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 helpful 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:

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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 UFMG, 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.

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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 which can be achieved by a closed-loop control system. This project targets the development of a system that 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

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 many 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], who focused on predicting undesired discharges (sparks) in the system occurred when the electric potential is too high. For that, a python software routine was developed to connect most of the peripherals and analyze the current data. The success of her work in applying statistical classifications corroborates the validation of this project motivating its continuation on development and optimization.

The main goals are shown:

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 to 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 the methodology.

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

In Chapter 5, presents results and engages in discussions comparing the knowledge acquired through literature review.

Finally, in Chapter 6, this document is concluded 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 phenomenon.[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 suggests 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 a certain stable spraying mode can be reached, as seen in the Figure 2.1.

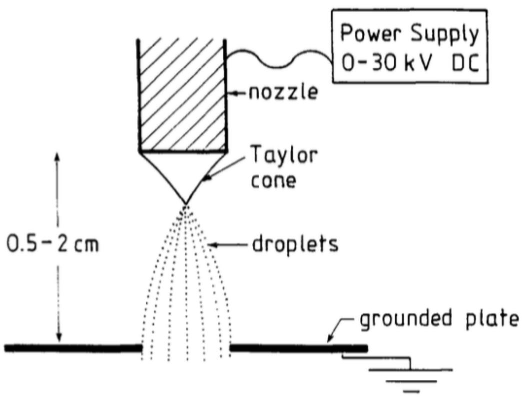

Figure 2.1: EHDA physical concept[2].

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.

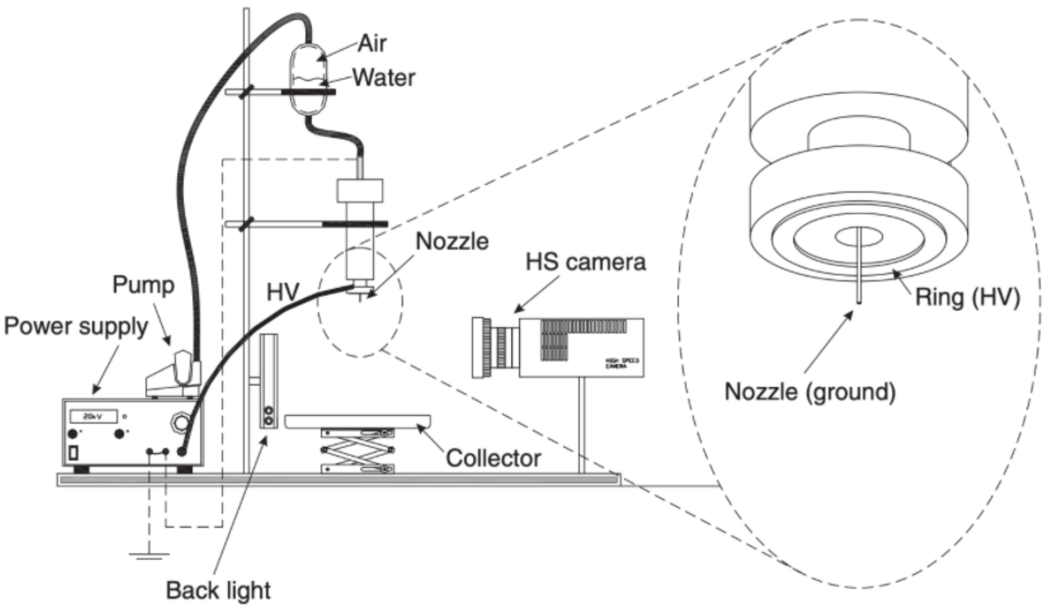

Figure 2.2: EHDA experiment setup[3].

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 gives valuable information about the spraying behavior. Figure 2.3 illustrate a sample of current measured in the system during a dripping of a charged liquid. The signal of two droplets generated in this time frame can be visually identified by the perturbation.

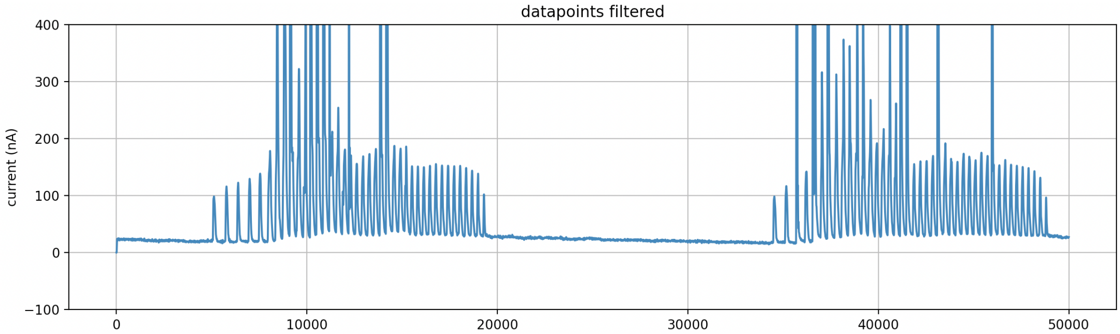

Figure 2.3: Current measurement sample of dripping spraying mode. This graph represents 0.5s sample.

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 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. Equation 2.1 calculates the current flowing through the charged liquid during a Taylor[9] cone jet.

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

Where,

$$I : current\ emitted\ [A]$$

$$Q : flow\ rate\ [m^3/s]$$

$$\gamma : surface\, tension\, [N/m]$$

$$\kappa : relative\, dieletric\, constant$$

$$K : electric\, conductivity\, [S/m]$$

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 circumstance 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 on what they have seen experimentally, and it is still being used as basis for EHDA researches.

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

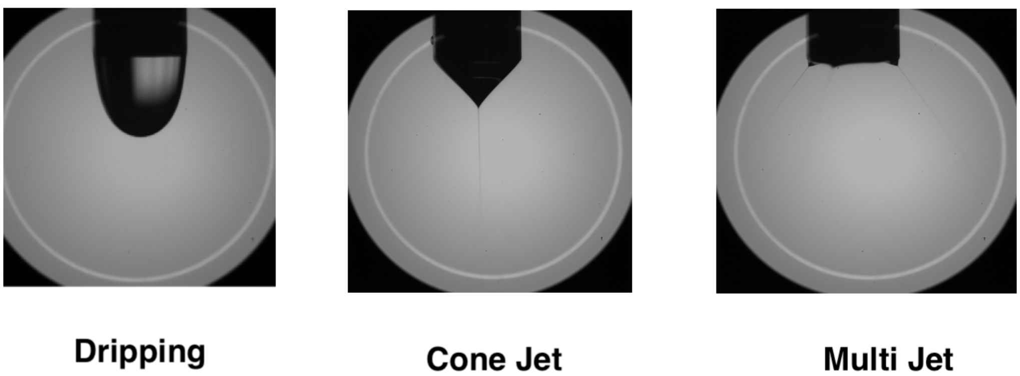

Figure 2.4: Fundamental electrospraying modes.

