AVR-GCC) constitutes the basic structure of Arduino.

Arduino software consists of a development environment (IDE) and libraries. The IDE was written in Java and is based on the environment of processing. The libraries are written in C and C ++ and they are compiled with AVR-GCC and AVR Libc. Arduino source codes are available in here. The Optiboot component is the bootloader component of Arduino. This is the component that enables the programming of the microcontroller on Arduino cards (URL 2).

The most important component that makes Arduino so popular is the Arduino libraries, which enable everyone to program without having to have detailed knowledge of the microcontroller. A list of Arduino libraries is available here. Arduino libraries come with the development environment and are located under the "libraries" folder. By examining the code, you can see how the microcontrollers are programmed and the structure of the libraries. Finally, it is used to program the compiled code in the AVRDude component (URL 2).

In this study, for programming of Arduino C language was used and speed arrangement was conducted with regulating of the voltage receiving by actuator. When the voltage was adjusted to 12 V, the actuator moved with a speed of 2 mm/s. The designed machine can be controlled with manual keypad and Bluetooth of mobile phone. There are four options defined with programming such as stop, downward, upward and wait 30 seconds before going upward. Small part of a code used for the downward movement of the carrier arm is given in Figure 4.

```
void loop() {
  key = keypad.getKey();
  veri = Serial3.read();

  if (key){
   Serial.println(key);
  }

  if(veri){
    Serial.println(veri);
  }

  if (veri == 'd' || key == '*')
  {
   lcd.clear();
   lcd.setCursor(0, 0);
   lcd.print("Going DOWN");
```

Key represents manual control

Veri represents blutooth control

**Figure 4.** A small part of code used for the downward movement of the carrier arm

### 2.3. Construction of Outline Profile

Outline platform appropriate for length of material which will be coated was constructed from iron rods with 2 m long. In Figure 5 the construction process of outline profile was shown.

### 2.4. Construction of Bottom Platform

A bottom platform was made to ensure balance between dipping arm and dipping container with screws. In Figure 5 the picture of bottom platform is given. The AutoCAD drawing and the final picture of the device is given in Figure 6.

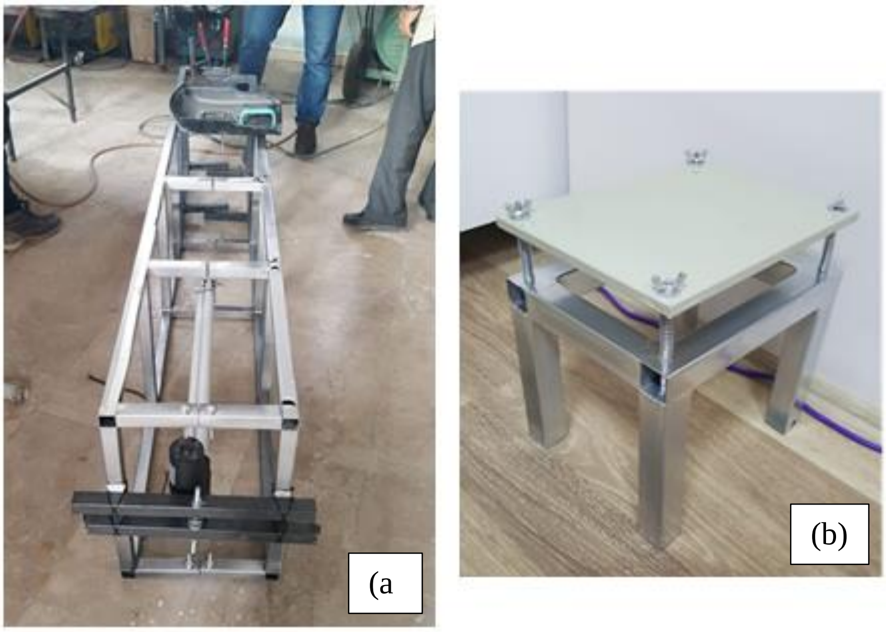


**Figure 5.** (a) Construction of outline profile of dip coater and (b)the picture of bottom platform


**Figure 6.** (a) AutoCAD drawing of dip coater and (b) the picture of final device

### 2.5. Performance Experiment

To Determine system performance, three experiment was conducted with different sols prepared with sol-gel method. The experiments were carried out with the procedure given below:

