

# DESIGN OF DIGITAL THERMOMETER AND APPLICATION IN ELECTROSPINNING

A THESIS SUBMITTED IN PARTIAL FULFILLMENT

OF THE REQUIREMENT FOR THE DEGREE OF

**BACHLEOR OF TECHNOLOGY** 

IN

### **BIO-MEDICAL ENGINEERING**

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# **Acknowledgement**

I express my sincere gratitude to Prof. K. Pramanik, Professor of the Department of Biotechnology Engineering, National Institute of Technology, Rourkela, for giving me this great opportunity to work under her guidance throughout the course of this work. I am also thankful for her valuable guidance and timely suggestion without which work would not have been possible

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# CERTIFICATE

This is to certify that the thesis entitled, "**DESIGN OF DIGITAL THERMOMETER AND APPLICATION IN ELECTROSPINNING**" submitted by **SAROJ KUMAR ROUT** in partial fulfilments for the requirements for the award of Bachelor of Technology Degree in Bio-Medical Engineering at National Institute of Technology, Rourkela (Deemed University) is an authentic work carried out by them under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University / Institute for the award of any Degree or Diploma.

Date: 11/05/2011

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### ABSTRACT

Electrospinning is currently available one of the best fibre forming technology with many advantages over previously available methods. But still this process requires a lot of optimization. In my project I mainly focused on design of digital thermometer which can be further used for real time temperature measurement during the run of electrospinning process. LM 35 sensor and At-mega 32 microcontroller was used. Seven segment display is used for the display of temperature. The thesis mainly covers the basics of electrospinning, factors affecting it and how to control the parameters for its optimization. But mainly the thesis focused on the design of digital thermometer. At different temperature SEM of fibre produced were taken and it was observed that at higher temperature fibre diameter decreases .So temperature control is an important aspect in electrospinning to control fibre diameter and for production of smooth fibers.

Keywords: Nano fibre, Digital temperature control, At mega 32,

# CHAPTER -1

# INTRODUCTION

# **1.1** <u>INTRODUCTION</u>

Traditional needle Electrospinnin available is common and versatile technique for the production of Nano fibers with diameters ranging from 10 to 1000 nm. It has wide advantage over the previously available fibre formation method because here electrostatic force is used instead of conventionally used mechanical force the production of Nano fibers. Moreover this process can produce fibers with high surface area to volume ratio and with high porosity level which has wide range of application in various fields. Still this elect spinning process needs quite a lot attention for its optimization for the production of fibers with high efficiency by optimization of various parameters. Although this electro spinning is widely used for the production of Nano fibers but still exploitation of technologies based on electro spinning is very limited due to poor understanding of the process and consequent limitation in process control, reproducibility and productivity.

### **1.2 OUTLINE OF THE PROJECT**

The current study deals with the study of brief overview of electro spinning process and the various parameters affecting the electro spinning process. Various steps for the optimization of the parameters are discussed. The project also deals with the design of digital thermometer for the environment temperature measurement which plays a crucial role in electro spun fibre yield. Digital thermometer monitors the environmental temp at a regular interval inside the electro spinning box which is later used as a part for the temperature control of the electro spinning process by heat exchange method

# **CHAPTER-2**

# **BASICS OF ELECTROSPINNING**

### 2.1 ELECTROSPINNING

Electro spinning is a process for the production of fibre by the use of electrostatic force in spite of previously used mechanical force. This method is an excellent fabrication process which can be used to form Nano fibers with diameter from 10-1000 nm from fibrous polymer materials. High voltage electric field is being used in this process to generate electro spun Nano fibers.