About the classification, there is not a convention in literature. Some authors can describe more classifications than others, hence this work will focus on 5 electrospraying modes as will be explained below.

2.4.1 Dripping

When the applied electric field is insufficient to alter the shape of the meniscus, the liquid droplet typically has a size larger than that of the capillary. Additionally, there are low-frequency intervals between each drop, indicating that the droplet is in a dripping mode. With the increase of electric field, starts a phenomenon called field enhanced dripping, where droplet size decrease and frequency starts to increase in order to keep the same volume of liquid flowing.

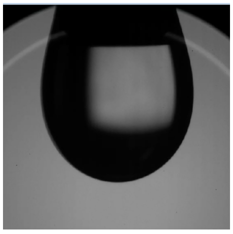

Figure 2.5: Dripping examples.

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, situation called electric 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 assume 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 when the electric potential increase, 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.

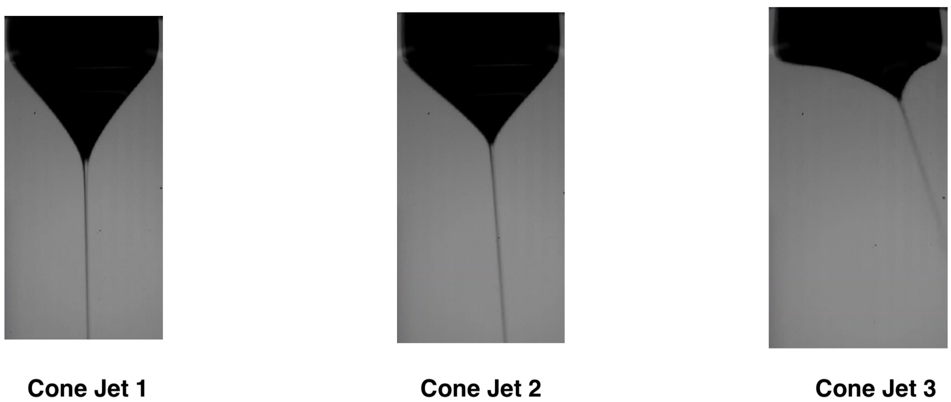

Figure 2.7: Cone Jet examples.

2.4.4 Multi Jet

Zeleny's (1915)[11] paper was the first reference to multi-jet mode. In his experiments it 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 it is called 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. It was 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 to as 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.

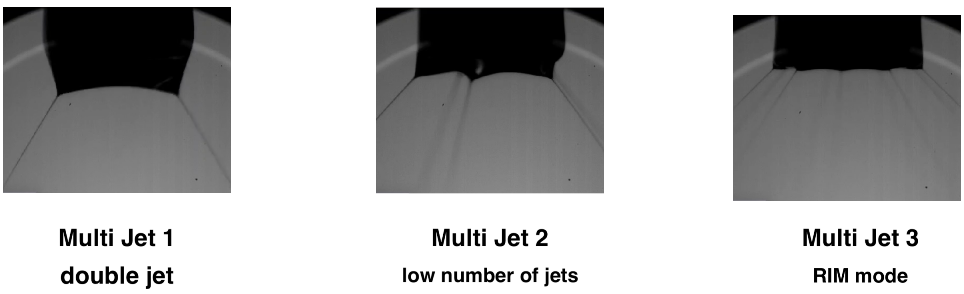

Figure 2.8: Multi Jet examples.

2.4.5 Corona

At even higher electric potentials, above of multi jet, the insulation between the electrodes start to breakdown, achieving an event of discharge, referred in this work as corona. 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. The previous student work was focused on how to predict that mode with the current analysis for safety in industrial applications.

2.5 Chapter conclusion

This chapter presents some electrospray theory that was important for the development of this project: Firstly, a brief review about the physical concept behind the experiment. Then, it is introduced how current analysis can be useful for applications, citing works already done with this approach. Finally, ending up the chapter defining what spraying modes classification will be considered and its properties.

Chapter 3

System Description

In this chapter, the equipment used will be discussed 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 the 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 measurements disconnected from an external power supply, which increases 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. In addition the WifiScope has an open source interface library for python, [13], making it easier to integrate with the automation routine.

c) Humidity and Temperature sensor

The stability of the system is affected by many physical effects. Evidently, it favors the system control having more parameters analyzed. The surface tension force, for example, is dependent of the liquid-gas interface on the meniscus, hence, the surrounding gas must be constant and so its humidity. Also, temperature is a variable that interferes 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 to the routine.

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 a new controllable variable, the flow rate. The spraying mode can be controlled using the two main variables that affect the system. It brings more complexity for the system since now it is a multivariable control. Controlling also the flow rate provides this project a new dimension in the system giving freedom to explore the flow rate properties. With this new input variable the control system is MISO (Multiple Inputs Single Output).

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

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

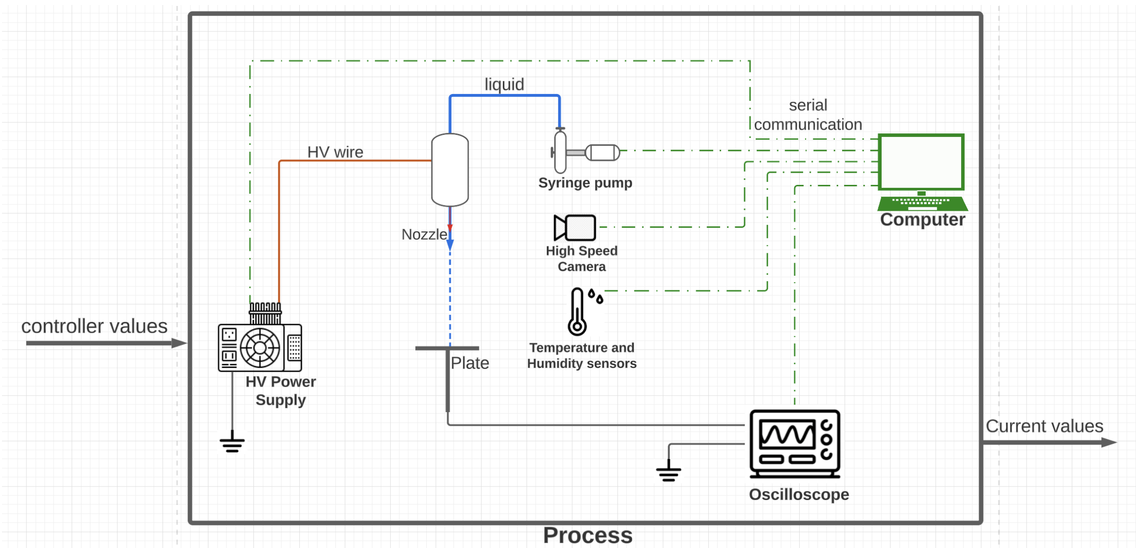

Figure 3.1: EHDA automation system setup.

