Improving the strength of **bio-based** direct ink writing



Master thesis

Improving the mechanical strength of bio-based direct ink writing

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Abstract

Direct ink writing is emerging as a sustainable alternative to conventional 3D printing techniques, promising significant reductions in energy consumption. This thesis presents a comprehensive investigation into the optimization of DIW processes, focusing on the development and evaluation of various ink formulations to identify the strongest recipes for producing materials suitable for prototyping within a circular economy.

A systematic methodology was devised to produce test samples both by casting and printing. For the DIW samples, achieving a balance between the required ink volume and the output volume was crucial to ensure consistent printing. Iterative adjustments to the g-code parameters and air pressure were made to fine-tune the printing process, resulting in the production of samples for mechanical testing.

Three-point bending tests were conducted on both casted and printed samples to evaluate their mechanical properties. Stress-strain curves obtained from these tests were analysed to determine the flexural strength and overall mechanical performance of each ink formulation. Among the various recipes tested, Carob combined with alginate emerged as the strongest, demonstrating mechanical properties comparable to those reported in existing literature. Confirming its potential as a viable candidate for use in prototyping.

The findings of this research underscore the importance of optimising both material composition and production parameters in DIW processes. By successfully identifying and validating an ink formulation, this work contributes to the advancement of sustainable 3D printing technologies.

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1. Introduction

This chapter initiates an investigation into the connections between Additive Manufacturing (AM), material strength, and the circular economy. Starting with a structured search plan, our inquiry delves into the foundational aspects of AM, providing an understanding of the parameters and considerations within AM. Subsequently, we delve into the Mechanical Properties of Materials, providing a foundational understanding essential for dissecting the impact of 3D printing on material strength.

Within the framework of the circular economy, our examination navigates the alignment of AM practices with principles of sustainability and resource efficiency. Moreover, an in-depth analysis of paste printing variables is undertaken, explaining the intricacies of recipe formulation and ingredient selection. Concluding this chapter, recommendations from research are summarised as well as a knowledge gap identified. The chapter ends with the research questions this report addresses.



1.1 | Search plan

In conducting this research, the search operator employed was:

"material? strength" OR "tensile strength" OR sturdiness OR "flexural strength" OR "bend" resistance"

AND

"liquid deposition modelling" OR "paste? extrusion" OR "additive manufacturing with paste?" OR "3D printing" OR "viscous material printing"

AND

binder?

AND NOT

concrete

The development of this search term is outlined in Appendix A. Additionally, the Web of Science platform has been used for the search, and the detailed protocol can be found in Appendix B. To further validate the findings, a literature overview authored by Romani et al. (2023) was consulted and as well as a database provided by Jeremy Faludi [personal communication] on which a comprehensive analysis was conducted as a double-check. Implementing the snowball method, the connected papers feature in the software was also leveraged, which led to the discovery of additional relevant papers.



Figure 2: Connected papers to Sauerwein et al. (2020)

1.2 | Additive Manufacturing and strength

1.2.1 Introduction to additive manufacturing

Additive manufacturing (AM), also known as 3D printing, represents a paradigm shift in modern manufacturing, reshaping the landscape of production processes. This approach involves building three-dimensional objects layer by layer from digital designs, offering flexibility and precision. Among the vast array of AM techniques, two prominent methods stand out for this research: Fused Deposition Modelling (FDM) and Direct Ink Writing (DIW).

FDM, a notable variant of AM, involves the extrusion of solid thermoplastics, known as filaments, through a heated nozzle to create semi-liquid material layers on a build platform. While FDM has been a staple in the 3D printing realm, its reliance on elevated temperatures limits its compatibility with certain materials and poses challenges for printing bio-based substances. However, FDM offers a wide selection of thermoplastics, including popular choices like PLA, PETG, and ABS, known for their ease of use and decent engineering properties. A downside of FDM is the large energy consumption of the heated elements (Faludi et al., 2019) and the circularity of printed objects.

In contrast, DIW distinguishes itself by its operation at room temperature, necessitating drying of printed objects. This characteristic makes DIW particularly well-suited for printing bio-based materials and substances with diverse viscosities. Its versatility enables the fabrication of structures with varied mechanical, electrical, or biological properties, making it indispensable across industries such as tissue engineering, microelectronics, and advanced manufacturing.



Figure 3: Schematic of FDM and DIW, adapted from Henssen (2023)

As the field of additive manufacturing continues to evolve, exploring the capabilities of both DIW and FDM provides invaluable insights into the diverse applications and possibilities of this technology. Whether it is pioneering sustainable manufacturing practices or pushing the boundaries of material science, these techniques pave the way for a future defined by efficiency, versatility, and sustainability in manufacturing.

In both FDM and DIW, several factors must be taken into account, including layer height, orientation, and infill pattern.

In FDM, layer height typically falls within the range of 0.1 mm to 0.25 mm. Common infill patterns include rectilinear and gyroid, although most slicing software provides a variety of options. The feasible layer height is dependent upon the machine, particularly the size of the nozzle in use. While a typical infill density is around 20%, values can vary from 0% (printing solely the outer shell) to 100% (a completely solid object).



Figure 6: Rectilinear infill pattern (Figure credit: Prusa Research, n.d.)





Figure 4: Schematic of parameters in FDM

1.2.2 Mechanical properties of materials

Materials belong to distinct families, each with numerous generalised properties. Michael Ashby describes various material characteristics, including general ones like density and market price, as well as mechanical properties such as strength and scratch resistance. Thermal properties encompass a material's response to temperature variations, while electrical and magnetic properties define its behaviour under respective conditions.



Figure 7: Material families (adapted from Ashby (2021)

In this study, crucial among mechanical properties are the elastic modulus, representing the slope of a graph and indicating a material's tendency to deform when subjected to stress, and the yield strength, marking the point where plastic deformation begins. And lastly the ultimate tensile strength, signifying the maximum stress a material can withstand. The ultimate tensile strength tends to be higher than the yield strength for metals and ductile polymers compared to ceramics and brittle polymers, where both strengths are comparable. From these properties the flexural modulus can be derived, which shows the tendency of a material to resist bending. It quantifies the relationship between stress and strain in the elastic region of the material's deformation. Higher flexural



Figure 8: Sample design for tensile, compression and 3 point bending tests

modulus values indicate a stiffer material that resists bending, while lower values indicate a more flexible material. These properties are assessed through various tests, most commonly; tensile, compression, and three-point bending, each probing different aspects of a material's behaviour.

For instance, concrete excels in compression but fares less impressively in tension or three-point bending. To address this lack of tensile strength reinforced concrete has been developed. Reinforced concrete, a composite material, demonstrates enhanced tensile strength and flexural strength but loses some compressive strength.

Each test generates a stress-strain curve . The characteristics are influenced by various factors, including anisotropy, wherein a material behaves differently depending on the direction of applied force. Wood, for instance, exhibits contrasting behaviour along and perpendicular to its grain, illustrating this phenomenon.



Figure 9: Anisotropy of wood

The stress-strain curves reveal key parameters like the elastic modulus, yield strength, and ultimate tensile strength. Each of these parameters occurs at a specific strain, the maximum strain a material can withstand is called strain at break. Materials exhibiting significant plastic deformation before failure are termed ductile, while those failing abruptly are considered brittle. Examples include steel as a ductile material and glass as a brittle one.



Strain [%]

Figure 10: Generalised stress-strain curve for ductile polymers (adapted from Ashby (2021))



Figure 11: Generalised stress-strain curve for ductile metals (adapted from Ashby (2021))

1.2.3 Effect of 3D printing on strength

The impact of 3D printing on material strength is a multifaceted subject, as delineated by various researchers. Dave et al. (2019) highlight that infill density plays a crucial role, with tensile strength being most significantly affected, followed by considerations of infill pattern and orientation. They found that a rectilinear print laid flat exhibits the highest strength under tension in the direction of the print lines. Lee et al. emphasise the anisotropic nature of materials produced through 3D printing, indicating that strength varies depending on the direction of force application. Furthermore, Giri et al. point out that factors such as layer thickness, cooling rate, and printing orientation have implications for build time, layer adhesion, and ultimately tensile strength. The correlations are illustrated in Figure 12, showcasing that there is an optimal region for maximum tensile strength. These insights illustrate the complex interplay between printing parameters and material properties, underscoring the need for meticulous control and consideration in 3D printing processes, similar to the precision found in traditional casting methods.



Figure 12: Correlations between cooling rate [%] and layer thickness [mm] and effect on tensile strength [MPa] for PLA printing. (Adapted from Giri et al. (2021))



1.3 | Circular economy frame

1.3.1 Relevance

Embracing circular economy design is crucial in addressing the climate crisis. Additionally, there exists a personal motivation for engaging in this approach, as it represents a systemic solution and not solely focused on minimising negative outcomes.

The butterfly diagram, depicting the circular economy system, illustrates the ongoing flow of materials within a circular economy. This system consists of two primary cycles: the technical cycle and the biological cycle. In the technical cycle, products and materials are sustained in circulation through practices such as reuse, repair, remanufacture, and recycling. On the other hand, the biological cycle involves returning nutrients from biodegradable materials to the Earth, contributing to the regeneration of nature (composting). For direct ink writing, both recycling and composting are interesting aspects (Ellen Macarthur Foundation, n.d.). "In our current economy, we take materials from the Earth, make products from them, and eventually throw them away as waste – the process is linear. In a circular economy, by contrast, we stop waste being produced in the first place."

- Ellen McArthur Foundation



Figure 14: Ellen McArthur Foundation Butterfly model. Figure credit: Ellen Macarthur Foundation (n.d.)

1

1.3.2 **3D printing in the Circular** Economy

The impact of 3D printing is multifaceted, with notable considerations in the domains of energy consumption, material usage, and embodied energy of the printer itself. Research conducted by (Faludi et al., 2019) reveals that among FDM printers, energy consumption has the most substantial impact, closely followed by the embodied energy of the printer itself. However, when printers are utilised more efficiently, the use of printing materials and waste increases in importance. The embodied energy is predominantly influenced by the electronics, while the energy consumption in ABS printing is attributed to heating.

Direct ink writing emerges as a promising solution to tackle aspects of traditional 3D printing. By relying on evaporation at room temperature, this method significantly diminishes the energy demand required to operate the printer. However, there is a marginal increase in energy consumption attributed to the regulation of air valves and fans for drying purposes as described by Faludi (2019). Moreover, the materials employed in paste printing exhibit lower embodied energy, although this requires thoughtful evaluation. Finally, the embodied energy of the printer may experience a reduction owing to the use of less impactful parts, this remains a subtle modification.



Figure 16: 3D printed helmet. Figure credit: HEXR (n.d.)

The key to achieving these reductions lies in utilising direct ink writing, essentially involving the development of a new material. This material has to be suitable for the application it is intended for. 3D printing offers diverse applications, ranging from crafting specialised jigs for car production to producing custom bicycle helmets (HEXR, n.d.) (Schwaar, 2021).



Figure 15: Impact of 3D printing. Figure credit: Faludi et al. (2019)

A substantial aspect of 3D printing is dedicated to prototyping, according to Chapman (2023). In the context of this project, emphasis is placed on material requirements specific to prototyping. This focus is justified primarily by its significant share in overall 3D printing. Additionally, products in prototyping are typically used for a brief duration as they are part of a fast paced iterative process, resulting in lower material demands, without the need for extensive water or UV resistance and often not requiring exceptional strength, as indicated by Chapman. The brief lifespan also entails swift discarding, making it particularly compelling to enhance sustainability in this aspect. Therefore, the decision to concentrate on prototyping is intentional.

