



ADHESIVES

A study of adhesives and their creation

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2 Preface

I have written this paper as my Matura project during my time at the Kantonsschule Zug from May 2021 to February 2022. The topics I have selected are the synthesis and in-depth analysis of adhesives.

My father, who is working in the cement business in Iran, became interested in expanding and buying a daughter firm of the well-known Swiss company Sika, which, among other fields, concerns itself with adhesives. Him telling me about it sparked my interest in the topic, leading me to learning more about adhesives. What I found most fascinating was the potential of adhesives, outperforming other bonding methods and still having lots of room to improve, with seemingly infinite new products being released.

A few months later I asked Mr. Landis to supervise my Matura project, since I was very interested in his chemistry lessons and contemplated studying it in university. He accepted and himself suggested adhesives among other topics. This reminded me of my previous interest and eventually motivated me to write about them and create some of them myself.

I thank everyone who helped me on my path to completing this paper: Urs Leisinger and Vivien Hofstetter for taking their time to read through and correct it, Norbert Spaar for supporting and helping me with some of my testing methods at the start, as well as providing me with space in the gymnasium's workshop. I would also like to thank my parents Irina and Nicolas Mathys, my sister Anastasia Mathys and her partner and friend of mine Kirill Volynskiy for reading and correcting my paper, as well as providing criticism to help me attain a satisfactory end product. Lastly, I express my gratitude to Heinrich Landis for agreeing to be my supervisor and providing the workspace in the laboratory, as well as for his ideas and his explanations to problems that occurred.

3 Abstract

The focus of this Matura paper was to understand the way adhesives work, as well as synthesize and analyze them.

During this project, different types of adhesives were synthesized, such as dispersion adhesives, all-purpose adhesives, hotmelt adhesives, and chemically curing adhesives. Commercially available adhesives, such as an all-purpose adhesive as well as a super glue, were also tested and then compared to the synthesized ones. To measure the adhesive force, the adhered parts were pulled apart with a 50 N dynamometer. The synthesized adhesives were also compared, and conclusions were drawn in combination with the written theory.

Starting off the research, a store-bought all-purpose adhesive was tested on its adhesive strength on different materials. These materials were wood, paper, aluminum, and polyamide. The adhesive was applied to the parts to be adhered and they were pulled apart using a dynamometer. This same method was used for various adhesives, some of which were synthesized for this project, while others were commercially available adhesives. Multiple all-purpose adhesives and a hotmelt adhesive were created. Store-bought super glue was tested as well, which was the only store-bought adhesive, besides the first all-purpose one.

The experimental data presented in chapter 5 supported, that different physically setting adhesives generally followed the same trend: the rougher surfaces adhered better than smooth ones. This is due to higher adhesion areas associated with rough surfaces. The setting time showed a significant effect on strength: The longer the adhesive was setting, the stronger would it hold, eventually reaching a maximal value. The adhesive strength over time was of logarithmic behavior. It was also observed that the higher the absorbance of the adhered material is, the faster does the adhesive set. Therefore, if the strength of adhesives in a short period of time is measured, absorbent materials like paper and wood will generally result in stronger adhesion.

In the final step of the research, it was attempted to synthesize a chemically curing adhesive. However, the lack of appropriate equipment to hold a constant influx of humidity made this impossible. Thus, instead of a truly chemically curing adhesive, a quickly adhering physically setting adhesive was synthesized with Gum Arabic and glycerin. The synthesized adhesive was however inferior to the chemically curing adhesive as its adhesive strength was approximately 10 times weaker on every tested material.

4 Introduction and Theory

In this section, it is explained what adhesives are and it is crucial for the understanding of the whole paper. This chapter includes the definition of adhesives, information about their functionality and it also goes into detail about the different types of adhesives, as well as their fields of use and advantages to other bonding methods.

4.1 Definitions

Per definition by DIN EN 923 “an adhesive is a non-metallic material, which can connect separate joining parts through surface adhesion and inner cohesion”.^[1] Adhesion is figuratively speaking the sticking effect between two parts to be adhered.

Cohesion is the connection and coherence of a single material and could be defined as a special case of adhesion, where the two adhering phases are the same material.^[1]

For convenience, “part to be adhered” will be abbreviated to PTBA over the course of the paper. “Parts to be adhered” will be abbreviated to PTBAs.

4.2 Adhesion

Adhesion is achieved by electrostatic forces, Van-der-Waals forces, dipole-dipole interactions, as well as through chemical bonding. Most commonly, Van-der-Waals forces and dipole-dipole interactions are the main driving forces of adhesion. Adhesive forces and interactions influence not only the adhesion zones, but also the transition zones between the adhesive and the two conjoined parts (Figure 1). Depending on the material, as well as the adhesive itself, the transition zone can be of varying size.^[1]

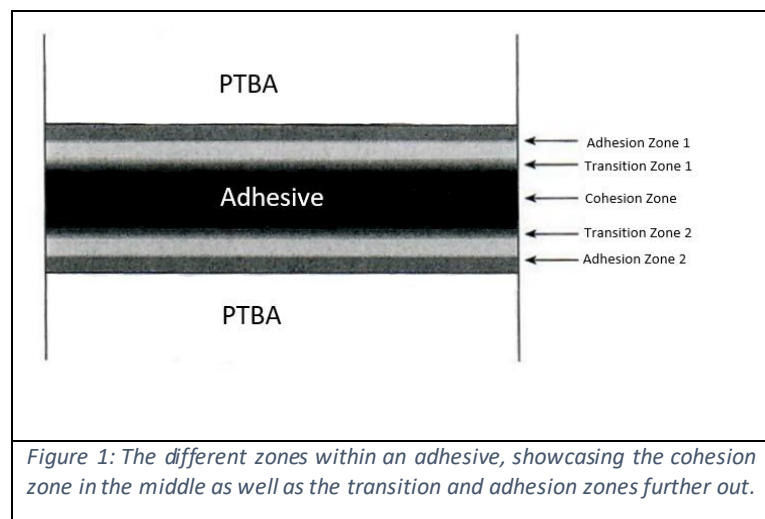


Figure 1: The different zones within an adhesive, showcasing the cohesion zone in the middle as well as the transition and adhesion zones further out.

For adhesion, the following conditions need to be met. Firstly, the distance between the adhesion zone and the PTBA must not exceed 0.5 nm. Additionally, the adhesive needs to be able to cover or moisten the adhesive surface. For this condition to be met, the adhesive’s surface tension may not be larger than the one of the materials to be adhered. The most important factor, however, is the size of the adhesive surface. The larger the adhesive area, the better the materials hold together.^[1]

4.3 Cohesion

Cohesion, like adhesion, consists of chemical bonds or intermolecular forces. The cohesion zone seen in Figure 1 is responsible for the inner strength of an adhesive. The properties in the cohesion zone might differ from the ones in the adhesion zone. They are caused by different intermolecular forces within the adhesive itself (Figure 2). The strings (1) are the polymer chains. At (2), two polymer chains form actual chemical bonds with one another. (3) visualizes the intermolecular forces between four strings which hold them together without a chemical reaction happening. Lastly, (4) shows that the long polymer strings can get tangled up which leads to additional cohesion.

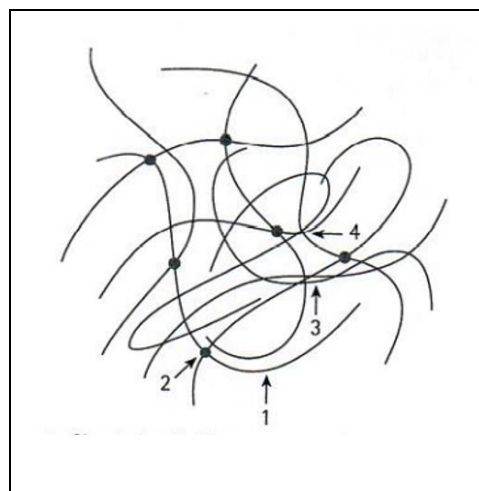


Figure 2: Showcase of the chemical bonds within an adhesive (1), chemical bonds which lead to interlinking of the polymers (2), intermolecular interactions between the molecules of the adhesive (3) and mechanical entanglement of the different molecules of the adhesive within the cohesion zone of the polymer (4).

For optimal cohesion, the adhesive is required to be solid. Thus, if a solvent is involved, it needs to fully evaporate first, before the adhesive has reached its full adhesive strength. When an adhesive is bought, the stated maximal force in Newton per square meter refers to cohesion, because, in comparison to adhesion, cohesion is the only constant value in adhesives. Even if adhesion is stronger than the cohesion in a specific adhesive, the connection breaks once the cohesion fails.^[1]

4.4 Polymerization

There are three types of polymerization. There is cationic, anionic, and radical polymerization. Polymerization is a chain reaction with the first step being a chain carrier initiation. The type of polymerization is determined by the substance which initiates the chain reaction. The initiating substance is commonly part of the synthesized polymer. However, due to its small size in comparison to the polymer, it does not affect its chemical properties in a significant way and thus can be seen as a mild impurity. In the chain reaction the propagated starting material reacts with the unpropagated starting material, thus propagating it, and making the reaction happen for several cycles. This results in the molecule being a series of one repeating unit (Figure 3).^[2]

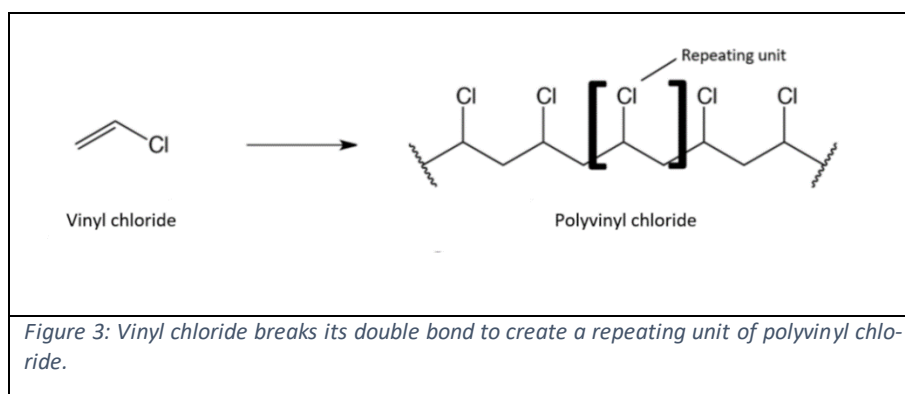
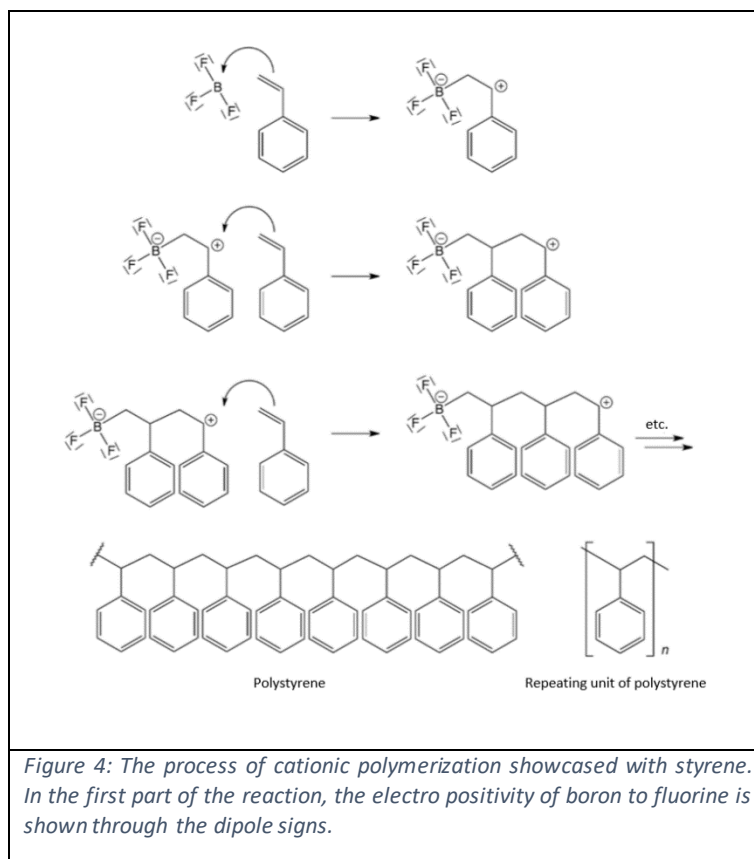


Figure 3: Vinyl chloride breaks its double bond to create a repeating unit of polyvinyl chloride.