There are also many minor variables that affect 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 below.

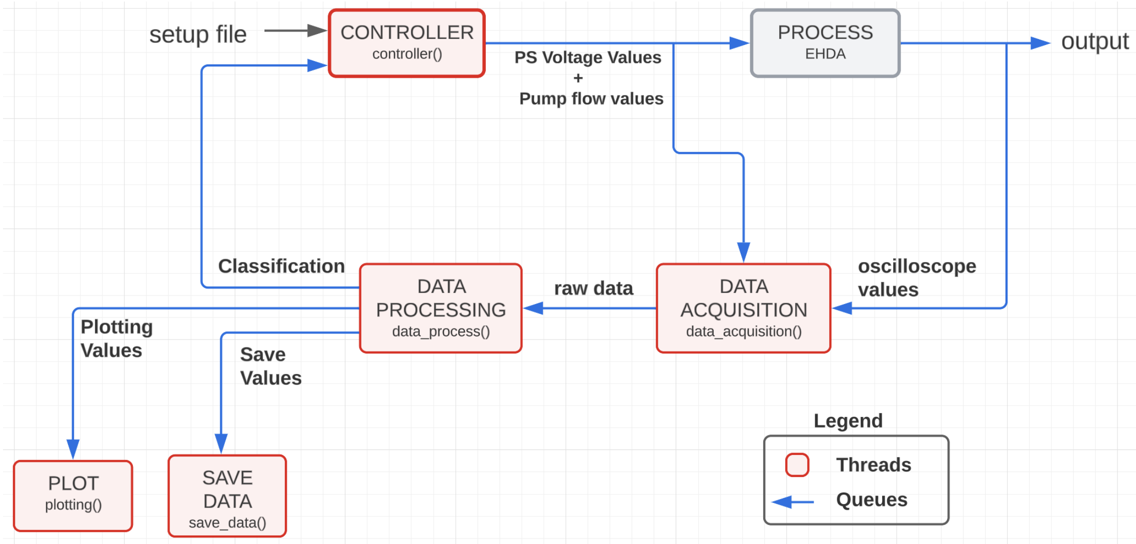

Figure 3.2: EHDA automation closed loop control system model implemented.

3.2.1 Threading and Queues

In order to implement this closed loop control model in 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.

The controller thread is responsible for sending to the power supply the set voltage values and to 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, the setup config file with information about the liquid and the setup used such as geometric parameters of the nozzle to plate system. As output, the commands to the actuators.

The data acquisition thread is responsible for concatenating the current, voltage, flow rate, humidity and temperature data and concatenate it into one sample. The data processing is responsible for calculating the statistical values from the raw data and classify it in the respective spray mode for that sample. 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, instead of having all data together in program memory to be saved in the end, it is saving each sample in real time during the experiment. 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 ensures that the data is stored separately in enumerated samples. This prevents any loss of data in the event of errors during experiments and significantly enhances the usability of the data for in-depth analysis. Each hour-long experiment generates approximately 6 GB of data, and effectively organizing such a substantial amount of data was a crucial enhancement in the software. To efficiently work with and analyze the data, the widely-used Python framework, pandas Dataframe, can be utilized with the following 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 slow to read and large to store. For that, saving the data frame in a compressed type of file called feather is much faster to work with it.

This is only running function that should run in the main thread because of the plotting library *matplotlib* incompatibilities with running outside the main function. The plotting function 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 it is explained 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 this routine and for being able to be used in multipurpose applications the 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. A current data frame of 0.5s is acquired with 100 kHz as sampling frequency. Therefore, each current sample holds 50 thousand current values.

Through statistical analysis in these signal such as mean, median and standard deviation the classification is done. Figure 4.1 illustrates data samples separated by statistical method, depending on the range in mean, mean/median and deviation/mean.

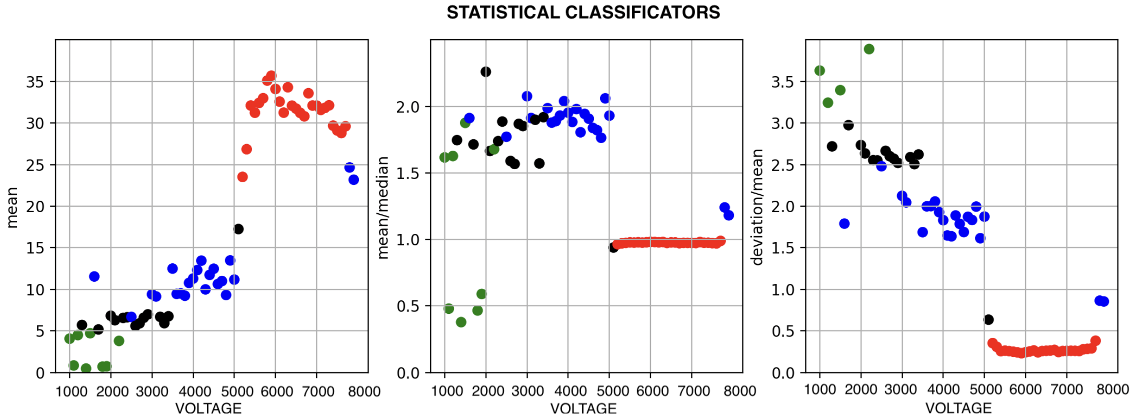

Figure 4.1: Sample classification using statistical values. Colors: Green = Dripping; Blue = Intermittent; Red = Cone Jet.

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 it was 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, the classification of the multi jet was implemented when the current mean is 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 ← "Undefined";
    mean ← sample.mean;
    deviation_mean ← sample.std_deviation/sample.mean;
    mean_median ← sample.mean/sample.median;
    if mean > 5 then                                              ▷ Sjaak classification [5]
        spray_mode ← "Dripping";
        if 2.5 < mean/std_deviation < 2.5 & mean/std_deviation > 0.3 then
            spray_mode ← "Intermittent";
        end if
    else if mean/std_deviation < 0.3 then
        spray_mode ← "ConeJet";
        cone_jet_mean ← 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 × mean then
            spray_mode ← "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 the control model in Figure3.2, 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_thread.

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.

