Charge stability in Triboelectric polymeric films

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I would like to thank my family. My parents who always supported my curiosity and without whom none of this would be possible. To my sister and grandparents, who always were there when I needed.

I would also like to thank my friends, whose support stopped me from going mad.

Resumo

Neste trabalho eu completei várias experiências de modo a testar a ração de diferentes TENG a várias temperaturas (25ºC a 100ºC) e humidades (30% a 95%), de modo a melhor entender como estas mudanças afetam o decaimento de cargas em camadas triboelétricas. Todos os TENG tinham uma camada triboelétrica, acima de uma camada de cobre, acima de uma camada de vidro. Os TENG foram pressionados durante um certo período de tempo (4.5 horas ou 30 minutos), antes de serem sujeitos a mudanças de temperatura ou humidade, depois das quais eles foram pressionados novamente (mesmo período de tempo) para determinar a diferença na voltagem máxima. A partir do estudo eu pude concluir que o aquecimento tem um efeito negative na voltagem máxima, este efeito é constante para temperaturas entre os 40ºC e os 100ºC. Para um TENG de nylon-PTFE, o efeito não surgiu aos 50ºC, possivelmente por ser negado por uma mudança de fase. Humidade não afetou os TENG de forma relevante. Por último, o aquecimento do TENG a 100ºC mostrou ser uma boa maneira de remover falhas na camada triboelétrica e de adicionar carga, provavelmente causado pela oxidação do cobre exposto.

Palavras-chave: Triboelectricity, TENG, Triboelectric properties, Charge decay, Spinning, Electrospinning.

Abstract

In this work I have done several experiments to test the reaction of different TENG to variations in temperature (25ºC to 100ºC) and humidity (30% to 95%), in order to determine how these changes effect the charge decay of triboelectric layers. All TENG had a triboelectric layer, atop a copper layer, atop a glass layer. The TENG were pressed for a given time (4.5 hours or 30 minutes) before being subjected a change in either temperature or humidity for 30 minutes, after which they would be pressed again (same time) to determine the difference in maximum voltage. From the study I was able to conclude that the heating had a negative effect on the maximum voltage, said effect remained constant in temperatures ranging from 40ºC to 100ºC. For a nylon-PTFE TENG, the effect was not noticeable at 50ºC, theorized to be counteracted by a phase sift. Humidity did not affect the sample in any major way. Lastly, heating the TENG at 100ºC showed to be a valid way to remove gaps in the upper layer and add charge, though to be due to the oxidation of exposed copper.

Keywords: Triboelectricity, TENG, Triboelectric properties, Charge decay, Spinning, Electrospinning.

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List of Abbreviations

TENG	TRIBOELECTRIC NANO GENERATOR
RPM	ROTATIONS PER MINUTE
PTFE	POLYTETRAFLUOROETHYLENE
PVDF	POLYVINYLIDENE
PDMS	POLYDIMETHYLSILOXANE

Introduction

In our modern world sensers are everywhere, when someone thinks about this fact their mind is immediately drawn to things like CO_2 sensors, cameras, or motion detectors, however, one of the common signal or devices receive from us is touch. In fact, if you are reading this on a computer, you are using one of these sensors right now to move the page, either a button, mouse wheel or a touch screen. These methods of man machine interaction are so common that we often don't even think of them as such.

Another reason people might not think much of these types of sensors is that their function is very user reliant. When a science fiction book describes a suit that senses the wearers every movement we don't imagine a suit with hundreds of buttons on the inside, clicking every time movement is attempted. We imagine a skintight suit, maybe more plastic looking than cloth, but whose sensors are not noticed in their operation.

This idea is, for now, fiction, but the seeds of its creation have already been planted in the form of triboelectric sensors. Triboelectric sensors are, as the name implies, sensors that work based on triboelectricity, where a charge is generated by movement, thus, they can be passive motion generators. Their structure allows for great flexibility, small size and a variety of modifications to suit different necessities.

These triboelectric generators have been proven to be able to power small devices, becoming an enticing addition to the green energy roster. When we add the fact triboelectricity can be generated by both gas-solid and liquid-solid paring, we immediately start thinking of new aero and hydro generators.

Of course, for this dream to become a reality, we need to understand how triboelectric generators work in the physical sense, as in, what forces guide their charge generation, and in the practical sense. The second is the one this thesis will focus on, to be more specific, the role variations in temperature and humidity have on charge loss. To be more general, how variations in temperature and humidity effect the electrical output of a generator.

To achieve this, several materials and temperatures were tested, their outputs gathered and analyse, which the reader shall see in due time, but first, we should start by giving a more complete explanation of the principals in use and the knowledge already gathered on the topic. Besides triboelectricity, there will also be a quick explanation on two fabrication methods used: spinning and electrospinning.

1. Triboelectric Nanogenerators

1.1. Triboelectricity

Triboelectrification is the transference and accumulation of charge that occurs when two materials are rubbed together. The materials in question can be solid, liquid or gas, with the intensity of charge accumulation varying from material to material.[1]

Static electricity is an example of this effect, with the rubbing of socks on carpet leading to charges travelling from one to the other, causing the person wearing the socks to become electrically charged. When said person touches something like a doorknob, the charges travel from one to another, creating a spark. Another example is lightning, where air particles rub on water droplets, causing them to gain charge, until it is released in the form of lightning.

The previous paragraphs may make the process seem simple, however, the act of rubbing two materials together results in many phenomena such as deformation (plastic/elastic), heating, and fracturing. Outside factors such as humidity and heat, as well as additional layers beneath the material will also affect the charge transference. Add all this influences to the fact that a complete theory would have to take into account solids, liquids and gases, and it becomes clear the difficulty in explaining triboelectricity.

Because of this difficulty, most studies are done experimentally. In recent times, the study of triboelectricity has gone from trying to prevent fires and accidental discharges, to the study of TENG, TriboElectric NanoGenerators.

1.1.1. Triboelectric Series

From the previous segments, one would realize how important the ability to gain or lose electrons is to the triboelectric effect, as such, the triboelectric series was formed to give insight into how materials would behave.[4]

A triboelectric series ranks materials based on their ability to gain or lose electrons, where positive mean the material tends to lose electrons, and negative that it tends to gain electrons. Meaning, to produce a good TENG, it is best to combine a positive material with a negative one.

The lists are not perfect, as some materials have shown to change behavior based on their partner, being possible for one material to become triboelectric with itself.[5]

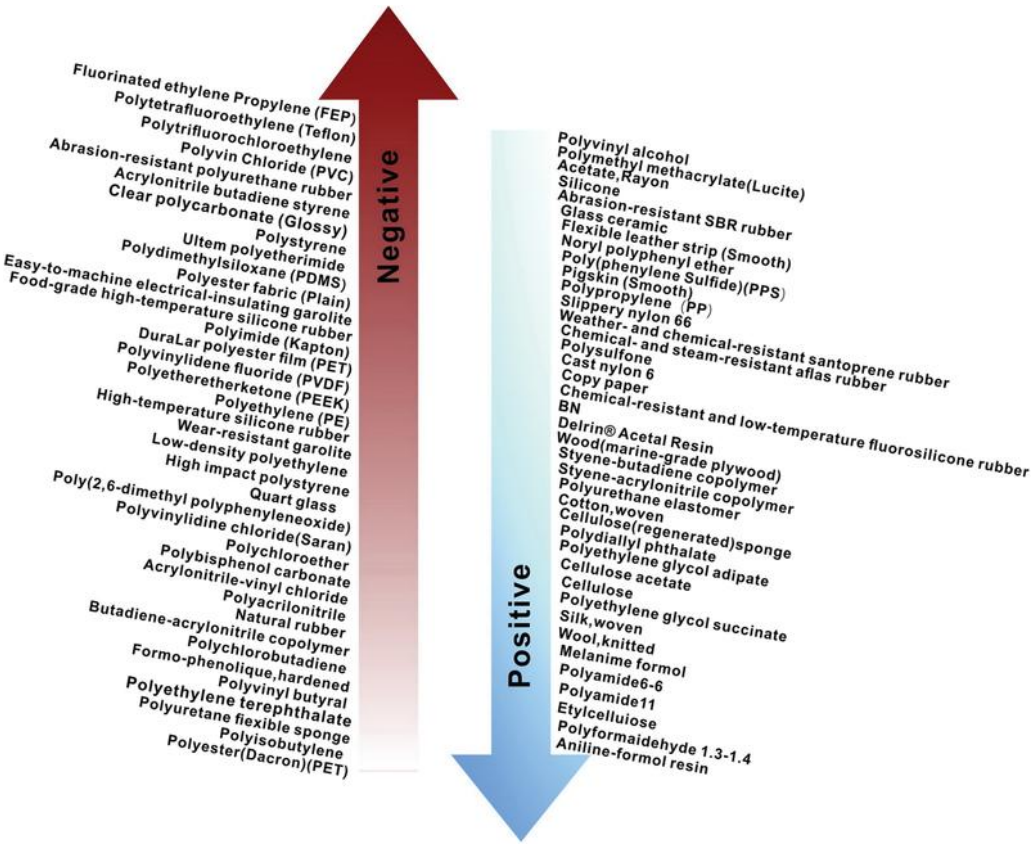

Figure 1 - Triboelectric series (taken from [3])

1.2. TENG

TENGs are devices that can generate an electrical current (and thus an electrical signal) through the triboelectric effect. In general, a TENG will utilize the different electric attractiveness of a material at different positions to create movement of charges, and thus current. In detail, there are five different modes, seen in figure 1. [2]

In the contact separation mode, two triboelectric layers are each connected to one conductive layer, these two conductive layers are then connected by a wire. When the two triboelectric layers touch, they will accumulate charges of opposite polarities. After that, the separation of the layers will result in a potential difference. Since both conductive layers are connected, the potential difference will lead to a movement of charges between them, thus resulting in an electrical current.

In lateral sliding mode, we have the same set-up as before, except we slide the two layers instead of separating them. This will still result in the creation of potential and thus an electrical current.

In single-electrode mode, we again go to the first scheme, except, instead of both triboelectric layers being connected by the conductive layers, only one side has a conductive layer, connected to the ground. Both layers still join and separate, but the current occurs between one layer and the ground, instead of between both layers.

In freestanding mode, we have a single triboelectric layer and two conductive layers, the conductive layers are connected by a wire. The triboelectric layer slides back and forth between the conductive layers. This leads to a potential between the conductive layers, and thus an electric current.

In non-contact mode, we have the same scheme as the first, except the layers do not touch each other after being charged. The difference in potential is only caused by relative motion. The charge in both layers tends to disappear with time, depending on several internal and external factors (temperature, humidity, other layers …).

