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Low Cost Emulsion Electrospinning Apparatus for Encapsulation of Bioactive Compounds

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***Abstract*---Bioactive compounds in foods are a growing area of research in today's world due to the lifestyle diseases and its numerous health benefits. There are a wide variety of compounds that can be incorporated into foods and encapsulation is one of the most successful methods. Electrospinning is said to be the best methods for obtaining nanofibers through encapsulation which can find its way directly into any nutraceutical and functional foods. Even when electrospinning is the most efficient option, the high cost stands as a limitation. So, the purpose of our project work was to design and develop a low cost electrospinning apparatus suitable for emulsions. The work involved setting and fabricating the components of electrospinning and assembling them to produce the desired results. Different approaches were taken and an Arduino controlled programmable syringe pump, high voltage power supply using flyback driver and MOSFET, and Aluminum plane plate collectors were fabricated at a low cost. Any kind of emulsion based bioactive compounds can be encapsulated using the device developed and we have used omega-3 enriched fish oil emulsion. Fish oil emulsions possesses large advantages but has limited applications due to oxidation. Therefore, electrospinning stands as the best approach for preserving the stability and availability of fish oil. The successful system validation was done using PVA and later fish oil, was successfully encapsulated and nanofibers were obtained. The nanofibers can be incorporated into infant foods and formulations which can be widely used.**

***Keywords*--- Bioactive compounds, Electrospinning, Fish oil emulsion, Flyback Driver, MOSFET, Arduino, Syringe Pump, Omega-3**

I. INTRODUCTION

The sweep of application of bioactive compounds ranges from antibodies, anticancer agents, neutraceuticals, enzyme immobilizer, food packaging material to biosensors. Even though their usage is often restricted by their unfavorable flavor, poor stability and solubility during food process, uncontrolled release profile, and low bioavailability in the upper gastrointestinal tract (GIT) [10]. An apt method for entrapping bioactive compounds is encapsulation, as it can safeguard these compounds from adverse environmental conditions or from the GIT (e.g. stomach acid), gain controlled release profile, intensify the solubility or dispersibility of lipophilic compounds and ameliorate the bioavailability of bioactive molecules during digestion [1, 2].

Methods including electrospinning, gelation, coprecipitation, layer-by-layer deposition, extrusion, coacervation, spray/freeze-drying, and emulsion formation have been reported for encapsulation of food ingredients. Among these approaches electrospinning represents a adaptable method for the production of micro and nano sized fibers and has been proposed as a practicable route for the encapsulation of various bioactive compounds like fish oil which own noteworthy nutritive values [6, 15]. The electrospinning process make use of high-voltage electric fields to fabricate electrically charged jets of bioactive compound loaded solutions and results in ultrathin fibers on the evaporation of the solvent.

Omega-3 polyunsaturated fatty acids (PUFA) are the major bioactive compounds present in fish oil, which have countless beneficial health effects such as improvement of the anti-inflammatory response, avoidance of cardiovascular disease, and development of brain and eye retina in infants. Since humans have a low conversion rate of the essential linolenic fatty acid (ALA) to EPA and DHA, these PUFA need to be ingested through the diet. Thus the development of a system to substitute omega-3 PUFA delivery, which is easy to diffuse and which lead to improved oxidative stability of omega-3 enriched food products are required [5]. Hence we selected fish oil as our process solution.

The laboratory setup, which we have developed consists of a syringe with needle, syringe pump which extrudes the polymer solution from syringe- needle assembly at defined flow rate, a high DC voltage supply usually in the range of 1-40kV with positive and negative polarity for charging the polymer solution and the conducting plane plate collector, for collecting the nanofibers [11]. The experiments were

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carried at varying conditions of solution flow rate, fish oil polymer solution concentration, collector-to-needle tip distance and voltage supply to optimize the parameters essential to obtain fine nanofibers from fish oil-polymer solution.