1. Substrate, which is glass sheets of 500 mm long, was cleaned with 2-propanol and purified water
2. Plates were immersed into sol with 2 mm/second velocity.
3. The plates were waited for 30 seconds into the sols before pulling out.
4. The plates were withdrawn from the sol with same speed (2 mm/second).
5. They were dried in a desiccator for 1 hour at room temperature.
6. Finally, they were subjected to heat treatment in an oven for 1 hours at 600 $^{o}$C.

The performance of device to produce thin films was evaluated according to the optic and atomic force microscopy images of this experiment and thin film thickness determined with equation 1.

## 3. RESULTS AND DISCUSSION

Totally 3 samples were prepared with dip coating experiments. Optic microscopy images of samples are given in the Figure 7, 8, 9. Different extension rates were used to take these images. 200µm, 100 µm, 20 µm and 10 µm are the zoom in ranges used for these samples

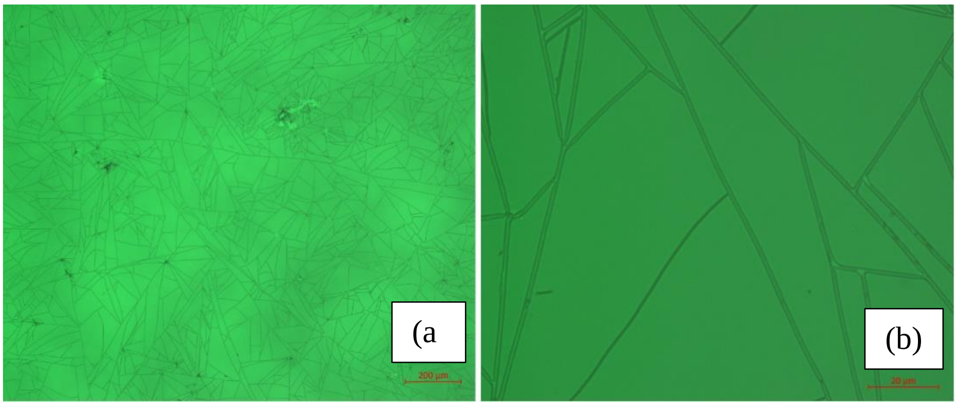


**Figure 7.** Optic microscopy images of samples (a) image of sample 1 with 200µm zoom in, (b) image of sample 1 with 20 µm zoom in

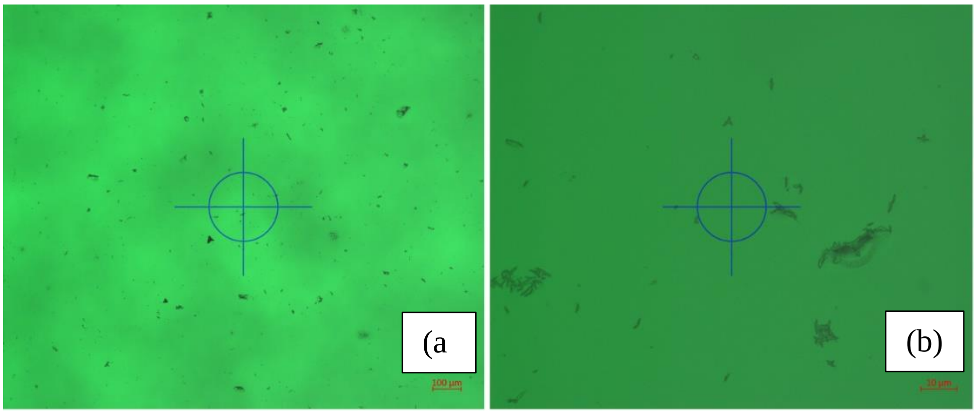


**Figure 8.** Optic microscopy images of samples (a) image of sample 2 with 100µm zoom in, (b) image of sample 2 with 10 µm zoom in