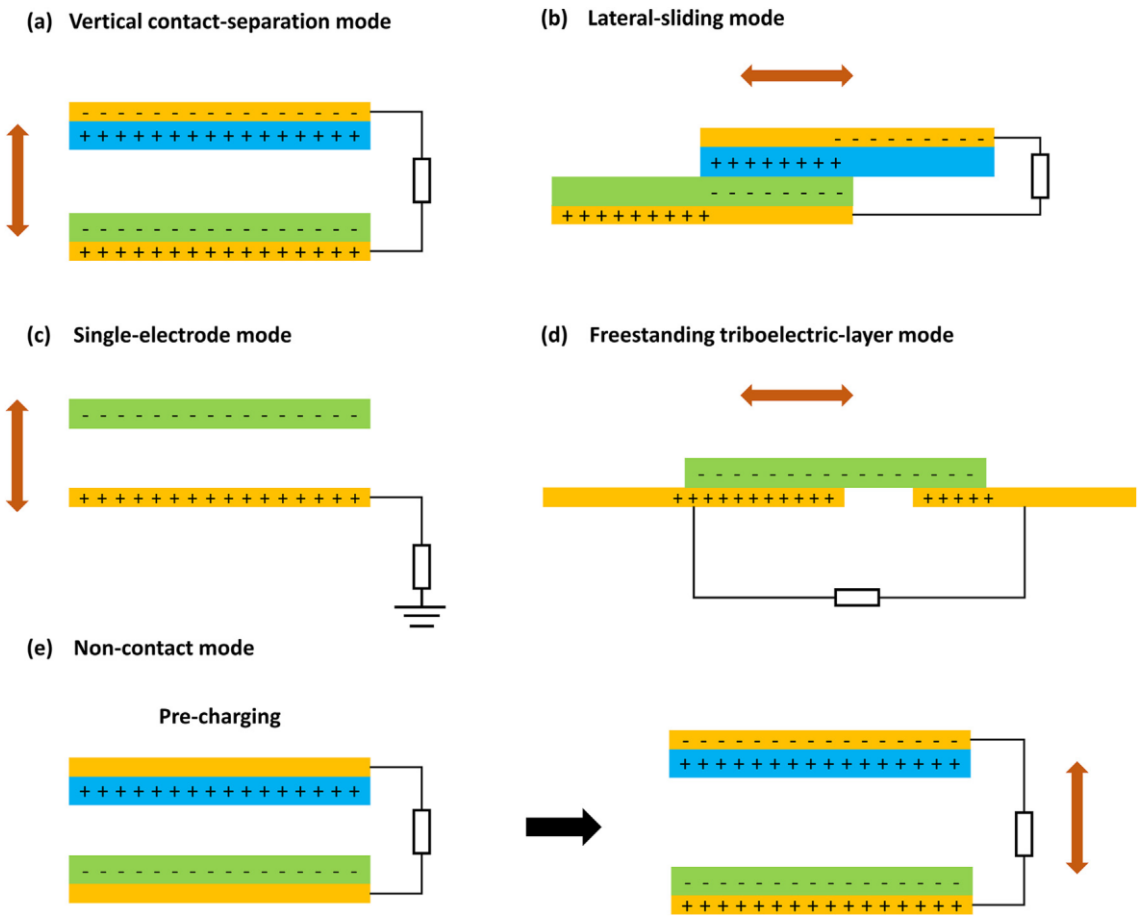

Figure 2 - Different configurations of TENG (taken from [2])

1.2.1. Charge dissipation

As stated previously, when two triboelectric materials touch, electrons will go from one to the other. This does not occur all at once, it takes several contact separating cycles until the TENG reaches a maximum voltage output. This saturation point depends on various factors, being a balance of charge accumulation, how much charge is gained, and charge dissipation, how much charge is lost.

Charge dissipation is the natural tendency of electrons to move to where there is less negative charge. Since the triboelectric materials will be charged, they will be unbalanced in relation to their environment and will either gain or lose electrons because of it. Said loss occurs in two main ways: air breakdown and dielectric breakdown.[6]

Air breakdown occurs when the charges in the material are strong enough to ionize the air, meanwhile, dielectric breakdown occurs when the charges are strong enough to turn an isolating material into a conductive one. In other words, they represent charge

dissipation into the air and into the ground, respectably, with the air breakdown being a bigger factor in the total charge dissipation. [7]

In addition, there are the more unpredictable effects such as tears in the layers, and the ambient influences of temperature and humidity which will be talked about in following sections.

1.2.2. Possible Uses For TENG

One possible use for TENGs is in biomonitoring. Since TENGs are thin and generate their own charge, it is possible to instal such a device inside a person to measure, per example, heartbeat or lung movement.

Another possibility is to integrate them in infrastructure. By putting TENGs on roads, we could be able to better manage traffic without the increase energy cost of cameras or lasers.

TENGs could also be installed in clothing, either to generate energy for devices such as watches, or for sensing data. A sensor on the sole could tell if a patient is walking correctly, helping in physiotherapy.

Continuing on the previous point, by setting up TENGs in areas with a lot of movement, such as the sea (waves), it would be possible to harvest green energy.

1.3. Influences on TENGs

Due to the complexity and number of influences in the triboelectric effect, few studies have been done on the impact of temperature and humidity on charge dissipation. This study will then try to counteract this fact.

1.3.1. Effects of humidity on triboelectric effect

Both charge accumulation and charge dissipation are affected by humidity. For the purposes of this work, the effects of humidity on charge dissipation will be more relevant.

As shown in [9] humidity increases both charge dissipation into the outside and charge lateral diffusion, with the latter only being relevant under higher humidities. These effects were seen in time periods of less than 30 minutes.

The increase in charge dissipation can be explained by the water molecules in the atmosphere neutralizing the charge via interaction with ions (air breakdown). The lateral diffusion would be caused by an increase in droplets deposited on the surface of the film, which would facilitate the movement of charges, this could also lead to charge loss due to absorption. Both factors would increase due to increased relative humidity, thus the increase in charge dissipation and lateral diffusion. [8]

The previous statements all referred to surface affects, however, if the layer are permeable, water droplets may also condense inside the TENG, increasing internal diffusion.

1.3.2. Effects of temperature on triboelectric effect

Much like any other material, when a TENG is heated up, its electrons will gain energy, making it easier for them to overcome the materials work function. The higher the temperature, the easier it will be. It is also important to consider the physical characteristics of the materials used, since high temperatures may lead to breakdowns in structure.[10][11]

When the Teng is being used, the rise in the energy of electros is both useful and detrimental, as it will cause charges to more easily travel from one side to the other, increasing maximum voltage, but it will also increase charge loss to the environment, reducing maximum voltage.

The influence of energy when the TENG is not in use is more obvious, as without the contact to reintroduce charge, the only thing left is the loss to the outside, which is worsened by the temperature. Perhaps for this reason, less studies have been done on this front.

1.4. Possible Materials for TENG

There are many materials suitable for the production of TENGs, with each having their own characteristics and strong suits. Below are the materials that were used in this thesis, alongside some context.

1.4.1. Nylon

In this thesis, nylon was selected as a triboelectric material due to its relatively high position in the triboelectric series, meaning it tends to lose electrons when in contact with other materials. Nylon also had the benefit of having been used previously by my co-supervisor, meaning that manufacturing procedures and raw material were already available.

As a member of the polyamide family, characterized by amide linkages, nylon exhibits high chemical resistance, tensile strength, and flexibility. These properties, together with their triboelectrification capabilities, make it particularly suitable for TENG applications.

1.4.2. PTFE

PTFE, or polytetrafluoroethylene, is a synthetic fluoropolymer of tetrafluoroethylene. Its main characteristics are its chemical inertness and low friction. For this study, it is also relevant to point out it begins to decompose at 260ºC and its propensity for creep. Unlike nylon, it has low position on the triboelectric series.

As stated previously, nylon occupies a high position on the series, meaning that nylon and PTFE form a great pair for TENG. When the two touch, nylon will have a tendency to lose electrons, while PTFE will tend to gain electrons.

1.4.3. PVDF

PVDF, or polyvinylidene difluoride, is another polymer that displays flexibility and chemical inertness, it also displays low density and thermal resistance. One of its most interesting characteristics may be the fact that it is piezoelectric.

Even if not to the same degree as PTFE, PVDF also occupies a low place in the triboelectric series, making it a good pair to nylon.

1.4.4. PDMS

PDMS, or polydimethylsiloxane, is a polymer with many uses, not only as a solid but also as a liquid, displaying low surface energy, chemical inertness, and flexibility. As a liquid, it is used for lubrication, as an antifoaming agent in fast food and in cosmetics. As a solid, it is used in hydrophobic coating and soft lithography.

In terms of the triboelectric series, PDMS is lower than PVDF, but not as low as PTFE, thus being a good polymer for testing.

2. Methods of fabrication

In order to better understand the nature of the processes taking place, various materials were used. The fabrication of suitable devices was undertaken via different methods, some used commercial material, some were electrospun, and others used spinning.

Despite this, all materials were placed atop a layer of copper, placed above a glass substrate. Wires were then soldered to the copper.

I will start by explaining the practices of electrospinning and spinning, as well as the set-ups necessary for this these. After, I will describe the processes for the fabrication of the samples (by material) as well as the results of said method.

2.1. Electrospinning

This section will start by explaining the idea of electrospinning, before describing the set-up I used for it.

2.1.1. Theory

Electrospinning is a method used to produce nanofibers using a high-voltage electric field. To do this, several items are required: a high-voltage power supply, a syringe pump, a spinneret (hypodermic needle with blunt tip) and a conductive collector. Additionally, having the system in a controlled environment, isolated environment is also advised. Finally, a solution containing the desired material is also necessary.[13]

To achieve electrospinning, we need the syringe pump to move the solution to the spinneret. A high voltage (0~30 kV) is then applied to said spinneret, causing an accumulation of charge in the fluid. When the accumulation reaches a critical point, the force of the electric field will overcome the surface tension, resulting in Taylor cone. A jet will be emitted from this cone, landing on the collector.

During the travel between the spinneret and the collector, the jet displays two distinct phases. During the near field, the jet will be continuously stretched as its diameter decreases, remaining parallel to the electric field thanks to the changing acceleration. On the other hand, surface tension and the viscoelastic force will prevent it from moving forwards, resulting in its deceleration.

Once the acceleration has stopped or become constant, instabilities will cause the jet to develop perpendicular motion, resulting in loops. Once this happens, we have reached the far field. Additionally, more loops may form during the travel, with each loop stretching the jet and thus thinning the resulting fibre.

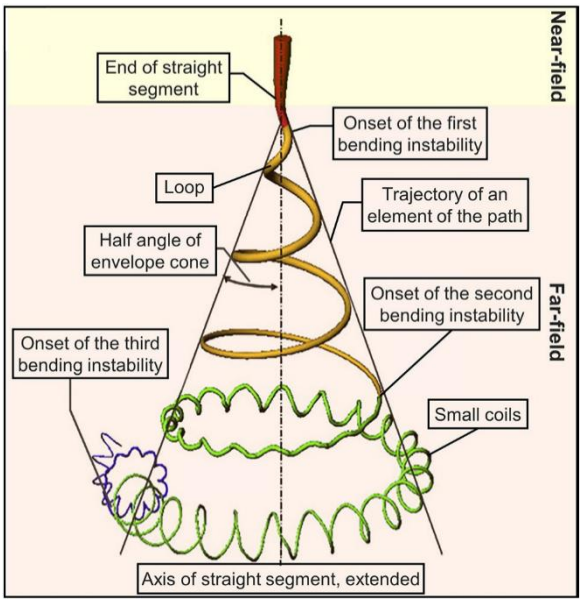

Figure 3 - Representation of the electrospinning jet (taken from [13])

All throughout the trip the solvent is evaporating and fibres are solidifying, so that, by the time the jet reaches the collector, we are depositing solid fibres.