### 2.2 STRUCTURAL DESIGN OF AN ELECTROSPINNING MACHINE

Traditionally available needle electrospinning apparatus mainly consists of following parts

- Syringe
- High voltage power supply
- Metallic needle with an orifice at the tip
- Polymer or composite solution
- Collector electrode



Fig 2.1 SCHEMATICS OF AN NEEDLE ELECTROSPINNING APPARTUS

The whole electro spinning setup is paced in a Plexiglas box that helps in limited exposure of the whole system to the exterior. This box helps in isolating the electro spinning process from unpredictable air current and from various ambient parameters such as humidity and pressure that can alter the fibre production process. The inside of the Plexiglas box also contains acetone bath used to saturate the electro spinning environment with acetone. A computer controlled device controls the electrode separation distance. Syringe location can also be adjusted. The syringe is driven by a syringe pump which is used to control the flow rate and volume of the polymer being ejected. The syringe being used is having a capacity of 1-mL. syringe is attached to a thin tube that leads to a thin needle that ejects the polymer solution. The electrode plate was placed on a stand made of acrylic acid. The electrode is generally grounded and is used for the collection of both random and aligned fibre.

### 2.3 WORKING PRINCIPLE OF AN ELECTROSPINNING MACHINE

Basically electro spinning process can be broadly explained by 5 steps, such as-

- a) Charging of the polymer fluid
- b) Formation of the cone jet (Taylor cone)
- c) Thinning of the jet in the presence of an electric field
- d) Instability of the jet
- e) Collection of the jet

### 2.3.1 Charging of the fluid:-

Syringe is filled with an polymer solution containing the spin-dope at an angle which prevents fluid discharge from the needle under its own weight. Needle orifice also plays an important role in this. The polymer solution is charged to a very high potential around 10 KV by contact with and flow across and electrode kept at very high potential (positive or negative) known as induction charging. Depending upon the nature of the fluid and polarity of the applied potential free electrons, ions or ion-pairs are generated as the charge carriers forming an electrical double layer. This induction charging is suitable for conducting fluid. But for non-conducting fluid charges may be directly injected into the fluid by the application of electrostatic field

### 2.3.2 Formation of the cone-Jet:-

The polarity of the fluid depends upon the voltage generator. Now in the polymer solution repulsion between the similar charges at the free electrical double layer work against the surface tension and fluid elasticity to deform the droplet into a conical shaped structure known as Taylor-cone. Beyond a critical charge density Taylor-cone becomes unstable and jet of fluid id emitted from the tip of the cone.

### 2.3.3 Thinning of the jet

This jet then seeks a path to the ground. This fluid jet then forms a slender continuous liquid filament. The charged fluid is accelerated in the presence of electrical field. This region of fluid is generally linear and thin.

### 2.3.4 Instability of the jet:-

These Fluid elements are accelerated and thus stretched and succumbs to one or more fluid instabilities which distort as they grow following many spiral and distort path before collected at the collector electrode. This region of instability is also known as whipping region.

### 2.3.5 Fibre collection:-

Charged electro spun fibers travel downfield until impact with a lower potential(grounded) collector plate. Orientation of the collector affects the alignment of the fibers. Different type of collector used are- Rotating drum collector, moving belt collector, rotating wheel with bevelled edge, multifilament thread, parallel bars, simple mesh collector etc.



# **CHAPTER-3**

# FACTORS AFFECTING ELECTRO SPINNING

### **3.1 PARAMETERS IN ELECTROSPINNING PROCESS**

Electro spinning is a method is affected by various parameters. Broadly these parameters can be classified into two categories

- a) Polymer solution parameters
- **b)** Process parameters

Solution parameters	<b>B) Processing parameters</b>
Molecular weight and solution viscosity	Voltage
Surface tension	Feed rate
Solution conductivity	Temperature
Dielectric effect of solvent	Effect of collector
	Diameter of the orifice of needle

