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Faculteit Industriële ingenieurswetenschappen

master in de industriële wetenschappen: verpakkingstechnologie

Masterthesis

Centrifuge fiber-spinning setup for production of micro/nano fibers

PROMOTOR :

Prof. dr. ir. Naveen REDDY

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Prof. dr. ir. Mieke BUNTINX

Willem Meurs

Scriptie ingediend tot het behalen van de graad van master in de industriële wetenschappen: verpakkingstechnologie

Gezamenlijke opleiding UHasselt en KU Leuven

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UHASSELT KU LEUVEN

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My master thesis is the final step of my education process as industrial engineer in packaging technology at the University of Hasselt. I was very lucky to be able to work on a recent technology, for which I designed a centrifuge. It became a design and building process that required all the skills I obtained during the past years as a student.

My main objective was designing and building a highly adjustable centrifuge, capable of spinning polymer nanofibers, with a future proof design allowing ongoing trials in the years to come. I used 3D modeling software to design the centrifuge. Working with this software, was a skill I acquired through self-tuition. Once the build plans were available, Johan Soogen from the workshop at building D on the UHasselt campus was a fantastic partner in the execution of the plans. He helped me out in terms of getting the plans up to an industry standard, while pushing me to keep working on achieving the results present in this research.

Firstly, I would like to thank Prof. dr. ir. Naveen REDDY for introducing this project to me. I am honored that he believed in my capabilities in terms of creating such a machine. Even though I was hesitant initially, I am happy that I accepted the challenge. It gave me the opportunity to learn a lot and to acquire some useful skills that will also help me in the future. I really appreciated the fact that he empowered me to take the lead in the design process and to define the timelines for execution. This approach, together with seeing the finished and operating centrifuge, created for me an increased sense of fulfillment and it boosted my self-confidence.

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GLOSSARY

3D	3 dimensional
Fa	Air friction force
DC	Direct Current
E-CS	Electrostatic assisted centrifugal spinning
g/hr	Grams per hour
KV	Kilovolt
LCD	Liquid Crystal Display
m	mass
mm	Millimeter
PAN	polyacrylonitrile
PMMA	Polymethyl methacrylate
PS	Polystyrene
r	Radius
rpm	Rotations Per Minute
THF	Tetrahydrofuran
V	Volt
ω	Rotation speed
wt%	Weight percentage
°C	Degrees Celsius

ABSTRACT

Fiber spinning using centrifugal forces is one of the more interesting techniques to produce polymer nanofibers. At present, this technique is still underdeveloped. More research is required to fine-tune the morphology of the nanofibers. Polymer nanofibers can be used for a whole range of applications, such as protective clothing, energy storage, drug delivery systems, filtration and tissue engineering. The goal of this research is to develop a stable and highly adjustable centrifuge, which can produce nanofibers from a solution of polystyrene (PS) and tetrahydrofuran (THF).

The emphasis in this research is on designing the centrifuge. This is facilitated using 3D modeling software. Drawings are created and sent to a workshop, where the centrifuge is manufactured using durable materials like aluminum and inox. The centrifuge is designed to be highly adjustable to enable easy operation. The size of the fibers can be adapted using a spinneret with different orifice sizes, measuring 0.3 mm, 0.6 mm and 1.0 mm. Different concentrations of PS in THF are tested ranging from 17.5 to 25 wt%. Other variables allow changing the morphology of the fibers, such as the collection distance and the adjustable rotation speed.

Preliminary testing reflects results from other studies, which indicates that the centrifuge is working properly. Further testing will reveal which variables have the highest impact on the morphology of the fiber. Variables concerned are the collection distance, the orifice size, the rotation speed and the concentration.

ABSTRACT IN DUTCH

Spinnen van vezels door centrifugale krachten is een interessante, maar vooralsnog onderontwikkelde, techniek om nanovezels te creëren uit polymeren. Meer onderzoek is nodig om het productieproces en de morfologie van de nanovezels te verfijnen. Nanovezels kunnen toegepast worden in beschermende kledij, opslag van energie, medische compressen, filtratie- en weefselmateriaal. Het doel van dit onderzoek is het ontwikkelen van een stabiele centrifuge die nanovezels kan produceren uit een oplossing van polystyreen en tetrahydrofuraan.

De focus van deze studie ligt op het ontwerpen van de centrifuge. Dit proces wordt ondersteund door 3D visualisatie software. De ontwerpen worden overgemaakt aan een werkplaats, waar de centrifuge wordt vervaardigd uit duurzame materialen zoals aluminium en inox. De centrifuge moet eenvoudig te bedienen en makkelijk instelbaar zijn. De dikte van de vezels kan aangepast worden door het gebruik van een spinneret met verschillende openingen: 0,3 mm, 0,6 mm en 1,0 mm. Verschillende concentraties gaande van 17,5 wt% tot 25 wt% worden getest. Andere variabelen laten toe om de morfologie van de vezels te veranderen, zoals de collectie-afstand en een aanpasbaar toerental.

De eerste testen bevestigen de resultaten van eerdere studies hetgeen aantoont dat de centrifuge naar behoren functioneert. Verdere testen zullen uitwijzen welke variabelen de morfologie van de vezels het meest beïnvloeden. Het betreft hier parameters zoals de collectie-afstand, het formaat van de opening, het toerental en de concentratie.

1. INTRODUCTION

1.1. Situating

Fiber spinning is a technique to make very thin polymer fibers. The polymer base material can be melted just before it is spun into fibers or it can be dissolved in a strong solvent that evaporates very quickly. The most commonly used techniques to produce nanofibers using solvents are centrifugal spinning and electrospinning. Other techniques are melt-blowing, bicomponent fiber spinning, phase separation, template synthesis and self-assembly [1].

Polymer nanofibers are used in applications such as filtration, tissue engineering, protective clothing, battery separators, energy storage and more. The high surface to volume properties of the nanofibers make them a good candidate for above mentioned applications.

Centrifugal fiber spinning is still less common than electrospinning even though it has some benefits over electrospinning. Centrifugal fiber spinning provides a platform that could easily be scaled up for industrial production. It also creates a safer work environment and allows the machine parts to be easily accessible for adjusting the settings or for maintenance. However, the results are not as good as the electrospinning setup. Because electrospinning allows the production of fibers with a better morphology on a lab scale, it is more widely used. More testing needs to be done to understand and manipulate the physics behind the centrifugal spinning process to generate better result.

1.2. Problem statement

To achieve a sturdier and more suitable setup for centrifugal spinning, the existing fiber production setup needs to be upgraded. The existing setup consists of a lab centrifuge with plastic Eppendorf pipettes with a needle protruding from them. The Eppendorf pipettes are filled with a solution of polystyrene (PS) dissolved in tetrahydrofuran (THF) with concentrations going from 17.5 wt% up to 25 wt%. This centrifuge operates on a fixed speed. When switched on, it rotates at about 6000 rpm. The centrifuge generates a centrifugal force by spinning, hereby creating micro/nanofibers that are collected on Polymethyl methacrylate (PMMA) posts (as shown in Figure 1). The only variable the operator can influence is the concentration of the solution in the Eppendorf pipettes. Other important variables cannot be adjusted, variables like:

- The collection distance: how far or how close the collection posts are positioned versus the spinneret,
- The rotation speed: the lab centrifuge has a set speed and cannot be changed,
- Orifice diameter: the needles connected to the pipettes have a fixed size,
- Collection height: fibers are collected on top of each other.

Figure 1: original centrifugal fiber spinning setup

The original centrifuge has no mechanism to collect fibers next to each other. Instead, fibers are collected on top of each other, making the characterization process harder. Focalizing on every layer of fibers is difficult with an optical microscope because of the way it is built. Additionally, overlaps are undesirable in many applications such as transparent conductive electrodes, sensors etc.

The lack of adjustments in the setup hinder in-depth research of the production and the characterization of micro/nanofibers. A more stable setup with adjustable rotation speed, with an adjustable spinning height and with a vibration-free base is required to make the research more precise and varied.

2. LITERATURE

2.1. What are nanofibers and what can they be used for?

Nanofibers are fibers with a diameter within the nanometer range (<1 µm). In this study we will focus on fabrication of polymer nanofibers. A detailed literature survey concerning applications and production methods of polymer nanofibers is provided below.

Polymer nanofibers can be made from virgin polymers or from plastic waste streams. The latter is particularly interesting as the current global demand of plastics is very high. The use of recycled plastics is growing linked to environmental concerns.
Recycled plastics are usually no match for their virgin material counterpart as high processing temperatures, shearing, aging and heterogeneity can be detrimental to the quality and mechanical properties of the recycled polymer. Other factors that also contribute to the mediocre quality are contaminants like paper, adhesives and additives and all the various grades of plastics that are collected at the same time. For recycled polymers, a combination of different grades of plastics is less desirable. Reprocessed polymers may experience viscosity changes from previous degradation processes like branching, crosslinking, crystallization behavior and chain scission [1]. Those disadvantages of producing recycled plastics should not be a large drawback when producing nanofibers for applications like, for instance, insulation. The different grades of plastics that are processed at the same time, may lead to interesting findings and properties of the nanofiber composites.

Nanofibers are particularly interesting to research because of their unique properties like high surface area-to volume ratio, high porosity, mechanical flexibility, directional strength and fully interconnected pore network [2], [3].

These characteristics make nanofibers a good building block in many applications. Examples are: medical use, electronics, filtration, insulation, textiles and much more. The most important applications of nanofibers will be discussed in the next section.

2.1.1 Biomedical tissue engineering

Nanofibers can be highly aligned, offering a good mechanical strength. They are highly porous and can be produced in a simple and cost-effective manner. Because of these properties, nanofibers can be used in the medical world, offering a means of drug delivery or aiding in the healing process of wounds. The polymer, used to make these nanofibers, needs to have a low chemical toxicity, a good complexion, good adhesion properties, an excellent physiological compatibility and a reasonable solubility to dissolve the polymer. Both the wound dressing and drug delivery system can be combined in the same patch of nanofibers. It replaces the traditional gauze used in medical applications [2].

2.1.1.1 Wound Dressing

A wound dressing helps to heal a wound by promoting cell growth, providing a barrier against micro-organisms and slowly releasing drugs into the wound to speed up the healing process. The nanofibers can also function as a tissue engineering scaffold. Aligned fibers will mimic the architecture of nerves, muscles and tendons. Random arrangements can mimic extracellular matrices, speeding up the healing process of very difficult to heal injuries. Certain criteria are associated with the ideal wound dressing. It must be porous enough, so bodily fluids can migrate through the microstructure of the nanofibers, which will promote and support initial cell adhesion.

The polymer used should also be hydrophilic to promote the biodegradability of the polymer itself. Initially it has to provide relevant mechanical strength for the cells to get attached and then it has to slowly degrade so the cells would stay put and carry out the rest of the healing process. This process is also known as in vivo tissue engineering [2], [4].

2.1.1.2 Drug delivery system

Nanofibers can act as a drug delivery system. Drugs can be added during the production of the nanofibers. In a second stage, these will be formed into pads, which can be used for the treatment of wounds. As the pads break down (as mentioned in section 2.1.1.1), the drugs can be slowly released into the wound over time. This eliminates the risk of an initial release of too much drugs. This method can be used for different kinds of drugs. Firstly, it can be used to release drugs that would aid the healing process. Secondly, the fiber mat could house anti-microbial substances that would keep the wound pathogen-free and make it heal faster [2]. Lastly, the fiber mat could house analgesic and inflammatory agents to give a soothing effect to open wounds, burns and skin abrasions [5].

2.1.2 Textiles

Nanofibers can be used to produce textiles using both virgin and recycled material.
Recycled fibers might have a mediocre quality, but they can still be used as insulation, wipes, bandages and other disposable and/or biodegradable fabrics. The small pores will trap air ensuring good insulation [1], which can be of use in the construction or in the clothing industry.
As mentioned, nanofibers are an optimal candidate for wipes and other absorbent textiles because of the capillary effect of the water, which draws itself into the pores between the nanofibers.

2.1.3 Filtration media

The machine, producing nanofibers, can be set to produce fibers of different diameters. The smaller the diameter of the fibers are, the closer the nanofibers could be in relation to each other. The tight network of fibers can form a good basis for a filtration medium. Like fiberglass, which is commonly used as a filter medium, nanofibers can be used in the same way. Smaller nanofibers tend to have smaller pores between them, making these optimal for filtering out fine particulate or even micro-organisms. Removing the micro-organisms from water can make it safe to drink.

Nanofibers that are produced on lab-scale are often loosely packed, making the pores between the fibers larger. Post production treatments can help to reach optimal properties. The post-treatments are meant to pack the fibers more closely and can be done by hot pressing or calendaring to make the nanofiber mat fit for the desired application [6].

2.1.4 Energy storage – Battery separators

A study at the North Carolina State University stated that heat treating centrifugally spun nanofibers could make them conductive and an optimal candidate for energy storage. They heat treated polyacrylonitrile (PAN)/PMMA nanofibers at 800 °C in an argon gas environment. The argon gas was used to flush out oxygen, so the nanofibers would only degrade thermally and not by oxidation. The PAN was converted into carbon at 800 °C and the PMMA acted as a pore generator. The result was a highly porous conductive nanofiber with a large surface area. This means it can be used as electrode material in supercapacitors [6].

Nanofibers could also be used for battery separators in high performance batteries. A battery separator is a critical component in a lithium-ion rechargeable battery. It prevents contact between the positive and negative poles of the battery. A short circuit within the battery is therefore avoided

but the transport of ions is still enabled. With the evolution of electric cars, the demand for high performance batteries is rising. Nanofibers would be an optimal candidate for use in higher-performance lithium-ion rechargeable batteries because they require a good electrolyte uptake capacity and enhanced separator-electrode adhesion. The pores of a nanofiber mat would greatly improve the battery separators that are currently on the market because of their good electrolyte uptake through capillary forces [7].

2.2. How are nanofibers made?

Polymer nanofibers can be made using different techniques. Often, the first step in making nanofibers is to fluidize the polymer so it can be processed into nanofibers. The two most common ways of fluidizing the polymer are to melt the polymer or to dissolve the polymer in a strong and appropriate solvent.

If melting is the chosen way of fluidizing the polymer, thermal degradation is a key factor to monitor. Heating the polymer up past its melting point, especially in the presence of oxygen, can have a devastating effect on the polymer chains and can lead to poor process-ability.

If dissolving is an option, good solvents are used to fluidize the polymer. By dissolving the polymer, chains will be free to move around in the solution due to Brownian motion. In the process of making the nanofibers, the fibers are aligned while the solvent evaporates. Choosing the correct solvent can alter the final properties of the fibers. Solvents with higher vapor pressure evaporate more quickly, which makes them a candidate if rapid solidification of the fibers is desirable, to prevent fibers from sticking to one another [8].
Solvents that can dissolve polymers are often toxic for human health. Nanofibers, made from solutions that are used for medical applications, often need another treatment after the fibers are formed. Typically, they are held under vacuum to remove any remaining solvent that is trapped within the nanofibers [6].

Polymer nanofibers can be produced in a lot of different ways. Below is the summary of the most commonly used practices. Many of these techniques suffer from scalability as they only exist in lab scale setups. They suffer from low production rates because of low polymer throughput or are labor intensive. Scaling up the production would greatly improve the general adaptability of polymer nanofibers into various applications. As described above, nanofibers are very interesting but the production process will need to be scaled up to make it mainstream and affordable.

2.2.1 Melt blowing

Melt blowing is the one of the leading non-woven fiber manufacturing processes. Fibers are generally larger than 1 µm but can be manufactured form different polymers. Controlling the different parameters of the melt blowing process is required for each type of polymer.

Melt blowing first melts the polymer and redirects it in a specially designed nozzle. The nozzle has 2 types of inlets. The first inlet is used to push the melted polymer through. Through the second inlet flows heated, high-velocity air. The two steams meet each other at the tip of the nozzle (as shown in Figure 2). The high-velocity air impacts the polymer melt and pulls it in the direction of the air stream. This draws a very thin fiber from the polymer melt to obtain microfibers. The structure of melt blown fibers can by altered by adjusting the flow rates of polymer melt and air, temperatures of the polymer melt and air, intrinsic properties of the polymer melt, nozzle geometry, etc. [4], [6].

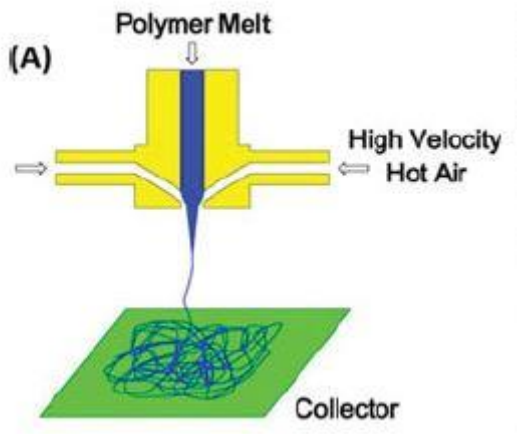


Figure 2:Schematic of melt blowing process [6, p. 680]

2.2.2 Bicomponent fiber spinning

Nanofibers can be produced through bicomponent fiber spinning. Two different immiscible polymers are used at the same time creating a fiber. Those polymers are melted and are co-extruded. They are then spun by a spinneret, creating a unique cross-section of the fiber. Different cross-sectional arrangements can be made by changing the spinneret. The nanofibers (providing that one of the polymers is extruded on a nanoscale) can then be obtained by splitting the different components or by removing one of the components of the bicomponent fibers.

The most common cross-sectional arrangements are island-in-the-sea, segmented pie and hollow segmented pie (shown in Figure 3A). A cross-section of island-in-the-sea shows multiple individual fibrils (islands) of one component surrounded by the other polymer. These fibrils are then removed by splitting or removing the other polymer, releasing the encapsulated fibers (shown in figure 3B). A segmented pie cross-section shows a shape much like an equally divided pie-chart where one polymer type alternates with the other. Hollow segmented pie's cross-section is similar to the one of the segmented pie, where one polymer alternates with the other, forming a pie. The difference is the hollow segmented pie creates a hollow tube [6].

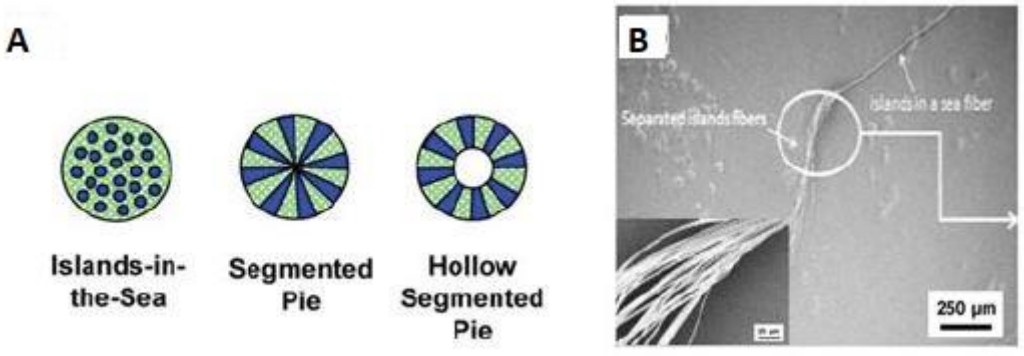


Figure 3:A) Typical cross-sectional shapes of bicomponent fibers used for producing nanofibers B)SEM image of Island-in-the-sea fibers being released from the surrounding polymer [6, p. 680]

2.2.3 Phase separation

Figure 4A shows the process of phase separation. The process is divided into five steps: 1) dissolving the polymer, 2) gelation, 3) phase separation, 4) solvent removal and 5) drying. First, the polymer is dissolved in a solvent to form a homogenous solution. This can be done at room temperature or at elevated temperature to speed up the dissolving process. The solution is then held at the gelation temperature, where the polymer solution forms a gel. The gel separates from the solvent creating a matrix of nanofibers. The solvent is then removed, followed by drying of the matrix. Finally only nanofibers are left. Figure 4B is a SEM (scanning electron microscope) image showing typical nanofibers produced by phase separation [6].

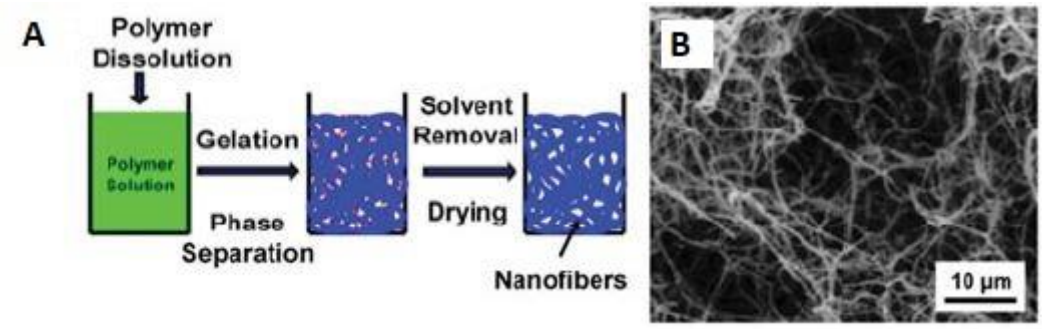


Figure 4: A)Schematic of the nanofiber production using phase separation process, B) SEM image from a typical formation of nanofibers produces by phase separation [6, p. 680]

2.2.4 Template synthesis

In template synthesis, a hollow porous template is created which acts as a mould for nanofibers. This template is later removed leaving only the nanofibers to be collected. First, a template is made from ceramic or polymer, followed by filling the template with monomers or polymers. Monomers are converted chemically or electrochemically to produce polymer nanofibers. Making nanofibers this way often results in a hollow structure because the synthesized polymer is prone to form onto the inner surface of the hollow channels (as shown in Figure 5B).