In pursuit of circularity in 3D printing, various strategies can be employed, essentially focusing on either of the two cycles as depicted by the butterfly model. One approach involves selecting materials that align with the biocycle, necessitating the material to be compostable. Although first focus should lie on cascading the materials by for example utilising waste stream food to create textiles as has been done by Orange Fiber (Orange Fiber, 2019). Using leftover citrus peels from juice production to manufacture textiles that can be transformed into clothing (Orange Fiber, 2019).

Alternatively, one can emphasise looping within the technical cycle. Particularly for the materials used, recycling assumes significance since each 3D printed part is inherently designed for its specific use, limiting possibilities for reuse, repair, or remanufacture. An excellent example of a material demonstrating a nearly closed technical loop is aluminium. Given its high value and the ability to undergo nearly infinite resmelting, the recycling rate for aluminium currently stands at 69% within the EU (EuRIC AISBL, n.d.). Notably, there are established policies to promote aluminium recycling, such as the recent inclusion of aluminium cans in the deposit system in the Netherlands (Ministerie van Algemene Zaken, 2024). This stands in contrast to PLA, which technically can be remelted but faces challenges in recycling due to costs and degradation (CE Delft, 2021). Thus a technical cycle for PLA is in theory possible but faces many practical hurdles, that is why this project aims to improve circularity by focusing on direct ink writing.



Figure 17: Orange Fiber example of fabric. Figure credit: Orange Fiber (2022)

In order to achieve more circularity an ideal material would be easily biocycle compatible and multiple times reprintable, in other words multiple loops within the technical cycle. It is important to include the biocycle in this statement because eventually the material will be discarded. And since it is highly unlikely that a low value material is capable of endlessly looping within the technical cycle, end of life needs to be thought of.

However to make the material demands more realistic for this project the focus should lie on the following parts. First of all biocycle compatible, meaning ultimately compostable (Ellen Macarthur Foundation, 2022). Secondly the material should be printable by room temperature, relying on either evaporation or chemical bonding. Lastly, the material should hold sufficient strength to meet the demands of most prototyping purposes. Testing can determine these values during research.

Narrowing the project's scope to these three facets inevitably results in excluding various other considerations. This encompasses aesthetics, the exploration of potential waste stream utilisation, the proximity of material sources, and even the concept of reprinting. While these factors are pivotal for mitigating the impact of 3D printing, especially reprinting, and enhancing the circularity of the technique, the project is constrained by limited time, making them secondary focal points. This is summarised below. These demands and wishes will serve as a guideline throughout the project.

Demands

- Paste printable
- Compostable
- Strong enough for prototyping

Wishes

- Sourced from a waste stream
- Local material supplychain
- Reprintable for 5+ cycles
- Abundant resource

1.4 | Polylactic Acid

1.4.1 Material properties

Polylactide, also known as PLA, is a biodegradable thermoplastic sourced from natural lactic acid derived from corn, maize, or milk. It bears a resemblance to clear polystyrene, offering favourable aesthetics with gloss and clarity. However, its inherent stiffness and brittleness necessitate modification through the incorporation of plasticizers for optimal use in various practical applications. PLA exhibits versatility in processing, enabling its transformation, like many thermoplastics, into fibres, films, and products through processes such as thermoforming, injection moulding or 3D printing (ANSYS, 2022).

In Table 1 the mechanical properties for PLA are shown as well as properties of 3D printed parts made of PLA. As can be seen 3D printing impacts the strength, the tensile strength is slightly below that of conventional PLA. This effect is highly dependent on the printing settings such as orientation, infill percentage, layer height, printing speed and the resulting layer adhesion (Ultimaker, 2022) used during production.

In Figure 18 the relative properties of PLA can be seen. PLA is ranged in the middle of plastics being slightly denser than polycarbonate and significantly denser and stronger than commonly used polypropylene. The tensile strength of PLA is higher than most other plastics, whereas 3D printing PLA is more in the middle of the material family. In Figure 18 the compressive strength of the plastics can be seen. Here PLA is among the stronger materials, being comparable to PC. 3D printed PLA would be more comparable to PET.

Property	Unit	PLA Material	PLA 3D printed	
Density	kg/m3	1.25E3	1.24E3	
Yield strength	Мра	50-55	52.9	
Tensile stress at break	МРа	55-72	45.5	
Compressive strength	Мра	66-87	57	
Elongation at yield	% strain	2-3.5	3.4	
Flexural modulus	GPa	3.1-3.6	3.0	

Table 1: Mechanical properties of PLA ((Ansys, 2022) (Ultimaker, 2022) (Kumar & Narayan, 2018) (PrusaPolymers, 2021))



Figure 18: Tensile strength plotted against density with PLA highlighted adapted from ANSYS (2022)



Figure 19: Compressive strength plotted against density with PLA highlighted adapted from ANSYS (2022)

1.4.2 Benchmark

In order to define a baseline a three point bending test has been conducted to conform to ISO 178:2019 on a Zwick Roell type Z010 machine. Samples were produced using a Prusa mini equipped with a 0.4mm nozzle and a layer height of 0.25mm. Prusament Recycled PLA obtained from Prusa Research was used as a filament.



Figure 20: Zwick Roell Z010 machine used for testing

In Figure 24 the stress strain curves of horizontal and vertically printed samples can be seen. For stress -strain curves of each sample refer to Appendix C. It is clear that a higher infill percentage results in a higher strength. The error bar for horizontally printed samples is much smaller than for vertically printed. Vertical fails along the layer lines, where layer adhesion determines strength causing more variance. Whereas horizontal fails perpendicular to the layers where the inert strength of PLA is more important and more consistent. Horizontally printed samples show more variance after the maximum stress is reached. For vertically printed the samples with 60% infill are much closer together than samples

with 80% infill where more variance can be observed. This benchmark shows different values for stress and strain and the resulting flexural modulus than literature for similar samples, as can be seen in Figure 22 and Figure 23. Considering that the difference is a factor 2,2 caution needs to be taken when interpreting data. Since future three point bending tests will be conducted in a similar manner as this benchmark these values will be used for reference as has been agreed upon with the supervisory team.

The strength of PLA is used as reference since it is the main material used for prototyping see Chapter 1.3 |. Materials that possess half the tensile strength of PLA are for example PHA, commonly used for shampoo bottles. On the lower end of plastics TPS commonly used for single use cutlery (ANSYS, 2022). Ideally paste printing equals PLA but lower tensile strength could also be sufficient depending on the kind of prototyping done. As described in Chapter 1.2 |strength has various aspects, it is important to keep that in mind when comparing materials.







Figure 24: Stress-Strain curve of PLA Benchmark

1.5 | Paste printing variables and constraints

This chapter explores the complex variables and challenges of direct ink writing. From viscosity fluctuations to buildability issues, and from shrinkage unpredictability to nozzle clogging, achieving optimal strength and durability in printed objects poses significant hurdles. The chapter concludes with a summary of recipes that have been evaluated by other researchers.

Printability

Printability describes the ability of a mixture to be successfully printed on the available machines. Navigating challenges encountered during direct ink writing presents significant complexities that impact the reliability and reproducibility of results. One primary issue observed is the fluctuating viscosity of the mixture, which affects printability. Despite utilising consistent code and batch, the printer produces inconsistent results, necessitating adjustments to air pressure within the same batch to accommodate viscosity changes, typically between 0,1 and 0,45 MPa. This is further complicated by the lack of real-time controls on the Eazoa Bio printer, which prevents pausing, slowing down, or increasing the flow during printing.

Shear-thickening (dilatant) liquids exhibit an increase in viscosity as the rate of shear strain rises. Conversely, shear-thinning liquids experience a

decrease in viscosity as the rate of shear strain increases. Thixotropic liquids become less viscous quickly when subjected to shaking, agitation, or stress. Thixotropy is important for 3D printing DIW pastes, because a slow response time to shear forces extruding the material from the syringe will cause poor extrusion at the beginning of every print line, only reaching adequately low viscosity after some time. Bingham plastics display solidlike behaviour at low stresses but transition into a viscous fluid state at high stresses.



Figure 25: Behaviour of fluids



The characteristic of shear thinning enables smoother extrusion through the nozzle and enhances control over ink flow. Such behaviour is essential for precise deposition, preventing discontinuous extrusion and minimising the risk of nozzle clogging (del-Mazo-Barbara & Ginebra, 2021).

The factors underlying fluctuations in viscosity are not fully understood. One potential factor could be the variability between batches of ingredients, as all ingredients are natural and therefore exhibit inherent variation. Another factor may involve molecular interactions between particles, which have yet to be thoroughly investigated. Attempts have been made to address these issues through adjustments to water and ethanol content; however, establishing a definitive cause-andeffect relationship remains elusive.

The most important parameters are;

- Air pressure
- Nozzle diameter
- Mix viscosity
- Printing speed



Vrequired depends on Vhead & Anozzle

Voutput depends on $p_{air} \& \mu$

> where μ is non-linearly correlated to pair



Vrequired = Voutput









Buildability

Achieving optimal buildability is another challenge, wherein the material must strike a balance between drying quickly enough to support subsequent layers and keep dimensional accuracy but not drying too rapidly, which could compromise adhesion between layers.

Inadequate drying leads to sagging, impacting layer height and causing issues across multiple layers.

The time a layer has to dry sufficiently is dependent on the printing speed and the area of the layer that is being printed. The larger the area the more time the material has to dry, the higher the speed the less time it has to dry. And the more layers you print on top of each other the stronger the effect of sagging becomes. Where the top layers create a pressure on the bottom layers causing the material to become liquid again due to shear thinning.

The rate of drying is also dependent on the environmental conditions, the most common solvent, water, evaporates at different rates at different temperatures and humidity. Similar behaviour can be observed in ethanol, a common solvent for enhancing buildability. Additionally fans have an impact on airflow that directly affects the evaporation rate. However, it is important to acknowledge that current machines lack control over these fans.

Henssen (2023) provides insight into the impact of environmental conditions on the printing process. Stating that environmental conditions influence the print quality but are not the sole factor explaining variance between prints.



Figure 29: Example of a sagging print



Figure 30: Printing speed and layer area correlation



Figure 31: Example of a too high printhead

Shrinkage

Shrinkage, especially in the Z-axis, poses a notable challenge that is difficult to anticipate and manage, leading to challenges in the reproducibility of printed objects. This shrinkage is caused by the evaporation of water and is more visible in recipes with a large share of water.

As outlined by (Kam et al., 2019), printing along different pathways induces anisotropic shrinkage, which in turn results in cracking. To mitigate this issue, Doron Kam (Kam et al., 2019) utilised a Teflon substrate, facilitating the easy removal of prints from the print bed prior to drying. Furthermore, the researcher placed the printed objects in a sealed chamber for 48 hours to ensure thorough drying. Both of these strategies can be employed to improve the number of successful prints.

Nozzle clogging

Nozzle clogging is also a concern, particularly when excessive extrusion occurs due to high air pressure, low viscosity, or slow printing speeds. The excessive extrusion causes a lump of the material to stick to the nozzle and dry out, causing the blockage. There are more factors that cause blockage, such as clumps in the mix or drying out of the mix in the syringe. For many cases of clogging no cause has been found and the most effective way forward is to replace the nozzle.



Figure 33: Nozzle with clumps

Air supply

Due to pressure in the syringe, material moves toward the air supply and dries up, as shown in Figure 32. In extreme cases, this can completely block the air supply. To resolve this issue, disassemble the air supply and clean out the tubes. Using clear tubing helps in identifying blockages early and taking timely action. Another option is to only fill the syringe till three quarters but that has as a downside that fewer prints can be made with one batch.

Figure 32: Clogged airsupply. From left to right, nozzle connector, supply line, internal tubing.



