EHDA closed loop control system based on real time non-visual spray mode classification

Monography

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Abstract

Electrohydrodynamic Atomization (EHDA), also called electrospray, is a liquid atomization technique that produces micro and nanometric charged droplets within a narrow size distribution by using high electric fields (kV/cm). According to Cloupeau and Prunet-Foch[1] (1994), electrosprays can generate droplets in different ways, which the authors named "electrospray modes". These modes may be adjusted by varying the strength of the electric field and flow rate, but also depend on liquid properties and system geometry. In their work, the authors proposed four possible EHDA modes: dripping, intermittent, cone-jet and multi-jet, which are generally distinguished visually.

This project develops a closed-loop control method for EHDA devices that uses real-time, electric current-based (hence non-visual) spray mode classification. The proposed electrospray system is entirely automatic, where all the peripherals, such as HV power supply and syringe pump, are controlled by a computer which executes their routines. The system classifies spray mode dynamics using real-time current data and changes EHDA operating parameters such as liquid flow rate and applied voltage to achieve and maintain the chosen spray mode. The electrospray modes are validated in real time by using a high-speed camera. As compared to conventional manual approaches, the control algorithm promises higher accuracy and lower transient time. Therefore, a completely autonomous EHDA system opens the door to potential industrial applications. In addition, the use of the electric current signal can be useful to further research in electrospray processes, leading to better control on droplet generation (frequency, size and charge).

Acknowledgments

I would like to express my heartfelt gratitude to the following individuals and institutions who have played a significant role in shaping me into the person I am today:

First and foremost, my beloved mother, whose support and belief in my potential has been my guiding light throughout my life. Her invaluable lessons on dedication, patience, and discipline have helped me overcome countless challenges and achieve my goals.

I also extend my gratitude to my father, whose teachings on the virtues of ambition have inspired me to strive for excellence. His relentless pursuit of success has been a constant source of energy for me.

I would like to acknowledge the outstanding education and training that I received from the UFMG university, which has equipped me with the necessary skills and knowledge to thrive in the global marketplace. I am grateful for the transformative experience that has prepared me for a fulfilling career and a meaningful life.

Moreover, I cannot forget my dear friends from República Casassanta, who have made my student life such a unique and unforgettable moment. Their camaraderie and support have been a source of joy and comfort during challenging times.

Lastly, I would like to thank myself for having the courage and resilience to pursue my dreams despite my anxieties and doubts. Through my perseverance, I have been able to savor every moment of my journey and give my best to achieve my goals.

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Chapter 1

Introduction

Electrohydrodynamic Atomization (EHDA) is a way to disintegrate a liquid into droplets by exposing it to a strong electric field.[1] The balance between forces on the charged liquid meniscus defines the electrospraying dynamics and droplet size. The electric current transported by the spray reveals characteristic shapes for different spray modes. Signal processing techniques can allow a non-visual classification of the spray mode based on the electric current shape.[5] The spray process imposes noise and random sequences on the measured signal making its classification a non-trivial task. Industrial applications demand automated stabilization of a spray mode. This can be achieved by a closed-loop control system. This project targets the development of a system which allows real time acquisition of the electrospray current, treat and process its values, define in which specific mode the electrospray is running and control the necessary hardware to change and/or stabilize the spray at a desired mode.

1.1 Motivation and Justify

EHDA research has contributed as an important tool for the development of technology. The advantage of using electrospray is precision and uniform size and shape of droplets creation. Specially in certain spraying modes.

Although there are applications of EHDA in industry, the stabilization of the conical jet spray mode is mostly done empirically and based on average current measurements.

Many applications such as gas odorization, spray coating and pharmaceutical industries require automated stabilization of a spray mode. This can be achieved by a closed system loop control system. An automated spray mode classification is a crucial part of the control system to work, as well as the development of an appropriate control algorithm.

1.2 Project Goals

This project aims to give continuity to the previous student work[6], Mônica, who focused in detecting undesired discharges (sparks) in the system accusing high electric potential. For that, she developed a python software routine to connect most of the peripherals and analyzed the current data. Her work corroborates the validation of this project motivating its continuation on development and optimization.

Bellow are shown the main goals listed:

Multipurpose applications for both scientific and industrial approaches.

Fully automated and intuitive system for EHDA.

Real time non-visual classification of electrospraying modes.

Control and stabilization on a desired spray.

System portability and versatility.

1.3 People involved

The NHL Stenden Water Technology group has been involved in previous projects that have successfully implemented automated signal processing techniques, resulting in highly ranked outcomes. However, further research is required to enhance the accuracy of the classification algorithm.

In order to achieve this, the Water Technology Group at NHL Stenden University of Applied Sciences, in collaboration with Dutch companies, is conducting extensive research and implement appropriate classification algorithms. The aim is to combine analytical capabilities with infrastructure knowledge and availability to achieve optimal results.

As a student from UFMG, I am now actively involved in this research project to improve the automation usability, classification accuracy and system stabilization with signal processing techniques.

1.4 Document Structure

This document is divided in 6 chapters.

Chapter 1 provides an introduction to EHDA and explains the inspiration behind the project.

Chapter 2 delves into the literature concepts and knowledge that were utilized in this project.

Chapter 3 describes the system that was implemented to make this project work, along with the instruments and models used to apply our methodology.

Chapter 4 presents the methodology applied in our experiments to validate our system.

In Chapter 5, we present our results and engage in discussions comparing the knowledge acquired through literature review.

Finally, in Chapter 6, we conclude our document with discussions about the goals achieved and areas for potential optimization.

Chapter 2

Literature Review

2.1 EHDA

The electrospraying of liquids herein is referred to as electrohydrodynamic atomization (EHDA). The atomization by primarily electrical (electro) forces of a liquid (hydro) that is moving (dynamic) during the atomization captures the essence of the phenomena.[7] That motion applies to the liquid certain velocity that is not enough to create the spray alone. Therefore, the electric field itself is the responsible for the spraying dynamics.[1] A stable balance between the capillary and field forces on the liquid suggest a *quasi static* dynamics. The feeding liquid is pulled through a small orifice by means of the applied voltage. The droplets are accelerated towards the grounded electrode by the electric field. The resulting spray is electrically charged, and the droplets repel one another. For this reason, with a controlled environment we can reach a certain stable spraying mode as can be seen in the Figure 2.1.

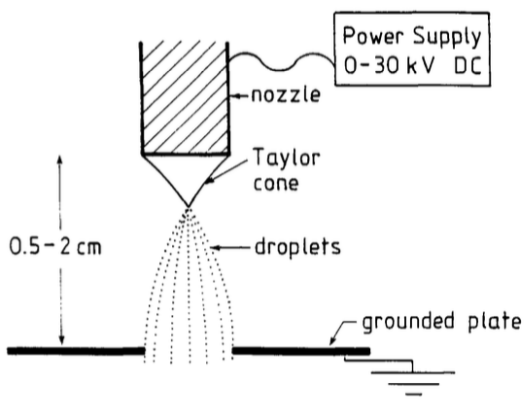

Figure 2.1: EHDA physical concept[2].

2.2 Non-visual classification

From the inception of EHDA until the present day, research has been carried out through manual means, involving the use of visual classification to determine the spraying mode either through cameras or by direct observation. It is advisable to employ a high-speed camera (HS) to accurately capture the spraying process as certain intermittent or dripping states may occur at a high frequency and be erroneously perceived as a stable condition. The setup in figure 2.2 shows the most common setup used for EHDA researchers.

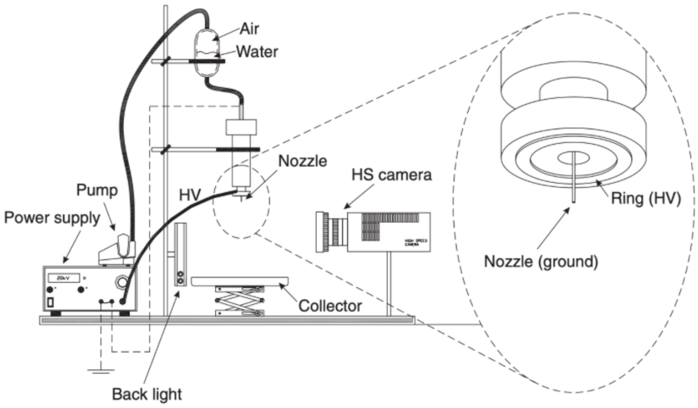

Figure 2.2: EHDA experiment setup[3].

Therefore, many researches were made about the classification of the spraying mode measuring the current flowing through the nozzle to plate[5][8]. Evaluating the current data can give us valuable information about the spraying behavior. Figure 2.3 illustrate a sample of current measured in our system during a dripping of a charged liquid. We can visually identify the signal of two droplets generated in this time frame.

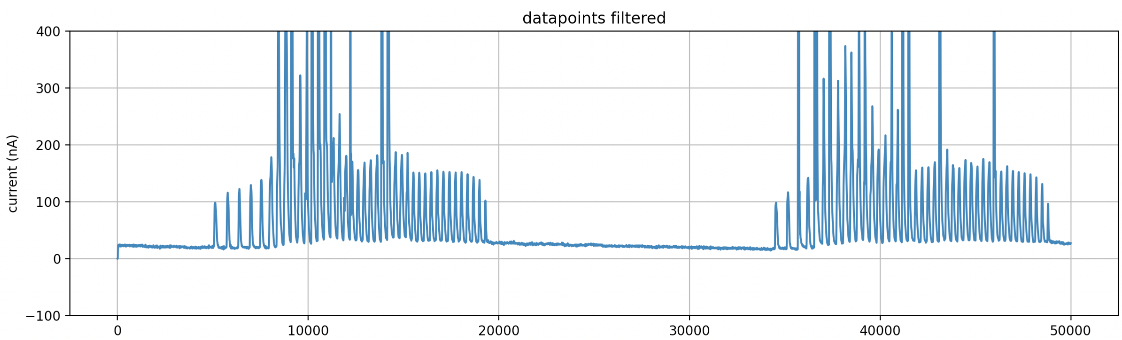

Figure 2.3: Current measurement sample of dripping spraying mode. This graph represents 0.5s sample.