Nanofibers can also be formed by starting off with a polymer solution. However, this will lead to thicker fibers due to the higher viscosity of the polymer solution making it harder for the solution to penetrate all the way into the template. Nanofibers will be formed after the evaporation of the solvent. After the nanofibers are formed, the template is dissolved or etched releasing the nanofibers (shown in Figure 5A) [6].

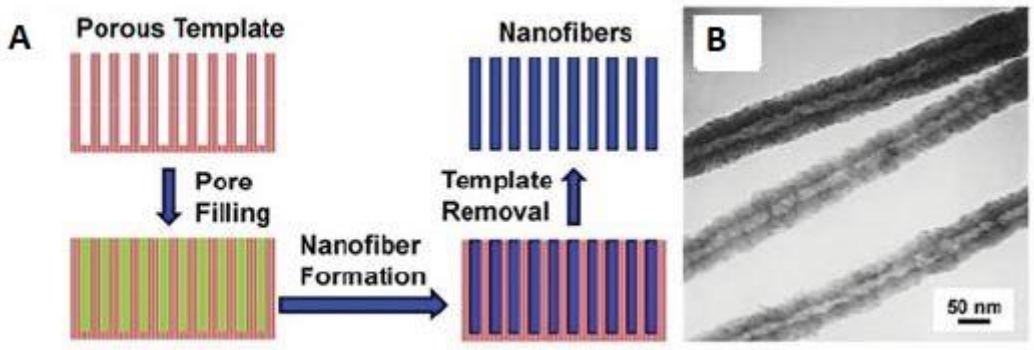


Figure 5: A) Schematic of template synthesis process, B) SEM image of fibers produced by template synthesis [6, p. 680]

2.2.5 Self-assembly

As the name implies, self-assembly is a technique where the nanofibers form due to self-assembly, caused by an external or an internal driving force. The most common mechanism is using a hydrogel that houses small molecules. These molecules are free to move in the gel and can interact with the other molecules. Inter-molecular forces draw these molecules together, causing them to 'self-assemble' into a long fiber. As the fibers form, the fiber phase and solid phase separates from the liquid phase (which is typically water). The fibers are then dried by evaporation of the liquid. Figure 6 shows how the mechanism works. The hydrogelator has both a hydrophilic and a hydrophobic end. The self-assembly works by achieving weak hydrogen bonds and hydrophobic interactions between the hydrogelator molecules forming a nanofiber [6].

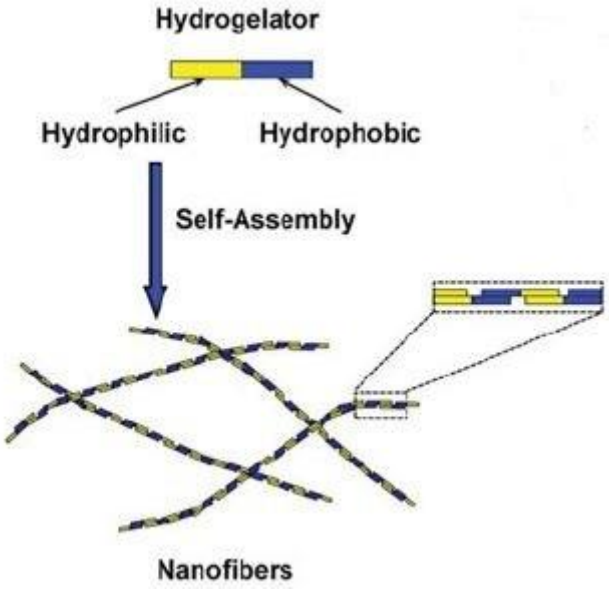


Figure 6: Schematic of self-assembly process [6, p. 680]

2.2.6 Electrospinning

Electrospinning is a commonly used technique in lab setups because of the high degree of uniformity of the fibers produced and its simplicity.

Electrospinning is a simple, non-mechanical technique based on a potential difference between a nozzle and a grounded plate. The potential difference between nozzle and the ground plate must be a *DC* voltage in the 10-50 KV range, making the setup simple, yet dangerous [9].

Electrospinning can only be used with dissolved polymers. This limits the number of polymers it can spin into a fiber. A syringe, connected to the nozzle, pushes the polymer solution at a slow rate so that it forms a pendent droplet. The applied charge between to the nozzle tip and the ground plate causes the pendent droplet to form a Taylor cone [10]. This droplet is then pulled downwards by the electric field, forming a jet. The electrically charged jet then undergoes a stretching and whipping process under the influence of the electric field. This elongates the jet and reduces the fiber's diameter to as low as tenths of nanometers. Figure 7A shows a schematic of the process, while Figure 7B shows a SEM image of nanofibers produced by the electrospinning process.

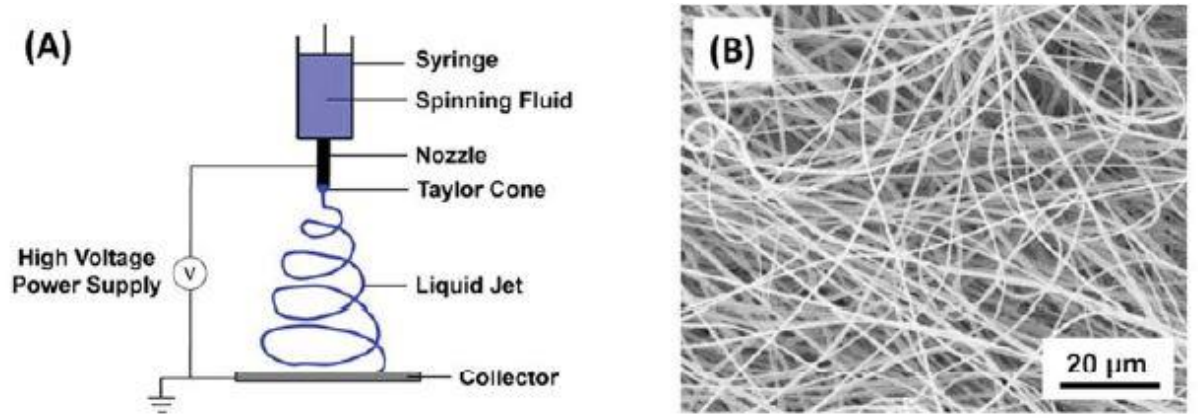


Figure 7:A) Schematic of the electrospinning process, B) SEM image of nanofibers produces by the electrospinning process [6, p. 680]

The morphology of the electrospinning fibers can be altered by changing relevant variables. Changing variables specific to the properties of the solution itself can have a significant impact on the outcome. It concerns variables such as polymer solution rheology, conductivity, surface tension, polymer molecular weight, and solution concentration. Variables that are specific to the setup can also be changed. Here it concerns variables such as electric field strength, solution flow rate, nozzle-collector distance and motion of the collector [6].

2.2.7 Centrifugal spinning

Centrifugal spinning, also known as Rotary Jet Spinning (RJS), Forcespinning (FS) or Centrifugal Jet Spinning (CJS), is a technique that uses centrifugal force to make nanofibers. Centrifugal spinning is a very versatile process because it can produce nanofibers from various polymers. It can produce nanofibers from polymers in a dissolved or molten state. By melting polymers, no harmful or toxic solvents are needed. Additionally, it allows spinning nanofibers that cannot be dissolved in strong solvents.

A centrifugal spinning setup is often comprised of very simple components. A *DC* motor is used to drive a cup, also called a spinneret, that spins at high *rpm* (rotations per minute). The speed of the motor can be easily controlled by changing the voltage going to the motor. A spinneret is basically a cup with holes in the side to let the fluid polymer through. Needles can be added to the cup. These can increase the path length of the polymer and they give different alignment characteristics to the fibers. For molten polymers, the cup itself can be heated to the melt temperature of the polymer inside the cup while spinning. The spun fiber is then collected on collecting posts, which catch the fibers when they are solidified [11]. A schematic of a basic setup is shown in Figure 8A. The produced fibers are strong and can be produced in a larger quantity compared to the electrospinning technique. Fibers are created more rapidly, and the spinneret can be constantly fed with molten or dissolved polymer. Collecting systems that constantly collect the fibers can also be set up to create non-woven nanofiber mats, so a constant production of fibers can be achieved.

Creating a nanofiber with centrifugal fiber spinning happens in three steps: jet initiation, jet extension and collection of the fibers. Jet initiation is where the base of the fiber is initiated at the orifice of the spinneret. This can only happen if the centrifugal force is large enough to overcome the capillary force and the surface tension of the spinning fluid. In the second stage, jet extension, the polymer solution gets elongated resulting into thin fibers. The fiber elongates by the centrifugal force and frictional force of the air acting on the fiber itself, thinning the fiber whilst the molten polymer solidifies (shown in Figure 8B). In the third and final stage, the fibers are collected on collection posts

or on a circular collector surrounding the spinneret. The fibers either continue to cool down or the solvent evaporates to solidify the nanofibers [3], [11].

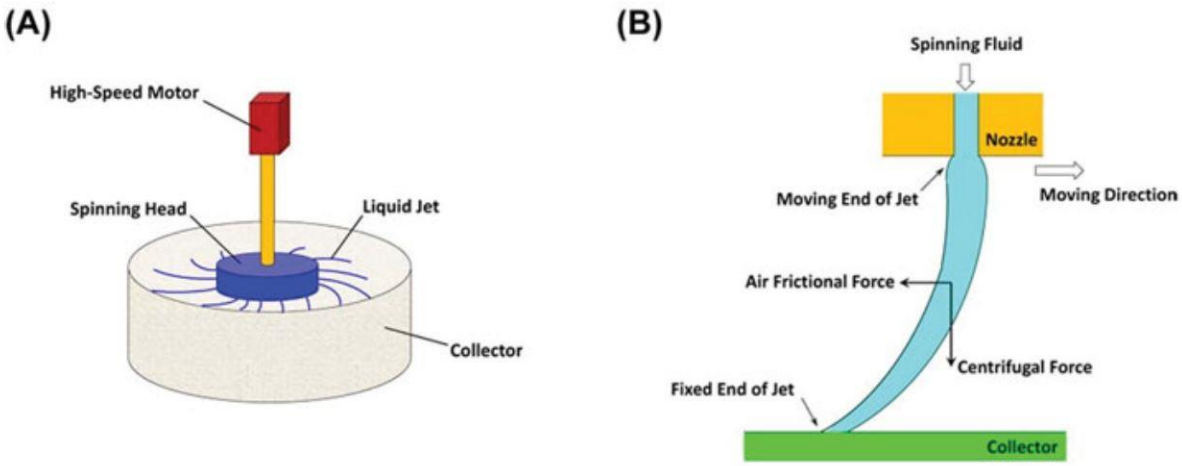


Figure 8: A schematic of a basic bench-top centrifugal spinning setup, B) the path of a liquid jet ejected from the nozzle tip during centrifugal spinning [6, p. 686]

Variables that can be changed to vary the size or uniformity of the fibers are the orifice size in the spinneret, the rotation speed of the spinneret, the viscosity of the polymer, the collector distance and the molecular weight of the polymer [11], [12].

2.3. Different setups of centrifugal nanofiber spinning

Currently, on a lab scale, electrospinning is a widely researched topic. Unlike other fiber making processes, electrospinning produces fibers made from single filaments whose dynamics can be easily followed. Excellent results can be achieved with electrospinning, but this technique lacks the scalability. Centrifugal fiber spinning is another interesting technique, but it is not commonly used in small scale nanofiber production. It is heavily being researched because of its scalable nature while achieving the production of uniform and thin fibers.
Centrifugal spinning is the main subject of this research. For this reason, only centrifugal fiber spinning setups will be presented here.

2.3.1 History

Spinning of fibers is not a new technology. Back in the 19th century the first widely spread application of fiber spinning was invented to make cotton candy [11]. Cotton candy is made of sugar fibers that are collected on a stick. It is a popular snack at amusement parks or fairs. A cotton candy machine consists of a fast spinning spinneret which is heated to melt the sugar. Through the holes in the side of the heated spinneret, molten sugar can escape once the centrifugal force overcomes the surface tension of the molten sugar. The sugar fibers are formed when the molten airborne flying sugar cools and solidifies. The airborne sugar fibers can be collected by twirling a stick around the spinneret, creating a large ball of stacked sugar fibers on the stick.

Centrifugal spinning is also used by the construction industry for producing insulation. As mentioned earlier in this research, thin fibers can trap air providing excellent insulation properties. The produced insulation material is called fiberglass or glass wool. Fiberglass is not only used for insulation but is used for multiple applications in the manufacturing process of boats, car body panels, roof panels and much more. To make fiberglass work for those applications, fiberglass is mixed with an epoxy resin which creates a very strong and light, yet quite flexible, panel. Like rebar in reinforced concrete, the glass fibers give structure to the epoxy resin. The glass fibers make the epoxy resin more resilient to bending forces that would normally break the epoxy. The production process of fiberglass is simple and is very similar to the production of cotton candy. A spinneret with thousands of holes, uniformly distributed in the circumference, is heated to 900-1000 ℃. The heating is required so molten glass wouldn't solidify in the spinneret with the risk of clogging the holes. Before adding molten glass, the spinneret spins at about 2000-3000 rpm. Molten glass is flung out of the holes in the spinneret. The fibers are being blown downwards by high-velocity air towards a conveyor belt, while being sprayed with a binder that allows the fibers to stick together. The high-velocity air also breaks the fibers into shorter fibers with a length of several centimeters. The conveyor belt moves the collected fiberglass mat into a curing oven where the binder is cured, making a coherent fiber mat [6]. Figure 9 shows a schematic of the fiberglass making process.

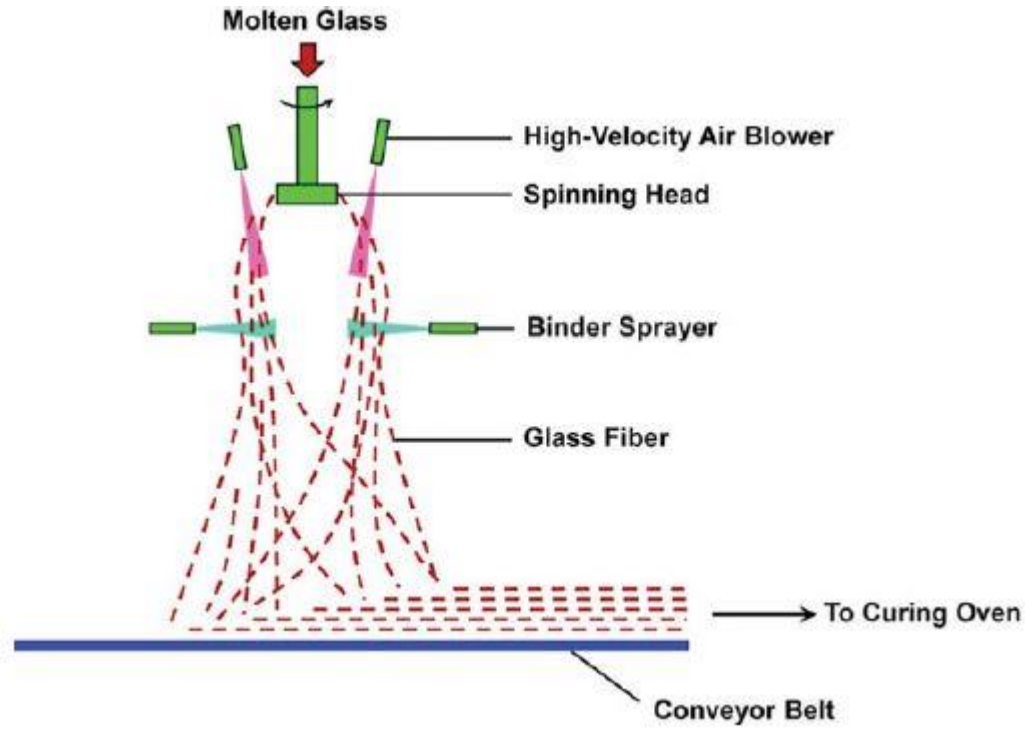


Figure 9: Schematic of a typical centrifugal spinning process used in the fiberglass industry [6, p. 684]

2.3.2 Different setups of lab scale centrifugal fiber spinning

Centrifugal spinning is a simple way of creating polymer nanofibers. The setup comprises two major components: the design of the spinneret and the way the fibers are collected.

2.3.2.1 The design of the spinneret

There are two types of spinnerets.

The first type uses a spinneret which is based on syringes. Two syringes are placed in a cradle holding the spinning liquid (shown in Figure 10). At the tip of the syringes different needles can be attached to change the size of the orifice the spinning fluid has to pass through. The size of the orifice can influence the final diameter of the spun nanofibers. Once the syringes are loaded, a motor drives the cradle to spin it at a high rotational speed. The centrifugal force acts upon the spinning fluid inside the syringes, forcing the spinning fluid outwards. The spinning solution is ejected from the needles creating nanofibers [6].

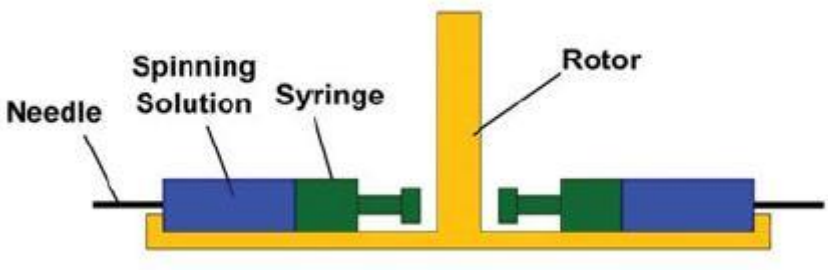


Figure 10: Schematic of a syringe based spinning head [6, p. 687]

The second type is more frequently used than the first one. Here, the spinning head is a cylindrical shaped spinneret especially designed for this process. A hollow cylinder is used as the base for this type of spinneret. In the circumference of the spinneret, holes are drilled to act as nozzles. In the top

of the cylinder, a hole is made to use as a spinning fluid inlet to feed the spinneret while at an operating rpm. The spinning fluid can escape through the holes in the sidewall of the cylinder. The size of those holes or nozzles can be chosen according to the desired properties of the spun fibers. Heating coils can also be added to the spinneret to enable it to produce nanofibers from polymer melt [6]. A schematic of this setup can be found in Figure 11.
Because of the use of heating coils, the cylindrical spinneret can produce fibers from polymer melt without dissolving them in a strong solvent. The fibers are therefore solvent free and nontoxic which is important for medical applications. There are a couple of advantages using the cylindrical setup over the syringes.
The use of syringes creates a challenge regarding balancing the cradle. A lack of balance can induce vibrations in the cradle. Those vibrations can cause inconsistencies in the nanofibers. However, an advantage of using the syringes is the rapid changing of different sized needles to generate different results.

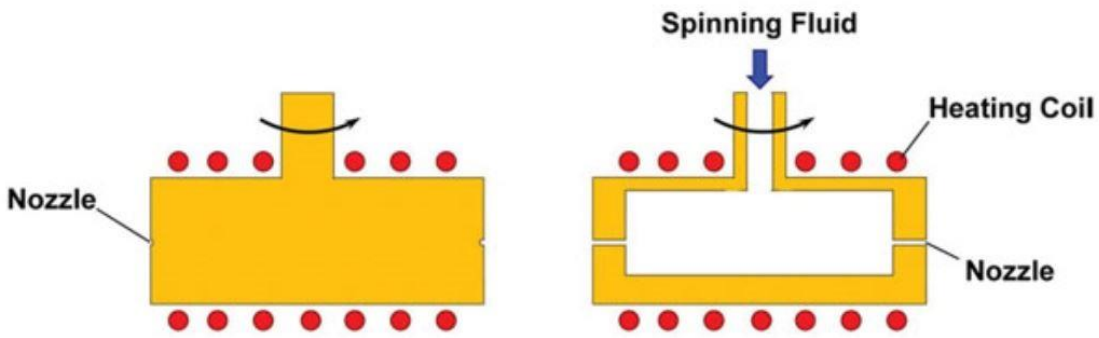


Figure 11: Schematic of a cylinder based spinning head [6, p. 687]

Combinations of the two philosophies are also possible. Having a cylindrical spinneret with needles functioning as nozzles is the most obvious choice. The spinneret does not suffer from balance issues as much as the syringe-based spinneret and still enables change needles by different sized ones to obtain desired results. Figure 12 shows an operational setup that has spun a nanofiber web. Another advantage of using this setup, besides the interchangeable nozzle sizes, is the ability to easily clean the equipment. The needles used, are often off the shelf parts and can be disposed of, leaving a larger hole in the spinneret which makes it easier to use utensils to clean the spinneret itself. By using a drilled nozzle, the size of the hole is often too small to use cleaning tools. Additionally, there is a risk of making the hole bigger during the cleaning process, possibly changing test results in future experiments.

