# Design and Implementation of a Threeoperational Mode Programmable Spin Coater for Deposition of Thin Films on Flat Substrates

Atule A. A., Amah A. N., Ahemen I. & Gesa F. N.

Abstract-A programmable spin coater with three operational modes (manual, single and multiple) has been successfully designed and constructed. Microcontroller (PIC19F876) was interfaced with a display (20x4), multiplexed keypad (input), d.c. motor, a mosfet driver (IR2100), regulated power supply as well as passive components to achieve the system. The controller was programmed with a code written in C-compiler for effective coordination of its software so as to enable the controller communicate with the d.c. motor according to the predefined speed algorithms. When powered and tested at supply voltages of 3 V, 6 V, 9 V, 12 V, 15 V, 18 V, 24 V and 28. 2 V, the motor generated varying spinning speeds of 522 rpm, 1044 rpm, 1620 rpm, 2088 rpm, 2609 rpm, 3132 rpm, 4176 rpm and 4906 rpm respectively. The system is capable of operating at the maximum speed of 4906 rpm against the designed speed of 5000 rpm for 10 bit resolution (100 % duty cycle) and minimum speed of 500 rpm. The slight variations observed is attributed to losses in the implemented system which however did not affect the overall performance of the system as evident in the coated thickness of the substrates. Using VeecoDektal 150 profilometer, the profilometric analyses of thin films coated with different concentration of pure CdO at varying speedsusing single mode operation and Eu-doped at constant speed at varying doped layers using multiple modes operation for 15 secs. show film thickness of 1100 nm, 800 nm, 450 nm, 400 nm and 390 nm for pure CdO and 510 nm, 470 nm, 450 nm, 300 nm and 270 nm for Eu doped CdO respectively. The results obtained collaborates speed versus film thickness trends reported by other researchers of nano materials which posited that film thickness varies inversely as the coating speed. The programmable spin coater here designed and implemented thus find useful applications in medical and semiconductor industries as well as nano materials research laboratories where the applications and usefulness of deposited thin films/substrates cannot be over-emphasized.

Index Terms- Programmable, Spin coating, deposition, thin film, characterization.



#### **1** INTRODUCTION

THIN film technology plays an important role in the development of integrated circuits [1]. Thin films are material layers ranging from fractions of a nanometer to several micrometers in thickness [2]. Application of uniform thin films to materials may be done using different techniques like Chemical Vapour Deposition (CVD), Physical Vapour Deposition (PVD), spin coating amongst others [3]. Of all these methods, spin coating has been identified as a fast, practical and relatively easy method of generating thin and homogeneous films [4].

In spin coating, a small amount of coating material is deposited on the centre of a substrate. The substrate is then rotated at high speed in order to spread the coating material by centrifugal force. In the coating process, rotation is continued while the fluid spins off the edges of the substrates until the desire thickness of the film is achieved. The applied solvent is usually volatile and simultaneously evaporates. The higher the angular speed of the system, the thinner the film thickness. Moreover, the film thickness depends on viscosity and concentration of the solution and solvent [5].

A good number of spin coaters are commercially available, they are however very expensive. Commercially available spin coaters include the use of microprocessors operating in one or two modes of operations. Relatively low cost spin coater units make use of analog/manual controls with the attendant challenges of manual system.

In the programmable spin coater, a D.C motor, microcontroller, liquid crystal display (LCD) crystal oscillator, optical encoder, key pad,

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transformer, linear regulator and motor driver form the most important parts of this device. A power supply is built not different from the manual power circuit, the regulated voltage acts as input to the microcontroller generating PWM signal at varying voltages which regulate the spinning speed.

The programmable spin coater has been regarded as a more favored method of coating thin films (substrates) due to it advantage of programming both spinning speed and time [6].

This research work, gives a clear procedure for designing and implementing a less expensive digital spin coating machine with a combination of single, manual and multiple operational modes for deposition of thin films on flat substrates. Additionally, the constructed spin coater will adds to research facilities and also encourage research in thin films technology both in our Universities and neighboring institutions across the globe.