The existing signal contains valuable data not only about dripping but also other modes of spraying, thus it can serve as a non-visual classifier. This project draws significant inspiration from Sjaak's[5] work, which identifies distinct statistical characteristics of electrical current for various spraying modes. A profound use of his method will be explained in section 4.1. Together with the current characteristics, visual observations and results from literature it was investigated whether generic trends are present that can be related to the actual spraying modes.

2.3 Scaling Laws

The electric spray current is a critical variable that has been extensively researched by multiple authors[1][8][4], resulting in scaling laws that express the relationship between liquid flow rate, conductivity, surface tension, density, and droplet diameter. These scaling laws can predict the size and charge of emitted droplets, making them useful tools for electrospraying practitioners. Obtaining scaling laws is necessary for characterizing an electrospraying system and gaining a better understanding of the process mechanisms. In equation 2.1 we have the current flowing through the charged liquid during a Taylor[9] cone jet.

$$I = (\frac{\gamma K Q}{\kappa})^{1/2} \tag{2.1}$$

2.4 Spraying modes

Since 1915 with his pioneering work in EHDA, Zeleny[10] observed several functioning modes with very different characteristics. Years later the same phenomena was noticed by other scientists, but the classification of these modes were still not well-defined by the community. For that Cloupeau and Prunet-Foch[1] proposed spray mode classifications based in what they have seen experimentally, and it's still being used as basis for EHDA researches.

Figure 2.4 shows 3 spraying dynamics that we are most interesting in this project.

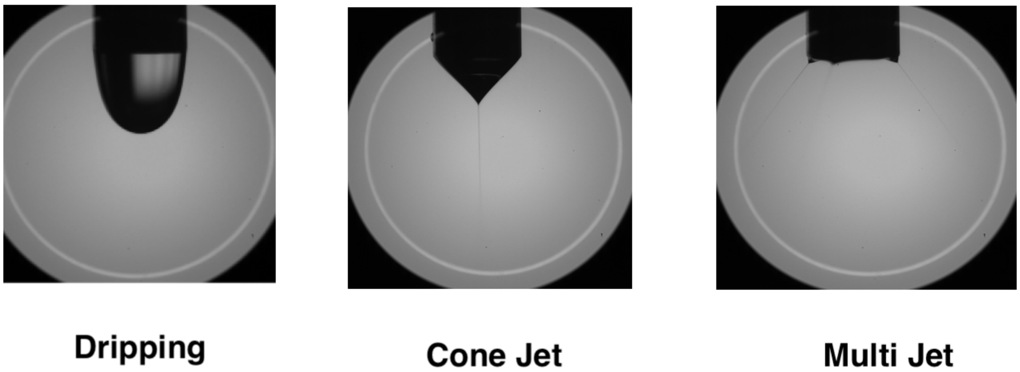

Figure 2.4: Fundamental electrospraying modes.

Through the various classifications and subclassifications of spraying defined in literature we are going to aggregate some of them and separate between 5 modes as reffered below in order of growing electric potential:

2.4.1 Dripping

Dripping mode happens when the electric field applied is not enough to change the meniscus shape, phenomena called field enhanced dripping. In that situation the liquid droplet has, in general, size bigger than the capillary and low frequency intervals between each drop.

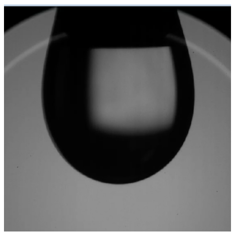

Figure 2.5: Dripping examples.

2.4.2 Intermittent

Intermittent mode is defined when the electric field forces start to have a considerable effect in the meniscus and droplet formation. This mode can be identified by its pulsating manner. In this mode the droplet size is smaller than the nozzle, phenomena called microdripping, and the dripping frequency increases with the increasing of the field applied. Figure 2.4.2 illustrates 3 different dynamics herein considered as intermittent.

Figure 2.6: Intermittent examples.

2.4.3 Cone Jet

The Cone jet mode is a particular interest in applications, where the meniscus emits a steady microscopic jet, resulting in uniformly sized droplets. Taylor (1964)[9] was the first to demonstrate that electrostatic pressure and capillary pressure can be balanced at any point on the surface of a liquid cone. The electrified meniscus can take on a conical shape within a certain range of applied voltage and injected liquid flow rate.

In Taylor cone, here referred as Cone Jet, the direction of the cone apex also varies depending on the balance of forces. With a fixed flow of liquid it is noticeable that the pointing direction curves and the meniscus reduce volume as we increase the electric potential until the moment the jet breaks into two or more jets going to the next classification.

Figures 2.4.3 shows three cone jets in different circumstances.

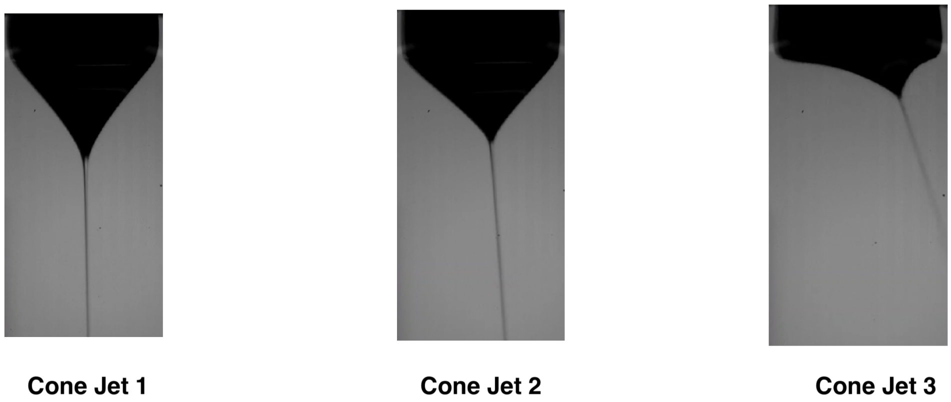

Figure 2.7: Cone Jet examples.

2.4.4 Multi Jet

Zeleny's (1915)[11] paper was the first reference to multi-jet phenomena. In his experiments was observed: *"for voltages well above that for which the steady conical surface changed into a blurred agitated one, the appearance became a number of fine points with their attendant dark streaks arranged along the circumference of the raised edge"*.

In recent times, researchers have made significant advancements in understanding multi-jet technology and have identified various profiles with distinct characteristics. Because of that, some authors can divide what we call multi jet in more classifications. The multi jet mode is considered in this work when two or more spraying jets can be visualized in the HS camera and the current shows a stable profile.

C.N. Ryan (2012)[12] focused his work in multi jet spraying mode, and its current properties. He detected that for a low number of jets emitted the meniscus is not bound to the rim of the capillary. This mode is seen to be less stable, in that each jet in multi-jet mode is unable to support the same volumetric flow as in the single jet mode. As the number of jets increases the mode becomes what Ryan refers at rim-mode electrospray. In this work, all modes with more than one stable jet will be classified as multi jet. Figure 2.4.4 shows three different variations of multi jet spraying mode.

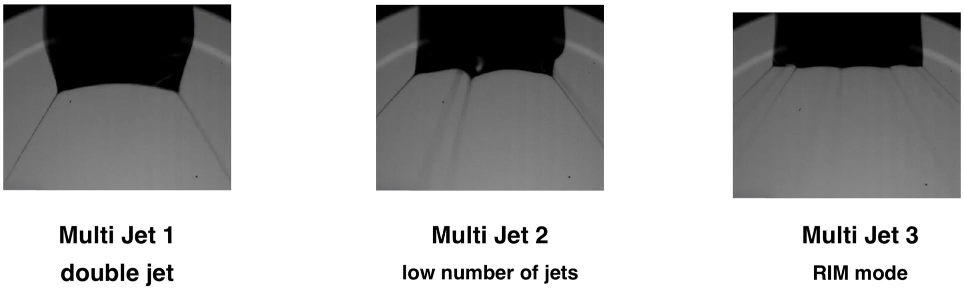

Figure 2.8: Multi Jet examples.

2.4.5 Corona

At even higher electric potentials, above of multi jet, the insulation between the electrodes start to breakdown, and we achieve a phenomenon of discharge. Those can be easily classified because of its high current value.

This mode is also divided by other classes such as streamer corona or transient sparks discharges, but it's not in the scope of this work. Mônicas[6] work was focused on how to predict that mode with the current analysis for safety in industrial applications.

2.5 Chapter conclusion

In this chapter we exhibit some electrospray theory that was important for the development of this project. Firstly, with a brief review about the physical concept behind the experiment. Then, I introduce how current analysis can be useful for our application citing works already done with this approach. Finally, I end up the chapter defining what spraying modes classification will be considered and its properties.

Chapter 3

System Description

In this chapter, we will discuss the equipment used in detail, including its integration and how it was modeled and implemented in both hardware and software. The python automation routine that is the focus of this project was started by a previous student[6], and the software itself was originally created as an electrospray multipurpose library.

3.1 Hardware model

3.1.1 Instrumentation

The main instruments used for this project are listed below:

a) High Voltage Power Supply (HVPS)

   - brand: FUG

   - model: HCP35-20kV

   The HVPS provides the electrical potential to the liquid, which can be applied by connecting the HVPS directly to the liquid feeding capillary or needle to a grounded electrode (usually a plate or a ring) located downstream.[6] The setup has the USB serial interface for controlling and polling measurements.

   The software has an interface to integrate the HVPS to our routine. This interface can be found in *FUG_function.py* file where is located the functions used to control and collect data from this instrument. In case of change of equipment, a new interface must be created within this file to match another manufacturer specifications.

b) Wireless Oscilloscope

- Brand: *TiePie engineering*

- model: TiePie WifiScope WS6 DIFF

The signal analysis with an oscilloscope using Wi-Fi technology allows an in-depth case study of the electric current signal. The current is measured via a TiePie WifiScope WS6 from TiePie engineering that is a battery powered oscilloscope capable of transmitting data via a Wi-Fi connection allowing it to be placed in the high voltage or ground path.