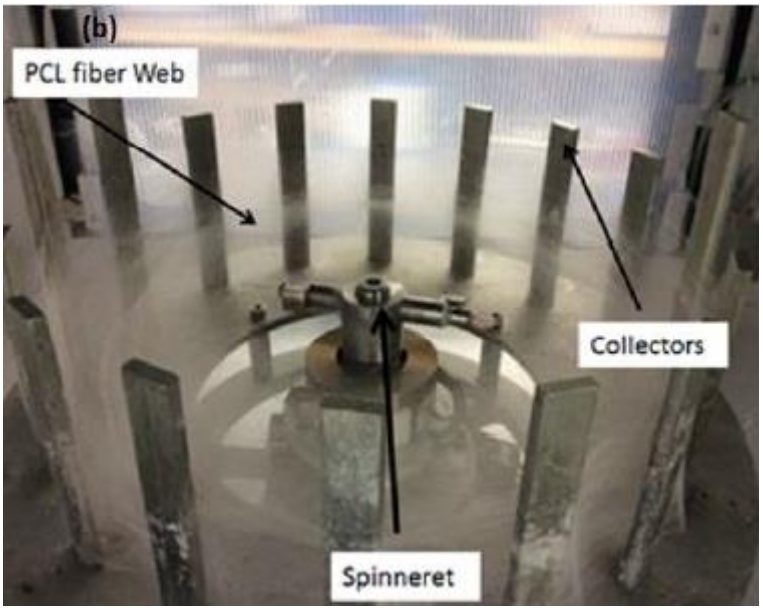


Figure 12: Operational setup with a cylinder-based spinneret with needles [8, p. 4]

2.3.2.2 Fiber collection system

The collection system of the nanofibers can differ between setups and depends on the requirements and desired test results. Three main principles present themselves as driving forces behind the collection systems.

The first principle uses centrifugal force to collect the fibers on posts surrounding the spinneret. The spinneret jets the polymer out of its nozzle(s) in a circular pattern as the centrifugal force acts upon them. The collection posts are commonly placed in a circular pattern surrounding the spinneret. The posts can be placed at different distances away from the spinneret to acquire different results. An airfoil can be added to the spinneret to manipulate the air flow and to improve the alignment of the collected fibers that would otherwise be collected at different locations. The alignment may be required for certain applications of the spun nanofibers. In Figure 13, a schematic drawing is shown of a centrifugal spinning setup that uses this principle. The setup also uses an aluminum sheet to surround the setup protecting the spinning process from air disturbances from the lab [11].

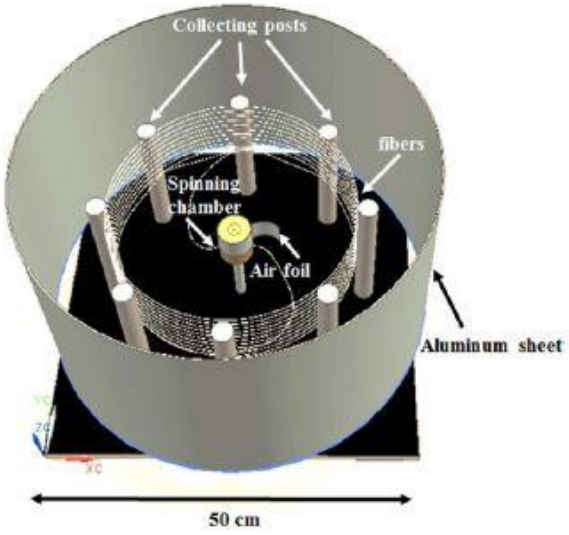


Figure 13: Schematic of a centrifugal spinning setup using centrifugal force as the collection system [11, p. 2594]

The second principle relies upon airflow pushing or pulling the spun nanofibers to the collector. As the fiber is spun, air jets or fans push the fibers towards a collector as the airflow grabs hold of them. Suction can also be used to pull the nanofibers against the collector. The collector can be static making the process less continuous. To make the collection process continuous, conveyor belts can be used to collect a continuous piece of nonwoven nanofiber mats. When using suction however, the conveyor belt needs to be porous, so air can pass through. Often these porous belts are made from textile fabric, paper or other porous membranes [6]. Figure 14, 15 and 16 depict schematic setups of centrifugal spinning setups using airflow as the basis of the collection system.

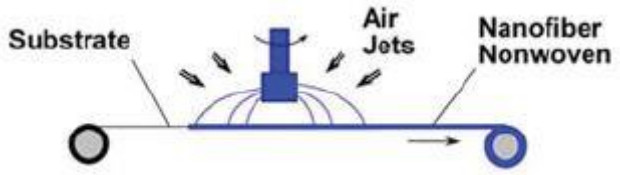


Figure 14: Schematic of an air jet based continuous collection system [6, p. 688]

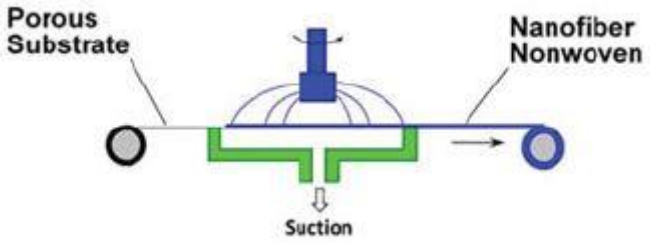


Figure 15: Schematic of a suction based continuous collection system [6, p. 688]

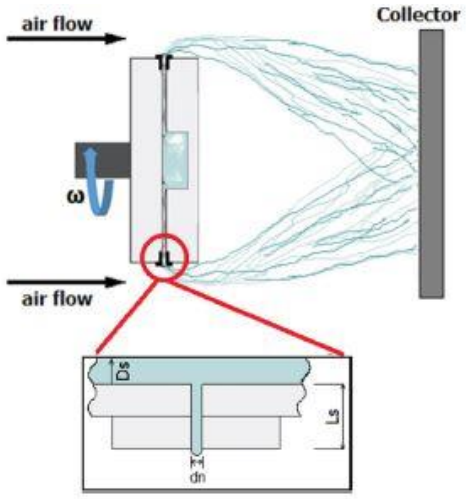


Figure 16: Schematic of an air flow-based collection system with static collector [13, p. 99]

The third and final commonly used collection technique is based on a potential difference between the spinneret and the collection surface. This technique is also known as an electrostatic-assisted centrifugal spinning setup (E-CS). It is a hybrid between electrospinning and centrifugal spinning.
A spinneret is used to create the nanofibers. Once the fibers eject from the spinneret, they are pulled towards the collection surface. A conveyor belt can be mounted on top of the collection surface to make a continuous collection of nonwoven nanofiber mat.
This setup creates more uniform fibers because the fibers are less impacted by the airflow surrounding the apparatus. Fibers are pulled towards the collection plate in a predictable and

gradual manner, hereby improving the uniformity of the collected fibers. The morphology of the fibers can be altered by changing the strength of the electric field like in an electrospinning setup [14]. Figure 17 provides a schematic depiction of an E-CS setup. With this setup, special attention is needed for the motor. If an incorrect or insufficient insulator is used, the operation of the motor could be disturbed due to the high electrical potential added to the shaft of the spinneret.

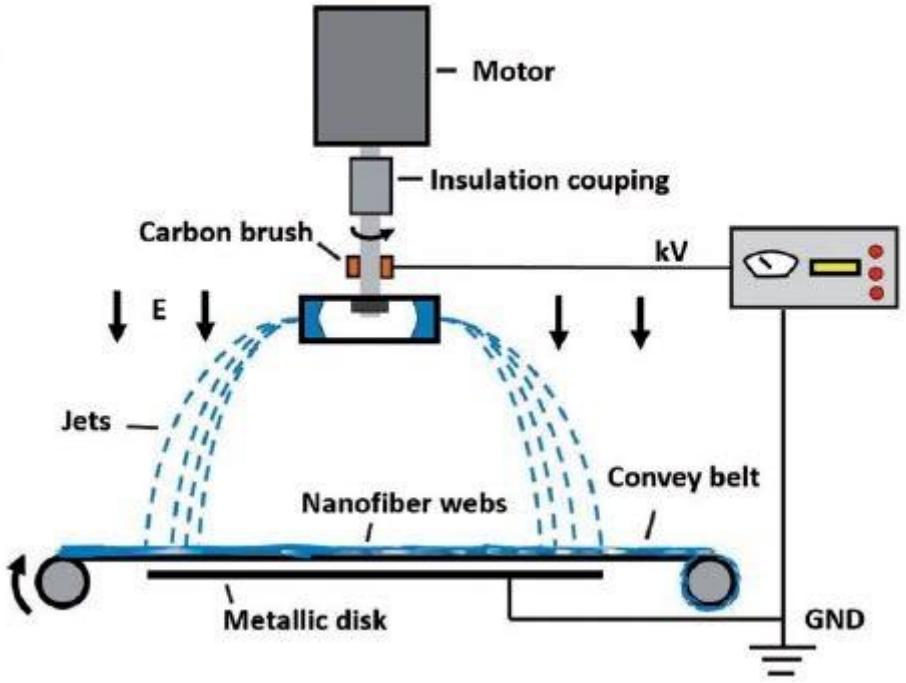


Figure 17: Schematic setup of an electrostatic-assisted centrifugal spinning setup [15, p. 3]

2.4. Why use centrifugal spinning?

To industrialize a certain technique or product, quality and quantity is needed. In the nanofiber industry a gap between supply and demand calls for an upscale of the existing nanofiber producing technologies with high cost-versus-yield efficiency and versatility [11]. Table 1 shows a comparison of production rate, material choice, safety and cost for different nanofiber production methods.

Table 1: Comparison of production rate, material choice, safety and cost for different nanofiber production methods [6, p. 682]

Method	Production Rate	Material Choice	Safety	Cost
Centrifugal Spinning	High	Broad	High	Low for industrial production, medium for academic research
Electrospinning	Low	Broad	Relatively low due to the use of high voltage	Low for academic research, high for industrial production of nanofibers due to low production rate
Melt Blowing	High	Narrow	High	Medium
Bicomponet Fiber Spinning	Medium	Narrow	High	Medium
Phase Separation*	N/A	Narrow	High	N/A
Template Synthesis*	N/A	Narrow	High	N/A
Self-Assembly*	N/A	Narrow	High	N/A

*These three methods have only been used for laboratory research and their production rate and cost are not reported.

Melt blowing, bicomponent fiber spinning, phase separation, template synthesis and self-assembly are not optimal to industrialize because of their narrow range of material choices.

Electrospinning

Electrospinning is the most commonly and widely used technique for spinning microfibers on a lab-scale operation because of its low cost, simple setup and great uniform results. The setup is simple to build with minimal equipment and the broad choice of materials makes it a very versatile machine. Because the technology is so commonly used, the techniques to operate the appliance are perfected, creating a great foundation for research.
The biggest drawback of using electrospinning is the low production rate at about 0,2 g/hr. and the ability to only spin nanofibers from a solution. This limits the material choice to only soluble polymers [1].
Safety is another big issue. Because of the high *DC* voltages used (10-50 kV), electrical shocks or burning the operator are a serious risk.

Ways to mitigate the electrical shock risk is the installation of safety systems like a safety hood or interrupting the power supply once opened. This lowers the risk of shocking the operator whilst insuring lowering the risk of turbulent air coming in from the lab. The issue of the limited production rate can be improved using nozzle-less electrospinning or multi-nozzle electrospinning. The production rate from those techniques however, is not able to meet the increasing global demand [6]. Upscaling the electrospinning process isn't possible due to the limitation of the high electric field strength needed to produce the fibers. Additionally, building many setups would imply a high investment, which wouldn't be sustainable.

Centrifugal spinning

Compared to electrospinning, centrifugal spinning can produce a variety of nanofibers and is more cost-effective to scale up the production to an industrial level. As discussed in the previous segment, the spinnerets can be designed to run both molten polymer and polymer solutions. This allows centrifugal spinning to produce nanofibers, destined for medical use, without the need for post-treatments to make it safe. Electrospinning nanofibers, which are created from a solution, need a post-treatment in a vacuum chamber to evaporate all the toxic chemicals. Another advantage of spinning fibers from a molten state, is the ability to spin fibers from polymers that cannot be easily dissolved by solvents.

Because centrifugal fiber spinning is not as widely tested as electrospinning, some issues regarding morphology of the fiber are still not understood. Testing must be done to discover the optimal working conditions and variables to improve the uniformity of fibers produced, and to decrease fiber diameters, before scaling up to industrial sized setups.

2.5. Test results with centrifugal spinning with changing variables

Utilization of nanofibers is mostly designed around one specific morphology of the nanofibers used in certain applications. Fibers must be uniformly shaped and need to have a specific diameter to ensure the projected properties of a specific application. Tests are required to understand irregularities in the nanofibers and to reduce them to a minimum. Irregularities like beads, beads-on-fiber and holes are common results identified in lab-scale testing [11] (shown in Figure 18).

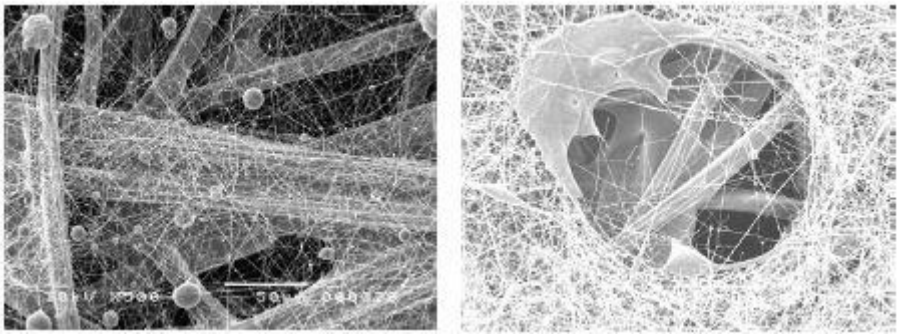


Figure 18: SEM image of the formation of beads (on the left and the formation of holes (on the right) [14, p. 85]

Holes are a major concern if they occur in nanofiber mats destined for use in filtration systems or in drug delivery systems. Holes are created when spun fibers, created out of a solution, reach the collection posts before they have had the chance to solidify. The solvent has not fully evaporated at the time the fiber reaches the collection posts. The solvent then dissolves both the newly spun fiber and the surrounding nanofibers on the collection posts, creating holes in the nanofiber mats [14]. Once the solvent fully evaporates, a hole with a hard edge is left in the nanofiber mat.

Beads can also be formed during the spinning process. Beads are irregularities that give the nanofiber mat properties that are not desired for any given applications. Figure 19A shows a schematic representation of the formation of a nanofiber during the fiber spinning process. T1 represents the time the jet is initiated from the nozzle. T2 represents a continuous slender jet, following a spiral trajectory between the nozzle and the collection posts. T3 shows the jet extension phase. In this phase the fiber is extended to create more surface area while the solvent evaporates. Fibers can shrink up to 1/3 of their diameter after solvent evaporation [3]. During this process, air

friction force (*Fa*) acts upon the surface of the fiber jet due to the circular airflow, while the axial drawing force elongates the fiber decreasing the fiber's diameter until it reaches a stable value. With the increase of the length of the jet, the gas-liquid interface of the jet (fiber solution to air) becomes unstable. This can lead to a radial deformation of the gas-liquid. If this radial deformation cannot be controlled, the bead density of the fiber will continuously increase until it reaches a stable state. Figure 19B shows the radial deformation process while Figure 19C shows the actual process of bead formation. The formation of those beads can be explained by the Rayleigh-Taylor instability. If the centrifugal force reaches a higher level than the viscous force can counter, the gas-liquid interface of the jet turns into an array of undulated jet, which causes a wave profile in the polymer jet. The weak deformations in the jet results in a concentration of tensile stress inside the jet, causing it to break up in to beads [15].

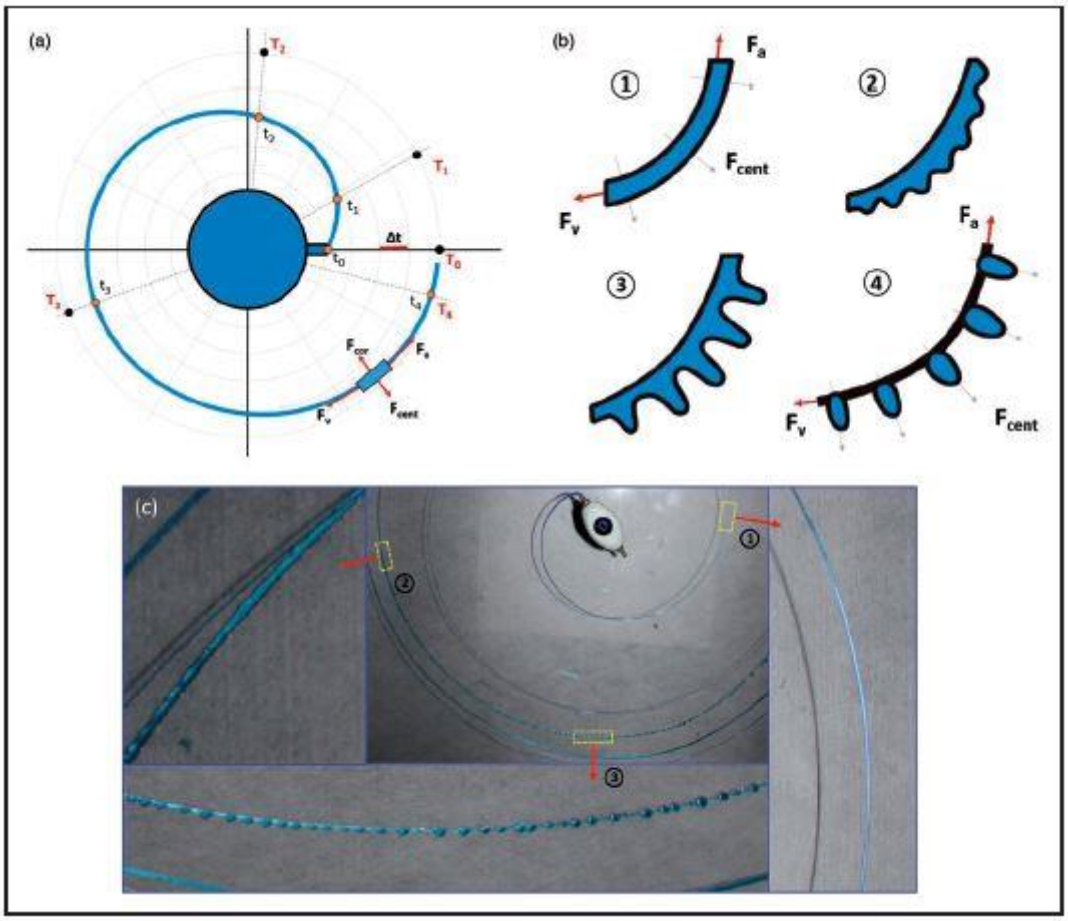


Figure 19: A) Typical process of a centrifugal fiber jet spinning, B) schematic of bead formation caused by Rayleigh-Taylor instability and C) actual test showing bead formation [15, p. 7]

Different variables can be changed to optimize morphology of the spun fibers. Fluid properties like viscosity or surface tension or operation conditions like rotating speed, spinning head diameter, nozzle diameter or collector distance can be varied to obtain the desired results.

Viscosity

The viscosity of the spinning solution can be altered by changing the weight percentage of the polymer in the solution, or by changing the temperature of the molten polymer. Heating the solution will lower the viscosity, while operating at lower temperatures will increase the viscosity. Likewise, reducing the weight percentage of the polymer in the solution will lower the viscosity, while adding more polymer to the solution will increase the viscosity.

A polymer in solution, however, is a non-Newtonian fluid. If shearing forces are applied, the polymer solution undergoes a process called shear thinning, lowering the viscosity. In the application of centrifugal spinning, shear thinning must be taken into account because of the centrifugal forces applied at the nozzles in the spinneret. At lower speeds, the centrifugal force is low, and the viscosity of the spinning fluid remains constant [1].

If the viscosity is too low, it means polymer chains in the spinning solution rarely overlap. This lack of chain entanglement results in fibers that are loosely aligned with large beads on them, forming a beads-on-fiber configuration [6]. Increasing the viscosity will resist the force of the surface tension, lowering the chances of bead formation [14]. The increase of the viscosity will increase the fiber diameter, resulting in a delicate balance between having a thicker fiber and having a bead-on-fiber morphology.

Surface tension

The surface tension is the driving force behind the formation of beads. It strives to lower the surface area of the liquid and forms spheres. Spheres have the lowest possible surface area for a given volume. Lowering the surface tension lowers the chance beads are formed, which means the viscosity can be lowered to create thinner fibers [6].

Centrifugal force

The centrifugal force is a force that is exerted on an object while it is rotating. The formula of the centrifugal force is listed below (Formula 1). The formula states that the centrifugal force(F_C) is equal to the mass (*m*) times the rotation speed (ω) to the power of 2 and multiplied by the radius (*r*).

$$F_c = m * \omega^2 * r \qquad (1)$$

This means that the centrifugal force rises exponentially when the rotation speed is increased and linearly when the radius is adjusted.
To spin nanofibers, centrifugal force needs to exceed the surface tension and the capillary force of the solution at the nozzle [3], [11]. This can be achieved in two ways. The first approach is to create a slow spinning spinneret with a large radius to increase the centrifugal force sufficiently to enable the creation of nanofibers. The other option is to lower the radius of the spinneret and increase the rotating speed.

