Bachelor`s Thesis

Mechanical Recycling of Bulk Molding Compound (BMC) Thermoset Biocomposites (Composite Inception)

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List of Terms

Term	**Meaning**
New (bio) composite	It is a composite that was created and tested during this Research.
New mixture	It is a mixture from which new (bio) composite was made.
Previous group	It is a group that was working on the same topic and on which results this Research was based.
Previous research (Minor Biobased Building: Structural Health)	It is the Research that was made by the previous group.
“Bridge parts”	It is BMC thermoset (bio) composite from which pedestrian bridge was made and that was used as a recyclate in this Research.
NPSP Company	NPSP company is the company that has requested this Research from HZ University of Applied Sciences Internet: www.npsp.nl
Initial material	In this Research the initial material is referred to as “bridge parts” from which filler for the new material was made.
Initial mixture	It is the mixture that NPSP company used for their NABASCO mixture.

List of Acronyms

Acronym	Meaning
BMC	Bulk molding compound
EU	European Union
FRP	Fibber reinforced composites
DMC	Dough molding compound
SEM	Structural equation modeling

Research Report

1. Introduction

1.1. Background and Context

Bulk molding compound (BMC) thermoset (bio) composites are difficult to recycle because they cannot be melted for reprocessing. However, this group of materials is often used in high-end applications where strength and lightweight materials are required, such as boat hulls, aircraft parts, car parts, windmill blades and others. At the end of the life cycle of these products there is no solution for recycling.

Waste management is now a high priority within the European Union (EU). Concern for the environment, both in terms of limiting the use of finite resources and the need of managing waste disposal, has led to increasing pressure to recycle materials at the end of their useful life. So far composite waste has been disposed in a landfill. A recent EU direction is to reduce the amount of organic material landfilled. As a consequence, it is already illegal to landfill composites waste in many EU countries. Therefore, there is a need for recycling routes for composite to be established and potential technologies to be developed.

1.2. Problem Statement

The current solution for the BMC thermoset (bio) composite issue is burying it as a landfill or burning it to generate energy and there is no recycling solution. And due to the recent EU directions of stopping landfill composites waste because it damages the environment, recycling methods should be developed. A new approach in relation to this topic was used: mechanical recycling by shredding of recyclate and its addition as a filler for the new bio composite. This approach showed a promising result as the newly created composite had slightly greater strength properties and was slightly more brittle. But it is clear that additional controlled testings are needed to help prove the properties of the material and potentially improve them by adjusting the mixture.

1.3. Research Questions

The analysis of this problem statement finds its expression in the following **Main Research Question:**

1.3.1. Main Research Question

What is the relation between the properties of the recycled BMC thermoset (bio) composite material and its mixture proportions?

For the purposes of this Research this Main Research Question shall be subdivided into the following **Research Sub-questions:**

1.3.2. Research Sub-questions

1. Can properties that are useful for the construction of the recycled composite material be adjusted and targeted by adjusting the mixture proportion?
2. What are the properties useful for the material used for civil engineering?
3. In what structures are composite materials used in the Netherlands?
4. What is the amount of composite materials used in those structures?

5. What are the possible applications for the building industry of recycled bio-composite material made?

6. What is the best method for recycling BMC thermoset (bio) composite into filler for a new composite in lab-scale?

1.3. Hypothesis of this Research

The following Research Objective and Research Questions find their expression in the following Working Research Hypothesis: It is expected to find the relation of the mixture of the recycled composite and its properties important for this type of material in civil engineering.

1.4. Methods of this Research

The theoretical basis of this Research are articles about thermoset BMC bio composites, their analysis aimed at looking for relevant information. The methodological basis of this Research is a recycling approach by grinding the initial material into powder and using it as filler for new composite, conducting tests on new made material to study its properties.

1.5. Secondary and Primary Data

Informational and statistical data was collected from articles, e-books and web pages on thermoset BMC bio composites, as follows.

- Information, received by the Author of this Research during NPSP company visits, during execution of this Research.
- Information, received by the Author of this Research during studies of the previous research.
- Information, received by the Author of this Research working on grinding the initial material, making new material and performing test on it.
- Information, received by the Author of this Research analysing results of the conducted tests.
- Information received by Author of this Research on the meetings with the supervisor of this Research.

1.4. Relevance and Importance of the Research

The results of this Research can lead to more deep knowledge in relation of mixture proportions of BMC thermoset composites and their properties. It can lead to finding the most desirable properties of the material that can perform as good or even better than the initial material.

As it was previously stated there are no proper end life solutions for the composite. One of the currently developed approaches in relation thereto is the end life solution used in this Research, but it is not fully developed yet and this Research can improve knowledge of grinding the initial material into powder to be used as a filler.

Hence, if this Research shows promising results in properties of new-made composite this method of recycling can be a proper environment-friendly solution. That shall lead to a much larger use of this full bio material in different fields including civil engineering.

The development of a proper recovering method can be beneficial to companies that nowadays use this material for their products like NPSP, because the biggest problem of this material is the lack of end life solution now.

Future studies can focus on finding on improving the mixture formula and finding a solution for grinding the material for production scale based on the findings of this Research.

1.5. NPSP Company

NPSP company is the company that has requested this Research from HZ University of Applied Sciences. And their material is used as a filler for recycled material namely NABASCO and "bridge parts" (fully bio bridge was built from that material). And their politicy is to use biobased and circular raw materials as much as possible. They can be recycled circularly after a long lifespan. Therefore, this Research is aimed at recycling of BMC thermoset bio composite (NABASCO and "bridge parts") as requested, because currently there is not a developed way of its recycling. NPSP develops and supplies innovative environmentally friendly composite materials and products for public space, construction, design, and mobility.

2. Theoretical Framework

2.1. Concepts

Concepts are topics that were created based on the above-mentioned Research Questions to be used for Chapter **2.2 Literature Review** to find out more information on this topic and understand it in detail.

These are the following Concepts:

1. BMC Thermoset bio composite
2. Existing recycling methods
3. Working principles of the ingredients
4. Possible and available bio ingredients on the market
5. The production process of composites material
6. Existing applications on the market of the BMC thermoset bio composite
7. Valuable properties of the material for civil engineering of the BMC thermoset bio composite

2.2 Literature Review

2.2.1. BMC Thermoset Bio Composite

BMC thermoset bio composite is a part of a larger group of Fibber Reinforced Composites (FRP). Composite at a basic level is a material that is composed of at least two elements working together, usually matrix (resin) and reinforcement (fibres) to produce material properties that are different to compare to the properties of the element on their own. BMC stands for Bulk Molding Compounds and it is a method where the matrix used as a bulk material and fibre reinforcement is added to increase strength and stiffness (Custom Formulated Sheet & Bulk Molding Compounds. Composites Intersessional, 2021). Fibre lengths and type can be customized which results in various strength characteristics (Robert et al., 2014). Thermoset polymers in particular are polymers that are cured into a solid form and cannot be returned to their original uncured form, those thermoset matrices are strong and have very good fatigue strength, but they are extremely brittle and have low impact-toughness making (Guide to composites, 2021). The term “bio” is used here to denote that the reinforcement and matrix are bio-based (Robert et al., 2014).

2.2.2. Existing Recycling Methods

“For BMC thermoset composites there are fundamentally two categories of the process: those that involve mechanical comminution techniques to reduce the size of the scrap to produce recyclates; and those that use thermal processes to break the scrap down into materials and energy (Pickering, 2006).” The biodegradability for bio for BMC thermoset bio composite is also a possibility by introducing materials that are improving biodegradability of the composite, by using bio-based plastics and natural fiber composites (Eddie et al., 2013).

2.2.3. Working Principals of the Ingredients

Overall, the properties of the composite are determined by four main factors: (1) the ratio of fibre to resin in the composite (Fibre Volume Fraction), (2) the geometry and the orientation of the fibres in the composite (3) and the properties of fibre and (4) properties of resin (Guide to composites, 2021).

It is important to emphasise the following about the Fibre Volume Fraction: In general, the higher the fibre volume fraction is, the higher will be the mechanical properties of the resultant composite, because the mechanical properties of the fibres are much higher than those of resins. But there are limits here, because for the fibre to be effective it should be fully covered in resin, so there is upper limit for ratio of fiber to resin generally with a range from 30 – 40% up to 70% (Guide to composites, 2021).

The geometry of the fibers in the composite is also important since having their highest mechanical properties across their length, then across their widths. This leads to the highly anisotropic properties of composite (Guide to composites, 2021).

The type of resin affects the various properties of the composite, including water resistance and degradation, resistance to other environmental degradation of the material, toughness, resilience and other mechanical properties (Guide to composites, 2021).

Fibres have two main factors that affect the composite: the basic mechanical properties of the fibres themselves, the surface interaction of fibre and resing (adhesive properties of resin also have an effect here). The higher those parameters are, the better properties the composite will receive (Guide to composites, 2021).

2.2.4. Possible and Available Bio Ingredients on the Market

There are three main components in the composite that are essential: matrix (resin), reinforcement (fibres) and filler. In this section, several of the most popular biomaterials in those three categories are taken into consideration, as well as additives that can be added to the mixture to further improve different qualities of composites.

The first main component is resin that is used to transfer stress between the reinforcing fibres, act as a glue to hold the fibres together and protect the fibres from mechanical and environmental damage (Composites lab, 2021). There are several different types available on the market, such as bio-based phenolic resins, bio-based epoxy, bio-based polyurethane, cellulose acetate, biopolyesters, biopolyolefins, etc. As an example of how those resins are made bio, the sustainable level of phenolic resins has been enhanced by both replacing petroleum-based phenol with biophenols and substituting carcinogenic formaldehyde with sugar-based furfural / hydroxymethylfurfural (Thabang et al., 2017).

As for fibres with the primary function to carry the load (Composites lab, 2021), there is even more of a variety: sugarcane bagasse, curaua, flax, hemp, jute, sisal, kenaf, Jute fabric, ramie-cotton fabric, and jute-cotton fabric. They are known to have high tensile strength and can be effectively used for composites (Thabang et al., 2017).

Fillers are used to improve performance and reduce the cost of a composite by lowering the compound costs of the significantly more expensive resin and imparting benefits, such as shrinkage control, surface smoothness and crack resistance. And there are also possible fillers like calcium carbonate, extracted cellulose fibers and cellulose nanowhiskers (Composites lab, 2021). Calcium carbonate was shown to increase mechanical properties, hardness and thermal properties (Fombuena et al., 2014). And cellulose fibres and cellulose nanowhiskers was shown to to increase the mechanical properties in wide variety of polymers. (Thabang et al., 2017).

Additives and modifier ingredients expand the usefulness of polymers, enhance their process ability or extend product durability. Those additives can be thixotropes, pigments&colorants, fire retardants, suppressants, UV inhibitors&stabilizers, conductive additives, release agents. This can bust different properties of the composite and make the production process easier (Composites lab, 2021).

2.2.5. The Production Process of BMC Thermoset Composites Material

The production process for BMC thermoset composite generally starts from winding / draping / forming, during which the fibres are placed in the desired lay-up configuration (this step can be neglected if necessary). The next step is filling or consolidation, where the reinforcement is impregnated with resin or the pre-impregnated material is consolidated (mixing) to reach the desired levels of fibre volume fraction and thickness. And the final step is curing during which the part undergoes the crosslinking reaction initiated by heating following cooling (Struzzieroa et al., 2019). During curing for BMC composites the mold set is mounted in a hydraulic or mechanical molding press and the molds are heated from 120°C to 200°C. A weighed charge of molding material is placed in the open mold. The two halves of the mold are closed and pressure is applied. The curing time ranges from less than a minute to about five minutes. After curing, the mold is opened and the finished part is removed (Composites lab, 2021).

2.2.6. Existing Applications of BMC Thermoset Bio Composite on the Civil Engineering Market

Fiber-reinforced plastic (FRP) composites have many applications in civil engineering, including crating, fenceposts, signposts, handrails, trashracks, baaring supports, baaring plates, ladders small pipas, cable tray racks & pipa hangers, light posts, sluice gates, sawer pipas, mechanical & electrical parts, electrical isolation structural members, noncritical load-bearing structures, culverts at small pump stations, culverts at levee outlet structures and light-gauge sheetpile. And there are even more possible applications that shall be developed. Engineering and design composite materials for civil engineering structures, 1997).

BMC thermoset composites so far take a small part in those applications. Illustrating examples can be door skins, fencing, roofing and window panels. Even more, those types of the composite can maintain critical physical properties under exposure to stressful conditions, thermoset composites for construction. They can also maintain an attractive appearance over time under exposure to stressful conditions (Market-construction, kitchen, and bath, 2021).

Bio composites of this group have been just recently developed and they are not even as common as their counterparts, but several companies (e.g. NPSP company) use prefabricated facades, road signs, outdoor sofas, bathroom floors for student houses and even constructed bio composite bridge with it (NPSP, 2021). That shows this material can be used for multiple purposes. Bio counterparts of BMC thermoset composite have almost the same properties and even so composite is still not used widely. And hopefully it will find more use with the development of proper recycling methods in the future.

2.2.7. Valuable Properties of the Material for Civil Engineering of the BMC Thermoset Bio Composite

There are many properties that are important for the material used in civil engineering most notably: physical (shape, size, density, specific gravity, etc.,), mechanical (strength, elasticity, plasticity, hardness, toughness, ductility, brittleness, creep, stiffness, fatigue, impact strength, etc.,), chemical (corrosion resistance, thermal degradation point, etc.,), optical (colour, light reflection, light transmission, etc.,), acoustical (sound absorption, transmission, and reflection), physiochemical (hygroscopicity, shrinkage, swell due to moisture changes water retention) and others (properties of building materials and their importance in construction, 2021).

The properties at which BMC thermoset composite excels are the following: heat resistant, corrosion-resistant, flame resistant, and being low shrink materials, dielectric and electrical insulators (Composites international, 2021). Especially, it excels at the ability to take the heat in a variety of demanding applications in which BMC thermoset composites have the highest-ever temperature rating in the thermoset polyester composites industry (Paul, 2009).

2.2.8. Preliminary Conclusion of this Chapter

The literature review shows that a mixture and ingredient of BMC thermoset biocomposite is responsible for its properties that leads to the conclusion that it is possible to adjust properties of the composite changing percentage of the filler in the composite.

It is also significant to notice that this composite can be used in the civil engineering field. And if the end life solution can be found here, it will lead to more use of this biomaterial in the future.

3. Methodology

The objective of the Research was to make the plates from (BMC) thermoset biocomposites from recicaled (BMC) thermoset biocomposites using it as a filler for new composite. Then perfroming test that has been chosen based on properties that are important for civil engenering and posibility to compare the results with the previous group that made the same material and tested it (Jimmy et al., 2021).

The detailed explanations about this process are discribed in this Capter **3. Methodology** as follows:

3.1. Method Updated

In this Chapter the following is explained in detail: (1) the reasons why the new method was developed and (2) how new mixtures are recalculated.

3.1.1. Changes in Research Proposal

The percentage of the filter used for the material in this Research first was based on the mixture percentage from the previous research. There were 3 mixtures with 42, 50 and 60 present filler. More detailed information on the amount of the ingredients used for one plate and for 100 g for those mixtures can be found in **Appendix A**.

Due to difficulty with grinding that has been unexpectedly harder and the failure of one recipe (as it will be shown in **Chapter 3.10.2 Failure of Recipe**) approach for deciding on mixture percentage and number of mixtures has changed (as it will be explained in further chapters).

Tests

A the beginning several different tests were considered to be performed on the material including (1) 3-point flexure test, (2) density test, (3) flame test and (4) DSC (differential scanning calorimetry). More detailed information on those tests including (1) parameters to be measured, (2) ISO norm, (3) the reasons why the test was chosen, (4) dimensions of the test samples according to the norms, (5) the number of the test samples according to the norms, (6) the number of the plates used per mixture and (7) the place where the test was conducted. This information can be seen in **Appendix B**.

Only two tests were made due to the lack of time and impossibility to compare those tests results with the research of the previous group. Only (1) 3-point flexure test and (2) density test that were also performed by the previous group were conducted (Jimmy et al., 2021).

3.1.2. Failure of the Recipe

After realizing how difficult and time consuming it was to grind the material it was decided to do two mixtures: one mixture with 60% and the other mixture with 50% amount of filler in g. Those two mixtures were tested by the previous group, but the number of plates used were fewer than 3 and results were confusing, because the performance of 50% mixture was worse than 42% and 60%. And therefore, it was decided to use those percentages and compare the results with a previous group (Jimmy et al., 2021).

But after understanding that while mixing the material with 60% the mixture looked not as it should look like, not as basic NABASCO recipe looks (as it is shown in Illustration 1), it looked more like soil comparing to NABASCO looking like clay. It was decided still to continue and heat press the plate after letting the mixture rest for two days in the fridge (as that improves the quality of the product), but after heat pressing it was clear that the plates had problems. They did not fully harden and there were some spots (gray parts) where there was not enough resin, so the plate could be broken by hands. Such a plate can be seen on Illustration 2 and an example of braking can be seen in the bottom right corner. After contacting NPSP company it was figured that problem was that relation of filler to resin was not correct and that mixture should have a ratio of around one to one of filler to resin and 60% recipe had almost 2,5 times more filler than resin. Therefore, the percentage of fillers was recalculated once more using the new approach that is shown in Chapter **3.2. General Approach.**

Illustration 1: 60% Mixture that was a Failure.

Source: Illustration made by the Author of this Research, 2021.

Illustration 2. 60% Mixture Plate that was a Failure.

Source: Illustration made by the Author of this Research, 2021.

3.2. General Approach

The first percentage of "bridge parts" filler was established. For that purpose, the first density of each ingredient used was found out. Then each ingredient volume was found out from the initial mass of each ingredient, from the initial mixture. Then the relation between the volume of filler was chosen (based on initial mixture repletion) and on the basis thereof the density mas in grams of filler was calculated. And from that and total mass of mixture percentage of filler in the mixture was calculated. This is shown in more detail in the following Chapter **3.10.4. Approach of Calculation of the Percentage of "Bridge Parts" Filler.**

3.2.1. First Method of Calculation

Initially while calculating to find out the amount of the filler needed another approach and the percentage of the filler in the mixtures were analysed. That previous approach turned out to be less precise and, therefore, the new approach that is described in following chapters was used. The previous approach and its results can be seen in **Appendix C**.