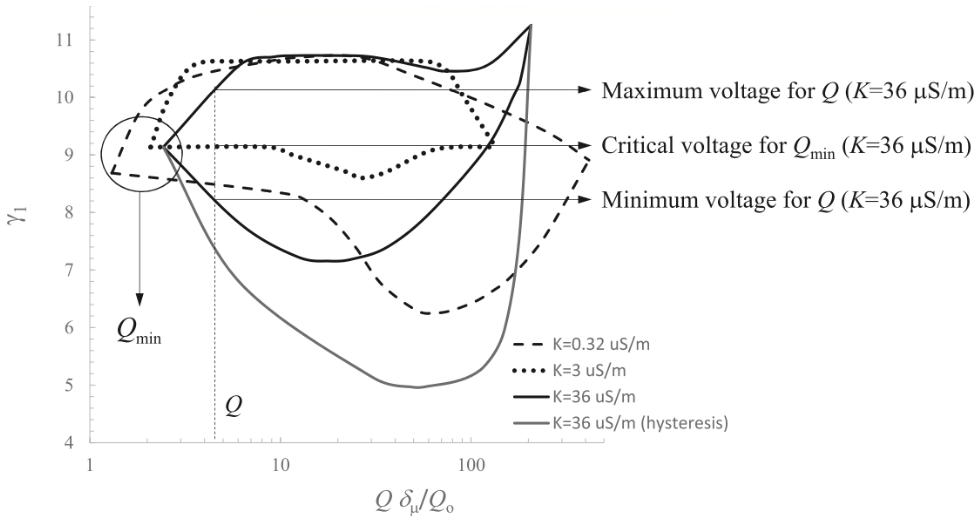

Figure 4.2: Domains of existence (stability) of Taylor Cone Jets. [4] . The window formed by these points is where the system operated in the stable cone-jet. Operational windows depend on the liquid and configuration setup. Different windows are represented for different liquid conductivity. The X and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

In Figure 4.3 it can be seen 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.

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

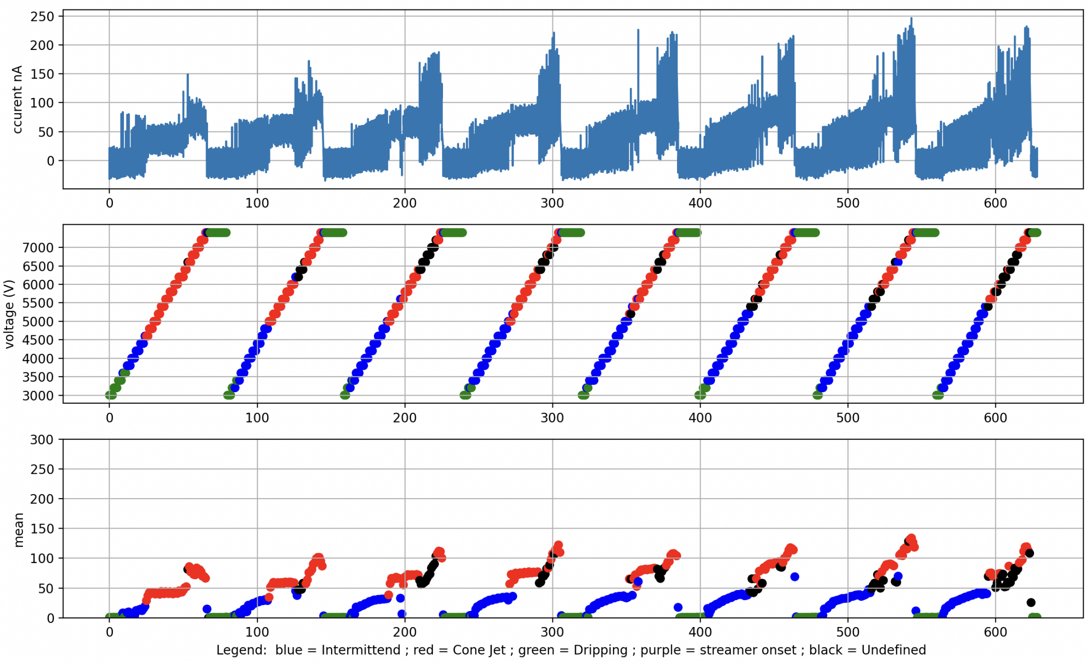

Figure 4.3: Mapping Experiment data collected. The Figure has 3 graphs with shared x-axis representing the samples collected. The first is the current values collected through all the experiment. The second is the voltage values applied in each window of data collected. The colors represent the spraying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

With all the data collected, classified and saved in real time, further analysis and studies can be done. For example, Figure 4.4 illustrate the data automatically classified and displayed in a Voltage X Flow rate range of spraying modes with a specific liquid setup so that a comparison on automatic results with previous researches can be done, such as showed in Figure 4.2, validating the algorithm.

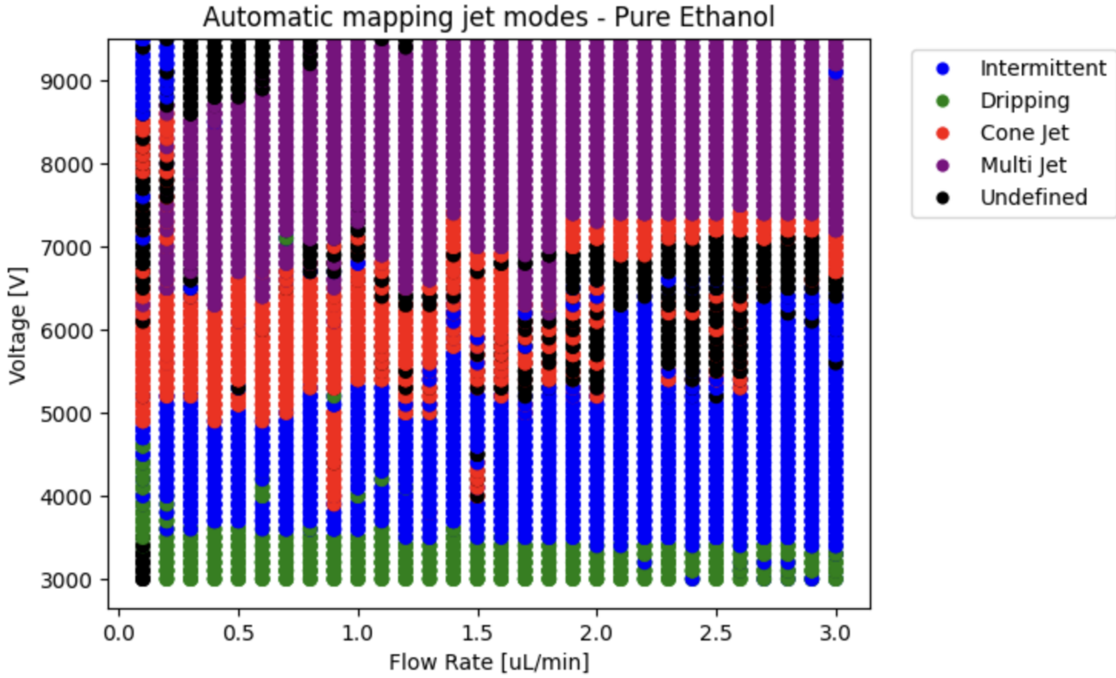

Figure 4.4: Mapping Experiment for pure ethanol in ambient conditions. The map shows the stability region of each electrospraying mode in the voltage and flow rate range.