Wireless communication allows us to make measurements disconnected to an external power supply, which increase safety when using high voltage potential references and also reduce the signal noise collected from external power lines. The current is routed directly via the input, hence the oscilloscope measures the voltage dropped via its input resistance (which can be switched between 1 or 2 Mohms). TiePie WifiScope WS6 has a resolution of up to 16 bit at a minimal input range of 200 mV, sufficient to measure currents down to 1 nA.

The interface with the software was made using the TiePie Library[13] and can be found in *configuration_tiepie.py*. Note that is also important to have the *printinfo.py* file in the project folder in order to work.

c) Humidity and Temperature sensor

The stability of the system is affected by many physical effects. Evidently having the more parameters analyzed favors the system control. The surface tension force is dependent of the liquid-gas interface on the meniscus. Hence, the surrounding gas must be constantly the same and so its humidity. Also, temperature is a variable that interfere in many phenomena in the system. Specially the liquid properties such as viscosity.

For that, a standard microcontroller development board (*Arduino Uno*) with a temperature and humidity sensor (DHT11) was configured to add that data in real time in the routine. The Arduino code can be found in the */peripherals* folder.

d) High Speed Camera

- Brand: *Photron*

- model: Photron fastcam mini

The High Speed Camera (HSC) was just used in this project for validation purposes.

e) Syringe pump

- Brand: *Master dual*

- model: WPI AL-1000

The pump integration in the automation algorithm was done in this work, bringing us a new controllable variable, the flow rate. We can control the spraying mode with the two main variables that affect the system. It brings more complexity for the system since now we are dealing with multivariable control. Controlling also the flow rate gives to this project a new dimension in the system giving us freedom to explore the flow rate properties. With this new input variable our control is a MISO (Multiple Inputs Single Output) system.

About the pump interface, as there was no good ready-to-use library, I developed a simple and intuitive interface to be used in our software routine. The communication protocol used is RS-232 and the pump commands list were found in the user manual.

In figure 3.1 we illustrate a diagram with all the key components of our system. The peripherals are connected to a computer running the software routine via serial communication. This diagram encapsulates our process system and will be used as a sub-system in our control model. The input of our process system are power supply voltage and pump machine flow rate, referred here as the controller values. The output is the oscilloscope current sample.

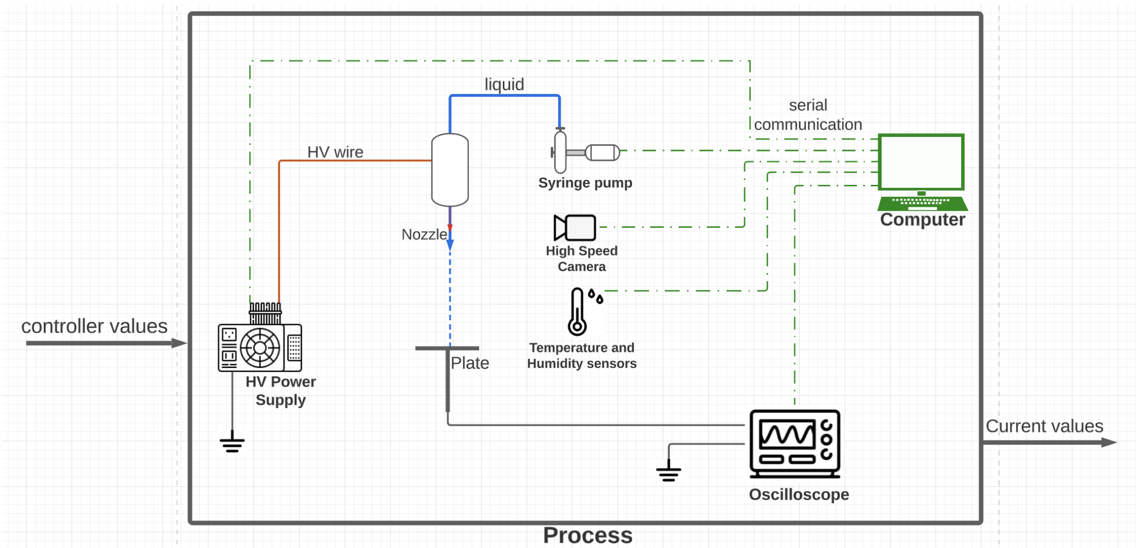

Figure 3.1: EHDA automation system setup.

There are also many minor variables that affects the experiment stability. More details are explained in appendix A.1.

3.2 Software Model

The software was reformulated on top of the control model represented in figure 3.2. The process of the control loop is the same as represented in figure 3.1. Each other subsystem in the control loop is a separate thread that will be explained bellow.

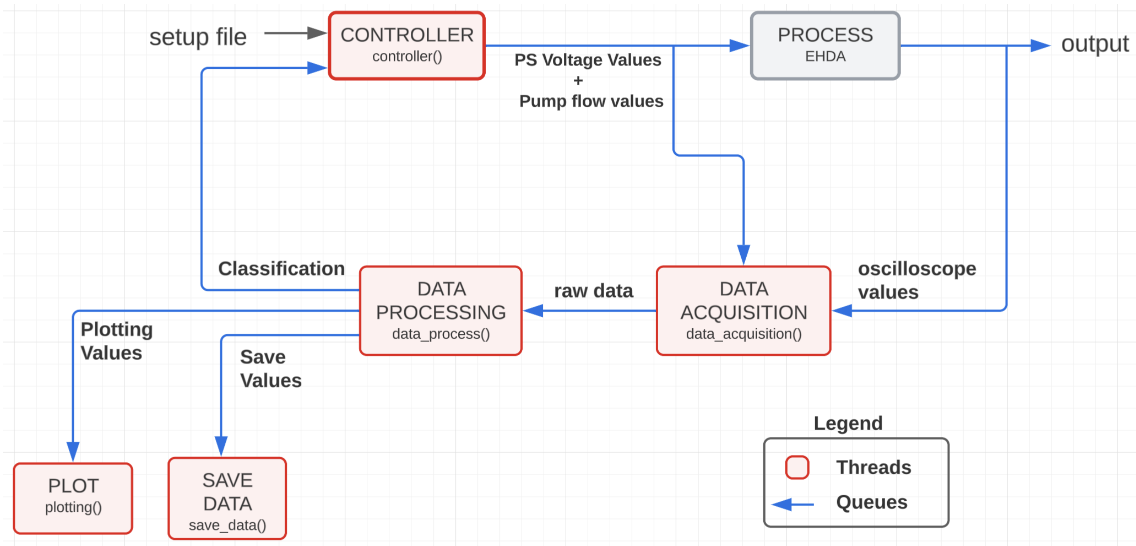

Figure 3.2: EHDA automation closed loop control system model implemented.

3.2.1 Threading and Queues

In order to implement this closed loop control model to the software and explore parallel processing, each sub-system was implemented as a separate Thread. For concurrency on flux of data between threads was used queues structures. A queue is an abstract data type that holds an ordered, linear sequence of items. You can describe it as a first in, first out (FIFO) structure.

3.2.2 Controller Thread

It is responsible for sending the power supply set voltage values and the syringe pump the flow rate set values according to the sequence selected. Also, responsible for sending the finish event command that end the routine and trigger the threads to close their routines. As input, we have the setup config file and the *feedback_queue*. As output, we have the values in the *controller_output_queue()*.

3.2.3 Data Acquisition Thread

It is responsible for reading the current data from the oscilloscope, humidity and temperature data from the DHT11 sensor, voltage from the power supply, flow rate from the pump and concatenate into one sample data. As output, we have the values in the *data_queue()*.

3.2.4 Data Processing Thread

It is responsible for calculating the statistical values from the raw data and classify it in the respective spray mode for that sample. As output, we have the values in the *save_data_queue()*, *plotting_queue()* and *feedback_queue()*.

3.2.5 Save Data Thread

After processing, the data is saved in real time in a json file using *jsonstreams* library to save one sample structure at a time. With the new streaming model of saving a new structure of the collected data were created. Instead of having all data measurements values and after all data processing values we now are saving for each sample the measurements and processing values. The data acquired in each sample of 0.5s is shown in figure 3.3.


```
"sample 0": {
    "name": "setup/liquid/ethanol",
    "current": [...
    ],
    "flow_rate": "0.97",
    "voltage": "4001.62",
    "current_PS": "-2.79252e-09",
    "temperature": "0",
    "humidity": "16.0",
    "date_and_time": "2023-03-16 14:26:53.012518",
    "target_voltage": 4000,
    "mean": 18.687360763549805,
    "variance": 506.2657470703125,
    "deviation": 22.500349044799805,
    "median": 9.559748649597168,
    "rms": 29.248645782470703,
    "spray_mode": "Intermittent"
},
```


Figure 3.3: Output data json structure.

This new way of saving the measurement and processing data altogether separate by enumerated samples prevents loss of data in case of error during experiments and, specially improve in the usability of the data for further deep analysis. One experiment of around 1 hour collects around 6 GB of data. Organizing that amount of data to make it fast and easy to use was an important improvement in the software. To work and analyze the data we can use pandas Data frame. A python widely used framework for data analysis. With the command:

pandas.read_json('PATH', orient='index').

It will construct a Data frame with all data organized by samples.

To conclude, json files are good as a system output because of its human readability. But as the database gets bigger json becomes to be a slow to read and heavy to store data. For that, saving the data frame in a compressed type of file called feather is much faster to work with it.

3.2.6 Plotting

The only running function that should run in the main thread because the plotting library *matplotlib* incompatibilities of running outside the main function. It is responsible for plotting in real time the current sample acquired, and its respective fast Fourier transform to evaluate the sample frequency spectrum. It takes as input the *plot_data_queue* from the *processing_thread()* and displays three graphs updated in real time on the screen during the experiment.

3.3 Chapter conclusion

In this chapter we described the system description detailing the hardware instruments, how it was modelled and its implementation in the software.

Chapter 4

Methodology

This chapter is about describing the methodology and developments to achieve the project goals. Firstly, by elaborating on the workings of classification process in the processing thread. Moving on, delving into the operational sequences of the controller thread. Lastly, providing an explanation on the implementation of the controller.