3.3. Dimensions of the Plate and Mold

The dimensions of the mold (as it can be seen in Table 1) are known and from that dimension of the future plates are assumed.

Table 1: Dimensions of the Plate/Mold and their Volume.

Plate dimensions of the mold	
h (mm)	3
l (mm)	160
w (mm)	140
Volume of plate (mm^3)	67200
Volume of plate (cm^3)	67,2

Source: Experimental trials conducted by the Author of this Research, 2021.

3.4. Approach of Calculation of the Percentage of "Bridge Parts" Filler

"The volume approach" was used to calculate the amount of ingredients in g per plate. Each ingredient volume was found out using the density of ingredients from the initial mass of each ingredient, from the initial mixture. Then from the volume of mold and total volume of ingredients volume of each ingredient required was calculated. And from that and the density of each ingredient, their mass in g was calculated. This is shown in more detail in Chapter **3.8. Needed amount calculation.**

3.5. Recipe With Calcite as Filler

The mixture that was used is the mixture of NABASCO from NPSP can be seen in Table 2. For testing the production method first calcite mixture shall be made and tested. The results of the tests shall be compared to the properties provided by NPSP.

Table 2: Amount of Material in g Needed for 100g of Calcite as a Filler Mixture.

Recipe in g with calcite as filler per 100g				
Resin (Hars)	**Harder (Peroxide)**	**Losmiddel (Zinkstearaat)**	**Riet (Vezel)**	**Vulstof (Calcite)**
28,26	0,41	1,43	9,9	60

Source: NPSP company.

3.6. Density of the Ingredients

The density of ingredients used to calculate the mixture is shown in Table 3 as follows.

Table 3: Densities of Ingredients in g/cm³.

Density of ingredients						
Ingredient	**Resin (Hars)**	**Riet (Vezel)**	**Filler (Bridge Parts)**	**Filler (Calciet)**	**Harder (Peroxide)**	**Losmiddel (Zinkstearaat)**
Density (g/cm^3)	1.4	1.4	1.13	2.71	1.45	1.1

Source: Experimental trials conducted by the Author of this Research, 2021.

3.7. Calculating the Percentage of "Bridge Parts" Filler

In this Chapter, all the ingredients in mass and volume are the same for all mixtures except for filler and only the amount of filler is changing.

In Table 4 initial mixture (NABASCO) in grams and volumes can be seen. Based on those relations all-new mixtures with new percentage of filler were calculated.

It is shown in Table 4 as follows.

Table 4: Ingredients of the Initial Mixture in Mass and Volumes.

Initial mixture (NABASCO)					
Ingredient	Resin (Hars)	Riet (Vezel)	Filler (Calciet)	Harder (Peroxide)	Losmiddel (Zinkstearaat)
Mass (g)	31.79	11.13	67	0.46	1.6
Volume (cm^3)	22.70714286	7.95	24.72324723	0.317241379	1.454545455

Source: Experimental trials conducted by the Author of this Research, 2021.

Table 5 shows the volume repletion of filler to the resin of initial mixture and new mixtures (Recipe 1 – 3) used to calculate the amount and percentage of the filler in the new mixture. And Recipe 4 is a failed mixture described in Chapter **3.1.2. Failure of the Recipe**.

Table 5: Volume Repletion of Filler to Resin.

Volume relations	
Mixture	**Volume relation**
Initial mixture	1.09
Recipe 1	1.07
Recipe 2	0.55
Recipe 3	1.49
Recipe 4	2.63

Source: Experimental trials conducted by the Author of this Research, 2021.

In Table 6 the filler amount in g and % to the mixture of new mixtures is shown used to calculate the amount of ingredients of the new mixture for one plate.

Table 6: Filler Amount in g and % to the Mixture of New Mixtures.

Filler amount		
Mixture	**Amount (g)**	**%**
Initial mixture	24.72	60
Recipe 1	24.4	38
Recipe 2	12.6	24
Recipe 3	33.9	46
Recipe 4	59.7	60

Source: Experimental trials conducted by the Author of this Research, 2021.

3.8. Needed Amount Calculation

The amount of each ingredient for each mixture for one plate was calculated using the filler amount in g and % to the mixture of new mixtures found in the previous chapter and method described in Chapter **3.10.9. Calculation of Percentage of "Bridge Parts" Filler.**

3.9. Calculation of Amount of "Bridge Parts" Filler

To calculate percentage of "Bridge Parts" Filler the volume of mold (Vm) of 67,2 cm^3 (140mm - 160mm - 3mm) was used find the total present volume (Vp) of each mixture and multiply each ingredient mass by deference in them (d). In Table 7 calculation can be seen as follows:

Table 7: Calculation of Differences in Volumes of Each Mixture and Volume of the Mold.

Difference in volumes		
Mixture	**Formula**	**Result**
Recipe 1	d1 = Vm / Vp1	1.18
Recipe 2	d2 = Vm / Vp2	1.491
Recipe 3	d3 = Vm / Vp3	1.01
Recipe 4	D4 = Vm / Vp4	0.73

Source: Experimental trials conducted by the Author of this Research, 2021.

Then from the difference in volumes (d) mass of each individual ingredient needed to make one plate for each mixture can be calculated using Formula 1.

Mif = Mi * d

Formula 1: Calculation of final mass of each ingredient of each mixture for making one plate.

Where:

Mif – the final mass of each ingredient in each mixture

Mi – the mass of each ingredient in initial mixtures

d – the difference in volumes of the corresponding mixture

Using this formula mass of each ingredient of each mixture for making one plate was calculated. The results thereof can be seen in Table 8.

Table 8: Mass of Each Ingredient of Each Mixture for Making one Plate and its Total Mass.

Mass of each ingredient of each mixture and total mass of each plate (in g)						
Mixtures	**Resin (Hars)**	**Riet (Vezel)**	**Filler (Calciet)**	**Harder (Peroxide)**	**Losmiddel (Zinkstearaat)**	**Total mass**
Recipe 1	37.6	13.2	32.6	0.54	1.89	85.8
Recipe 2	47.5	16.6	21.2	0.69	2.39	88.4
Recipe 3	32.2	11.3	38.8	0.47	1.62	84.4
Recipe 4	23.2	8.1	49.2	0.34	1.17	82.0

Source: Experimental trials conducted by the Author of this Research, 2021.

To make the mixture, the total amount of ingredients was calculated by multiplying the mass of each ingredient of each mixture for making one plate by the number of plates (4 in this case 3 for 3-point flexure test and Density test and 1 for Flame test) and by 1,05 to make sure that there shall be enough of mixture for making the required number of plates, because some amount can be lost in preparation and mixing process. In Table 9 the results of the calculation can be seen. All the amounts (in g) in this table are the exact amounts that were put in the mixture.

Table 9: Mass of Each Ingredient of Each Mixture for Making the Required Number of Plates.

Total mass of each ingredient of each mixture (in g)					
Mixtures	**Resin (Hars)**	**Riet (Vezel)**	**Filler (Calciet)**	**Harder (Peroxide)**	**Losmiddel (Zinkstearaat)**
Recipe 1	158	55	137	2.3	7.9
Recipe 2	199	70	89	2.9	10.0
Recipe 3	135	47	163	2.0	6.8
Recipe 4	97	34	207	1.4	4.9

Source: Experimental trials conducted by the Author of this Research, 2021.

To understand how much filler powder should be prepared, total amount of filler was calculated (Mft) by summing up the required amount of fillers of each mixture for 4 plates multiplied by 1,05.

Mft = 596 g

3.10. Preparing for Grinding the "Bridge Parts"

It is necessary to prepare the "bridge parts" for their grinding. The previous approach to prepare them can be seen in **Appendix D**. And the developed methods for preparing the "bridge parts" can be seen in Chapter **4.1. "Bridge Parts" Grinding Methods**.

3.11. Grinding the "Bridge Parts"

The material-specific machinery is needed for grinding. Therefore, grinding was conducted at the NPSP company during a visit there. The developed methods for grinding the "bridge parts" can be seen in Chapter **4.1. "Bridge Parts" Grinding Methods**.

3.12. Making the Material

In this section general approach to making the material is described. The first step to make the material is to mix the resin (hars) and hardliner (peroxide). Then losmiddel (zinkstearaat) is added to the mixture and put in the mixer. Then while mixing one half of the recicalent (bridge parts) is added. And as the next step, after it is well mixed, the other half is added. Finally, when mixed fibres (riet) are added in thirds each time adding after being well mixed. After that the mixing material is put into the form that is preheated for around 170 C (after preheating losmiddel (zinkstearaat) was applied to mould to prevent sticking of the material). Then the form is put into the machine and heated at the temperature at around 170 C for around 5 minutes under the pressure of 80 bar. Then made material is taken out from the machine and let it cool down at room temperature. And then it is ready to be tested. This approach has been used previous group.

3.13. Cutting Plates into Samples

For 3-point flexure test material is required to have specific dimensions (l: 80 ± 2 mm, b: 10,0 ± 0,2 mm, h: 4,0 ± 0,2 mm) for those plates with dimensions of l: 160 mm, b: 140 mm, h: 3 mm in seven pieces from which five best shall be used. The plates shall be cut using water get.

3.14. Evaluation of Results

The results shall be evaluated by comparing the test results of each mixture between each other. As each result is important to understand and find the best performing mixture if possible or at least the mixture that performed best in each test and, if possible, find the ratio of mixture proportions (% of the amount of filter) and its properties.

3.15. Preliminary Conclusion of this Chapter

The amount of the ingredients in each mixture and the amount of filler that should be grind were calculated in this chapter. And following the calculation, it can be possible to make several different mixtures and try to find out which of them can perform the best.

4. Results

4.1. "Bridge Parts" Grinding Methods

This Chapter describes the following: (1) what machinery (and its settings), grinding process and what input partial sizes were used for the purposes of this Research, (2) the most efficient approaches for those parameters for grinding the material in the lab scale and (3) how grinding can be upscaled to the production scale.

4.1.1. Machinery Grinding

In this Chapter the following is explained in detail: (1) machinery that was sued for making the powder from initial material and (2) its components.

Machine

The machine that was used to grind the material is Planetary Mono Mill PULVERISETTE 6 classic lines. This machine is suitable for grinding material for lab-scale material as it can grind a small amount of material very fast.

The Planetary Mono Mill PULVERISETTE 6 classic line is a high-performance Planetary Ball Mill with a single grinding bowl mount and practical, easily adjustable imbalance compensation.
At the same time, the PULVERISETTE 6 classic line is ideal for mechanical alloying or for mixing and perfect homogenizing of emulsions and pastes.

Bowl

There are many different bows that can be used for the machinery that can affect the performance of grinding of different materials. For the purposes of this Research the comparison of the bow used (it can be seen in Table 10) and a desirable bow is chosen. Desirable #1 bowl is taken from what was used in Fritsch experiment that has milled in 1 min 20 g of material with 67% (g) usable (under 125 µm), which is a much better result compared to the achieved results with the used bowl (the achieved result can be seen in **Chapter 3. Testing Different Settings and Particle Sizes**). It is necessary to test Desirable #2 bowl as balls from the same material are to be tested, as well. The reason for it is clarified in Chapter **Balls**. The volume of the bowl can be different, but the bigger the bowl is, the bigger amount of material can be input, the faster grinding process theoretically.

Table 10: Comparison between the Used Bowl and two Desired Bowls.

Comparison of Bowls			
	Used	**Desirable #1**	**Desirable #2**
Description	500 ml grinding bowl made of hardened, stainless steel (fe – cr) Useful capacity: 80 - 225 ml Abrasion resistance: good	80 ml grinding bowl made of zirconium oxide (zro2) Useful capacity: 10 - 30 ml Abrasion resistance: very good	250 ml grinding bowl made of hard metal tungsten carbide (wc) Useful capacity: 30 - 125 ml Abrasion resistance: very good
Suitable for	Hard, medium-hard, brittle samples	Fibrous, abrasive samples	Hard, abrasive samples

Source: Experimental trials conducted by the Author of this Research, 2021.

Balls

There are many different balls that can be used for the machinery that can affect the performance of grinding of different materials. For the purposes of this Research the comparison of balls used (it can be seen in Table 11) and desirable balls is chosen. Desirable #1 balls are taken from what was used in Fritsch experiment that has milled in 1 min 20 g of material with 67% (g) usable (under 125 µm), which is a much better result comparing to the achieved results with used balls (the achieved result can be seen in **Chapter 3. Testing Different Settings and Particle Sizes**). Desirable #2 balls are based on that mass of the balls has a direct impact on kinetic energy that balls are generating while moving (K = m*v2/2 /=> more mass more energy), therefore, balls with bigger density and the same diameter will generate more energy and, therefore, theoretically will be better for grinding the material. Thus, hard metal tungsten carbide with almost two times bigger density than used hardened, stainless steel should be also tried. The number of balls is based on the saithe of the bowl. The diameter can be different, but changing diameter the amount should be also be changed.

Table 11: Comparison between Used Balls and two Desired Balls.

Comparison of Balls			
	Used	**Desirable #1**	**Desirable #2**
Description	25 x 20 mm ø Fe-Cr grinding balls Material: hardened, stainless steel - Fe-Cr Abrasion resistance: good	5 x 20 mm ø zro2 grinding balls Material: zirconium oxide - ZrO2 Abrasion resistance: very good	10 x 20 mm ø WC grinding balls Material: hardmetal tungsten carbide - WC Abrasion resistance: very good
Suitable for	Hard, medium-hard, brittle samples	Fibrous, abrasive samples	Hard, abrasive samples
Density	7.7 g/cm³	5.7 g/cm³	14.3 g/cm³
Hardness	~ 60 HRC	1200 (HV10)	89.7 HRA

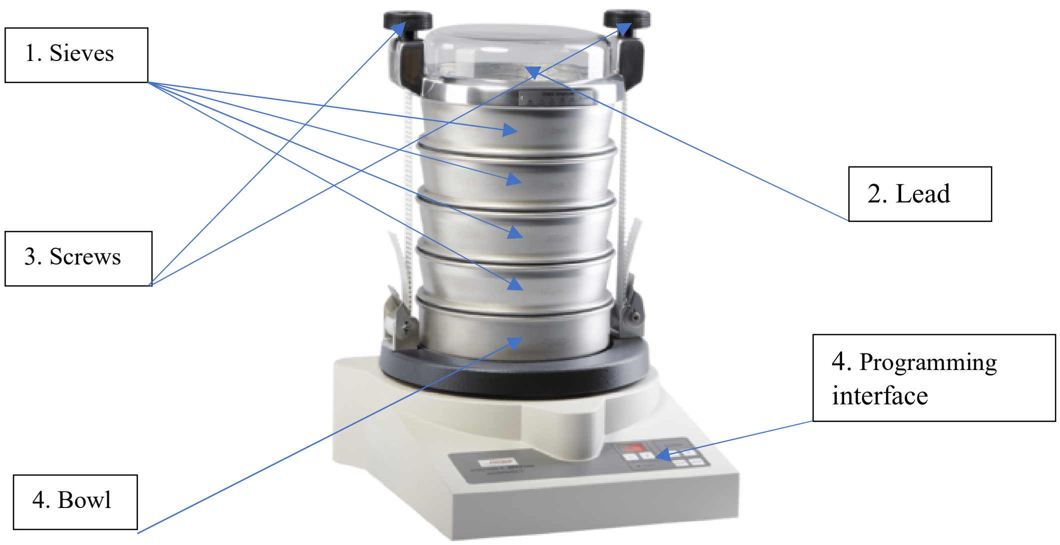
Source: Experimental trials conducted by the Author of this Research, 2021.

4.1.2. Machinery Sieving

Machine

Vibratory Sieve Shaker ANALYSETTE 3 SPARTAN for all typical sieving tasks in the laboratory with optical adjustment of the amplitude on the running instrument.
The ANALYSETTE 3 SPARTAN is suitable for quantitative particle size analysis of solids and suspensions of all kinds through dry or wet sieving with woven test sieves. It can be seen on Illustration 3.

Illustration 3: Sieve Shaker ANALYSETTE 3 SPARTAN.



Source: Picture taken by the Author of this Research, 2021.

Working Principal

The ANALYSETTE 3 is a "shaking sieve" system in the classic sense in which an electromagnetic drive causes the sieves to oscillate in a vertical direction. The material to be sieved is periodically propelled upward off the sieve fabric and is forced through the mesh of the attached test sieve as it falls back down.

Sieves

There are four different sieves used the sieving machine: (1) 500 µm, (2) 250 µm, (3) 125 µm and finally (4) 50 µm.

General Approach

First, put about 500 ml of grinded dust into top 1. Sieve. Then close the lid ad screw the 3. Screws until the lead cut rotates. Then turn on the machine and increase the frequency until 0,5 mm separated bars on the cape visually became single line. The machine is present for 2 minutes of work on 4. Programming interface. Then after the machine is done sieving turns of the screws and each sieve has its corresponding particle size. For the research, only particles of 125 µm or smaller were used.

4.1.3. Grinding Process

The whole grinding process was constructed using the same machinery described above. Therefore, all the results can be different when different machines or/and machinery equipment are used. All the steps mentioned below are specific to the machinery and machinery equipment used.