Often, the rotation speed is chosen over the radius as the variable to be adapted. The rotation speed can be easily changed and has a bigger impact on the centrifugal force. Higher rotating speeds result in higher centrifugal and air frictional forces acting upon the spun fiber. This elongates the fiber even more, creating thinner fibers.

It must be noted that, if the rotating speed is too high, the speed at which the spun fiber moves through the air is higher. The fibers will reach the collection posts sooner because the flight time of these fibers is reduced. If the solvent did not have enough time to evaporate, the remaining solvent on the fiber will dissolve other fibers on the collection posts. This will increase the risk of holes.
The very high rotating speeds can also have the negative side effect of increasing the mass throughput, contributing to a larger fiber diameter [6].

Nozzle diameter

The nozzle diameter dictates how much of the spinning fluid can flow out of the nozzle. Larger nozzle diameters increase the flow rate of the spinning fluid resulting in a larger fiber diameter. Likewise, smaller nozzle diameters decrease the flowrate, resulting in thinner fibers [13]. If the nozzle diameter is too thin, the flow of the polymer solution is hindered due to the high capillary force. This stops the flow of the solution completely, making it impossible to produce nanofibers.

Nozzle-collector distance

The distance between the nozzle and collection post can be changed by moving them apart. A larger collection distance increases the collection time. The increased flight time gives the spun nanofibers time to fully evaporate the solvent while stretching the fiber. This results in thinner and more uniformly shaped nanofibers.

Conclusion

It can be concluded that the above parameters can have a significant effect on the fiber's morphology. Table 2 summarizes the impact of increasing parameters on the fiber morphology. A delicate balance between the design of the machine, machine settings and properties of the spinning fluid must be achieved to create uniform and thin fibers with the desired morphology.

Table 2: Impact of increasing parameters on fiber morphology [14, p. 88]

Increasing Parameter	Diameter	Holes	Beads
Polymer concentration	↑	↑	↓
Distance	↓	↓	↑
Flow rate	↓	↓	↑
Rotation speed	↓	↑	↑

3. BUILDING THE CENTRIFUGE

3.1. Requirements

The first step in the process of building a centrifuge is identifying the requirements. These will then drive decisions on the design of the machinery, which will need to meet the requirements.
Main requirements of this centrifuge are: sturdy build, variable collection distance, dynamic collection system, variable rotation speed, variable orifice diameter and ease of use.

The machine should include a flexible setup and provide the opportunity to obtain a lot of test results with minimum time and effort. The way this was executed will be addressed in the next sections.

3.1.1 Sturdy build

A sturdy apparatus will reduce the impact of small vibrations coming from the centrifuge itself or from the lifting system.
Constructing the centrifuge out of metal will make the machine heavy, which will make it less vulnerable to vibrations or turbulence induced from the centrifuge while spinning or from external sources.

Vibrations can lead to changes in the morphology of the spun fibers. The vibrations can induce a resonant frequency resulting in the formation of beads and/or broken fibers. Strong vibrations while spinning can also reduce the lifespan and safety of the machine. Once the resonant frequency of the machine is reached, the machine vibrates heavily which can result in the fasteners becoming undone, creating a potentially hazardous situation.

3.1.2 Variable collection distance

As mentioned in section 2.5, collector distance can influence the diameter of the fibers.
If fibers reach the collection posts at a larger distance, fibers tend to be more uniform and thinner than fibers collected closer to the orifice of the spinneret. Therefore, designing a centrifugal spinning setup where the collector distance can easily be changed, is desired to make the centrifuge suitable for many experiments. Literature study indicates that collection systems in a circular pattern are commonly used. It is also clear that adaptability is an important feature because many experiments will be required to see and understand changes in fiber morphology by changing the collection distance.

For this research, a design where the collection posts can be set up in an ellipsoidal pattern around the spinneret is preferred. This allows the researcher to take samples from different collector distances out of just one experiment.

3.1.3 Dynamic collection system

The requirements of this centrifuge also call for a dynamic collection system. The dynamic collection system will make sure that the spun fibers are not collected on top of each other. This will reduce the risk of presence of unevaporated solvent (that could be present in the fibers) creating holes in the spun fibers.
Without a dynamic collection system, the fibers would collect on top of each other creating a donut bundle of spun fibers, comparable to a spool of wire. Bundled fibers are not preferred when doing a centrifugal spinning research because of the difficulty of analyzing the fiber morphology. All the different layers of stacked fibers make it hard to visualize the fibers under an optical microscope.

A dynamic collection system ensures that the spun fibers will reach the collection posts next to each other instead of on top of one another and hence avoids bundling of the fibers. Such a system can be created by either lifting the spinneret or by moving the collection posts.
Ideally this dynamic collection system creates a nanofiber 'mat' with a thickness of 1 fiber. This facilitates easy characterization of the fibers. The nanofibers being in the same plane make it easier to focus on the fibers using an optical microscope.

3.1.4 Variable rotation speed

Along with the radius of the spinneret, the rotation speed alters the centrifugal force. As specified in section 2.5, a change in rotation speed changes the centrifugal force exponentially. Being able to adjust the rotation speed will allow for precise control over the amount of centrifugal force exerted on the spinning fluid. Larger centrifugal forces can help overcome the surface tension and capillary forces to generate fibers, especially when using a small orifice diameter.
Higher rotation speed will also cause an amplification of potential instabilities present in the centrifuge itself. During the building process, special care regarding stability needs to be taken. The spinneret needs to be as balanced as possible to limit the vibrations that could alter the fiber's morphology.

3.1.5 Variable orifice diameter

Developing a cup with different sizes of orifices, creates an opportunity in terms of flexible and cost-effective production and execution. A flexible tool, that covers all relevant orifices, avoids producing multiple tools to achieve the same goals. Also, these additional tools might get misplaced during the test process and will increase overall cost.
Having different orifice diameters in the setup, allows for quick changeover times. This machine setup will allow the user to easily switch between diameters and to quickly analyze the different results linked to individual orifice diameters. Finally, this setup will limit the risk of environmental changes that might occur in a lab setting. The tests will result in data that are more comparable than data of experiments executed under different environmental conditions.

3.1.6 Ease of use

Creating a device that requires a specific setting will not allow for a wide range of experiments. The ideal apparatus needs to be adaptable. Being able to change settings on the fly, will help to fine tune the process for optimal results.
The ease of use plays a big role when adjusting the machine settings. It must be easy for an uninitiated person to operate the centrifuge based on a limited set of instructions. Every handling that needs to be done, must be easy to understand and easy to execute. This will ensure making repetitive tests with different settings, viable in a short timeframe.

The ease of use also assumes that the machine can be easily assembled and disassembled. This implies that the cleaning process has been made easy and fast to carry out, and that the repetitive nature of the fiber spinning process is facilitated.

3.2. Design

Once the requirements of the centrifuge are identified, documented and agreed, the design process for the centrifuge can begin.
The design needs to ensure that the set requirements are met. In this phase, the machine parts are being identified, drawn up and agreed. Only once the design is optimized, with a full machine drawing as a result, and the machine parts defined, the building process can begin.
In Figure 20 a 3D model of the centrifuge is shown. It consists of 2 levels. The top level contains the

actual fiber spinning equipment which consist of a spinneret (attached to a motor) in the center and collection posts held up by the top plate. The lower level houses the electronics and the lifting mechanism.

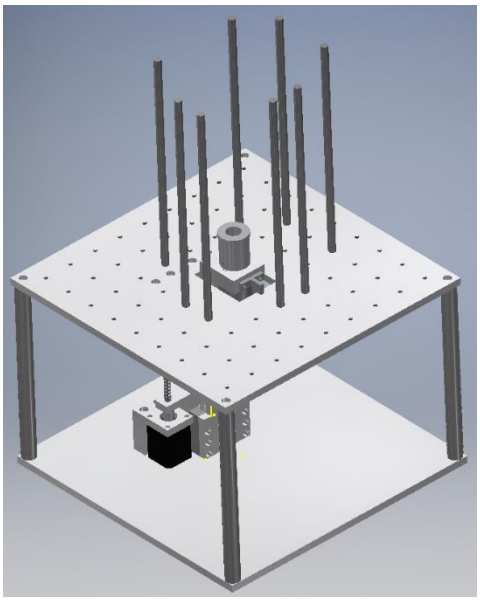


Figure 20: Design of the centrifuge using 3D modelling software

The next section describes the design process in further detail. The build plans, for the parts that needed to be built or customized, are provided in Appendix A.

3.2.1. Sturdy build

As discussed in section 3.1, sturdiness is a major requirement for the optimal operation of the centrifuge. For this reason, the centrifuge is built using heavy gauge material. This way, torsional and lateral movement of the machine which can induce unwanted vibrations, is reduced to a minimum. Two horizontally placed plates are used. These are separated from each other by thick rods that will help eliminate torsional forces that may occur. In the center, the two plates are supported by a guiding rail. This guiding rail is used for a dynamic collection system (section 3.2.3) but also acts as additional support for the two plates. Additionally, it limits the movement of the dynamic collection system to only one axis and resists movement from the other two axes. This will reduce the vibrations coming from the spinneret. Finally, provisions are made to add rubber feet on the bottom of the apparatus to cancel out any vibrations that might come from the lab environment.

3.2.2. Variable collection distance

Figure 21 provides a view of the top plate of the machine. In the middle of the plate, a large provision is made for the spinneret to pass through. Holes are drilled (and tapped) in the top plate, in a grid style pattern. These are placed at 40 mm intervals, which makes the collector distance easy to calculate. Another advantage of the grid style pattern is the ability to place the collection posts in an ellipsoidal pattern. This allows the researcher to take samples from different collection distances from only one experiment. At the same time, it eliminates the changes of environmental conditions that might occur between multiple experiments.
In total there are 72 holes where collection posts can be fastened to, which gives the researcher a broad choice where to place them. It allows for a large range of different collection distances, helping to fine-tune the fiber's morphology.

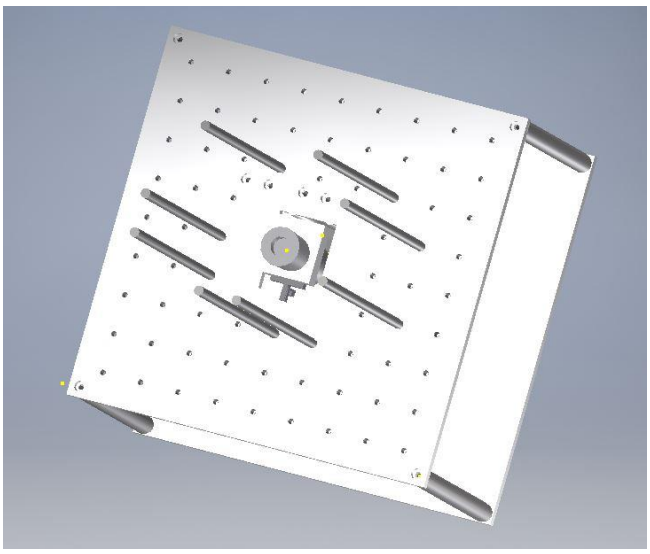


Figure 21: Ellipsoidal setup of the collection posts using 3D modelling software

Because the holes in the plate are threaded, collection post can be easily changed from one position to the next. It can be done without the help of tools and ensures a quick and seamless process, facilitating the iterative process of testing. The holes that are not used are blocked off using small bolts screwed in to the empty provisions. Blocking off the remaining holes will prevent spinning solution to gum up the threads in the holes, which would make it hard to screw in the collection posts for future testing.

3.2.3. Dynamic collection system

The dynamic collection will help prevent fibers from being collected on top of each other, making the samples more suitable for analysis using an optical microscope. The design of the lifting system is illustrated in Figure 22. The dynamic system operates by using a lifting system for the spinneret/motor assembly. This lifting system lifts the spinneret upwards or downwards while the spinneret is spinning, so the fibers are collected next to each other on the collection posts.

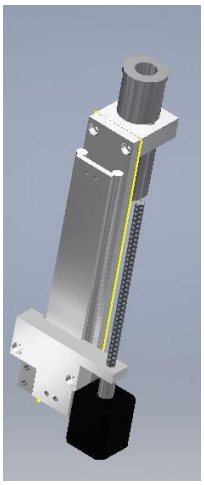


Figure 22: Schematic of the lifting system using 3D modelling software

The assembly is lifted by a stepper motor and a threaded rod. A stepper motor is a brushless DC motor that can be precisely controlled. The motor divides a full rotation into equal steps, making the position of the motor highly tunable. This tuning-ability will ensure precise controlling of the speed of the motor, and thus of the lifting speed.

The motor & spinneret assembly consist of a lifting arm, a motor support and a motor holder. The motor support and motor holder cradle the motor and are connected to a guiderail. The guiderail provides a smooth lifting motion but prevents moving from side to side, resisting possible vibrations coming from the motor and spinneret.

The lifting arm is attached to the back of the guiderail and connects the motor assembly with the threaded rod on the stepper motor. A threaded insert in the lifting arm makes sure that the rotational forces of the stepper motor/threaded rod are translated into lifting the assembly without too much frictional losses. The insert is made from stainless steel, which will ensure that there is minimal wear and that the machine will last going through many cycles.

3.2.4. Variable rotation speed

A DC motor is used to spin the spinneret (shown in Figure 23). DC motors have the property that their speed can be controlled by varying the voltage. A higher voltage spins the motor faster, while a lower voltage will make the motor spin slower. The voltage delivery will be done by a variable power supply. The variable power supply can be set to whatever voltage (and in turn, the rotation speed) is desired to run the experiment.

Figure 23: DC motor used for spinning of the centrifuge

3.2.5. Variable orifice diameter

A variable orifice diameter can be achieved by using a spinneret with multiple sets of different orifice sizes. During the testing phase, it appears that the first design was limited in its use because of its smaller diameter. Therefore, a second design was required for a better spinneret design. Both versions are shown below.

3.2.5.1. Version 1

Version 1 is a very simple design (shown in Figure 24). It is a spinning cup with a lid. The cup is fixed onto the shaft of the motor with a setscrew to ensure the cup stays in position whilst spinning at high rpm. In the sides of the cup, 2 sets of 3 different size holes are drilled (6 holes in total). The holes are drilled on the lower end of the fluid reservoir, extracting all the spinning fluid in the process. The sizes of the holes are respectively 0,3 mm, 0,6 mm and 1,0 mm. These sizes are derived from the testing on the original setup with the lab centrifuge.

Figure 24: Spinneret design version 1

The lid of the spinneret closes the cup off using an O-ring. This prevents the spinning fluid to climb up the sidewalls of the cup and spill out from the top. In the lid there is a hole through which the spinning fluid can be added while the spinneret is spinning. The O-ring however (not visible in the picture, only the provision is visible), needs to be resistant to the chemicals used in the spinning fluid to ensure a longer lifespan. Making the lid removable (cup and lid) will help the cleaning process so contamination is kept to a minimum.

3.2.5.2. Version 2

Version 2, shown in Figure 25, has several improvements over the first version.
The first improvement is related to the size. The length of the spinneret in version 2 is more than twice as big as the diameter of the cup in version 1. The larger size will equate to a larger centrifugal force at the orifice exit compared to version 1 using the same rotation speed. The same method as version 1 is used to fasten the spinneret to the motor. Especially with the larger size, the importance of a solid connection is even more important in version 2.

Figure 25: Spinneret design version 2

A second improvement is related to the tips of the spinneret. Tips with different channel sizes can be fitted to vary the orifice diameter by screwing or unscrewing them (shown in Figure 26). In this case only 0,3 mm and 0,6 mm are used to see what test results these would produce before building new ones. Tips can be unscrewed to open the spinneret and facilitate the cleaning process. The interchangeable tips will also allow for additional tests with alternative orifice sizes, without the need for the development of a new spinneret (shown in Figure 27).
In the top of the spinneret, there is a provision to drip fill the spinneret with the spinning fluid, either when spinning or when it is stationary.

Figure 26: Spinneret design version 2 with threads showing

Figure 27: Interchangeable tips version 2

3.2.6. Ease of use

The previous sections explain how every variable in the machine setup can be changed with ease by the operator. The construction of the machine is very simple because all the parts that need to be fixed are bolted to each other. All the parts that can be adjusted, can be removed with one screw (the spinneret) or can be undone by hand (the collection posts and the screws in the top plate).

Because all the parts are robust and easily accessible, cleaning them is made easy. Additionally, it ensures minimal risk of contamination between samples from different tests.

3.3. Materials and methods

The construction of the centrifuge can be split up into 3 main phases. In the initial phase, all the machine parts are designed using a 3D modeling software. In the second phase, the designs from the 3D modeling software are converted into build plans. The designed components are fabricated according to the plans. The third and final phase is to combine all the components to a fully constructed and operational centrifuge.

3.3.1 Design

The design of the centrifuge is done using 3D modeling software, Autodesk Inventor 2018. 3D modeling software is a powerful tool to visualize a prototype. It visualizes the parts in a 3D virtual space, so interferences can be checked. These interferences can be addressed before the prototyping and production begins. This way of working is efficient, saves time and is cost effective.

Every part of the 3D model is then exported to a set of build plans (see Appendix A). These build plans specify the dimensions of the different parts. A workshop can translate these plans into physical objects as designed in the 3D model.

3.3.2 Construction

3.3.2.1 Material choices

Making the centrifuge sturdy requires using robust materials. Two types of materials are chosen for the build.
Aluminum is used on almost all components. It provides a solid base to limit the transfer of vibrations through the setup. Aluminum is easier to work with compared to other metals like steel, and still provides ample strength to cut threads and make strong connections.
Some parts, like the threaded rod for the lifting system, the threaded insert for the lifting arm and the collection posts, are made from stainless steel. In case of the insert for the lifting arm and the lifting system, stainless steel is chosen for its higher resistance to wear. The lifting system copes with high stresses trying to lift the spinneret and motor assembly. Having the lifting system made from stainless steel will insure a long lifespan. The collection posts are also made from stainless steel. In this case, the objective is not to have a wear resistant property, but primarily to have a smooth and clean surface. Stainless steel is less prone to have chemical reactions with the air or with solvents used. Aluminum forms a strong thin oxide layer on the surface which prevents rust from forming and gives the metal a protective layer. Another advantage of using stainless steel is the roughness of the surface. Stainless steel has a very smooth surface making the cleanup process much easier.

3.3.2.2 Fabrication

Once available, the build plans are investigated in great depth. The outcome of this study is a plan of approach for the construction of the machine, hereby considering efficient use of raw materials. The build plans are then printed to size. This allows for checking the work along the way and for making sure the manufacturer is on the right track.

The fabrication process includes shaping round objects on a lathe, milling flat objects down, drilling holes and tapping holes. These processes allow the manufacturer to shape the parts to the desired dimensions and/or shapes.

The parts for this project are produced by Johan Soogen in the workshop of building D on the campus of UHasselt Diepenbeek.

3.3.3 Electronics

Electronics are needed to operate the lifting system and to measure the rotation speed of the motor/spinneret. For this project an Arduino Uno is selected as the brains of the electronics. The Arduino is a microcomputer that can be programmed with the Arduino IDE software. It is an ideal circuit board to make prototypes of any kind because of the comprehensible programming language and the great support from forums online. The code for this project can be found in Appendix B.

The lifting is done via a stepper motor. As discussed, a stepper motor is a motor that divides a full rotation into equal steps. Those equal steps can be set at required intervals, allowing a very precise control over the speed of the motor. The electric signals are controlled via a stepper motor driver, in this case a Pololu A4988. The Pololu gets 12V from a separate power supply to drive the stepper motor. Additionally, it receives 5V and signals to control the logic on the driver from the Arduino via jumper leads. The stepper driver then gets controlled by a simple code that is written on the Arduino. A potentiometer is fitted to the electrical system and is programmed to control the speed of the stepper motor. The operator can choose at which lifting speed the experiment needs to be carried out.

To measure the speed of the spinneret, a generic infrared interruption signal from an infrared sensor is used. A small piece of reflective foil is attached on the rotating assembly. Every time the assembly

rotates, the infrared light is bounced of the reflective foil and is captured by the sensor. This completes a circuit prompting that a full revolution has been completed. The Arduino has an 'interruption' channel built in. This channel measures how many times the circuit of the IR (infrared) sensor is closed and opened in a certain period. The signals can then be converted into *rpm* (rotations per minute), to return a number relevant to the operator.

The Arduino also drives an LCD (Liquid Crystal Display). The display provides a digital read-out of the status of the experiment. The display provides information regarding the *RPM* of the motor which can be noted for the analysis afterwards. A second read-out provided by the display, is the percentage of the speed at which the stepper motor is performing. A hundred percent would be the maximum speed, while zero percent projects the minimum speed at which the lifting system can run.

All the electronics are fixed to a 3D-printed base to house all the components (see Figure 28). This makes the setup less fragile while transporting the equipment or when movement from the environment occurs. Examples of unexpected impacts are a misplaced hand from the operator or something hitting the machine accidentally.