II. MATERIALS AND METHODS

The electrospinning apparatus (Fig. 2.1) consist of mainly 3 parts. They are a high voltage power source, syringe pump and a collector. Fish oil emulsion is used to run the apparatus to produce fibre. Our objective is to design and develop an cost effective electrospinning apparatus by producing cost effective components of the apparatus. This apparatus can be used to encapsulate any bioactive compounds and we have used fish oil emulsion to encapsulate.

2.1. Syringe Pump

A syringe pump is a device used to transfer small amount of liquid accurately. The syringe pump is driven by the stepper motor. 3D printed chasis forms the frame work of the syringe pump onto which stepper motor is fixed. The stepper motor rotates the lead screw threaded through a 3D printed pushing block. The two linear rod keeps the pushing block in horizontal direction. The liquid ejects from the tip of the syringe as the pushing block, holding the plunger, moves forward.

Arduino uno is used to operate the stepper motor. The program to run the stepper motor to deliver required amount of liquid is uploaded onto the Arduino uno [3, 4]. The assembly of syringe pump is shown in fig. 2.2.

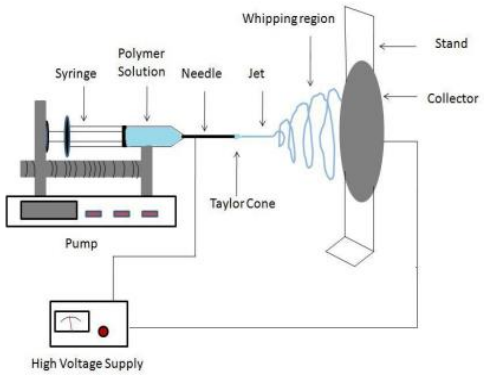

Figure 2.1. Schematic diagram of electrospinning setup consisting of syringe pump, high voltage power supply and the collector (adapted from [8])

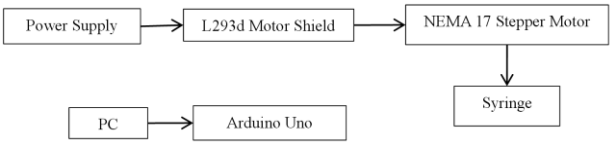

Figure 2.2. System diagram of syringe pump

2.2. High Voltage Power Supply

High voltage source is required in electrospinning to obtain long nanoscale fibers. High voltage determines bead and fiber formations. High voltage required varied based on compounds and we require around 20kV for successful fish oil encapsulation.

We developed a high voltage power supply initially using CFL and a flyback transformer and performed trial runs using chitosan. This method was unsuccessful as within short span of time the CFL was blown out due to high voltage generation when capacitor was introduced also a DC supply was required. So a new supply was designed using flyback driver [14].

2.2.1. Flyback driver using power MOSFET

This high voltage supply consisted of a flyback transformer with switching frequency of 15kHz, IRFZ44N MOSFET, rrechargeable battery (3.7V, 2A), resistor of 220 ohm and a switch.

The low voltage DC applied to MOSFET can generate high voltage which can drive a flyback. The flyback consists of primary and feedback windings, which were identified through continuity check using a multimeter and resistances were also analysed. High voltage applied to the primary winding of flyback created electromagnetic induction which is stored in the capacitor and on reaching secondary coil high frequency high voltage low ampere output is obtained. The circuit is self-oscillating and generates high voltage in the form of a plasma arc. From the dielectric breakdown theory of gases, we found out that the 2cm arc formed indicates 20kV which was efficient for our purpose. Connections were made as per the circuit diagram shown in fig. 2.3 [16].