New ingredients

The introduction of new ingredients can alter the viscosity, necessitating a re-evaluation of ingredient ratios to obtain a shear-thinning mixture for successful extrusion. The variability introduced by these ingredients presents additional challenges where expertise is needed to make the necessary adjustments to one of the factors, for example printing speed, mentioned above.



Figure 34: Print test with high concentration gluten Other challenges

Literature research highlights further challenges in paste printing, including inconsistencies in surface finish, wobbliness of prints, and extended drying times. Klemmt et al., (2022) discusses the coarse surface finish of prints attributed to fibre content. Similarly, in previous research, graduating student Ennio Donders (2022) encountered instability in his prints, leading to their collapse. Both Donders and Edwin van Tongeren (2020) highlighted the prolonged drying time associated with paste printing, which can extend up to a week. This extended drying period is noteworthy, considering that 3D printing, including direct ink writing, is primarily utilised for rapid prototyping, deriving many of its advantages from its speed.

Although the focus of this research is on enhancing strength, these printing challenges significantly impact the final outcomes. For example, despite the potential improvements in strength observed with certain additives like gluten, their nonprintability in high concentration renders them unsuitable for the research objectives.



Figure 35: Rough surface finish of prints made from plant fibre, adapted from Klemmt et al. (2022)

The overall strength of the printed bars remains a significant concern, with the risk of breakage during basic handling. This brittleness, comparable to unfired clay objects, is intensified by the thin walls and lack of solidity in the prints, an inherent part of many 3D printed objects . While some compounds exhibit promising strength properties, they must also be printable and align with the established research framework described in Chapter 1.3.

In summary, the research faces several challenges, including varying viscosity, buildability issues, unpredictable shrinkage, and nozzle clogging, among others. Understanding and addressing these challenges are crucial for advancing the field of paste printing and achieving the desired strength and durability in printed objects.



Figure 36: Wobbliness prints rolling behaviour of layers; (a) ideal situation, (b) situation for overhang, (c) situation for extrusion inconsistencies. Adapted from

Examples of strong recipes

A search has been performed to assess the state of the art in strong DIW recipes using the search protocol outlined in Chapter 1.1. All the papers analysed were assessed based on their alignment with the Circular Economy framework outlined in Chapter 1.3. Strength data from these papers was collected, and a method was developed to compare the diverse reporting styles employed. For a more comprehensive overview, including all analysed papers, please consult Appendix D. The table presented below showcases the most effective recipes tested by different researchers, listing the ingredients utilised in another table below. The entries in the table are not ranked, as each researcher presented their data differently, often not including more than two strength tests, making direct comparisons challenging to execute.

Table 2 shows that Sauerwein et al. (2020) is the closest to PLA values, particularly in Young's modulus where they have reached 64% of PLA. Sanandiya et al. (2018) excels in various strengths, while Scaffaro's recipes (2022b) outperform in tensile behaviour. This analysis shows that while excellence in a specific part of strength has been achieved, making a paste printed object that resembles PLA in all areas has not been done yet. It is important to remember that a trade-off between various aspects of strengths in inevitable and careful considerations must be made.

Sanandiya et al. (2018)			
FLAM CC (1:8)			
Chitosan [w/w]	1		
Wood flour [w/w]	8		
Sauerwein e	et al.,(2020)		
Mussel alginate	[wt%]		
Sodium Alginate	3		
H2O	36		
Mussel shell powder	61		
Scaffaro et	al., (2022b)		
FDM_MB/OFI-B	[wt%]		
MB	90		
OFI	10		
Scaffaro et	al., (2022b)		
FDM_MB/NPK-b	[wt%]		
MB	90		
NPK	10		

Table 3: If	ngredients	of strongest	recipes
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Table 2: Topfourstrongestrecipes,including PLA for reference

Paper	Recipe	Tensile strength [MPa]	Compressive strength [MPa]	Flexural strength [MPa]	Young modulus [MPa]
Sanandiya et al. (2018)	1.	11,31	15,31	15,03	244,1
Sauerwein et al. (2020)	Musselshell	-	-	9,8	2100
Scaffaro et al. (2022)	FDM_MB/ OFI-B	18	-	-	128
Scaffaro et al. (2022)	FDM_MB/ NPK-b	20	-	-	120
PLA (3D printed)	-	45,5	57	82	3250



Figure 38: Product made by Sanandiya et al. (2018). Image credit: Sanandiya et al. (2018)

1.6 | **Paste printing ingrediens**

In direct ink writing four main groups of ingredients can be used to formulate inks, referred to in this research as recipes. The four main groups are;

- Fillers
- Binders
- Solvents
- Additives

In Figure 39, the roles of each group are illustrated. It is important to recognize that the categorization of an ingredient within a group is not fixed. For instance, an additive could also function as a solvent if it serves as the primary fluid.

Fillers

Fillers are the main structural ingredients, providing mechanical performance and dimensional stability of printed objects. Cellulose biomass, sourced from agricultural waste or wood, represents a sustainable filler option. Its incorporation into ink formulations addresses concerns regarding resource competition and pollution associated with traditional fillers. Examples like pecan shell flour and tangerine peel demonstrate the versatility of biomass-derived fillers, offering not only mechanical reinforcement but also eco-friendly alternatives to conventional fillers. Additionally calcium based fillers such as eggshells or oyster shells provide an interesting aspect forming an organic matrix, improving strength.



Figure 39: Roles of each group of ingredients

Binders

Binders hold together the fillers, and the strength with which they bind them also limits the maximum strength of the material.Polysaccharides, found abundantly in biomass, offer a rich pool of materials for binder formulation. These polymers, comprising monosaccharides or sugars, demonstrate varying printing characteristics influenced by factors like concentration, chemical structure, and flow behaviour. Sodium alginate, chitin, starches, and others serve as common polysaccharide-based binders in DIW.

Proteins, composed of amino acids, exhibit remarkable potential in DIW ink binder formulations. Collagen, gelatine, and other proteins find applications in tissue engineering and food printing, where their hierarchical architectures and intermolecular bonding functionalities contribute to complex structure formation.

Solvents

Lastly, solvents play a pivotal role in ink formulation by dissolving and dispersing ink components while providing the necessary fluidity for extrusion. Controlling solvent evaporation rates influences ink solidification, affecting print quality, printing speed and structural integrity. Understanding the interplay between ink components and solvent properties is essential for achieving optimal printing conditions and desired material properties.

Additives

Additives further enhance ink quality and functionality, facilitating adjustments in rheological behaviour, stability, and additional functionalities. Rheology modifiers like Xanthan Gum and Guar Gum, dispersants, surfactants, and crosslinking agents contribute to ink stability, printability, and mechanical properties. Their strategic incorporation optimises ink performance, ensuring precise deposition and layer adhesion critical for high-quality prints. In summary, exploring the diverse array of biobased ingredients for DIW ink formulations holds potential for sustainable additive manufacturing. From polysaccharides and proteins to cellulose biomass and innovative additives, each ingredient contributes to environmentally friendly and highperformance printing solutions, paving the way for a more sustainable future in 3D printing.

Binders	Fillers	Solvents	Additives
Alginate	Cellulose	Ethanol	Calcium ions (calcium chloride)
Cement	Cellulose NanoCrystal (CNC)	Isopropyl alcohol	Glycerol
Chitin	Coffee grounds	Water	Nano- particles
Chitosan	Egg shell		Vinegar
Collagen	Hardwood flour		
Corn starch	Maple wood Flour		
Gelatin	Methyl cellulose		
Guar gum	Mussel shell		
Gypsum	Olive pomace		
Potato starch	Oyster shell		
PVA	Pecan flour		
Sodium silicate	Pine wood flour		
Sugar	Soft wood flour		
Xanthan Gum (XG)	Walnut shells		
	Wood Flour (WF)		

Table 4: Fillers, binders, solvents and additives

 currently used

1.7 | **Project Goal**

1.7.1 Recommendations from literature

Prioritising improvements in mechanical strength and dimensional stability analysis lays the foundation for understanding the material's structural integrity under varying conditions, as described by Rosenthal et al. (2022). Concurrently, enhancing mechanical strength is crucial to fortify the material against external stresses, ensuring its reliability in real-world applications (Whyte et al., 2019). Addressing lower brittleness, as described by Donders (2022), is one of the vital focus points within material strength as it is the greatest challenge presently.

Romani et al., (2023) puts emphasis on fostering knowledge sharing among stakeholders, which is essential for collective understanding and continuous improvement, allowing insights to be disseminated and applied effectively across teams.

Investigating curing additives (Klemmt et al., 2022) and exploring material properties (Sauerwein et al., 2020) are essential steps towards enhancing the material's functionality and versatility, enabling tailored solutions to specific requirements. Necessitating further testing with materials already investigated and potentially making new combinations. Implementing in-situ solidification methods (Henssen, 2023) could be a path forward for this.

Measuring printer energy consumption (Donders, 2022) and optimising rheology (Henssen, 2023) contribute to sustainable production practices, minimising resource consumption while maximising efficiency.

Conducting tests on strength after recycling (van Tongeren, 2020 and Sauerwein et al., 2020) underscores the importance of sustainability, allowing for the assessment and optimization of recycling processes to minimise waste and environmental impact. Furthermore, controlling environmental conditions (Henssen, 2023) ensures consistency and reliability in material

properties, mitigating potential deviations caused by external factors.

Exploring faster paste creation processes (van Tongeren, 2020) contributes to streamlining production workflows, reducing lead times, and enhancing overall efficiency.

1.7.2 Knowledge gap

The exploration of mechanical strength, additives, and ingredient interactions is crucial in the context of paste printing. While numerous researchers have experimented with various fillers and binders, there is a notable gap in mechanical strength achieved when compared to commodity plastics.

Additional knowledge gaps within the realm of paste printing encompass reliable printing aspects such as nozzle clogging, paste viscosity, and environmental conditions. Furthermore, the challenges related to recyclability and energy consumption remain areas that warrant further investigation. These gaps in understanding pose opportunities for comprehensive exploration and refinement in the field of paste printing technology.

The primary objective of this study is to address a significant challenge in paste printing, for that reason the focus lies on improving the mechanical strength. This choice was made considering the timeframe of the project and specific strengths that the researcher has.

1.7.3 **Research question**

RQ1 How can the mechanical strength of a printed object using bio-based direct ink writing be enhanced?

- RQ1.1 Which ingredient (s), or combinations of ingredients can provide more strength?
- RQ1.2 How can the new ingredients be printed?



2. Methodology

A fundamental aspect of this methodological approach is balancing insights gained from literature with practical experimentation. While established knowledge is often the base, ventures beyond conventional wisdom are also explored, occasionally challenging it to explore innovative solutions. The aim is to utilise waste stream ingredients where possible, but if they alone cannot provide the desired quality, necessitating the exploration of alternative sources. In summary, this method involves a comprehensive approach to ingredient selection, testing, and documentation, ultimately aimed at enhancing the strength of paste printed parts.



Figure 42: Mould used for casting, a test sample. PLA printed in dark green, held together by nuts and bolds to enable reuse.



Figure 41: Flowchart of methodology

2.1 | Material development

2.1.1 **Overview**

The overarching goal of this method chapter is to detail how new samples are developed with a focus on addressing issues of brittleness. In order to do this new ingredients are selected that show potential for addressing brittleness. In other words that have ductile behaviour. While selecting new ingredients the frame explained in Chapter 1.3 is kept in mind, which details which kind of ingredients belong to the options for this research.

The process begins by combining the new ingredient with water, followed by ethanol if relevant. Water, being an abundant, non toxic and accessible solvent plus ethanol for faster evaporation, aiding buildability. The next step is iteration on this base by incorporating filler ingredients, such as gluten and pecan shell flour, adjusting viscosity as needed. Details on these tests as well as background information on materials used can be found in Appendix E. Intermediate tests were casted in moulds to assess behaviour and gain insight.