4.1 Classification

The classification is a key step in our routine and for being able to be used in multi-purpose applications our classification must be able to run in real-time, which means it must be fast and automatic. The goal is to improve and apply approaches of non-visual spraying classification using the current data collected from the system.

4.1.1 Statistical Analysis

In Sjaaks[5] work, the author exposed signal characteristics that can be used to classify the actual spraying mode with a sample of measured current using both time and frequency domain analysis. We acquire the current data frame of 0.5s with 100 kHz as sampling frequency, with each current sample as 50 thousand current values. Through statistical analysis in these values such as mean and standard deviation we apply in our automatic classification the relative standard deviation. Which is referred as the sample standard deviation divided by the sample mean values. Figure 4.1 illustrate data samples being classified using Sjaaks method.

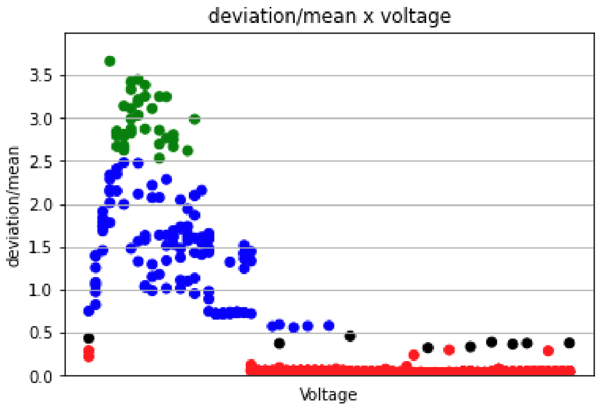

Figure 4.1: Sample classification using statistical values. Colors: Green = Dripping; Blue = Intermittent; Red = Cone Jet.

This classification by statistical analysis was already implemented in the software library made by the previous student [6]. This method was capable of only classifying dripping, intermittent and cone jet modes. Also in her work, Monica extended the classification to corona or discharge detection. My contribution was to integrate her work on my control loop model and extend the classification for the multi jet.

To classify the multi jet, after running many steps experiments I noticed that the multi jet signal has a similar shape as the cone jet but with a step in its current mean, also the cone jet current was almost fixed in all its voltage range. These effects were exposed by Ryan[12]. In his work he defines the relation between current per jet in both cone jet and multi jet modes.

With that, I implemented the classification of the multi jet when the current mean is 1.14 above the expected cone jet current mean. The cone jet current can be calculated by the scaling laws 2.3 formula. The factor value of 1.14 was found by repeating a lot of experiments with the same liquid (pure ethanol), but varying the flow rate and the voltage. This factor value is just applied for pure ethanol, the liquid used in all experiments in this work. The classification in the algorithm was divided in three steps as shown below.

- Sjaak Classification -> Classifies Dripping, Intermittent and Cone Jet

- Monica Classification -> Classifies Corona Sparks

- João Classification -> Classifies Multi Jet

The algorithm 1 shows the implemented classification and works in the following way:

Algorithm 1 Statistical Classification

function STATISTICAL_CLASSIFICATION($sample$)
  $spray\_mode \leftarrow "Undefined"$;
  $mean \leftarrow sample.mean$;
  $std\_deviation \leftarrow sample.std\_deviation$;
  $median \leftarrow sample.median$;
  if $mean/std\_deviation > 2.5$ **then** ▷ Sjaak classification [5]
    $spray\_mode \leftarrow "Dripping"$;
  else if $2.5 < mean/std\_deviation < 2.5$ & $mean/std\_deviation > 0.3$ **then**
    $spray\_mode \leftarrow "Intermittent"$;
  else if $mean/std\_deviation < 0.3$ **then**
    $spray\_mode \leftarrow "ConeJet"$;
    $cone\_jet\_mean \leftarrow mean$;
  end if
  if $mean/std\_deviation > 2.5$ **then** ▷ Monica classification [6]
  end if
  if $spray\_mode == "ConeJet"$ **then** ▷ João classification
    if $cone\_jet\_mean > 1.14 \times mean$ **then**
      $spray\_mode \leftarrow "MultiJet"$;
    end if
  end if
  return $spray\_mode$;
end function

4.2 Routine Sequences

The program developed is capable of running different types of routines and with an easy way to implement new strategies. Continuing the methodology, in the setup json file there is a "sequence" attribute which can be chosen between "ramp", "step", "map" or "control". The controller thread will manage what the algorithm must do for each sequence. Following our control model3.2, the controller outputs (voltage and flow rate) are the actuators signal. The sampling rate is fixed to 0.5 seconds for all the experiments, and it's managed by the data_acquisiton_thread3.2.3.

4.2.1 Ramp

The ramp sequence is simply done by sending one command to the power supply to perform a ramp in the voltage with initial, final and slope values. The flow rate for this sequence is constant, and the experiment consists in a voltage range scan.

4.2.2 Step

The step sequence is done by the same command send to the power supply but with the maximum slope value in order to proximate of a step signal. By waiting a certain defined *step_time* and repeating the command in a loop until it gets into the final voltage, as represented in algorithm 2.

Algorithm 2 STEP sequence in controller thread

```
procedure STEP(voltage_start, voltage_stop)
    voltage ← voltage_start
    while voltage ≤ voltage_stop do                ▷ scanning voltage range
        SEND_VOLTAGE_COMMAND(voltage)
        SLEEP(step _time)
        voltage ← voltage + step_size
    end while
end procedure
```

4.2.3 Map

The map that will be explained below is the most relevant sequence in this work. This type of experiment saves human work and time, create a precise analysis and can be compared with previous works for validation of methodology. The purpose is to map the operational window, seen in Figure 4.4, that can be defined where the cone jet spraying mode can be stabilized based on the flow rate, voltage and the setup configuration.

The map was inspired in Gañan-Calvo[4] work where he points how liquid conductivity influences the cone jet stability island.

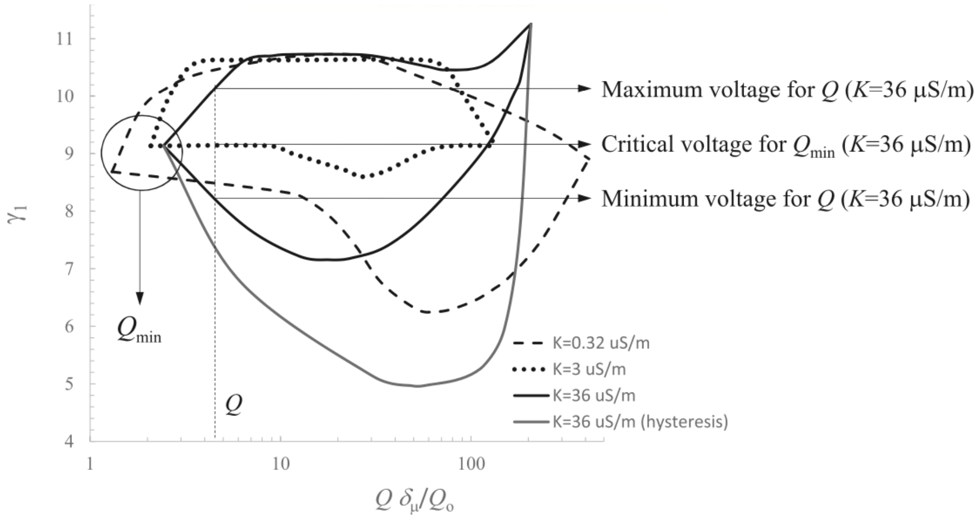

Figure 4.2: Domains of existence (stability) of Taylor Cone Jets. [4] . The window formed by these points is where the system operated in the stable cone-jet. Operational windows depend on the liquid and configuration setup. Different windows are represented for different liquid conductivity. The X and Y axis are non-dimensional representation of electric potential and liquid flow rate, respectively.

To reach certain map it is necessary to traverse flow rate and voltage values acquiring samples. The flow rate (X axis) and voltage (Y axis) values for each experiment can be configured in the setup.json file. The algorithm for this sequence is a loop of the step sequence4.2.2 for each flow rate chosen, and it is represented in the algorithm 3.

Algorithm 3 MAP sequence in controller thread

procedure MAP(*flowrate_values*)
  for all flowrate_values **do** ▷ scanning in the flowrate range
    SEND_FLOWRATE_COMMAND(flowrate)
    $voltage \leftarrow voltage\_start$
    while $voltage \leq voltage\_stop$ **do** ▷ scanning in the voltage range
      SEND_VOLTAGE_COMMAND(voltage)
      SLEEP(step _time)
      $voltage \leftarrow voltage + step\_size$
    end while
  end for
end procedure

In Figure 4.3 we can see the data acquired in this mapping experiments. The liquid used is pure ethanol. Note that the experiment is composed of loops that increase voltage, change flow rate and repeat.

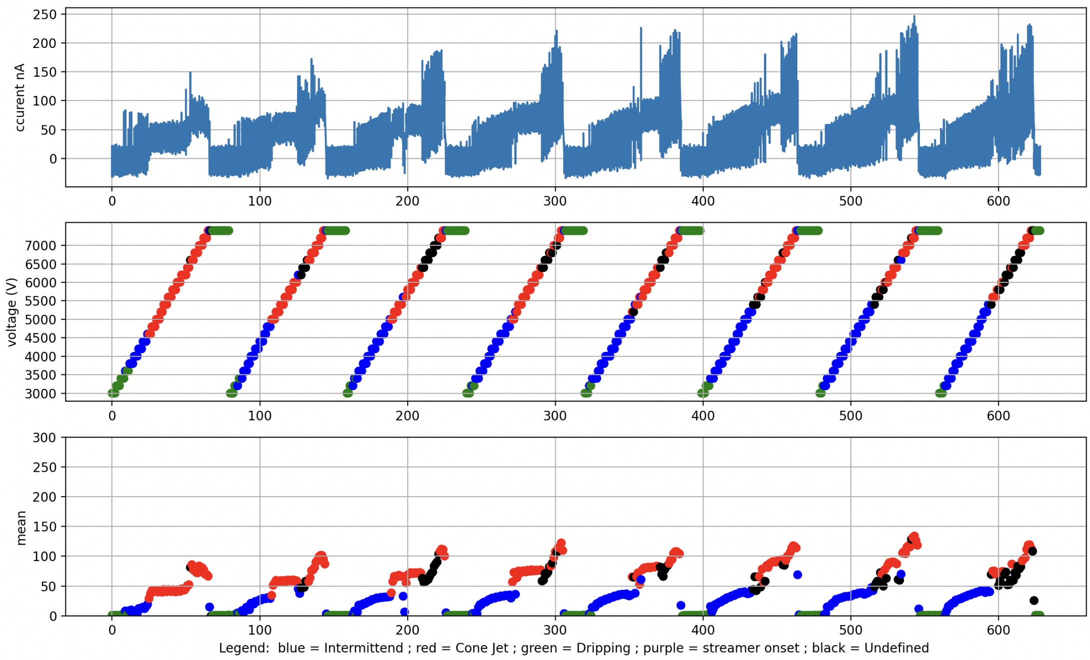

Figure 4.3: Mapping Experiment data collected. The figure has 3 graphs with shared x-axis representing the samples collected. The first is the current values collected through all the experiment. The second is the voltage values applied in each window of data collected. The colors represent the spraying classification defined by our routine. The third graph shows the current mean value of each data sample.