### **3.2 POLYMER SOLUTION PARAMETERS**

### 3.2.1 Molecular weight and solution viscosity:

Higher the molecular weight of the polymer being dissolved in the solvent, higher is the viscosity of the solution because with higher molecular weight the polymer chain length is higher and hence the molecular entanglement increases causing increase in viscosity. Higher viscosity prevents the electro spun jet breakup during its stretch up to collector electrode leading to continuous fibre generation. And hence solution made up of monomeric polymer can't be used to form fibers. But very high viscosity may make it difficult to pump the solution and also may lead to the drying of the solution at the needle tip lowering the yield of the process Very low viscosity may also lead to bead formation in the resultant electro spun fibre .High viscosity also lead to the formation of fibre of increased diameter because of the resistance of the solution for stretching. Higher viscosity also is the reason for smaller deposition area because it prevents bending of resultant fibre .At higher viscosity there is a stable secondary jet formation which leads fibers of smaller diameter along with fibre of higher diameter erupting from main jet

#### **3.2.2 SURFACE TENSION**

At lower viscosity Surface tension leads to the formation of bead formation along the fibre length because it leads to the decrease of the surface area. But at higher viscosity effect of surface tension is nullified because of the uniform distribution of the polymer solution over the entangled polymer molecules .Lower surface tension leads to the smooth fibre formation. Lower surface tension can be achieved by addition of surfactant to the polymer solution

#### 3.2.3 SOLUTION CONDUCTIVITY

Increased conductivity of the solution lead to the distribution of higher charge on the electro spinning jet which leads to the increased stretching of the solution during fibre formation leading to the yield of smaller diameter smooth fibre. Increased conductivity of the polymer solution also lowers the critical voltage for the electro spinning. Increased charge leads to the higher bending instability leading to the higher deposition area of the fibre being formed; as a result jet path is increased lading to formation of finer fibre. Solution conductivity can be increased by the addition of a salt or polyelectrolyte. It can also be increased by the addition of drugs and proteins which dissociate into ions when dissolved increasing the conductivity of the solution. When polymers of smaller diameter is dissolved then due to its greater mobility under external electrostatic field, there is greater elongation force leading to formation of smaller diameter fibre

### 3.2.4 Dielectric effect of solvent

Higher the dielectric property of the solution lesser is the chance of bead formation and smaller is the diameter of electro spun fibre. With increase in dielectric property there is increase in the bending instability of the jet leading the increased deposition area of fibre. As a result jet path length is increased leading to the formation of finer fibers

### 3.3 PROCESSING CONDITION PARAMETERS

### 3.3.1 VOLTAGE:

Taylor cone stability is based upon applied voltage. With higher voltage greater amount of charge cause the jet to accelerate faster leading to smaller and unstable Taylor cone. Higher voltage lead to greater stretching of the solution due to the greater columbic forces forming smaller diameter fibre. Higher voltage may be the reason of dry fibre formation because of high evaporation rate. At lower voltage the flight time of the fibre to collector plate increase leading to the formation of finer fibers.

With higher voltage there is greater tendency to bead formation because of increased instability of the Taylor cone, and even at very high voltage theses beads join to form thick diameter fibre. Higher voltage means higher electrostatic field induce better crysttalinity in the fibre. But very high voltage reduce the crysttalinity of the fibre being formed, because with very high voltage acceleration of fibre increase leading to reduced flight time and due to this polymer molecules don't find sufficient time to align themselves leading to fibre of less crysttalinity.

Instead of DC if AC voltage is provided for electro spinning it forms thicker fibers . because when AC voltage is used the different segments of jet contains opposite charge , so there is reduced repulsive force between different segment , thereby reducing the bending instability of the jet leading to the less stretching which results in higher diameter fibre. In AC supply there is less accumulation of like-charges after fibers are being deposited, which is useful for accumulation of thicker layers of electro spun fibre on collector plate

### **3.3.2 FEEDRATE**

With increased federate there is increase in the fibre diameter because greater volume of solution being drawn from the needle tip. But with increased federate there is also corresponding increase in the charges and thus corresponding increase in the stretching of the polymer solution which counters the increase in diameter due to increased federate. But with increase diameter there is increase in size of the beads because more volume of the solvents drawn and thus don't get sufficient time to evaporate leading to the fusion of the fibers which increases the bead size