As the material progresses there is continual refinement of g-codes, modifying parameters like printing speed, layer height, width, and wall line count to optimise the printing process, see Appendix F for details on the g-codes used. All samples were printed on a Eazoa Bio with a 20 gauge nozzle and a syringe of 50 mL. The printer is operated using a pneumatic system, supplied by the lab airline. If a material proved difficult to print, moulds were used, in order to still gain insights and determine if solving the printing issues would be a viable path.



Figure 43: Airsupply controls

However, not all prints are successful. When failures occur, the reasons are meticulously documented, some of which remain unknown and are detailed in the appendix G. From these experiences, core lessons are extracted and implemented into the next steps. The most influential lessons are detailed on the next page.

After priting samples were either air dried, or put in the climate chamber to control the drying conditions. Air dried samples were put in a cabinet in the lab exposed to ambient temperature and humidty, exact numbers are in Appendix F. The climate chamber used was a Hielkema Espec set to 22.5 °C and 65% humidity. Samples were left to dry for at least one week, exact time per sample can be found in Appendix F.



Figure 44: Samples drying in the climate chamber

The material exhibited distinct behaviours: while the corners manifested a sharp, well-defined structure, the central regions displayed sagging as can be seen in Figure 45. This phenomenon was indicative of shear thinning, attributable to the material being subjected to its own weight. A certain degree of shear thinning is desirable for pushing the material through the nozzle. However, too much shear thinning negatively affected the material's buildability, necessitating a reevaluation and subsequent adjustment of the formulation to optimise the buildability.

A secondary issue identified was that of under extrusion, as evidenced by the presence of fragmented lines in the printed output, as can be seen in Figure 46. This was attributed to the nozzle exerting excessive force on the previously deposited material caused by the speed of the printhead, leading to line discontinuities. To mitigate this, an increase in air pressure during the printing process was implemented.

Regarding nozzle calibration and print height, efforts were directed towards maintaining a uniform 1 millimetre distance between the nozzle and the print surface at the onset of each print job. However, intermittent discrepancies were noted, potentially stemming from factors such as vibrations or human error, which resulted in the nozzle being positioned at an elevated height relative to the preceding print layers as can be seen in Figure 48. This led to material dropping down onto the previously printed layers and a subsequent decline in print quality over the duration of the print job. Additionally, despite best effort to calibrate the printer before each print, some first layers were not printed optimally as can be seen in Figure 47. This issue manifested as a wavering skirt line due to an excessive nozzleto-bed distance and has been fixed by restarting the print.

For additional illustrative examples, readers are directed to consult the appendix G.



Figure 45: Example of sagging print



Figure 46: Example of underextrusion



Figure 47: Nozzle is too high



Figure 48: Wavering skirt line
2.1.2 Ingredient ranges

Throughout the process an array of ingredients have been tested which can be seen in Figure 49. Details can be found in Appendix F. Carob and Alginate only have a single value since the first quantity tested with printing was successful.

The recipes that have been explored include PSF, PSF + G, and Carob. Refer to Appendix F for iterations of these recipes. The baseline of pecan shell flour was chosen based on the result from database analysis and the success of another graduate student, Anne Henssen (2023). Integrating gluten showed promise based on the approach detailed previously. Carob was tested due to promising results from a research partner (N. Yusufova, personal communication, April 8, 2024).

PSF				
Pecan shell flour	11			
All binder	27			
H2O	13,75			
Ethanol	8,75			

Table 5: Ingredients of PSF recipe in grams

PSF + G				
Pecan shell flour	6,16			
All binder	27			
H2O	13,75			
Ethanol	8,75			
Gluten	4,62			

Table 6: Ingredients of PSF + G recipe in grams

Carob				
Carob	12			
Alginate	3			
H2O	35			

Table 7: Ingredients of Carob recipe in grams



Figure 49: Ranges of ingredients tested, arrows indicates which quantity was most successful

2.1.3 Printing ranges

The most important printing ranges are;

- Layer height
- Printing speed
- Flow

As can be seen in Figure 50 various ranges were tested to get to successful prints. It is essential to note that iterations ended when prints were successful, this research does not focus on the best print quality but on sufficient quality for testing mechanical strength.

See appendix F for the details on g-code. PSF, PSF + G and Carob samples 1-6 were printed using a CAD model V3 dimensioned to 6,4 mm in height to adjust for shrinkage in z-direction. Carob samples 7-11 were printed using model V4 to account for the larger shrinkage in all directions that was observed with Carob samples 1-6. See Figure 51.



Figure 50: Ranges of gcode parameters, arrow indicates which value was most succesful



Figure 51: Adjustment of CAD model for shrinkage

2.2 | **3 Point bending test**

The rationale behind conducting three-point bending tests lies in the unique properties and behaviours of the materials under study. Firstly, these tests are employed due to the inherent brittleness observed in similar materials, as indicated by previous literature see Chapter 1.5 |. This brittleness often makes it challenging to shape the material into a dog bone configuration and subsequently subject it to tensile testing without premature sample failure. Secondly, the interest is in examining the overall behaviour of the material, rather than focusing solely on compressive or tensile properties. Three-point bending is an exemplary method to achieve this comprehensive understanding.

In a three-point bending test, a rectangular bar, with dimensions specified as 4 millimetres in height, 10 millimetres in width, and 80 millimetres in length, is supported by two fixed points and subjected to bending forces using a moving head equipped with a circular attachment. This configuration is illustrated in a sketch for clarity, see Figure 52. The tests are conducted on a Zwick Z010 machine to track the head's travel against the applied force. Machine setup and sample dimension is as outlined in ISO standard 178.2019. Adjustments to the standard were made for Carob P AD samples 1-6 because of shrinkage of samples. The span was adjusted to 54 mm. Samples that bowed during drying were all placed with the bow facing upwards, as indicated in Figure 53. Details on the bow can be found in appendix H.



Figure 53: Bow facing upwards



Figure 52: Sample dimensions according to ISO 178.2019

From this testing setup, we can derive valuable data. By analysing the stress-strain curves for each sample, we gain insights into the material's mechanical properties under bending. Using the same dataset, we can derive the flexural strength. Flexural strength is the maximum stress a material can withstand before failure occurs. Additionally the flexural modulus can be calculated.

Formula used for calculating;



Where;

F = force [N]

L= support span [mm]

b= width of test beam [mm]

d= thickness of test beam [mm]

D= maximum deflection [mm]

(Callister, 2015)

Thus, the three-point bending tests provide a comprehensive evaluation of the material's behaviour, offering valuable information on strength and brittleness.

Compression

Tension

Figure 54: Behaviour of sample during 3 points bending

2.3 | **Documentation**

Lastly, documentation is a crucial part of this method. Testing was done in a structured system including R-code, V-code, trial code, and date to correlate with lab sessions and mixing protocols. See on the right.

The effectiveness of the documentation method hinges on its comprehensive and searchable nature, addressing the complexity of 3D printing processes. Given the vast array of combinations possible in paste printing, each with its distinct advantages, disadvantages, and varying outcomes in terms of print quality and strength, there is an extensive body of prior research from students at this faculty, graduating students, and researchers worldwide. To avoid redundant work and ensure the repeatability and verifiability of our tests, it's crucial to document the processes meticulously. Searchability is an important aspect here as it fosters collaboration between researchers, allowing access to previously studied combinations with ease.

Key elements of this documentation include detailing the types and quantities of ingredients used, the specific mixing protocols and machinery employed, as well as the printer settings and G-codes utilised during printing. This can be found in Appendix F.

Furthermore, the drying process and environmental conditions under which the object was cured should also be documented, as these both impact the results significantly. This level of detail not only ensures traceability but also facilitates future research and replication of the experiments. This can also be found in Appendix F.

Another pivotal aspect of this documentation method is attributing authorship, allowing for accountability and trustworthiness of the research. Knowing who conducted the tests enables peers to ask questions and assess the reliability of the findings. Moreover, such a systematic documentation approach could



Figure 55: Explanation of documentation

empower even bachelor's students to contribute to the database, provided they adhere to the same rigorous documentation standards. This standard has been developed for this research specifically, when broader implemented adjustments can be made, for example in denotion of dates from dd-mm to yyyy-mm-dd to allow for better structuring. Overall, the method's strength lies in its thoroughness, searchability, and collaborative potential, fostering a culture of transparent and credible research within the academic community.v

While documentation is essential for scientific rigour, it can often be difficult to understand. To improve clarity, the conversion table below is provided to make the results more accessible.

Name in the report	Code used during
	testing
PSF	R1.4
PSF + G	R1.9
PSF + G	R1.10
Carob	R2.0
Р	Vx.x
С	Mould
CD	Climate Chamber
AD	Air dried

Table 8: Conversion from testing coding to names in this report

3. **Results**

3.1 | **Printing**

Prints exhibited consistent line extrusion and good buildability, as shown in the images. The most successful G-codes were V2.6, V2.7, and V3.0, with extrusion pressure ranging from 0.1 to 0.45 MPa, adjusted during printing. The recipes that printed well were:

- PSF
- PSF + G
- Carob





Figure 61: Correct line width, printed with PSF + G

Figure 59: Layers are not wobbly, printed with

PSF + G

Figure 60: Correct layer height, printed with Carob

Figure 62: Good balance between Vrequired and Vout, printed with PSF + G

3.2 | Stress-strain curves

In appendix I stress-strain curves with original coding can be found. Here are stress strain curves of;

- PSF C AD
- PSF C CD
- PSF + G P AD
- PSF + G C AD
- Carob P AD
- Carob P CD

To note that there are no stress strain curves of printed PSF as all samples broke during production.



In Figure 64 it can be seen that samples failed at various stresses and strains, showing large variance. The highest stress being 8 MPa at 0,075 strain.

In Figure 65 it can be seen that samples 2 and 6 can withstand a stress of 3 MPa. The rest of the serie lies closer together but fails at approximately 1 MPa.



Figure 64: Stress strain curve of samples made with pecan shell flour and all binder, casted, climate controlled dried. Samples 1-7 were produced with the same method.



Figure 65: Stress strain curve of samples made with pecan shell flour and all binder, casted, air dried. Samples 1-8 were produced with the same method.

It can be seen that samples 2-7 lie close together. Having a similar slope and failing at approximately a strain of 0.005. Sample 1 failed at a higher strain of 0,0058 and can withstand 7 MPA. Sample 8 can withstand 5 MPa but fails at a strain of 0.003.

In Figure 67 it can be seen that samples 1-4 have similar slope but sample 4 failed at a higher strain of 0.01.



Figure 66: Stress strain curve of recipe made with pecan shell flour, gluten and all binder, printed and air dried. Samples 1-7 were produced with the same method.



Figure 67: Stress strain curve of samples made with pecan shell flour, gluten and all binder, casted, air dried. Samples 1-4 were produced with the same method.

In Figure 68 is a stress strain curve of samples made with Carob and alginate, printed and climate controlled dried. See Chapter 2.1 for details on production procedure. Samples 11 and 12 were deformed during drying. In Figure 69 it can be seen that sample 7, 11 and 12 failed at stresses below 8 MPa but withstood strains twice as high as sample 8 and 9. The other samples show large variance ranging from 11 MPa to 18 MPa.



Figure 68: Sress strain curve of samples made with Carob and alginate, printed and climate controlled dried. See Chapter 2.1 for details on production procedure. Samples 11 and 12 were deformed during drying.



Figure 69: Stress strain curve of samples made with Carob and alginate, printed and air dried. See Chapter 2.1 | for details on production procedure.