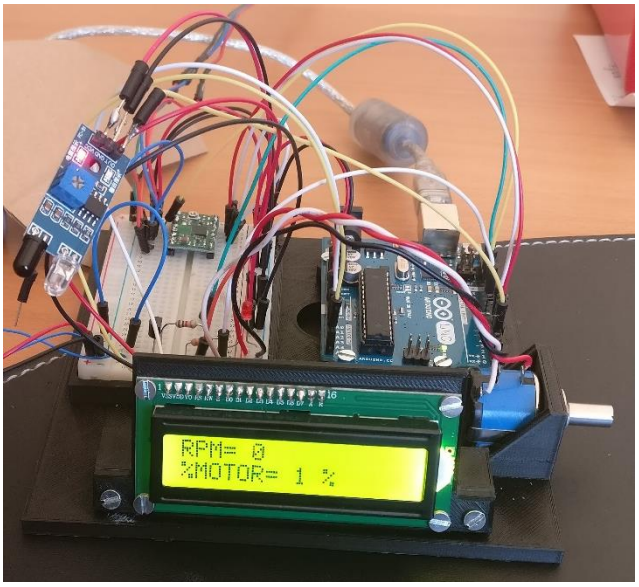


Figure 28: Electronics controlled via an Arduino on a 3D printed base

3.4. Fully assembled centrifuge: Thoughts and impressions

All the machine parts are carefully manufactured so that the assembled result meets all the requirements identified. The assembled centrifuge is depicted in Figure 29.

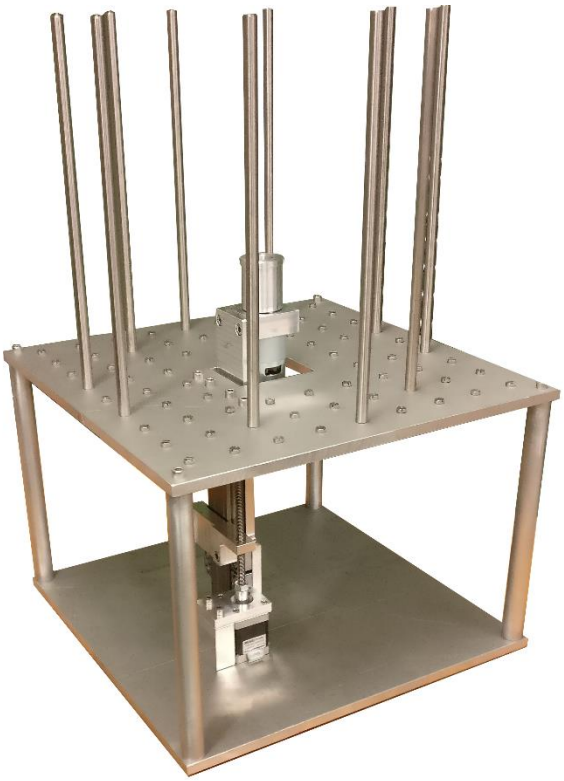


Figure 29: Fully assembled centrifuge

3.4.1 Thoughts and impressions

The centrifuge has been crafted using premium materials which demonstrates the level of care put in to the fabrication of the different components. All parts were very precisely manufactured which ensured a smooth assembly. Every component exactly matched the ones designed in the 3D model, so no parts had to be redesigned nor remanufactured.

Because the machine was built using high quality aluminum, the assembled apparatus is a very sturdy construction. A smooth operating lifting system and collection posts that can be rearranged very easily, ensures the adaptability of the centrifuge. The heft of the materials lowers the susceptibility to vibrations induced by the environment.

Looking back, there is one further improvement that can be made to the setup. The point at which the motor assembly is lifted, lies next to the guiderail at the meeting point where the lifting arm connects to the threaded rod. Because it connects to the side of the motor assembly, torsional force is induced to the threaded rod through the lifting arm, providing more friction. A better solution would be to create a lifting arm that connects to the threaded rod behind the guide rail. This setup would lower the torsional force on the guide rail, hereby reducing the friction on the guiderail, making the lifting mechanism operate even more smoothly.

4. PRODUCTION AND CHARACTERIZATION OF FIBERS

4.1. Materials and Methods

The produced nanofibers are made from PS (polystyrene). The PS is dissolved in THF (Tetrahydrofuran) to be able to spin nanofibers with simple equipment, given there is no need for a heating element at the spinneret. THF is a fast evaporating solvent. Once fibers are spun, the solvent quickly evaporates, creating solid polymer nanofibers. Solutions using different weight percentages are required to enable the production of nanofibers.

4.1.1. Mixing solutions

The solutions used for fiber spinning are 17,5 wt% (weight percentage), 20 wt%, 22,5 wt% and 25 wt%. The solutions are made using a scale. For example, in case of 20 wt%, 20 % of the solutions weight consists of PS while 80 % of the weight consists of THF.

THF is a potential carcinogenic solvent. It belongs to IARC group 2B carcinogens [16]. Caution needs to be taken when handling the substance. Making solutions and running experiments needs to be done under a fume hood to exhaust the evaporated THF. Another precautionary step is to put on gloves, as skin contact with THF and PS will irritate the skin.

First, the PS pellets are weighed inside a recipient. PS is weighed first because the THF would evaporate at a high rate while in the process of weighing the PS, leading to an inaccurate measurement of the mixture. In this case, the weight percentage of the mixture would be higher than intended.
PS pellets are carefully added with a spatula to make sure the correct amount is weighed. THF is added with the use of a pipet to ensure an accurate amount is weighed.

Vigorous shaking or vibration is needed to agitate the components and fully mix the PS and the THF. The use of a lab mixer, ultrasonic mixer or vibration table may help speed up the mixing process. Overall, solutions with a higher weight percentage takes more time to mix.

4.1.2. Producing fibers

The first step in the nanofiber production process is to clean the spinneret thoroughly using a spatula and THF to scrape off and to dissolve any remaining PS in the spinneret. The spinneret is then fixed to the output shaft of the motor. A collection pattern for the collection posts is chosen depending on the variables that are being tested. Finally, before the spinning process begins, the holes that are not used on the spinneret (version 1) are taped off so no fluid can escape from those holes. In case of spinneret version 2, the correct end pieces with the desired orifice size need to be screwed on for the test.

The electronics are programmed in a way that the lifting mechanism returns to its starting position once the machine is switched on. When starting the fiber spinning process, the spinneret is spun up to the desired speed. This speed can be measured by the frequency at which the spinneret triggers the IR sensor.
The solution, containing PS and THF, is then added to the spinneret using a pipette to limit the dosing and to limit the risk of too much mass hitting the spinneret causing it to slow down. The formed fibers are collected and then placed between microscope slide glasses. It is important that, when collecting the fibers, these are cut rather than pulled off. Ripping the fibers would stretch them even

more, hereby reducing the accuracy of the measurements performed during the characterizing process.

Precautionary measures need to be taken when running the experiments.
Firstly, the fume hood needs to be closed to the marked workspace level to ensure a safe operating lab environment.
Secondly, the spinneret cannot be filled with the solution before spinning up the spinneret. Filling up the spinneret earlier may cause fibers to be spun at lower rpm making the test results inconclusive. Another drawback of filling up the spinneret too early is that the rpm is difficult to set. Not meeting the desired rpm also lowers the accuracy of the experiment.
Finally, the collection of the fibers needs to be done very carefully and cautiously in order not to damage these fibers. The aim is to cut the fibers rather than ripping them. Cutting the fibers does not change their morphology. Ripping them, however, will stretch and elongate these fibers, resulting in a less accurate measurement when characterizing them under the microscope.

Every test must be repeated 3 times to ensure the results are conclusive.

4.2. Characterization

Characterizing is done to evaluate the nanofibers produced during the experiments. Great care needs to be taken when handling the samples, so no samples and/or test results are mixed up.

In the characterizing process, the overall morphology of the fibers is assessed. Before the actual computer aided characterizing process, a visual judgment can be made. Visually, the following can be assessed: quantity of fibers formed, potential presence of beads, thickness of the fibers, texture of the fibers etc. Those judgements can be made before visualizing the fibers under a microscope.

4.2.1 Microscopic analysis and characterization using ImageJ Software

To start the characterizing process, a cover slide is placed on top of the fibers that are resting on the slide glass. The function of the cover slide is providing a compacting force on the fibers. The weight of the cover glass will slightly compact the fibers beneath. This compacting process brings the fibers in the same plane and enables the optical microscope to create a clear image. The optical microscope struggles to focus on different depth levels due to the way it operates by focusing with lenses.

The optical microscope is fitted with a digital camera connected to a computer. Software on the computer will allow the image of the camera to be displayed on the monitor.

A specimen is chosen and put under the microscope. A magnification is chosen to display the fibers. Once the image on the monitor displays a desired picture, the image is frozen and captured. An example of a picture taken with the microscope is shown in Figure 30. The software, camera and microscope are calibrated to accurately scale the image. The scale is captured on the bottom right of the picture to document the relative size of the picture and magnification.
This process needs to be repeated 5 times for each specimen in order to improve the statistical accuracy.

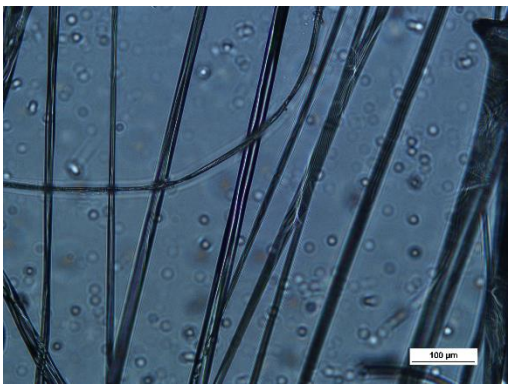


Figure 30: Example of a picture of the spun fibers taken with a microscope

In practice it is hard to ensure that all the fibers in the image are focalized. Refocusing will make the other fibers more visible. The images can be combined using image processing software to form the clearest picture possible.

The actual characterization is done using ImageJ software. ImageJ is an imaging processing software used for optical and computational instrumentation. The size of the fibers is determined with the DiameterJ Plugin.

The first step of the process is to select the image, created by combining the pictures of fibers in focus, and converting the image to an 8-bit version of this picture (seen in Figure 31). Using the DiameterJ segmenting Plugin, the 8-bit image is segmented.
The result of the segmentation process is a set of black and white versions of the 8-bit image which are all different from each other. The best image is chosen and put in the 'best segmentation' folder (seen in Figure 32). If the image needs to be cleaned up, it can easily be done using the ImageJ-software.

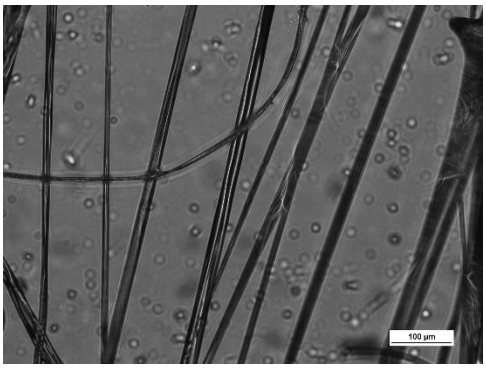


Figure 31: 8-bit image generated by the ImageJ software

Figure 32: Segmented image generated by the DiameterJ Plugin

Finally, the DiameterJ Plugin is used to analyze the black and white segmented picture resulting in a histogram and an Excel-file that show the mean diameter together with the standard deviation.

All these steps are repeated for every picture taken with the microscope.

4.3. Results

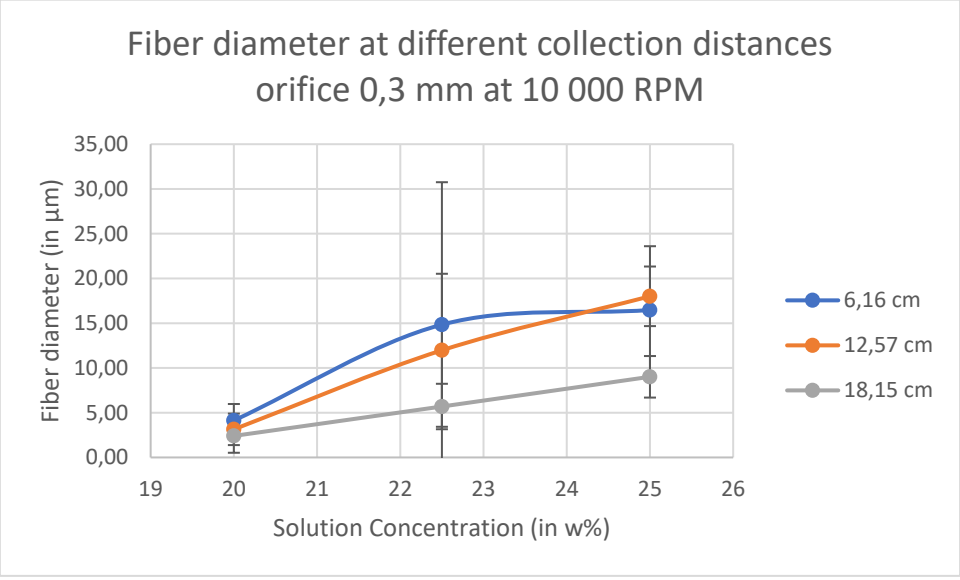
All the results of the tests performed, referred to in this section, were obtained using version 1 of the spinneret running at a speed of 10 000 rpm. Due to the time constraints of this thesis, version 2 of the spinneret was designed but not fully tested. Further research will be done on version 2 of the spinneret in a later stage.

4.3.1. Changing collection distance

Graph 1 below represents the fiber diameters of fibers produced in a test with changing collection distances. As previously described, the collection posts are positioned in an ellipsoid to generate multiple samples out of one test. In this case, the collection distances were 6,16 cm, 12,57 cm and 18,15 cm. This test was carried out with 3 different concentrations of spinning fluid: 20 wt%, 22,5 wt% and 25 wt%. The test results confirm the findings of similar tests done in other researches. The further the collection distance, the lower the fiber average diameter. Another finding is that the concentration has a definite effect on fiber diameter. The higher the concentration, the higher the fiber diameter.

The average fiber diameter is indicated by the colored curves and the standard deviation is given at every data point. The standard deviation is large, meaning that both thin and thick fibers are produced. The thick and thin fiber production is a reoccurring pattern in most samples taken. This can be called a 'bimodal' fiber distribution. See section 8.1 regarding hypotheses on the reasons why bimodal fiber distribution might happen.

It needs highlighting that these results were gathered from a small number of tests performed, thus it concerns limited statistics. However, the graph reveals a general trend of test results that can be obtained for given settings.



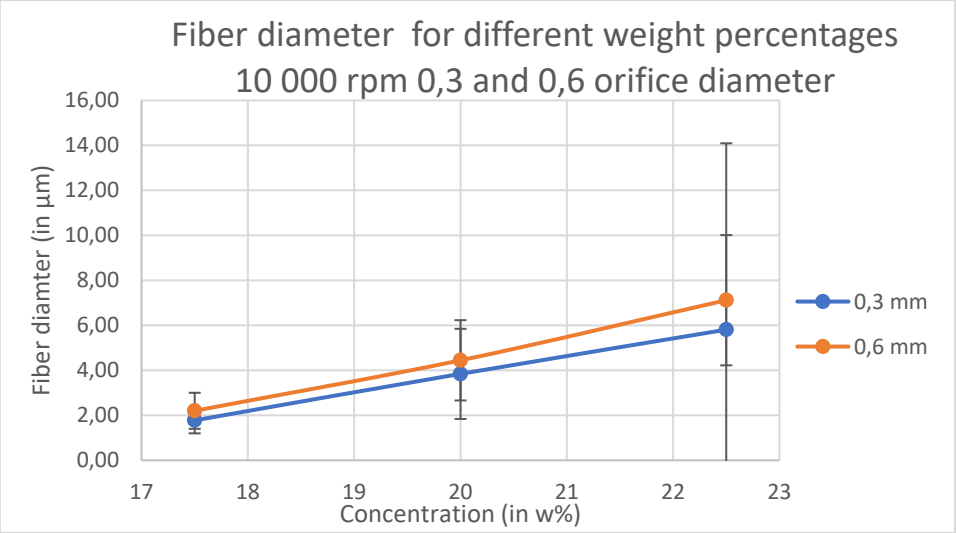
Graph 1: Results of fiber diameter at different collection distances using different concentrations

4.3.2. Changing orifice diameter

Graph 2 below reveals the results of a test performed with different orifice diameters. The test was done at a collection distance of 12 cm, with the spinneret rotating at 10 000 rpm. The orifice diameters were 0,3 mm and 0,6 mm. Different concentrations of spinning fluid were used: 17,5 w%, 20 w% and 22,5 w%.

The test results show that the diameter of the orifice has an impact on the thickness of the fibers. On average, the fibers are thicker when produced with the larger orifice. Increasing the concentration of the spinning fluid has a more significant effect on the average fiber thickness. Higher spinning fluid concentrations generally result in thicker fibers.

The standard deviation on the average fiber thickness is higher for higher concentrations of spinning fluid. Both thick and thin fibers are identified on the pictures from the microscope. The bimodal fiber thickness (see section 8.1) is also present in this test, and the orifice diameter does not seem to have an influence on the bimodal distribution.



Graph 2: Results of fiber diameter from different orifice diameters for different concentrations

5. OUTLOOK

In this section, an outlook is provided for technology regarding fiber spinning in general and for relevant testing. As described in the Literature chapter (section 2), centrifugal fiber spinning is a very promising technology. A lot of potential applications have been identified, but the technology needs to become more mainstream to make the applications economically viable.

5.1. Bimodal fiber diameter distribution

Test results show a strange pattern emerging in the distribution of the fiber diameter. This phenomenon is identified in both the analysis of the data and in the pictures made via the microscope. Test results show that both thick and thin fibers are produced within one spinning process. Figure 33 illustrates this result. Some hypotheses might explain why the bimodal fiber production happens. These are explained below.

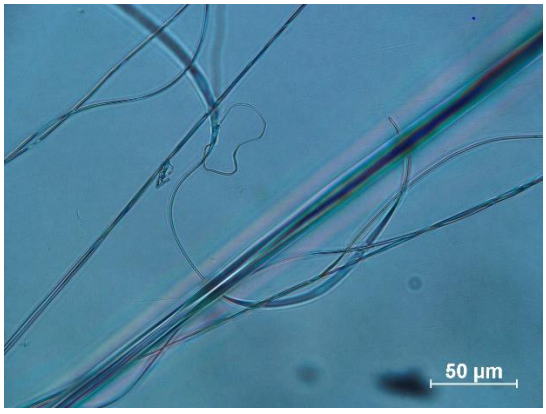


Figure 33: Example of a Bimodal fiber distribution during testing

5.1.1. Hypothesis 1: Slip

As the fluidized polymer moves through the nozzle of the spinneret, friction will slow the fluid down around the edges of the tube of the nozzle. This causes the center of the moving fluid to be slightly ahead of the fluid on the edges of the tube because it does not experience any friction from the wall. The fluid, that is slightly ahead, looks like the beginning of the formation of a droplet. Once the fluid reaches the edge of the nozzle, the start of the droplet will be the start of the fiber. Since the start of the fiber is smaller than the orifice diameter, it leads to thinner fibers.

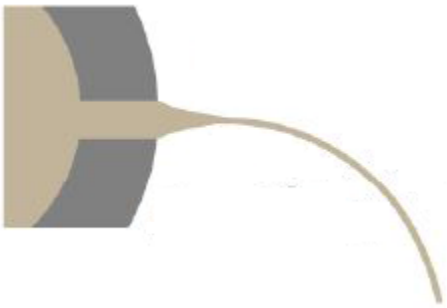


Figure 34: Visual representation of a fiber starting from a small diameter [3, p. 9]

However, if there is slip at the walls, the friction at the edges of the nozzle will be less. In this case, the polymer that comes out of the nozzle has the same size as the orifice diameter, leading to thicker fibers at the collection posts.

Figure 35: Visual representation of slip [3, p. 9]

Fibers with these two thicknesses are collected and show the bimodal fiber diameters seen in the test results (see Figure 33).

5.1.2. Hypothesis 2: Die swell

In the industry, die swell is a phenomenon that occurs in the injection molding process. Non-Newtonian fluid rheology is the explanation behind this technology. Die swell happens when aligned polymer chains curl up with entropy as the driving force behind this process.

When fluidized polymer is forced through a tube, the polymer chains along the sides of the tube align due to friction, while the chains in the center remain curly. When the friction and pressure in the tube stop at the orifice, the aligned fibers curl back up causing an expansion in volume. This means that the diameter of the fiber is larger than the orifice diameter once the polymer exits the nozzle.

5.1.3. Hypothesis 3: Stretching of thicker fibers

Because thicker fibers have more mass, the acceleration towards the collection posts is higher. If a thicker fiber is ejected from the spinneret, the centrifugal force will act upon that fiber. This will pull the thicker fiber faster towards the collection posts than a thin fiber. The thicker fiber will then stretch itself to a thin fiber due to the continuous rotation of the spinneret. This results in a thick fiber in the beginning and a thin fiber towards the end. If a thicker fiber will be ejected during the spinning process, a bimodal fiber diameter will be noticed when characterizing the spun fibers.