With all the data collected, classified and saved in real time, we can do further analysis and studies. For example, Figure 4.4 illustrate the data classified by our algorithm and displayed in a Voltage X Flow rate range of spraying modes with a specific liquid setup so that we can compare the automatic results with previous researches, such as showed in figure 4.2 and validate the algorithm.

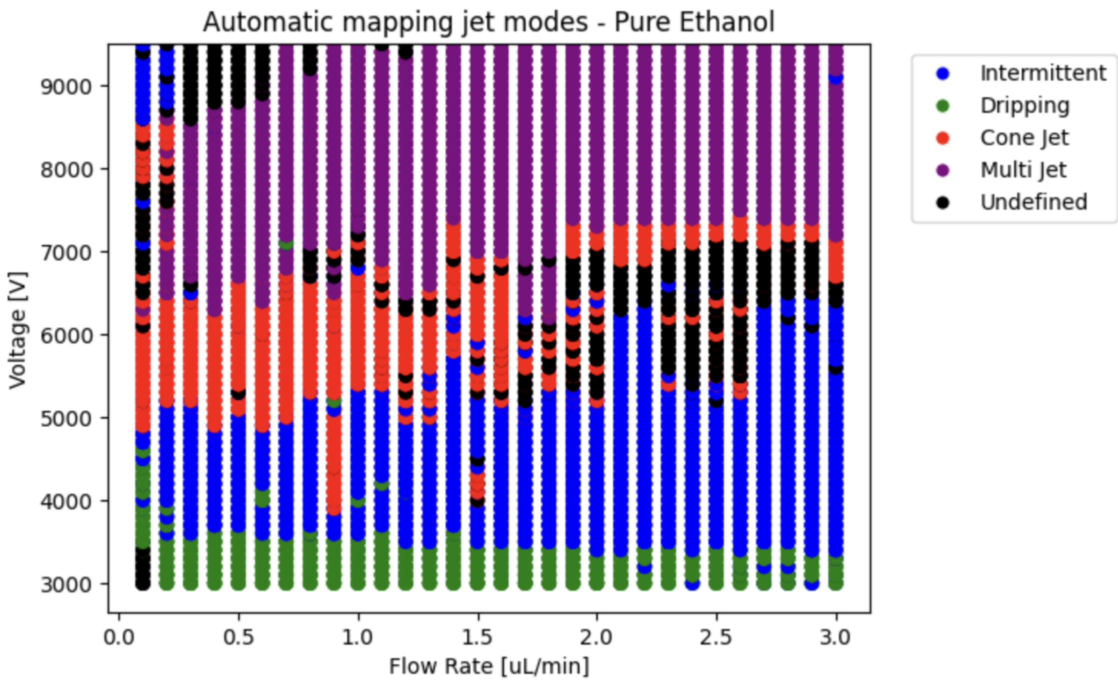

Figure 4.4: Mapping Experiment for pure ethanol in ambient conditions with our capillary setup. The map shows the stability region of each electrospraying mode in the voltage and flow rate range.

4.2.4 Control

The control sequence is the only from our list of sequences that actually uses the feedback classification value. As it is a closed loop control, the controller must be able to stabilize the system in the desired conditions. A simple controller algorithm is represented in algorithm 4, and it was used as a proof of concept. Its results can be seen in results section 5.3.

Algorithm 4 simple controller

```
function CONTROLLER(spray_mode)
    if spray_mode = 'Intermittent' or spray_mode = 'Dripping' then
        SEND_VOLTAGE_COMMAND(voltage + 100)
    else if spray_mode = 'MultiJet' or spray_mode = 'Corona' then
        SEND_VOLTAGE_COMMAND(voltage - 100)
    else if  spray_mode = "ConeJet" then                    ▷ Keep Stable
    end if
end function
```

4.3 Chapter conclusion

This chapter 4 exhibits how the knowledge from chapter 2 was implemented with a control and automation engineering approach in order to achieve our goals. Next chapter 5, we will discuss the results reached.

Chapter 5

Results

In this chapter, we will present the results obtained from our project. Firstly, we will demonstrate how the automation routine enhanced the research experiments. Next, we will discuss the validation of the classification results. Finally, we will showcase the performance of the implemented controller algorithms.

5.1 Automation routine

The automated experiment routine is capable of acquiring a significantly more precise and extensive amount of data than what can be achieved by a human. With the save data by streaming in saving thread 3.2.5, we achieved less program memory during the experiment and, specially, safety for not losing the data in case the program fail during an experiment.

Also, most power supplies rely on manual potentiometer adjustment to select the set point, which results in imprecise potential selection by a person and takes some time to achieve the desired voltage. Moreover, the electrospray phenomena has a known hysteresis, that can be seen in figure 4.2, and can perform different results depending on the previous electric potential. By running a computer routine that automatically sends commands to the power supply and pump machine we can achieve faster and more reliable experiment data points.

The visual interface for the user, seen in figure 5.1, shows all the important sensor data and signal analysis that can be interesting to the operator in real time, serving as a supervisory system.

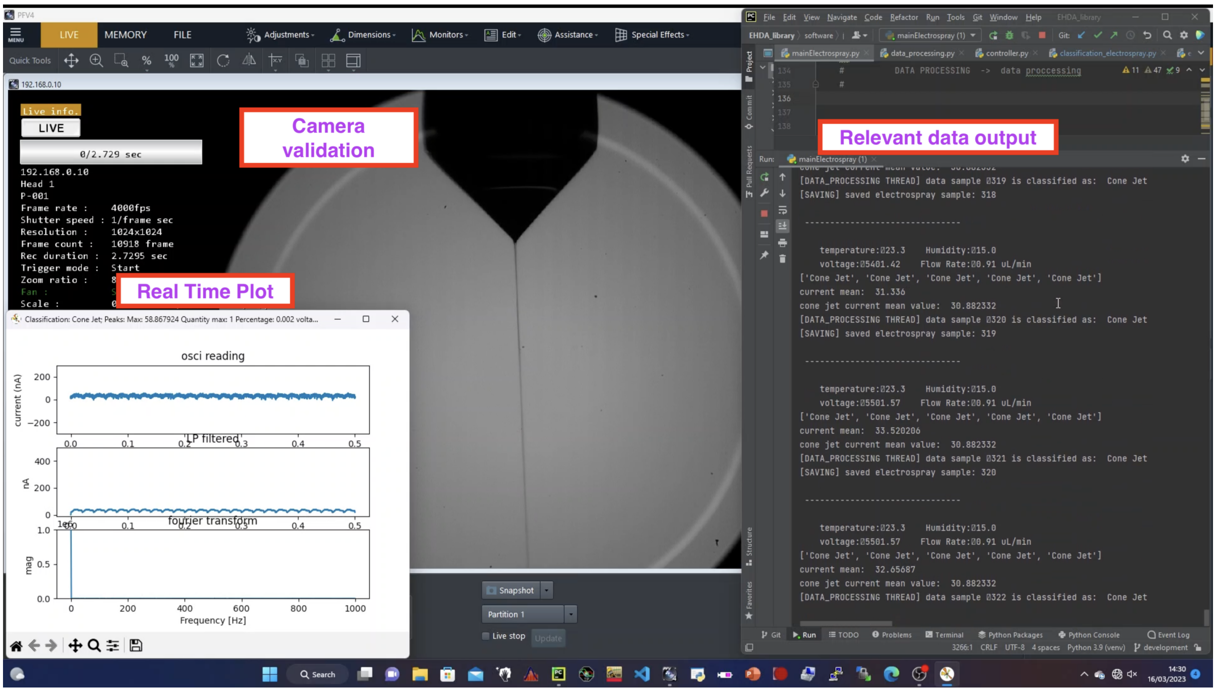

Figure 5.1: Print screen of the window shows user interface during the experiment. We can see the image generated by the camera in the background. The routine code running in pycharm software on the right side. And also real time signal plottings of the current data on the left side.

5.2 Classification

The classification results in our algorithm implemented as described in section 4.1 showed good accuracy for all the classifications described in section 2.4. Nevertheless, the multi jet classification made by a current value factor of 1.14 above the cone jet is just effective for pure ethanol, liquid used in all this project.

Even if the multi jet classification just works for pure ethanol, the data saved with correct classification in ethanol can be used to extract any other signal information about the multi jet or to train a black box classification algorithm.

We will categorize our classification results into two main groups - the step routine and the map routine. These two routines have showed valuable insights and comprehensible results of the electrospraying process.

5.2.1 Step Sequence

Our first classification results were made exploring the voltage ranges. For that, we fixed a flow rate to 0.7 uL/min. and ran a step routine as defined in 4.2.2. The figure 5.2 shows three graphs.

The first shows the controller output signal as an input voltage of the process. As it is a step routine we implemented a increasing voltage with steps of sizes 50V and time between each step of 5 seconds. The voltage range is between 3k-10k Volts.