4. Discussion

This chapter discusses the mechanical properties of tested recipes, focusing on stress-strain curves, flexural strength, and flexural modulus. The stress-strain curve will be examined to understand material deformation under applied forces, providing insights into ductility and flexural strength. Flexural modulus is analysed to assess the material's ability to resist bending.

Comparisons are made with existing literature and PLA to contextualise the findings. The chapter also addresses the limitations of the research, acknowledging constraints that may have affected results. The chapter concludes with answering the research question and summarising key insights. To denote the following has been used;

- (a) = brittle fracture
- (b) = inconsistencies
- (c) = consistent
- (d) = dutctile fracture
- (e) = similar slope
- (f) = maximum stress
- (g) = large varience

Deserves b					
VID 23-0 VIB 31-0		1 U24 6-11	UZ STAN	U.r.	(ha 41-3
OLO TAIL	V2.1 31-0 910 T2	U24 6-1	UL CON	Uz.q 5-3	UZ 4 7-3 RLS 62
100 23-10	NIO TC	-	212 FILM	U1.4 5-3	07.5 7-3 R15 F1
RIDE I	V21 31-0 210 TV V22 31-6 V22 31-6		UL12 5-3 mesh 012 62 005	Rt4 62	Drus als
VIRTEN	0 T1 210 23	-	U26 8-5	R1.4 E4	026
VI.B.740 RLO	UZZ 5 UZ4 6-11 RLO TX3 RIO 62			oue the	Empty "
026 11-5 RL4 EI RLB U26 11	11 6 21-3 R1100 21-3 R1100 21-3		121	20 20-S	No.lda 15-5
15 10 R143	U 21-3 RIND 62 RI.4	Malde 145	vator 5	R20 EZ	REALD ET
	21.10 C5 2Ly	HUNE HAS	U16 23	USL 2015	NL
	21/7 -3 ULA 24	May 15-5 220 E2	220 24	RED COS	Renzey
4 t2 10-3 t4 15-3		READ ISS	R2.0 5	210 15	CE COM
RLS 65 RLS EIZ		Bese, ta	ALL 156	Exte DI	R3.1 EL

4.1 | Stress Strain curves

Here the stress strain curves presentent in the previous chapter are discussed and annotated. In the stress strain curves, behaviour of different recipes can be observed. For the PSF recipes, we notice a brittle behaviour denoted at point (a). This brittle fracture is evident from the lack of deformation after the maximum stress is reached. Both air dried and climate dried exhibit very low strain resistance, staying below 0,001 mm. Additionally, there is a large inconsistency in the slope of the lines, indicating variations between different samples, denoted at point (b). Variability is expected due to the natural materials and the printing process. The overall strength is low, with air dried reaching only up to 3 MPa, and climate dried just more than 8 MPa at most.



The PSF + G show similar brittle fractures with low strains, see point (a). However, PSF + G C AD is more consistent compared to both PSF. Even though the lines are not at the same level, they have a similar slope, indicating consistency in their behaviour, denoted at point (c). This is not the case for PSF + G P AD as can be seen at point (b).



Figure 73: Stress-strain curve of PSF + G P AD



Figure 74: Stress-strain curve of PSF + C AD

Carob shows significantly higher stresses and strains, with failure strains resembling PLA at 60% infill (see Chapter 1.4). Unlike the PSF and PSF + G, Carob shows greater ductility, as evidenced by the deformation after the maximum stress is reached, point (d) in the graph. The maximum stress of carob samples varies, with the top most sample reaching nearly 18 MPa, see point (f), while the lowest is barely over 10 MPa. Despite variance, the initial slopes of the lines are consistent, denoted at point (e). The bottom three lines in P CD represent samples that have cracked, specifically samples 11 and 12, which condensed, resulting in high strain with minimal stress resistance. Sample 7, being extremely bowed, did not produce a similar stress curve due to likely contact with the machine base instead of the supports. Still in both Carob variants large varience can be seen, denoted at point (g).



0.12

0.16

0.14

Sample 11

0.18

-0.02

0.02

Sample 7

0.04

0.06

Sample 8

0.08

Strain [x]

Sample 9

0.1

Sample 10

Figure 76: Stress-strain curve of Carob P CD

As can be seen in the stress-strain curve below, Carob P AD and Carob P CD reach higher stresses and strains than PSF C AD, PSF C CD, PSF + G P AD and PSF + G C AD.



Figure 77: Combined stress-strain curve with zoomed-in detail

4.2 | Flexural strength

Based on the stress-strain curves the flexural strength of each recipe is determined and summarised in Figure 78. See appendix J for a table of all bar charts.

When looking at the results it is clear that Carob P AD performs best reaching 15,4 MPa on average, being a 200% higher than the strongest PSF + G formulation. Carob P CD is weaker, reaching 11,1 MPa, but it is to be noted that the number of samples is limited (n=3).

The results of PSF C indicate that climate drying (CD) produces prints that are 240% stronger compared to air drying (AD). In contrast when the same comparison is made with Carob P the strength of climate controlled is only 72% of air dried. These results are conflicting and indicate that other factors must influence the difference.

When comparing casted air-dried samples, the strength values of PSF C AD and PSF + G C AD reveal that the addition of gluten enhances the strength by 250%. This enhancement underscores the beneficial impact of gluten incorporation for improving mechanical strength. Interestingly, both PSF + G formulations perform inferior to PSF C CD, showing that even though ingredients are important, so is the drying process.

Furthermore, comparing the casted and printed formulations with gluten shows that the casted version exhibits slightly lower strength, although the difference is not significant.



Figure 78: Flexural strength of tested recipes

4.3 | Flexural modulus

For flexural modulus, the PSF C CD is much stiffer than other formulations. Carob P AD shows a flexural modulus that is at 43% of the PSF +G P AD, indicating greater flexibility. Calculations are done as detailed in Chapter 2.2. Considering the brittleness observed with PSF the flexibility of Carob is development in the desired direction.

Flexural Modulus 4,00 3,00 Flexural Modulus [GPa] 2,00 1,00 0,00 PSF C CD PSF + G PAD PSF + G C AD PSECAD Carob PAD Carob P CD n=8n=8n=3n=6n=7n=3Figure 79: Flexural modulus of tested recipes





rigure so: Plexulai strength of lecipes compared to interature

When comparing flexural strength to literature values, the Carob P AD recipe shows comparable strength to the findings of Sanadiya et al. (2018), with only a 2.5% difference. The carob recipe is 57% higher than the findings of Sauerwein et al. (2020), which is promising. However, both pecan shell flour recipes (PSF C CD and PSF + G P AD) are only 52% and 44% respectively of the strength of Sauerwein et al., making them less impressive. Other research did not report flexural strength, making the comparison unequal.

4.5 | Comparison to PLA



Figure 81: Flexural strength of recipes compared to PLA

When compared to PLA, the strength of Carob P AD is much lower, reaching only 13%. Despite discrepancies between literature PLA and the PLA benchmark (Chapter 1.4 |), the overall strength is low.

The PSF C CD reached 40% of the flexural modulus of PLA, while PSF + G P ADachieved 19% of PLA, and carob only reached 8%.

In summary, Carob P AD shows higher flexural strength and lower modulus compared to PSF C CD, aligning well with certain plastics, making it potentially suitable for specific prototyping applications.



Figure 82: Flexural modulus of recipes compared to PLA

The carob recipe's flexural strength is comparable to PVC and PE, while both PSF C CD and PSF + G P AD align more closely with wood. With the PSF + G P AD being similar to maple, andPSF C CD resembling oak. Carob P AD is on the lower end for the flexural strength of plastics but aligns well with natural materials. This suggests that Carob P AD might be adequate for prototyping depending on the load case. Carob P AD demonstrates greater flexibility, comparable to elastomers (ANSYS, 2022). PSF C CD falls within the range of unfilled plastics, while PSF + G P AD is situated between elastomers and unfilled plastics. See Figure 83 for flexural strength and flexural modulus compared to materials families.



Figure 83: Flexural strength plotted against flexural modulus for material families, adapted from ANSYS (2022)

Flexural modulus [GPa]

4.6 | Limitations

First and foremost, the values for flexural strength and modulus presented in the research are unreliable. The primary reason is that the benchmark performed with PLA, as described in Chapter 1.4, does not align with the values found in the literature. This discrepancy, discussed with the supervisory team, is 28% (31 MPa). Consequently, the percentages for the developed recipes could be off by 28%, though the exact deviation is unclear and could be less or more.

Secondly, the recipe tests were conducted with a very limited number of samples, resulting in high variance. This necessitates further testing of similar samples to increase reliability. Some samples were not quantified due to extreme brittleness and consequent breaking during handling, and thus were excluded from the analysis. Additionally, the printing process was inconsistent, leading to visible holes between print lines and cracks on the print bed surfaces, which could indicate the presence of microcracks not visible to the naked eye. Both affect strength measurements and have to be addressed for that reason.

Moreover, some samples showed outliers despite being produced in the same manner, a potential explanation would be a different rate of drying due to drying conditions or inconsistencies in mix preparation. These inconsistencies have contributed to unreliable data. Most samples had to be adjusted from the ISO 178.2019 standard dimensions. Rough surfaces caused measurement to vary greatly and quite a few samples were bowed requiring adjustment to the test set-up. As a result, these adjustments mean the results are not fully validated. All samples were approximations of ISO 178.2019 but did not match perfectly due to sample shrinkage. This research focused solely on three point bending, which combines tension and compression. Thus, it did not account for pure tensile or pure compression tests, making comparison with literature less adequate. Analysing these tests could provide valuable insights for further research and development.

Not all recipe alternatives have been thoroughly investigated. While the research has focused on a specific approach, it did not capture the entire scope of possibilities. Additionally, although the g-code developed permits successful printing, no strength evaluations have been conducted on different g-codes. This means that while the prints may be produced, no optimasation step has been made is this regard.

Lastly, during this research only rectangular bars were printed and for that shape printability was optimised. However, recipes were not evaluated for printability of more complex shapes, such as higher models or thin walled objects.



Figure 84: Bowed sample in 3 point bending test

4.7 | **Conclusion**

4.7.1 Which ingredient (s), or combinations of ingredients can provide more strength?

Firstly, recipes based on carob and alginate binders are significantly less brittle than those based on pecan shell flour and all binder, failing a a simliar strain as PLA. Compared to PLA Carob P AD is 13% of the flexural strength. Second, the ingredient that improves flexural strength is adding a small percentage of gluten to a recipe based on pecan shell flour and all binder, by 250%. Results on the effect on climate controlled are conflicting, see Chapter 4.2 |.

There are a few things that have proven ineffective for improving strength. Arrowroot, oyster shell and olive pit powder do not yield valuable properties, demonstrating slight shear thickening. For example, samples made with oyster shells and olive pit power were cast due to their thickening behaviour but exhibited very brittle behaviour and were not pursued further for that reason.

4.7.2 How can the new ingredients be printed?

To assess the suitability of the new ingredients for printing, it is crucial to reference Chapter 1.5 |, where the relationship between air pressure, nozzle diameter, mixed viscosity, and printing speed is explained. This relationship, the balance between Vrequired and Voutput is also illustrated in Figure 85. Additionally, it is essential to ensure that the mixture exhibits the desired properties as described in the methodology, Chapter 2.1 |. Specifically shear thinning, allowing easy extrusion through the nozzle. Adhering to these parameters, iterations to achieve a printable mixture can be conducted. However, it is important to note that adherence to these guidelines does not guarantee successful objects. When direct ink writing with bio-based materials, it is important to consider the drying process. Results for climate-controlled drying are conflicting, but issues such as warping and cracking has been common for most prints. Additionally, scaling, as described in Chapter 2.1 |, has been crucial to ensure the correct dimensions. This highlights that printability is not the only factor to consider. Proper drying techniques and adjustments for scaling are also essential to achieve the desired results.