4.4.1 Cationic Polymerization

Cationic polymerization needs an electrophile chain initiator. In Figure 4, cationic polymerization of styrene with boron trifluoride as the electrophile is visualized.

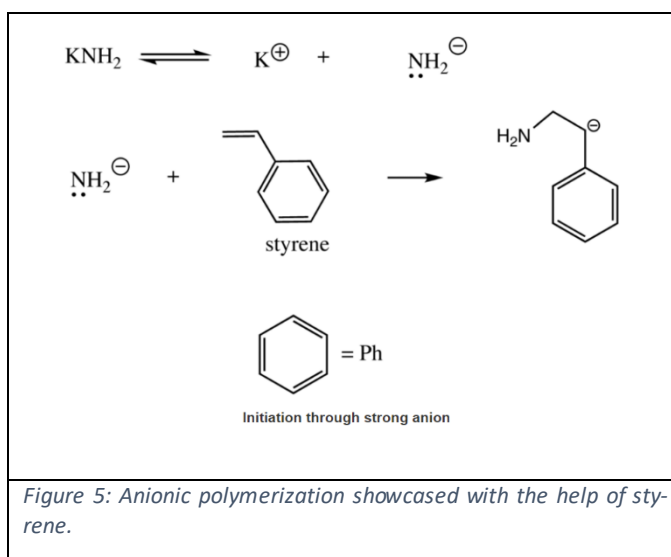
The boron is electrophile whereas styrene's double bond is nucleophile, thus allowing a reaction between them. This leads to the styrene's second carbon atom becoming positively charged, and thus electrophile. This allows the same type of reaction with the positively charged carbon as the electrophile and another styrene's double bond as the nucleophile. This process continues until the resulting polymer connects to another polymer with boron trifluoride at the end, which balances out the electrical charges.



4.4.2 Anionic Polymerization

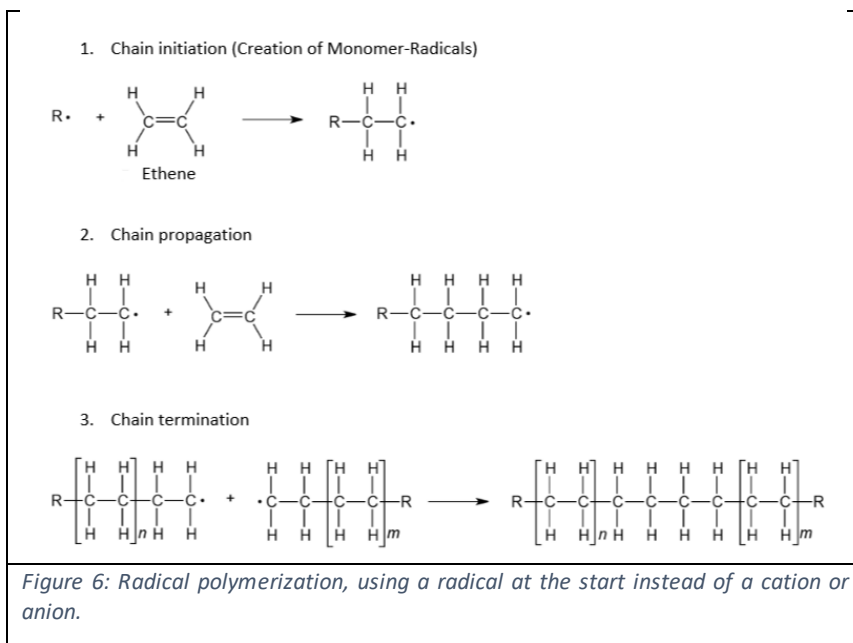
Anionic polymerization is very similar to cationic polymerization, however, it requires a nucleophile, to start the reaction. Figure 5 visualizes an example with styrene as the starting material and azanide as the chain initiator.

The salt KNH_2 is used for this reaction to attain the negatively charged azanide. The rest of the reaction is very similar to the cationic polymerization, except for the double bond now reacting as an electrophile.



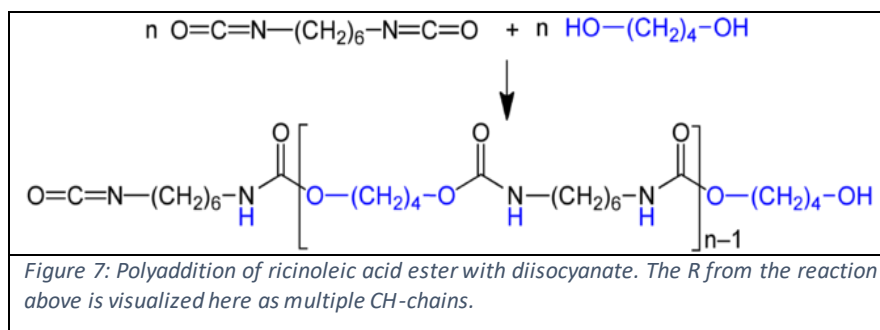
4.4.3 Radical Polymerization

The third type of polymerization is radical polymerization. It is visualized in Figure 6. First, the starting molecule must be a radical, meaning it needs to have an unpaired electron, which makes it highly reactive. The mechanism of the reaction essentially remains the same, yet no charges are involved. In this case the radical acts as the nucleophile and the double bond as the electrophile. During chain initiation an electron from the ethene's double bond connects to the radical, whereas the other electron stays on the other carbon atom. The intermediate is a radical. In chain propagation, the intermediate bonds with another ethene molecule, resulting in a longer chain. This process continues until another radicalized chain of ethene molecules connects to the original chain, equalizing the radicalization of both molecules and creating a polymer chain.



4.5 Polyaddition

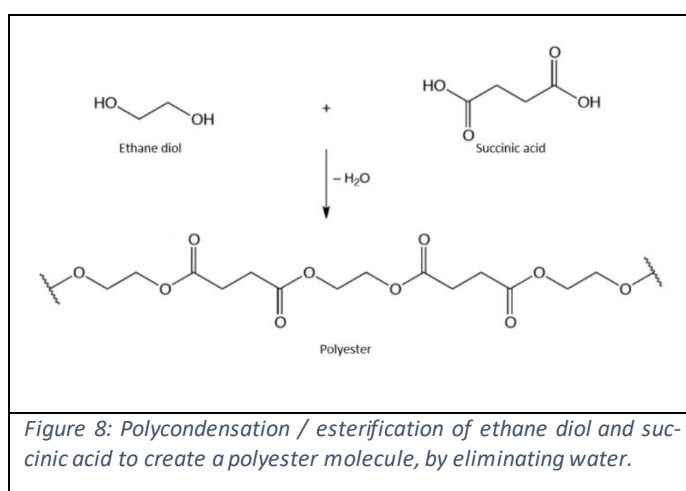
Polyaddition is a form of polymer formation where two molecules of different functional groups are linked with the transfer of protons (H^+) from one group to the other. There are no byproducts in polyaddition. An example of polyaddition is portrayed in Figure 7. [3]



The double bond between the carbon atom and the oxygen atom of diisocyanate bonds with an oxygen atom of ricinoleic acid, which connects the two molecules. In order to equalize the charges, a proton from the previously mentioned oxygen atom of ricinoleic acid connects to a diisocyanate's nitrogen atom.

4.6 Polycondensation

When a polycondensation is taking place, an addition-elimination reaction is taking place. The split off molecule is usually a good leaving group. A condensation, where a carboxylic acid reacts with an alcohol, and water gets eliminated, is called esterification. Figure 8 is an example of this reaction, where ethane-1,2-diol (the alcohol) reacts with succinic acid (the carboxylic acid).



The middle C of the carboxyl group is attacked by the hydroxyl group where in the end a water molecule is eliminated. This reaction occurs multiple times between the polymer and the repeating units. [2]

4.7 Types of Adhesives

There are different types of adhesives. They are grouped by their chemical basis and their solidification mechanism. Most adhesives used today are based on plastics, which will be referred to as polymers in this paper. Adhesives based on these polymers can be divided into two major sections: The physically setting and the chemically curing adhesives. ^[1]

4.7.1 Physically Setting Adhesives

When applying physically setting adhesives, the polymers are already formed. Usually, physically setting adhesives are liquefied before application. There are, however, notable exceptions, such as adhesive films. Nonetheless this section will focus on adhesives that are applied in liquid form. Examples for these kinds of adhesives, are dispersion adhesives, contact adhesives and hotmelt adhesives. The liquid physically setting adhesives are applied to the gluing surface, where the solvent evaporates, leaving only the previously solved adhesive. Another method is to apply a melted adhesive without the use of a solvent and let it cure. ^[1]
[4]

4.7.1.1 Dispersion Adhesives

Dispersion adhesives are a typical example for physical setting adhesives. The polymer particles are dissolved and stay apart through the solvent and surfactants. Surfactants are generally molecules that consist of a hydrophilic and hydrophobic part. In the case of dispersion adhesives, the hydrophilic head points outwards, towards the liquid, whereas the hydrophobic tail points inwards (Figure 9). The purpose of surfactants is to maintain distance between the polymer particles, as to stop them from adhering before being applied. ^[1]^[5]

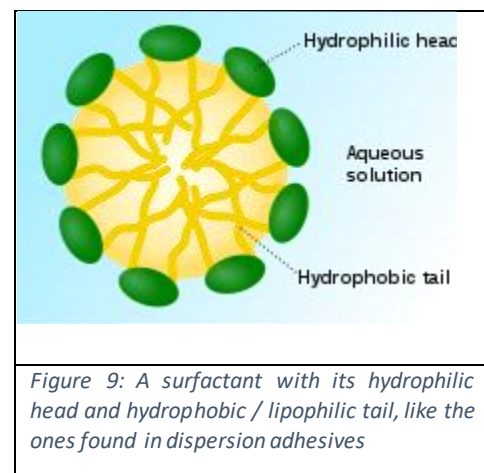


Figure 9: A surfactant with its hydrophilic head and hydrophobic / lipophilic tail, like the ones found in dispersion adhesives

The liquified adhesive consists of anywhere between 40% to 70% of polymers, the rest being the solvent. The setting of the adhesive happens through the solvent's removal, which can occur in two different ways. Very often the solvent evaporates with time, leaving behind the adhering mass. In some cases, the solvent may soak into the material of the PTBAs, making the process of adhesion faster. Through the solvent's removal the concentration of the polymer particles increases until they are close enough to create a unified polymer layer. This is visualized in Figure 10. ^[1]

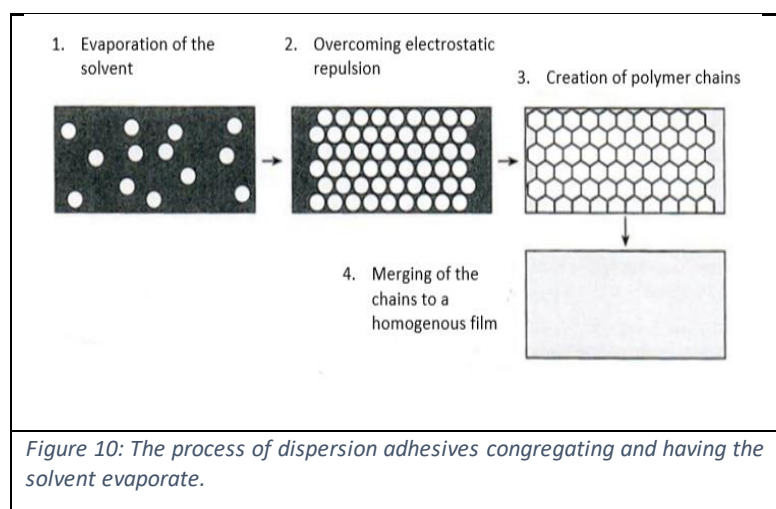


Figure 10: The process of dispersion adhesives congregating and having the solvent evaporate.

The advantage of dispersion adhesives is mainly that the solvent is water. This makes them applicable for situations, which require an incombustible solvent due to the heat involved. Additionally, water is advantageous in fields that have strong environmental regulations since the evaporating solvent should be environmentally friendly.