### 2 MATERIALS AND METHODS

### 2.1 Materials

2.1.1 Programmable spin coater materials

The following materials/components were used in the design and construction of the programmable spin coater. Such as Multiplexed key pad, Regulators (LM317, LM7805), Microcontroller (PIC16F876), Liquid crystal display (204ZAF), Optical encoder, Mosfet (motor driver) IR2110, D.C motor (NF003SG-001), Crystal oscillators, Resistors, Capacitors, Teflon material, Metal casing, Light emitted diode (LED), Plastic material, Vero and bread board, Switch, PIC C compiler software program (CCS). 2.1.2 Sol-gel and thin films development materials

Cadmium chloride (Cdcl2), Ethanol (C2H5OH),Triethanolamine (C6H15NO3), Eurapium (Eu), Micro slide glasses (substrates),Electric hot pan plate,Oven,Furnace were used for the preparation, deposition of the sol-gel and development of thin films.

#### 2.1.3 Testing devices

The following devices used for testing of selected parameters of the work include Tachometer, Multimeter, Profilometer.

2.2 Methods

2.2.1 Programmable spin coater design

Figure 2 shows the programmable spin coater design. In the design, a switch mode transformer embedded with regulators (LM7805 and LM317) were used to regulate constant 5 V for system operation and regulates effective (desirable) output voltages up to 29.0 V for D.C motor operation. However micro-controller (PIC16F876) was interfaced with multiplex key pad, liquid crystal display (LCD), mosfet drive (IR2110) and D.C motor for optimal function of the system. The speed of the D.C motor was designed to be controlled by varying the Pulse Width Modulation (PWM) signal of varying frequency and duty cycle generated from PIC16F876. Mosfet driver further scaled up the PWM signals to high signal pulses that drive the D.C motor at varying voltages ranging from 1.25 V - 29.0 V. Multiplexed key pad was used to input selected values into the system while the LCD was used to convert analog signal of PWM to digital signal (Pulse) to enable viewing of selected values.

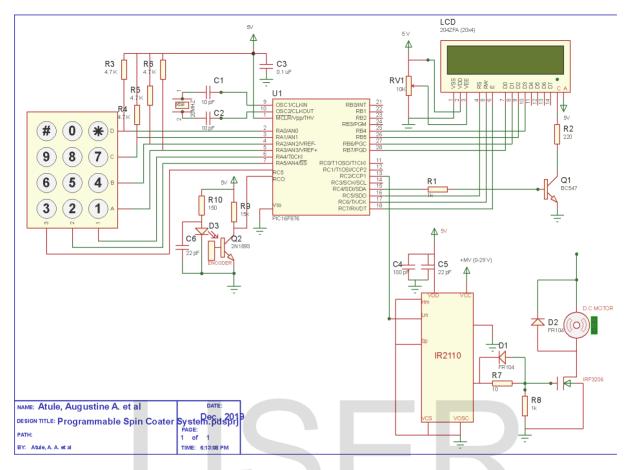
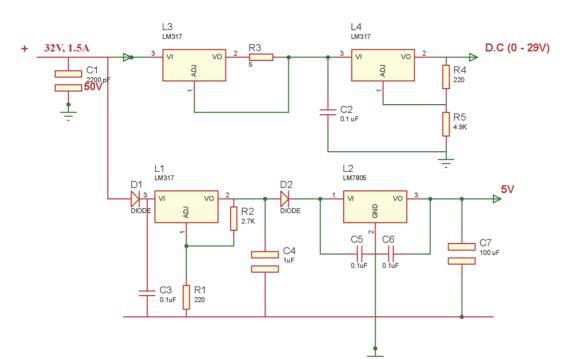


Figure 2: Design of Programmable Spin Coater

#### 2.2.2 Power supply design

The power supply was designed to tolerate 100 V – 240 V and 1300 mA a.c input. Regulators,

capacitors, resistors and a switch mode power supply of 32 V and 1.5 A output was used in the system due to its ability to regulate required minimum or maximum input voltage supply for effective results. The power supply circuit consists of two sections as shown in Figure 3.



#### Figure 3: Power Supply Circuit.

2.2.3. Construction of programmable spin coater

Construction of programmable spin coater was successful with the used of metal casing, plastics container, Teflon material, bolts and nuts. Both the circuit and D. C motor were mounted in a metal casing with D.C motor shalf passing out on the upper surface of the casing. A teflon material was used as spinning disk with adjustable nuts and bolts for holding substrate in fixed position while spinning. This was done so that it can be easily remove for cleaning and maintenance. LCD and multiplex key pad were screwed in front of the spin coater. A transparent plastic material was used to enclose the spinning disk to avoid sprinkling of solvent. Behind the system, a fixed switch key used for turning on/off the system and fixed Light emitted diode (LED) red in color used to indicate light when the system is on. The constructed spin coater is shown in plate 1.To activate the hardware for coating activities, a programme written in C and Micro C was encoded in the controller to enable the components communicate effectively as desired. The software algorithm is shown in appendix A while the source code is attached as appendix B.