From the above description it becomes clear that many factors affect the electrospinning process. From the solution, we have parameters such as viscosity, surface tension, conductivity, molecular weight and concentration. On the fabrication side: tip to collector distance, voltage, spinneret diameter and feed rate. Finaly, the ambient temperature and humidity.

All the aforementioned parameters will influence the resulting fibres, with many having interactions between each other. For example, decreasing the tip to collector distance will cause the same voltage to display a higher influence; a higher feed rate will most likely necessitate a higher voltage to prevent accumulation.

To achieve the desired result, one needs to vary the different parameters, not just the voltage or the feed rate. Furthermore, it is useful to have a controlled atmosphere, such as in a hotte, so as to prevent changing temperature and humidity from altering the characteristics of the solution (namely rate of solvent evaporation), and thus, the fibers. Additionally, a ventilation system is highly advised to prevent the accumulation of evaporated solvent, especially if the solvent is harmful.

My set-up has a static collector which is covered in a substrate (i.e. aluminium), however, other types of collectors exist, such as spinning collectors. There is also the possibility of having multiple spinnerets, or of having coaxial needles to create fibres of two different materials. Still, for our purposes, a single simple spinneret was enough.

2.1.2. Set-up

The set-up I used can be seen in figures 4 and 5. The first figure shows the initial set-up where almost all of nylon testing was done, while the second shows the set-up for PVDF. As the reader can see, the second set-up is in a hotte and possesses a camera.

The change to a hotte had three underlying reasons: first was safety, as being located inside a hotte would further prevent gases from affecting the user; second was control, as a hotte would have better control over temperature and humidity[1]; third was ventilation, as a hotte would allow for better handling of gases, as oppose of a tube going out of a window.

To better understand the atmospheric parameters, a thermometer was put inside the device.

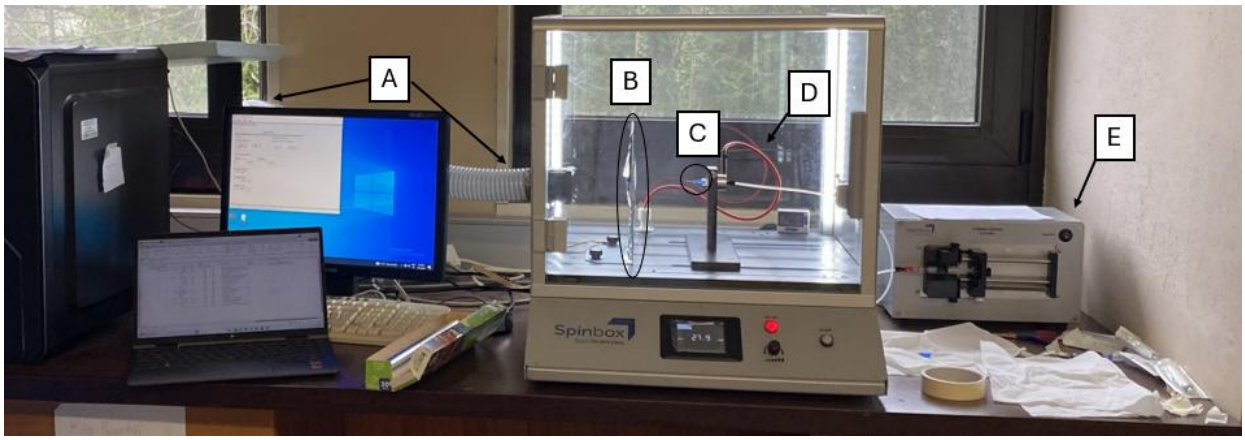

Figure 4 - Initial electrospinin set-up; A) exhaust; B) collector covered in tin foil; C) Spinneret; D) Cables connecting voltage to spinneret; E) Pump with syringe

[1] Hottes do not serve to control temperature or humidity, however, its use allowed the system to be put on an enclosed room, were the temperature and humidity could be better controlled.

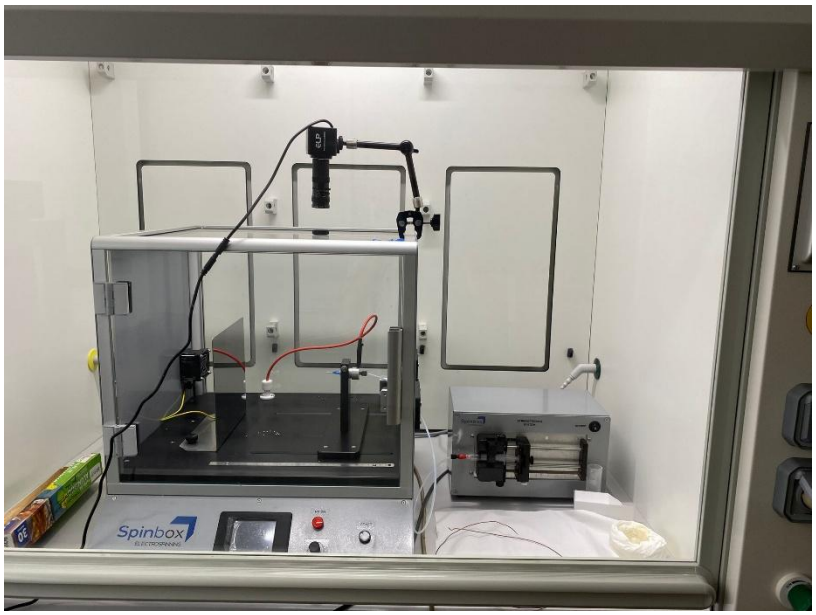

Figure 5 - Electrospinning set-up in the hotte, camera also added

As stated in previous chapter, our objective is to create a layer of overlapping, homogenous, nanofibers. As such, we need our jet to reach the far field. If it breaks during in the near field, we would be doing electro spraying, and would be unable to produce the desired fibres. Additionally, we also need to watch out for beads in the fibres.

To this end, it is very helpful to see how the liquid evolves as it leaves the needle. Such is possible with the naked eye, though it can be difficult and tiring. A better alternative in the use of a camera as can be seen in figure 8. Mounted outside the device to avoid dirtiness, its high amplification is of great help. Its high amplification and mounting position also make it rather difficult to point at the right location. Small movements led to big changes, and wrong distances led to the image being unfocused. Still, with a bit of perseverance one gets there.

2.2. Spinning

Spin coating is a technique utilized to manufacture thin, uniform films in the range of nm to μm. It consists in covering a substrate with a certain solution and then rotating said substrate to level the solution. It is used in microfabrication as, per example, a precursor to photolithography in the manufacturing of microprocessors.

This technique can be divided into four steps: deposition, spin up, spin off and evaporation.[12]

Deposition is the part where we drop the solution into the substrate. This can be done with the substrate spinning (dynamic spin coating), or with it still (static spin coating). In either case, the solution should be dropped in the centre, as the rotation will move it to the edges.

Spin up is the moment where the rotation speeds up to the desired value. The speed up process can be immediate, or it can be incremental. The fluid will eventually match the speed of the substrate due to drag forces.

Spin off occurs as the velocity of the liquid and the substrate begin to match. On this step, the liquid will level, with a portion of it being expelled, as the viscosity, surface tension and centrifugal force try to achieve balance.

Evaporation is the main effect to take hold once the spin off has ended. As the name implies, it is due to the evaporation of the solvent, thus being affected by the solvent volatility, vapour pressure, and ambient conditions.

From the above description, we can conclude that the process is rather simple, while having several nuances. The most obvious ones will probably be the rotation speed and duration. The latter will mainly depend on the duration of the evaporation phase, which, due to the small quantities in question, can be as fast as thirty seconds for water or acetone. Other solvents may require more time

The rotational speed plays a major role in determining the thickness of the substrate, where a rough relation of proportionality can be stabilised:

$h = \frac{1}{\sqrt{w}}$, where h is the thickness (nm) and w the rotational speed (rpm).

More complete models have been such as the Meyerhofer model, that supports this proportionality, however, they still make assumptions, meaning the true results stray away from it.

The substrate is usually stuck in place by vacuum, which may result in deformities if said substrate is too weak. In my experiments, the substrate was too small for the spinning apparatus, meaning I had to stick it on top of a wafer with duct tape. This process resulted in some wasted space, especially for higher speeds.

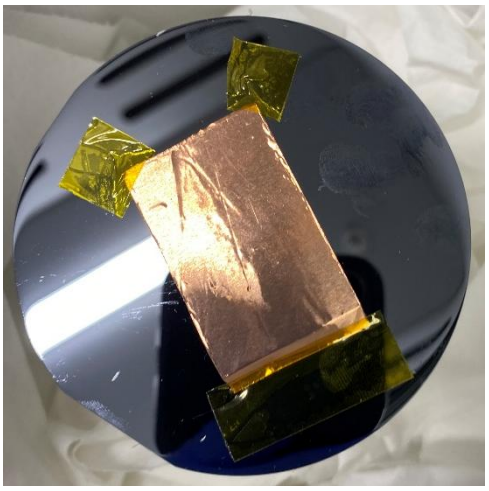

Figure 6 - Substrate duct taped to a wafer on the spinner.

It is important that the substrate is clean, as any debris can cause holes in the film or create comet tails. Deformities may also occur if the solvent is too volatile, or not volatile enough.

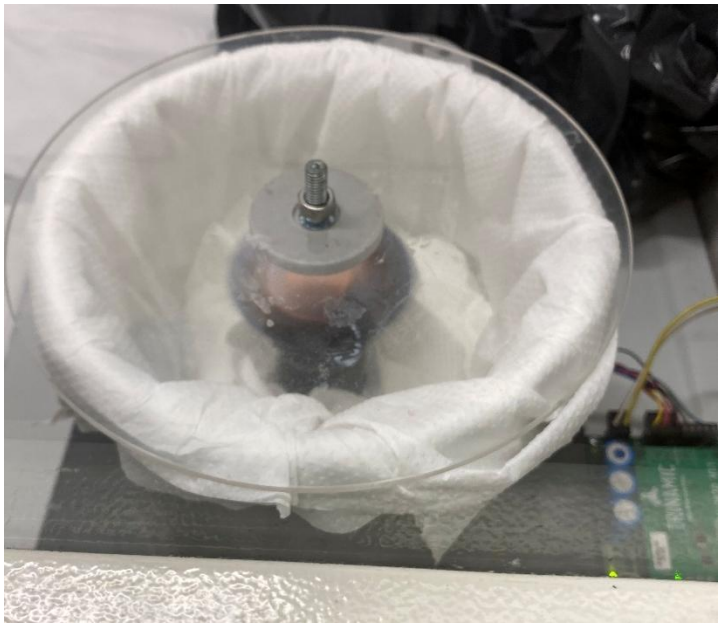

Figure 7 - Spinner set-up mid rotation

Lastly, there is benefited to adding extra layers of different material, but, for this study, a single layer was considered enough.

2.3. Triboelectric pneumatic system

As described in the previous chapter, testing the TENGs requires repeated and uniform contact, which is difficult to achieve manually. This was accomplished using a triboelectric pneumatic system, operating in constant cycles to ensure consistent contact. The applied force was not directly measured or controlled; however, the system completed approximately 0.83 cycles per second, estimated empirically from the number of cycles observed during testing.