The second is the raw output data collected by the oscilloscope in *data_acquisition_thread()*. The sampling rate is 100kHz. Therefore, this experiment of 700s has 70 Million data points just of current data. This is an example of how scalable the data collected can be depending on the experiment time. This will be even more noticeable in mapping experiments.

The Third graph is the same data as the second after the classification procedure done by *data_processing_thread()*.

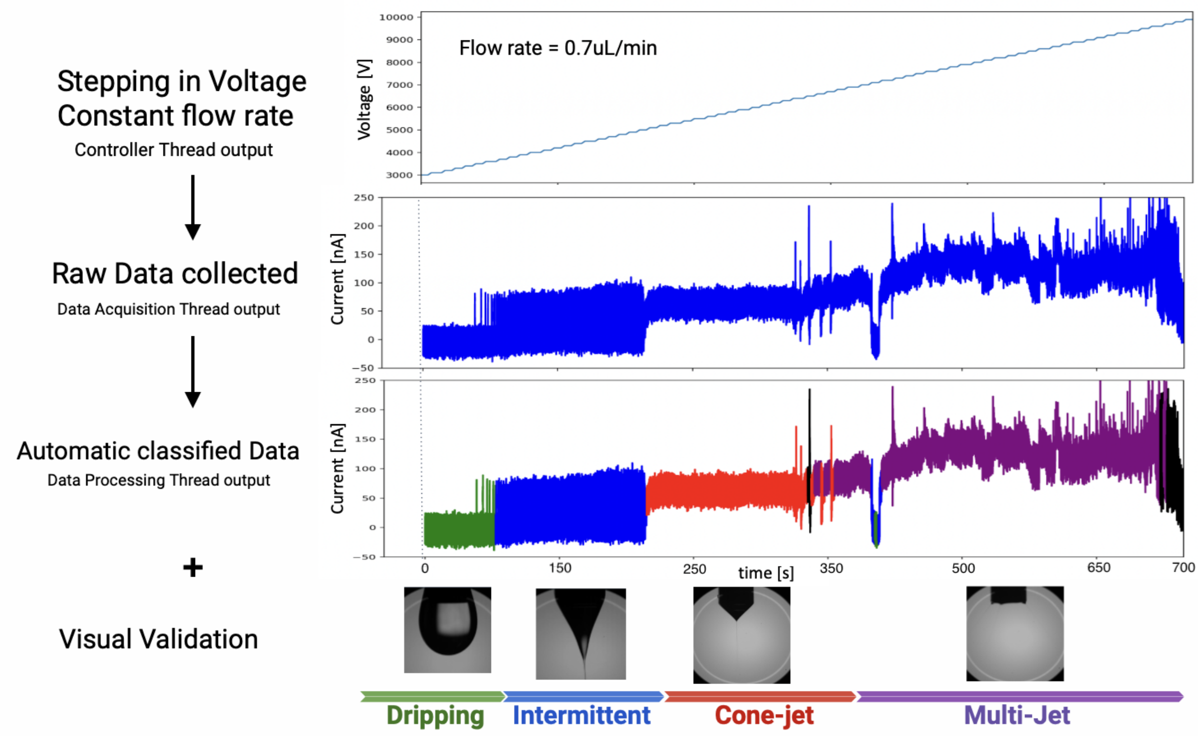

Figure 5.2: Automatic electrospray classification through the step routine.

The graph of voltage scan has a common shape for different liquids and parameters. After having familiarity with it, is even possible to classify the spraying modes by visual analysis.

For example, dripping mode has a current mean of 0V. The Intermittent state has a high variation of values that can be seen by the increased thickness on the graph. The Cone jet is a thinner graph because of its constant signal. Multi Jet has the same shape as Cone jet but with a higher mean value. Corona sparks are not showed in the graph because its discharges has a high current value above the axis limits.

5.2.2 Map Sequence

For validation with literature and also to expose the benefits of the automated routine and classification, the map sequence proof itself the best result of this work.

Initially, for better understand pure ethanol classification regions through voltage and flow rate ranges, I made a manual map seen in figure 5.2.2.

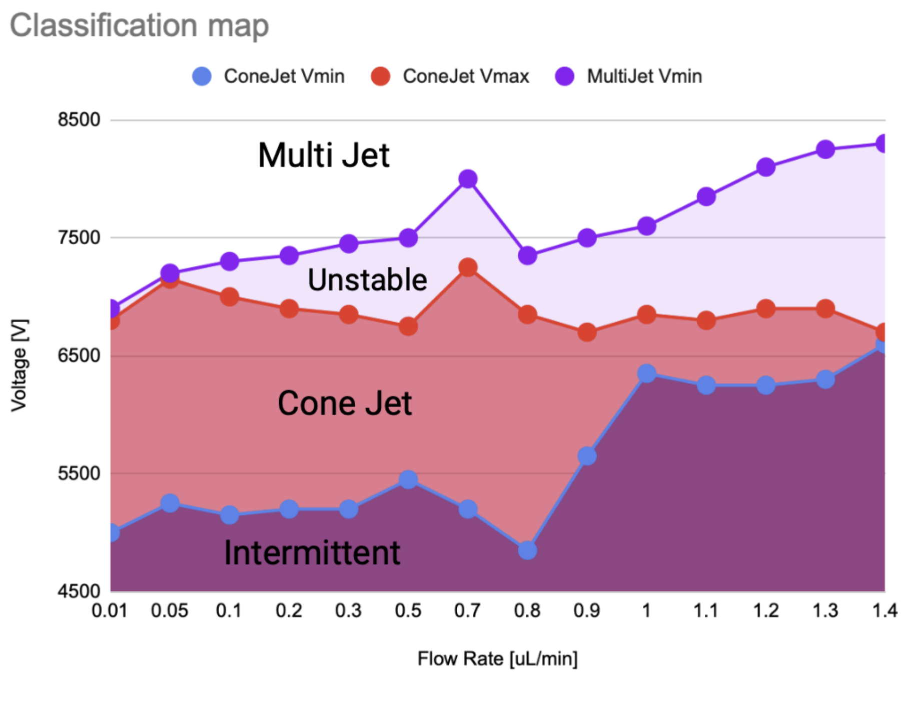

Figure 5.3: Experimental spraying modes regions of pure ethanol.

In order to validate our automatic classification, experiments were made comparing both visual and automatic stability island on the same experiment. Figure 5.4 shows that automatic stable cone jet region could be identified in the same region as visually seen by the high speed camera (data acquired manually).

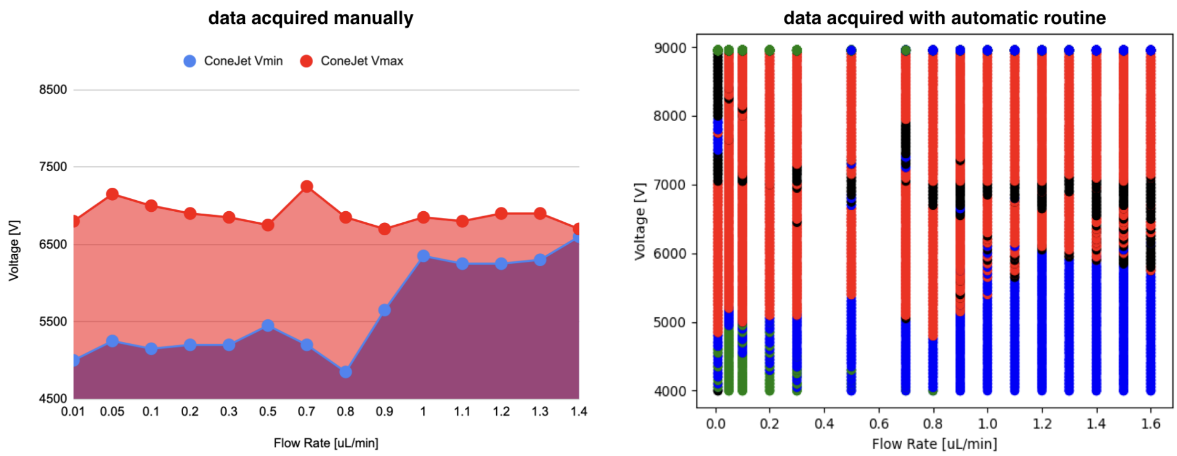

Figure 5.4: Cone jet stability region for pure ethanol experiment 1.

With the development of a Multi Jet classification using the logic explained in section 4.1, I repeated the same experiment as shown in Figure 5.5.

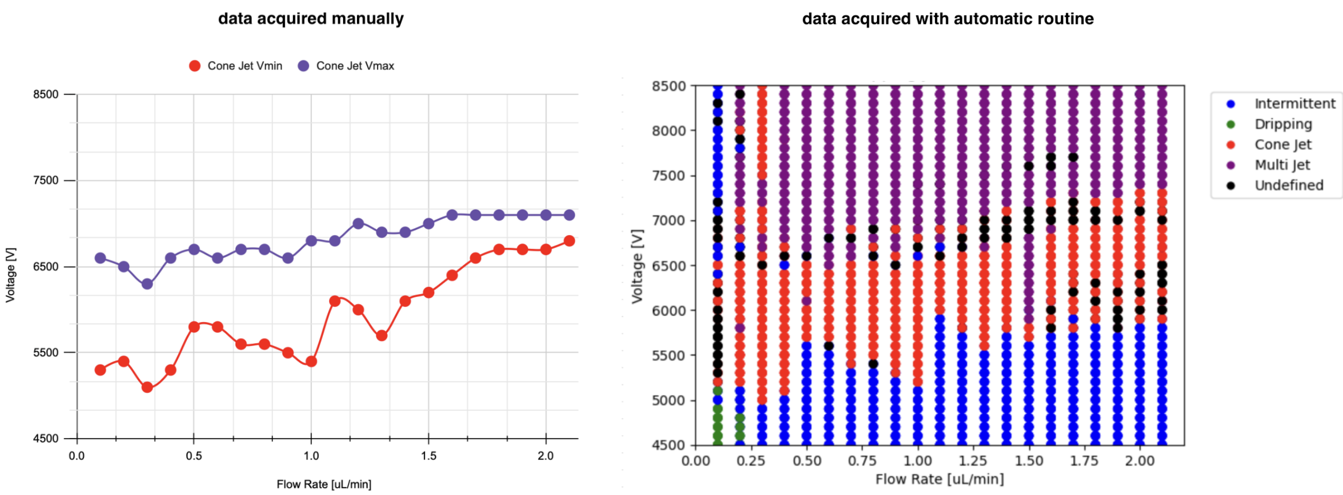

Figure 5.5: Cone jet stability region for pure ethanol experiment 2.