4.7.1.2 Hotmelt Adhesives

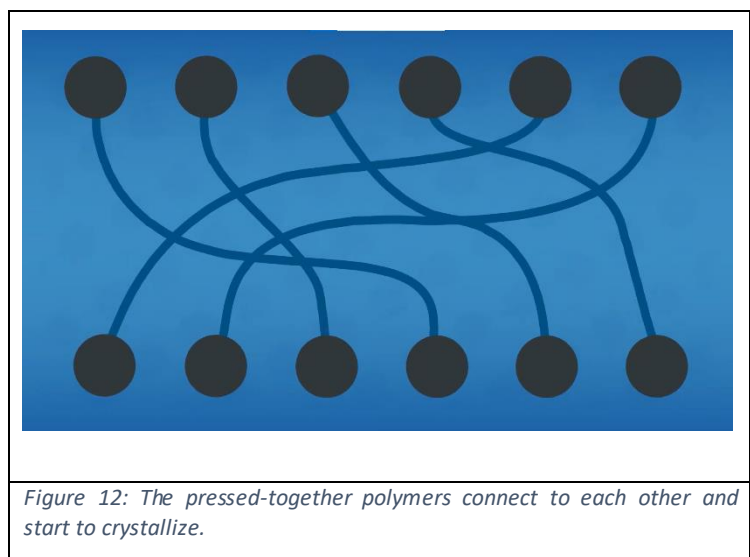
Hotmelt adhesives are thermoplastics, which are solid at rest temperatures and are melted before being applied. These adhesives must be applied quickly, so that the adhesive does not solidify before having covered the entire gluing area. It is advisable to heat up the PTBAs before applying the adhesive, especially when working with cold metals, because of the speed of the solidification process. These adhesives are used not only for their adhesive power, but also as sealants, due to the lack of solvents and their solid form. The most common use of hotmelt adhesives is glue guns, where the solid adhesive stick is melted in the gun and applied in liquid form. [6]



In comparison to dispersion adhesives, hotmelt adhesives do not lose out on adhesive surface since no solvent needs to evaporate. This may lead to parts of the surface not being covered with the polymer. Up to 70% of the volume may evaporate, especially if the share of polymer particles in the adhesive is rather small. Hotmelt adhesives have a relatively short setting time, whereas dispersion adhesives may only adhere once the solvent has evaporated. However, depending on the combustibility of the PTBAs, hotmelt adhesives may not be an option, as they could damage the PTBA. Should the adhered material be exposed to higher temperatures, the bond strength may be impaired, which could lead to melting of the adhesive and complete loss of adhesive power. [6][7]

4.7.1.3 Contact Adhesives

A contact adhesive is a bonding agent, which is applied dissolved to both PTBAs. Once the solvent mostly evaporates, the PTBAs must be pressed together. The pressure leads to the crystallization of the adhesive and thus its solidification. This process is seen in Figure 12. Contact adhesives set relatively quickly. This is due to the immediate crystallization. Contact adhesives are also commonly used for their great adhe-



sion to different materials. The concentration of polymer particles in commercially available contact adhesives lies at around 20% - 40% with the rest being the solvent. ^{[8][9]}

Temperature and humidity are important factors for the setting of the adhesive. High temperatures and low humidity lead to the solvent evaporating quicker. If the humidity is high, contact adhesives are not as efficient due to the speed of setting decreasing. These adhesives are generally solved in flammable or toxic solvents. Thus, they are difficult to operate at high temperatures or in industries with strict environmental regulations, unlike the two previously mentioned adhesives. ^[9]

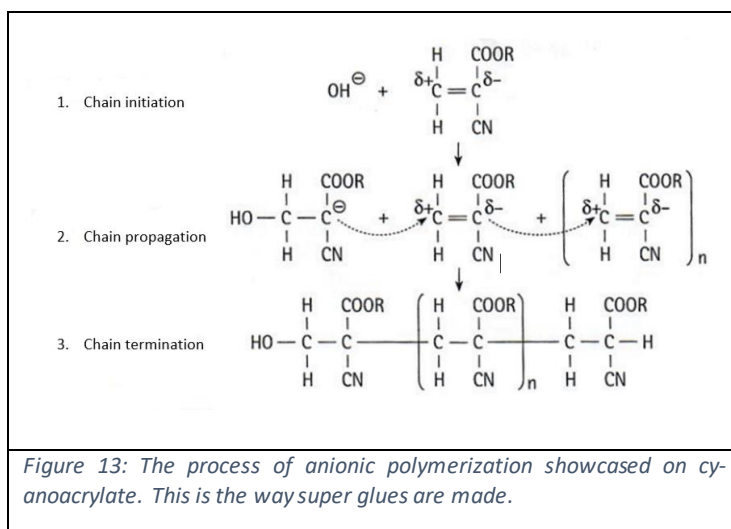
4.7.2 Chemically Curing Adhesives

Unlike physically setting adhesives, the polymers within the chemically curing ones only form within the glue joint. The polymer chains are constructed with the classic principles of polymer forming: polymerization, polyaddition and polycondensation. Unlike physically setting adhesives, they require chemical reactions to harden, whereas physically setting ones simply needed their solvents to evaporate. Generally, chemically curing adhesives resist temperature and humidity really well, which usually gives them an advantage in these aspects. [1]

4.7.2.1 Super glue

Super glues consist of cyanoacrylate esters. Their curing process is based on the previously mentioned anionic polymerization. The process specific to super glues is shown in Figure 13.

The cyan- and ester groups are electronegative, which attracts the nucleophile OH^- , which is



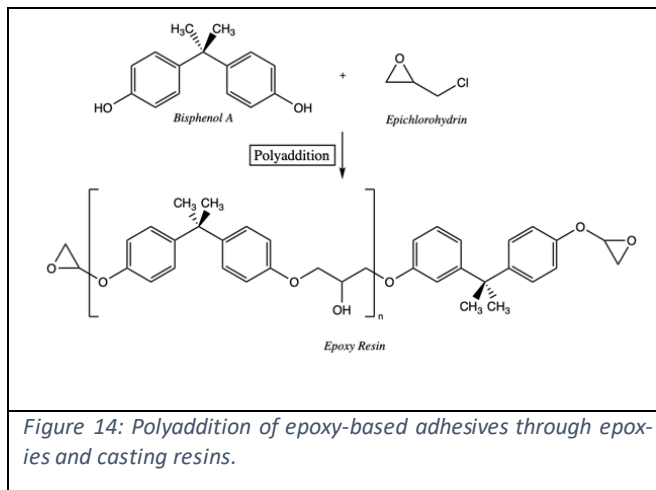
available in Water. Even a humidity of 50% provides enough OH^- to make the reaction possible. The rest of the reaction works the same as the previously mentioned ones. Since OH^- acts as a base, super glue works much better in basic than in acidic environments. Once there is enough OH^- available, be it through humidity or simply water being involved in the reaction, the polymerization of the liquid cyanoacrylate happens within a few seconds.

While super glue set quickly and have strong adhesion to a variety of substances. However, the thermal and chemical stability is lacking, which means that heat and chemicals can negatively influence this glue's adhesion. Furthermore, this adhesive cannot efficiently seal gaps due to the polymerization process. This limits its use, since other adhesives, such as hotmelt adhesives, do not have this drawback.

4.7.2.2 Epoxy-based adhesives

Epoxy-based adhesives always consist of an epoxy part and a hardener. The epoxy-part is an organic connection, with one oxygen-atom bonding with two carbon-atoms, creating a ring.

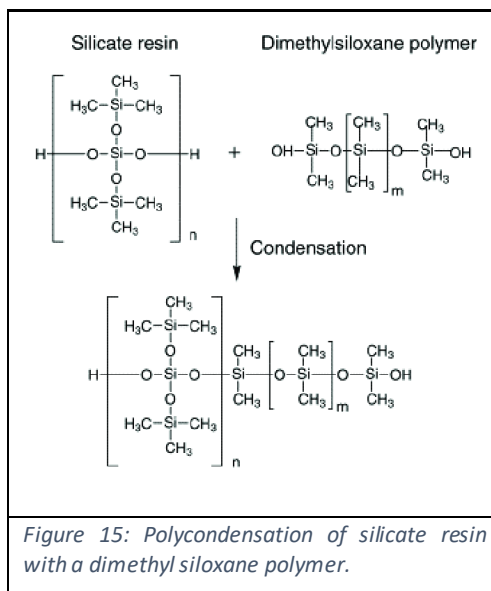
The hardener is usually in form of a casting resin. Those two components then conduct a polyaddition and become an epoxy-based adhesive. This process is made clear in Figure 14. [10]



Epoxy-based adhesives are advantageous in adhering metals and generally flat surfaces, which is why they are often used in aerospace adhesion. Additionally, they can withstand a wide range of humidity and temperature. Their polymerization and curing speed is relatively slow. They are also expensive and harmful to the environment, which makes their use very limited and explains why they only make up around 5% of the market share for adhesives. [11]

4.7.2.3 Silicone adhesives

Silicone adhesives cure according to the principle of polycondensation, where two polymers condensate and water gets eliminated. Where the two polymers connect, the OH of the dimethyl siloxane polymer bonds with the H of the silicate resin and creates H₂O, which is then discarded.



Silicone adhesives differ from other chemically curing adhesives because their adhesive strength is limited. However, they offer great resistance to humidity and weathering in general, as well as heat. Additionally, it has high flexibility and elasticity. These properties mean that silicone adhesives are more often used as sealants than primarily adhesives. [12]

4.8 Fields of use

Because of their variety and effectiveness, chemically curing adhesives are much more commonly used than physically setting ones. In construction physically setting adhesives are usually used, such as all-purpose adhesives or hotmelt adhesives. However, in most other industries chemically curing adhesives are more common. Especially in the railway or aerospace industry, adhesives have overtaken other joining methods. In trains, the aim is to reduce their weight, while maintaining or increasing their performance. Adhesives are especially suited for this, because of their lightness. In the shipping industry, where stability is crucial, using adhesives instead of the heavy rivets reduces the weight of the ship significantly.

As the railway and shipping industry aim to decrease more weight, using thin materials is beneficial. To connect thin materials, using rivets or other joining methods gets increasingly more difficult. Using adhesives, however, circumvents that problem. ^[1]

4.9 Advantages and disadvantages

There are several other joining techniques besides adhesives, such as welding and riveting. This chapter compares them to adhesives and discusses the advantages and disadvantages of adhesives.

The process of welding metal requires a lot of heat, which alters the metals properties within the heat-affected zone. Riveting only allows force to be transferred at certain points. Moreover, drilling holes in the material leads to weakening of the material. Most adhesives avoid these problems. Most adhesives do not require heat and therefore do not alter the joined material. Secondly, they do not necessitate the drilling of holes and thus do not destroy and weaken the material's structural integrity.

Consequently, adhesives will especially gain importance in fields like microelectronics,

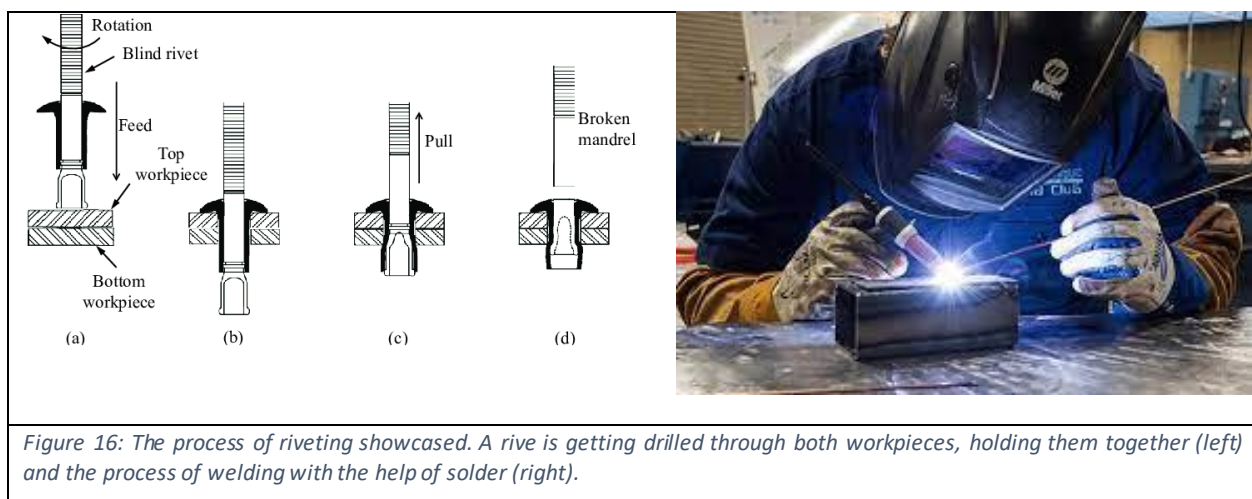


Figure 16: The process of riveting showcased. A rivet is getting drilled through both workpieces, holding them together (left) and the process of welding with the help of solder (right).

where drilling a hole in order to join two parts is rarely feasible.

Because of the variety of adhesives and their physicochemical properties, their use can be highly diverse and allows for different conditions. ^[13]

5 Results

To test the force of the adhesives, they were applied in two different ways: on wooden and aluminum blocks with a surface of 2.25 cm^2 as well as on screw nuts and clothespins. To measure the force to break the adhesive bonds, the used PTBAs were pulled apart. The force was measured with a 50 N/cm^2 dynamometer. Every experiment series consisted of 5 measurements per material.