To measure the signal generated by the TENG, an electrometer (Keithley 6517B) and a 1 GΩ resistor are used. Each sample is connected to one end of the resistor, while the electrometer is connected across it. The electrometer is linked to a computer running a LabVIEW program, which records and stores the signals in 30-second cycles with 5-second intervals between them.

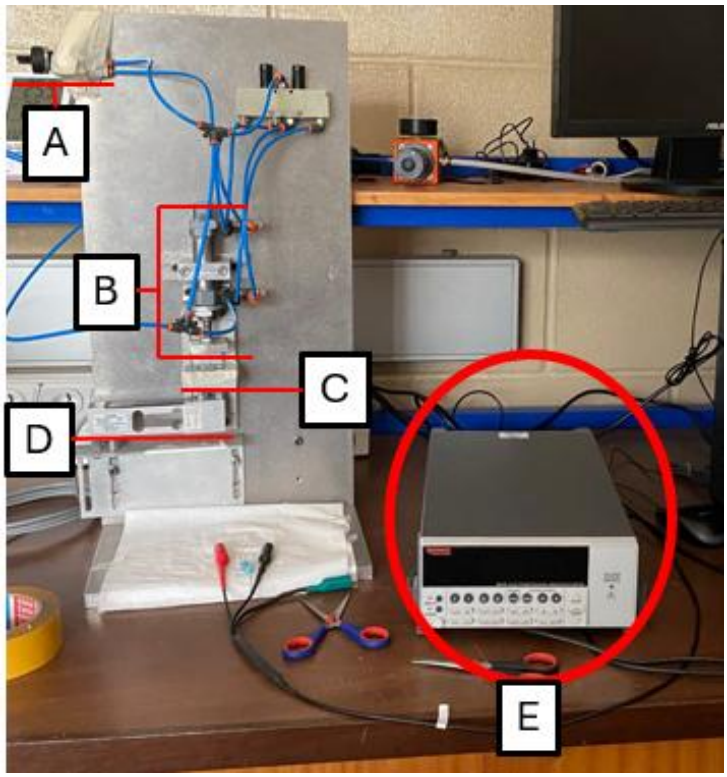

Figure 8 - TENG testing pneumatic set-up; A - on/off button (taped in place), B - piston, C - base, D - load cell, E - electrometer

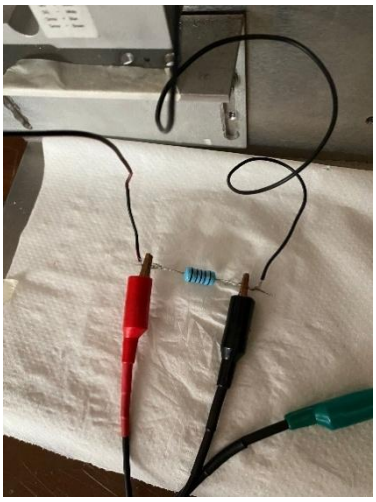

Figure 9 - Example of the connection between samples and electrometer

2.3.1. Pneumatic device

The homemade pneumatic device is, as the name implies, an air powered piston, made by the university. It works well enough, it goes up and down in constant cycles, and since the piston always goes down the same length, the force should be the same.

The ”should” in the first paragraph is very hopeful, as prolong use of the device would lead to audible leaks. While on the topic of force, said force could be changed by moving the base up or down, or by altering the frequency of the piston. Neither of these two options were utilized because I had no way of knowing the force, thus changing the force would make any results impossible to compare with the precious ones.

The base, however, is connected to a load cell connected to an amplifier, so I should have been able to measure the force. And I did, with the help of an Arduino and an online tutorial. As I did some calibrations, I notice that there always appeared to be a relevant error factor. I noticed it was, in part, due to the load cell scraping something.

After removing the load cell, I saw a screw sticking out at an odd angle from its hole. I removed it noticing it didn’t connect to anything, as the hole was not deep enough to connect with another peace. When I reconnect everything again, the load cell no longer worked.

I tried many things in the following days, including putting the screw back in, but nothing worked. Thus, I remained unable to change the force, or frequency. The former was of 0.83 cycles per second, taken empirically as I saw 25 cycles in 30 seconds. The latter remained unknown.

2.4. Fabrication of nylon samples

To investigate its performance in TENGs, three different fabrication methods were used to prepare nylon samples: commercial, spinning and electrospinning. Only one layer was used for spinning.

2.4.1. Fabrication

The nylon solution was prepared made by dissolving Nylon 6.6 powder (5-50 micron, GoodFellow) in adding formic acid (≥96%, Sigma-Aldrich) and stirring for 3 hours in a 60ºC in oil bath. This method was chosen as it was already used in a previous work. The ratio between nylon and acid was a variable to optimize the solution, having been tested the ratios of 5%wt, 10%wt, 15%wt, 20%wt. The solution was then electrospun to form a nanofibers film, with voltage and needle-to-substrate distance as variables.

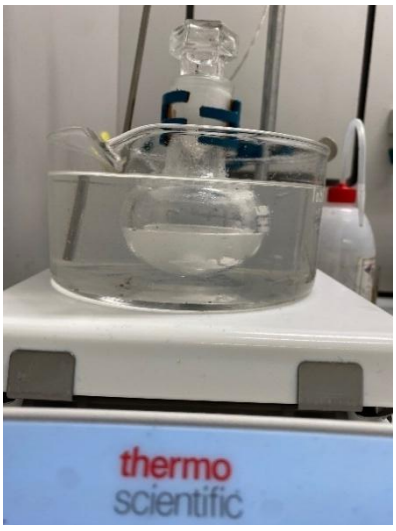

Figure 10 - Nylon solution while in the bath

The same nylon solution was used for spinning, allowing direct comparison with electrospinning. Nylon films were produced at different rotational velocity and concentrations.

Commercial Nylon 6 films (0.060 mm thick, GoodFellow) were also used. Glass substrates were prepared with conductive copper tape, on top of which the commercial nylon films were placed. The nylon was cut leaving extra area around the copper to ensure that the electrode contacted only the intended layer and to prevent unwanted charge transfer.

For all fabrication methods, a cable was soldered to the copper layer. In the electrospun and thin films, soldering was performed after adding the nylon layer, while in the commercial films, it was done before assembly

2.4.2. Results

This solution had already been used, so I had some idea of what the ideal parameter would be. Still, the plan was to investigate the influences of the different voltages and distances, so I would need to test the parameters beyond the ideal.

Unfortunately, this was quickly proven to be impossible. The first evidence was the necessity of the voltage to be much higher than expected to reach the desired results, i.e, instead of requiring 10kV it would require 30kV. I then noticed electric arcs going from the syringe to the pump, as well as an inconsistency in the voltage of the machine.

Figure 11 - Examples of arcing.

I believed this problem was caused by an incomplete isolation of the pump combined with the high conductivity of the nylon solution. This lack of insulation caused the current to not accumulate at the needle, but instead to flow through the tube, and then arc to the pump.

Several attempts were made to solve this issue, mainly the covering of the syringe in plastic and cardboard so as to isolate it. The attempted isolation saw limited success,

making any actual experimentation unreliable at best, and dangerous at worst. A different pump was also tried without success.

The failures occurred while the machine was in its first placement, but they persisted when it was moved to the hotte. A talk with the manufacturer lead to the belief that the liquid was simply too conductive to be electrospun, at least by that machine.

However, this solution was used before in that same machine without issue, and the conductivity seemed a problem in spite of the concentration as solutions of 5, 10, 15 and 20 %wt where tested. Perhaps time or mishandling caused the machine to become more sensitive, causing this solution to pass beyond the threshold of conductivity. Nylon has an increased conductivity due to its amide bonds

The spinning method had better results, in the sense that I was able to produce samples for different rpm's and concentrations, as can be seen in figure 12. However, they would end up not being used due to a change in approach.

Nevertheless, we can still see that the thickness increases with an increase in concentration a decrease in rpm, as was to be expected. In these high concentration, low speed condition, the borders also become more visible, as there are accumulations on those spots. Additionally, at lower concentrations, spots can be detected at the surface of the samples, most likely due to debris left on them.

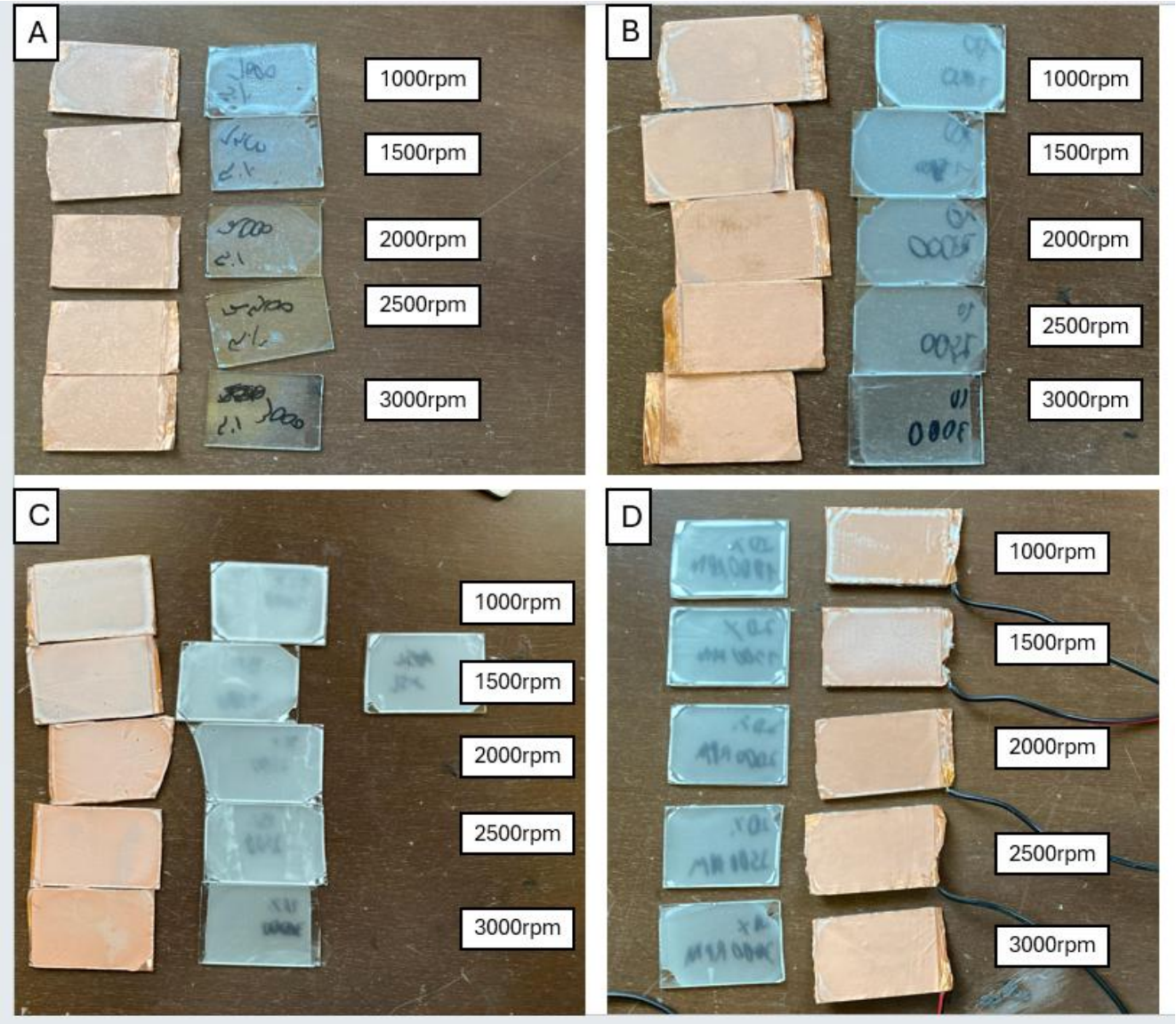

Figure 12 - The results from the spinning of nylon at different concentrations and rpm. A) 5%wt ; B) 10%wt ; C) 15%wt ; D) 20%wt

The commercial films worked, and the extra area provided proved to be a relevant addition, as one experiment lacked it causing the results to be unusable.