#### **3.3.3 TEMPERATURE:**

Higher temperature leads to lower viscosity and higher evaporation rate. At higher temperature the fibre being produced are of uniform diameter due to lower viscosity and higher solubility which allows greater stretching of the solution. At higher temperature due to low viscosity columbic force exert a greater stretching. Increased polymer mobility due to higher temperature just helps this stretching. But due care should be taken while dealing with biological substances such as enzymes and proteins because it may be the reason for loosing functionality

### 3.3.4 EFFECT OF COLLECTOR

In electro spinning collector material is made of conductive material being electrically grounded to create stable potential difference between needle and collector. Had a nonconducting material been used then the charges on the electro spinning jet would have been developed an opposite charge on the collector reducing the amount of fibre being deposited with lower packing density. But in case of conducting collector there is accumulation of closely packed fibers with higher packing density. Porus collector yields fibers with lower packing density as compared to non-porus collector plate. In porus collector plate the surface area is increased so residual solvent molecules gets evaporated fast as compared to non-porus collector where there occurs accumulation of residual solvent around fibers leading to a dense packed structure. In porus surface the residual charges remain on the fibre repelling each fibre leading to lower packing density but in case of uniform surface residual charges conducts away to collector leading to less repulsion so more packing density. Rotating collector is useful in getting dry fibers as it provides more time to the solvents to evaporate. It also increase fibre morphology

### 3.3.5 DIAMETER OF PIPETTE ORIFICE

Orifice of smaller internal diameter reduces the clogging effect due to less exposure of solution to the atmosphere. Small orifice leads to the formation of smaller diameter fibers . However very small orifice has the disadvantage that it creates problem in extruding od droplet of solution at the tip of the orifice

# **CHAPTER-4**

# MATERIALS AND METHODOLOGY

### **4.1 MATERIALS USED**

<u>Materials</u>	<u>Number of items</u>
At mega 32 microcontroller:	1
Temperature sensor (LM35)	1
7 segment display	3
Simple SPDT switch	1
Batter (9V)	1
IC 7805	1

Soldering kit, Vero board

### 4.2 BLOCK DIAGARM:-

The following flow chart provides the general principle of operation of a digital thermometer. Each section is described in details in the following sections



The building blocks such as temperature sensor, ADC conversion and display system is explained one by one .

### 4.3 <u>TEMPERATURE SENSOR</u>

The LM35 temperature sensor is a precision integrated circuit temperature sensor whose output voltage is linearly proportional to the Celsius temperature. It can be used to measure environment temperature in degree centigrade with 0.5 degree centigrade accuracy. LM35 temperature sensor has the advantage over the other temperature measuring sensor because it doesn't use mercury (like old thermometers), bimetallic strips (like in some home thermometers or stoves), nor do they use thermistors (temperature sensitive resistors). LM35 doesn't require external calibration.

### **4.3.1 PIN CONFIGURATION AND CONNECTION**

Vcc = 4 Volt - 20 volt

Vout =  $0 \text{ mv} + 10.0 \text{ mv} / ^{\circ}\text{C}$ 

The voltage at output pin is in analog form. It is first converted into digital values. For this the output pin of LM35 is connected to the A0 of the A port (ADC port) of the At mega 32 microcontroller where it is converted into its corresponding digital values so that it can be readable by MCU

#### **4.3.2 OPERTING PRINCIPLE OF LM35**

In LM35 temperature sensor as the temperature increases the voltage across a diode increases at a constant rate because of the voltage drop between the base and the emitter. This voltage change is directly related to the temperature change.