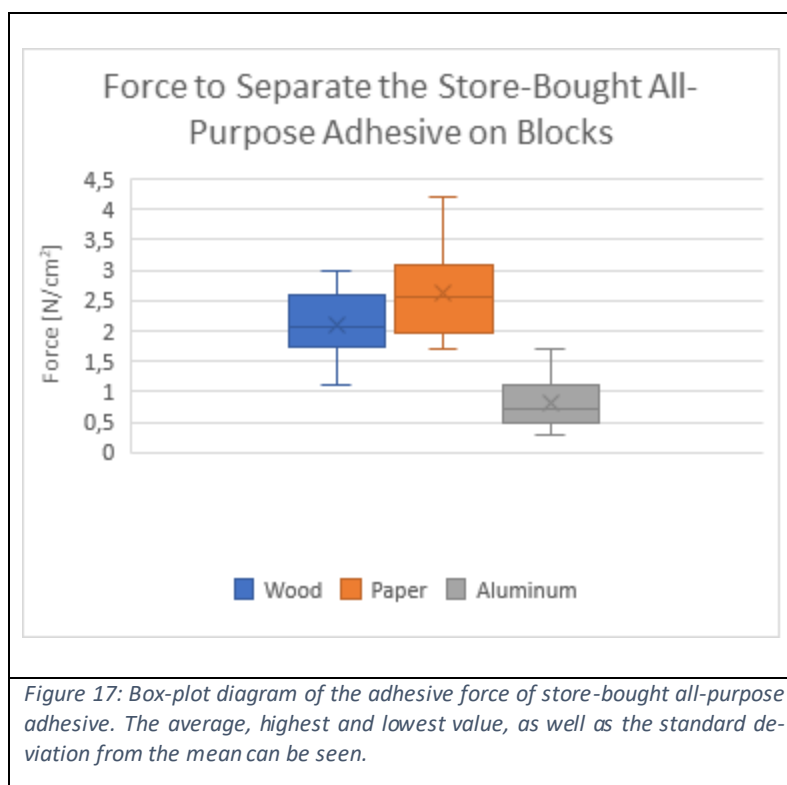
The experiments on wooden blocks were conducted only once due to high production time and are exclusively discussed in chapter 5.1.1. In all the other experiments, the adhesives were tested on screw nuts and clothespins. The screw nuts were made of aluminum and polyamide and the clothespins were made of wood. The clothespins were used for the testing of wood and paper. The adhesive had a setting time of approximately 24 hours. The amount of adhesive used for all experiments laid between 0.004 g and 0.005 g .

5.1 Adhesive Strength of Store-Bought All-Purpose Adhesive

Experiments with a store-bought all-purpose adhesive were performed and the results will be displayed in the following two sub-chapters. The adhesive used was made by the German company UHU, which specifies in all-purpose adhesives. The one used is called “Der Alleskleber”.

5.1.1 Adhesive Strength on Blocks

In this series of experiments, the store-bought all-purpose adhesive was tested on wood, paper, and aluminum surfaces with an area of 4 cm^2 . The adhesive had a setting time of approximately one hour.



In Figure 17, Paper visualized the highest average value and also the biggest single deviation from the average, as well as largest range in general. Wood had the second highest average, whereas aluminum had by far the lowest values measured with the smallest discrepancies of all.

However, because the adhesive on the blocks did not dry completely, right before testing their adhesive force, the experiments were conducted again, this time however, instead of letting set for only 40 min to 60 min, at least 24 h were given in order to make

sure that every adhesive on every material was completely dry and at its peak adhesion.

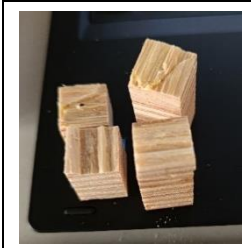


Figure 18: The broken blocks of plywood. The differently colored layers, where the wood broke, can be seen (light and dark brown).

In this experiment all the blocks held together way too strongly, wherefore it was not possible to pull them apart at all, let alone try to measure the adhesive force with a dynamometer of only 50 N/cm^2 . When more force was applied, the links between the layers of different types of wood within the plywood broke (Figure 18). Thus, it was not possible to measure how much force was required.

The gluing area was then reduced to 2.25 cm^2 , which produced a single value of under 50 N/cm^2 . Paper on average took 40 N/cm^2 to separate. However, the rest of the wooden blocks was not measurable.

5.1.2 Adhesive Strength on Screw Nuts and Clothespins

In this series of experiments, the store-bought all-purpose adhesive was tested on wood, paper, aluminum, and polyamide surfaces. The adhesive was left to set for 24 hours.

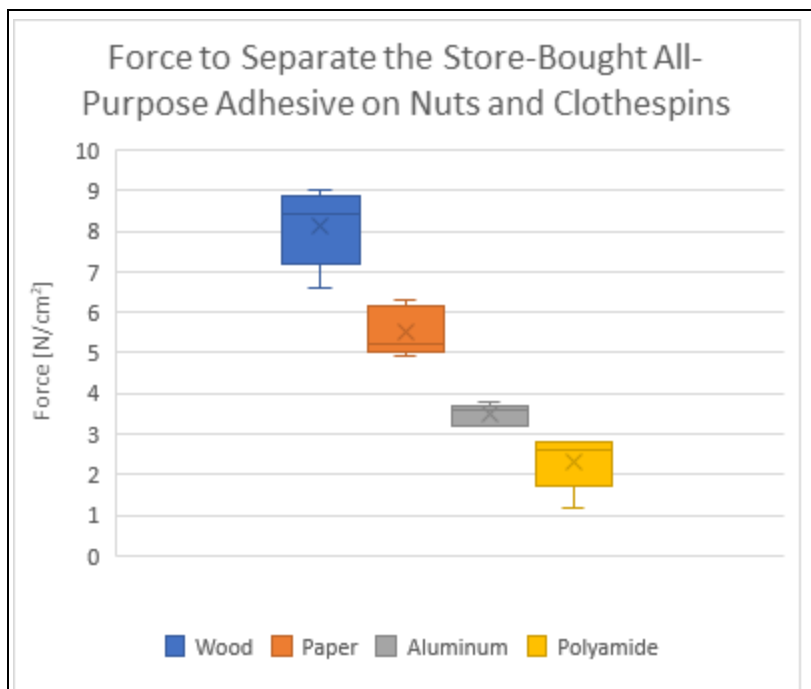
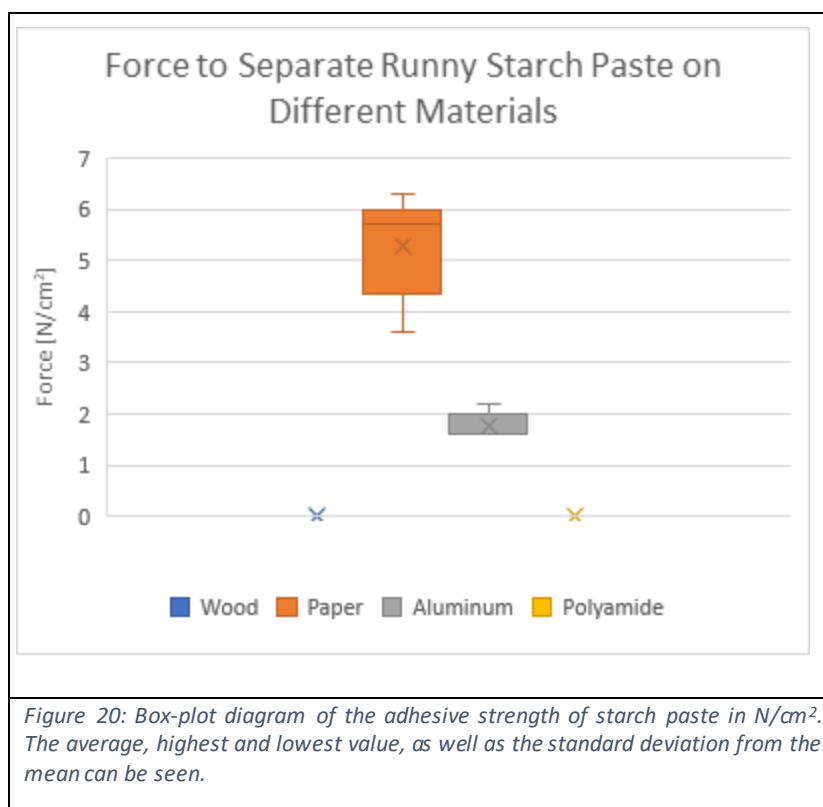


Figure 19: Box-plot diagram of the adhesive force of store-bought all-purpose adhesive. Polyamide was measured with the help of screw nuts. The average, highest and lowest value, as well as the standard deviation from the mean can be seen.

The highest adhesive force was measured for wood with values between 6.6 N/cm^2 and 9 N/cm^2 , with an average of 8.1 N/cm^2 . The adhesive force between paper surfaces laid in the range between 4.9 N/cm^2 and 6.3 N/cm^2 , with an average of 5.5 N/cm^2 . Aluminum had the smallest range of values, going from 3.2 N/cm^2 to 3.8 N/cm^2 with an average of 3.5 N/cm^2 . Polyamide gave the lowest values with discrepancies ranging from 1.2 N/cm^2 to 2.8 N/cm^2 , averaging at 2.3 N/cm^2 .

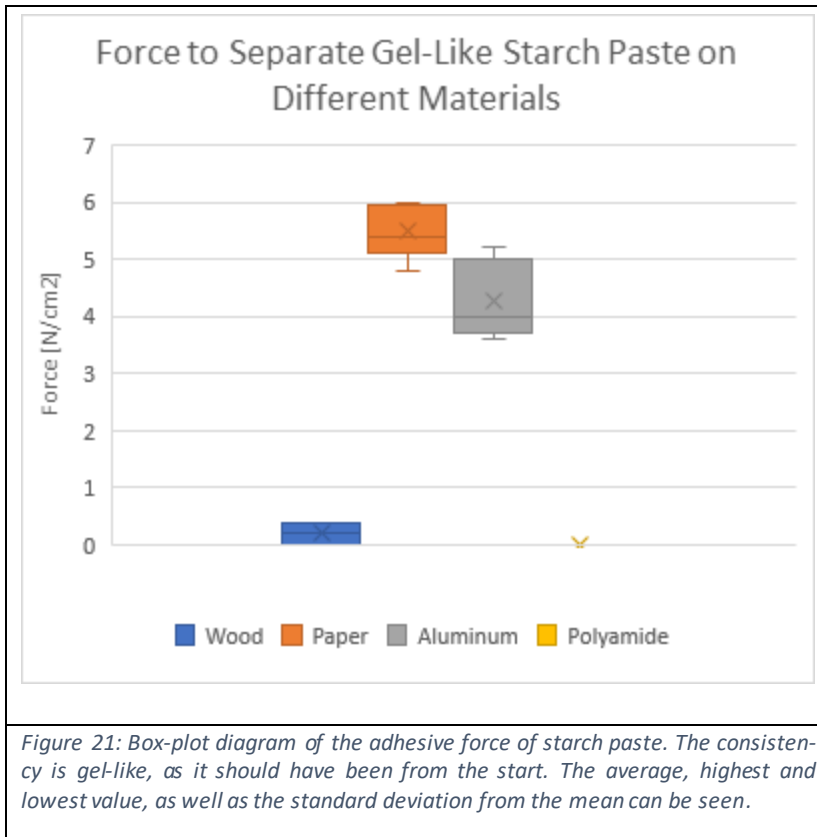
5.2 Adhesive Strength of Starch Paste

To synthesize starch paste, potato starch (10 g; 62 mmol; 1.0 Eq) was dissolved in water (130 mL; 7200 mmol; 120 Eq) while heating the mixture at around 85 °C. After cooling down to room temperature, the product had a watery, slightly sticky consistency. The product was tested on its adhesive strength.



Wood and polyamide did not adhere. The starch paste crumbled and was not sticky enough. However, paper PTBAs and aluminum PTBAs did stick. Aluminum parts adhered with an average force of 1.8 N/cm², reaching as high as 2.2 N/cm² and as low as 1.6 N/cm². The force measured on paper had a broader distribution. It reached from 3.6 N/cm² to 6.3 N/cm² with an average of 5.3 N/cm².

After leaving the liquid starch paste for a day, letting some of the water evaporate, the adhesive became gel-like. The tests were also conducted with this concentrated paste.