Working Principal

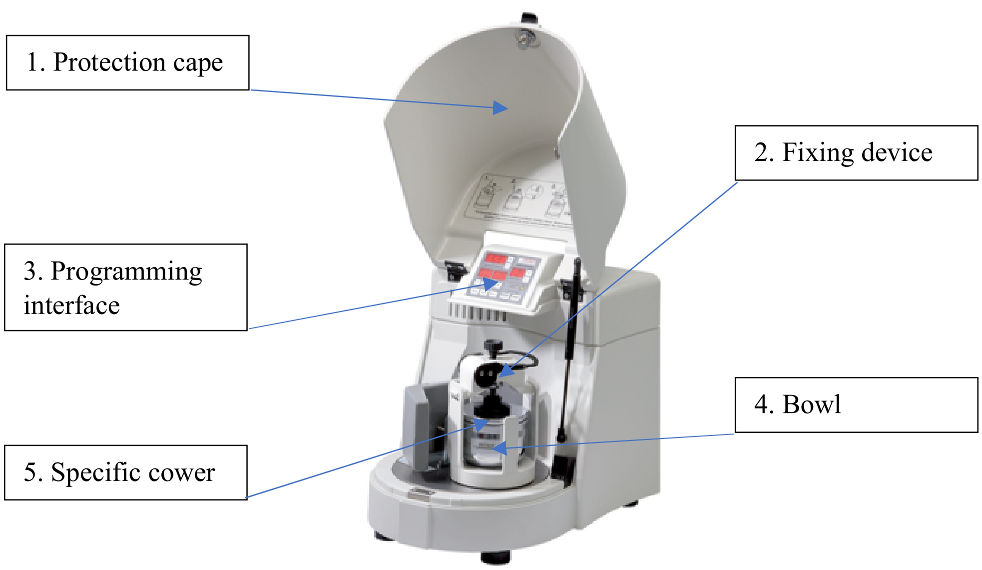
The comminution of the material to be ground takes place primarily through the high-energy impact of grinding balls. To achieve this, the grinding bowl, containing the material to be ground and grinding balls, rotates around its own axis on a main disk while rotating rapidly in the opposite

direction. At a certain speed, the centrifugal force causes the ground sample material and grinding balls to separate from the inner wall of the grinding bowl. Then the grinding balls cross the bowl at high speed and further grind the sample material by impact against the opposite bowl wall. In addition, the impact between the balls themselves on the sample material adds to the size reduction process.
The PULVERISETTE 6 classic line achieves a centrifugal acceleration of up to 29 g due to the enormous rotational speed of the main disk – up to 650 rpm.

General Approach

To grind the material first grinding balls are put in the bowl then in between 200 ml and 250 ml of material are put in the 4. Bowl. Then 4. Bowl is closed with the 5. Specific cower with rubber bent in between to prevent the escape of powder and get the better connection of bow and cower. Then bow is put in the machine and fixed with the 2. Fixing device. Then 1. The protection cape is closed, and the machine is tern on. The machine can be programmed on how many reps and for how long they should be and if rest in between is needed and how long it should be by using 3. Programming interface. Then after the machine is done 1. Protection cape can be open and the 4. Bowl can be put out. After that bowl is open and grinded material is put out using a sieving tool to separate balls and not grinded material from powdered material.

Illustration 4: Planetary Mono Mill PULVERISETTE 6 Classic Lines.



Source: Picture taken by the Author of this Research, 2021.

Rotation Speed

The grinding balls will distribute better on the entire grinding bowl surface and result in the best possible grinding effect. If the rotational speeds are too low, the grinding balls only remain in the lower area of the bowl or only move across the bottom to the inner radius of the bowl. The necessary grinding duration will be the briefest and, therefore, the unavoidable contamination and abrasion will be the lowest. With a low rotational speed, the grinding powers / energy of the grinding balls are much reduced, the grinding duration will be infinitely too long and the wear will enormously increase.

Amount of Rotations

The number of rotations was from 1 to 5 each taking one minute and with one-minute rest in between each rotation. Table 12 shows the number of rotations used and the time it takes to do the full grind. If while using the machinery on programming interface it says “repetitions” not “rotations”, thus, it is necessary to find how many rotations are completed, to find it, add one to the number of “repetitions”.

Table 12: Time of Grinding per Number of Rotations.

Time per Number of Rotations	
Number of rotations	**Time**
1	1
2	3
3	5
4	7
5	9

Source: Experimental trials conducted of the Author of this Research, 2021.

Heat Up

Due to the high grinding energy, the grinding system heats up quickly, the hot grinding bowl has to be cooled down, thus, the evolved steam pressure inside the grinding bowl does not release the suspension through the grinding lid seal.

4.1.4. Testing Different Settings and Input Particle Sizes and Shape

In this Chapter different settings and input particle sizes and shape of material to be grinded are clarified in detail. The most efficient settings and imput particle are found out.

Input Particle Sizes and Shape (Grinding)

Used particle size and shape also affect the result as much as bowl and balls used and settings applied. In this Chapter it will be clarified what particles size and shape was used and how it affected the grinding process.

In Table 13 the comparison of all the particle shapes and sizes and their grinding performance (best-found combinations of machinery settings and input amount are used) can be seen. It is to note that the speed on the machine used was 600 rpm for all input particle sizes and shapes.

Table 13: Comparison of all the Particle Shapes and Sizes and their Grinding Performance.

Input particle sizes and shape						
Particle shape and size	**Cubes and rectangles (0,5 – 1 x 0,5 – 1 x 0,5 – 1 cm^3)**	**Tubes and plates (0,2 – 0,3 x 0,2 – 1 x 0,5 – 1 cm^3)**	**Drill sawdust**	**Flakes planed (big)**	**Flakes planed (small)**	**Planed (very small)**
Illustrations						
Cutting and preparation process time for 100 g of material (min)	5 – 10 min	10 – 15 min	5 – 10 min	15 – 20 min	15 – 20 min	15 – 20 min
The amount that can be put in (g)	200 g	100 g	15 g	22 g	40 g	50 g
Amount of powder after	7 g	85 g	14 g	20 - 22 g	38 - 40 g	48 g

grinding (g)						
Rotations to fully grind (if possible)	4 (not fully grinded)	4 (not fully grinded)	2 (fully grinded)	3 (almost fully grinded)	3 (fully grinded)	4 (fully grinded)
Amount of “good” powder (particles smaller than 125 µm) (g)	4,2 g*[1]	22,6 g*[1]	9,4 g*[1]	6,9 g*[1]	16,7 g*[1]	24 g*[1]

*[1]These results are based on the sieving of powder from those materials. Sieving results can be seen in Chapter **Sieving Results.**

Source: Experimental trials conducted by the Author of this Research, 2021.

Testing of Different Settings (Grinding)

The objective of this Research was to find the most efficient way to grind the material. Efficient in that case is minimizing the time to grind and minimizing the waist (not “good” particle size (over 125 µm). For some input particle sizes and shapes, it was obvious that it did not perform in a sufficient way. Therefore, most of the tests were conducted on “promising” input particle sizes and shape: drill sawdust, flakes planed (big), flakes planed (small). As for cubes and rectangles (0,5 – 1 x 0,5 – 1 x 0,5 – 1 mm3) after 4 rotations it gives the smallest amount of powder and it is only 3 - 4% of input amount. It is not efficient to compare to the others. Both tubes and plates (0,2 – 0,3 x 0,2 – 1 x 0,5 – 1 mm^3) giving the biggest amount of powder after 4 rotations do make a lot of “good” particle size (under 125 µm) around 12 % of input amount and that is also not efficient to compare to others. In Table 14 testing of different partial sizes with different input amounts, rotations and powder results are shown.

There is no use of regrinding already grinded particles using this machinery, because from the first grind most of the material that can be grind/easily grind is already grinded. And regrinding it shall not be efficient.

It is significant to note that the speed on the machine used was 600 rpm for all the tests.

Table 14: Testing of Grinding of Different Particle Shape and Size on Different Number of Rotations with Different Amounts of Input Material.

Testing			
Particle shape and size	**The amount that can be put in (g)**	**Amount of powder after grinding (g)**	**Rotations to fully grind (if possible)**
Tubes and plates (0,2 – 0,3 x 0,2 – 1 x 0,5 – 1 cm^3)	90 g	73 g	5 (very badly grinded (too hot material starts to stick at the end))
	90 g	57 g	4 (lower quality than the 5 rotations (too hot material starts to stick at the end possible cause overheating of the bowl))
	100 g	64 g	5 (slightly worse that 90 g (too hot material starts to stick at the end))
	29 g	19 g	5 (almost fully grinded (too hot material starts to stick at the end))
Drill sawdust	15 g	10	4 (fully grinded)
	15 g	11	3 (fully grinded)
	15 g	13	2 (fully grinded)
	15 g	14	1 (not fully grinded)
Flakes planed (big)	22 g	18 g	2 (almost not

			grinded)
	22 g	22 g	3 (almost fully grinded)
	40 g	40 g	4 (not fully grinded)
Flakes planed (small)	35 g	32 g	2 (not fully grinded)
	35 g	34 g	3 (fully grinded)
	40 g	38 - 40 g	3 (fully grinded)
	40 g	38 g	4 (fully grinded (but no significant difference from 3 rot)
Flakes planned (very small)	40 g	38 g	3 (fully grinded)
	50 g	48 g	4 (fully grinded)

Source: Experimental trials conducted by the Author of this Research, 2021.

It is also important to note the following: Drill sawdust input amount can be larger (up to 50 g), because during its testing it was not compacted as it was done with flakes planed (very small) which significantly increased the amount that was put in. As the properties of both of them are quite the same and probably the sawdust is even more compatible that can increase input amount even more.

__**Sieving Results**__

In Table 15 the results of sieving of different input materials powder and sieving percentage are shown. All the servings were made in the same setting mentioned in Chapter **4.1.2. Machinery Sieving.**

Table 15. Results of Sieving of Different Input Material Powder with Calculation of Sieving Percentage.

Sieving results and Sieving percentage			
Material	**Input amount**	**Output**	**Sieving percentage (%)*2**
Cubes and rectangles (0,5 – 1 x 0,5 – 1 x 0,5 – 1 cm^3)	20 g	12 g	60
Tubes and plates (0,2 – 0,3 x 0,2 – 1 x 0,5 – 1 cm^3)	200 g	14 g (way too much everything is stack at the top sieve. Stack part can be seen on Illustration 5.)	7
	79 g	21 g (re-sieved)	26,5
Drill sawdust	Not tested due to lack of grinded material		67,3 Calculation based on relations (can be slightly different in reality)
Drill sawdust / Flakes planed (big) (50/50)	203 g	90 g	44,3
Flakes planed (big)	Not tested due to lack of grinded material		32,9 Calculation based

			on relations (can be slightly different in reality)
Flakes planed (small, big 50/50)	103 g	39 g	37,9
Flakes planed (small)	105 g	45 g	42,9
Flakes planed (very small)	Not tested due to lack of grinded material		50 (Estimation based on grinded powder quality)

*2Ii is percentage of usable powder to input powder on average.

Source: Experimental trials conducted by the Author of this Research, 2021.

It is important to mention sieving more than 100 g of powder is not recommended, because the material can be stuck on in the top sieve (especially if powder has visually many big partials (bigger than 500 µm, it can be seen on Illustration 5). In case it got stuck, separate the powder and regrind.

Illustration 5: Stack on the top sieve particles. Particles bigger than 500 µm.

Source: Picture taken by the Author of this Research, 2021.

“Good” and “Bad” Pre-Sieved Powder

In Table 16 the difference of “Good” and “Bad” pre-sieved powder is shown as follows.

Table 16: Comparison of "Good" and "Bad" Pre-sieved Powder.

"Good" and "Bad" pre sieved powder		
Comparison	**"Good"**	**"Bad"**
Illustration	 Small lumps can be seen in the illustration, they are stuck together due to the hit, but they can be sieved without problems.	
Description	Good powder almost does not have any partials that are bigger than 500 µm (it can be seen on Illustration 5)	Bad powder have many partials that are bigger than 500 µm (it can be seen on Illustration 5)
Sieving percentage	The sieving percentage of good powder is generally around 50%	The sieving percentage of bad powder is generally around 30%
Stuck and kluge possibility	Have a low chance of kludging top sieve. Allowing most of the partials to go through corresponding sieves through	Have a high chance of kludging top sieve and not allowing some of the usable powder to go through
How to get	Grinding flakes planed (small) or drill sawdust generally led to good output powder	Grinding cubes and rectangles (0,5 – 1 x 0,5 – 1 x 0,5 – 1 cm^3) or tubes and plates (0,2 – 0,3 x 0,2 – 1 x 0,5 – 1 cm^3) or flakes planed (big) generally led to bad output powder

Source: Experimental trials conducted by the Author of this Research, 2021.

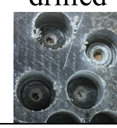
One of the goals of upscaling shall be getting good powder from grinding, because it is less wasteful and easier to sieve.

<u>Advantages and Disadvantages of Each Input Particle Size Based on Previous Data</u>

In Table 17 advantages and disadvantages of each input particle size based on previous data can be seen.

Table 17. Advantages and Disadvantages of Each Input Partials and their Preparation Process.

Advantages and disadvantages of each input particle						
Particle shape and size	**Cubes and rectangles (0,5 – 1 x 0,5 – 1 x 0,5 – 1 cm^3)**	**Tubes and plates (0,2 – 0,3 x 0,2 – 1 x 0,5 – 1 cm^3)**	**Drill sawdust**	**Flakes planed (big)**	**Flakes planed (small)**	**Flakes planed (very small)**
Process	First cutting strips with a width of about 1 cm and length of about 1 m from the “bridge par” on vertical band sawing machine (that is 1 cm thick), then cutting it into cubes of about 1 x 1 x 1 cm on metal band sawing machine	First cutting strips with a width of about 0,3 cm and length of about 1 m from the “bridge par” on vertical band sawing machine (that is 1 cm thick), then breaking it into tubes and plates (0,2 – 0,3 x 0,2 – 1 x 0,5 – 1 cm^3)	Darling the material with a drill with a drill for wood	Doing the same as for cubes and rectangles (0,5 – 1 x 0,5 – 1 x 0,5 – 1 cm^3) and then planing it using a big plane and plates with a width of >1,0 mm	Doing the same as for tubes and plates (0,2 – 0,3 x 0,2 – 1 x 0,5 – 1 cm^3). And then planing it using a small plane and plates with a width of 0,5 – 1,0 mm	Doing the same as for tubes and plates (0,2 – 0,3 x 0,2 – 1 x 0,5 – 1 cm^3) and then planing it using a small plane and plates with a width of 0,3 – 0,5 mm
Advantages	- Fast and easy to prepare - Good Sieving percentage[*2]	- Relatively fast and easy to prepare	- Fast and easy to prepare - Almost all the material is grinded - More than 50% makes usable powder	- Almost all the material is grinded - A bit less than 50% makes usable powder after sieving - No waisted material after cutting and	- Almost all the material is grinded - A bit less than 50% makes usable powder after sieving - No waisted material after cutting and preparation - Fully grinded after	- Almost all the material is grinded - A bit less than 50% makes usable powder after sieving - No waisted material after cutting and

			after sieving - After drilling powder that can be sieved is collected - Fast to grind fully (only 2 rotations)	preparation	3 rotations (40 g)	preparation - Fully grinded after 4 rotations (50 g)
Disadvantages	- Does not grind properly in used machinery	- A lot of material is vested, and cut be used as filler (around 90%) - Cannot be fully grinded - Takes time to grind therefore it causes overheating of the bowl	- There are material remains after drilling that cannot be drilled 	- Quite slow and energy-consuming to prepare by hand - Does not fully grind (even after 3 rotations)	- Quite slow and energy-consuming to prepare by hand	- Quite slow and energy-consuming to prepare by hand

Source: Experimental trials conducted by the Author of this Research, 2021.

Grinding “bridge parts“ using current equipment is not efficient. But if grinding is required and only current equipment is present, there are two “best” approaches: (1) Tubes and plates (0,2 – 0,3 x 0,2 – 1 x 0,5 – 1 cm^3), because it makes the biggest amount of usable powder in the shortest time and fast and easy to prepare, but they have the biggest amount of unusable / waisted material and (2) flakes planed (small) do not have as big amount of unusable material after one grind, but they have almost one half of usable powder from the input amount and it does not take much effort to plane them.

4.1.5. Problems with Recycling

There were three major problems occurred during recycling at the lab scale: (1) overheating, (2) density and (3) usable material percentage shall be discussed in this Chapter.

Overheating

The big problem with used machinery after about 6 – 8 rotations if it was not used previously gets to the temperature where it starts to negatively affect grinding after which it has to be cooled with water which takes about 3 minutes. But it does not cool fully with water, therefore, after the first cooling it starts to overhit after each 3 – 4 rotations. Cooling takes much time in the grinding process (almost half of the time) and if this situation could be improved, it will significantly increase the efficiency of grinding. Probably changing the bowl and balls of the grinding machine to desirable types will improve the situation.

Density

The density of the material is very low (1,13), therefore, even if the whole bowl is filed (about 250 ml), it makes only 40 g of powder after grinding. This increases the number of grinding circles and, thus, time and effort that is needed to grind and recycle "bridge parts".

Usable Material Percentage

There is on average less than 50% of usable (under 125 μm) powder after each sieve. It means that only less than 50% of the material can be recycled using this method. That can also change, if desirable types of bowls and balls shall be used, because in Fritsch experiment it made around 67% of the usable powder.

Summery

To make the grinding process easier and more efficient at laboratory scale, new and more suitable, as it is described in Chapter **4.1.1. Machinery Grinding**, the bowl and balls should be bought. Hence, a different approach than cooling with water should be tried out, because cooling with water is time-consuming. An efficient cooling spray can be an option for overhitting problem, and this approach should be tried out in the next research.

Additionally, extra testing with sieving should be done because the current data are not enough to make a solid conclusion.

4.1.6. Recycling at the Production Scale

To make powder at the production suitable machinery should be chosen, for cutting and grinding. The cutting machine should cut material and cut it into pisses as big or smaller than the maximum fid size of the grinding machine. And the grinding machine should grind the initial material and grind it in the required particle size (125 μm or smaller) at the maximum efficiency rate. There are many different options of the machinery that can be used for this purpose and to find the best combination of the machines. Further research is necessary, because the knowledge acquired in this Chapter is relevant only for a laboratory scale.