4.2.4 Control

The control sequence is the only from the 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 the project goals. Next chapter 5, it will be discussed the results reached.

Chapter 5

Results

This chapter will present the results obtained from this project. Firstly, it will be demonstrated how the automation routine enhanced the research experiments. Next, a discussion on the validation of the classification results. Finally, a showcase of performance on 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, it was 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. Running a computer routine that automatically sends commands to the power supply and pump machine allowed obtaining 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.

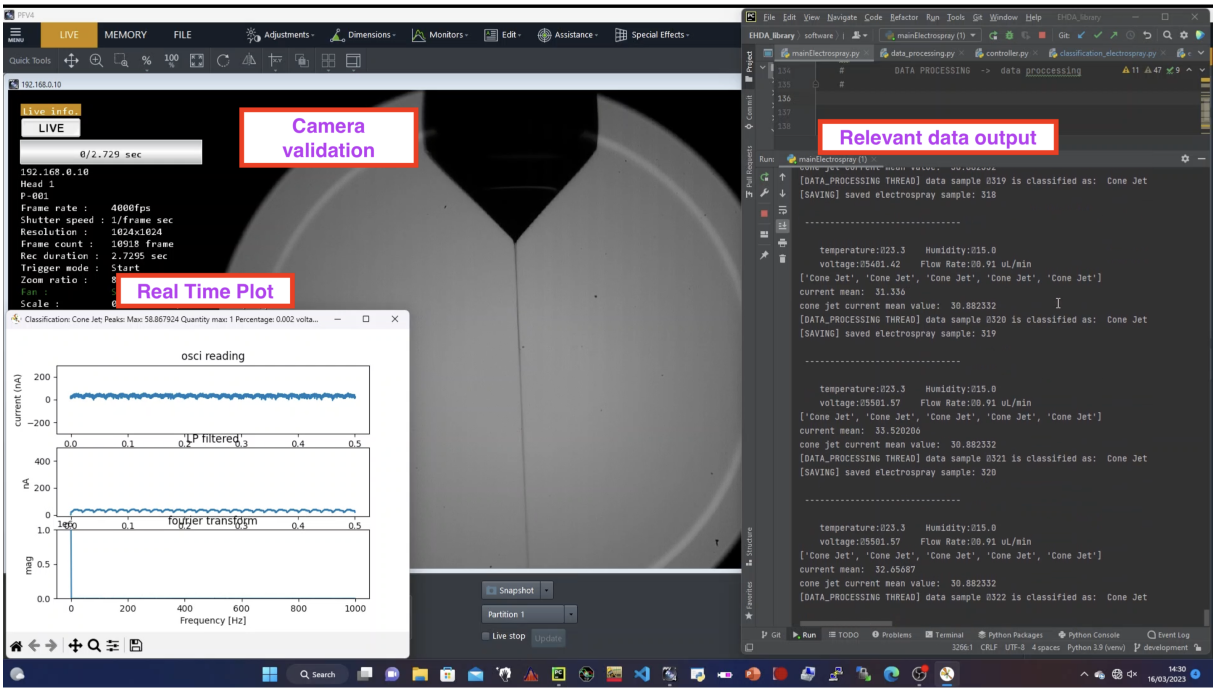

Figure 5.1: Print screen of the window shows user interface during the experiment. The image generated by the high speed camera can be seen in the background. The routine code running in pycharm software on the right side. And also real time signal plotting of the current data on the left side.

5.2 Classification

The classification results in the 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.

The classification result will be categorized 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

The first classification results were made exploring the voltage ranges. For that, a flow rate was fixed 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, an increasing voltage with steps of sizes 50V and time between each step of 5 seconds was implemented. 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()*.

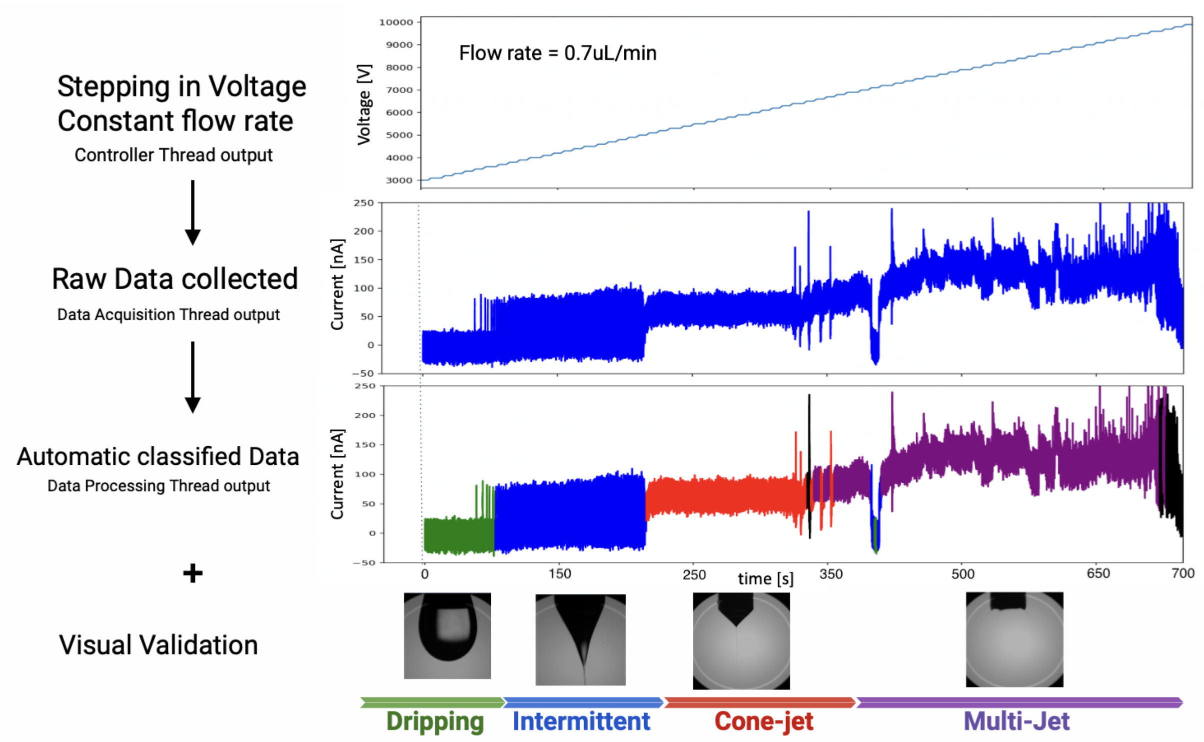

Figure 5.2: Automatic electrospray classification through the step routine.

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, it was made a manual map seen in figure 5.2.2.