Iterations on the G-codes impact printability and sample production quality which can impact measured strength.

4.7.3 How can the mechanical strength of a printed object using bio-based direct ink writing be enhanced?

Enhancing the mechanical strength of bio-based direct ink writing can be achieved by using Carob, alginate and H2O employing a G-code with a 0.7 mm layer height, a speed of 6 mm/s, 140% flow rate, and allowing the ink to air dry. This approach yielded the strongest outcomes in this research of 15,4 MPa.



5. **Recommendations**

Recommendations are split between material improvements and manufacturing. However, there is some overlap between the two as improvements in manufacturing can aid the material development and vice versa. The chapter starts with three recommendations that have the most priority for improving the mothod towards printing for prototyping.

5.1 | **Focus**

To improve the process in the future, there are three key areas to focus on. First, developing controlled printing and drying techniques is essential to prevent warping, control variables, and potentially improve mechanical strength. This is crucial because significant time is lost due to print failures, and reducing the failure rate is necessary for successful application in the prototyping industry. Additionally tests have shown controlled drying could improve the strength thus further testing in this direction is advisable.

Second, integrating gluten into the carob recipe holds promise, as gluten has shown beneficial effects when combined with pecan shell flour. Observing similar improvements with carob, which already demonstrates potential, could lead to enhanced material properties. Third, it is important to make more samples for testing tension and compression and to increase the number of samples overall. Fatique, cyclic behaviour and impact tests could also be included. Additionally, having a higher number of samples is necessary for proper quantification, requiring the success of the first recommendation.



Figure 86: Samples of Carob drying in the climate chamber



Figure 87: Sample of musselshell and olive pit powder (not used)

5.2 | Material

Currently, the origen of the strength measured remains unkown, and understanding which aspects need improvement can guide future developments.

Testing different suppliers of ingredients to assess their impact and potential benefits can provide insight into their effect and potentially open new routes for material improvement. Experimenting with combinations of binders can help leverage the advantages of each type, leading to improved material properties. Additionally, generalising the recipe to enable the printing of a diverse range of objects, including taller models and thinwalled structures, will increase versatility. Lastly, ensuring proper compostability according to the latest guidelines has to be tested, although that is only relevant once sufficient progress has been made on mechanical strength.

5.3 | Manufacturing

To advance the process, several key improvements are recommended. Developing a guide to reduce the learning curve for working with direct ink writing is essential, as this technology is not comparable to regular 3D printing and remains an evolving field. Where conventional printing has sources such as YouTube and blogs full of community generated advice, direct ink writing is still being developed and as such knowledge about the process is hard to come by. Additionally, enhancing the printer by incorporating automated bed leveling, simplifying print removal, and enabling real-time control will significantly improve reliability. Quantifying shrinkage and making necessary adjustments in printing code or CAD design will ensure dimensional accuracy in the printed objects. Investigating the environmental impact of high-temperature drying to assess the potential and sustainability, could show promise, similar to firing clay objects in a kiln. Conducting heat treatment studies on gluten and analysing the mixing effect will help optimise material properties. Furthermore, developing a G-code capable of printing the highest possible concentration of gluten will enhance print quality and performance, contributing to overall process improvement.



6. **References**

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7. Appendix

A - Search operator scopus

Material strength				
Sturdiness				
Robustness				
Tensile strength				
Stretch resistance				
Pulling strength				
Force				
Flexural strength				
3 points bending				
Bend resistance				
Load bearing capacity				
Compressive properties				
Compression testing				
Print quality?				
"Material? strength" OR "Tensile strength" C Studiness OR "Flexural strength" OR "Ben resistance"				
				Paste printed parts
Liquid deposition modelling				
Paste extrusion				
Eazoa				
3D printing with pastes				
Viscous material printing				
Paste-based fabrication				

Additve manufacturing with pastes

"Liquid deposition modelling" OR "Paste? extrusion" OR "additive manufacturing with paste?"

Binders

Honig alles binder

Fillers?

Glue

Adhesives

Bonding agents

Binder? OR adhesive? OR "bonding agent?"

Rounds of search query

Round 1

"material? strength" OR "tensile strength" OR sturdiness OR "flexural strength" OR "bend" resistance"

AND "liquid deposition modelling" OR "paste? extrusion" OR "additive manufacturing with paste?"

AND binder? OR adhesive? OR "bonding agent?"

Result is only one paper, Rosenthal 2022 Objective: about 80% has to be relevant (not 100%!)

Round 2

"material? strength" OR "tensile strength" OR sturdiness OR "flexural strength" OR "bend" resistance"

AND "liquid deposition modelling" OR "paste? extrusion" OR "additive manufacturing with paste?" OR "3D printing" OR "viscous material printing"

AND binder? OR adhesive? OR "bonding agent?" OR filler?

219 documents found

This looks pretty good, some papers are not interesting but most are. Too many about fossil based wood filled so I'm including AND NOTs

Round 3

"material? strength" OR "tensile strength" OR sturdiness OR "flexural strength" OR "bend" resistance"

AND "liquid deposition modelling" OR "paste? extrusion" OR "additive manufacturing with paste?" OR "3D printing" OR "viscous material printing"

AND binder? OR adhesive? OR "bonding agent?" OR filler?

AND NOT pla

AND NOT abs

AND NOT peek

AND NOT "polylactic acid"

AND NOT petg

145 documents

The AND NOTs might exclude comparing papers so this isn't a good idea

Round 4

"material? strength" OR "tensile strength" OR sturdiness OR "flexural strength" OR "bend" resistance"

AND "liquid deposition modelling" OR "paste? extrusion" OR "additive manufacturing with paste?" OR "3D printing" OR "viscous material printing"

AND binder?

AND NOT concrete

26 results, interesting papers saved in search query 1 (search saved & alert set)

B - Web of sciences

Search terms Sustainable 3d printing Paste printing Biodegradable paste printing Additive manufacturing wood flours Additive manufacturing new materials Additive manufacturing Additive manufacturing net zero Result: list of 8 papers @ scopus (3)



C - Stress-strain curve of PLA



D - Strength analysis of literature

Paper	Recipe	Tensile [MPa]	Compressive [MPa]	Flexural [MPa]	E-Modulus [MPa]	Young's modulus [MPa]
0.4 Sanandiya	1.	11,31	15,31	15,03		244,1
2.11 Bakker	Musselshell	-	-	9,8	2100	
4.8 Scaffaro	FDM_MB/ OFI-B	18			128	
4.8 Scaffaro	FDM_MB/ NPK-b	20			120	
0.2 Rosenthal 2022	Gy6	-	14	8,13		
0.5 Final	MCC	-				6587
0.5 Final	Sawdust C200 4	0,00565				4050
0.5 Final	Walnut	0,00175				3123
2.1 Rech	XG 1.8wt% 0.1M	2,4				175
4.8 Scaffaro	FDM_MB/ OFI-A/ NPK-A	17			168	
4.8 Scaffaro	FDM_MB/ OFI-B/ NPK-B	15			162	
4.8 Scaffaro	FDM_MB/ NPK-A	19			116	
4.8 Scaffaro	FDM_MB/ OFI-A	17			113	
4.8 Scaffaro	FDM_MB	20			89	
0.1 Faludi	Rice & IPA	-	10,0	-		
4.6 Nida Moses	3D (1:1)			10,74		
0.2 Rosenthal 2022	Gy1	-	3,17	8,13		
0.7 Cellulose	MCC+WG	13,39	38,5			
0.7 Cellulose	UF+WG	13,3	19,72			
2.1 Rech	XG 1.8wt% 0.2M	2,3				140

0.3 Kam et al	75	3,4	-		44
0.3 Kam et al	0:1	-	1		18
0.1 Faludi	Pecan + Oak	4,7	7,9	-	
0.1 Faludi	Pecan	9,0	5,8	-	
0.2 Rosenthal 2022	SO	-	5,19	7,94	
0.2 Rosenthal 2022	Sn1	-	5,5	7,48	
4.6 Nida Moses	3D (9:1)			6,98	
1.12 Vaezi	1.	3,562		6,65	

E - Hand tests

Test	Date	Description	Observations	Insights
Gluten 1	8-3-2024	Combined 8,37 grams of leftover R1.4 with 0,9 grams gluten and 0,9 grams H2O. Gluten is roughly 10% of the mixture of R1.4. Handmixed till combined	- with 18gauge needle mix is hard to extrude - no nozzle extrudes fine - mix show good buildability	Gluten don't show any funky reaction with R1.4. Flow needs to be investigated. Question that remains is water absorption by the gluten.
Gluten & AB t1	8-3-2024	Combined 2,57 grams of AllBinder (leftovers of the package) with 0,64 grams of gluten (which is 25%) and 1,3 grams of H2O (same ratio Ab:H2O as in R1.4)	Formed a cube and let it dry. Once it dried (12- 3) it is hard. Does not form bubles and it smells like bread.	No specific interaction between gluten and AllBinder.

Gluten and H2O t2	8-3-2024	Combined 2 grams of gluten with varying amounts of water (H2O:Gluten) (t2.1 - t2.5) to test absoption. H2O was at room temperature	With a little stirring all mixtures formed a ball. Tried stretching the material of t2.1 by hand for a few minutes and strechyness was reduced and the colour became	Water content between t2.1 and t2.2 seems best. It leaves the least extra. Focus on kneading as little as possible to prevent reduction in stretchyness.
			the weekend the sample started to mold. t2.2-t2.5 are fine.	
		t2.1 1:1 2 grams H2O	Quite firm and springy - After 1 hour of resting mixture can form a ball and shows discolourations.	
		t2.2 2:1 4,3 grams H2O (measuring error)	After an hour of drying has excess water that sticks to my fingers, you can pull it apart	
		t2.3 3:1 6 grams H2O	Bubbles start to form, mixture is not homogenous - After 1 hour of drying has excess water in the glass. similar strecht and springyness as t2.2	
		t2.4 5:1 10 grams H2O	Mixture is liquid - after an hour of resting has more excess water that t2.3 and feels less trechy than t2.1	
		t2.5 6:1 12 grams H2O	Mixture is seperating - After one hour of resting still has water laying on top	
Gluten t3	8-3-2024	Combined t2.2. t2.3, t2.4 and t2.5 (21,93 grams) with 2 grams of ethanol (10% of total) Left the excess water out	Ethanol seems to coat the material. Springyness reduces. The mixture feels like 2.1	Ethanol does not majorly affect the mixture.
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Blob R1.9	14-3-2024	excess midxture handfromed into a "cube"	Is not dry on 15-3	Drying time is more than a week
Hand Drawn R1.9 t1	14-3-2024	Excess extruded on 0,4 MPa, attempted to make a thin walled cube	There was very little left in the syringe	-
Mould 3 R1.9 t1	14-3-2024	Excess material after hand drawen into a mould	not dry on 15-3	No new
Mould 3 R1.9 t2	15-3-2024	Excess mixture from production in a mould	not dry on 15-3	No new
Removing prints	21-3	removing V2.6 R1.9 t9 using water	Works but affects the bottom layer	Putty knife works best for removing stuck prints
		removing V2.6 R1.9 t2 using a putty knife	with patience works good	
R1.11 Hand drawn	25-3	Material extruded on 0.4 MPa to test flow	Flows awfully, very inconsistent. Likely due to non homogenous material	R1.11 does not work for printing with this machine and syringe diameter
Alginate t1	14-5	"Ratio 1:8 A:H2O 3 [g] Alginate 24 [g] H20 Alginate to H2O, strirred rigourously"	Formed stiff peaks after 10 minutes.	More water is a more liquid gel.