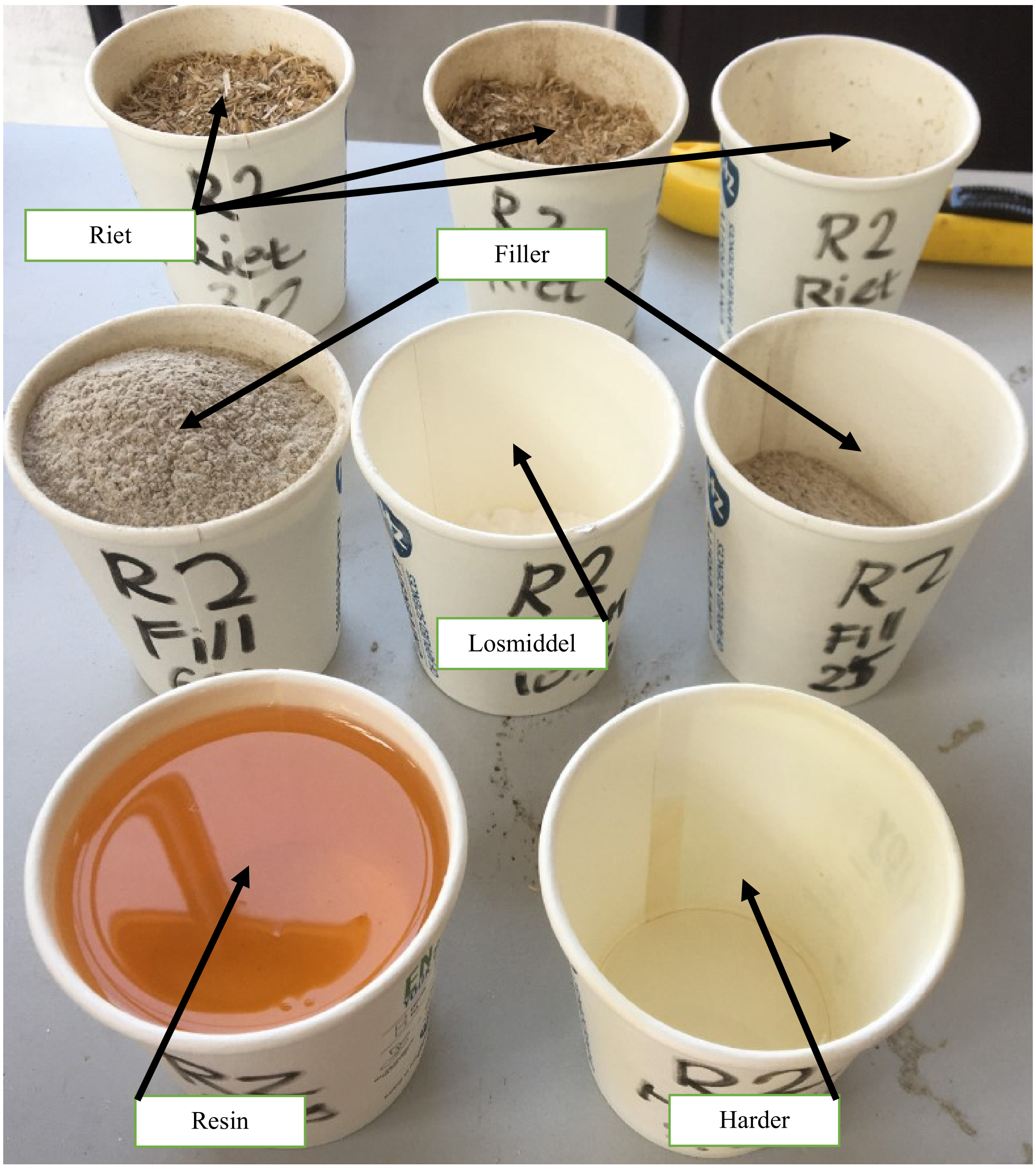
4.1.7 Preliminary Conclusion of this Chapter

New ways for grinding the material should be developed, because the current equipment is not efficient. That can be overcome by testing a new equipment that was mentioned in Chapter **4.1.1 Machinery Grinding.** And that even two «best solutions» for grinding that were found in Chapter **4.1.4. Testing Different Settings and Input Particle Sizes and Shapes**: they are tubes and plates (0,2 – 0,3 x 0,2 – 1 x 0,5 – 1 cm^3) and Flakes planed (small) that have some flows as mentioned in this Chapter.

4.2. Plates Making
In this Chapter the following aspects are clarified: (1) heat pressing process, (2) the machinery that was used, (3) how the ingredients were measured, mixed and heat pressed. (4) In addition, the resulting plates and conditions how they were made are also described.

4.2.1. Measuring the Amount of Ingredients
After the amount of the material needed for each mixture was calculated, it is necessary to be measured. Precision for all ingredients while measuring was 1.0 g and for hardener it was 0,1 g. All the ingredients were put into caps and weighted. On Illustration 6 the ingredients in the caps for mixture 2 are shown as follows.

Illustration 6: Ingredients in the Caps for Mixture 2.



Source: Picture taken by the Author of this Research, 2021.

It is important to mention that while measuring ingredients volume relation between filler powder and resin was probably not as it was calculated as in first mixture, where the volume repletion between those ingredients was 1,07 to 1, but there were 2,25 cups of filler and only 1 cup of resigning. The same situation was with other mixtures. A possible explanation can be that the powder was not compressed enough or density of it that was used for calculation was not correct, but further research is needed.

4.2.2. Mixing

The mixing of the material is done using the Mixing machine (Illustration 7). The method of mixing is the same as described in Chapter **3.12. Making the Material.**

Illustration 7: Mixing Machine.

Source: Picture taken by the Author of this Research, 2021.

After mixing all the ingredients resulting mixtures for each recipe can be seen on Illustrations 8, 9 and 10 as follows.

Illustration 8: Mixture for Recipe 1.

Source: Picture taken by the Author of this Research, 2021.

Illustration 9: Mixture for Recipe 2.

Source: Picture taken by the Author of this Research, 2021.

Illustration 10: Mixture for Recipe 3.

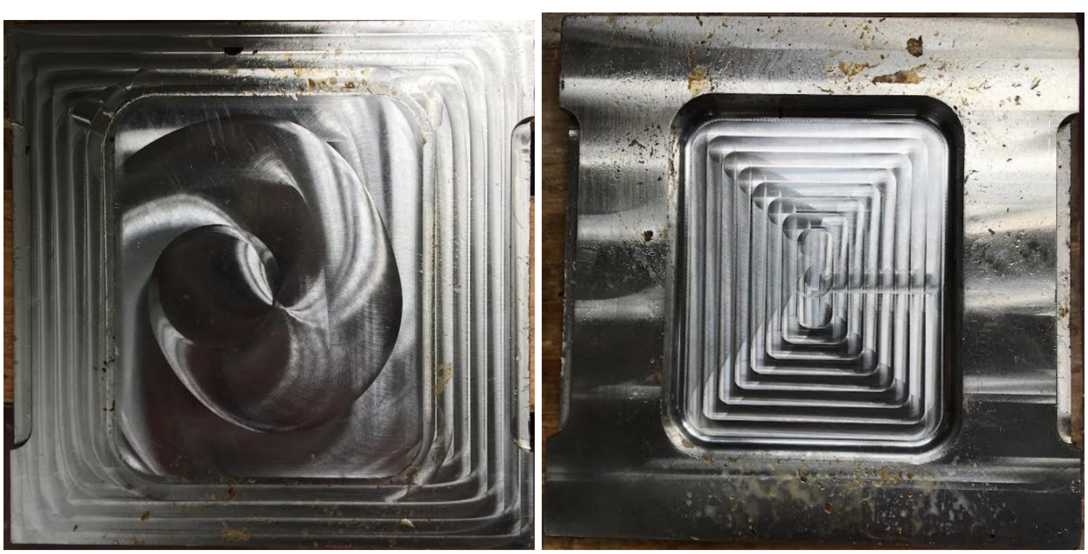
Source: Picture taken by the Author of this Research, 2021.

All the mixtures look less dry than the mixture of recipe 4 (60% mixture, that did not work out) That is described in detail in Chapter **3.1.2. Failure of the Recipe.**

4.2.3. Heat Parsing (BMC)

On the Illustration 11 the top part with the cape of the mold can be seen on the left Image and the bottom part with recess can be seen on the right one.

Illustration 11: Mold.

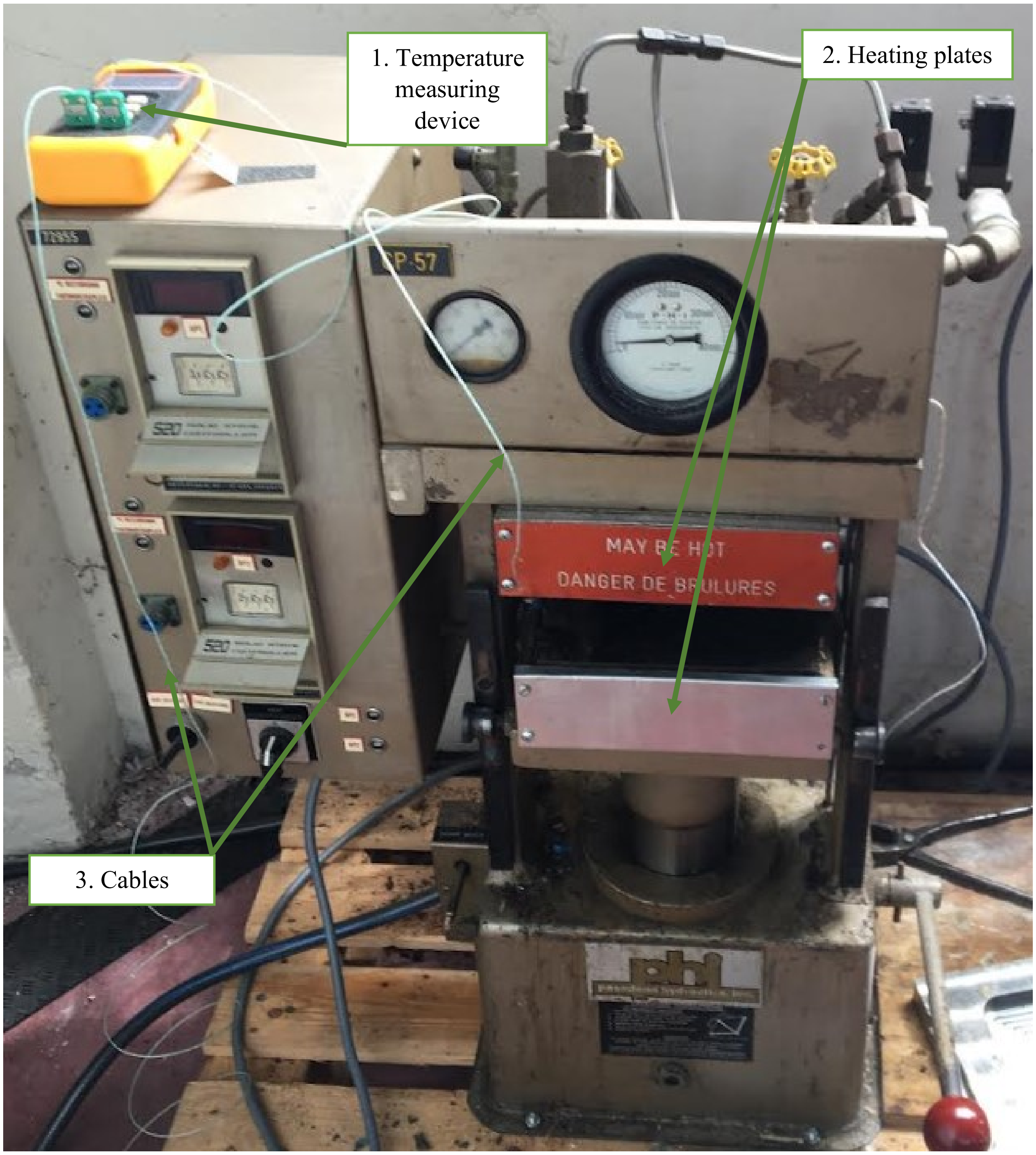


Source: Picture taken by the Author of this Research, 2021.

The materials were hit pressed on the heat pressing machine (Illustration 12) using the mold (Illustration 12). Before putting the material in the mold top and the boot parts are sprinkled with Losmiddel (Zinkstearaat) and then the mixture is evenly distributed (if possible) in the bottom part. Then the mold is closed and put in the pressing machine. Then the pressure is applied on the mold using the machine. This process is described in Chapter **3.8 Making the Material.**

The heat pressing machine used to make plates can be seen on Illustration 12. The specific temperature of the mold is required to be 170 C to make the plate. To check the temperature of the mold the temperature was measured with the temperature measuring device (1) by the connecting cables (3) to the heating plates (2), because an internal measuring device that is installed in the machine was proven not to be reliable, because it is located far from the actual surface of heating plates (2) and is closer to the heating element and always give a higher temperature than at the heating plates (1) and therefore mold.

Illustration 12: Heat Pressing Machine.



Source: Picture taken by the Author of this Research, 2021.

4.2.4. Resulted Plates

Each plate for each mixture was marked and conditions (pressure, temperature and the mixture that was put in the mold), on which all plates were made, were noted. These conditions can be seen in Table 18. Each resulting plate can be seen on Illustrations 13, 14 and 15. As it can be seen in each mixture, there are plates that were better and worse in terms of how good they healed. On Illustrations 13, 14 and 15 areas that did not have enough resin and have the same problems that they can be broken by hands are marked with rad circles. Their quality (including mixture, plate number, pressure, temperature, weight of mixture that was put in the mold, weight of resulting plates and quality of resulting plates) can be seen in Table 18 as follows.

Table 18: Shows the conditions under which plates were heat pressed.

Plates conditions						
Mixture	**Plate №**	**Pressure**	**Temperature**	**Weight of mixture that was put in the mold**	**Weight of resulting plates**	**Quality**
Recipe 1	1	40000 PHI*[2] (80 bars)	Top - 179 Bot - 170	86	86	Fully cured/set
	2	40000 PHI*[2] (80 bars)	Top - 179 Bot - 170	80 (not enough mixture)	79	Have many defects
	3	40000 PHI*[2] (80 bars)	Top - 180 Bot - 170	86	83	Have some defects
	4	40000 PHI*[2] (80 bars)	Top - 168 Bot - 166	86	85	Fully cured/set
Recipe 2	1	40000 PHI*[2] (80 bars)	Top - 175 Bot - 169	89	85	Fully cured/set
	2	40000 PHI*[2] (80 bars)	Top - 169 Bot - 165	83 (not enough mixture)	83	Have a small defect as not enough mixture was present
	3	40000 PHI*[2] (80 bars)	Top - 173 Bot - 170	89	87	Fully cured/set
	4	40000 PHI*[2] (80 bars)	Top - 168 Bot - 166	89	85	Fully cured/set
Recipe 3	1	40000 PHI*[2] (80 bars)	Top - 173 Bot -*[1]	85	81	Almost did not cured/set
	2	40000 PHI*[2] (80 bars)	Top - 169 Bot -*[1]	85	84	Have some defects
	3	40000 PHI*[2] (80 bars)	Top - 166 Bot -*[1]	85	82	Have moderate amount

						of defects
	4	40000 PHI*[2] (80 bars)	Top - 182 Bot -*[1]	85	83	Have moderate amount of defects

*[1]Bottom cable have unglued from the bottom heating plate, therefore temperature, was not able to be measured.

*[2]40000 PHI was calculated from 80 bars (the required pressure in the original mixture) using the dimension of the mold, because the machine operates with PHI.

Source: Experimental trials conducted by the Author of this Research, 2021.

Illustration 13: Plates from Recipe 1.

Source: Picture taken by the Author of this Research, 2021.

Illustration 14: Plates from Recipe 2.

Source: Picture taken by the Author of this Research, 2021.

Illustration 15: Plates from Recipe 3.

Source: Picture taken by the Author of this Research, 2021.

It is important to note that the plates were marked with a marker that could have slightly affected the performance negatively.

4.2.6. Preliminary Conclusion of this Chapter

The only mixture that had only 24 % (in g) of filler did not have any uncured / unset parts that can be caused by filler to resin ratio. Some plates in the same mixture appeared to be better than the others (they did not have or did not have very many defects) and there is no reasonable explanation for this fact (except for plate №2 in Recipe 1 and 2, where there was no enough material that was put in the mold, and plate №1 in Recipe 3, where the problem probably connected with the production that was not found out appeared).

Possible causes of these defects can be the following. Firstly, something during the production state was not executed correctly and was not noted. Secondly, the problem was with the initial calculation of recipes percentage of filler and volume ratio of filler and resin, as it was mentioned in Chapter **4.2.1 Measuring the Amount of Ingredients** amount of filler compared to resin visually was much bigger than calculated. Therefore, to find out what was the cause of the problem it is recommendable to make plates with the same amount of ingredients as in Recipe 1 (as it has the same calculated volume ratio as the original (NABASCO) recipe) and to see what the results shall be. If the results shall be the same, it means that there is a problem with calculation. But if the results shall be different, it means that there is probably a problem with the production. Additionally, it is advised to recalculate the density of the "bridge parts" itself, because the value for that was taken from previous research and can be different in the reality (Jimmy et al., 2021).

Hence, it is advised to proceed in the following way: while making the recipes multiply **not** by 1,05, but by 1,10, because the amount of mixture was not enough for all the 4 plates in 2 out of 3 recipes. It is also recommendable to place in the mold a slightly bigger amount of mixture than what was required, because it can also be the reason of the problem.

Summary

While making the plates several different problems occurred: (1) the initially expected presence of filler in the mixture (in g) was too much and even much smaller amounts (38% and 46% comparing for initial mixtures 60%) were too much, thus, the plates did not fully cure and had some defects and (2) the plates in the same mixture had different amounts of defects, the cause for that can be problems during the production process.