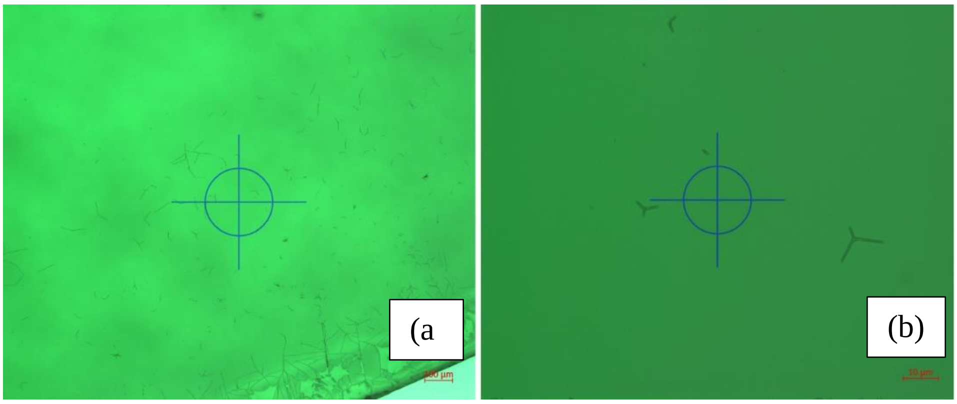


**Figure 9.** Optic microscopy images of samples (a) image of sample 3 with 100µm zoom in, (b) image of sample 3 with 10 µm zoom in

According to optic microscopy images, there were cracks formed on the surface of sample 1. In the other samples, cracks are not so apparent. The main reason of these cracks may be the heat treatment at high temperatures. Atomic force microscopy image of samples is given in Figure 10, 11, 12. Moreover, from atomic force microscopy images roughness of the samples were calculated. The average roughness value of samples is 1.797 nm for sample 1, 1.98 nm for sample 2 and 1.678 nm for sample 3. These images show that the surface of the samples is smooth with a few peak points. Especially in sample 3, peaks are less than the other samples. When the roughness values of samples were evaluated, they are acceptable and show that the surface of thin films produced with this machine are very smooth (Mardare and Hones, 1999).