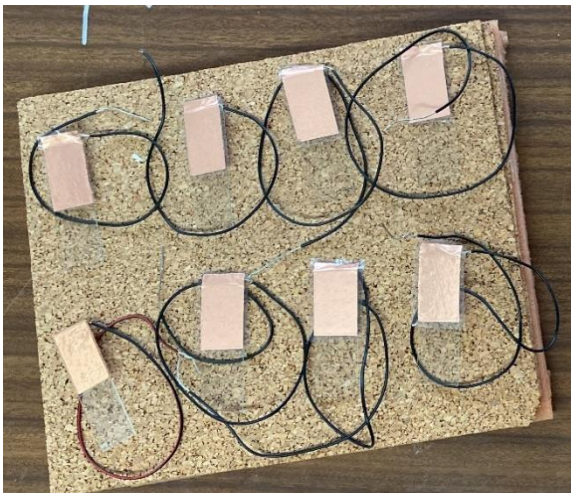

Figure 13 – Examples of samples using commercial nylon film

2.5. Fabrication of PTFE samples

In this thesis, PTFE samples were prepared exclusively from commercial films.

2.5.1. Fabrication

The preparation of PTFE samples followed the same procedure used for commercial nylon films. Glass substrates were prepared with a layer of conductive copper tape acting as the electrode and commercial PTFE films (0.05 mm thick, GoodFellow) were then placed on top of the adhesive side. Extra material around the copper was left to prevent unwanted electrical contact.

2.5.2. Results

The opaque nature of PTFE allowed better visualization of the sample, revealing the presence of bubbles. Avoiding these defects proved difficult, as the material adhered strongly to the copper, and applying excessive force risked rupturing or altering the film

structure. These defects may cause irregularities in the signals, leading to fluctuating max voltages.

Figure 14 - Examples of samples using commercial PTFE film

2.6. Fabrication of PVDF samples

The PVDF samples were prepared using electrospinning and spinning, no commercials films were used. Only one layer was used for spinning.

2.6.1. Fabrication

The solution for PVDF was made by first measuring a certain amount of thin PVDF powder (Sigma-Aldrich), then adding DMF (≥99.5, Fisher Scientific), then pure acetone (Valente & Ribeiro). The resulting mixture was then put in a 60ºC bath and stirred until homogeneity.

The ratio of DMT to acetone was 6:4, and the concentration used was 10%wt.

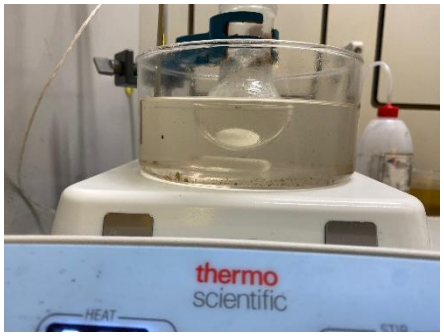

Figure 15 - PVDF solution in the bath

To determine the best parameters for electrospinning I went through various positions, namely 10 cm, 15 cm and 20 cm, and in each I varied the voltage from 0 kV to 30 kV so as to see the behaviour of the fluid at the tip. I did this process for three different pumping rates, 0.1 ml/h, 0.2 ml/h and 0.05 ml/h. For each sample the machine was kept working for 20 minutes.

Ambient conditions such as temperature and humidity were also recorded, as they influence the results.

In terms of spinning, several rotational speeds were used, 500 rpm, 1000 rpm, 1500 rpm, 2000 rpm, 2500 rpm, with which a sample of cooper and one of glass were made. Due to the way the samples had to be stuck to the wafer during spinning, a portion of the sample was left without a PVDF layer. This would leave the copper exposed, resulting in sort circuits. To remedy the situation, the segment was cut off, leaving only non-conductive glass.

2.6.2. Results

In general, the behaviour of the electrospinning process could be divided into three stages, one where the voltage was too weak to pull the fluid, at most pulling droplets; a second where the voltage could pull the fluid, but the flow was unstable; a third where the field was so strong it pulled the fluid to fast, making strings impossible.

The ideal parameters would be somewhere in the second stage, where the fluid could be pulled at a steady, continuous rate.

Several samples were made, most of which were too thin to be used for triboelectric testing. To remedy this, the pump rate was increased to 0.5ml/h to get one sample. As can be seen in figure 17 the PVDF did not stick well to the copper substrate, and this

seemed to worsen with an increased pump rate, i.e. an increased thickness. The last sample would be used for triboelectric testing.

Figure 16 - Results of electrospinning PDVF

In figure 17 we can also see that the increase in distance leads to an increase in dispersion of the fibres in the substrate, such can be seen by noticing that the white circle (fibres) is more clear (larger amount of fibres), and has a shorter radius. This may be because the increase in distance allows the cone to grow more before landing.

Higher voltage decreases the dispersion, making the sample thicker. This led to the curious case of 10 cm 15 kV, where the cone had so little time to grow, it ended up concentrating on two spots

The sample made from the 0.5 ml/h fibres can be seen in figure 18. It shows clear irregularities in the surface, which could not be removed without risking destroying the layer. These irregularities will likely lead to a weaker layer and a more unstable signal.

Figure 17 - A sample created using electrospun PVDF

A summary of the electrospinning results can be seen in table 1.

Table 1 - Summarized conclusions of spinning PVDF

Temperature	Humidity	Flow Rate	Tip to sbstrate Distance	Voltage	Observation
ºC	%	ml/h	cm	kV	all samples took 20 minutes
		0,05			
28	36		10	7--30	too strong, string not visible, droplets not visible
28	36			4--7	string visible, potencially stable
28	36			0-4	too weak to pull fluid, pulls big droplets at most
28	36		15	7--30	too strong, string not visible, droplets not visible
28	36			5--7	string visible, potencially stable
28	36			0-5	too weak to pull fluid, pulls big droplets at most
28	36		20	10--30	too strong, string not visible, droplets not visible
28	36			7--10	string visible, potencially stable
28	36			0-7	too weak to pull fluid, pulls big droplets at most
		0,1			
28	36		10	8--30	too strong, string not visible, droplets not visible
28	36			15	aluminum sample and video taken
28	36			5--8	string visible, potencially stable
27	52			7	aluminum sample and video taken
28	36			0-5	too weak to pull fluid, pulls big droplets at most
27	54			4	video taken
28	36		15	9--30	too strong, string not visible, droplets not visible
28	48			15	aluminum sample and video taken
28	36			6--9	string visible, potencially stable
28	48			7	aluminum sample and video taken
28	36			0-6	too weak to pull fluid, pulls big droplets at most
28	48			4	video taken
31	35			7,7	cooper and aluminum sample taken, too thin
28	36		20	9--30	too strong, string not visible, droplets not visible
28	48			15	aluminum sample and video taken
28	36			6--9	string visible, potencially stable
28	48			7	aluminum sample and video taken
28	36			0-6	too weak to pull fluid, pulls big droplets at most
28	48			4	video taken
		0.2			
28	36		10	9--30	too strong, string not visible, droplets not visible
28	36			5--9	string visible, potencially stable
28	36			0-5	too weak to pull fluid, pulls big droplets at most
28	36		15	9--30	too strong, string not visible, droplets not visible
28	36			5--9	string visible, potencially stable
28	36			0-5	too weak to pull fluid, pulls big droplets at most
34	35			8,9	cooper and aluminum sample taken, thin, slight peeling
28	36		20	10--30	too strong, string not visible, droplets not visible
28	36			6--10	string visible, potencially stable
28	36			0-6	too weak to pull fluid, pulls big droplets at most
34	34	0.5	15	11	cooper and aluminum sample taken, peeling

For the spinning, we can see in figure 19 that the increase in rpm leads to a diminishing thickness of the PVDF layer, as was to be expected. The lower thickness is indicated by an increase in the copper coloration, with less PVDF, copper is more visible.

Additionally, PVDF seems to have had more difficulty sticking to glass than to copper, though, much like in the electrospinning, a higher thickness seems to lead to a lower stickiness, as can be seen by the light peeling for 500rpm.

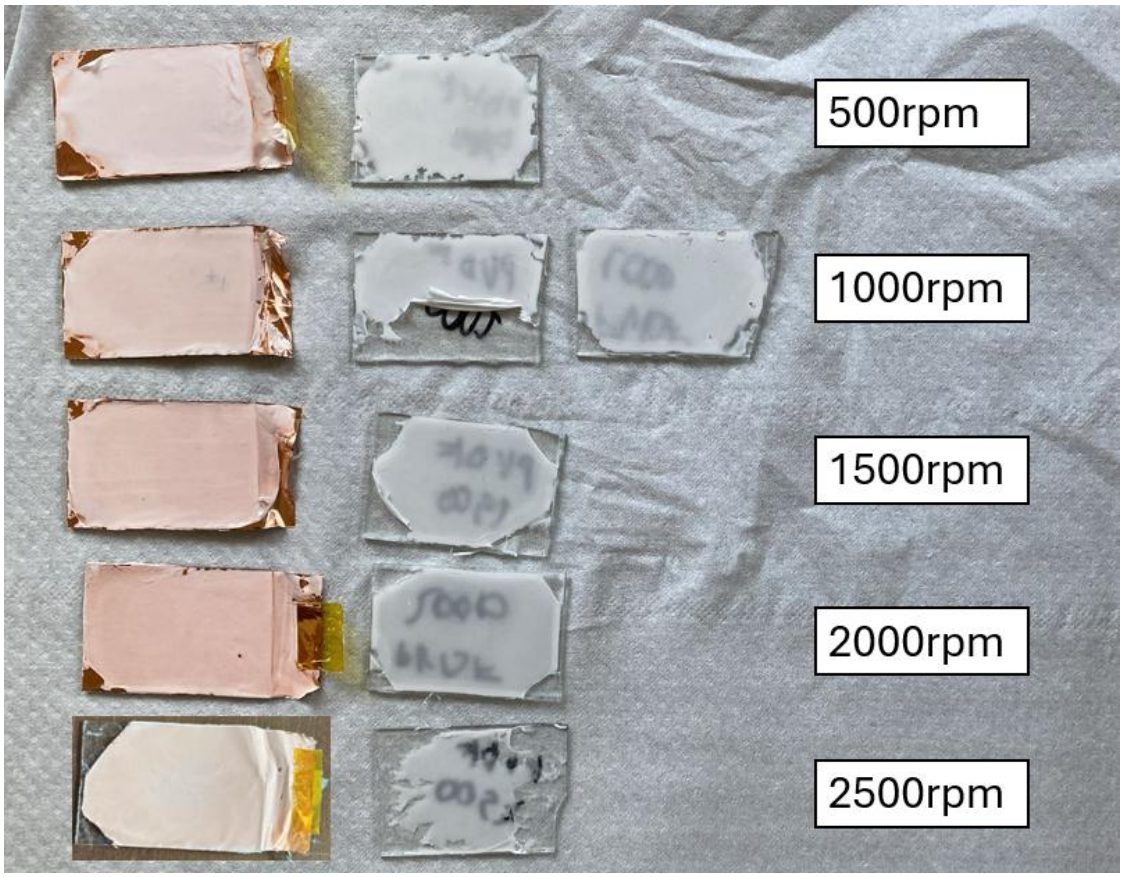

Figure 18 - Results of spinning PVDF. 2500rpm's photo taken on different day.