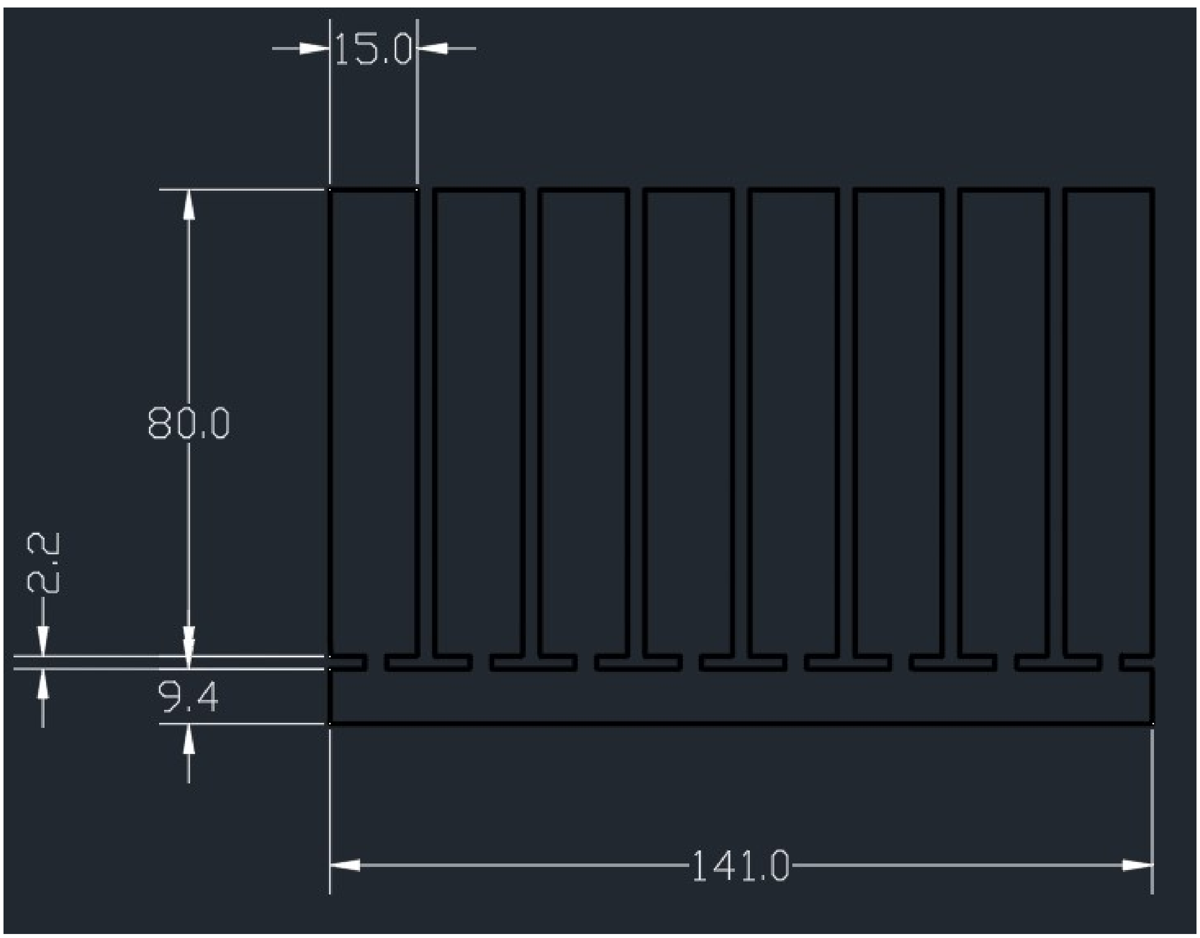
4.3. Cutting
In this Chapter the machinery for cutting, cutting pattern and the cutting results are explained in detail.

4.3.1. Machinery
For cutting the plates into the required shape and number of samples, the 3 best plates (with listed defects) of each mixture were cut using a water jet cutter in order to do the 3-point bending test specific dimensions of the samples required. It will be clarified in detail in Chapter **4.3.2. Cutting Pattern and Dimensions of Samples**.

Additionally, it was considered to use the laser cutter instead of the water jet cuter, because the water jet was not available at the time, but after making a research it was found out that laser cutting decreased bending strength of plastic composites and was not preferable in this research (Dirk et al., 2008). Therefore, water jet cutting was used.

4.3.2. Cutting Pattern and Dimensions of Samples
To make the dimensions a specific pattern that can be seen on Illustration 16 was used to cut the plates with the water jet from which 8 samples were received. The thickness of the samples was determined by the mold, because it was not cut with the water jet.

Illustration 16: Dimensions Used for Water Jet Cutting.

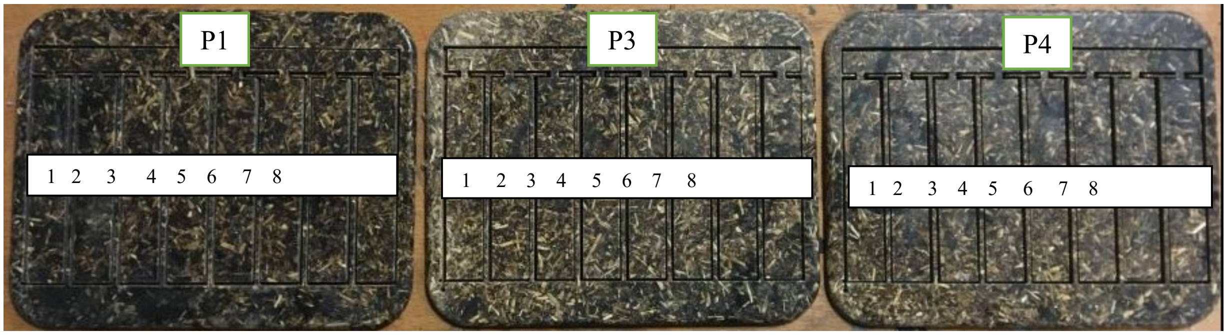


Source: Picture taken by the Author of this Research, 2021.

4.3.3. Results of Cutting

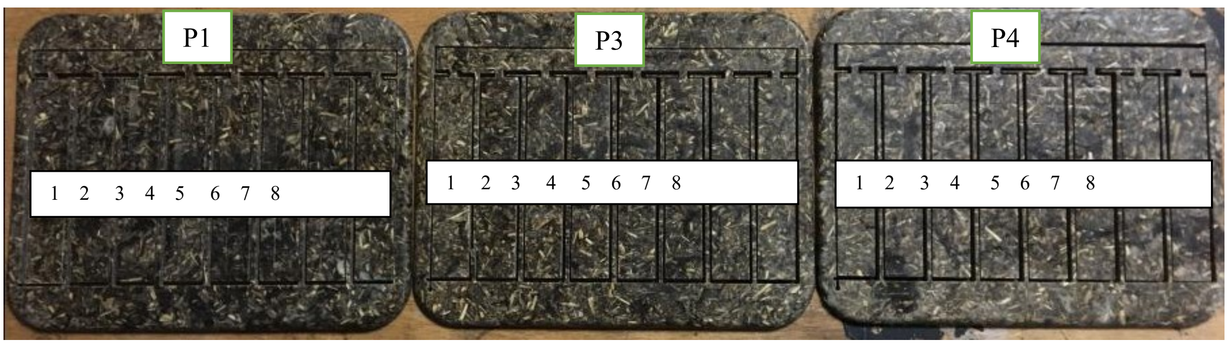
The catted plates for each mixture can be seen on Illustrations 17, 18 and 19. Each plate sample is marked with its number that is further used for testing.

Illustration 17: Plates from Recipe 1.



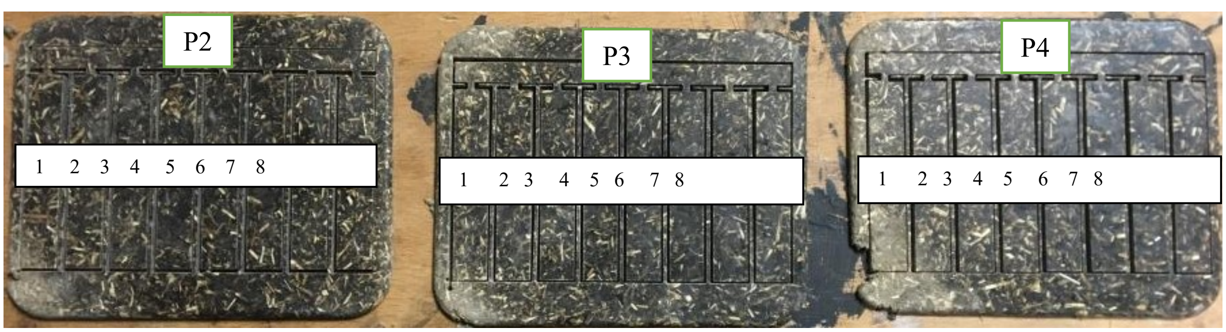
Source: Picture taken by the Author of this Research, 2021.

Illustration 18: Plates from Recipe 2.



Source: Picture taken by the Author of this Research, 2021.

Illustration 19: Plates from Recipe 3.



Source: Picture taken by the Author of this Research, 2021.

As it can be seen on these Illustrations, all of the plates have a defect on the same side in relation to the cut. Samples from 8 to 6 were used for the density test and samples from 8 to 4 were used for the 3-point flexure test. It was conducted with the purpose to find the best performers of the mixture.

4.3.4. Preliminary Conclusion of this Chapter

In comparison to the measuring of samples results shown in **Appendix E** it can be seen that on average the length is within 2 mm of the allowed fluctuation, and the thickness that appeared on average being 3,81 mm and being in 0.2 mm allowed fluctuation. Yet the width which was 15 mm is not the required 10 mm and the fluctuation therefrom is larger than the allowed 0.2 mm. The thickness preferably needs to be fixed in the next research by adjusting the preset and regulating the water jet cutter. Also, to fix the problem with the width, the width of the mold should be changed and the new mold shall be made thinner (10 mm instead of 15 mm) for the next researches. Additionally, as the same number of samples are required, it will decrease the needed amount of the mixture for 1 plate and, therefore, for the filler. As previously indicated, it is very time-consuming to prepare the filler.

4.4. Tests

In this Chapter the conducted tests and their resalts are explained.

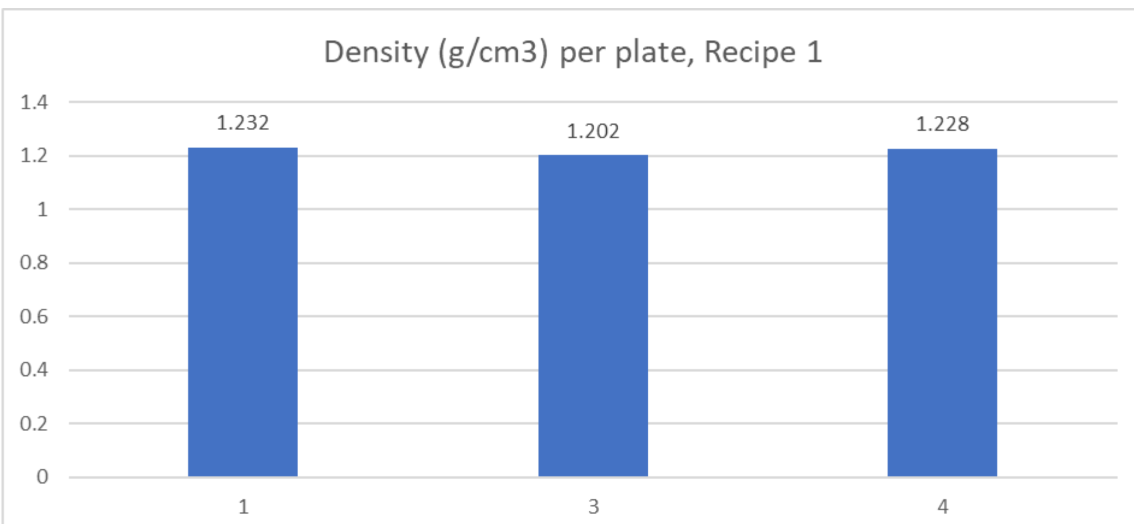
4.4.1. Density Test

Results

To find the density of each plate of each mixture, three samples of each plate of each mixture were measured and then weighted. On this base, the density of each sample was calculated and then the average plate density and the average mixture density were found. Table 26 Density and Average Density Calculation for Mixtures represents the results with calculations and data, it can be seen in **Appendix E**.

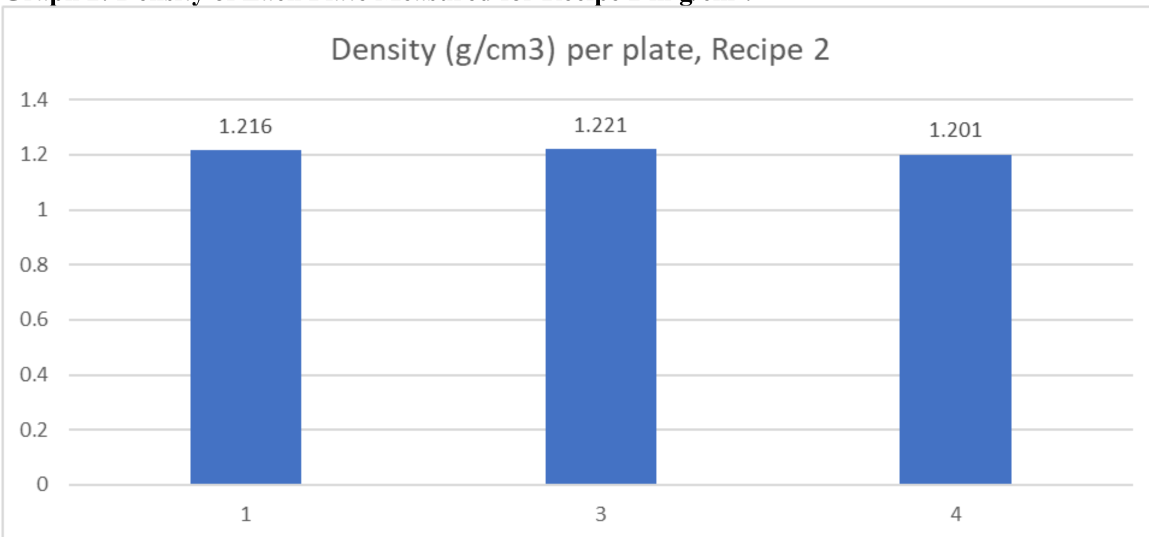
Graphs 1, 2 and 3 represent the density for each plate for each mixture and can be seen below. Graph 4 shows the density of each mixture comparison with the anticipated density that was calculated (The detailed calculation can be seen in **Appendix E**.)

Graph 1: Density of Each Plate Measured for Recipe 1 in g/cm^3.



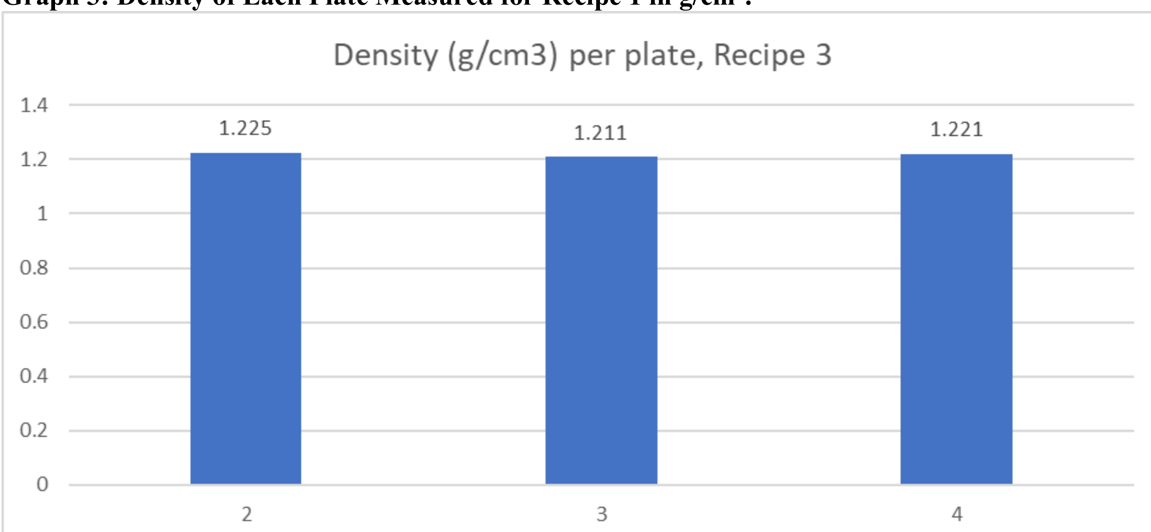
Source: Graph made by the Author of this Research, 2021.

Graph 2: Density of Each Plate Measured for Recipe 2 in g/cm^3.



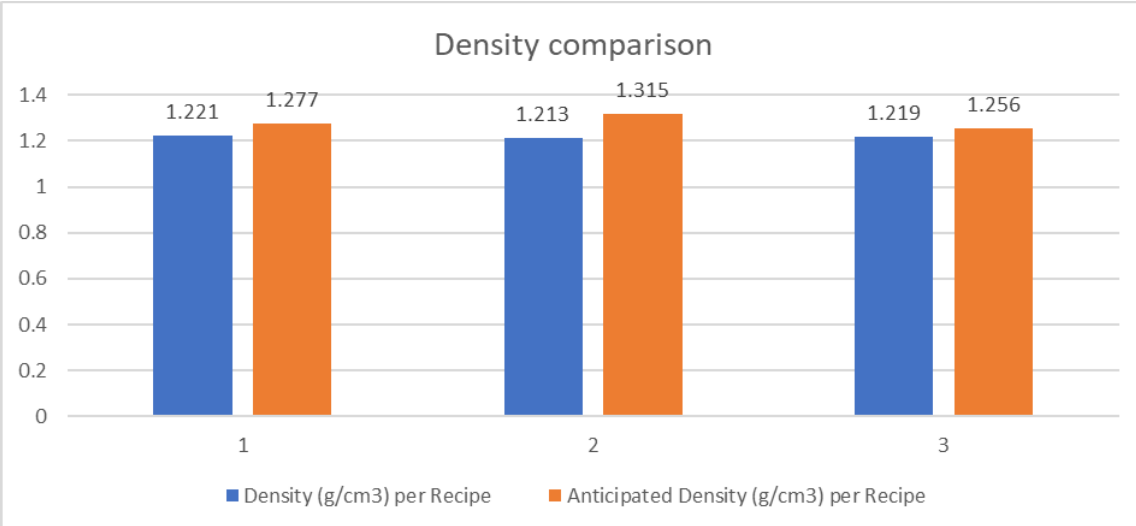
Source: Graph made by the Author of this Research, 2021.