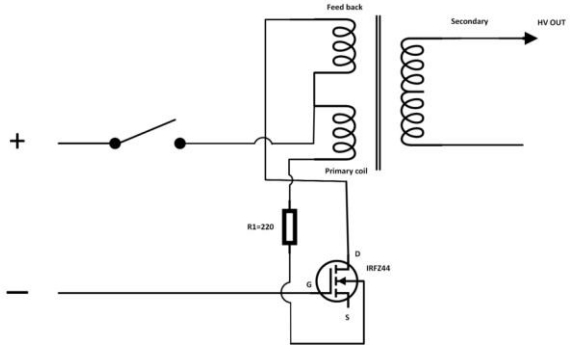

Figure 2.3. Circuit diagram of flyback driver

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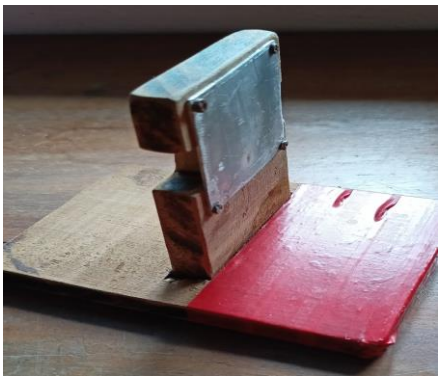

Figure 2.4. Plate collector

2.3. Collector

Collectors (fig. 2.4) are designed to collect fibre ejected from the Taylor cone. Collectors may contain stationary or rotating platform. A simple plane plate collector with rectangular aluminium plate (5×5 cm) is used in electrospinning apparatus. The aluminium plate is attached to a wooden base [11].

2.4. Solutions for conducting trail

The working of the electrospinning apparatus is examined using two solutions. They are,

a) Poly Vinyl Alcohol (PVA) - PVA (15% w/w) and acetic acid (1% w/w) are dissolved in distilled water (adjusted at pH 2 by addition of 0.1 N HCl) at 80 °C under constant stirring for 3 hrs. The solution is allowed to cool to room temperature under stirring for 2 hrs and then overnight without stirring [14].

b) Fish oil in PVA emulsion - Aqueous phases are prepared by dissolving Whey Protein Isolate (WPI) (1%, w/w), which is adjusted to pH 2 by addition of 0.1 N HCl and stirred overnight at 5 °C. Primary homogenization is done by adding the fish oil slowly to the aqueous phase and mixing at 16,000 rpm for 3 min. PVA (15%, w/w) and acetic acid (1%, w/w) are dissolved in distilled water (adjusted at pH 2 by addition of 0.1N HCl) at 80 °C under constant stirring for 3 hr. This PVA polymer solution is blended with fish oil-in-water emulsions in order to obtain electrospinning solution with required PVA concentration [10].

III. RESULTS AND DISCUSSIONS

A fully functional prototype device was built as depicted in fig. 3.1 and the results were demonstrated. The results were obtained after performing and adopting the methodologies as described earlier. Each of the components were separately designed and calibration and validation of each were conducted and discussed catering to our specific needs. The observations and analysis were done and summarized thereof.