The adhesive force between polyamide PTBAs was still too low to be measured. However, the concentrated paste slightly adhered to wood. The measured force was ranging from 0 N/cm² to 0.4 N/cm² with an average value of 0.2 N/cm². The force on paper PTBAs ranged from 4.8 N/cm² to 6 N/cm² with an average value of 5.5 N/cm². Aluminum PTBAs generated smaller forces with a higher discrepancy between them, ranging from 3.7 N/cm² to 5.2 N/cm², 4.3 N/cm² being the average.

Comparing the differently concentrated pastes, it is significant, that the higher concentration of potato starch results in adhesion between wooden parts and that aluminum slightly increases in adherence, whereas the values for paper and polyamide surfaces stay approximately the same.

5.3 Adhesive Strength of Polyvinyl Alcohol All-Purpose Adhesive

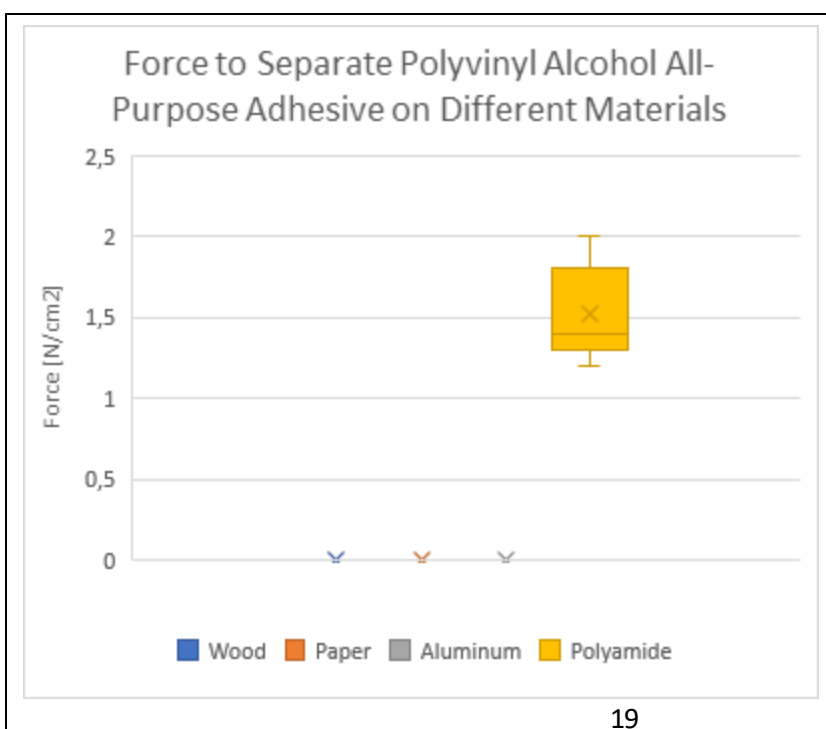


Figure 22: Box-plot diagram of the adhesive force polyvinyl alcohol all-purpose adhesive. The average, highest and lowest value, as well as the standard deviation from the mean can be seen.

For this experiment polyvinyl alcohol (3.0 g; 68 mmol; 1.0 Eq) was dissolved in

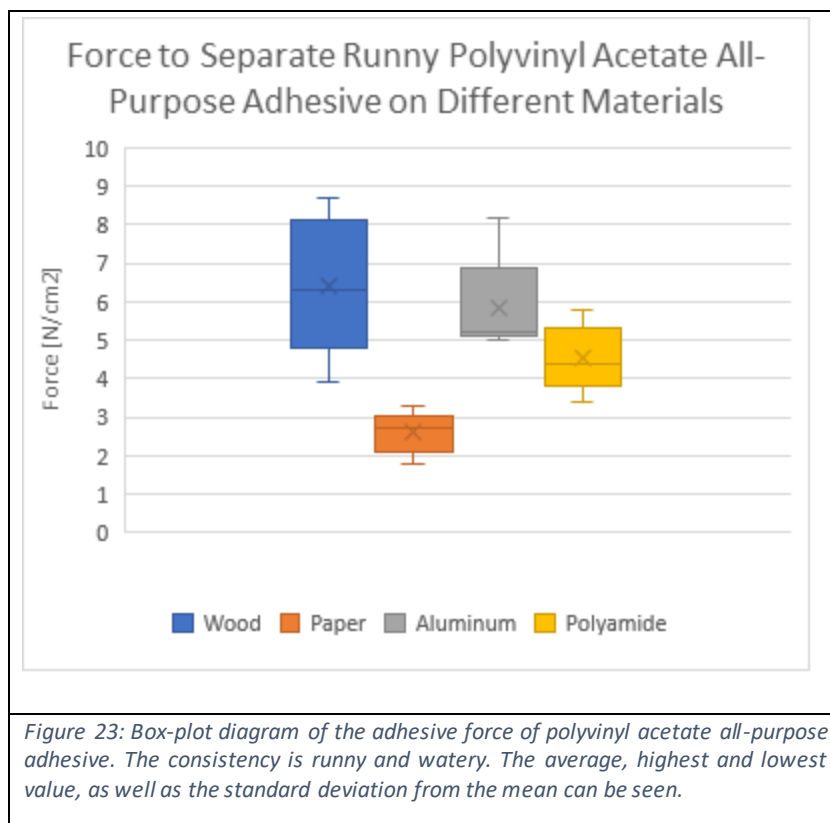
deionized water (50 mL; 2800 mmol; 40 Eq). It became a very runny, slightly sticky adhesive. Most of the polyvinyl alcohol could not dissolve in water, even after heating it up to 85 °C.

Polyamide was the only material which adhered. With wood, paper and aluminum, this polyvinyl alcohol adhesive did not adhere at all. This problem can most likely be traced back to the polyvinyl alcohol barely dissolving.

The same experiment was attempted with ethyl acetate as the solvent. However, still only very little polyvinyl alcohol ended up dissolving and thus the results of the experiment did not change.

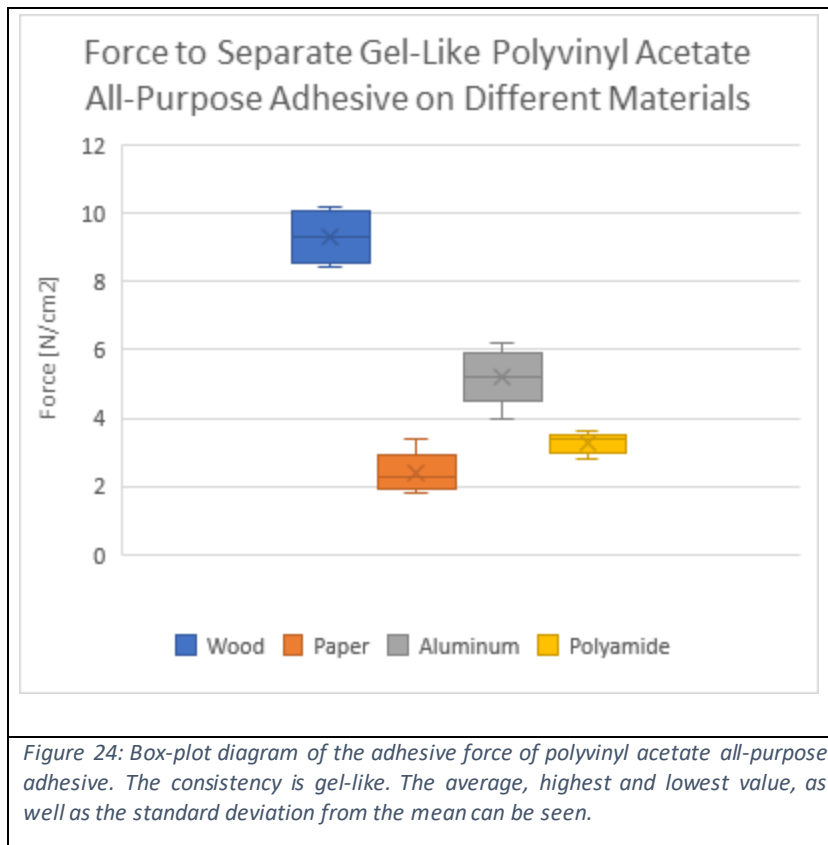
5.4 Adhesive Strength of Polyvinyl Acetate All-Purpose Adhesive

To try out another self-made all-purpose adhesive, a polyvinyl acetate adhesive was synthesized. Polyvinyl acetate (8.0 g; 93 mmol; 1.0 Eq) was dissolved in ethyl acetate (30 mL; 310 mmol; 3.3 Eq) while heated to 40 °- 60 °C. The solution was homogeneous. After the adhesive had cooled down, it was of low viscosity.



Unlike the previous adhesive, this one adhered all the materials. Wood adhered the best, with high discrepancies. The values ranged from 3.9 N/cm² to 8.7 N/cm² with an average of 6.4 N/cm². Aluminum adhered with smaller discrepancies. The values for aluminum ranged from 5 N/cm² to 8.2 N/cm². The average was 5.8 N/cm². The values for the polyamide's adhesive strength had low discrepancies: They lay between 3.4 N/cm² and 5.8 N/cm², the average being 4.5 N/cm². Lastly, paper adhered the worst with the

smallest discrepancies, ranging from 1.8 N/cm² to 3.3 N/cm². The average was 2.7 N/cm².



The adhesive was tested again after a 24-hour period, letting the paste concentrate through evaporation of solvent.

Wood adhered the best with its values ranging from 8.4 N/cm² to 10.2 N/cm². The average was 9.3 N/cm². Adhesive strength on aluminum amounted to values between 4 N/cm² and 6.2 N/cm², averaging 5.2 N/cm². Polyamide adhered with force ranging from 2.8 N/cm² to 3.6 N/cm². The average value was 3.3 N/cm². Values on paper laid between 1.8 N/cm² and 3.4 N/cm²

with an average of 2.4 N/cm². The discrepancies for all materials were rather small for this adhesive paste.

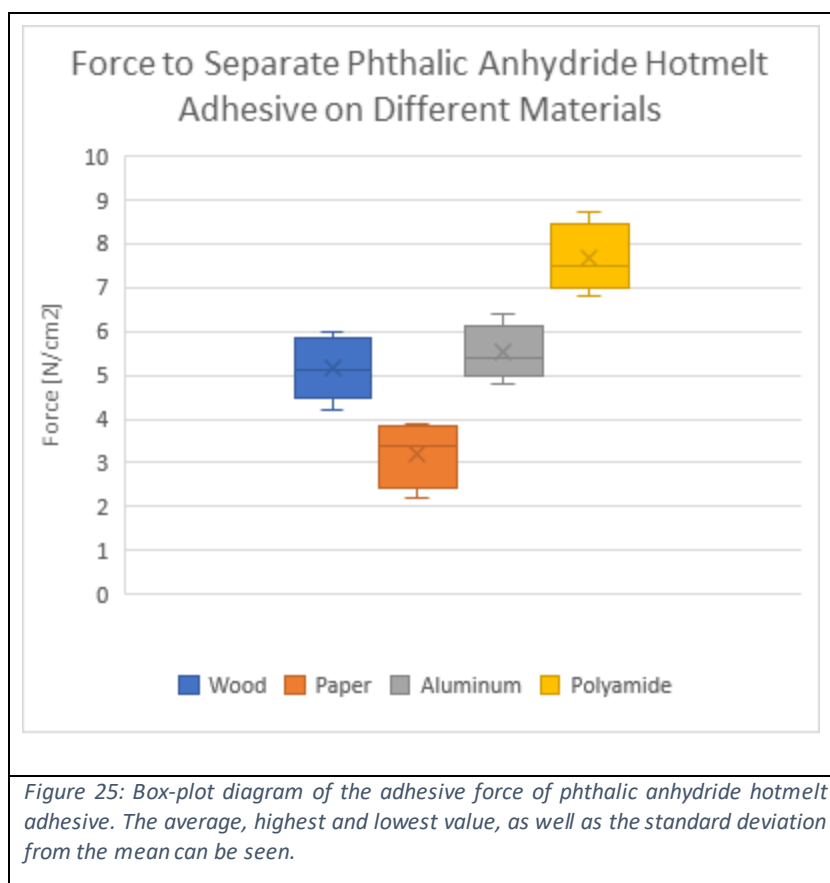
5.5 Adhesive Strength of Phthalic Anhydride Hotmelt Adhesive

To synthesize a hotmelt adhesive, phthalic anhydride (1.5 g; 10 mmol; 1.0 Eq) was dissolved in ethanediol (1.0 mL; 18 mmol; 1.8 Eq) over a Bunsen burner in a test tube.