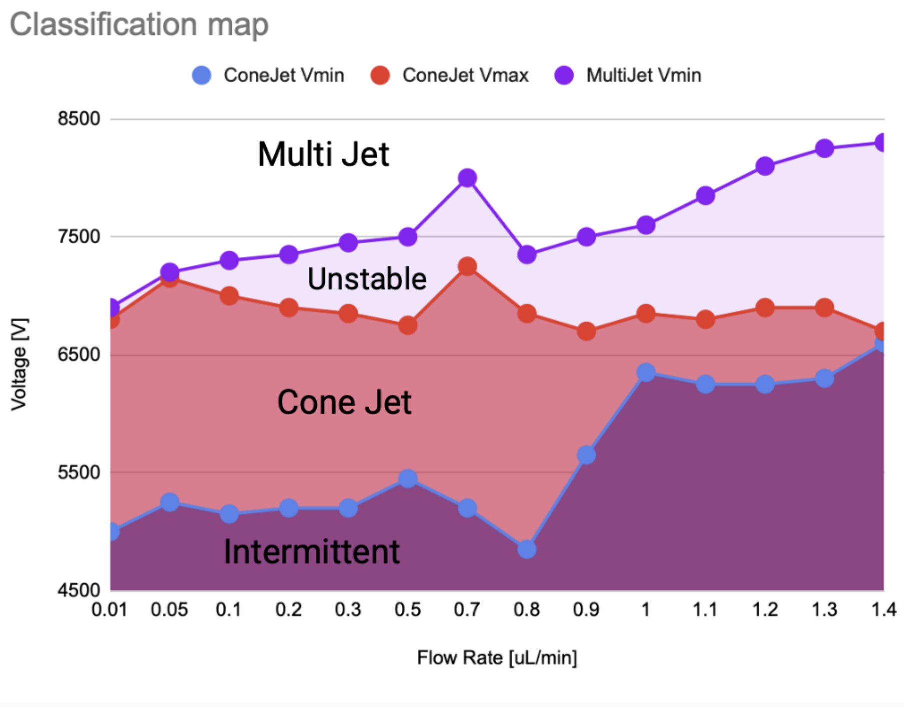

Figure 5.3: Experimental spraying modes regions of pure ethanol.

In order to validate the 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).

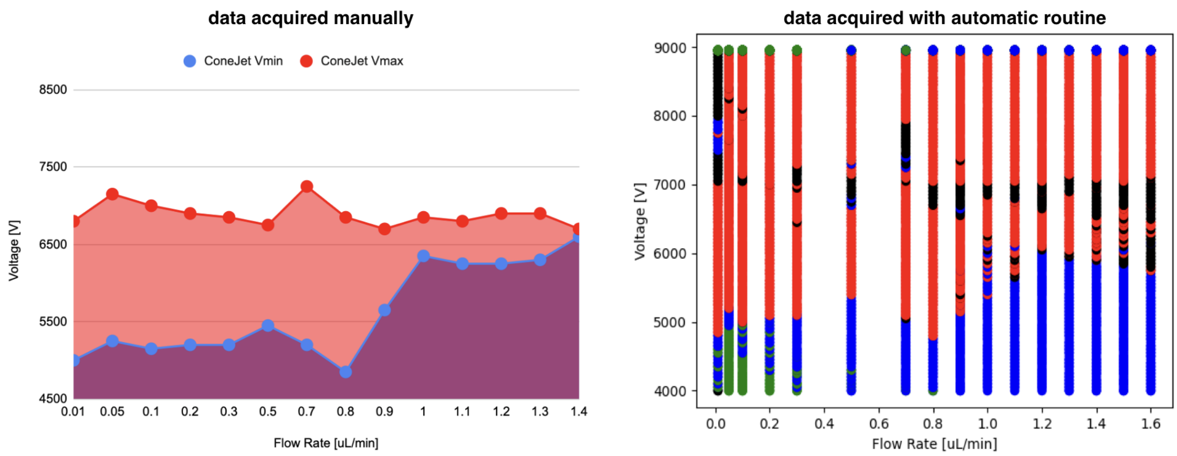

Figure 5.4: Cone jet stability region for pure ethanol experiment 1.

With the development of a multi jet classification using the logic explained in section 4.1, the same experiment was repeated, as shown in Figure 5.5.

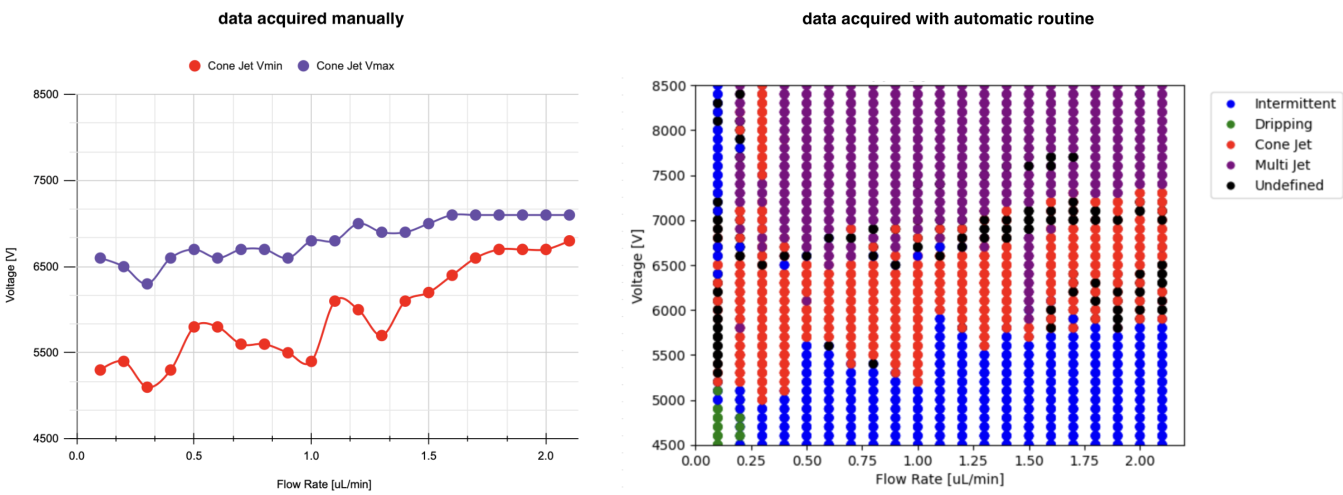

Figure 5.5: Cone jet stability region for pure ethanol experiment 2.

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, the data displayed in Figure 5.6 using the non-dimensional numbers used in his work[4].