2.7. Fabrication of PDMS samples

Only spinning was used for the creation of samples, with the solution using Sylgard 184 Silicone Elastomer Kit. Only one layer was used for spinning.

2.7.1. Fabrication

The solution was prepared by first combining the elastomer and the curing agent in a 10:1 ratio. The solution was then hand stirred for around 2 minutes; this created a high amount of bubbles. Then it went into a desiccator for 1 hour.

Spinning of PDMS was only done for 1500 rpm. After the spinning, the samples were left in the hotte for 30 minutes, before being put in the oven for 35 minutes at 100ºC. The solution was very viscous.

Same as in PVDF, a portion of the sample was left without a PDMS layer. This would leave the copper exposed, resulting in sort circuits. To remedy the situation, the segment was cut off, leaving only non-conductive glass.

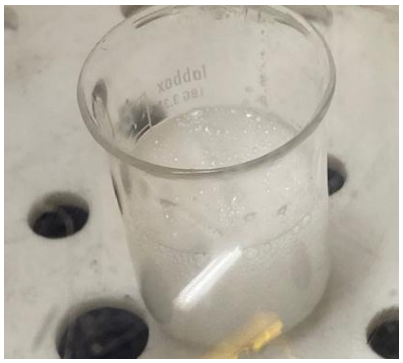

Figure 19 - Solution in the desiccator

2.7.2. Results

Unlike previous entries, the results of spinning do not allow for any sort of comparisons to be made, as only one rpm was used. Still, we can see a stark difference to the other results from spinning: PDMS is transparent.

The PDMS layer is also thicker, as will be proven later, and it seemed to stick to both glass and copper much more easily.

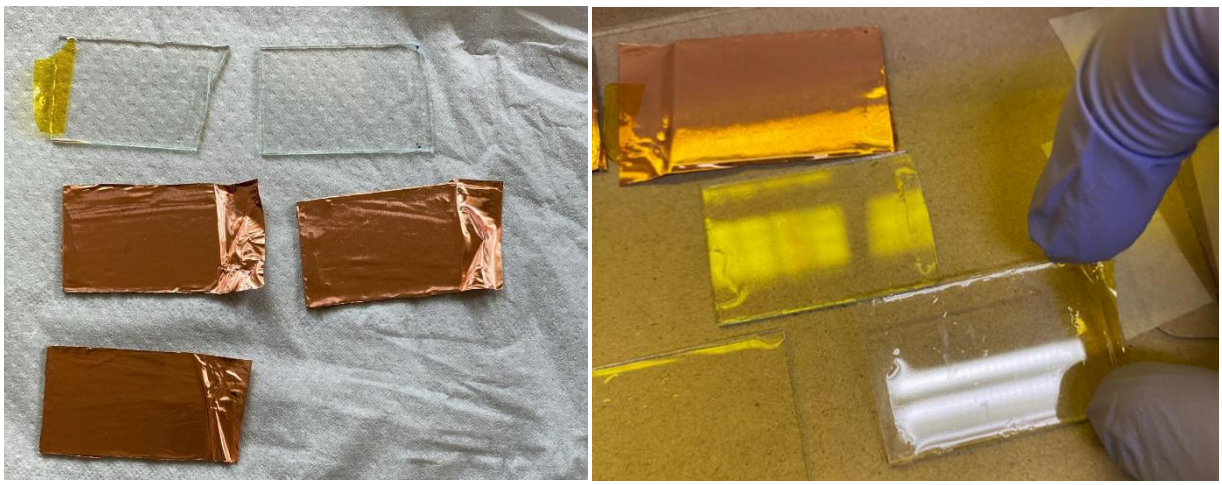

Figure 20 - Results of spinning PDMS

3. Procedures and Results

This chapter will explain the experimental procedures and analyse their results, so as to take real conclusions on the effect of temperature and humidity on the voltage output.

3.1. Temperature Variation with Nylon and PTFE

3.1.1. Procedure

In these experiments a pair of samples, one with a nylon layer, another with an PTFE layer, were repeatedly pressed together and separated at a constant frequency and force for 450 full measurements, on the pneumatic device. After this initial stage, the samples were stored until the next day, when they were put in the oven for a set temperature for 30 minutes and subsequently tested again. Both samples came from commercial films, due to the large number of experiments and the necessity of them to be uniform.

The large number of measurements in the first part of the process was to saturate the sample. In other words, reach the point where the maximum voltage of the sample stabilized. Each full measurements lasted about 30 seconds with around 5 seconds between each one, resulting in 4 to 4.5 hours, corresponding to 11205 to 13072 contact-separation movements. (Frequency of 0.83 Hz)

Fewer cycles were performed in the second stage, as the main objective was to evaluate the reduction of maximum voltage, however, a large number of cycles were still taken to check if the behaviour of samples had significantly changed.

For each temperature, a new pair of samples was used, to ensure the temperature was the only affecting factor. For some temperatures, two tests were run on different days and with new samples. In the case of 25ºC, the samples did not go int the oven, and, in the first test, were kept in the set-up, in other words, not moved.

3.1.2. Voltage evolution over time

Two examples of the behaviour seen in these tests can be seen in figures 21 and 22.

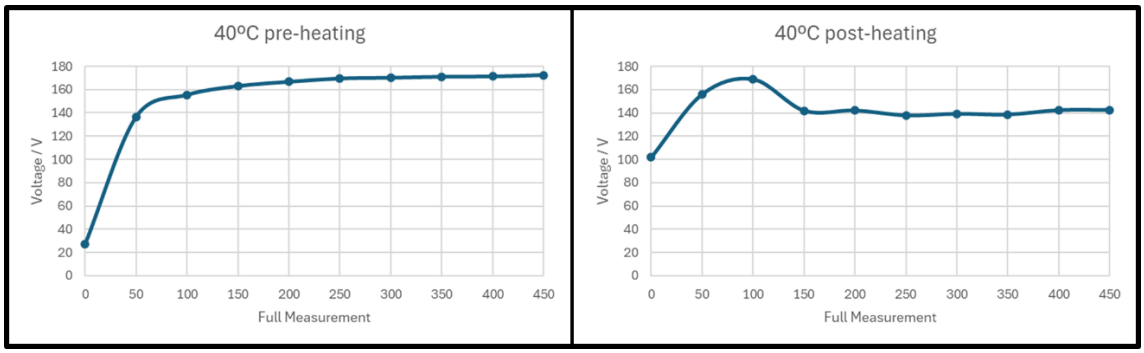

Figure 21 - Graphs showing the evolution of maximum voltage throughout the full measurements for 40ºC. On the left is the first day (pre heating) and on the right is the second day (post heating). We can see that the substrate broke somewhere between the 100 and 150 full measurements on the second day.

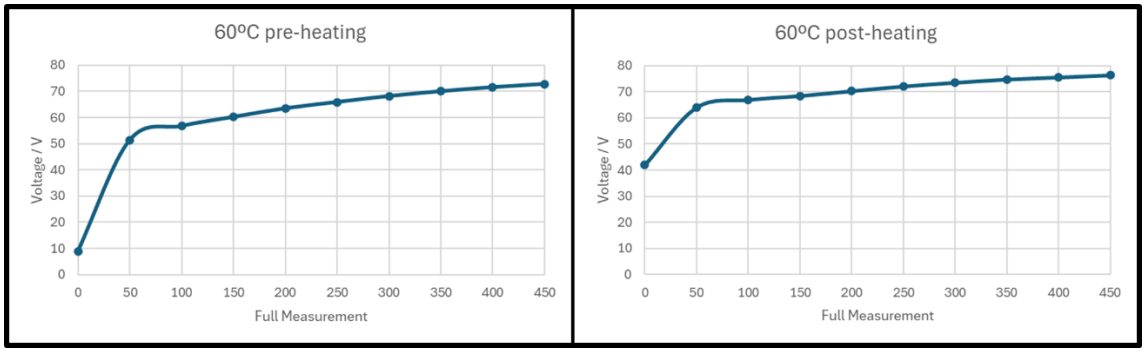

Figure 22 - Graphs showing the evolution of maximum voltage throughout the full measurements for 60ºC. On then left is the first day (pre heating) and on the right is the second day (post heating).

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An initial observation from the voltage measurements obtained by pressing two samples together is the second cycle generates a higher voltage than the first. In other words, each cycle of pressing and releasing increases the voltage from the samples.

This behaviour can be attributed to two main factors. The first is due to charge migration between the two samples: the more the samples connect, the more opportunities the charges will have to migrate from one to the other, the more migrate, the higher the resulting voltage.

The second, rather general, reason is alterations in the structure of the sample. As stated previously, the intensity of the effect depends on various factors, one of them being the area of contact. If, per example, the contact stretches the sample, the area will increase and may even expose new areas if the material was not already stretched.

Additionally, there is the possibility of the substrate breaking. This will occur in several experiments, despite my best attempts to avoid it, being visible on Figure 21 (right at 100-150). The immediate consequence seems to be a lowering in the maximum voltage and an increase in variations between maximums. The other consequence is that, since the substrate is glued to the base, removing it without further damaging it becomes very hard. Lastly, during transportation and handling, features make it easier to alter the sample, i.e. stretching and bending, which can further alter the results. These make fracture an undesirable occurrence

Another thing that quickly becomes apparent is the reduction of maximum voltage when the samples spend some time without touching. Said reduction will become apparent in subsequent chapters and has two main causes. First, there is the fact that a charged sample will, as the name implies, be charged and, much like any charged object, will tend to lose this charge.

Since the sample is isolated by the glass, this loss will be a slow diffusion to the air and ground, rather than something like a spark. After being charged, the samples were stored atop a cork (isolating material) base.

In addition to charge drift, many of the physical transformations that occurred to the sample (stretching) may be undone or diminished without the constant reinforcement, further diminishing maximum voltage output.

3.1.3. Charge Loss Ratio

Bearing in mind the idea that the loss of maximum voltage is due to charge drift/diffusion, and that such phenomena are correlated with temperature, it makes sense to test the reduction of maximum voltage in relation to temperature. To do so, the ratio between the second day's first maximum voltage, and the first day's last maximum voltage was calculated and graphed. This percentage means that if after 450 full measurements we have a maximum voltage of A, and the next day the first maximum voltage is B, the percentage will be $100 * \frac{B}{A}$.