Plate 1: Constructed Spin Coater System

## 3 THEORY OF THE PROGRAMMABLE SPIN COATER SYSTEM

### 3.1 Power Supply

The power supply unit comprises of two sections, in section one, regulator LM317 used to regulate the input voltage to 16.6 V output voltage which is further regulated by LM7805 to a constant output voltage of 5 V suitable for Microcontroller (PIC16F787) and display (LCD) operations. The resistor value R2 =  $2.7K\Omega$  (preferred value) used to set effective output voltage of 16. 6 V by LM317 is calculated using equation 1(a) where Vref = 1.25V and R1 =  $220\Omega$  (selected).

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$$V_{out} = V_{ref} \left( 1 + \frac{R_2}{R_1} \right)$$
 1(a)

$$V_{out} = V_{ref} \left( 1 + \frac{R_5}{R_4} \right)$$
 1(b)

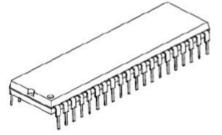
Similarly, in section 2 of the power supply unit, two LM317 were set to regulate effective (desirable) current and variable voltage (Vout = 0 to 29 V) for D.C motor operation as expressed using equation 1(b). Vref = 1.25V, R4 =  $220\Omega$ (chosen), R5 was calculated to be  $4.9k\Omega$  (preferred value).

On the other hand, to obtain the targeted regulated output current (Iout) of 0.25A, R3=  $5.0\Omega$  was calculated using equation 2 where Vref = 1.25V.

Iout 
$$=\frac{V_{ref}}{R_3}$$
 2

#### 3.2 The Microcontroller

Microcontroller is a programmable device that takes in numbers as input, performs arithmetic and logic operations on them according to programs stored memory and produces the result as output. It is programmable in the sense that it performs a given set of operation based on the sequence of instructions given to it [7]. In such a device, data is taken in through the use of input devices like the mouse, keyboard, switches etc. figure 1 is a typical diagram of Microcontroller.



$$f = \frac{1}{T_{Osc}}$$

Tosc = 50nS

In the design PIC16F876 was calibrated at 16 MHz because is within human hearing threshold hence no noise is needed, the period of oscillation was calculated using equation 3;  $T_{osc}$ = 62.5 nS.

Similarly, the PWM period of oscillation, T is expressed using equation 4.

$$T = (PR_2 + 1)X 4ToscxTMR_2PrescaleValue$$

where TMR2 is timer 2 and PR2 is period 2 register.

Chosen TMR2 prescaler as 1, period 2 (PR2) register as FFh (i.e 255 in decimal), the PWM period of oscillation T, was calculated using equation 4

T= 65 mS

The frequency of PWM period, T was obtained using equation 3;

 $\mathrm{F}=15.63~\mathrm{kHz}$ 

The pulse width at TMR2 prescaler 1 can be expressed using equation 5:

(CCPR1L, DC1B1, DC1B0)XToscXTMR2 PrescaleValue 5

Figure 1: A Typical Micro Controller diagram, microchipPulsewidth =  $6.4 \times 10-5$ 

data sheet (2010)

#### 3.2.1 Pulse width modulation (PWM)

A microcontroller (PIC16F876) is interfaced with quartz crystal oscillator of 20 MHz to generate pulse width modulated signals of varying frequencies and duty cycle that drives the D.C motor at varying speeds. With a crystal frequency, f = 20 MHz, the period, Tosc of oscillation is .expressed using equation 3, At the prescaler value of 16, period 2 register of FFh, the PWM period T, was obtained to be 1.03x10-3 S, frequency F = 0.97 kHz and pulse width 1.03 x10<sup>-3</sup>.