Non-dimensional axis

To have a better comparison with literature, specifically the Gañán-Calvo[4] stability islands showed in figure 4.2, with a visual juxtaposition of the shapes, we displayed in figure 5.6 the data using the non-dimensional numbers used in his work[4].

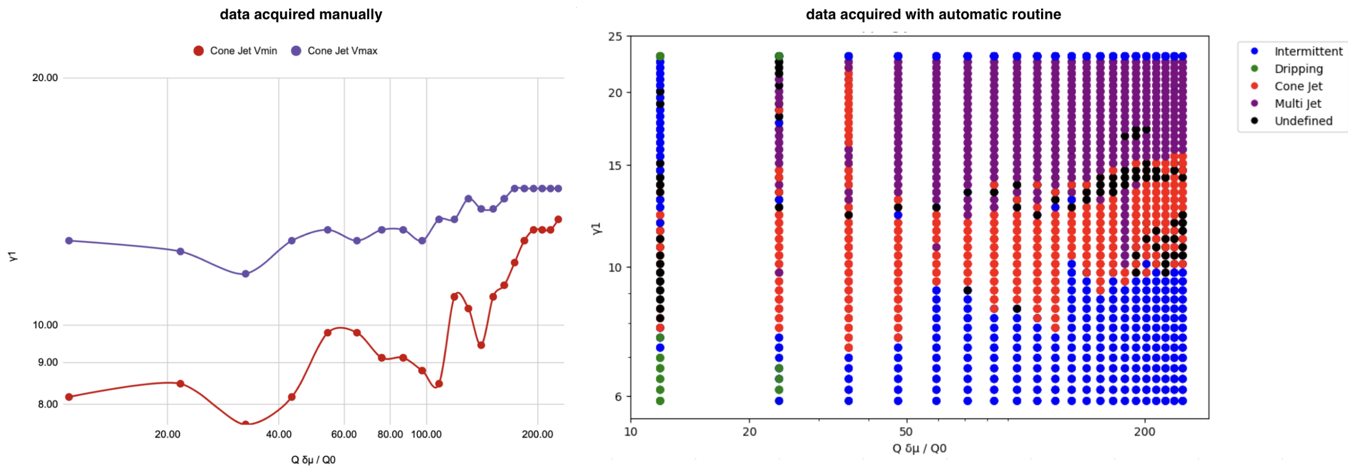

Figure 5.6: Cone jet island manual experiment 4.

The results of mapping experiment corresponded visual, literature and automatic regions of classification, giving authentication to our method.

5.3 Controller

The results of the classification process were favorable for experiment automation, however, the inaccuracy of the classification by statistical methods, specially the Multi Jet2.4.4, limited the development of a complete control project. Together with the amount of variables that need to be syntonized makes it hard to stabilize in a desired mode.

Even with all those problems we could implement a simple controller project, described in 3.2, that validated the efforts of remodeling all software into a closed loop control model.

The controller not only stabilized in Cone Jet mode, but also could reject perturbation, as seen in Figure 5.7, where I manually changed the flow rate. First graph shows current acquired. It starts as intermittent state, with an oscillating signal. We can validate this with the blue colored sample on the third graph representing its automatic classification. The second graph shows the controller actuating increasing the voltage to achieve a stable cone jet mode. After a period of time in cone jet, I manually increased the flow rate, serving as a perturbation of the system, and we can see that the effect of that was to return to intermittent state with the oscillating signal. The controller again, actuate increasing the voltage to reach the cone jet stability island.

This procedure was repeated after some seconds of stabilization in cone jet, increasing again the flow rate manually, and we can see the system automatically adjusting the voltage.

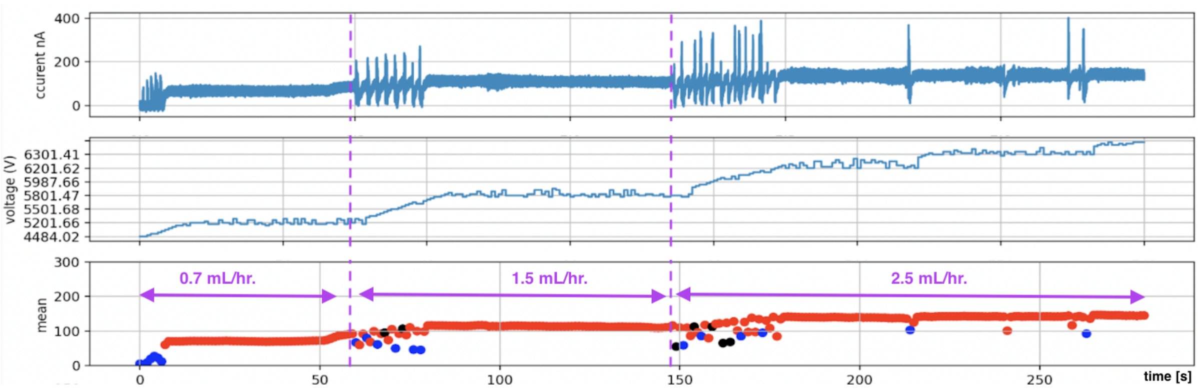

Figure 5.7: Simple controller validation experiment. For this experiment we waited the system to stabilize in the desired spraying mode. After that we made a perturbation test changing the flow rate. The classification legend follows the same color reference in all project.

After 200s of experiment we had some breaks up of the cone jet into a droplet and stabilized again. This is represented by the peak values in current signal. The classification correctly identified as intermittent, seen as blue dots in the third graph, and the controller reacted increasing the voltage, in a situation that can be considered as noise. A filter can be applied in a more complex controller project to avoid this.

5.4 Chapter conclusion

In this chapter we exposed the results of this project. The automatic routine, real time classification and control were all achieved and implemented. However, they can all be improved. In the next Chapter I will conclude this document with discussions about the results and proposal of continuation.

Chapter 6

Conclusion

In conclusion, the implementation of a Python routine for complete automation and integration of the system has improved the precision of experiments and enabled real-time collection of extensive data without requiring the expertise and time of a specialized scientist. This project underscores the potential of automation in enhancing scientific research and accelerating progress towards achieving research goals. In addition to its potential in scientific research, the software developed in this project can also be leveraged by industrial approaches. Furthermore, the classification methods and system control implemented in this project have yielded promising results and can be further optimized in the future. Overall, this project serves as a contribution towards the advancement of automated systems and the optimization of experimental processes.

6.1 Proposal for continuation

To continue, my proposal would be to prioritize the improvement of the classification step. Ideally, we could use machine learning or another algorithm to ensure accurate classification with a broad range of liquids. By achieving a reliable classification, we can then implement more effective controller logic or approaches. In this regard, I would like to present the concept of a fuzzy controller as a potential solution.

6.1.1 Machine Learning

When it comes to classifying spraying modes, machine learning represents a sophisticated and trendy approach. This project was designed in order to substitute our statistical classifier for a more general and accurate algorithm.

The data collected in this project was saved in 0.5s samples together with its classification allowing it to be used for supervised learning. Experimental trials were conducted to explore machine learning algorithms to classify the data. Unfortunately, despite efforts, an accurate method of distinguishing between classifications could not be identified.

6.1.2 Fuzzy Controller

One challenge in implementing the controller in this project is that we don't have a real value as feedback, instead, the feedback from the controller loop is a classification, which makes it difficult to apply many of the principles of control theory that are designed for continuous control. As this project involves logical control, it requires a different approach.

The controller project that will be presented is an attempt to quantify the classification and fit it in a fuzzy control model. That approach is for an open loop control system and the input and outputs of the controller must be fuzzyfied.

Firstly, the fuzzyfication and defuzzyfication machines were implemented using the data acquired in the experiment of step routine4.2.2, mapping the area of each spraying mode according to its potential. Figure 6.1 shows the steps of fuzzyfication and defuzzyfication within the input and output of the controller.

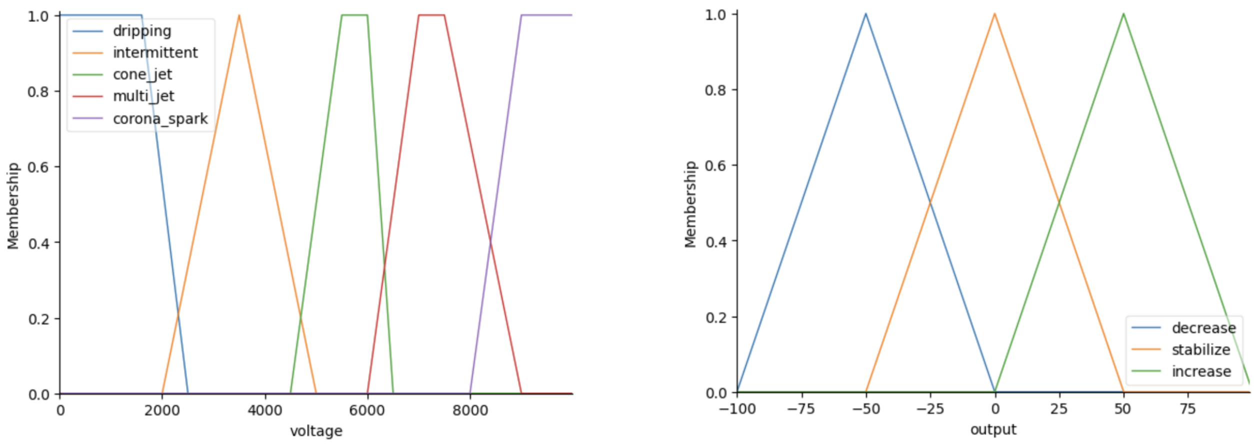

Figure 6.1: fuzzyfication of input and defuzzyfication of output.