A graph of the data collected can be seen in figure 24, while the raw data can be seen in table 2.

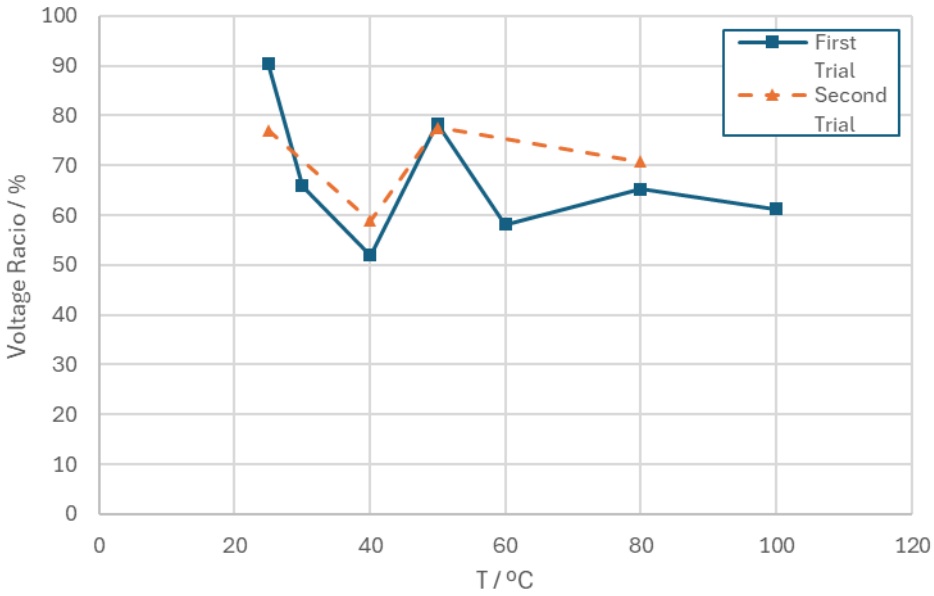

Figure 23 - Graph showing the relation between the last maximum voltage of the first day and the first maximum voltage of the second day, after being heated up at different temperatures. 25ºC was considered ambient temperature.

Table 2 - Data from the Nylon-PTFE temperature experiments, chapter 3.1. Light blue shows the results used in the figure prior. Yellow and green are for the figure after, where green is those who did 450 full measurements on the second day, yellow those who did not (not graphed). Light green is one who broke in the middle of the experiment, making it unreliable

Temperature	1º Day		2º Day		Diference betwen 1ºDay 2ºMax and 2ºDay 1ºMax		
ºC	1º Max	Last Max	1º Max	Last Max	absolute	relative	relative inverted
25	12,267	173,661	157,018	175,552	16,642	9,583	90,417
30	23,402	107,652	70,847	127,419	36,804	34,188	65,812
40	10,686	104,347	54,267	91,165	50,080	47,994	52,006
50	15,817	77,634	60,848	166,635	16,786	21,622	78,378
60	8,869	72,122	41,895	76,000	30,227	41,911	58,089
80	7,863	95,075	61,967	131,764	33,108	34,823	65,177
100	25,140	123,564	75,642	177,181	47,922	38,783	61,217
2º Time							
25	16,760	131,442	101,377	172,910	30,065	22,873	77,127
40	27,412	172,909	101,871	143,561	71,038	41,084	58,916
50	28,707	100,456	77,918	131,669	22,538	22,436	77,564
80	26,789	153,697	108,852	157,567	44,844	29,177	70,823

The first thing we can see is that no value goes above 100%, this makes sense as going above 100% would mean the samples gained charge while in storage. It would be like a battery charging in the drawer. The non-heated results of 25ºC remained hight, this could be due to the PTFE's propensity for creep, as it would make changes more permanent.

The second thing is the difference between the two ambient temperatures (25ºC), which shows the act of moving increases the charge loss of the sample. This may be due to the movements required to remove the duct tape from the sample may result in new stresses to the sample. There is also the possibility that the top layer will touch something, further increasing the rate of charge drifting.

There is a further reduction when the samples are heated to 30ºC, and another for 40ºC. This would seem in accordance with the idea of loss through drift, as higher temperatures would increase this loss.

The 50ºC increased is very curious, only made more so by how close the two trials were. It is too far away to be considered a result of normal variation, and the similarity of the two trials pushes away the possibility of this being the result of an error.

The following temperatures of 60ºC, 80ºC and 100ºC follow neither the diminishing that occurred for 25ºC, 30ºC and 40ºC, nor the increase of 50ºC. They instead fluctuate around 60%.

Ignoring the 50ºC, temperature does seem to have an effect on the samples, however, this does not vary steadily with varying temperature. One possibility is that the effect varies in such a way that it only needs to be heated a little to start, but then it varies very slowly.

A simpler explanation is that the oven itself helps the process. The movement required to transport the samples to the oven could diminishes the number of charges. The dry air may increase the chances of charge diffusion more than the temperature itself. The metal bars the samples are put on may be conductive enough to increase charge migration. Temperature would then only play a minor role.

Locking into the 50ºC spike, it could be due to a phase transition in the PTFE, as it has phases II below 19°C, IV between 19°C and 30°C, I above 30°C and III at higher pressures. When the sample is put in the oven, the 50ºC temperature causes the phase transition which prevents loss of charge, for higher temperatures this prevention could be countered by other factors, resulting in a unique spike at 50ºC.

3.1.4. End Voltage Increase

Locking now at table 2 we can see the values of the first and last maximum voltage of the two parts for every temperature. By looking at the last values of each day, it is possible to see that the last maximum value increases, though the increase does not correlate with temperature, as we can see in figure 24. Note the increase at 50ºC is an outlier in this case, as only one of the experiments displays said increase.

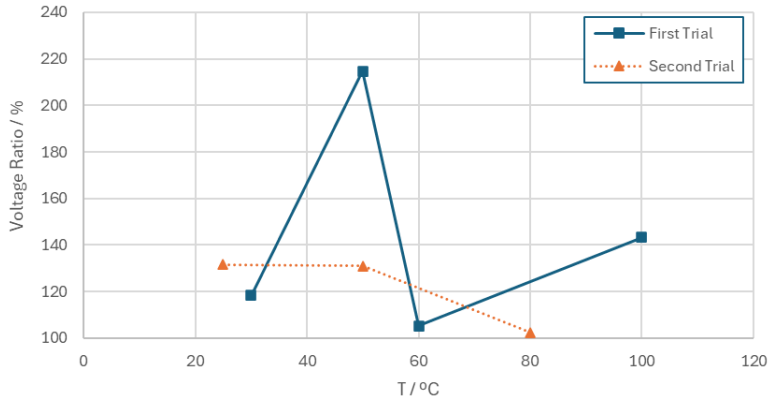

Figure 24 - Relative difference between the maximum voltage of the first and second day. Several points have been omitted due to not having done the same number of measurements the second time.

3.2. Humidity Variation with Nylon and PTFE

3.2.1. Procedure

For this experiment a specialized machine (desiccator) was used that could regulate both humidity and temperature. Since the main objective was to determine the influence of humidity, the temperature was set to 25ºC, as this was a rough approximation of room temperature. I tested the relative humidities of 30%, 50%, 55%, 75%, 90% and 95%.

In one experiment, temperature was set to 50ºC, and the humidity at 65%, since any higher would cause problems due to vapor pressure.

The samples were subjected to 450 full measurements, much like the previous experiment, however, they only rested for about an hour before being put in the specified humidity for 30 minutes. After said exposure, they were immediately pressed again for 150 full measurements. Like in the previous section, both used commercial films.

The second set of testing has less full measurements because its purpose was not to saturate the samples, but to measure the first maximum voltage. The still large number of measurements is to still get an understanding of the voltage's evolution, this way, if the sample had been significantly altered, I wouldn't miss it.

Lastly, ambient humidity was between 50% and 55%, varying from day to day.

3.2.2. Results

The results of the humidity experiments can be seen in figure 26 and the raw data on table 3.

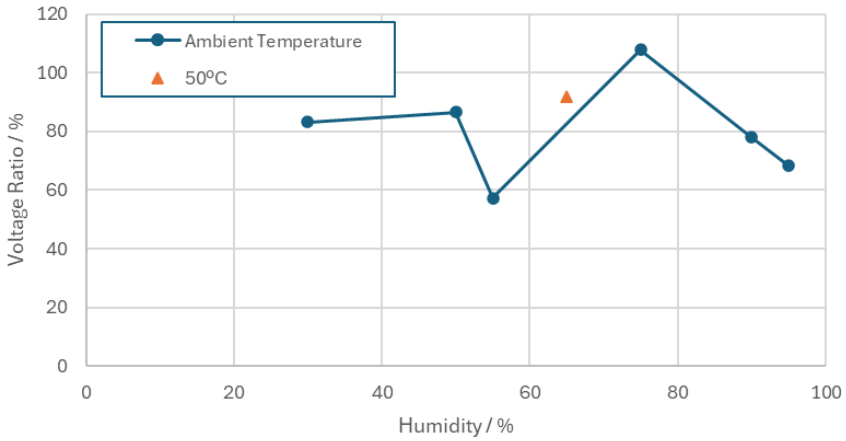

Figure 25 - Graph showing the relation between the last maximum voltage before humidity and the first maximum voltage after humidity. 25ºC and 50% were considered ambient conditions

Table 3 - Data from the Nylon-PTFE humidity experiments, chapter 3.2. Light blue shows the results used in the figure prior. Grey line refers to the data with temperature of 50ºC

Humidity	Pre-Machine		Post-Machine		Diference betwen Pre-M 2ºMax and Post-M 1ºMax		
%	1º Max	Last Max	1º Max	Last Max	Absolute	Relative	Relative Inverted
30	22.293	128.015	106.555	106.790	21.460	16.763	83.236
50	34.819	140.954	122.051	164.030	18.903	13.410	86.589
55	23.508	174.644	99.839	159.022	74.805	42.832	57.167
75	30.931	76.015	81.945	97.000	-5.930	-7.801	107.801
90	36.101	155.553	121.192	181.514	34.361	22.089	77.910
95	24.781	187.122	127.676	168.906	59.446	31.768	68.231
65	5.194	95.567	87.710	101.081	7.856	8.221	91.778

In the graph we can see the percentage keeping at 80%V[2], until a sharp decline in the 55%RH. It starts rising until 75%RH, from which it decreases in the 90%RH and 95%RH.

With an increase in %RH, it was expected that the %V would be reduced, as the water in the air would contribute to the charge drift, and any water that was deposited on the

[2] %RH - % of Humidity; %V – ratio voltage, “relative inverted” portion of the graph

sample would absorb some charge. If we take the 50%RH as the baseline, this reduction was seen only in the very high values of 90%RH and 95%RH. The reduction at 55%RH seems too sudden, though it may be like what happened at 50ºC in the previous chapter.

The 75%RH is especially intriguing as its value implies it gained charge between the two parts, which should be impossible. Either the ambient was charged enough to charge the samples, or the sample was physically altered by the transportation, or by the increase in humidity. During the transportation the samples may have been pressed or stretched, which would increase charge. Thought it is also possible that droplets formed on the surface of the material would charge it through the triboelectric effect.