5.1.4. Testing the hypotheses

In order to understand in further detail which of the above hypotheses is correct, tests must be done with version 2 of the spinneret, which has Teflon caps. Teflon is inherently smooth and has a low coefficient of friction.

Spinning fibers with the Teflon caps will yield different results following the above described hypotheses.

In case of hypothesis 1, the smooth nature of the tube from the nozzle will result in more thicker fibers because the slip will happen more frequently due to the lowered friction with the edges of the tube. Because the friction is lower, the droplet will not start to form, resulting in more thicker fibers.

In case of hypothesis 2, the resulting fiber diameter should be smaller. Due to the lower friction, the Teflon will not align the fibers along the edge of the tube. Therefore, the ejected polymer from the spinneret will have the same diameter as the orifice. The produced fiber will have a smaller diameter from the start, lowering the overall diameter of the collected fiber. This will result in the collection of smaller fibers.

Hypothesis 3 describes the formation of fibers after the ejection, whereas the two other hypotheses relate to the formation if fibers during the time spent in the nozzle. It is difficult to draw conclusions regarding this hypothesis, since the rotation speed is an important variable. The higher the rotation

speed, the lower the diameter of the fiber. Thicker fibers may be produced at the beginning of the fiber spinning process and these can be stretched as the fiber spinning process progresses. The lifting mechanism is a key factor in the determination as the fiber thickness lowers towards the end of the process. Great care needs to be taken when collecting the fibers.

5.2. Spinneret version 2

Further testing needs to be done with the second version of the spinneret. The added path length of the nozzle will yield different results compared to version 1. This may lead to an increased or reduced bimodal distribution of fiber diameters in test results, helping to explain how and why this phenomenon occurs with the aid of the above described hypotheses.

6. CONCLUSION

Preliminary testing with the new centrifuge reveals that the setup is working as it was designed and intended. It will offer a good platform for further research about centrifugal fiber spinning. No signs of wear or malfunctioning of the machine have been observed in the testing phase. Expectation is that the machine is durable and that it will sustain a lot of testing cycles in the years to come.

Testing with the Teflon tips, integrated in version 2 of the spinneret, will reveal the reason behind the bimodal fiber distribution that was observed. It will become one of the first studies to explain why the phenomenon of bimodal fiber distribution occurs in the centrifugal fiber spinning setup.

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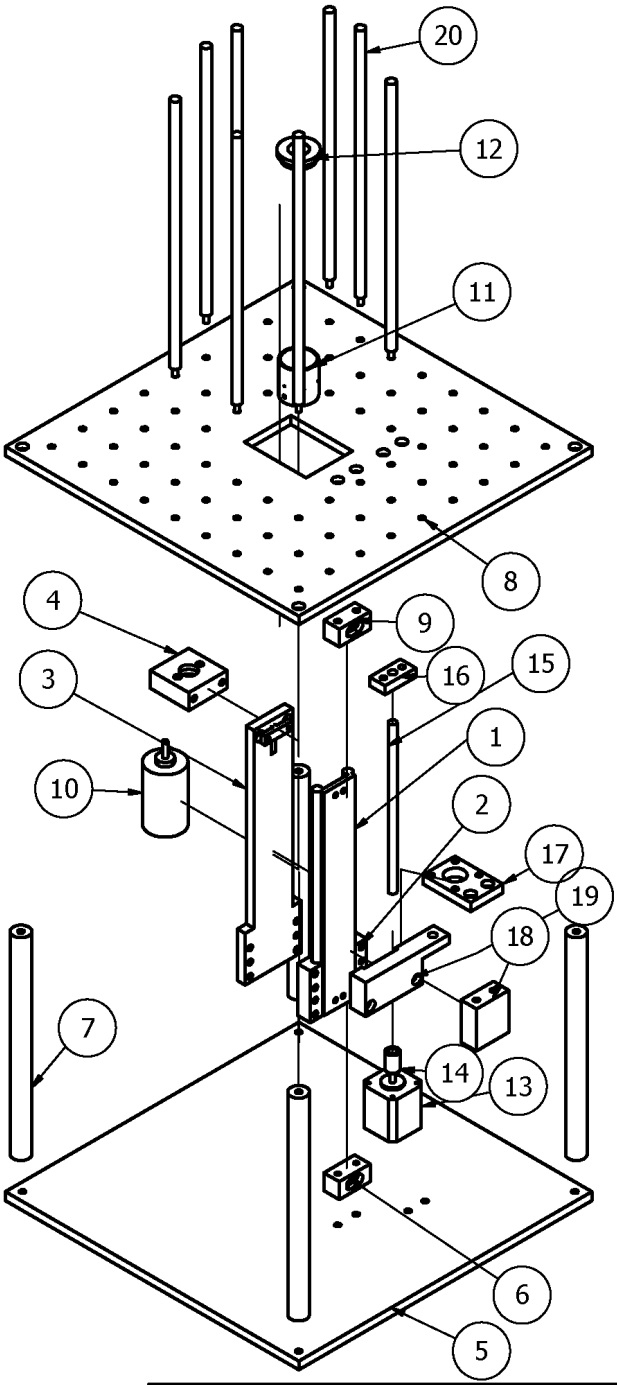
APPENDIX LIST

APPENDIX A: BUILD PLANS



PARTS LIST		
ITEM	QTY	PART NUMBER
1	1	Guide Rail
2	1	Motor Guide
3	1	Motor Holder
4	1	Motor Holder Top
5	1	Bottom Plate
6	1	Guide Rail Holder Bottom
7	4	Standoffs
8	1	Top Plate
9	1	Guide Rail Holder Top
10	1	DC Motor
11	1	Cup
12	1	Lid
13	1	Stepper Motor
14	1	Threaded Rod Sleeve
15	1	Threaded Rod
16	1	Threaded Rod Holder
17	1	Stepper Motor Holder Top
18	1	Stepper Motor Holder Bottom
19	1	Lifting Arm
20	10	Collection Posts

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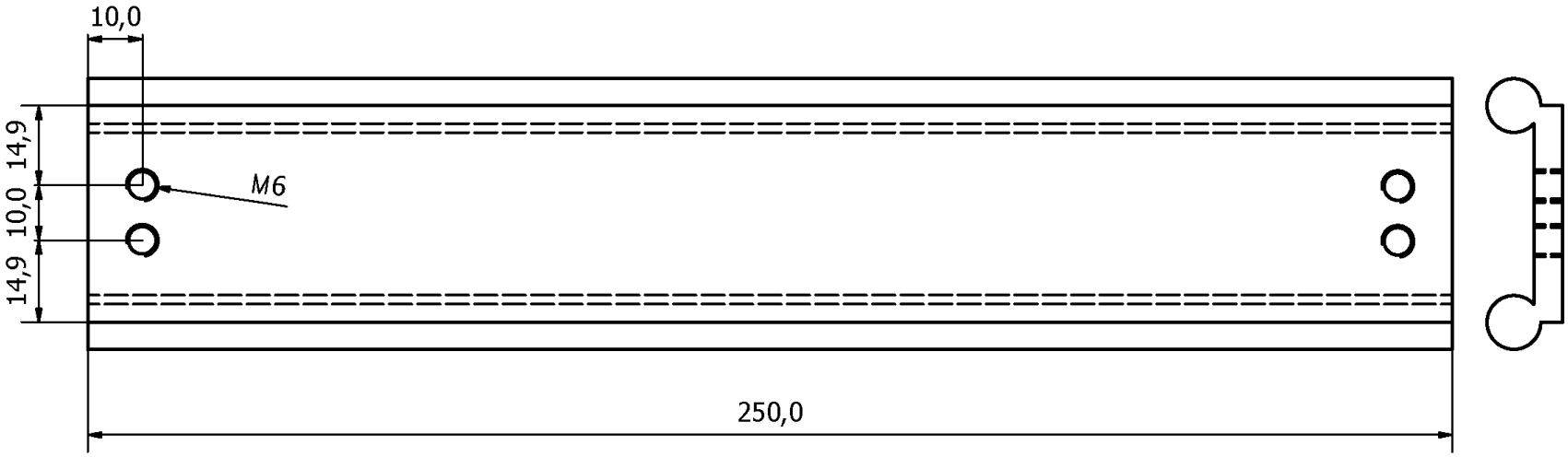
Exploded View Centrifuge

A4 thesis tekeningen | Edition | Sheet 1 / 19



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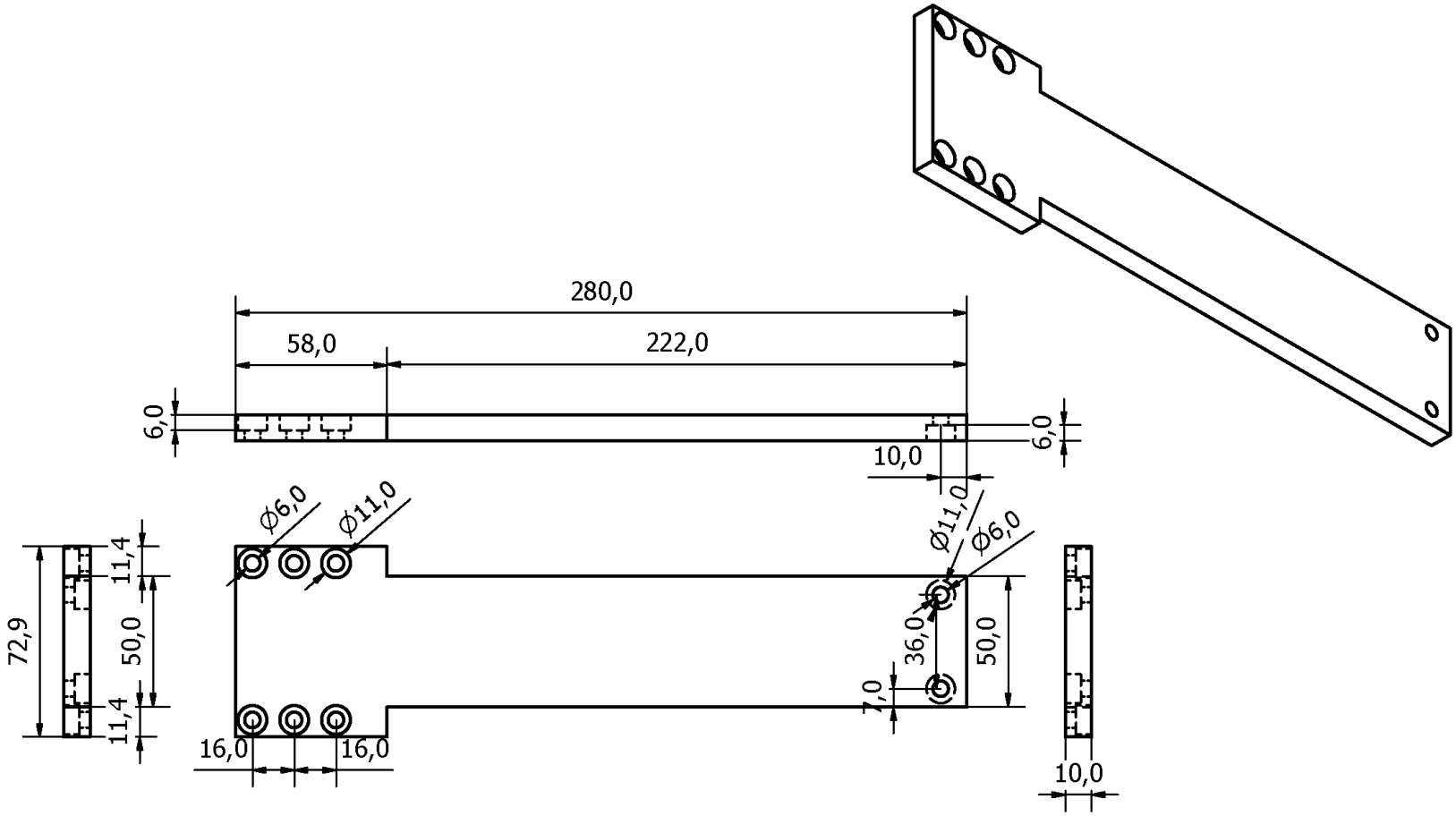

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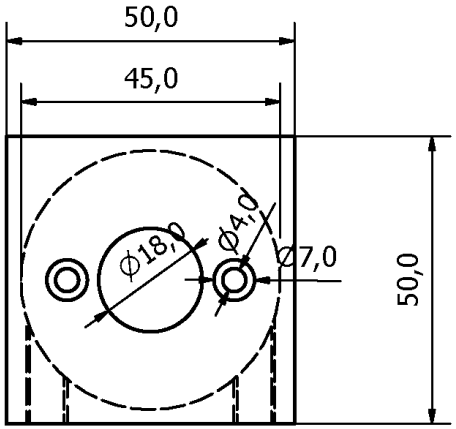

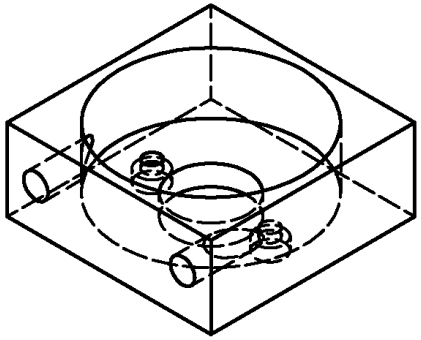

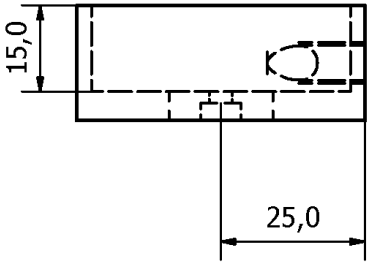

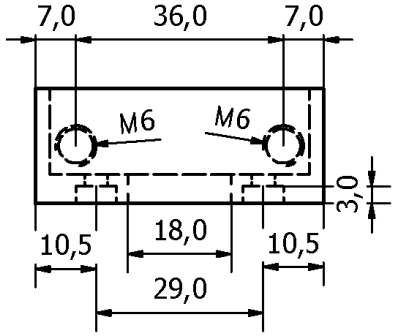

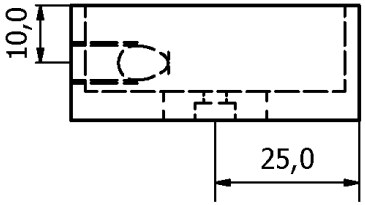

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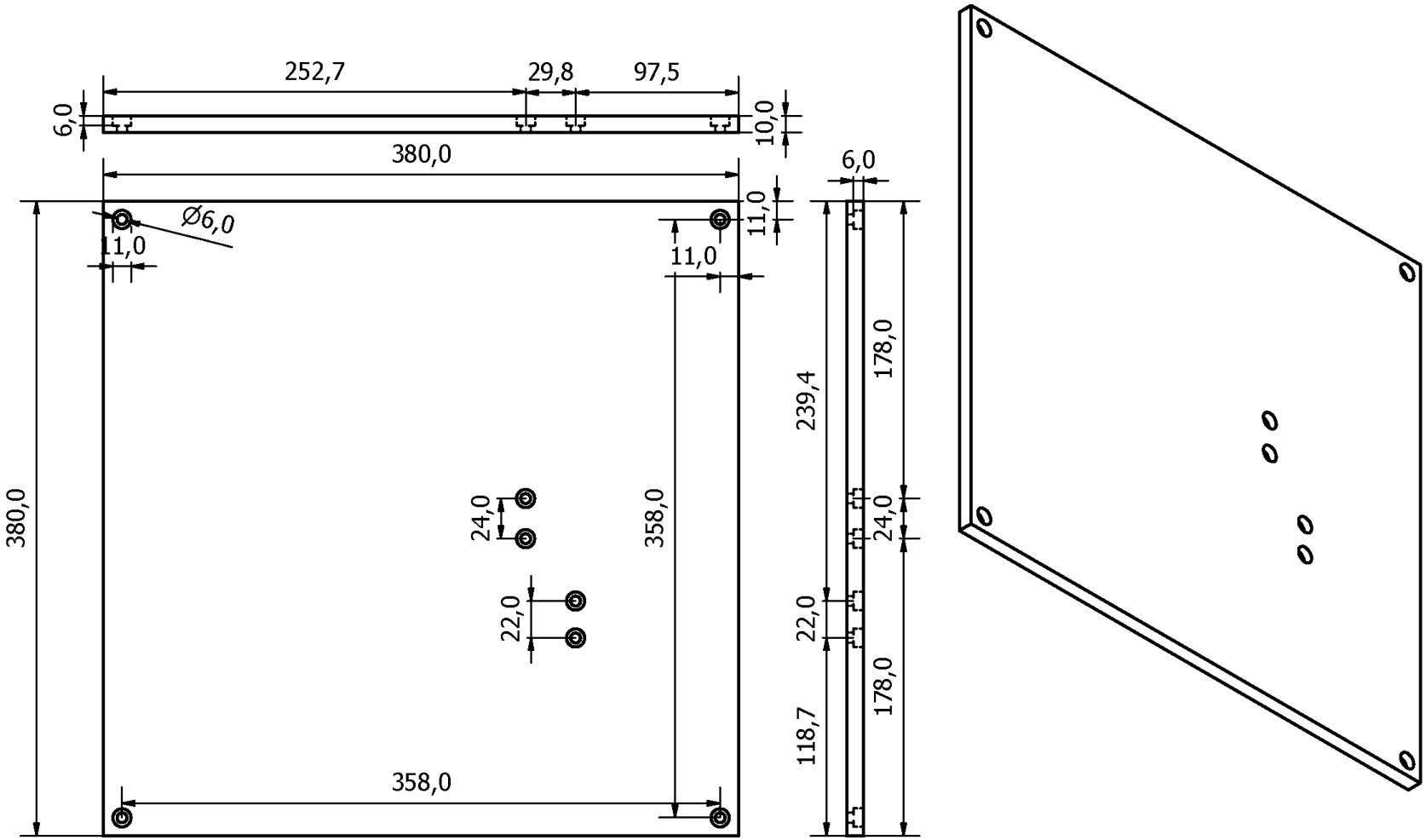

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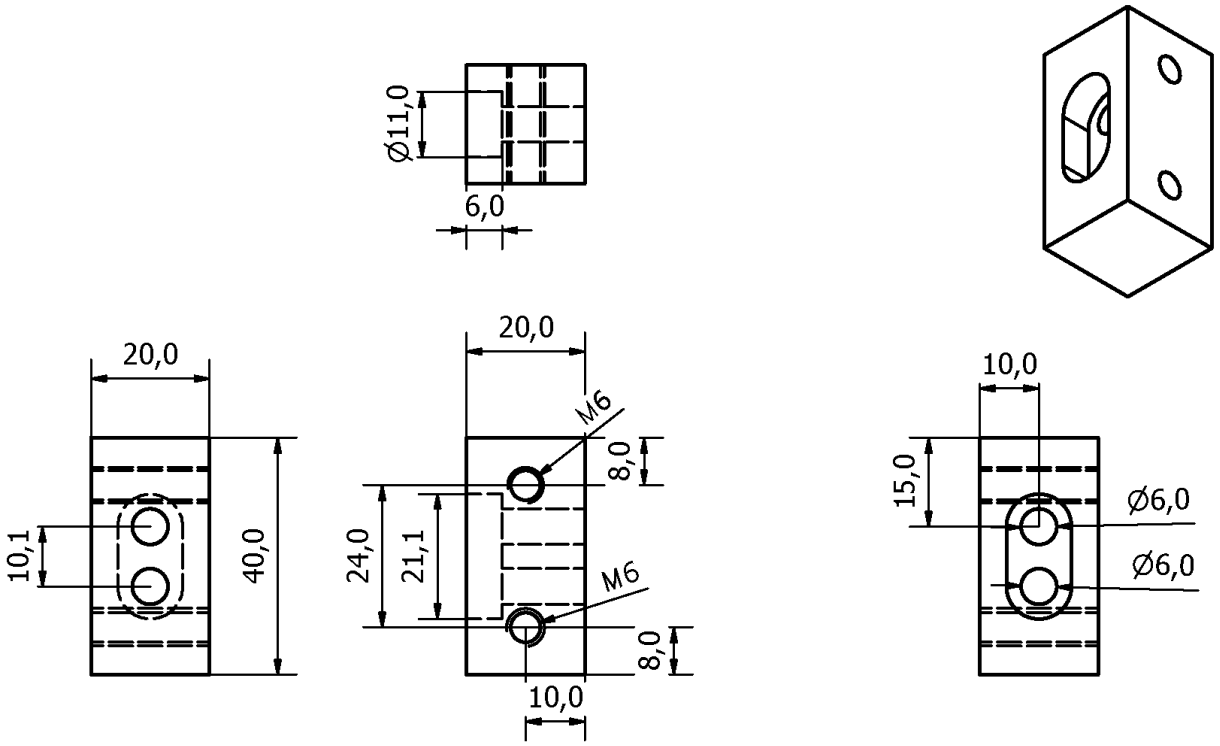