Since the adhesive was a hotmelt adhesive, it had to be applied in a molten state in order to adhere. The adhesive was transported to a beaker, where it was heated to around 130 °C. All the materials adhered at room temperature except for aluminum. Thus, aluminum was

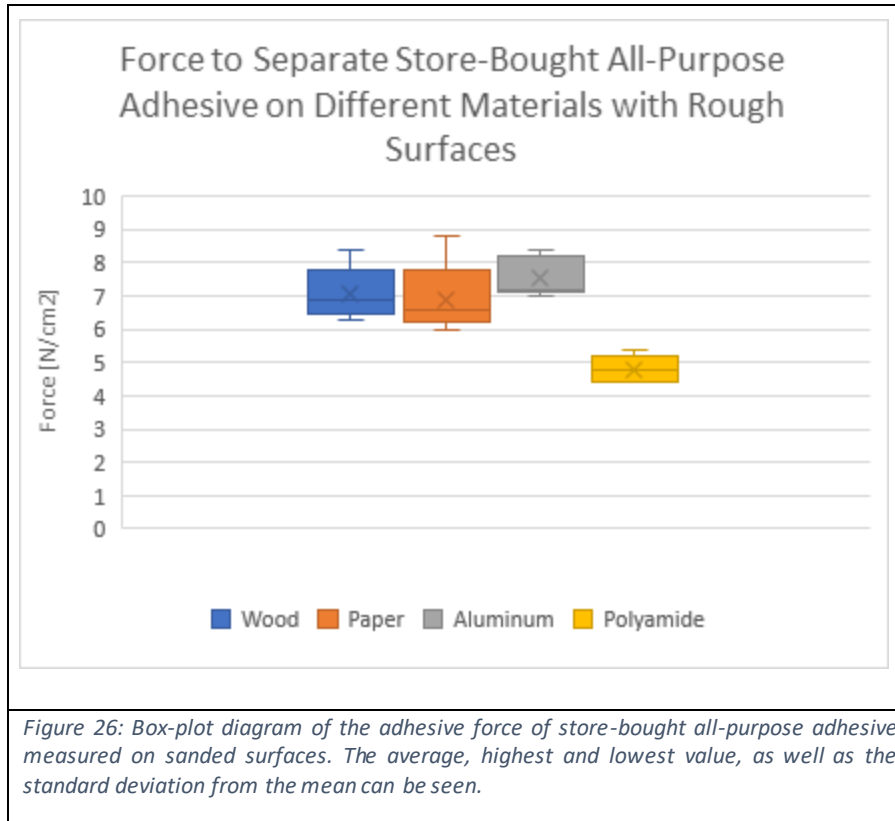
warmed up on the heating plate and the adhesive was applied.

This hotmelt adhesive made polyamide adhere the best, with values from 6.8 N/cm² to 8.7 N/cm² and an average of 7.7 N/cm². Wood and aluminum had similar values, ranging from 4.2 N/cm² to 6 N/cm² for wood and 4.8 N/cm² to 6.4 N/cm² for aluminum with an average of 5.1 N/cm² and 5.5 N/cm² respectively. Paper adhered the worst, the highest value being 3.9 N/cm² and the lowest 2.2 N/cm², with an average of 3.2 N/cm².



5.6 Adhesive Strength of Store-Bought All-Purpose Adhesive on Sanded Surfaces

To test if sanded surfaces affect adhesive strength experiments with the store-bought all-purpose adhesive discussed in chapter 5.1 were performed on such surfaces. All of the PTBAs' surfaces were sanded.



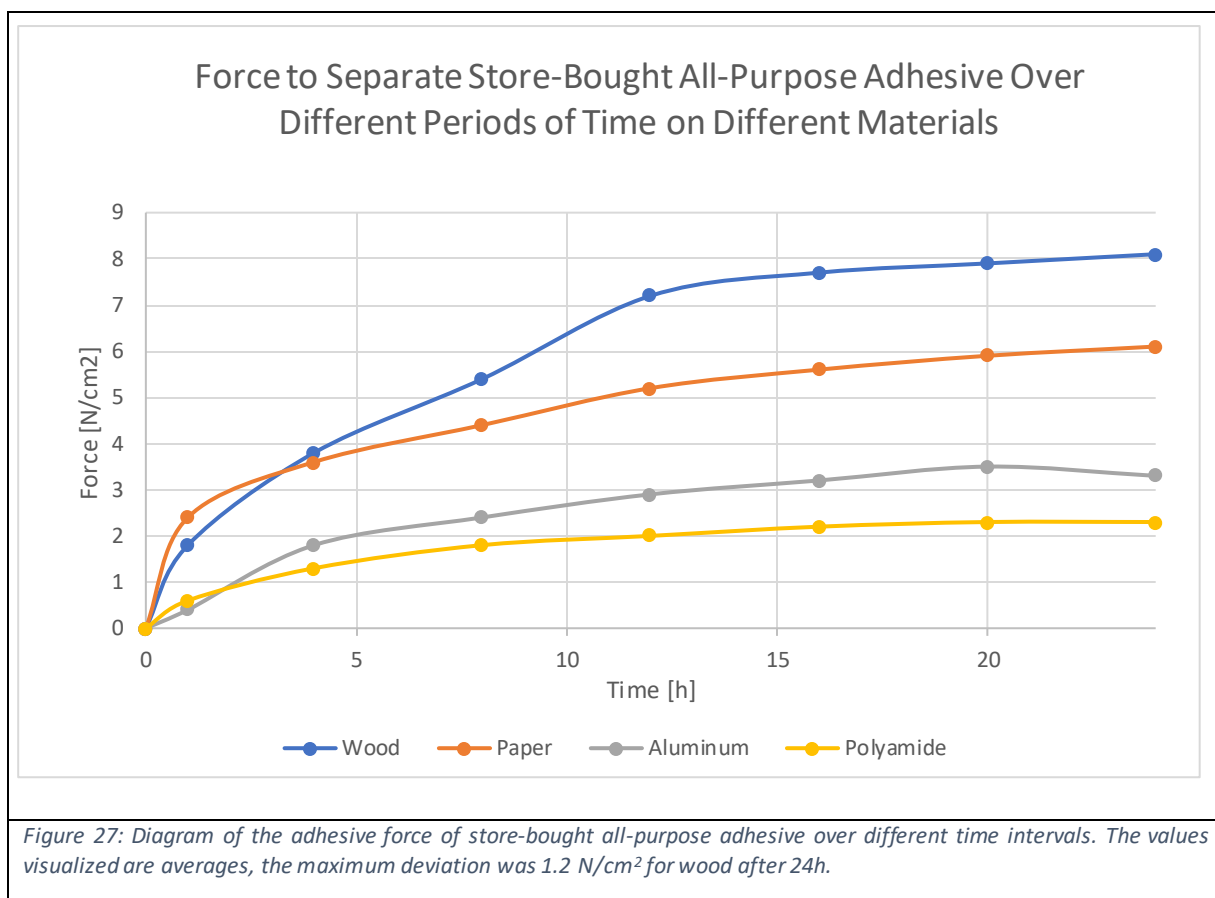
The values for wood, paper and aluminum were relatively similar. Values ranged from 6.3 N/cm² to 8.4 N/cm² for wood and 6 N/cm² to 8.8 N/cm² for paper with a respective average of 7.1 N/cm² and 6.9 N/cm². Aluminum averaged slightly higher values of 7.6 N/cm² with a much smaller range between 7 N/cm² and 8.4 N/cm². This adhesive on sanded polyamide measured significantly lower adhesive strength, ranging from 4.4 N/cm² to 5.4 N/cm² with an average value of 4.8 N/cm².

seive strength, ranging from 4.4 N/cm² to 5.4 N/cm² with an average value of 4.8 N/cm².

5.7 Adhesive Strength of Store-Bought All-Purpose Adhesive Over Different Periods of Time of Time

Given the results of the adhesion of wood and paper discussed in chapters 5.1.1 and 5.1.2 with different time intervals, the strength of the same store-bought all-purpose adhesive was tested depending on its setting time.

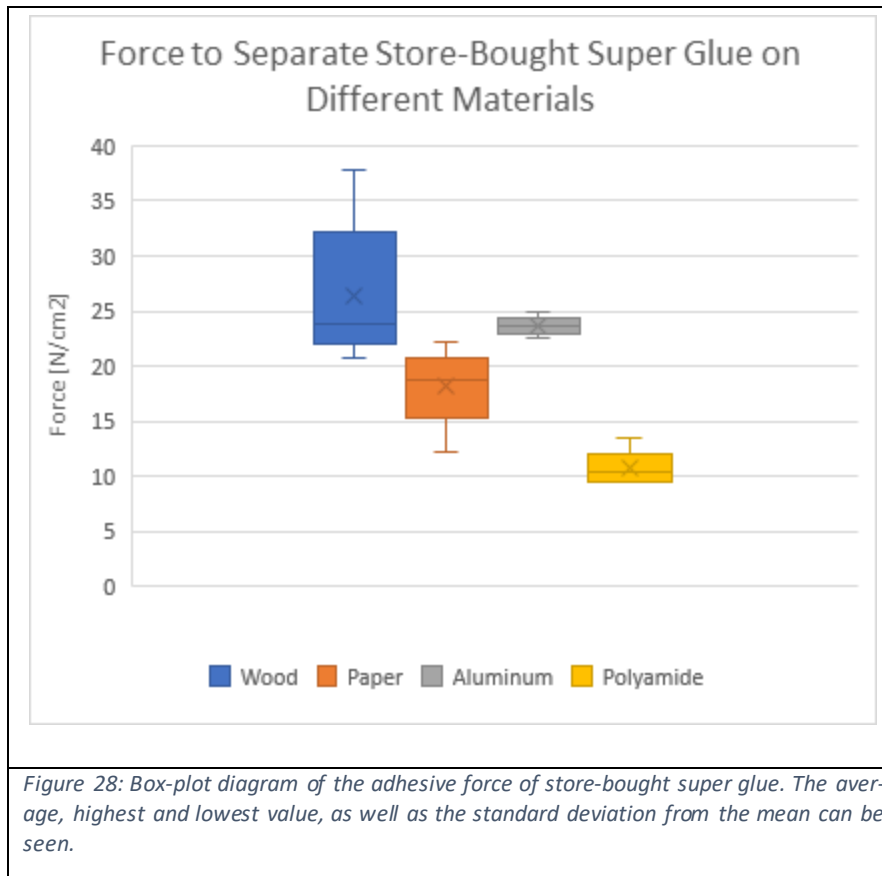
The development of the adhesive strength showed a similar trend on all the materials. Whereas paper adhered better at the start, wood held better over a longer period of time. The same was observed between aluminum and polyamide, where polyamide adhered better at first, but adhered worse than aluminum over a longer period of time. The results are



visualized Figure 27.

5.8 Adhesive Strength of Store-Bought Super Glue

Due to synthesis of chemically curing adhesives being too expensive for this project, a store-bought super glue was tested. It was the “UHU Sekundenkleber Pipette” by the German adhesive company UHU.



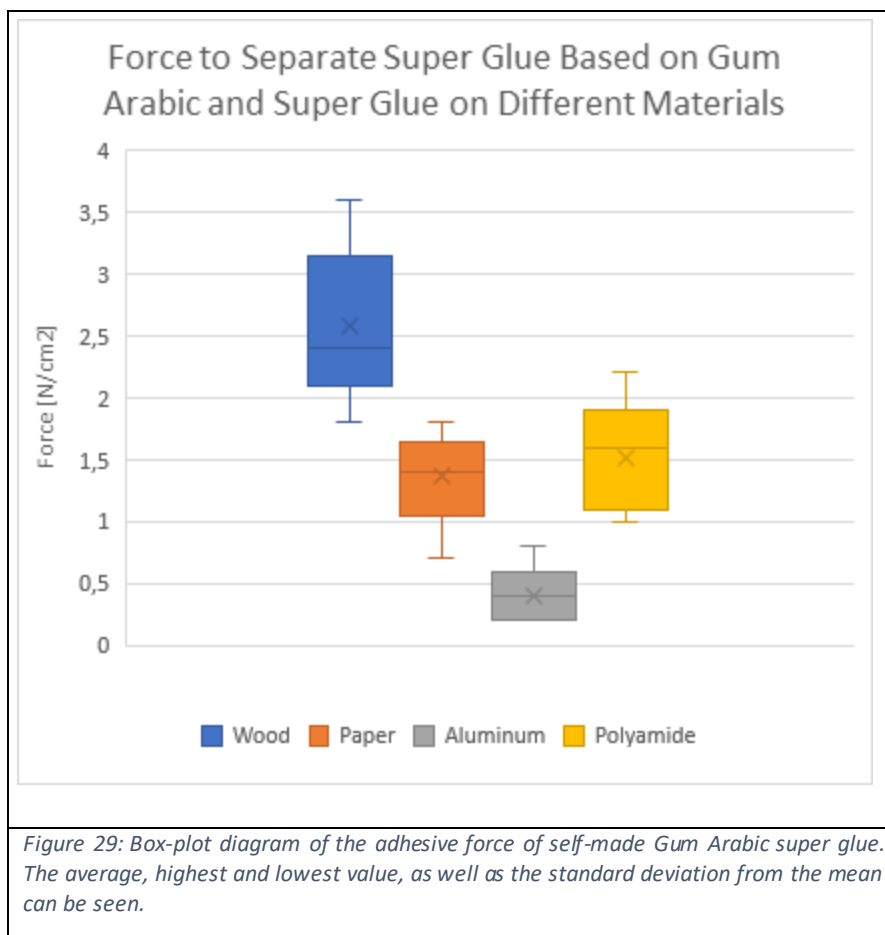
The values measured with the store-bought super glue showed higher values than any previously tested adhesive. The lowest value for wood was 20.7 N/cm². The highest value was 37.8 N/cm², and the average was 26.4 N/cm². The second-highest values were measured for aluminum. It had low discrepancies compared to wood with values ranging from 22.6 N/cm² to 25.0 N/cm², with an average of 23.6 N/cm².