Alginate t2	14-5	"Ratio 1:10 A:H2O 3 [g] Alginate 30 [g] H20 Alginate to H2O, strirred rigourously"	Has a little excess water after 10 minutes, looks promising depending of filler H2O absorption. After an hour firm	
		0	alike t1. After 2 hours almost as thick as t1.	
Alginate t3	14-5	"Ratio 1:12 A:H2O 3 [g] Alginate 36 [g] H2O Alginate to H2O, strirred rigourously"	Is a properly a liquid after 10 minutes, looks promising depending on filler H2O absorption. Afer an hour less liquid than before. After 2 hours has absorpt excess water and is becoming thicker	
Carob t1	14-5	"Ratio 1:2.3 C:H2O Based on R2.0 3 [g] Carob 6.9 [g] H20 H2O to Carob, strirred rigourously"	Thicker than t2 & t3. After an hour thickend into a paste Smells strongly of dark chocolate and has a similar colour, the powder sticks a little to itself	
Carob t2	14-5	"Ratio 1:2.9 C:H2O Based on R2.0 3 [g] Carob 8.7 [g] H20 H2O to Carob, strirred rigourously"	Consistency of water. Smells strongly of dark chocolate and has a similar colour, the powder sticks a little to itself Does not change over time	

			Ï	
Carob t3	14-5	"Ratio 1:3.5 C:H2O Based on R2.0 3 [g] Carob 11.5 [g] H2O H2O to Carob, strirred rigourously"	Consistency of water, after an hour slight separation of Carob and H2O. Smells strongly of dark chocolate and has a similar colour, the powder sticks a little to itself. Does not change over time	
OP t1	14-5	"Olive Pit Powder (OP) 3 [g] Olive pit powder 1.29 [g] H2O (Ratio 1:0.43) + 0.5 [g] H2O (Ratio 1:0.6)"	1st Ratio had far too little water, it was the consistency of wet sand. 2nd ratio formed a ball like apple pie dough. No changes over time.	
OP t2	14-5	"Olive Pit Powder (OP) 3 [g] Olive pit powder 2.4 [g] H2O (Ratio 1:0.81) "	Properly forms a dough like paste. Afer 2 hours is granier.	
OP t3	14-5	"Olive Pit Powder (OP) 3 [g] Olive pit powder 3.0[g] H2O (Ratio 1:1) "	Fully liquid, no specific shear thinning behaviour. Potentially shear thickening but not very obivious. After 30 minutes thicker mixture, more like a paste. Seems to shear thicken alike arrowroot. After 2 hours seems to shear thicken and is still paste like.	

OS t1	14-5	"Oyster Shell Powder (OS) 3 [g] Oyster shell powder 1.29 [g] H2O (Ratio 1:0.43) H2O to OS "	Similar to OP t3, feels like cement. after 1 hours show shear thickening, forms a ball	
OS t2	14-5	"Oyster Shell Powder (OS) 3 [g] Oyster shell powder 1.8 [g] H2O (Ratio 1:0.0.6) H2O to OS "	Liquid a little bit like paste. Thickening after 20 minutes. After 1 hour is still liquid and shows to shear thinning or thickening	
OS t3	14-5	"Oyster Shell Powder (OS) 3 [g] Oyster shell powder 2.4 [g] H2O (Ratio 1:0.81) H2O to OS "	Liquid. After 1 hour has water on top and is fully liquid	
Coffee t3	14-5	"10.557 [g] Coffee t2 from 8-4 (siffed the clumps out) 34.275 [g] Alginate t3 Combined by stirring"		

F - Testing documentation

Recipe	Filler	Quantity [g]	Binder	Quantity [g]	Solvent #1	Quantity [g]	Solvent #2	Quantity [g]	Additve #1	Quantity [g]
1.0	Pecan shell flour	11	All binder	27	H2O	11	Ethanol	7	-	-
1.1	Pecan shell flour	11	All binder	27	H2O	22	Ethanol	7	-	-
1.2	Pecan shell flour	17,46	All binder	6,04	H2O	4,92	Ethanol	1,57	-	-
1.3	Pecan shell flour	11	All binder	27	H2O	16,5	Ethanol	7	-	-
1.4	Pecan shell flour	11	All binder	27	H2O	13,75	Ethanol	8,75	-	-
1.5	Pecan shell flour	11	All binder	27	H2O	13,75	Ethanol	8,75	-	-
1.6	Pecan shell flour	2,79	All binder	6,85	H2O	7,61	Ethanol	3	Gluten	2,75
1.7	Pecan shell flour	2,71	All binder	6,66	H2O	11,64	Ethanol	3	Gluten	5,5
1.8	Pecan shell flour	3,36	All binder	8,25	H2O	21,70	Ethanol	3	Gluten	11
1.9	Pecan shell flour	6,16	All binder	27	H2O	13,75	Ethanol	8,75	Gluten	4,62
1.10	Pecan shell flour	6,16	All binder	27	H2O	13,75	Ethanol	8,75	Gluten	4,62
1.11	Pecan shell flour	3,7	All binder	9	H2O	3,7	Ethanol	3	Gluten	11
1.12	Pecan shell flour	11	All binder	27	H2O	13,75	Ethanol	8,75	-	-
2.0	Carob	12	Alginate	3	H2O	35	-	-	-	-
3.0	Oyster Shell	43,5	Alginate	2	H2O	41,5	-	-	Olive pit	3
3.1	Oyster Shell	33,03	Alginate	1,52	H2O	42,36	-	-	Olive pit	2,28

| 77

M0	M1	M2	M3	M4	M5	M6	M7	M8
Measure all ingredients seperately	Measure all ingredients seperately	Measure all ingredients seperately (except ethanol)	Measure all ingredients seperately	Measure all ingredients seperately (except ethanol)	Measure all ingredients seperately (except ethanol)	to be determined	Measure all ingredients seperately (except ethanol)	Measure ingredie seperate
Mix solvents for 20 seconds on speed 1	Mix solvents (H2O and ethanol) for 20 seconds on speed 1	Start with H2O	Take basemix (all binder, pecan shell flour and ethanol) from M2	Combine pecan shell flour and gluten and stir with a spoon until throughly mixed	Combine pecan shell flour and gluten and strir with a spoon until throughly mixed		Add all H2O to all binder and mix on speed 2 till combined	Add Alg to H2O, by scoop in betwe
Add allbBinder and mix on speed 1	Add all binder spoon by spoon through a siff and mix on speed 2 unitl combined between each scoop	Add all binder spoon by spoon through a siff and mix on speed 2 unitl combined between each scoop. Twirl the cup when needed to get all material.	Add H2O all at once	Start with H2O	Start with H2O (part 1)		Add half of pecan shell flour using a siff.	Stir with spoon ti combine (3-5 rou enough)
Pecan shell flour is sieved in and mixed on speed 1	Add pecan shell flour spoon by spoon through a siff and mix on speed 2 unitl combined between each scoop	Add pecan shell flour spoon by spoon through a siff and mix on speed 2 unitl combined between each scoop. Twirl the cup when needed to get all material.	Combine by hand	Add all binder spoon by spoon through a siff and mix on speed 2 unitl combined between each scoop. Twirl the cup when needed to get all material.	Add all binder spoon by spoon through a siff and mix on speed 2 unitl combined between each scoop. Twirl the cup when needed to get all material.		Mix on speed 2 till combined, twirling the cup	Let sit a gel up fo minutes after 10

Avlix the Avlix the Avlix the Avliant till a Whomogenous paste is <u>created</u> nate scoop . Stir en a 1 d nds is	Mix the material till a homogenous paste is created	Measure out ethanol	Add gluten	Gradually add pecan shell flour and gluten mixture mixture (1 st half with a siff, 2nd half without). Mix on speed 2 in between scoops, mix until combined.	Gradually add pecan shell flour and gluten mixture mixture. Add H2O (from part 2) when needed. Mix on speed 2 in between scoops, mix until combined and right consistency is reached.	Add other half of pecan shell flour using siff	Add filler scoop by scoop, stirring in between
		Add all ethanol at once	Combine by hand	Measure ethanol	Measure ethanol	Mix on speed2 till combined, twirling the cup	Stir with a spoon till combined (3-5 rounds is enough)
nd r 30 stir		Mix the material on speed2 till a homogenous paste is created	Add additional ethanol	Add all ethanol at once	Add all ethanol at once	Measure ethanol	
			Combine by hand	Mix the material on speed 2 till a homogenous paste is created	Mix the material on speed 2 till a homogenous paste is created	Add all ethanol	
						Mix on speed 2 till most ethanol is combined	
			Based on assumption that gluten needs to mixed as little as possible and ethanol evaporates fast				

Date	Time	Temperature [Celcius]	Humidity [%]
5-3	9:05	20,3	38
5-3	12:40	21,1	38
7-3	9:37	20	36
8-3	10:16	20,3	28
8-3	12:29	20,8	28
11-3	9:24	20,3	39
12-3	15:48	21,1	42
14-3	9:05	20,7	47
14-3	9:31	20,7	47
14-3	11:46	21,1	53
14-3	11:58	21,5	49
14-3	12:38	21,8	48
14-3	14:03	21,4	47
14-3	14:53	21,8	46
14-3	15:30	21,9	45
14-3	16:13	22	45
15-3	9:31	21,3	49
18-3	11:56	21,1	42
21-3	9:26	21,1	41
25-3	9:56	20,1	37
28-3	10:50	21,4	37
14-5	9:54	22,2	65
15-5	11:16	22,7	63
15-5	12:36	23,1	62
15-5	15:50	23,3	61
15-5	17:30	23,4	62
16-5	9:45	22	66
21-5	9:23	21,8	63
22-5	9:50	21,6	63
6-6	10:50	20,7	47
10-6	17:08	20,2	53
10-6	20:12	20,7	48
11-6	15:15	20,3	45
12-6	9:46	19,6	46
17-6	13:02	20,8	56
Max		23,4	66
Min		19,6	28

Product	Supplier	Date purchased	link	Price (Euro)
Alginate	Junai	6-5-2024	https://www. junai.earth/shop	100,00/kg
All binder	Albert Heijn - Koopmans	15-9-2023	n.a.	7,75/kg
Arrowroot	Albert Heijn - Smaakt	10-3-2023	n.a.	19,45/kg
Carob	Pit & Pit	5-4-2024	https://nl.pit-pit. com/products/ carobe-poeder-bio	12,50 / kg
Coffee	Caffeinc	n.a.	n.a.	n.a.
Ethanol	Cee-Bee Cleaning	1-7-2022	https://www.bol. com/nl/nl/p/cee- bee	12,95/L
Gluten	The Notenshop	n.a.	https://www. denotenshop.nl/ tarwegluten.html	6,95/kg
Olive pit powder	Junai	6-5-2024	https://www. junai.earth/shop	40,00/kg
Oyster shell powder	n.a.	n.a.	n.a.	n.a.
Pecan shell flour	South eastern reduction company	n.a	n.a.	n.a.