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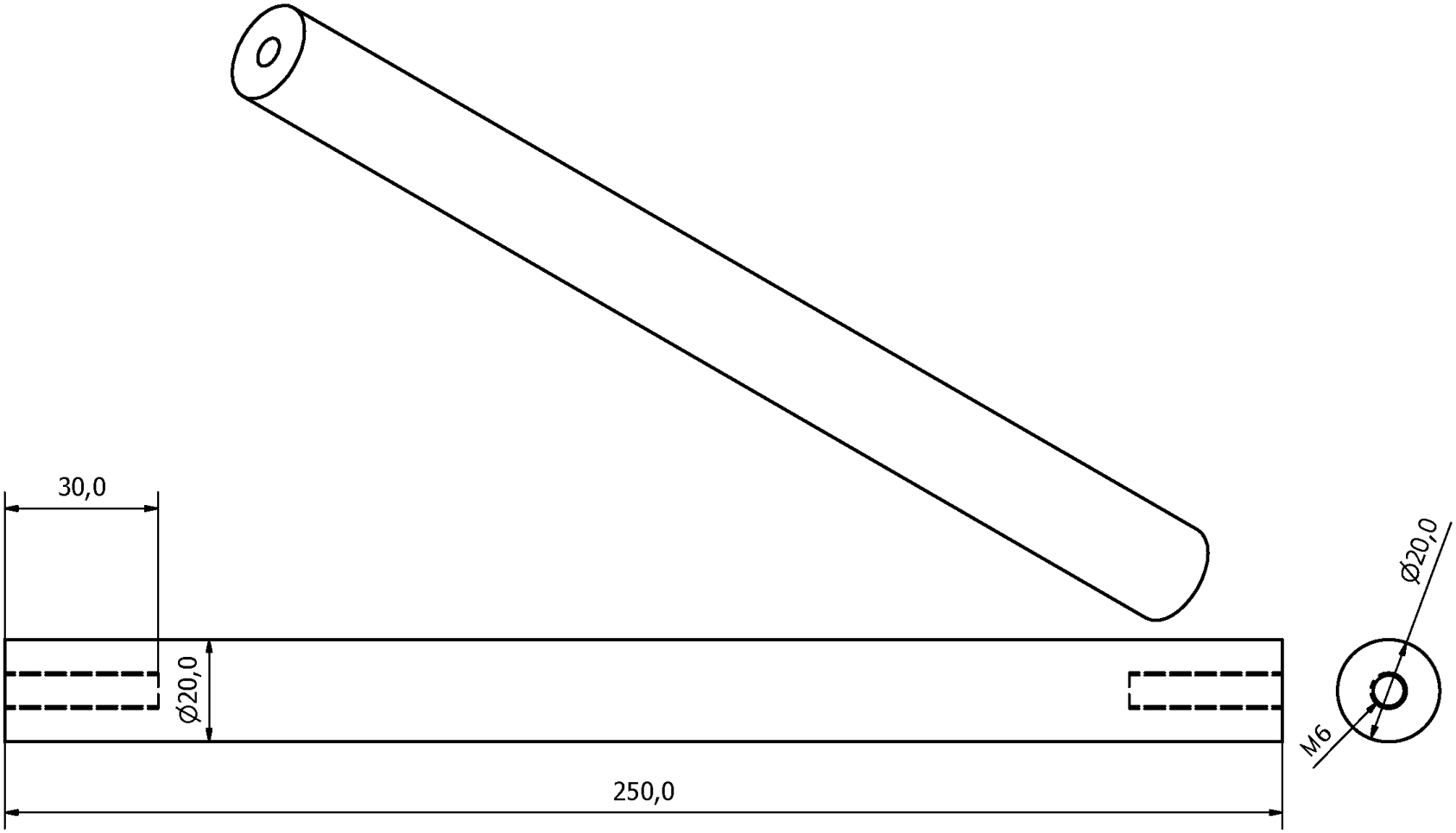

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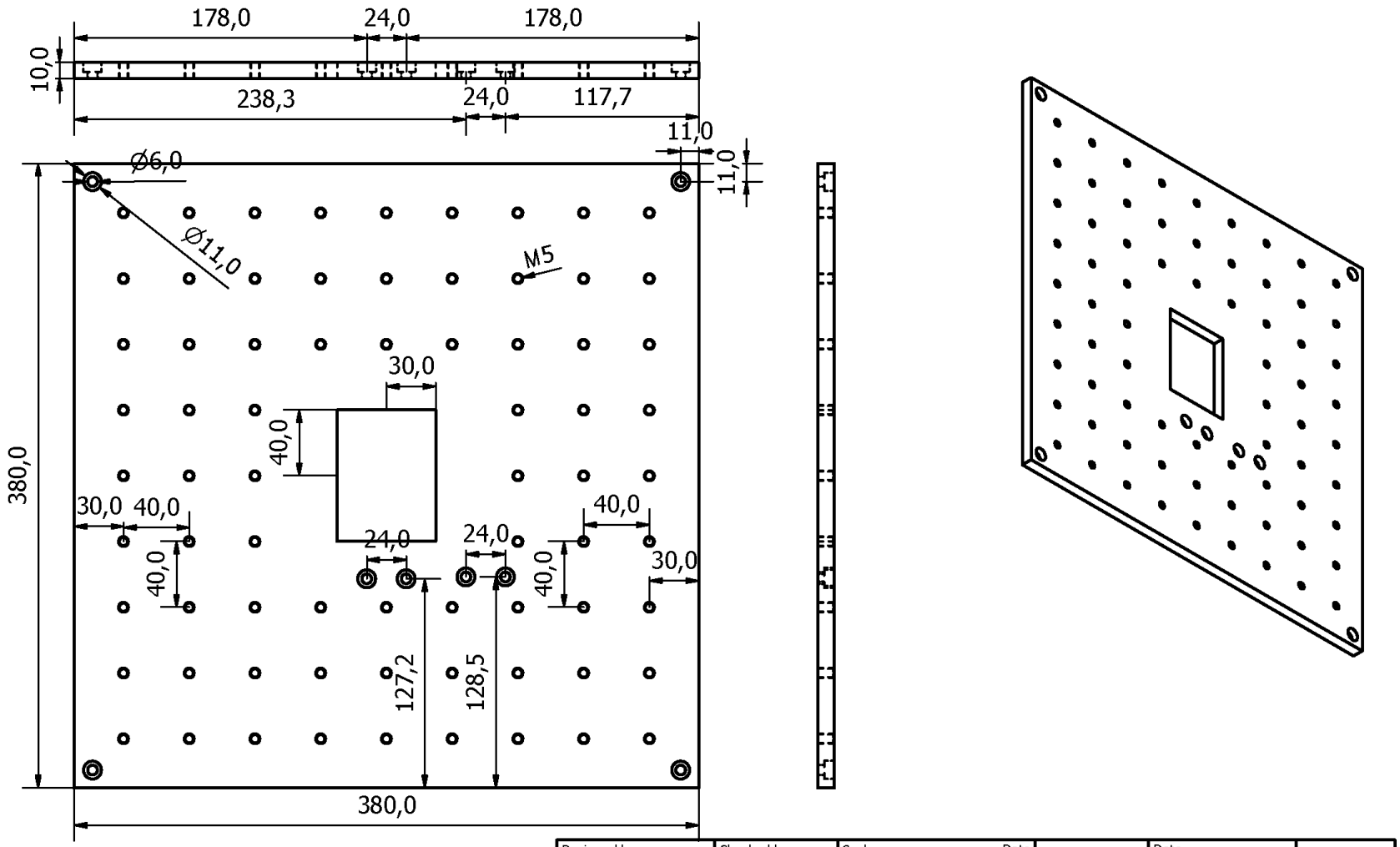

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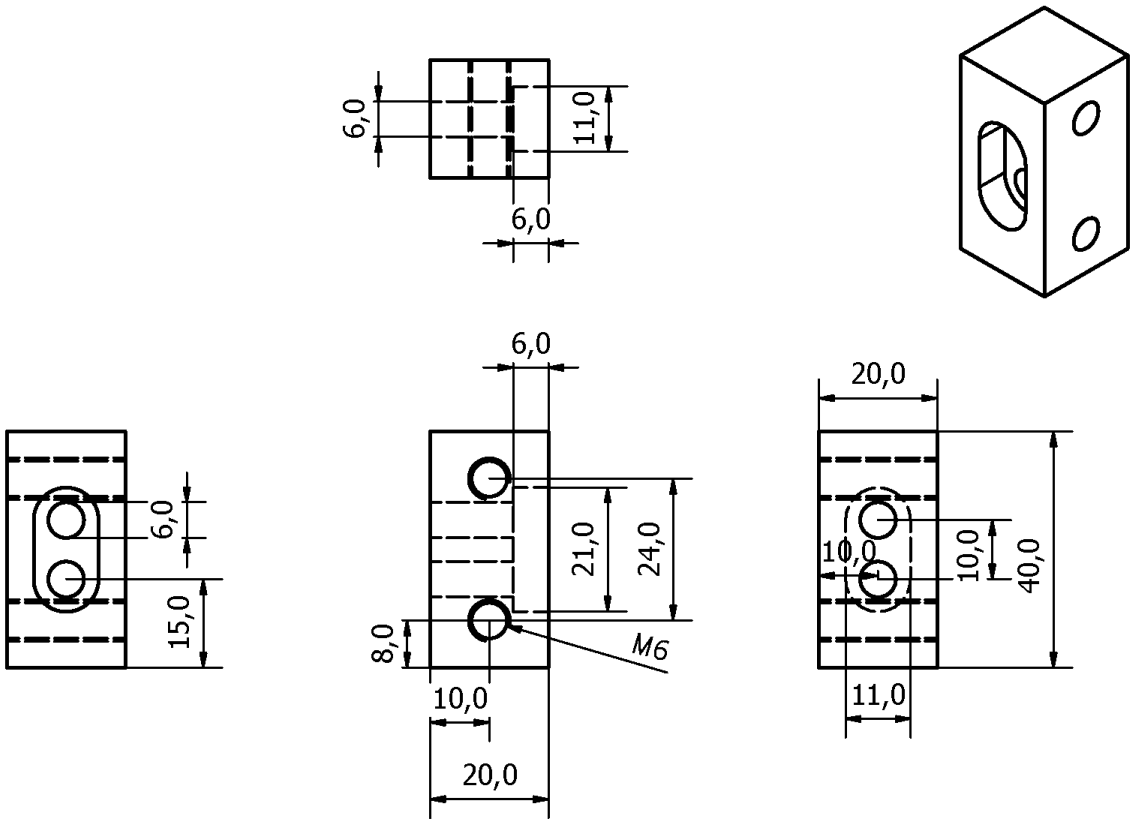

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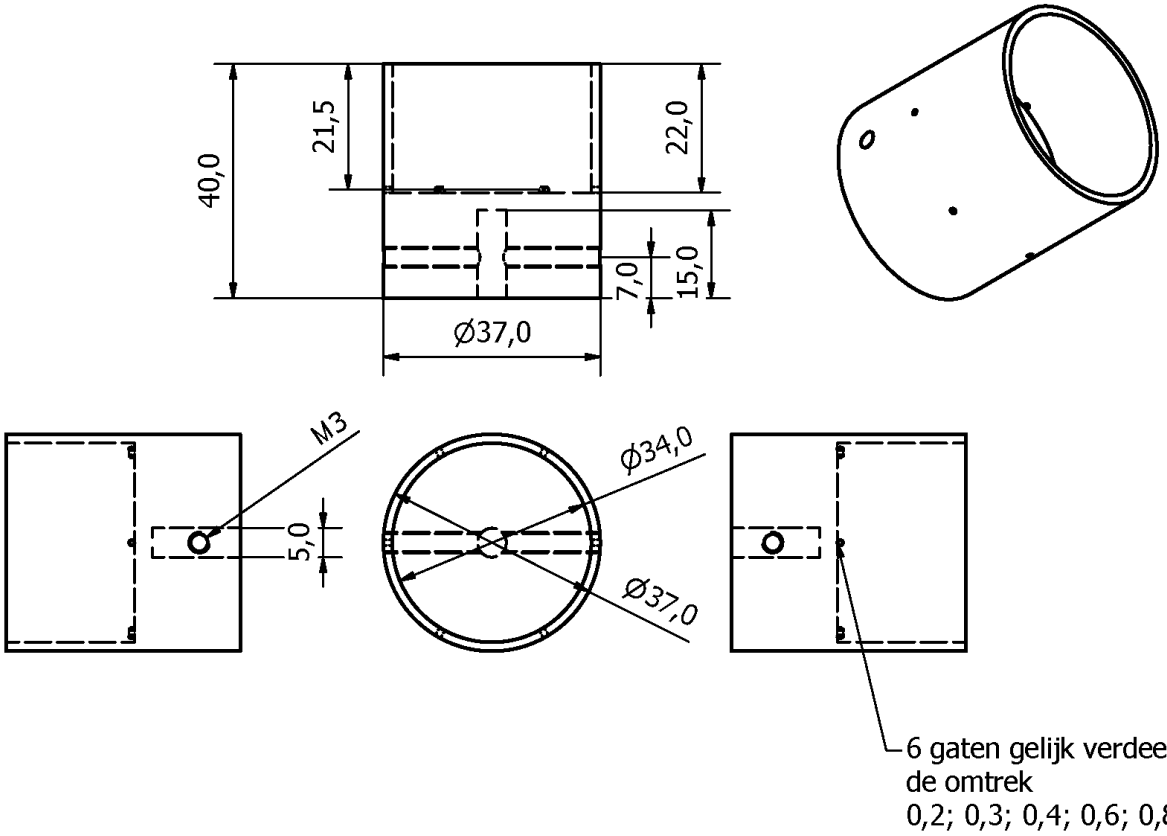

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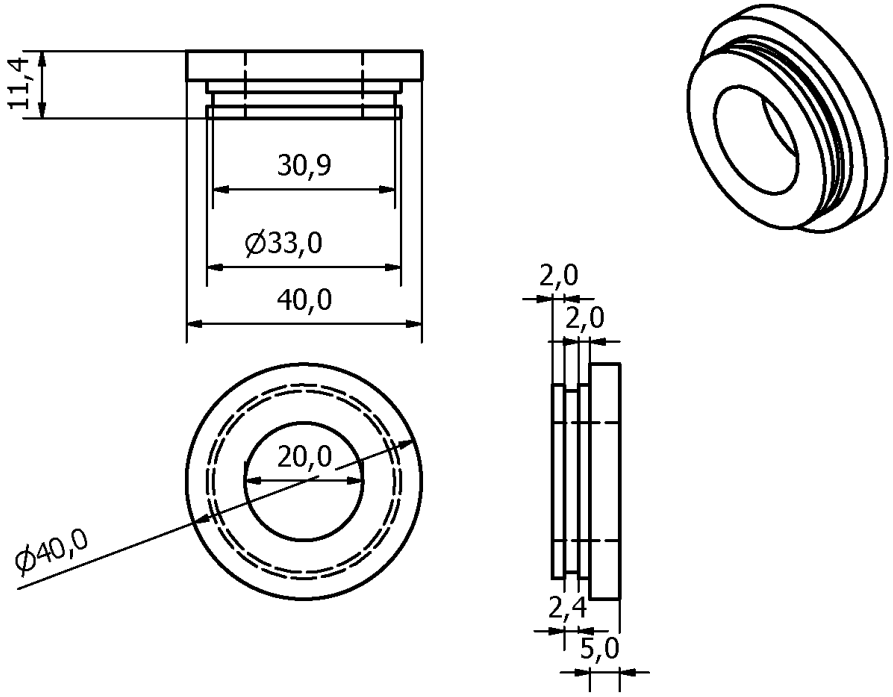

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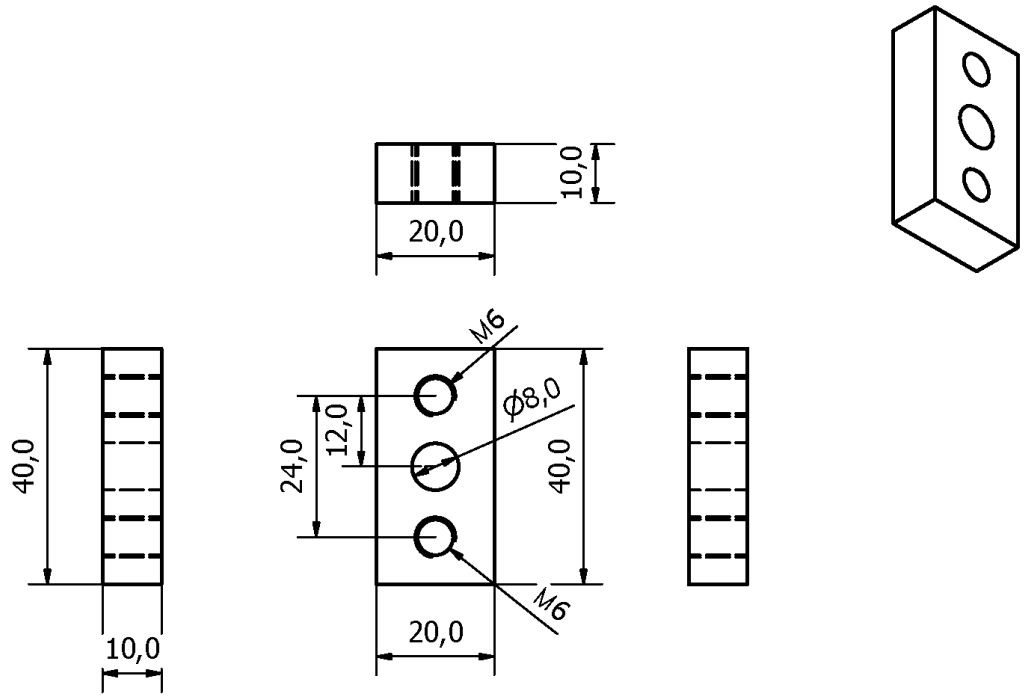

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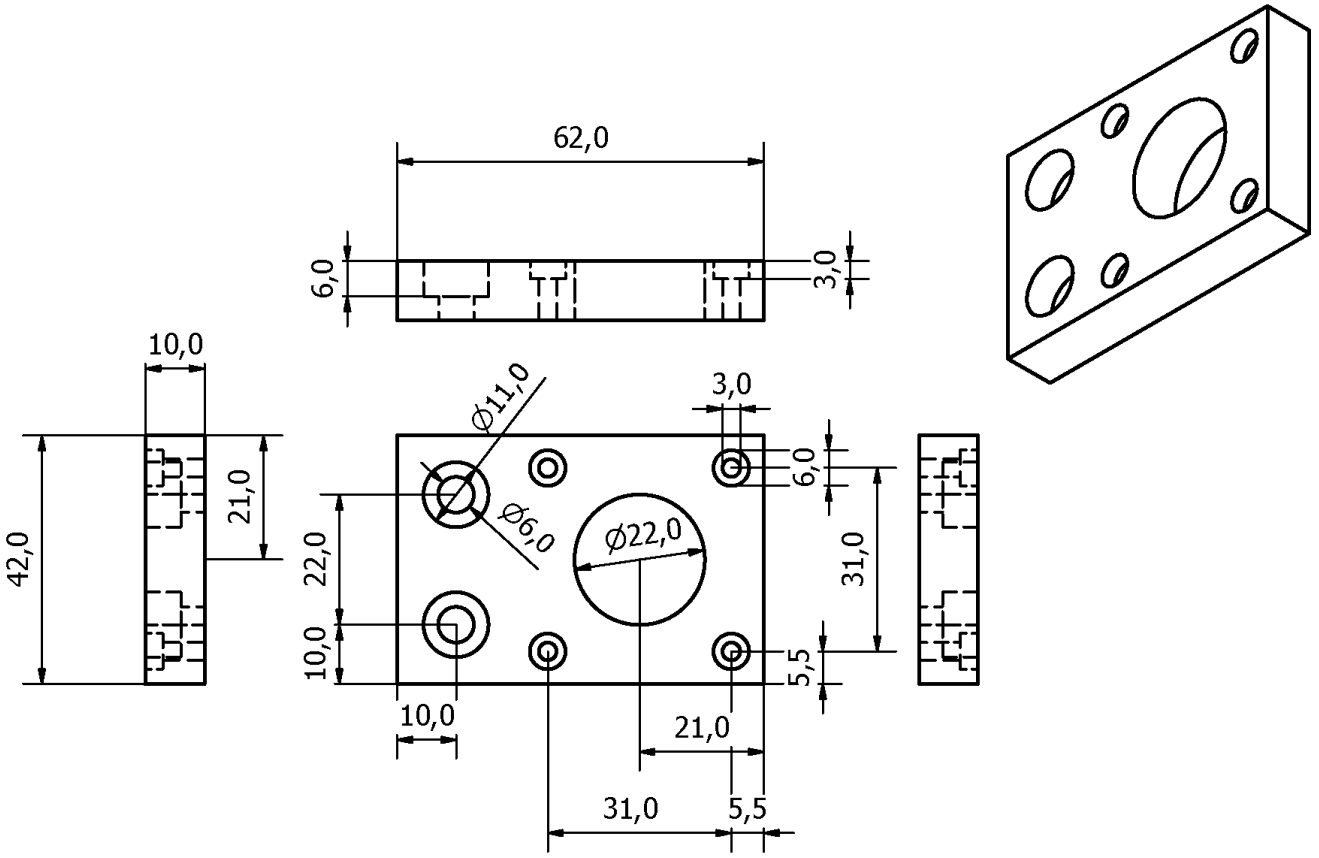