The adhesive force of paper was also relatively high, with values ranging from 12.2 N/cm² to 22.2 N/cm² while averaging 18.1 N/cm². Lastly, polyamide had values between 9.5 N/cm² and 13.4 N/cm² with an average of 10.7 N/cm².

5.9 Adhesive Strength of Super Glue Based on Gum Arabic and Glycerin

The following adhesive was synthesized according to the literature procedure [14]. In order to make this super glue, Gum Arabic (24 g; 99 mmol [15]; 1.0 Eq) was dissolved in glycerin (15 mL; 210 mmol; 2.1 Eq) and deionized water (5.0 mL; 280 mmol; 2.8 Eq) on a heating plate. This resulted in a sticky, highly viscous substance that acted like super glue.

The values measured for wood were the highest, ranging from 1.8 N/cm² to 3.6 N/cm², averaging at 2.6 N/cm².



The second-highest values came from polyamide. The average was 1.5 N/cm² and the range lay between 1.0 N/cm² and 2.2 N/cm². Paper adhered with an average force of 1.4 N/cm², with values between 0.7 N/cm² and 1.8 N/cm². The values for aluminum were the lowest. They laid between 0.2 N/cm² and 0.8 N/cm² and averaged at 0.4 N/cm².

6 Discussion

In the following chapter all the results depicted and visualized in chapter 5 are discussed. The discussion concerns itself with the physical reasoning behind the setting process and adhesive strength, as well as the chemical bonding between polymers and the solvent and the PTBAs.

6.1 Adhesive Strength of Store-Bought All-Purpose Adhesive

As it was visualized in chapter 5.1.1, the store-bought all-purpose adhesive adhered the best to paper, followed by wood and aluminum. However, as shown in chapter 5.1.2, after a longer setting time, wood adhered better than paper. The wood generally adhering better might be due to its gluing surface being rougher than the one of aluminum or paper. The sanded surface gives the wood more surface area to adhere. It is possible that stronger adhesive force may be achieved by sanding the gluing surface of paper and aluminum with sandpaper. [4]

Another approach at explaining this phenomenon is that the general setting time on paper is faster than on wood, since it can absorb the solvent faster than wood or other materials. This results in paper adhering faster, since the solvent is absorbed by the paper and more polymers connect to the PTBAs.

The setting time of an adhesive also has a noticeable effect on the adhesive strength, which is further discussed in chapter 6.6.

As mentioned in chapter 5, the surface area of 2.25 cm² was too large to measure the required force with the available dynamometer. For convenience reasons, instead of using the self-made blocks, they were replaced with commercially available screw nuts and clothespins.

Factors like humidity or temperature could not have played a part in the relative speed of evaporation of the solvent, since all the experiments were performed on the same day within a few hours.

6.2 Adhesive Strength of Starch Paste

When comparing the results presented in chapter 5.2, there are significant differences. On average, aluminum adhered better by 2.5 N/cm² (140 % increase) with the concentrated adhesive. Paper adhered better with an average increase of 0.3 N/cm² (5.8% increase). With the concentrated adhesive, wood PTBAs showed mild adhesion, whereas this was not the case with the diluted adhesive. Polyamide could not be adhered in either experiment.

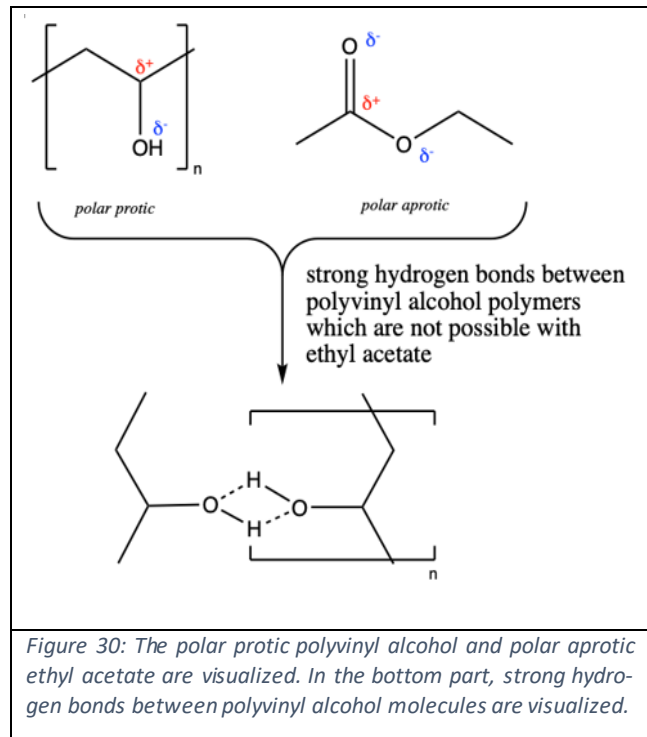
The diluted adhesive had a higher solvent concentration than the gel-like one. Thus, there were more polymers to hold the PTBAs together. Therefore, when the solvent evaporated, the PTBAs adhered better with the higher concentration of polymers. The more diluted the polymers during application, the less polymers there are in total, and thus, the worse the adhesion.

The gel-like paste, having a higher concentration of polymers led to generally better adhesion. The adhesive force on paper did not change significantly, as it absorbed much of the

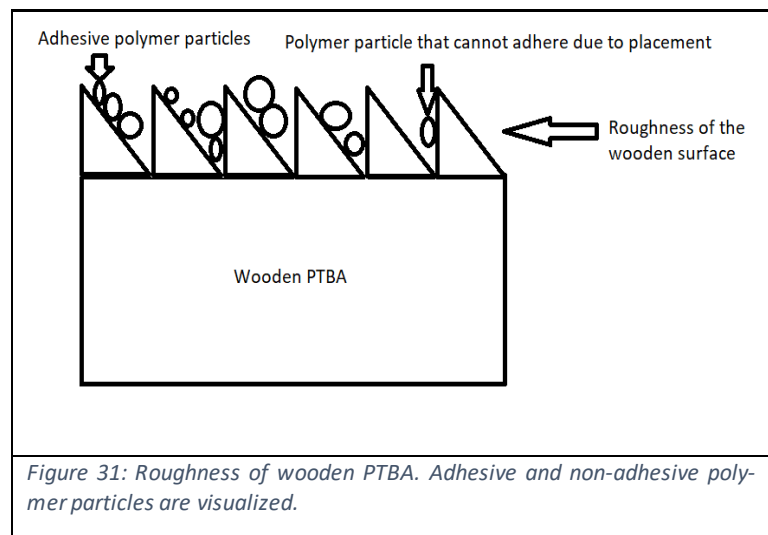
solvent when it was applied. This led to more adhesive being applied (approximately 1 mg) to cover the whole surface area. This increased the number of applied polymers and led to paper sticking approximately the same as with the concentrated adhesive.

6.3 Adhesive Strength of Polyvinyl Alcohol All-Purpose Adhesive and Polyvinyl Acetate All-Purpose Adhesive

As mentioned in chapter 5.3, the polyvinyl alcohol adhesive dissolved poorly in ethyl acetate. The reason, why the polyvinyl alcohol could not dissolve in ethyl acetate, are Van der Waals forces as well as hydrogen bonds. Polyvinyl alcohol is a rather large molecule with a big surface resulting in strong Van der Waals forces between the polyvinyl alcohol molecules. These forces are strengthened further by hydrogen bonds between the hydroxy groups of polyvinyl alcohol. Whereas an ethyl acetate molecule is polar aprotic and can only act as an acceptor for hydrogen bonds, a hydroxy group is polar protic and can be both a donor as well as an acceptor, strengthening the interaction between the polyvinyl alcohol. The hydrogen bonds as well as the two molecules are visualized in Figure 30.



For the polyvinyl acetate all-purpose adhesive, whose results were discussed in chapter 5.4, two series of experiments were conducted: experiment on diluted adhesive as well as on a more concentrated one. The concentrated adhesive was prepared by letting the solvent evaporate from the diluted polyvinyl acetate over 24 hours. The concentrated adhesive resulted in approximately the same adhesive forces as the diluted one. However, the measured values for the concentrated polyvinyl acetate adhesive had significantly lower discrepancies.



Wooden surfaces adhered better in concentrated polyvinyl acetate adhesives on average by 2.9 N/cm² (45 % increase). Due to the higher concentration of ethyl acetate in the diluted adhesive as well as the poor solvation of the adhesive in the solvent, some spots on the ad-

hesive surface were not covered in polymers (Figure 31). This was visually confirmed after the cohesion zone was broken apart.

The lower discrepancies are due to the gel-like concentrated adhesive being better mixed than the diluted adhesive. Thus, the concentrated adhesive reduces the number of spots without any adhesive, making the results easier to imitate and more consistent.

In comparison to the polyvinyl alcohol adhesive, the polyvinyl acetate dissolved in ethyl acetate. Unlike in the previous experiment, the chemical characters of polyvinyl acetate and ethyl acetate are similar allowing them to create a homogeneous solution.

6.4 Adhesive Strength of Phthalic Anhydride Hotmelt Adhesive

Due to quick setting of the hotmelt adhesive, the application of the adhesive was rather inconsistent. This is due to the adhesive being applied to all materials one after another without reheating the adhesive. Thus, the adhesive could not be applied to the PTBAs with a consistent temperature. Therefore, the results are to be interpreted with respect to this fact.

The adhesive was first applied on wood, followed by polyamide, aluminum, paper, and aluminum again. The aluminum had to be done twice because due to thermal conductivity of aluminum the hotmelt adhesive cured before the second PTBA could be positioned. To prevent that, aluminum PTBAs were heated on a heating plate while the adhesive was applied to paper. Thus, one of the reasons for paper having the lowest adhesion force is the fact that it was the adhesive was the coldest during its application.

The hotmelt adhesive adhered polyamide the best, followed by warm aluminum, wood and paper. The reason for polyamide adhering the best, might be that polyamide might have softened due to high temperatures and deformed, thus increasing the adhesion area. Aluminum most likely adhered better due to the PTBA being heated up. Because the surface was still hot, the adhesive could distribute itself more equally, allowing better adhesion.

6.5 Adhesive Strength of Store-Bought All-Purpose Adhesive on Sanded Surfaces

In comparison to the results in chapter 5.1 the results presented in chapter 5.6 are considerably different. Whereas wood's adhesion deteriorated, all the other materials adhered significantly better.

Wood's average adhesion force decreased by 1.0 N/cm^2 (12% decrease). By sanding the wooden PTBAs, they became smoother. Thus, the adhesion area was decreased and the PTBAs adhered worse.

With the other materials, the opposite happened: the adhesion surfaces became rougher. Thus, the adhesion area was increased and the PTBAs adhered better. The average adhesion forces for paper, aluminum and polyamide increased by 1.4 N/cm^2 (25% increase), 4.1 N/cm^2 (117% increase) and 2.5 N/cm^2 (109% increase) respectively.

Aluminum and polyamide show the biggest improvements in their adhesiveness. This is due to aluminum and polyamide being relatively smooth materials. Thus, the sanding roughened their surfaces significantly more than paper. These results indicate that the carves and notches on the adhesion surface increase the adhesion area and thus, the adhesion force.

6.6 Adhesive Strength of Store-Bought All-Purpose Adhesive over Different Periods of Time

To test how much of an influence the setting time has on the adhesion force, store-bought all-purpose adhesive was investigated. The adhesive force was tested over the course of 24 hours. As visualized in Figure 27, the results show a tendency of the adhesive strength increasing, the longer the adhesive sets. However, the trend is logarithmic and thus, the effect becomes less significant the more time goes on.