Graph 3: Density of Each Plate Measured for Recipe 1 in g/cm^3.



Source: Graph made by the Author of this Research, 2021.

Graph 4: Average Density of Each Mixture Compared to the Anticipated Density in g/cm^3.



Source: Graph made by the Author of this Research, 2021.

The average density of each plate in each mixture is quite similar (with the maximum difference being less than 0,3 g/cm^3) which can prove that the mixture was well mixed and plates should have relatively the same qualities in each recipe.

The overall density of each mixture was quite similar and that can mean that the amount of filler does not affect the mixture very much in terms of density. And all plates in each mixture had a quite similar density which means that process of making plates was conducted in a similar manner for all plates in at least one mixture. All mixture appeared to have lover density than anticipated. Therefore, it is possible that some of the ingredients had different densities than that was used for calculation (especially grinded filler).

When comparing anticipated and resulting density of each mixture, it can be seen that if anticipated it has higher density, especially in mixture 2 (being almost 0.1 higher) which can mean that densities that were used for calculation were not completely correct, as mixture 2 was anticipated to have the highest density. As it had the highest content of resin that is almost the densest component of the mixture (being second after hardener) such a small relative amount in the mixture that does not have any significant effect on the density of the mixture) and in theory, therefore, should have the highest density, but mixture 2 had the smallest density. Hence, it is possible that some of the materials have different densities than those that were used in the calculation. It is necessary to be checked for the next research on that topic.

Additionally, it can be seen that the density of the new mixture did not increase significantly (only 0,9 g/cm^3 max) compared to the initial mixture. It means after one recycling it can possibly be used for the same purposes as the original mixture, if the other parameters shall be sufficient (bending strength, flexural modulus). But further research is needed to clarify these issues in more detail.

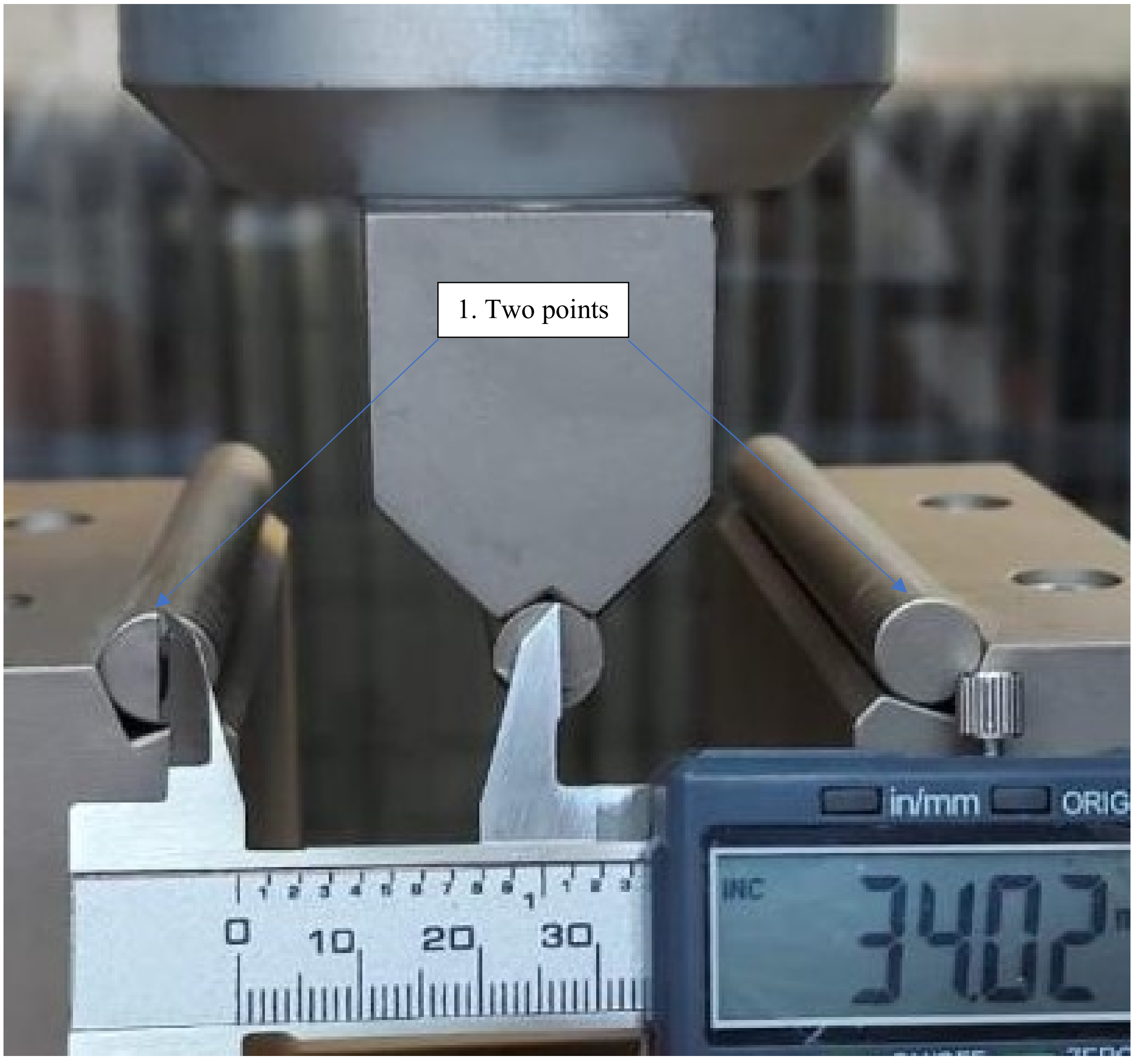
4.4.2. 3-Point Flexure Test

Preset

A 3-point bending test is conducted to find out the bending strength and flexural modulus. Five samples from each plate from each recipe were tested to do that. As it was mentioned that samples 4 – 8 were tested, because those samples were from the side of the plate that has either the lowest number of defects or it has no defects at all.

The dimensions that were preset in the machine were the following: 14.62 mm for width (w) and 3.8 mm for thickness (th). For calculating the bending strength (B) and flexural modulus (F) length (l) between 1. Two points at 3-point bending machine were used, that is equal to 68 mm, as it can be seen on Illustration 20 as follows.

Illustration 20: 3-Point Bending Machine Preset.



Source: Picture taken by the Author of this Research, 2021.

<u>Results</u>

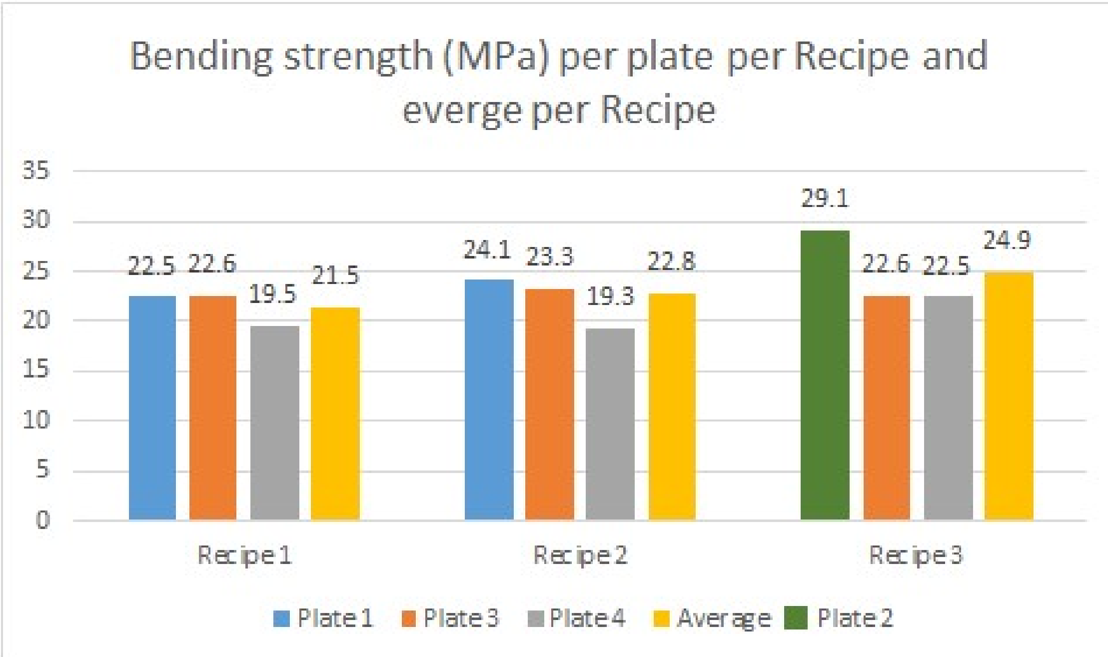
The tables with results (Table 27, 28 and 29) with calculations and data can be seen in **Appendix F**.

Graph 5 showing the bending strength of each plate for each recipe and average bending strength for each recipe can be seen below.

Graph 6 showing the flexural modulus each plate for each recipe and average flexural modulus for each recipe can be seen below, as well.

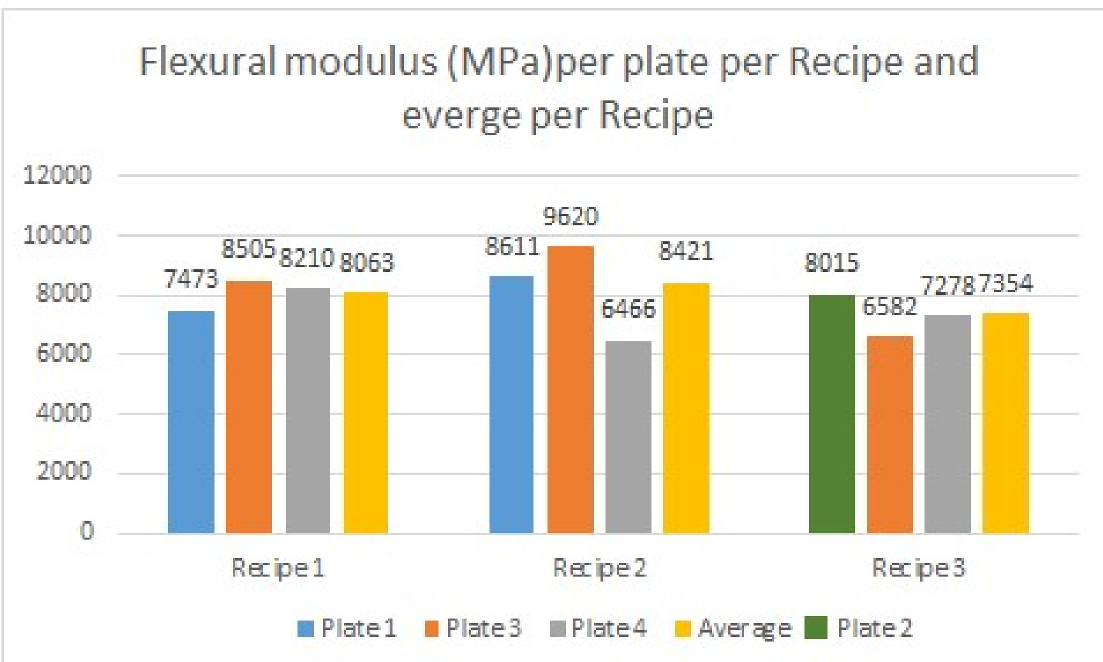
All calculations for this data can be seen in **Appendix F**.

Graph 5: Average per Recipe and per Plate per Recipe Bending Strength in MPa.



Source: Graph made by the Author of this Research, 2021.

Graph 6: Average per Recipe and per Plate per Recipe Flexural Modulus in MPa.

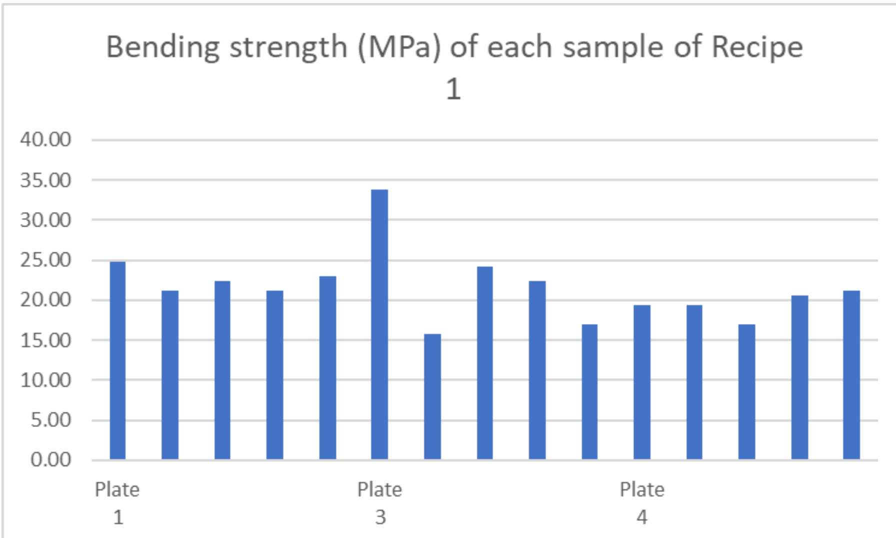


Source: Graph made by the Author of this Research, 2021.

All the results per plate in the same mixture are mostly similar, but there are some exceptions, e.g., bending strength for plate 2 in Recipe 4 that is more than 16% bigger than the average one and flexural modulus for plate 4 in Recipe 2 that is more than 23% smaller. These are quite huge deviations from the average for the mixture (even though only three plates were tested). It is possible that there were some deviations with the production of those plats, but there is no visible correlation between the temperature of the production of the plate and its density and performers of samples.

On the Graph 7 bending strength (MPa) of Each Sample starting from left two right starting from sample number 8 up to 4, then the next plat starts, for Recipe 1.

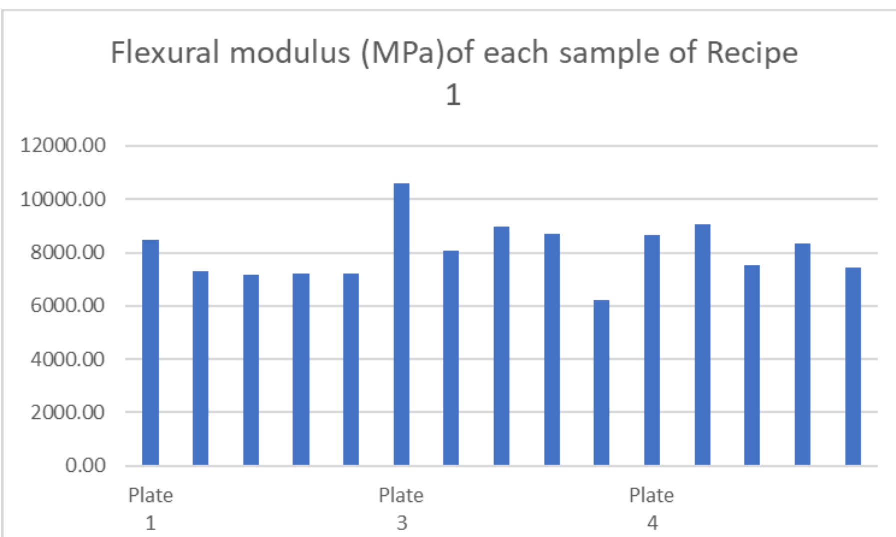
Graph 7: Bending Strength (MPa).



Source: Graph made by the Author of this Research, 2021.

On the Graph 8 Flexural Modulus (MPa) of Each Sample starting from left two right starting from sample number 8 up to 4, then the next plat starts, for Recipe 1

Graph 8: Flexural Modulus (MPa).



Source: Graph made by the Author of this Research, 2021.

It can be observed in Graph 7 and Graph 8 (for Recipe 1 and for the rest it can be seen in **Appendix F**) that there is a correlation between the placement of the samples within the plate and their properties in general: the closer the number of samples goes to 1, the worse is the performance (lower bending strength and flexural modulus). Especially, it can be seen at flexural modulus where all the plates are representing it at some degree. As mentioned previously, most of the plates had defects on the side with sample numbers closer two one, and that is probably the reason for the decrease in the performance.

As far as the comparison between mixtures is concerned, the following can be observed: Recipe 2 had the best flexural modulus result followed by Recipe 1 and finishing with Recipe 3, but the result was not so much different. The difference between the best and the worst results totals only by 15 %.

On comparing the mixtures it is clear that Mixture 3 has the highest bending strength performance on average, followed by Mixture 2 (Recipe 2) and then by Mixture 1 (Recipe 1). These results are not as expected, because Recipe 3 had the biggest problems with defects on the plates and that result was caused mainly by overperforming (comparing to all the other plates) plate number 2 in Recipe 3. Therefore, it can be assumed, Recipe 3 may not be that highly performing.

As the result in general it can be noted that Recipe 2 having the highest flexural modulus and second high bending strength can be considered as the best performing recipe overall. And so that the less filer there is, the better is the performance, at least at a certain point. This should be further investigated in the next research.