#### 3.2.2 PWM resolution

PWM signal is nothing more than the pulse sequence with varying duty cycle. For one specified frequency (number of pulses per second), there is a limited number of duty cycle combinations. This number is called resolution

3

IJSER © 2020 http://www.ijser.org measured in bits. For example, a 10-bit resolution result in 1024 discrete duty cycles, whereas an 8bit resolution will result in 256 discrete duty cycles etc. In relation to microcontroller, the resolution is specified in PR2 register. The maximum value is obtained by FFh number.

PWM resolution was determined at varying speeds of the designed programmable spin coater at varying supplied voltages. At 6 V supply the D.C motor speed was 1044 rpm, at 9 V supplies the D.C motor speed was 1620 rpm and at maximum voltage supply of 29 V the speed of the D.C motor was 5044 rpm for 100% duty cycle, 10 bit resolution (1024 discrete value). This implies that:

For 1044 rpm, PWM resolution was obtained using the relation given below

where x is the PWM resolution for 1044 rpm at 6 V.

Therefore PWM resolution for 1044 rpm at 6 V, 1620 rpm at 9 V, and 5044 rpm at 29 V were 21 %, 32 % and 100 % respectively.

#### 3.2.3 Timer zero (TMR0)

Timer zero counter was calibrated in PS2, PS1, PS0 (PS means prescaler) bit at the ratio of 1:1:1, which has prescaler values of 256, 255 and 76 for 8 bits wide respectively. The external clock of 20 MHz is used to generate pulses of arbitrary duration, with oscillation time of 200 nS internally. TMR0 can obtain by equation 6

Timer zero counter was calibrated in PS2, PS1, PS0 (PS means prescaler) bit at the ratio of 1:1:1, which has prescaler values of 256, 255 and 76 for 8 bits wide respectively. External clock of 20 MHz was used to generate pulses of arbitrary duration, with oscillation time of 200 nS internally. With this TMR0 can obtained as

#### TMR0 = 200nS X 256 ( i.e Tosc X prescaler value) = 5.12 X 10-5 sec

TMR0 at 256 PS was transferred to 255 PS value hence interruption has not occurred and this was obtained as

TMR0 =  $5.12 \times 10-5 \times 255 = 0.013056$  sec

Again TMR0 at 0.013056 was transferred to 76 PS value then interruption occurred and this was obtained as,

 $TMR0 = 0.013056 \sec X 76 = 0.992256 \sec C$ 

The value 0.992256 sec is counting for 1 second internally.

#### 3.2.4 Timer one (TMR1)

Timer TMR1 was configured at a prescaler value of 1:1, so it's counts immediately, once the time is set for counting.

### 3.3 Motor Speed

D.C motor has two distinct circuits; field and armature circuits. The input is electric power and output is mechanical power. The speed in revolution per minute N, is related to the angular speed  $\omega$  (in radians per second) by;



Where N is the r/min or rpm, both mean the same thing. The equation 7 can be used to determine the speed of the D.C motor at different operating conditions.

3.4 Test of the System

3.4.1 Power and Speed

The power supplied circuit, the output of selected parameters like switch mode transformer, L1, L2, L3 and L4 were tested using multimeter. The result is presented in table 1.

The tachometer was used for testing the speed at varying voltages of 3V, 6V, 9V, 12V, 15V, 18V, 24V and 28.2 Volts. The result is presented in table 2.

3.5 Sol-gel preparation and thin films coating In order to investigate the working accuracy and performance of the system, after performing several test of the device, pure cadmium oxide (CdO) and Europium (Eu) layer doped CdO solgels solution were prepared, deposited on 2.5 mm by 3.5 mm microslide substrates at different speeds rates using single and multiple operational modes of the system.

#### i Pure CdO sol-gel preparation

Pure CdO sol gel was prepared by firstly dissolving 0.1 M (2.30 g) of cadmium chloride Cdcl2 into 50 ml of ethanol (C2H5OH), with constant stirring using magnetic stirrer for 2 hours, 4 ml of triethanolamine (C6H15NO3) was added to the solution and stirred again for an hour.

ii Eu layer doped CdO sol-gel preparation The Eu layer doped CdO sol-gel concentration was prepared by dissolving 0.05 M (1.15 g) Cdcl2 into 25 ml of ethanol with constant stirring for 2 hours, 4 ml of triethanolamine was added to the solution and stirred again for an hour using magnetic stirrer. 5 % (0.041g) of Eu was dissolved in another 25 ml of ethanol and stirred for 2 hours and a clear mixture was obtained. Both Eu and CdO solutions were kept separately. All the prepared solutions were kept for 24 hours at room temperature for aging and were ready for use.



iii Coating, annealing and profilometry The single operational mode was tested by coating prepared pure CdO sol-gel solution at varying speeds of 500, 1000, 2000, 3000, 4000 rpm for constant time of 15 secs.