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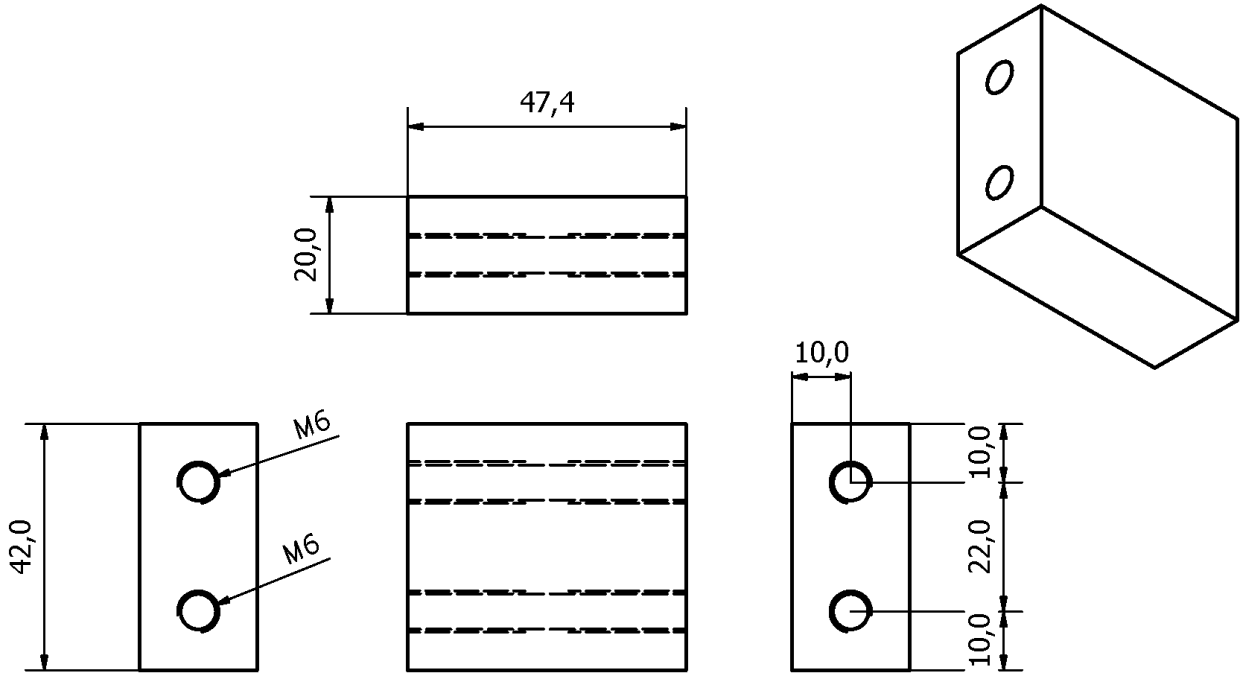

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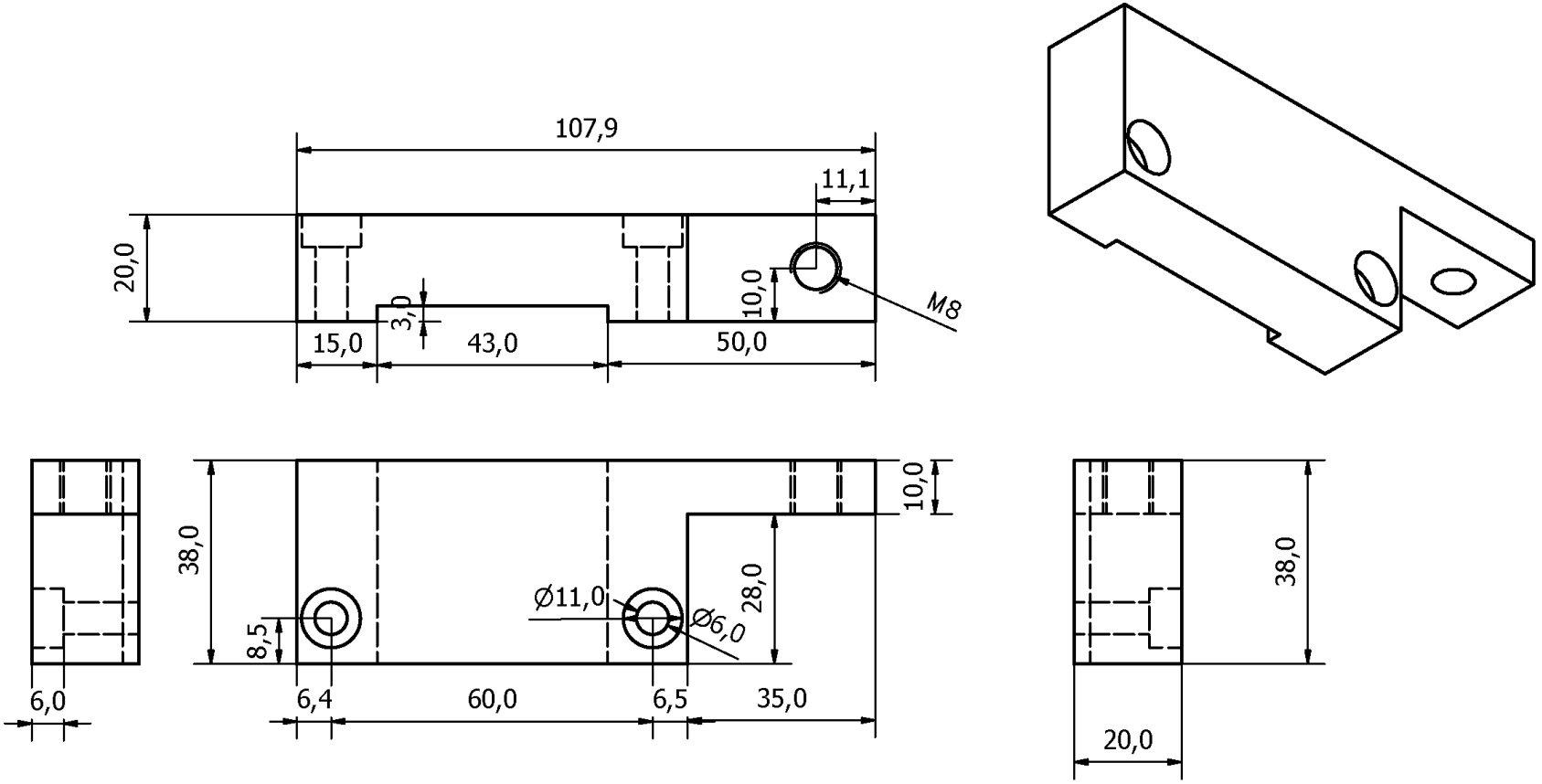

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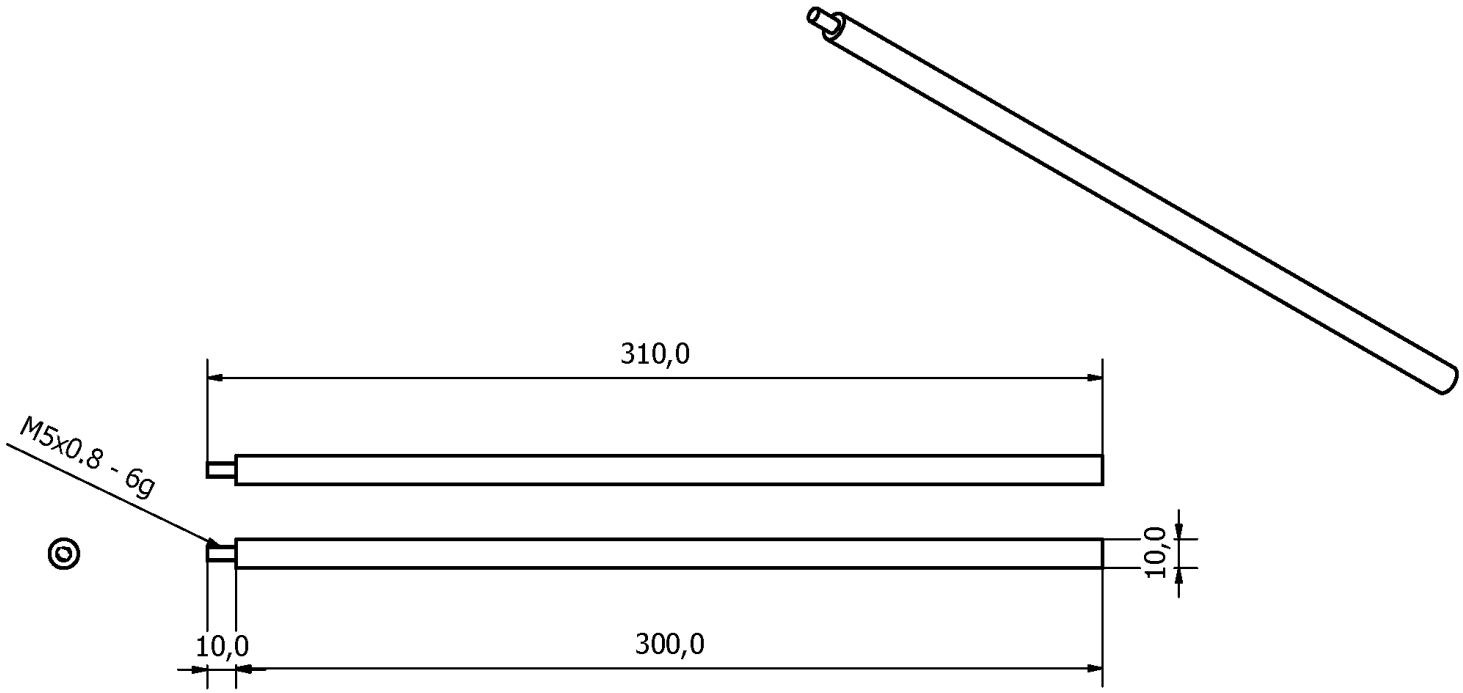

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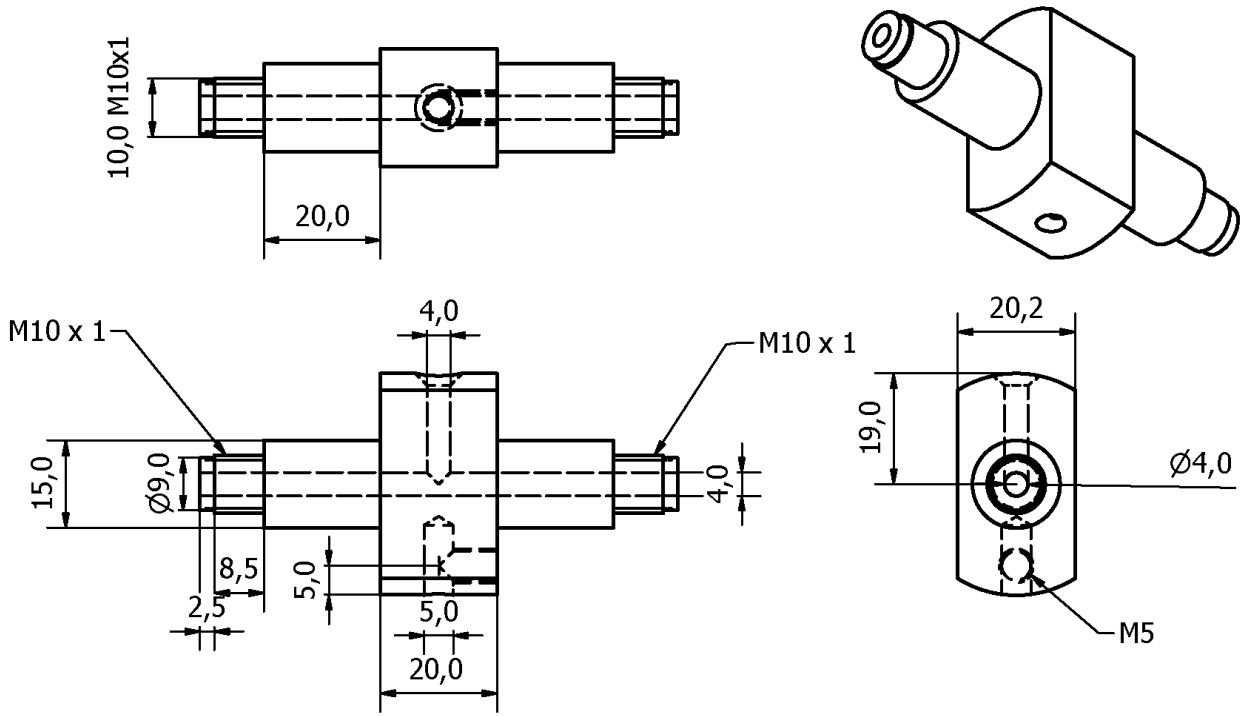

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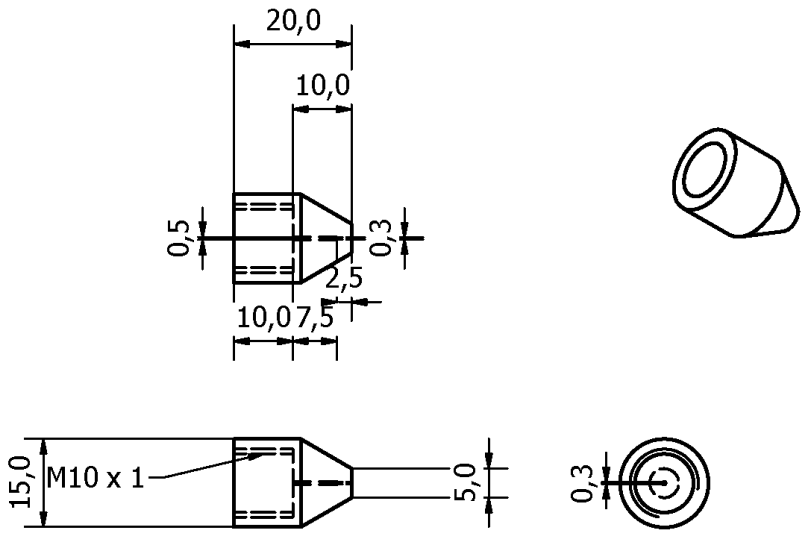

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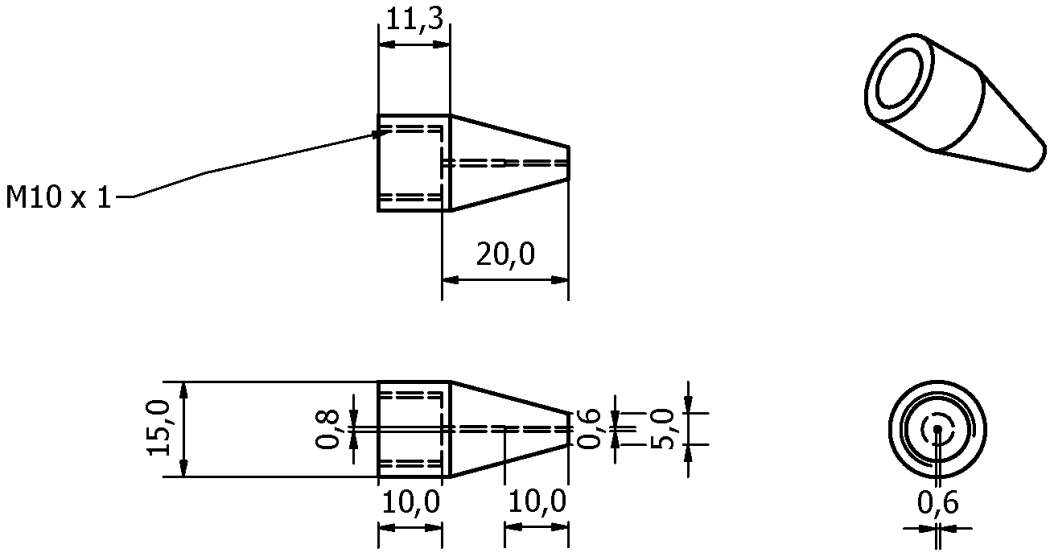

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				A4 thesis tekeningen	Edition	Sheet 19 / 19

APPENDX B: ARDUINO CODE

```
#include <LiquidCrystal_I2C.h>

#include <Wire.h>

LiquidCrystal_I2C lcd(0x27, 2, 1, 0, 4 ,5, 6, 7, 3, POSITIVE);

//Potentiometer
const int analogInPin = A0;
//Analog input pin that the potentiometer is attached to

const int analogOutPin = 9; //Analog output pin that the LED is attached to

int sensorValue = 0;        // value read from the pot

double motorpercentage = 0; //Declaring variable

//toerental
float value=0;//Declaring floating variable

float rev=0;//Declaring floating revolution variable

int rpm;//Declaring variable

int oldtime=0;//Declaring time variable

int time;//Declaring time


//stepper
const int stepPin = 4;
//declaring variable and Arduino output/input pin - step pin for stepper
motor

const int dirPin = 5;
//declaring variable and Arduino output/input pin - direction pin for
stepper motor

const int endBeneden = 7;
//declaring variable and Arduino output/input pin - end switch bottom

const int endBoven = 6;
//declaring variable and Arduino output/input pin - end switch top

const int startknop=12;
//declaring variable and Arduino output/input pin - starting button

int customDelay,customDelayMapped;
// Defines variables
```

```
void isr()
//interrupt service routine - counts the amount of times IR sensor is
tripped by revolution
{
rev++;
}

void setup()
{
  //lcd +toerental
lcd.begin(16,2);                //initialize LCD
attachInterrupt(0,isr,RISING);  //attaching the interrupt

//defining pinmodes of stepper motor and end switches
pinMode(stepPin,OUTPUT);
  pinMode(dirPin,OUTPUT);
  pinMode(endBeneden,INPUT);
  pinMode(endBoven,INPUT);
  pinMode(startknop,INPUT);

//moving lifting system to top position to start experiment
  while(!digitalRead(endBoven)){
    digitalWrite(dirPin,LOW);
    digitalWrite(stepPin,HIGH);
    delayMicroseconds(750);  //delay steps of stepper motor to control
speed
    digitalWrite(stepPin,LOW);
    delayMicroseconds(750);

  }
}

//rev counter function
void toerenteller(){
  //lcd +toerental
  // read the analog in value of potentiometer:
  sensorValue = analogRead(analogInPin);
  // map it to the range of the analog out:
  // interpolating potentiometer value to analog value
  motorpercentage = map(sensorValue, 0, 1023, 0, 255);


  detachInterrupt(0);           //detaches the interrupting signal from IR
sensor
  time=millis()-oldtime;        //finds the time
  rpm=(rev/time)*60000;         //calculates rpm
  oldtime=millis();             //saves the current time
  rev=0;                        //set rev to zero for next calculation

  //printing text to LCD screen
  lcd.clear();                  //clear LCD screen
  lcd.setCursor(0,0);           //set LCD cursor to top left corner
  lcd.print("RPM= ");           //print word RPM

  lcd.print(     rpm);          //print the rpm value next to rpm
```

```
  lcd.setCursor(0,1);            //set LCD cursor on bottom left corner
  lcd.print("lift speed= ");     //print words lift speed
  lcd.print(round((motorpercentage/255)*100)+5);
//print the speed percentage of motor
  lcd.print(" %  ");             // print % symbol
  attachInterrupt(0,isr,RISING);
// the interupt of the IR signal is done on rising signal

  delay(1000);                   // delay of 1sec, readout every second
}

void loop(){
  if(!digitalRead(startknop)){
//only executes the revcounter program when start button is off
    toerenteller();
    digitalWrite(dirPin,LOW);
    digitalWrite(stepPin,LOW);

  }

  if(digitalRead(startknop)){
          while (!digitalRead(endBeneden ) && digitalRead(startknop)) {
//execute when bottom switch isn't pressed and start button is in run
position
            int customDelay = analogRead(A0); // Reads the potentiometer
            int newCustom = map(customDelay, 0, 1023, 800,400);
// Converts the read values of the potentiometer from 0 to 1023 into
desired delay values (300 to 4000)

            digitalWrite(dirPin,HIGH);
//certain spinning direction of the stepper motor

             digitalWrite(stepPin,HIGH);
//initiate step
            delayMicroseconds(newCustom);
//new custom = custom speed provided by potentiometer

            digitalWrite(stepPin,LOW);
// end step
            delayMicroseconds(newCustom);

                  if(digitalRead(endBeneden)){                          //
break if loop when lifiting system reaches the bottom
                    break;
                  }
            }

            delay(800); // delay at change of direction (going up or down)

            while (!digitalRead(endBoven )&& digitalRead(startknop)) {
             int customDelay = analogRead(A0); // Reads the potentiometer
              int newCustom = map(customDelay, 0, 1023, 800,400); //
Converts the read values of the potentiometer from 0 to 1023 into desired
delay values (300 to 4000)
             digitalWrite(dirPin, LOW);
             digitalWrite(stepPin,HIGH);
```

```
      delayMicroseconds(newCustom);
      digitalWrite(stepPin,LOW);
      delayMicroseconds(newCustom);


      if(digitalRead(endBoven)){
        break;
      }
     }
    delay(800);// delay at change of direction (going up or down)

  }
}
```