4.4.3. Preliminary Conclusion of this Chapter

The overall density of each mixture was quite similar and that can mean that the amount of filler does not affect the mixture very much in terms of density. And all the plates in each mixture had a quite similar density which means that process of making plates was conducted in a similar manner for all the plates in at least one mixture. All the mixtures appeared to have lover density than it was anticipated. Therefore, it is possible that some of the ingredients had different densities than used for calculation (especially grinded filler) or possibly moist content of ingredients was not considered.

In general, the results show that the more filer there is in the mixture, the worth recipe performs (except of Plate 2 Recipe 3 that showed unexpectedly high performance), but the difference is not so considerable. And as most of the plates had defects on one side that was proved to worsen the performance of samples closer to the problematic sight that can lead to some inconsistency in recipes performance. The results can be slightly different and the difference between recipes performance in terms of flexural modulus and bending strength can increase or decrees. But it can be noticed that the pattern is clear and the more filler there are in the mixture, the worth it performs at least in the scope of % of filler that was tested (24% to 46%) in this Research.

4.5. Comparison to Previous Research

In this Chapter the comparison to the execution method and the results of the previous research and this Research are clarified.

4.5.1. Plates Making

There is a significant problem connected with comparing results with the previous research – the amount of filler in the new mixture. As mentioned in Chapter **3.10 Method Updates**, the amount of filler that was claimed to be used in the previous research was tested. And the plates that were made from the mixture with the same amount of filer (60%) were unusable and obviously had too much filler in them. Even when using 46% mixture that had a bit higher percentage compared to 42% that was the smallest used mixture in the previous research, there were still problems and it also had too much filler. That shows that there was some error in calculations or production methods in the previous report and that can cause the difference in the comparison of the two results.

4.5.2. Tests

While conducting a 3-point flexure test, different equipment on which test was conducted was used. That can also lead to the difference in results.

4.5.3. Comparison of Results

A comparison shall be made between this Research and the previous research (Jimmy et al., 2021) average results of all mixtures, as the results were quite similar and there is a difference in filler percentage in mixtures between the two types of research.

<u>**Density Test**</u>

In this Research the average density between mixtures is 1,217 g/cm^3 compared to 1,28 g/cm^3 in the precious research which is quite close to the average anticipated density in this Research which is 1,282 g/cm^3. The difference can be caused by the possibility that in previous research the percentage of filler was much smaller than what was stated as a filler (1.13 g/cm^3) is lighter than the resin (1.4 g/cm^3) (both having the biggest percentage amount in the mixture and, therefore, contributing to the density the most) and seeing that density in the previous research being bigger, theoretically, should have on the average higher amount of resin comparing to this one, the contrary on what is stated in the previous research.

<u>**3-Point Flexure Test**</u>

This Research shows average bending strength being 23.1 MPa and flexural modulus 7945,8 MPa compared to 40.6 MPa and 3990 MPa respectively for the previous research. This a huge difference in results in having almost two times smaller bending strength and two times bigger flexural modulus. This difference can be caused by the difference in equipment types on which the research was conducted, because in the case of this Research both parameters were calculated using formula and for the previous research it was calculated by machine on which their test was conducted.

4.5.4. Preliminary Conclusion of the Chapter

This Chapter shows that compared to the previous research it is very difficult as (1) data that was provided there was not corresponding to the findings of this Research or (2) the equipment on which some tests were performed was different. Those facts could lead to different results.

5. Discussion

In this Chapter the results of this Research are explained and compared to the existing literature on the topic and to the previous research.

5.1. Discussion

The results of this Research indicate that in terms of bending strength and flexural modulus performance the more filler there are in the mixture, the worth it performs. It is relevant at least in this particular filler percentages in the mixture that were tested. As for density all mixtures had similar results and close to the anticipated densities always being slightly smaller.

The amount of filler where bending strength and flexural modulus of the mixture shall be the highest, was not found, but the results showed the tendency to have higher performance of the mixture with decries of the filler percentage. This shows that nest research should focus on smaller percentage of filler and possibly find the percentage amount of filler where (if it goes lower) the results will be valuable. Having similar densities in mixtures represents that mixture was mixed well and all the plates were made as similar as it was possible. It shows that when finding density of the ingredients some of the density can be incorrect (especially, the density of the filler powder) or the moist content is not considered.

This Research was limited by lack of time and some unexpected problems with initial design of the amount of fuller in mixture, as mentioned in Chapter **3.1. Method Updated.** Thus, possible improvements such es making more mixtures and finding possible “best” filler percentage or finding more correct densities for the ingredients could be made. But it is important to note that already this Research showed promising results and with further development it should be possible to find the “perfect” amount of filler and use this method as the bio end life solution for the bio composite.

5.2. Comparison to the Existing Literature

In this Chapter the results of this Research are compared to the results of the previous research and literature on this topic.

5.2.1. Previous Research

As it was indicated, the comparison to the previous research is very complicated and inconclusive due to the afore-mentioned different problems, including problems with the amount of powder in material to the difference in equipment. And a proper comparison was not possible to be made.

5.2.2. Literature on the Topic

With the help of the specific grinding method useful recyclate can be produced, which can be used to replace existing reinforcing fibers in a new composite with minimal effect on the mechanical properties for dough molding compound (DMC) (or BMC) and structural equation modeling (SEM). Changing the mixing procedure reveals a significant increase in the new composites mechanical strength, it makes it have the same mechanical properties as initial composite. (J. Palmer et al., 2009). And shows that through finding perfect relation of compounds in mixture new bio composite possibly can be as mechanically capable as an old one that.

This Research shows that different mixtures in bio composites affect mechanical properties of composite. Hence, further research shall be done, perfect relation can be found at the same way it was for non-bio counterpart and new made bio composite can be as mechanically capable as from which it was made of.

6. Conclusion and Recommendations

In this Chapter the final conclusion of this Research is presented and final recommendations are given.

6.1. Final Conclusion

As expected and formulated in the Hypothesis of this Research, there is a relation between the filler amount and properties (flexural modulus and bending strength) of recycled BMC thermoset biocomposite, and it shows that the less filler, the better recipe shall perform. But the point (if any), where this stopes to be true and the properties become worse, was not found out and the pattern established cannot be for the whole mixture. The topic shows promising results and if all the details that were mentioned in this Research and in Chapter **6.2. Recommendations** shall be addressed, it is possible that this type of solution can be the end life solution for this type of biomaterial and it will find more use in civil engineering sphere that it currently has.

Nowadays, there are not so many areas where BMC thermoset composite is used, because it is a relatively new material and its bio counterpart is used even less. Examples of using bio one can be facades, road signs, and outdoor sofas. But to use this recycled bio composites more tests are needed to be conducted, because physical and mechanical properties that were tested need further research. And there are many other properties of the materials that are important for civil engineering (like chemical, optical, acoustical and physiochemical) that shall be found out in further research to use this recycled biocomposite in the civil engineering field.

6.2. Recommendations

Possible development for grinding solutions on the lab-scale should be made, because current methods are not very efficient. And all the developed solutions are focused only on lab-scale grinding of the material now. That is why production scale solutions should be developed for further production development.

Production process problems should be further investigated in the next research. Additionally, the density of all the ingredients should be checked (and remeasured in case of grinded filler) and adjusted in the calculation, if necessary.

For the next research new possible percentage of filler in g (less than 38%, as it is possible that 24% was not the maximum possible relation of filler and mixture) to find out the best combination (best performing at flexural modulus and bending strength) and to find out the point (if any) where the amount of filler is its minimum and making it smaller will negatively affect performance (at flexural modulus and bending strength).

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Appendices

Appendix A

Minor Biobased Building: Structural Health Mixtures

There were 3 mixtures from which material was made in the previous research. All the mixtures have the same percentage amount of ingredients, the only parameter that is changing is the percentage amount of filler (Brugdeel/calcite). Table 19 shows the amount of each ingredient in g for one plate of their material as follows:

Table 19: Amount of Ingredients for 1 Plate.

Ingredients	Amount (g)		
Resin (Hars)	31,79		
Harder (Peroxide)	0,46		
Losmiddel (Zinkstearaat)	1,61		
Riet (Vezel)	11,13		
	Recipe 1 (42%)	**Recipe 2 (50%)**	**Recipe 3 (60%)**
Vulstof (Brugdeel)	32,51	45,00	67,50

Source: Experimental trials conducted by the Author of this Research, 2021.

Table 20 shows the amount of each ingredient per 100 g for one plate of their material as follows:

Table 20: Amount of ingredients per 100 g.

Ingredients	Amount (g)		
	Recipe 1 (42%)	**Recipe 2 (50%)**	**Recipe 3 (60%)**
Resin (Hars)	41,02	35,33	28,26
Harder (Peroxide)	0,59	0,51	0,41
Losmiddel (Zinkstearaat)	2,08	1,79	1,43
Riet (Vezel)	14,36	12,37	9,89
Vulstof (Brugdeel)	41,95	50,01	60,01

Source: Experimental trials conducted by the Author of this Research, 2021.

Appendix B

Tests

Several different tests have been performed on the material. Some of the specifications of this tests are shown in **Table 21.**

Table 21: Illustration of the Performed Tests and their Specifications.

Test	Parameters to be measured	ISO Norm	The reasons why the test was chosen	Dimensions of test samples according to norms	Number of test samples according to norms	Number of plates used per mixture	The place where the test was conducted
3-point flexure test	The flexural test measures the force required to bend a beam under three-point loading conditions.	ISO 14125:1998 ISO 178:2019,IDT	These test results show most of the mechanical properties of the material. Knowing that fact is crucial for material that is can be used for the construction.	Length, l: 80 ± 2 mm Width, b: 10,0 ± 0,2 mm Thickness, h: 4,0 ± 0,2 mm	>=5 per plate (> if extra precision needed)	3 (to ensure the reliability of data)	Laboratory of HZ University of Applied Science
Density test	$Density\left(g/cm^3\right) = \frac{m}{v}$ where m is the mass of the composites and v is the volume of composites.	ISO 1183-1:2019	Knowing the density of the material allows to calculate the weight of this material of any shape because almost any form can be cast of this material. And knowing the weight of construction material is very important.	Methods a, b, and c: a) Specimens may be in any void-free form except for powder. They shall be of a convenient size to give adequate clearance between the specimen and the immersion vessel and should preferably have a	No specification	0 (The same plates as for banding test were used.)	Laboratory of HZ University of Applied Science

				mass of at least 1 g. b) Specimens of powders, granules or flakes shall be measured in the form in which they are received. The specimen mass shall be in the range of 1 g to 5 g C) Specimens shall be in a suitable void-free form.			
Flame test	Methods for the thermal analysis of polymers and polymer blends	ISO 11925-2:2010,IDT NEN-EN 13501-1 (en)	Flame resistance is one of the most important factors on deciding to use the material or not, because it gives knowledge on how fast the fire will spread through this material.	The dimensions of the test specimens shall be (250 +-2) mm long by (90 +-2) mm wide.	For each exposure condition, a minimum of six representative specimens of the product shall be tested. Three specimens shall be cut lengthwise and three crosswise.	1 (is enough)	Laboratory of HZ University of Applied Science
DSC (Differential scanning calorimetry)	This is the method for the thermal analysis of polymers and polymer blends physical transitions intended for the observation and measurement of various properties thereof, and phenomena such as	ISO 11357-1:2016	This test is important, because on the base of this parameter ideal production method in terms of heating temperature can be found.	-	-	0 (It remains from 3-point flexure test were used.)	-

	physical transitions (glass transition, phase transitions such as melting and crystallization, polymorphic transitions), chemical reactions (polymerization, crosslinking and curing of elastomers and thermosets, etc.), the stability to oxidation and the heat capacity.						

Source: Experimental trials conducted by the Author of this Research, 2021.

Appendix C

Old Approach Filler Amount

There were 4 mixtures from which material was made, the only difference shall be the percentage of recyclent. Table 22 shows the amount of the ingredients in g for one plate of the material.

Table 22: Amount of Ingredients for 1 Plate.

Ingredients	Amount (g)			
Resin (Hars)	31,79			
Harder (Peroxide)	0,46			
Losmiddel (Zinkstearaat)	1,61			
Riet (Vezel)	11,13			
	Recipe 1 (35%)	**Recipe 2 (45%)**	**Recipe 3 (55%)**	**Recipe 4 (65%)**
Vulstof (Brugdeel)	24,23	36,81	54,99	83,55

Source: Experimental trials conducted by the Author of this Research, 2021.

And Table 23 illustrates the amount of each ingredient in g per 100g of the material.

Table 23: Amount of Each Ingredient per 100 g for one Plate of the Material.

Ingredients	Amount (g)			
	Recipe 1 (35%)	**Recipe 2 (45%)**	**Recipe 3 (55%)**	**Recipe 4 (65%)**
Resin (Hars)	45,93	38,86	31,8	24,73
Harder (Peroxide)	0,66	0,56	0,46	0,36
Losmiddel (Zinkstearaat)	2,33	1,97	1,61	1,25
Riet (Vezel)	16,08	13,61	11,13	8,66
Vulstof (Brugdeel)	35	45	55	65

Source: Experimental trials conducted by the Author of this Research, 2021.

Average Anticipated Weight of One Plate

The calculation of weight of one plate has been made to show the amount of recyclent needed for the projects (in Chapter **Amount of Recyclent Needed).** The calculation can be seen in Table 24. The density of the plates has been taken from Minor Biobased Building: Structural Health.

Table 24: Method Showing how to Calculate the Average Anticipated Weight of one Plate.

Average anticipated weight of one plate				
	Desity (g/cm^3)	**Average density (g/cm^3)**	**Average weight per % (g)**	**Average weight (g)**
Brug 42% plate 1	1,31	1,28	= Average density * Volume of plate = 86,02	86,02
Brug 42% plate 2	1,26			
Brug 42% plate 3	1,27			

Brug 50% plate 1	1,27		= Average density * Volume of plate = 85,34	
Brug 50% plate 2	1,27	1,27		
Brug 60% plate 1	1,29	1,29	= Average density * Volume of plate = 86,69	

Source: Experimental trials conducted by the Author of this Research, 2021.

Amount of Recyclent Needed

The amount of recyclent should be calculated to make the material. Table 25 shows how it has been calculated as follows:

Table 25: Calculation of Minimum Amount of Recycling for Testing.

Given	-	Amount		Unit
Number of plates per mixture (n)		4		-
Weight of 1 plate (w)		86		g
Amount of recyclent per mixture		Recipe 1 (w1)	35	g/100g
		Recipe 2 (w2)	45	g/100g
		Recipe 3 (w3)	55	g/100g
		Recipe 3 (w4)	65	g/100g
What is calculated	**Formula**	**Result**		**Unit**
Total amount of recyclent per one test (W)	= (w1 + w2 + w3+w4) * n * w / 100	688		g
Total amount with 100% extra safety (Wf)*[1]	= Wt * 2	1376		g

*1 100% extra recicalent is taken to make sure that it will be enough and there is no need to cut and crush.

Source: Experimental trials conducted by the Author of this Research, 2021.

Appendix D

Cutting the Recyclent "Bridge Parts"

First, to make a filter out of the recycled it should be cut into pieces of around 1x1x1 mm^3 so that the machine can crush it into the powder. It is difficult to break the "bridge parts" into pieces, therefore, even small fragments have been cut. To cut it first from a big piece of material it has been cut into smaller strips on the bandsaw and afterwards from those strips it has been cut into cubes on the mechanical hacksaw. In Illustration 21 the result can be seen as follows.

Illustration 21: Cut Material.

Source: Illustration made by the Author of this Research, 2021.

Appendix E

Density and Average Density Calculation for Samples, Plates and Mixtures

In Table 26 calculations and data for the density of each plate of each recipe and average of each recipe can be seen. The results can be seen in **4.4.2 Density Test.**

Table 26: Density and Average Density Calculation for Mixtures.

Density and average density calculation for samples, plates and mixtures											
Recipes	Plates	Samples	b (mm)	h (mm)	w (mm)	V (mm^3)	V (cm^3)	m (g)	Samples Density (g/cm^3)	Plates Density (g/cm^3)	Recipes Density (g/cm^3)
Recipe 1	Plate 1	8	14.64	79.17	3.65	4231	4.23	5.18	1.224	1.232	1.221
		7	14.57	79.28	3.67	4239	4.24	5.21	1.229		
		6	14.56	79.35	3.64	4205	4.21	5.23	1.244		
	Plate 3	8	14.64	79.15	4.12	4774	4.77	5.8	1.215	1.202	
		7	14.7	79.3	4.07	4744	4.74	5.63	1.187		
		6	14.6	79.3	3.95	4573	4.57	5.51	1.205		
	Plate 4	8	14.69	79.15	3.96	4604	4.6	5.66	1.229	1.228	
		7	14.6	79.31	3.93	4551	4.55	5.59	1.228		
		6	14.6	79.3	3.85	4457	4.46	5.46	1.225		
Recipe 2	Plate 1		14.74	79.26	4.06	4743	4.74	5.66	1.193	1.216	1.213
			14.61	79.36	3.91	4533	4.53	5.55	1.224		
			14.56	79.35	3.82	4413	4.41	5.43	1.23		
	Plate 3		14.65	79.25	4.14	4807	4.81	5.83	1.213	1.221	
			14.58	79.27	4.02	4646	4.65	5.71	1.229		
			14.54	79.21	3.95	4549	4.55	5.55	1.22		
	Plate 4		14.65	79.11	3.44	3987	3.99	4.78	1.199	1.201	
			14.63	79.27	3.56	4129	4.13	4.95	1.199		
			14.53	79.33	3.65	4207	4.21	5.07	1.205		

	Plate 2	8	14.7	79.2	3.85	4482	4.48	5.47	1.22		
		7	14.7	79.35	3.8	4432	4.43	5.44	1.227		
		6	14.6	79.53	3.77	4377	4.38	5.37	1.227	1.225	
	Plate 3	8	14.7	79.13	3.63	4222	4.22	5.07	1.201		
		7	14.62	79.25	3.56	4125	4.12	5.09	1.234		
		6	14.58	79.3	3.7	4278	4.28	5.12	1.197	1.211	
Recipe 3	**Plate 4**	8	14.6	79.4	3.73	4324	4.32	5.34	1.235		
		7	14.5	79.2	3.8	4364	4.36	5.29	1.212		
		6	14.62	79.2	3.76	4354	4.35	5.29	1.215	1.221	1.219
Average			14.62	79.27	3.81						

Source: Experimental trials conducted by the Author of this Research, 2021.