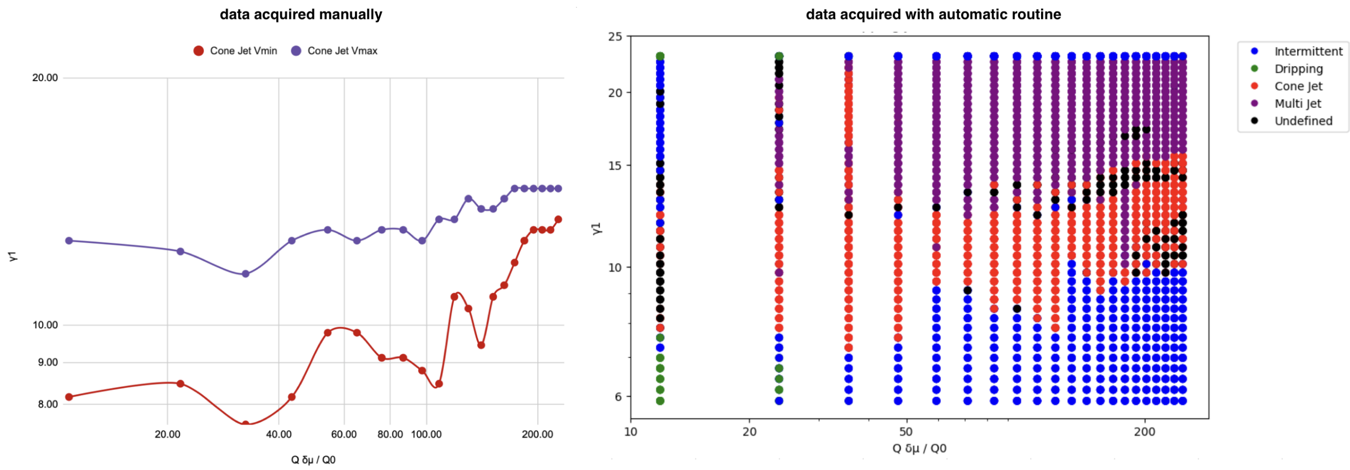

Figure 5.6: Cone jet island manual experiment 4.

The results of mapping experiment corresponded visual, literature and automatic regions of classification, giving authentication to the 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, a simple controller project could be implemented, as described in section 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 the flow rate was manually changed. First graph shows current acquired. It starts as intermittent state, with an oscillating signal. This can be validated 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, a manual increase in the flow rate was done, serving as a perturbation of the system, and it can be seen 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 once more the flow rate manually, and again the system automatically adjusting the voltage.

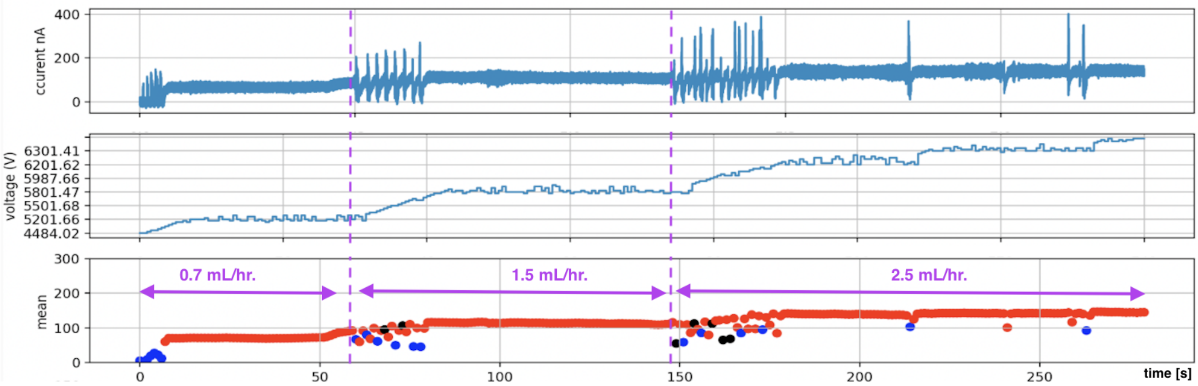

Figure 5.7: Simple controller validation experiment. For this experiment, after waiting the system to stabilize in the desired spraying mode it was made a perturbation test changing the flow rate. The classification legend follows the same color reference in all project.

After 200s of experiment the system 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 the results of this project were exposed. The automatic routine, real time classification and control were all achieved and implemented. However, they can all be improved. Next Chapter 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, a proposal is to prioritize the improvement of the classification step. Ideally using machine learning or another algorithm to ensure accurate classification with a broad range of liquids. By achieving a reliable classification, a more effective controller logic or approaches can be implemented. In this regard, a concept of a fuzzy controller will be presented 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 the 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 it does not 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.

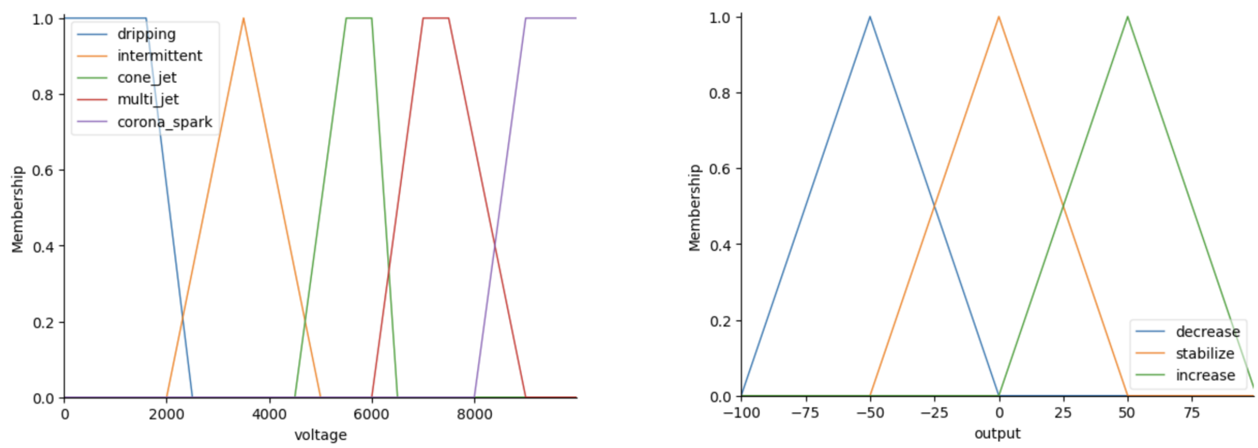

Figure 6.1: fuzzyfication of input and defuzzyfication of output.

The controller is an algorithm that calculate the output with a geometrical calculus on the membership functions of the input and output according to the 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.