The multiple operational mode was tested by coating pure CdO on glass substrate at first stage, heated and dried, then Eu solution was layer doped on the surface of the coated pure CdO layer. This was done by coating Eu against pure CdO step wisely up to 4 varying doped layer samples at a constant speed of 2000 rpm for constant time of 15 secs.

All the coated samples were heated on a hot plate at 75oC for 30 seconds to removed solvent and residuals. The coated samples were annealed in a furnace at 450oC for an hour. Some of the coated films samples are shown in plate 2. The resulting films were characterized using VeecoDektal 150 profilometer.



Plate 2: Samples of some coated films

#### 4 RESULT AND DISCUSSION

#### 4.1 Power Output Results

The output results obtained from the tested selected parameters of power circuit are shown in table 1, while the speeds at various voltages are shown in table 2.

Parameter

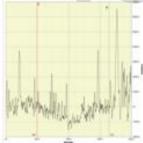
Result

Switch mode transformer output voltage	32 V
Switch mode transformer current output	1.5 A
L1 output voltage	16.6 V
L2 output voltage	5 V
L3 output current	0.25 A
L4 output voltage	28.2 V

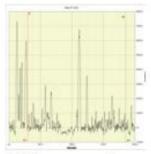
Table 1: Output of Power Circuit Parameters

From the obtained output results as shown in Table 1, the system performs optimally at 5 V, 0.25 mA when tested, similar voltage were used by several authors in powering their spin coating system such as [8], [1]. While the output results by LM317 for D.C motor operation was less than the target value due to its load regulation error of 0.1 % as recommended in LM317 data sheet [9], although the output voltage is not far from the expected voltage and it had become 28.2 ±1.0 V which keep the D.C motor at it optimal functions.

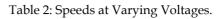
Voltage (V)	Speed (rpm)
28.2	4906.8
24.0	4176.0
18.0	3132.0
16.0	2784.0
12.0	2088.0
9.0	1620.0
6.0	1044.0
3.0	522.0



500 rpm speed



3000 rpm speed

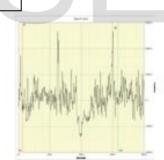


The results of speed obtained at varying voltages as shown in Table 2 reveals that's peeds vary directly to the supplied voltages. The maximum speed of the system was 4906 rpm at the maximum voltage of 28.2 V for 10 bits resolution (100 % duty cycle), this shows that at the speed of 1000 rpm the duty cycle is 20 % which is half of 1000 rpm duty cycle of Aguira system [8]. Aguilar system had maximum speed of 10000 rpm for 100 % duty cycle, but ours is approximately half in speed to Aguilar spin coating system, so if we had reach a maximum speed of 10000 rpm then we could had 100 % duty cycle, therefore this proved that the programmable spin coater work optimally (accurately) in line with [8] spin coating system.

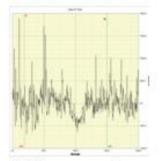
#### 4.2 **Profilometry Results**

4.2.1 Profilometry results for single mode operation

The profilometry results (Film thickness) obtained for pure CdO thin films are shown in Figure 5 and has been interpreted and the values of thickness and rpm displayed in Table 3 and the accompanying Figure 6.



1000 rpm speed



4000 rpm speed

2000 rpm speed

Figure 5: Film thickness of	pure CdO at varving speeds	for constant time of 15 secs.

Speed (rpm)	Thickness (nm)
500	1100
1000	800
2000	450
3000	400
4000	390

Table 3: Film Thickness Results obtained for pure CdO
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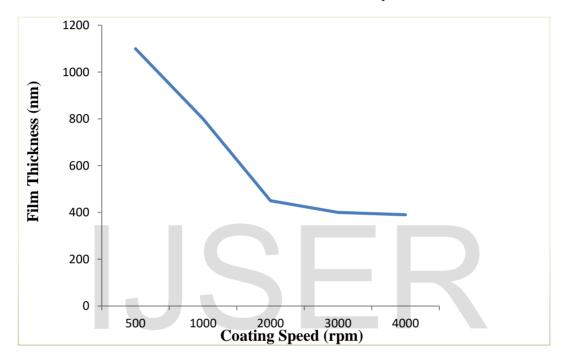


Figure 6: Film Thickness against Coating Speed for pure CdO Thin Films.