#### THE OUTPUT VOLATGE IS 10 MILLIVOLT PER DEREE CENTIGRADE

#### So temperature in ° C= (Vout in millivolt) /10

The analog output of the LM35 temperature sensor is converted into corresponding digital values using the ADC port of ATMEGA 32 microcontroller

LM35

FIG

### 4.4 ANALOG TO DIGITAL CONVERTION

The voltage output of the LM35 sensor is in analog in analog domain so that it can be read, calculated and stored in the microcontroller. Most of the modern MCUs contains in build ADC port. In At mega 32 also there is an inbuilt ADC port (Port A). Port A has 8 inputs( PA0-PA7). That means it can take analog signals from 8 different sensors at same time and can convert them to its digital domain. In At mega 32 microcontroller the ADC is of 10 bit resolution. So it has a range of 0- 1023. That means the whole Vcc (5V) is divided into 1023 parts. The reference voltage is set at maximum input voltage i.e. 5V

When Vin = GND, the ADC value = 0

Vin= Vref(5 V), the ADC value= 1023



ARef=5V

### **4.5 CALIBRATION AND SIGNAL PROCESSING**

In At mega 32 microcontroller ADC is of 10 bit resolution and Vref= 5 V. hence

#### Resolution in voltage = 5/1024 = 4.88 mv

So first we need to calculate the ADC value to calculate the voltage. ADC value can be calculated using the command ReadADC (ch). Where Ch. is the channel number. If the analog input is taken at channel 0 (PA0) of At mega 32 microcontroller then

#### adc\_val= ReadADC (0)

This will store the ADC value in adc\_val and its range can be from 0-1023 Now we have already see\n that for At mega 32 microcontroller with 0 bit resolution ADC port 1 unit of ADC value= 5 mv approx..

#### 5 mv= 1 unit of ADC

#### 10 mv =2 unit of ADC

Now for LM 35 temperature sensor

 $1 \circ c = 10 \text{ mv}$ 

So  $1^{\circ}$  C= 2unit of ADC

Hence form this it can be derived that

Temp= (adc\_val)/2

### 4.6 DISPLAY SYSTEM (SEVEN SEGMENT DISPLAY)

Seven segment display is used to display numerical output in many electronics devices. Seven segment display is divided into 7 different segment which can be switched on or off independently according to a user defined program. Different combinations of segments switched on produces different numbers. 7 segment displays also contain a decimal point. Basically the seven segment display contains 7 segments (a,b,c,d,e,f.g) and a decimal point(DP).





Fig 4.3 seven segment display Pin configuration

Fig 4.4 Real time picture of a seven segment display

The one common end of all the LEDs are connected while the rest are available. Depending upon whether anode or cathode of all the LEDs are connected these are of 2 types, -

- a) Common cathode seven segment display
- b) Common anode seven segment display



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### 4.6.2 PIN CONFIGURATION OF SEVEN SEGMENT DIPALY

Seven segment display contain 10 pins. The middle two pins are internally connected and rest ach pin is the free end of each LED.

Three common anode seven segment displays are connected in parallel. But first the first cell is powered and the other two remain unpowered. Then the second one is powered on keeping the other two off and same for the third seven segment display.



# CHAPTER-5

# **RESULTS AND DISCUSSIONS**

### **5.1 RESULT**

After soldering all the components and powering on by 9V power supply the temperature sensor reads the atmosphere temperature converts it into analog form and then send it to the microcontroller where it is converted into its corresponding digital values. Then according to the calibration and formula provided in the chapter 4 we can measure the temperature in degree Celsius. The temperature values so detected are shown in three seven segment display as described above.

The main aim to design thermometer for measuring atmosphere temperature is that it can give visual and easily understandable atmosphere temperature. So that we can maintain the inside temperature of the Electrospinnin box to the optimal value so that fibre morphology can be used. In future prospect The whole electro spinning box can be surrounded with the pipes which can flow hot or cold water according to our need to either increase and decrease the inside temperature of the Electrospinnin box to get optimal temperature value.,. And thermometers becomes handy in measuring the inside atmospheric temperature.