Our controller is an algorithm that calculate the output with a geometrical calculus on the membership functions of the input and output according to our rules listed below.

Fuzzy Rules:

-> IF dripping THEN increase

-> IF intermittent THEN increase

-> IF cone jet THEN stabilize

-> IF multi jet THEN decrease

-> IF corona THEN decrease

In figure 6.2, two tests were made. Test 1 with a higher voltage of 7000V accusing it to be 100% in multi jet and the output of decrease voltage. Test 2 shows the opposite case when the voltage is lower than expected.

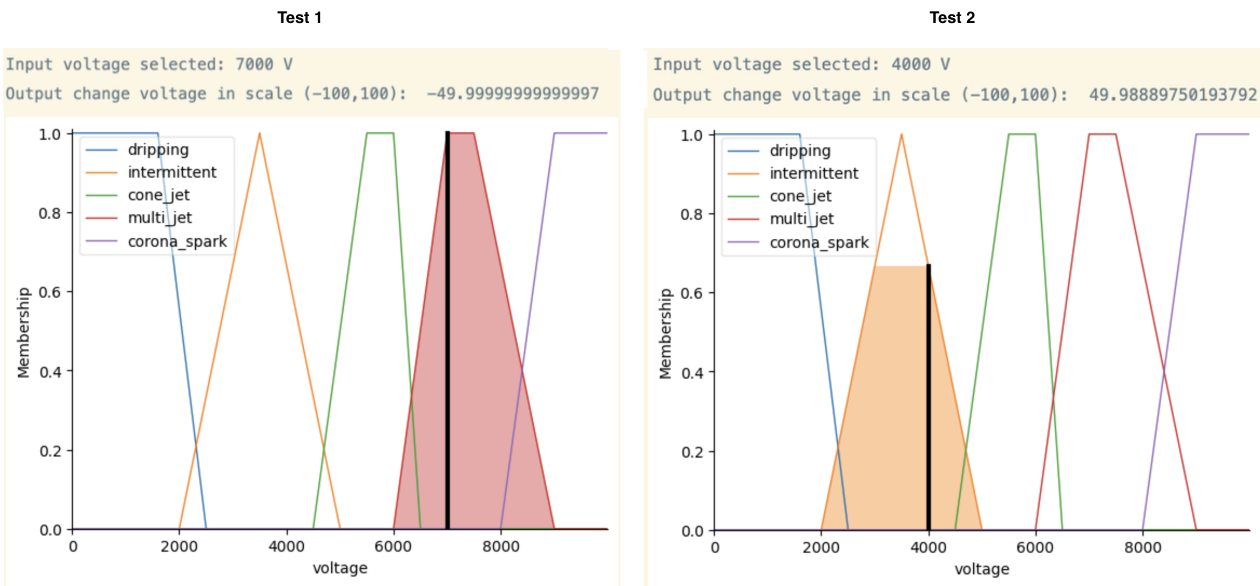

Figure 6.2: Test implemented to validate the concept of fuzzy controller in this project.

6.2 Final Discussions

The works presented in this report represent a continuation and refinement of previous efforts to develop a more precise and versatile automation routine that can be effectively applied in both industrial and research applications.

The previous student work focused on predicting corona streamers or spark discharges[6] and her automation routine provided a valuable proof-of-concept.

The current project builds on this foundation and highlights several key results, including the integration of a liquid pump into the software and the development of a routine that can be easily modeled to fit a control model, making it straightforward to implement new control algorithms or experiment routines. The software was also remodeled to support threads, allowing for the separation of each subsystem and the exchange of data between them using queue data structures. A classification for multi-jet spraying mode was developed, along with a simple controller to demonstrate proof of concept. Additionally, the saving of data was optimized to a real-time streaming format and expanded to include more sensor data. Finally, the algorithm usability was restructured to make it more intuitive for users through the use of a setup file.

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Appendix A

System Details

The electroplating system is an unstable system that relies on multiple parameters being within their respective optimal ranges to achieve a desired mode. Several tips can help reduce noise and stabilize the system, such as avoiding the use of an oscilloscope with the charger to avoid power line noise. Mechanical noise in the pumping machine can be minimized by using a syringe pump tilted with a bubble inside. External electrical noise can be picked up by antennas or connections in the circuit. To stabilize internal humidity, turn on the gas pipeline with air into the chamber.

The picture in figure A.1 shows the setup used in all this project. Figure A shows the nozzle and plate with the charged liquid being electrosprayed in cone jet mode.

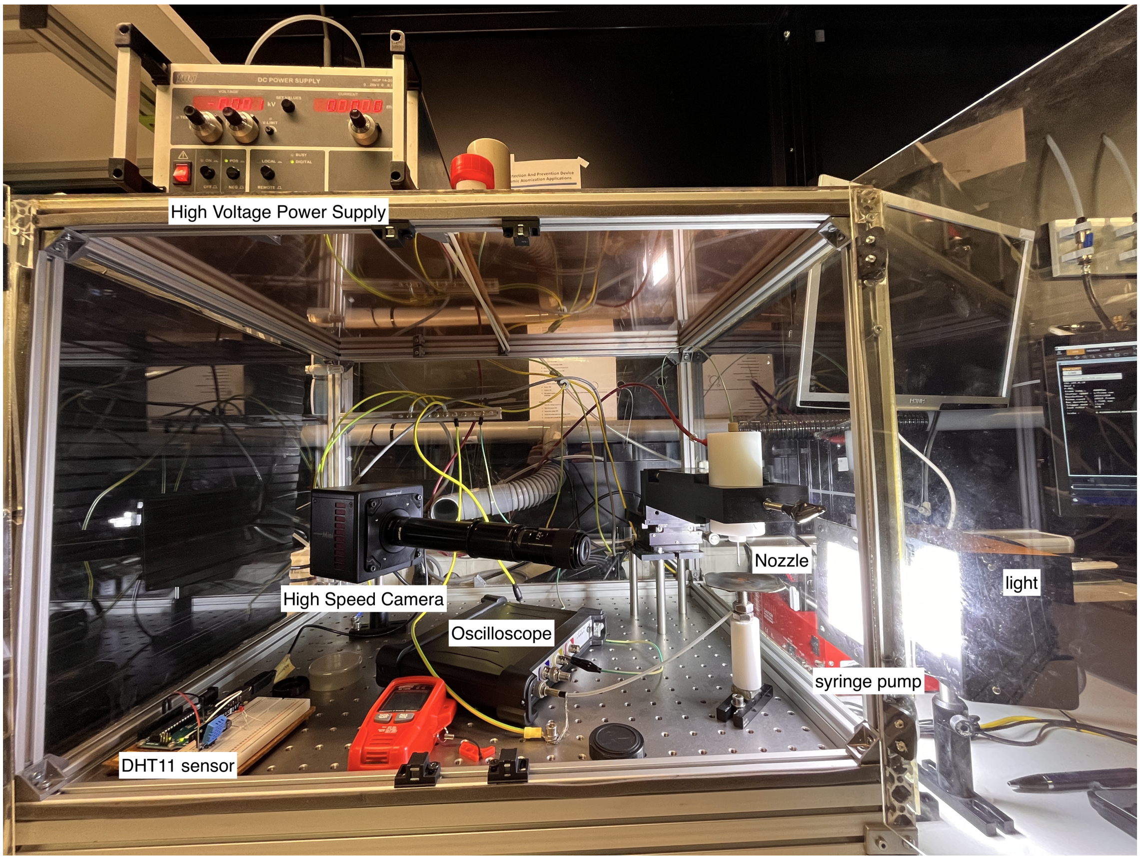

Figure A.1: EHDA automation system setup used for experiments.

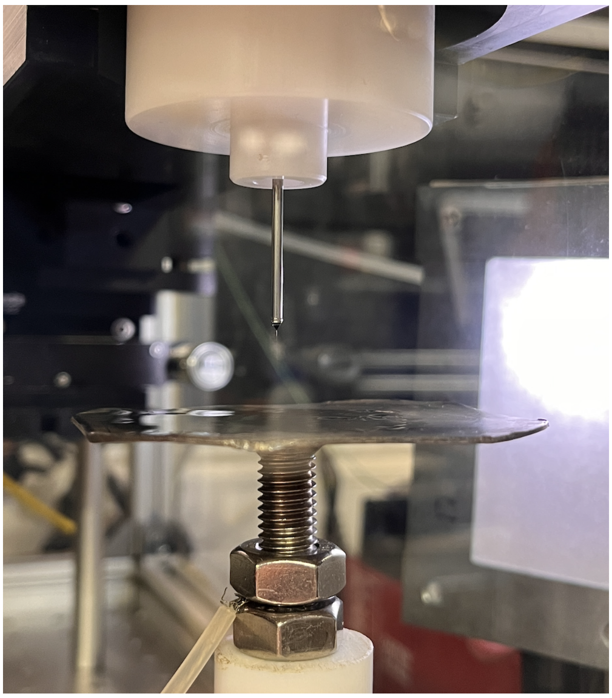

Figure A.2: EHDA picture of the electrified syringe.

A.1 Setup Validation

It is recommended to do initial tests in order to verify the setup assembly and the automation routine integration. This is an important step to understand in practice how electrospray works. Factors like geometry, polarity, material properties and occurring discharges are reflected in the system current. Also, liquid properties such as surface tension, dielectric constant, viscosity, density, electrical conductivity and vacuum permittivity. And also physical variables such as flow rate, system impedance, system temperature, system humidity, nozzle to plate distance, nozzle dimensions and applied voltage.

A.2 Oscilloscope Impedance

The current being measured by the Oscilloscope use its internal impedance. The *TiePie* oscilloscope model has two impedance options, 1Mohm or 2Mohms. Selecting one or other will multiply or divide your current measurement by 2. By default, we are using the 2Mohm differential input, however it was noticed that using the 1Mohm resistance might reduce noise. A proposal is to configure the 1Mohm resistance in the *configuration_ tiepie.py* file and evaluate its performance.