The curves resemble a logarithmic function. This is due to the polymers being created fastest at first, when there is a lot of starting material and chain termination possibilities. This process however slows down and stops once the equilibrium is reached. The equilibrium can be followed by observing the increase of adhesive strength.

In Figure 27, it is visible, that paper and wood adhere faster than aluminum and polyamide. This is due to the absorbing effects of these materials: by absorbing the solvent, the concentration of the polymers increases and thus the polymers set quicker.

6.7 Adhesive Strength of Store-Bought Super Glue and Self-Made Gum Arabic Super Glue on

As demonstrated in chapter 5.8, the store-bought super glue adhered the best to all materials. The lowest adhesion force with the store-bought super glue was measured for smooth polyamide PTBAs averaging at 9.5 N/cm^2 . This value is comparable to the highest adhesion forces achieved with the other tested adhesives.

Such differences in the adhesion forces can be explained by the different mechanisms of the adhesives. Whereas the other tested adhesives were physically setting, the store-bought super glue was a chemically curing adhesives. Since there was not too little solvent to evaporate and OH^- was not a limiting factor, the adhesive set over approximately 5 minutes.

In comparison to the store-bought super glue, the synthesized Gum Arabic and glycerin adhesive resulted in significantly lower adhesion forces which were approximately ten times lower. This is partially due to the fact, that the Gum Arabic and glycerin adhesive was a physically setting adhesive and not a chemically curing one. The available equipment was not sufficient to synthesize a chemically curing adhesive.

With the Gum Arabic and glycerin adhesive, the wooden PTBAs adhered the best, followed by polyamide, paper and aluminum. With the store-bought super glue, the wooden PTBAs adhered the best followed by aluminum, paper, and polyamide. Wood adhering the best can generally be attributed to its rougher surface and many possibilities to create hydrogen bonds with other molecules, due to their many OH-groups.

7 Materials and Methods

This chapter concerns itself with all the different experiments and methods used in order to synthesize and analyze the different adhesives. Additionally, it lists which equipment and which chemicals are required in order to perform the experiments. This chapter also provides the procedures used to synthesize the different adhesives.

7.1 Materials Used for the Synthesis of Starch Paste

For the synthesis of Starch Paste the following chemical substances were used:

Potato starch; Water; Salicylic acid

The Laboratory equipment used for the synthesis:

Beaker, 500 mL; Beaker, 250 mL; Heating plate; Measuring Cylinder 100 mL; Spatula; Analytical Balance

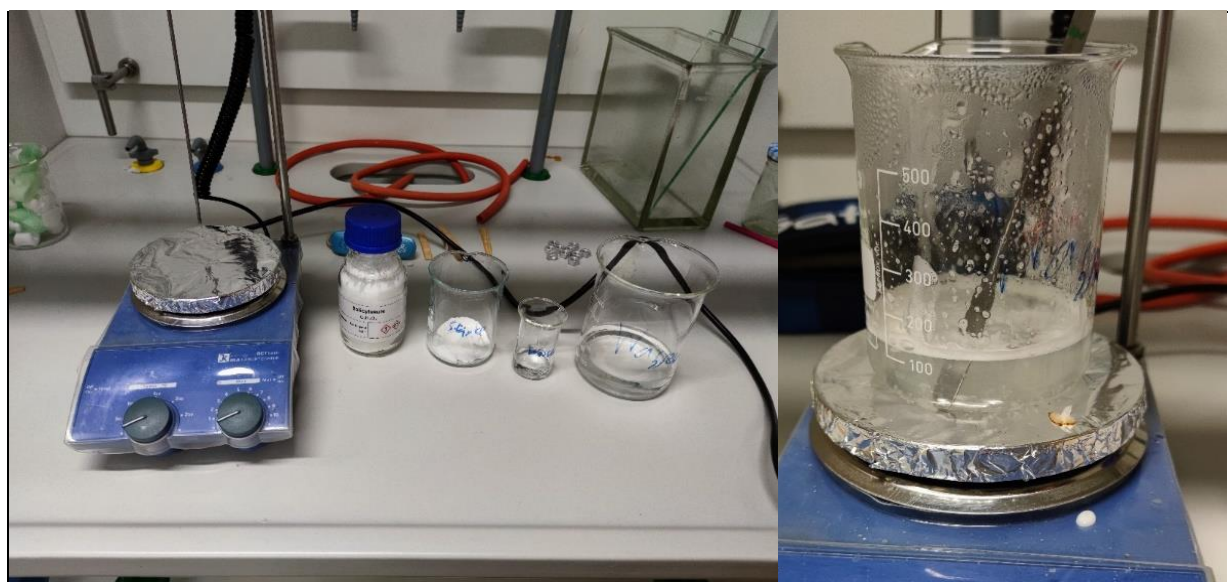


Figure 32: All materials used for the creation of this adhesive (left); the starch-water mix dissolved in heated water, creating a starch paste (right).

Method:

1. Potato starch (10 g; 62 mmol; 1.0 eq) was weighed on an analytical balance inside a 250 mL beaker and dissolved in cold deionized water (10 mL; 600 mmol; 10 eq), which was measured in a 100 mL measuring cylinder. It became a rather solid substance, that was very difficult to stir.
2. Deionized water (120 mL; 6600 mmol; 110 eq) was added to a 500 mL Beaker with the help of the 100 mL Measuring Cylinder and heated on the Heating plate, up until it started boiling.
3. The starch-water mix was dissolved in the boiling water and boiled, until it became a thick mass.

4. A little salicylic acid was added as a preservative, in order to be able to use the adhesive for a longer period of time without having to worry about microorganisms destroying it. [4] [16]

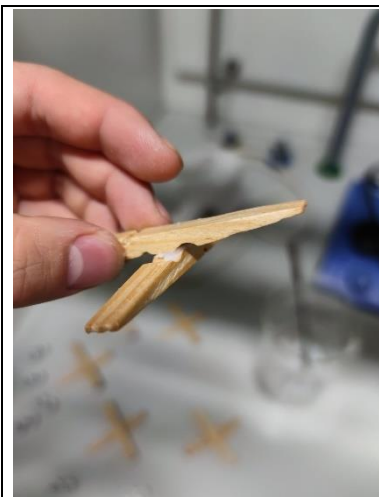


Figure 33: The starch paste adhering to two wooden clothespins.

7.2 Materials Used for the Synthesis of All-Purpose Adhesive Made with Polyvinyl Alcohol

For the synthesis of this type of all-purpose adhesive the following chemical substances were used:

Polyvinyl alcohol; Deionized water

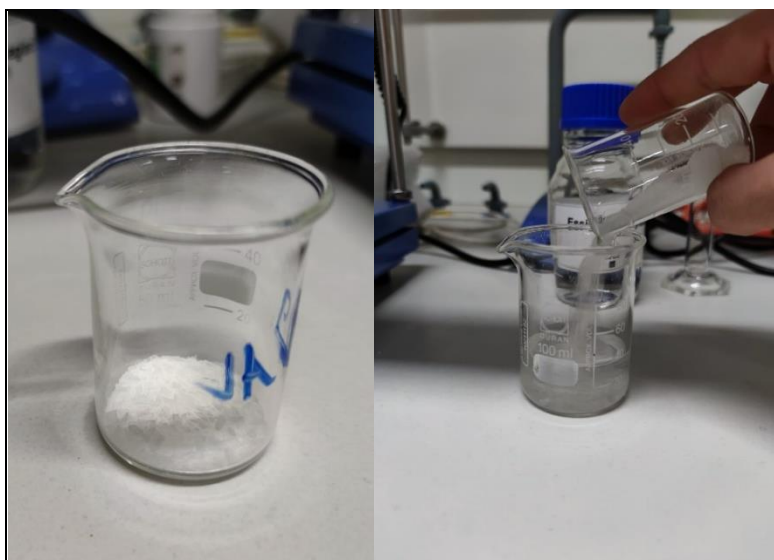


Figure 34: Polyvinyl alcohol in a beaker, in form of crystal shards (left); polyvinyl alcohol being poured into deionized water (right).

The Laboratory equipment used for the synthesis:

Beaker, 100 mL; Spatula; Heating plate; Measuring Cylinder, 50 mL; Analytical Balance

Method:

1. Deionized water (50 mL; 2800 mmol; 40 Eq) was heated in a beaker at 85 °C.

2. Polyvinyl alcohol (2.99 g; 68 mmol; 1.0 Eq) was added to the water and dissolved through slow stirring.

3. The solution was cooled down before the tests were conducted. [16]

7.3 Materials Used for the Synthesis of All-Purpose Adhesive Made with Polyvinyl Acetate

For the synthesis of this type of all-purpose adhesive the following chemical substances were used:

Polyvinyl acetate; Ethyl acetate

The Laboratory equipment used for the synthesis:

Beaker, 100 mL; Spatula; Heating plate; Measuring Cylinder, 50 mL; Analytical Balance



Figure 35: 7.99 g of polyvinyl acetate being weighed for the creation of all-purpose adhesive (left); 30 mL Ethyl acetate being poured into the polyvinyl acetate, before being heated up (right).

Method:

1. Polyvinyl acetate (7.99 g; 93 mmol; 1.0 Eq) was slowly dissolved in ethyl acetate (30 mL; 310 mmol; 3.3 Eq). The temperature was kept lower than 60 °C since ethyl acetate has a low boiling point.

2. The dissolved polyvinyl acetate was cooled down and left standing for some time. ^[16]

7.4 Materials Used for the Synthesis of Hotmelt Adhesive

For the synthesis of this type of hotmelt adhesive the following chemical substances were used:

Phthalic anhydride; Ethanediol; Boiling chips

The Laboratory equipment used for the synthesis:

Beaker, 100 mL; Spatula; Bunsen burner; Pipette; Analytical Balance; Test tube

Method:

1. Phthalic anhydride (1.54 g; 10 mmol; 1.0 Eq) was added to a test tube.
2. Ethanediol (1.0 mL; 18 mmol; 1.8 Eq) was then added to the same test tube.
3. The test tube was heated over the Bunsen burner, until the solution cleared up, and the phthalic anhydride dissolved itself in the ethanediol.
4. Two boiling chips were added.
5. The test tube was heated until it attained a slight yellow touch.
6. The adhesive was immediately applied before it could cool down. ^[16]



Figure 36: Mix of phthalic anhydride and ethanediol being heated over a Bunsen burner (left); the created yellowish hotmelt adhesive in a heated-up beaker, for it to remain in a liquid state (right).

7.5 Materials Used for the Synthesis of Super Glue

For the synthesis of this type of super glue the following chemical substances were used:

Gum Arabic; Glycerin; Deionized water

The Laboratory equipment used for the synthesis:

Beaker, 250 mL; Spatula; Analytical balance; Measuring cylinder, 100 mL; Pipette; Heating plate

Method:

1. Gum Arabic (24.77 g; 99 mmol; 1.0 Eq) was mixed with glycerin (15 mL; 210 mmol; 2.1 Eq) and deionized water (5 mL; 280 mmol; 2.8 Eq).
2. This mixture was stirred under 40 °C for around 20 minutes.
3. The adhesive was cooled down and then put to use. ^[16]

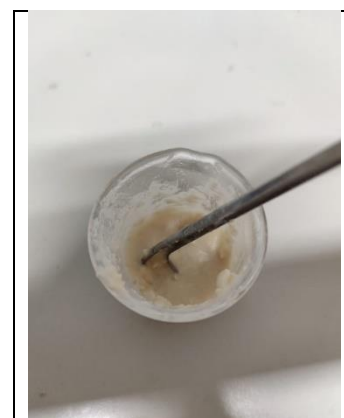


Figure 37: The created super glue. It is a yellowish, sticky mass.

8 Sources of Figures

Title page: <https://www.ecoenclose.com/blog/ecofriendly-adhesives-in-the-world-of-sustainable-packaging/> [accessed on 03.02.2022]

Figure 1: [1]

Figure 2: [1]

Figure 3: [2]

Figure 4: [2]

Figure 5: <https://www.differencebetween.com/difference-between-anionic-and-vs-cationic-polymerization/> [accessed on 26.01.2022]

Figure 6: [2]

Figure 7: <https://de.wikipedia.org/wiki/Polyaddition> [accessed on 03.02.2022]

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Figure 10: [1]

Figure 11: https://en.wikipedia.org/wiki/Hot-melt_adhesive [accessed on 04.01.2022]

Figure 12: <https://www.weicon.de/kontaktklebstoffe#> [accessed on 04.01.2022]

Figure 13: [1]

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All other figures have been created by me.

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