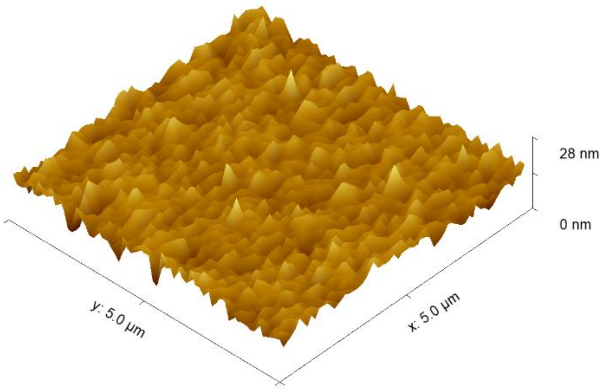


**Figure 10.** Atomic force microscopy image of sample 1

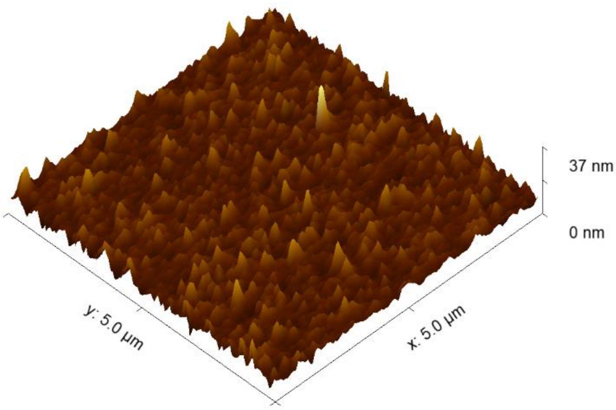


**Figure 11.** Atomic force microscopy image of sample 2

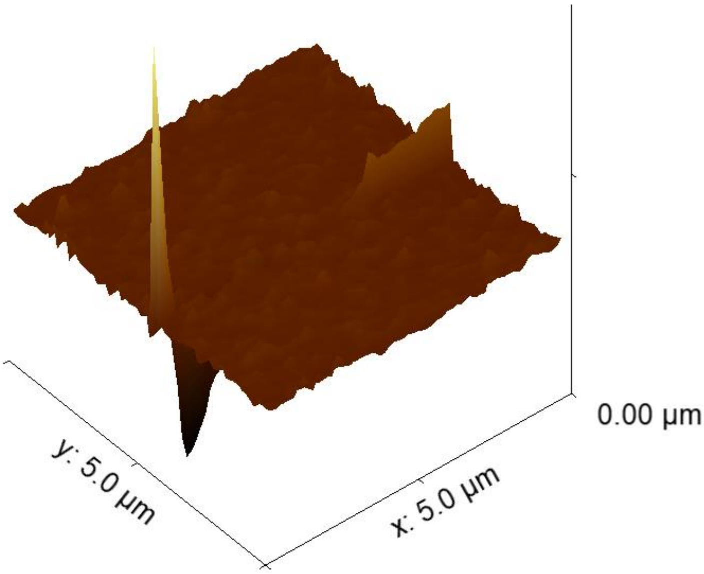


**Figure 12.** Atomic force microscopy image of sample 3

Also, thin film thickness was calculated according to Equation 1 and it was measured with profilometer. In Table 1, results of calculated and measured film thickness values and parameters used for calculation are given. These results are acceptable because the coating layer thickness is getting thicker closed to uncovered area of the glasses. Therefore, measured film thickness was found smaller than the thickness calculated with equation 1. The features of the sol are one of the main factors affecting the thin film. The dilution of chemicals and solvents used for the preparation of sol may affect the thin film thickness. Furthermore, the withdrawal speed is also important, the smaller speeds reduce the thin film thickness.

**Table 1.** Calculated and measured thin film thickness values of samples

| Samples | Parameters | Values | Calculated Thin Film Thickness (µm) | Measured Thin Film Thickness (µm) |
|---|---|---|---|---|
| **Sample 1** | Viscosity (mPa.s) | 1.5 | 6 | 0.5 |
| | Surface Tension (dyn/cm) | 23.5 | | |
| | Density ($g/cm^3$) | 0.83 | | |
| | Dipping speed (mm/s) | 2 | | |
| **Sample 2** | Viscosity (mPa.s) | 2 | 7.2 | 0.5 |
| | Surface Tension (dyn/cm) | 23.5 | | |
| | Density ($g/cm^3$) | 0.84 | | |
| | Dipping speed (mm/s) | 2 | | |
| **Sample 3** | Viscosity (mPa.s) | 2.5 | 8.3 | 0.5 |
| | Surface Tension (dyn/cm) | 21.7 | | |
| | Density ($g/cm^3$) | 0.88 | | |
| | Dipping speed (mm/s) | 2 | | |

## 4. CONCLUSION

Dip coating is an industrial coating process to manufacture bulk products such as coated fabrics and specialized coatings for example in the biomedical field. Moreover, it is one of the mostly used method to prepare coated materials with thin films especially for research purposes. By the help of this process, it is possible to produce thin films on any object. In this study, the homemade dip coating device, which has an ability to coat long objects up to 500 mm, was designed and built in laboratory environment with very cheap cost. The cost of this device is 10 times lower than similar products in the market and all equipment used for this device was very easy to find. According to optic and atomic force microscopy images, thin film thickness determination and roughness measurements, performance of this new machine was found successful and suitable to use for coating of long materials.

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