### EFFECT OF TEMPERATURE ON FIBRE MORPHOLOGY

Higher temperature leads to lower viscosity and higher evaporation rate. At higher temperature the fibre being produced are of uniform diameter due to lower viscosity and higher solubility which allows greater stretching of the solution. At higher temperature due to low viscosity columbic force exert a greater stretching. Increased polymer mobility due to higher temperature just helps this stretching. But due care should be taken while dealing with biological substances such as enzymes and proteins because it may be the reason for loosing functionality

Different temperature affects fibre morphology. Here I have provided 3 different SEM images of the fibre being produced at different temperature (T1, T2, and T3) and the different fibre morphology is clearly visible in the pictures. T1>T2>T3. Frome the SEM images it is clearly understood that with increasing temperature fibre diameter is reducing because of lowering of viscosity. But temperature should not be kept very high because it may lead to the melting of the electro spun fibers being produced



FIG 5.1 SEM OF ELECTRO SPUN FIBER AT TEMP T1



FIG 5.2 SEM OF ELECTRO SPUN FIBER AT TEMP T2



## Fig 5.3 SEM OF ELECTRO SPUN FIBER AT TEMP T3

Below are the two snapshots of my thermometer with two different temperatures -





### **5.2 PARAMETERS FOR OPTIMIZTION OF ELECTROSPINNIN PROCESS**

From chapter 3 we came to know about various parameters that affect the electro spinning process. By changing these parameters value we can get fibres of our need. Its morphology, diameter, structure can be adjusted by varying the value of the parameters. Here I have tried to classify these parameters in simple way according to the change we want to make

### 5.2.1 TO INCREASE THE FIBER DIAMETER

- Viscosity of the solution should be increased
- Surface tension of the solution should be decreased by the addition of various surfactant
- Conductivity of the polymer solution should be increased either by the addition of polyelectrolyte or by the addition of drugs and proteins
- Voltage supply should be decreased to reduce the stretching
- AC supply should not be used as it always results thicker fibers
- Feed rate of the solution should be increased
- Operating temperature should be low
- Diameter of needle orifice should be high but shouldn't be very high enough

### 5.2.2 DECRESE IN BEAD FORMATION

- Viscosity of the polymer solution should be low
- Solution conductivity should be increased by addition of polyelectrolyte or by addition of drugs and proteins
- Voltage supply should be high enough
- Feed rate should be increased
- Operating Temperature should be high
- Rotating or moving collector can be used to increase the flight time to decrease bead size
- Diameter of the needle orifice should be low
- Distance between the needle tip and collector should be decreased

### 5.2.3 SMALLR DEPOSITION AREA

Smaller deposition area is always desirable in Electro spinning process. Because if the deposition area is larger then fibers may not be collected at the electrode completely leading to the wastage of fibre and also the fibre diameter also decreases due to increased flight time. Smaller deposition area can be achieved by

- Viscosity of the solution should be increased
- Conductivity of the polymer solution should be decreased
- No addition of surfactant should be there so that surface tension should be kept minimal
- High voltage which leads to higher field strength

### **5.3 OUTLOOK**

Further different modification of the existing electro spinning machine can be achieved by various methods. Here is the brief overview of what can be done further

- A) Use of heat exchanger system around the box of the electro spinning to regulate the temperature of the electro spinning process and thereby regulating fibre morphology
- B) Use of rack pinion mechanism to control the distance between the TIP and collector so that fibre morphology can be adjusted according to parameters and our need
- C) Use of rack pinion mechanism to move the collector to control the fibre alignment thereby reducing fibre wastage by reducing the deposition are
- D) Use of High electric field sidewise on the fibre jet so as to compress it decrease the width of the jet thereby reducing the deposition are which ultimately reduce fibre wastage

# CHAPTER 6 CONCLUSION

### **6.1 CONCLUSION**

The study reports the various parameters that need to be modified according to the need of the process for the production of Nano fibers with high yield and optimum quality according to the use. Also successfully digital thermometer is designed for the design of environment temperature. This can be further used for the real time temperature measurement during electro spinning process so that by temperature feedback method electro spinning temperature can be set to optimum value.

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