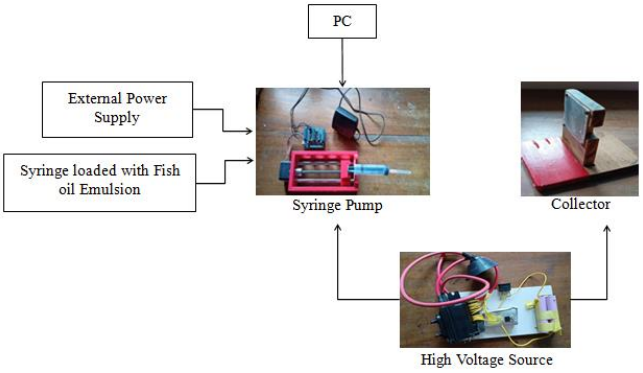

Figure 3.1. An experimental setup for electrospinning apparatus

3.1. Final Electrospinning Setup

A typical horizontal electrospinning system developed in this project is illustrated in Fig. 3.2. It consists of a low cost Arduino controlled programmable syringe pump which extrudes the polymer solution from the 20 ml syringe (Dispo van) at a definite flow rate. The syringe encapsulates a 4 inch 18 gauge blunt tip metal. Constant high voltage was provided by high power supply developed using flyback driver and power MOSFET. The needle and the collector were connected with the positive and the negative terminal of the high voltage DC power supply, respectively. The height and distance of the collector plate on the stand can be varied by adjusting and sliding the collector plate according to the fibre formation. The flow rate was varied according to the solution and kept at 0.02 mL/min for encapsulation of fish oil and voltage supply maintained at 20 kV [11]. Collector distance was varied until beadless nanofibers were obtained which was 4 cm. In order to verify the functionality of the setup system validation was done using PVA and beadless nanofibers were obtained.

Figure 3.2. Electrospinning Apparatus

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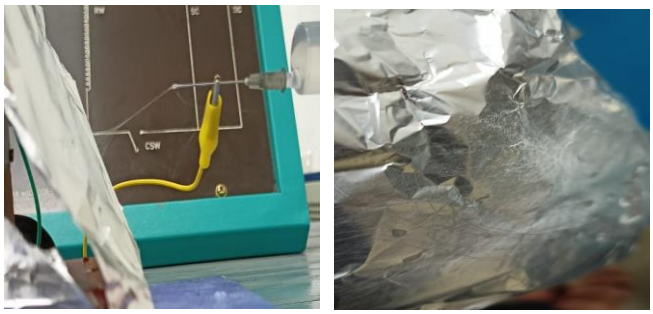

(a) (b)

Figure 3.3. PVA fiber deposition (15% PVA) - (a) emerging from needle to aluminum foil placed in collector at 0.01mL/min and 3.5 collector distance (b) in aluminum foil at 0.02mL/min and 3.7 cm collector distance.

3.2. System Validation using PVA

The developed electrospinning system was validated using preparing a PVA solution as mentioned in the methodology. 15 % PVA solution maintained at a constant voltage of 20 kV was allowed to run at varied flow rates of 0.01 and 0.02 mL/min, and at varied collector distances of 3.5 and 3.7 cm.

Different parameters like operating voltage, concentration, collector distance and flow rate had profound effect in fiber formation. Based on the previous experiments conducted in chitosan electrospinning, we concluded that 20 kv is the optimum voltage required for proper fiber formation and at both high and low voltage, bead formation occurred [12]. So, we studied the effect of flow rate and collector distance in PVA fiber formation and graphical analysis was done.

From the validation we analyzed that at 0.01 mL/min, beads were formed along with the fiber and at 0.02 mL/min, more thinner fibers were formed with less beads. Also optimum fiber formation was obtained at 3.7 cm. So an efficient fiber formation was obtained at 0.02 mL/min at a collector distance of 3.7 cm which is shown in the fig. 3.3.

3.3. Electrospinning Of Fish Oil In PVA Emulsion

Electrospinning of fish oil were conducted at different flow rates, collector to tip distance and different PVA to oil emulsion concentration. Emulsion containing 5% fish load and PVA are prepared in 3 concentration i.e., (i) 7.5% PVA - 2.5% oil emulsion, (ii) 10.5% PVA - 1.5% oil emulsion and (iii) 70% PVA - 10% oil emulsion, are exposed to different conditions for examining optimum process condition. Process was done at multiple flow rates, collector distances and concentration while 20 kV voltage was maintained. We arrived at optimum results after performing several trials.

We concluded that for 70% PVA solution optimum fiber was formed at 0.02 mL/min and at a collector distance of 4 cm. In case of 10.5% PVA, fiber formation was quite less compared to 70% PVA even when conditions were varied which is depicted through a graphical analysis on fig. 3.4. Also we found out that at lower concentration of 7.5% (w/w) PVA - 2.5% (w/w) oil, more beads are formed with little amount of fibre and as concentration was increased to 70% (w/w) PVA - 10% (w/w) oil, more thinner nanofibers were obtained. The results of fiber formation at optimum condition for different concentration od PVA and fish oil emusion are shown in fig. 3.5.