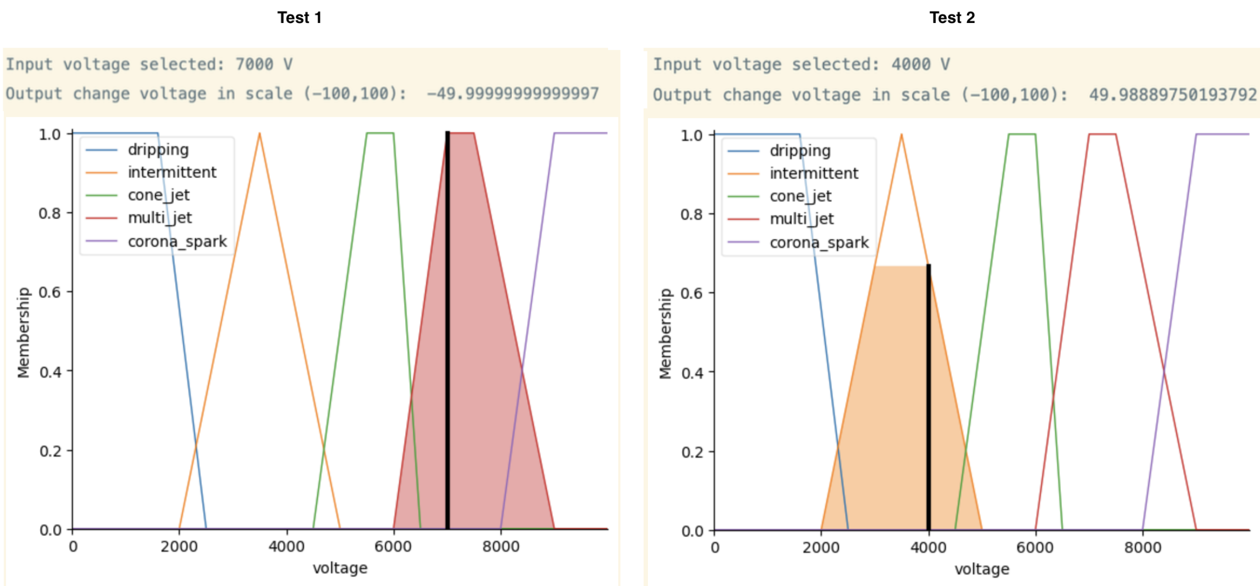

Figure 6.2: Test implemented to validate the concept of fuzzy controller in this project.

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.

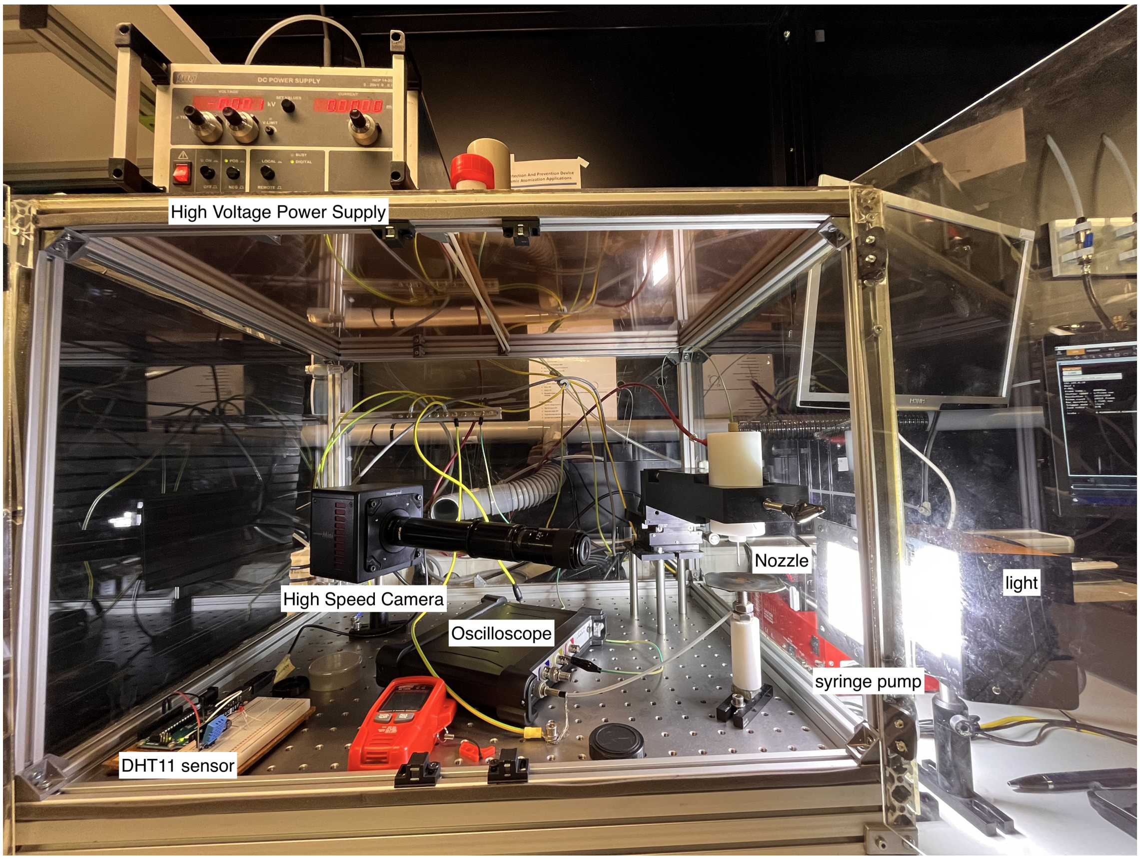

Figure A.1: EHDA automation system setup used for experiments.

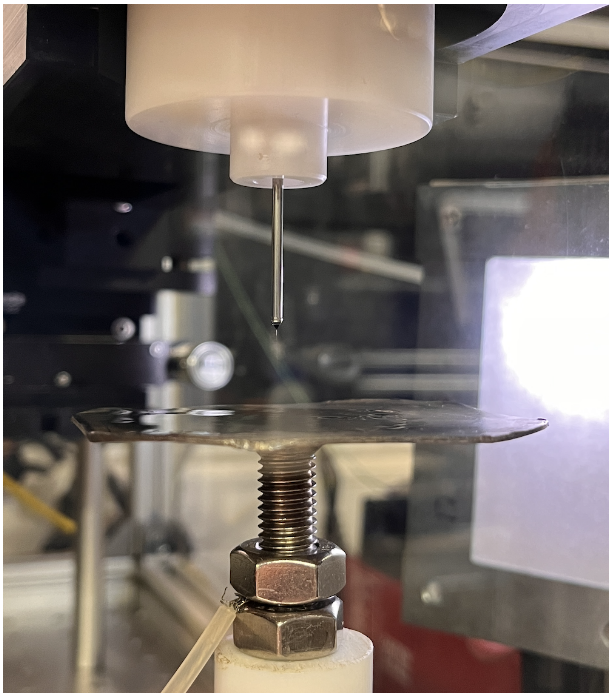

Figure A.2: EHDA picture of the electrified syringe.

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, it is being used 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.