Calculation of each anticipated density (ρa) for each recipe was made using the following formula:

ρa = ma/va

where,

ma = total mass of the plate that was calculated in Chapter **3.10.9 Calculation of Percentage of "Bridge Parts" Filler**

va = anticipated volume of each plate which is equal to volume of the mold (67.2 cm^3)

ρa1 = 85.8 / 67.2 = 1.277 g/cm^3 (density Recipe 1)

ρa2 = 88.4 / 67.2 = 1.315 g/cm^3 (density Recipe 2)

ρa3 = 84.4 / 67.2 = 1.256 g/cm^3 (density Recipe 3)

Appendix F

Calculations and Data of Bending Strength (MPa) and Flexural Modulus (MPa)

In Table 27 calculations and data for flexural modulus and bending strength of each plate of Recipe 1 and the average of each recipe can be seen.

In Table 28 calculations and data for flexural modulus and bending strength of each plate of Recipe 2 and the average of each recipe can be seen.

In Table 29 calculations and data for flexural modulus and bending strength of each plate of Recipe 3 and the average of each recipe can be seen.

The results of those data and calculations can be seen in Chapter **4.4.3. 3-Point Flexure Test**

Table 27: Calculations and Data of Bending Strength (MPa) and Flexural Modulus (MPa) and their Average Results for Each Plate in Recipe 1 and Recipe 1 Average Result.

Calculations and data of Bending strength (MPa) and Flexural modulus (MPa) for Recipe 1											
Recipes	**Plates**	**Samples**	**Stress (N/mm^2)**	**Force at maximum Flexural strength (N)**	**Displacement at break (mm)**	**Length (mm)**	**Width (mm)**	**Thickness (mm)**	**Bending strength (MPa)**	**Flexural modulus (MPa)**
		8	0.92	51.3	2.25	68	14.62	3.8	24.79	8489.60
		7	0.79	43.8	2.23	68	14.62	3.8	21.16	7313.44
		6	0.83	46.3	2.41	68	14.62	3.8	22.37	7153.46
		5	0.79	43.8	2.26	68	14.62	3.8	21.16	7216.35
		4	0.85	47.5	2.46	68	14.62	3.8	22.95	7189.70
	Plate 1	Average	0.836	46.54	2.322				22.5	7472.509
		8	1.26	70	2.46	68	14.62	3.8	33.82	10595.35
		7	0.58	32.5	1.5	68	14.62	3.8	15.70	8067.60
		6	0.9	50	2.08	68	14.62	3.8	24.16	8950.74
		5	0.83	46.3	1.98	68	14.62	3.8	22.37	8706.99
		4	0.63	35	2.1	68	14.62	3.8	16.91	6205.85
	Plate 3	Average	0.84	46.76	2.024				22.6	8505.303
		8	0.72	40	1.72	68	14.62	3.8	19.33	8659.32
		7	0.72	40	1.64	68	14.62	3.8	19.33	9081.72
		6	0.63	35	1.73	68	14.62	3.8	16.91	7533.11
		5	0.76	42.5	1.9	68	14.62	3.8	20.53	8328.90
		4	0.79	43.8	2.19	68	14.62	3.8	21.16	7447.01
	Plate 4	Average	0.724	40.26	1.836				19.5	8210.012
Recipe 1	**Average**		0.8	44.52	2.06				21.5	8062.608

Source: Experimental trials conducted by the Author of this Research, 2021.

Table 28: Calculations and Data of Bending Strength (MPa) and Flexural Modulus (MPa) and their Average Results for Each Plate in Recipe 2 and Recipe 2 Average Result.

Calculations and data of Bending strength (MPa) and Flexural modulus (MPa) for Recipe 2										
Recipes	**Plates**	**Samples**	**Stress (N/mm^2)**	**Force at maximum Flexural strength (N)**	**Displacement at break (mm)**	**Length (mm)**	**Width (mm)**	**Thickness (mm)**	**Bending strength (MPa)**	**Flexural modulus (MPa)**
		8	1.14	63.8	2.29	68	14.62	3.8	30.83	10373.79
		7	1.1	57.5	2.07	68	14.62	3.8	27.78	10343.08
		6	0.86	45	1.89	68	14.62	3.8	21.74	8865.49
		5	0.84	43.8	2.16	68	14.62	3.8	21.16	7550.44
		4	0.74	38.8	2.44	68	14.62	3.8	18.75	5920.99
	Plate 1	Average	0.936	49.78	2.17				24.05	8610.758
		8	0.72	40	1.38	68	14.62	3.8	19.33	10792.77
		7	0.94	52.5	1.86	68	14.62	3.8	25.37	10509.90
		6	1.15	60	2.35	68	14.62	3.8	28.99	9506.83
		5	0.81	45	1.81	68	14.62	3.8	21.74	9257.34
		4	0.79	43.8	2.03	68	14.62	3.8	21.16	8033.97
	Plate 3	Average	0.882	48.26	1.886				23.32	9620.162
		8	0.72	40	2.7	68	14.62	3.8	19.33	5516.31
		7	0.74	41.3	2.6	68	14.62	3.8	19.95	5914.65
		6	0.63	35	2.33	68	14.62	3.8	16.91	5593.25
		5	0.79	43.8	2.16	68	14.62	3.8	21.16	7550.44
		4	0.72	40	1.92	68	14.62	3.8	19.33	7757.31
Recipe 2	**Plate 4**	Average	0.72	40.02	2.342				19.34	6466.391
	Average		0.846	46.02	2.13				22.76	8421.30

Source: Experimental trials conducted by the Author of this Research, 2021.

Table 29: Calculations and Data of Bending Strength (MPa) and Flexural Modulus (MPa) and their Average Results for Each Plate in Recipe 3 and Recipe 3 Average Result.

Calculations and data of Bending strength (MPa) and Flexural modulus (MPa) for Recipe 3										
Recipes	**Plates**	**Samples**	**Stress (N/mm^2)**	**Force at maximum Flexural strength (N)**	**Displacement at break (mm)**	**Length (mm)**	**Width (mm)**	**Thickness (mm)**	**Bending strength (MPa)**	**Flexural modulus (MPa)**
		8	1.24	68.8	2.69	68	14.62	3.8	33.24	9523.32
		7	1.24	68.8	3.09	68	14.62	3.8	33.24	8290.53
		6	1.08	60	2.84	68	14.62	3.8	28.99	7866.56
		5	0.99	55	2.63	68	14.62	3.8	26.57	7786.80
		4	0.88	48.8	2.75	68	14.62	3.8	23.58	6607.53
	Plate 2	Average	1.086	60.28	2.8				29.12	8014.949
		8	0.99	55	2.87	68	14.62	3.8	26.57	7135.64
		7	0.9	50	2.71	68	14.62	3.8	24.16	6869.94
		6	0.52	28.8	1.77	68	14.62	3.8	13.91	6058.59
		5	0.97	53.8	3.1	68	14.62	3.8	25.99	6462.09
		4	0.89	46.3	2.7	68	14.62	3.8	22.37	6385.13
	Plate 3	Average	0.854	46.78	2.63				22.60	6582.276
		8	0.85	47.5	2.21	68	14.62	3.8	22.95	8003.01
		7	0.76	42.5	2.04	68	14.62	3.8	20.53	7757.31
		6	0.82	42.5	2.14	68	14.62	3.8	20.53	7394.82
		5	0.92	51.3	2.74	68	14.62	3.8	24.79	6971.38
		4	0.88	48.8	2.9	68	14.62	3.8	23.58	6265.76
Recipe 3	**Plate 4**	Average	0.846	46.52	2.406				22.47	7278.456
	Average		0.928667	51.19333	2.612				24.93	7353.585

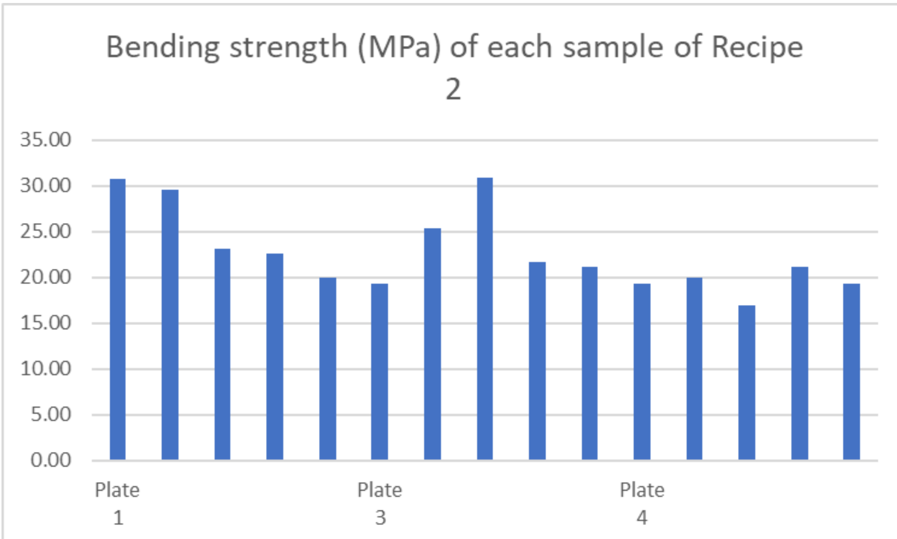
Source: Experimental trials conducted by the Author of this Research, 2021.

Graphs of Bending Strength (MPa) and Flexural Modulus (MPa)

Graphical comparison of the bending strength (MPa) and flexural modulus (MPa) for each sample for each plate for each mixture can be seen on Graphs 9, 10, 11 and 12 below.

On Graph 7 bending strength (MPa) of Each Sample starting from left two right starting from sample number 8 up to 4, then the next plat starts, for Recipe 2 can be seen.

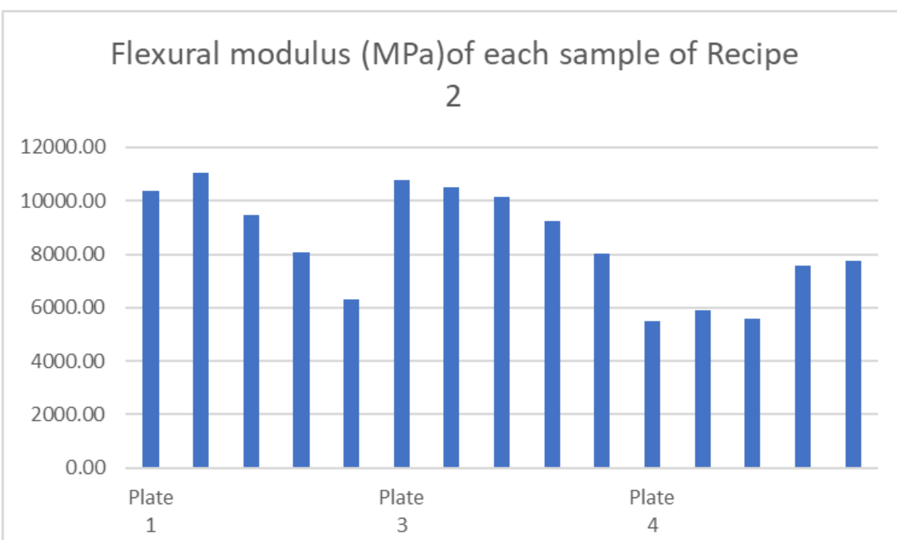
Graph 9: Bending Strength (MPa) of Each Sample



Source: Graph made by the Author of this Research, 2021.

On Graph 8 Flexural Modulus (MPa) of Each Sample starting from left two right starting from sample number 8 up to 4, then the next plat starts, for Recipe 2 can be seen.

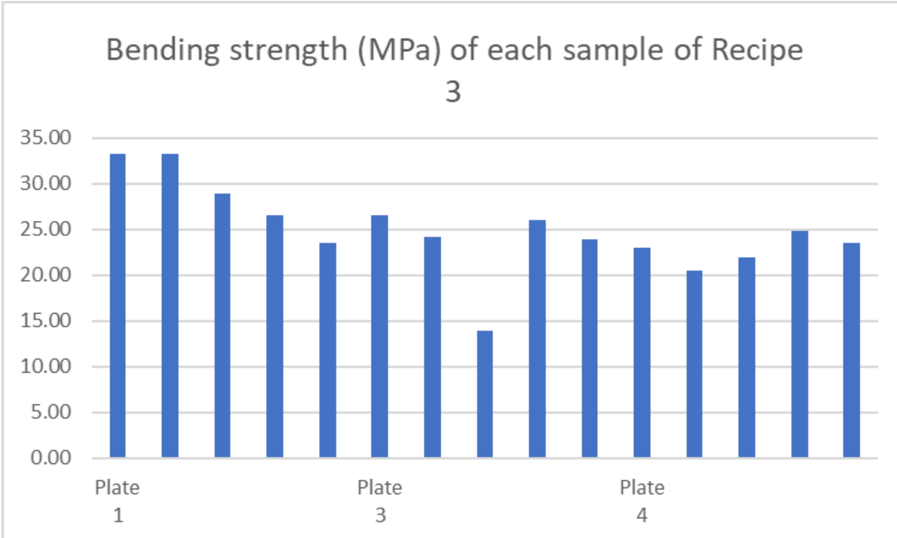
Graph 10: Flexural Modulus (MPa) of Each Sample.



Source: Graph made by the Author of this Research, 2021.

On Graph 7 bending strength (MPa) of Each Sample starting from left two right starting from sample number 8 up to 4, then the next plat starts, for Recipe 3 can be seen.

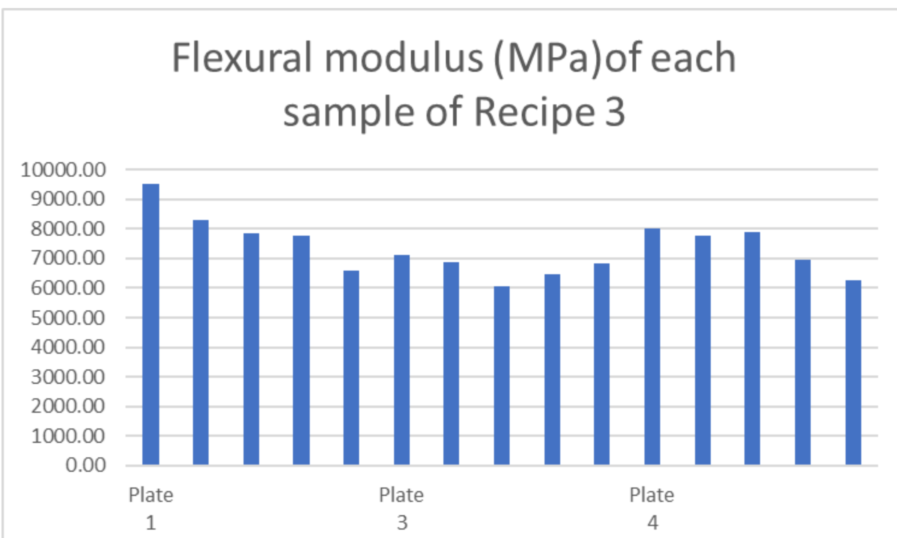
Graph 11: Bending Strength (MPa) of Each Sample.



Source: Graph made by the Author of this Research, 2021.

On Graph 8 Flexural Modulus (MPa) of Each Sample starting from left two right starting from sample number 8 up to 4, then the next plat starts, for Recipe 3 can be seen.

Graph 12: Flexural Modulus (MPa).



Source: Graph made by the Author of this Research, 2021.

Appendix G

Planning

Methodology plan

To explain how this Research shall be carried out Table 37 has been made to show the deliverables and sub-deliverables of each phase. After that for each phase actions to make, deliverables and sub-deliverables are indicated.

Table 37: Plan of Objectives to be Done in Each Phase and Sub-actions to Achieve it

Phase	Deliverable	Sub-deliverable	Action
Start-up phase	Research proposal	Introduction part	Writing the Introduction part
		Limits	Writing the Limits
		Theoretical framework	Writing the theoretical framework
		Methodology	Writing the methodology
		Research schedule	Writing the Research schedule
		Upload project proposal draft/final	Disclosing research proposal with coach
			Fixing and improving Research proposal
			Submitting Research
	Mid-term presentation		Making the mid-term presentation
			Presenting the mid-term presentation
	Initial material		Making the sample of the initial material
	Meeting HZ graduation lecturer		Organizing the meeting
			Having the meeting
	Initial knowledge about the project		Talking with the client
Working phase	Research results	Test results	Cutting the recyclent
			Crushing the cut recyclent
			Making the material plates
			Cutting plates into samples
			Conducting tests[1]
			Analysing the result
		Relation between the properties of the recycled thermoset (bio) composite material and its mixture	Analysing the effect of the mixture on material and finding correlation, if any
		Samples of the material	Making a sample of best performing mixture
		The properties useful for the material used for civil engineering	Making desk research on what properties are important for civil engineering

		Structures composite materials are used in the Netherlands	Making desk research on what composite materials are used in the Netherlands
		Number of composite materials used in those structures	Making desk research on the number of composite materials used in those structures
		The possible applications for the building industry of recycled biocomposite materials	Making desk research on applications for the building industry of recycled composite materials
			Analysing data and results of test properties and finding what bio recycled composite can be used for in the civil engineering department
Finishing phase	Research Paper	Finished Research paper	Combining all the parts of the Research together
			Checking with the coach if the Research is complete
			Adding missing parts if necessary, polishing, formatting the final document
			Submitting the final version of the Research
		Final presentation	Making a final presentation
			Presenting a final presentation

[1] Tests to be conducted are explained in detail in Chapter **4.4. Tests.**

Source: Plan conducted by the Author of this Research, 2021.