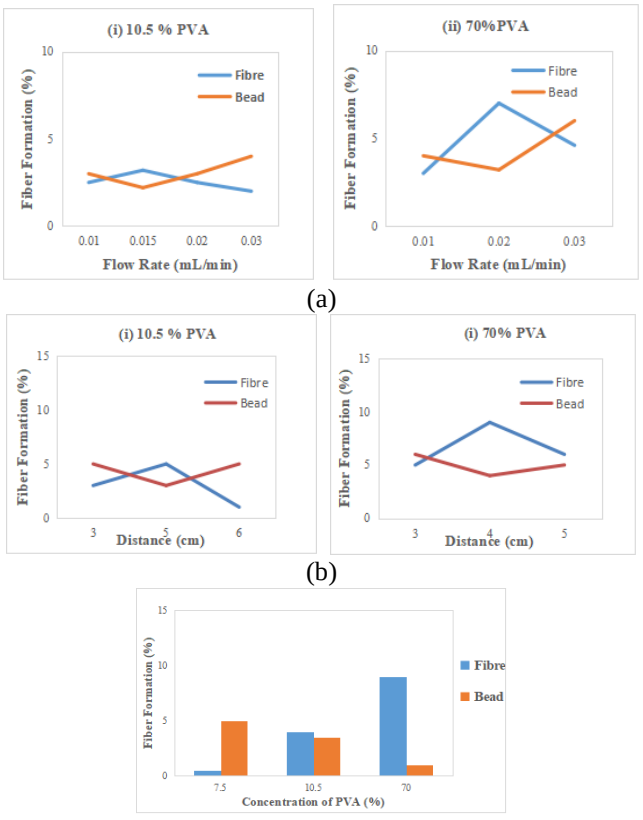

Figure 3.4. Extent of Fibre/Bead formation vs (a) Flow rate of solution, (b) collector to tip distance and (c) concentration

(a) (b)

(c)

Figure 3.5. Results of fish oil in PVA emulsion electrospinning (a) 7.5% (w/w) PVA - 2.5% (w/w) oil emulsion, (b) 10.5% (w/w) PVA - 1.5% (w/w) oil emulsion and (c) 70% (w/w) PVA - 10% oil emulsion

3.4 Cost Estimation

Electrospinning is an expensive process and a single unit electrospinning machine in the market cost around 10 lakhs. So we have adopted an innovative and feasible way and developed the machine by combining each parts. We have successfully developed all the components at a low cost with cheap and novel materials. The total equipment cost was only around 8K rupees. The equipment can also be improved through many methods and can be directly used in the market for research purposes and for encapsulation techniques at a very low cost. This is an immediate requirement of today's world where healthcare and nutraceutical industries are moving at a large pace.

IV. CONCLUSION

In the present scenario, bioactive compounds and its application in nutraceuticals is gaining demand. But a feasible and cost effective approach didn't came forward. So we have developed a low cost system which can encapsulate any bioactive compounds. We conducted literature studies and found out that electrospinning is the most efficient method for encapsulation. We found out that the electrospinning parameters like flow rate, collector distance, concentration of solution and electric field significantly affects the fiber formation [7]. Optimum conditions were achieved by conducted using trial runs and were plotted graphically for better understanding. The nanofibers developed possess high encapsulation efficiency and has a high oil load compared to the other methods. Also they are not prone to oxidation which is the major issue with fish oil. We could successfully produce nanofibers enriched with omega-3 and could utilize its health benefits. The fibers can be used in infant formulations, immune boosters and in capsules where the foul smell and taste of fish oil will not be present while all the benefits present in the fish oil will be utilized. Successful encapsulation of omega-3 delivery systems was done and studied using the designed model [5, 13]. Apart from omega-3 delivery systems, any kinds of emulsion based bioactive compounds like nanocurcumin, gingerol can be successfully encapsulated for nanofiber production. So there is a definite scope of improvement for the developed model by bringing out changes in the fabrication and design.

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