For the 50ºC experiment, the counterintuitive increase has an explanation. As we saw in the previous chapter, the temperature of 50ºC increases the %V, in relation to its neighbours, the same must have happened here. The smaller difference may be due to smaller the smaller losses that occurred, or some of the increase was balance by the decrease due to humidity.

The inconclusiveness may be due to insufficient time in the humidity.

3.3. Temperature Variation with Nylon and PDVF

3.3.1. Procedure

Since PDVF was both spun and electrospun, I was able to compare the two processes. As a reminder, the electrospun fibres had to be glued to the copper layer (like the commercial films) because they wouldn’t naturally stick to it, and the spun sample had to be trimmed down to prevent short circuits.

Due to lack of time and inability to produce enough samples, the experiment would be limited to only one intense temperature. Similarly with the commercial films, the sample would be subjected to 450 full measurements, at the same frequency, then left in storage overnight, before being heated at 100ºC for 30 minutes and pressed again.

3.3.2. Results using PDVF Fibres

The electrospun film started to tear away due to the repeated forces, forcing the first part of the experiment to be stop at 150 full measurements, a predictable outcome as its weakness was felt while glueing it to the copper layer. The first sign of tearing was the

low voltages the samples was generating, a characteristic also seen when commercial samples were not properly prepared.

A conclusion we can take from this is that the layer has to be thicker in order to resist the forces created by the connection-separation cycles.

The most interesting part of the experiment came when, for mere curiosity/completion, the sample was put in the oven at 100ºC for 30 minutes. After that, the readings improved immensely, even in the first maximum. The first maximum increase is especially confusing as it means the samples gained charges during the time between the two parts, something that should not occur, and did not occur before, except for one sample pair in the humidity chapter.

The lack of short circuit is also intriguing, since no new material was added. A possible explanation may be the fact that the glue was left exposed, and then heated, possibly making it into a non-conductive layer.

Figure 26 – PDVF fibre samples before (left) and after (centre, right) use. Exposed copper highlighted

3.3.3. Results using PDVF Film

The film component of PVDF fared better with its voltage, however, the sample kept rotating, needing to be realigned on a constant basis. During said realignment, it was starting to become obvious that the PVDF layer was being scraped off. This did not seem to have the same effect on voltage that the previous lack of upper layer had, perhaps because the PVDF layer was never completely removed, stopping serious short-circuits.

It is unknown how much the rotation affected the durability of the upper layer, but the added tangential forces certainly made it worst. Since the voltage did not seem to increase, the first part of the experiment was stopped at 362 full measurements.

This layer also seems to need to be thicker.

After heating up the samples, the voltage increased as it had for the fibres, again demonstrating a, supposedly, impossible increase in the charge.

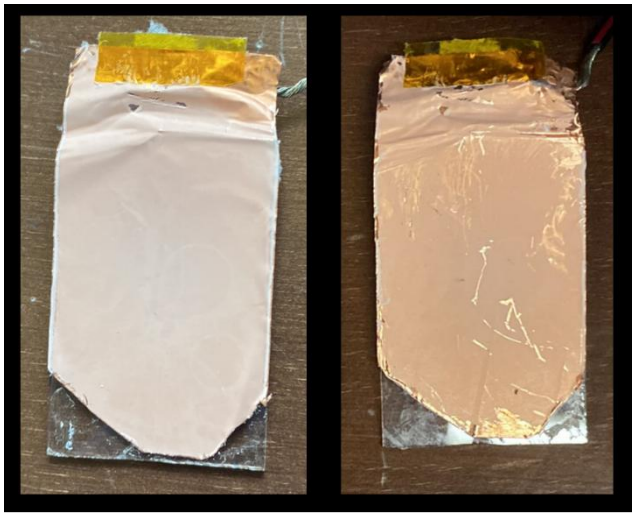

Figure 27 – PDVF film before (left) and after (right) use. Scratches can be seen on the left one

3.3.4. Possible Explanations for the Charge Increase

A possible explanation is that the copper layer oxidised. Heat helps copper oxidise with oxygen, and, unlike in the other experiments, this copper was (partially) exposed while being heated up to 100ºC in an environment with oxygen. Since oxidised copper is not conductive this would explain the lack of short circuit. Furthermore, electrons would be released, during oxidation, which could go on to latch on to the PVDF layer, explaining the increase in charge. Another possibility for the sudden increase in charge is a reorientation of PVDF's dipoles during the heating/cooling, leading to an enhanced piezoelectric effect on the triboelectric layer.

Table 4 - Data from the PVDF experiments

	1º Day		2º Day		Diference betwen 1ºDay 2ºMax and 2ºDay 1ºMax		
	1º Max	Last Max	1º Max	Last Max	Absolute	Relative	Relative Inverted
PVDF Fibras	14.935	23.357	70.705	118.460	-47.348	-202.716	302.716
PVDF Film	17.626	57.366	114.530	132.251	-57.163	-99.647	199.647

3.4. Temperature Variation with Nylon and PDMS

3.4.1. Procedure

For the final pair, I measure PDMS under two different protocols to evaluate its response to temperature variation. The first method was the same procedure used for PTFE, 450 full measurements, then stored overnight, then heated at 100ºC for 30 min and then pressed again. This was done once.

The second protocol aimed to test multiple temperatures within a single day using the same sample pair. Here, 50 full measurements were first performed, followed by a 30-minute oven exposure, and then another 50 measurements. This process was repeated at different temperatures, allowing the evaluation of the entire temperature range while minimizing sample fatigue.

3.4.2. Results

This experiment proved to be more doable than the previous ones since, as stated in its chapter, the spinning of PDMS led to a thicker layer, which translated into a more resistant layer. Even without the risk of short circuits, there was still the problem of the rotation of the sample. Said rotation was more of a problem during the first part since it was longer, but it still came up during the smaller segments. The correction was to remove the sample and readjust it, which alters the results, but it was deemed less problematic than letting the sample rotate.

Another detail was that the reduced size of the spinning samples made me have to put the sample on top of the nylon and then start the machine, whereas before the top sample was simply stuck to the top base. This should not make a big difference but should be pointed out. It was also the case for the spinning PVDF, but it is more relevant here since I am positioning the sample more often.

I will also point out that the ambient temperature went from 25ºC to 30ºC simply due to the weather.

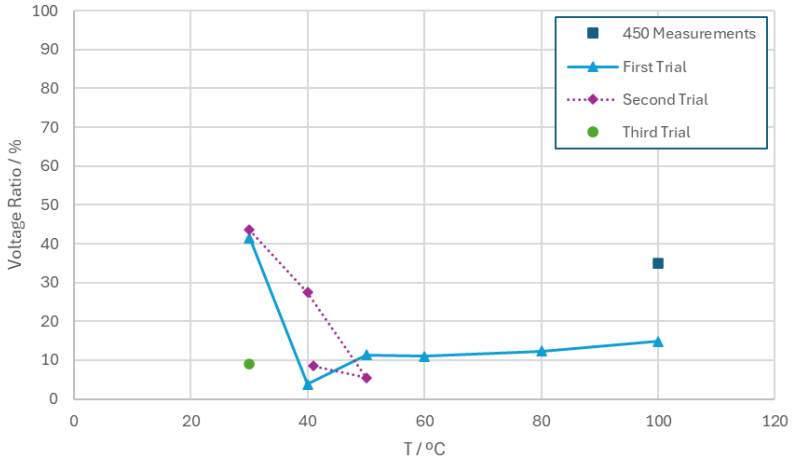

Figure 28 - The relative values of the last voltage pre-heating and the first voltage post-heating. Each shape/colour signifies a set of experiments done on the same day. The dark blue square is the experiment with the 450 full measurements, the rest represent the 50 full measurements tests

The dark blue dot is the experiment with the 450 full measurements, the rest represent the 50 full measurements tests, all done with the same sample, where each colour shows the testes done on the same day. The purple dots show an experiment that went ambient, 40ºC, 50ºC, 40ºC (shown as 41 for distinction). The green dot is the ambient temperature of 30ºC but done in the oven. The blue dot at 40ºC was left in the oven for 1 hour due to an issue, the rest were 30 minutes.

One thing one can immediately tell from figure 28 is how low the values are, even for the first experiment that was the same as the Nylon and PTFE. This could be due to PDMS reacting in a different way to the given stimuli, maybe it is more affected by the temperature or loses charge more quickly or it is because it suffers less from creep than PTFE, making changes less permanent.

Comparing the two 100ºC, we see that the 50 full measurements is smaller than the 100 full measurements, even though the 100 full measurements had a longer time to lose charge. Perhaps the idea that the sample suffers physical changes is true, and those changes don't appear in the 50 full measurements, making it easier for charges to dispel.

However, looking at the purple points, one can see that the 40ºC that followed the 30ºC is much higher than the 40ºC that followed the 50ºC (41ºC on the graph). This gives the idea that previous trips to the oven mater, making the sample have an easier time losing charge.

The green dot seems to show that the oven removes charge, even without an increase in temperature, however, this does not coincide with the data of the purple points and is lower than other points put through higher temperatures, so it is possible something else might have happed. This was the last experiment performed with this sample, so maybe it was suffering some sort of exhaustion after 10 trials.

Considering that the apparent tendency is for the results to lower until 50 or 60ºC, starting from which they will increase, the PDMS shows a different behaviour to PTFE, one that seems to improve with temperature and lacks the sudden rise at 50ºC. The purple points

help disprove the idea that the improvement could be due to previous temperatures, unless the improvement only comes from rising temperatures

4. Conclusion

Several conclusions can be taken from this study. First, is that heating a Teng will lead to a decrease in accumulated charge, but this decrease is does not noticeably change between 40ºC and 100ºC. Second, is that humidity does not decrease charge. Lastly, heating a TENG may oxidize exposed parts of the conductive layer, which may prevent short-circuits.

In more detail, I saw that the solution of nylon and formic acid was very conductive, necessitating a different solution, or special precautions, to do electrospinning with it. Continuing with electrospinning, PDVF seems to have difficulty sticking to copper, a characteristic that becomes more pronounce as the layer becomes thicker. It also needs a lot of time for electrospinning a thick layer, I used 0.5mL/h for 20 minutes and it still was weak.

Moving on to the experiments, from the PTFE and nylon I saw that the temperature had a starting effect of diminishing the maximum voltage of a given sample. This reduction, however, was sudden until the 40ºC, where it stabilized until 100ºC. At 50ºC a spike occurs, believed to be caused by a phase change in the PTFE. Humidity does not seem to affect the samples' maximum voltage.

The PVDF experiment's main takeaway is that heating the sample may be a good way to remove the danger of short circuits due to gaps on the upper layer, while also adding charge to the layer. After heating, the samples showed remarkable improvement in maximum voltage and stability, believed to be cause by the oxidation of the exposed copper, which created a layer of non-conductive oxidised copper and gave electrons to the upper layer.

From the PDMS, we saw a behaviour similar to that of the PTFE, except without the peak at 50ºC, this furthers the belief that the behaviour seen is in fact general. There was also evidence for the maximum voltage values being altered based on the order in which the samples were heated, in which case the previous confusions could be put into question. Still, the values were all lower than with PTFE, which adds to the idea that the values seen are not only due to charge migrations, but also due to physical alterations on the sample.

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