The result shows film thickness of 1100 nm, 800 nm, 450 nm, 400 nm and 390 nm at speeds of 500 rpm, 1000 rpm, 2000 rpm, 3000 rpm and 4000 rpm respectively with mean thickness value of 628 nm for a constant operational time of 15 seconds.

From the values of film thicknesses obtained, a decreasing trend is immediately observed as speed of rotation increases. This spells an inverse proportionality relationship between speed of rotation and film thickness. This deduction is in agreement with that made by [10].

From the plotted graph in Figure 6, it can be seen that the rate of decrease in film thickness is nonuniform as it slows down with increasing speed of rotation. This non uniform trend is attributable to increase in adhesion of the sol-gel due to aging.

4.2.2 Profilometry results for multiple mode of operation

To achieve Eu layer doped CdO thin films, the multiple operational mode of the constructed system was utilized. The system was preprogrammed to spin at given speeds and for preset times according to the number of spin times required to achieve the various layers considered.

The profilometry results obtained for Eu layer doped CdO and presented in Figures 6 has been interpreted and the values of thickness and number of layers displayed in Table 4 and the accompanying Figure 7.

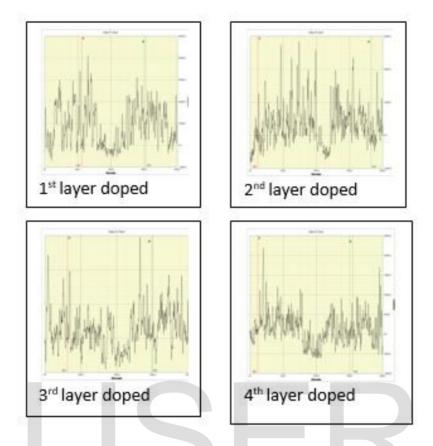


Figure 7: Film thickness of varying Eu layer doped CdO at constant speed of 2000 rpm for 15 secs.

The results shows film thicknesses of 450 nm, 530 nm, 560 nm, and 600 nm for 1st, 2nd, 3rd and 4th layers doping respectively at constant speed of 2000 rpm for 15 seconds.

From the trend of the plotted graph in Figure 8, the results reveal that film thickness increased with increased in numbers of coating layers. Rajammal, et al. [11] reports a similar observation from his work on the influence of number of coatings on properties of thin film.

The result obtained in this section basically confirms the functionality of the multiple operational mode of the constructed system.

Layers	Thickness (nm)
1st layer	480
2nd layer	530
3rd layer	560
4th layer	600

Table 4: Film Thickness Results Obtained for Eu Layer Doped CdO

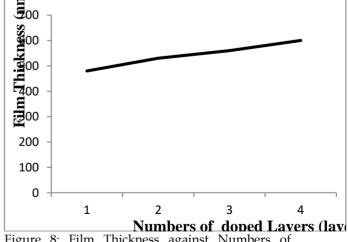


Figure 8: Film Thickness against Numbers of doped Layer for Eu Layer doped CdO Thin Films.

#### 5 CONCLUSION AND RECOMMENDATION

5.1 Conclusion

The design and construction of a programmable spin coater with three operational modes (manual, single and multiples modes) was successfully achieved, the system has maximum speed of 4906 rpm with the efficiency of 98.12 %, and the minimum speed of the device is set at 500 rpm. Test carried out on the system proved the system as reliable, efficient and in optimal functioning state. The system therefore finds useful application in research areas like semiconductor, medical and photographic industries among others.

### 5.2 Recommendation

Several characteristics of the thin films such as morphology, electrical, optical and mechanical properties of speed varying coated samples were not studied as they were outside the scope of this work. It is therefore recommended that future researchers venture into the characterization of several other features of films produced by the constructed spin coater. Additionally, future researchers should go beyond constructing a spin coating device and include multiple dispensers to enable easy depositions of multiple samples on substrates.

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# 6.0 Appendices

Appendix A:	Spin Coater Software Flow Chart
Algorithms	



Appendix A: Spin

# Coater Flow Chart Continuation