File used	V2		V2	V3	V3	V3	V3	V3	V3		V4														
Minimum wall line width [mm]	1,275	1,275	1,275	1,275	1,275	1,275	1,275	1,275	1,275	1,275	1,275	1,275	1,275	1,275	1,275		1,275	1,275	1,275	1,275	1,275	1,275	0,8		0,8
Skirt height [x]	n.a.		1	1	1	1	1	1	1		1														
Skirt	no		yes	yes	yes	yes	yes	yes	yes		yes														
Retraction	no		no	no	no	no	no	no	no		no														
Flow [%]	100	100	100	100	100	100	100	100	130	130	130	130	130	130	130		130	130	130	130	140	140	140		140
Travel speed [mm/s]	20	20	20	20	20	20	20	20	20	20	20	20	20	20	20		20	20	20	20	20	10	10		10
Inner wall speed [mm/s]	15	5	5	5	5	15	10	15	15	15	15	15	15	15	15		15	15	15	15	15	6	6		6
Outer wall speed [mm/s]	15	5	5	5	5	15	10	15	15	15	15	15	15	15	15		15	15	15	15	15	9	9		6
Print speed [mm/s]	15	5	5	5	5	15	10	15	15	15	15	15	15	15	15		15	15	15	15	12	6	6		6
Infill pattern	n.a.		Lines	Lines	n.a.	n.a.	n.a.	n.a.	n.a.		n.a.														
Infill density [%]	n.a.		100	100	100	100	100	100	100		100														
Wall line count [x]	9	6	6	6	6	9	9	6	6	9	9	9	6	6	6		2	2	7	6	6	9	7		7
Line width	0,84	0,84	0,84	0,84	0,84	0,84	0,84	0,84	0,84	0,84	0,84	0,84	0,84	0,84	0,84		0,84	0,84	0,84	0,84	0,84	0,84	0,84		0,84
Initial layer height	0,42	0,42	0,42	0,42	0,42	0,42	0,42	0,42	0,42	0,42	1,00	1,00	1,00	1,00	1,00		1,00	1,00	1,00	0,70	0,70	0,70	0,70		0,70
Layer height	0,42	0,42	0,42	0,42	0,36	0,36	0,42	0,42	0,42	1,00	1,00	0,80	0,70	0,65	0,60		0,70	0,70	0,70	0,70	0,70	0,70	0,70		0,70
EZ V1.x	0	2	3	4	5	6	7	8	6	10	11	12	13	14	15	EZ_ V2.x	1	2	3	4	5	9	7	EZ_ V3.x	0

G - Printing observations











Name R2.0 R2.0 R2.0 R2.0 V3.0 V3.0 V3.0 V3.0 10-6 t4 10-6 t3 10-6)	>		0	٦	T0	TT	12	L3	14
V3.0 V3.0 V3.0 V3.0 10-6 t4 10-6 t3 10-6	R2.0	R2.0	R2.0 V3.0	R2.0	R2.0	R2.0	R2.0	R2.0	R2.0	R2.0	R2.0
10-6 t4 10-6 t3 10-6 ¹	V3.0	V3.0	6-6 t1	V2.7	V2.7	V2.7	V2.7	V2.7	V2.7	Mould	Mould
	t1 20-5 t2	20-5 t4		15-5	15-5	15-5	15-5	15-5	15-5 t6	11-6 t1	11-6 t4
				t1	t2	t3	t4	t5			
WIQTN איזמדה אישט איז אישנע איז אישנע איז אישנע איז אישנע איז	5 10,06	9,41	9,62	7,61	6,87	6,53	7,24	8,11	6,65	10,03	9,22
Length 82,52 82,53 83,86	5 81,13	78,31	80,11	57,93	56,19	56,83	57,41	56,95	58,92	59,03	66,58
Thickness 5,03 5,66 4,67	3,66	4,31	5,42	5,28	4,45	4,4	4,3	5,13	3,45	4,41	3,92
Bow 14,03 11,27 14,33	3 6,8	6	13,91	10,81	9,77	9,61	11,17	10,41	10,26	22,64	23,1
Notes touching the base			touching base	Rough top texture				Rough top texture	thinner lines on top		
Too Too Too cracked cracked crack	ed cupped	Didn't set aprroach zero		Spanto 50mm	Spanto 50mm	Spanto 50mm	Spanto 50mm	Spanto 50mm	Spanto 50mm	Extreme bow	Extreme bow
Span: 64mm	Entered with wrong									Spanto 50mm	Spanto 50mm
	video									Put 'on pressure	Put 'on pressure
										VERY inaccurate	VERY inaccurate
-											

H - Dimensions of samples (mechanical)

	Mould Test 1	Mesh Test Up 4	Mesh Test Up 2	Mesh Test Down 3	Mesh Test Down 1	DryTest 1 Chamber 5	Dry Test 1 Chamber 2	DryTest 1 Mould Time 6	DryTest MouldTime 4	DryTest 1 Control 1
Breedte	9,95	9,53	10,76	9,97	10,03	10	9.58	X	9,83	9,55
Lengte	79,79	75,43	75,08	76,41	76,23	76,61	77	X	77,59	76,53
Dikte	3	3,72	4,375	3,65	3,57	3,92	3,11	X	3,92	4,04
Bow		6,1	6,485							

	Climate	Climate	Climate	Climate	Climate	Climate	Climate	Climate	Climate	Climate
	Lid	Cast 1	Cast 2	Cast	Cast	Syringe 1	Syringe 2	Syringe	Syringe	Syringe
				Mould 1	Mould 2			Mould 1	Mould 2	Mould 3
Breedte	9,79	9,74	9,65	10,52	X	9,57	9,75	X	9,68	9,67
Lengte	77,21	77,28	75,85	77,59	Х	76,13	76,73	X	77,15	77,32
Dikte	3,59	3,94	3,25	3,57	Χ	3,9	3,38	X	3,69	3,49
Bow										

I

Number	1	2	3	4	5	6	7	8	6	10	11	12	13
Name	t1 V2.6	t2 V2.6	t7 V2.6	t8 V2.6	t9 V2.6	t10	t12	t1 V2.6	t2 V2.6	t1	t2	t3	t1
	R1.9	R1.9	R1.9	R1.9	R1.9	V2.6	V2.6	R1.10	R1.10	Mould3	Mould3	Mould3	Mould4
	14-3	14-3	14-3	14-3	15-3	R1.9	R1.9	21-3	21-3	R1.9	R1.9	R1.9	R1.10
						15-3	15-3			14-3	15-3	15-3	21-3
Breedte Bottom	9,87	9,41	10,66	11,59	9,25	9,5	10,08	60'6	11,29	10,15	10,13	10,01	9,57
Breedte	7,81	8,07	8,34	8,94	8,29	7,95	8,28	8,19	11,3	X	X		
TUP													
Zwick	8,84	8,74	9,5	10,265	8,77	8,725	9,18	8,64	11,295	Х	Х	Х	Х
value													
(average)													
Length	78,28	77,71	78,79	78,76	77,89	77,31	77,69	77,18	79,93	78,42	78,38	78,26	76,33
Bottom													
Length	76,86	76,12	76,52	76,4	77,26	76,29	76,67	75,65	79,92	X	X		
Top													
Thickness	5,4	5,565	6,165	5,87	6,22	6,04	6,665	5,855	5,77	2,85	2,72	2,92	2,5
Bow	Х	7,82	Х	X	Х	x	X	X	Х	X	5,42	6,36	
Notes	Moved	Has	Has	Has	Has a			Broke	Rough				reverse
	to to	gaps	"dipped"	"dipped"	different			stringy,	top				bow
	just not		top	top	colur			not along	texture				
	touching							one					
								plane but					
								rather per					
								individual					
								line					



I - Coded stress-strain curves









J - Tables of bar charts

Recipe	Flexural strength [MPa]	n	sd
"PSF C AD "	1,5	8	0,9242
PSF C CD	5,1	7	1,7729
PSF+G P AD	4,3	7	1,2887
PSF+G C AD	5,2	4	0,5280
Carob P AD	15,4	6	1,9709
Carob P CD	11,1	3	2,8105

Recipe	Flexural Modulus [GPa]	SD	n
PSF C AD	1,07	802	8
PSF C CD	2,77	598	7
PSF + G P AD	1,29	390	8
PSF + G C AD	1,58	149	3
Carob P AD	0,55	144	6
Carob P CD	0,56	313	3

K - Project brief



IDE TU Delft - E&SA Department /// Graduation project brief & study overview /// 2018-01 v30

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Procedural Checks - IDE Master Graduation

chair <u>Jeleny Faludi</u> date		signature	
CHECK STUDY PROGRESS To be filled in by the SSC E&SA (Shared Service Center, Edu The study progress will be checked for a 2nd time just befo	ication & Student re the green light	Affairs), after approval of the p meeting.	roject brief by the Chair.
Master electives no. of EC accumulated in total: Of which, taking the conditional requirements into account, can be part of the exam programme List of electives obtained before the third semester without approval of the BoE	- EC - EC	YES all 1st year	r master courses passed year master courses are:
name date FORMAL APPROVAL GRADUATION PROJECT To be filled in by the Board of Examiners of IDE TU Delft. Pli Determined by the Delft. Pli Determined by the Delft. Pli Del		signature pervisory team and study the pr	arts of the brief marked **
 Next, please assess, (dis)approve and sign this Project Brie Does the project fit within the (MSc)-programme of the student (taking into account, if described, the activities done next to the obligatory MSc specific courses)? Is the level of the project challenging enough for a MSc IDE graduating student? Is the project expected to be doable within 100 working days/20 weeks ? Does the composition of the supervisory team comply with the regulations and fit the assignment ? 	r, by using the crit	APPROVED	NOT APPROVED

Personal Project Brief - IDE Master Graduation

Improving the mechanical strength of bio material paste printing

Please state the title of your graduation project (above) and the start date and end date (below). Keep the title compact and simple. Do not use abbreviations. The remainder of this document allows you to define and clarify your graduation project.

start date 04 - 09 - 2023

<u>15 - 03 - 2024</u> end date

fuDelft

INTRODUCTION **

Please describe, the context of your project, and address the main stakeholders (interests) within this context in a concise yet complete manner. Who are involved, what do they value and how do they currently operate within the given context? What are the main opportunities and limitations you are currently aware of (cultural- and social norms, resources (time, money,...), technology, ...).

In the early 2000's patents on additive manufacturing expired and the technique has matured since then (Chapman, 2023). Currently a wide variety of materials can be printed including metals, concrete and even food (Turney, 2021). However over the recent decades the world has seen a new challenge arise.

The climate crisis is well known and every industry is looking for ways to reduce environmental impact, including the additive manufacturing industry (Víšek, 2022). Most additive manufacturing techniques use plastic or metal, using heat to fuse them together.

By using a paste for printing two major environmental factors are addressed. Firstly, the embodied energy of the material for the prints themselves. Paste made of waste streams such as pecan shells not only reduces the waste sent to landfill but also gives it a second life (Rosenthal et al., 2022). Secondly the energy consumption of the printer. Since instead of melting material the evaporation of water used in during printing. This only applies to specific recipes but is vital for the reduction of energy consumption (Faludi, 2017).

Though it has to be noted that many companies are working on making more sustainable filaments (Castaneda, 2023) and that materials used to build printers in the first place also have a large impact.

The faculty of Industrial Design has conducted research on the topic in the past and in this thesis I am going to expand and build upon that knowledge. My focus will lie on evaluating and improving the mechanical strength of printed parts, currently the greatest challenge for the industrialization of paste printing (Faludi, 2018). For society and the transition that our government is making towards a circular economy (Ministerie van Infrastructuur en Waterstaat, 2023) it is vital that new methods of production are developed that allow materials to loop through the system . An important role lies here for universities specifically, as the hubs of research and with the concentration of expertise and facilities they have. So in short, the industry of additive manufacturing is looking towards more sustainable practices, in part pushed by the ambition of both the Dutch and European governments. Industrial Design aims to design sustainably (TU Delft, n.d.). And lastly I am motivated, both academically and personally, to contribute to the circular economy. The combination of all elements mentions above results in my proposed thesis topic of mechanical strength improvement of paste printing.

space available for images / figures on next page

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Initials & Name	M.C. Coster	Student number 4663020	
Title of Project	Improving the mechanical strength of bio material pas	te printing	

TUDelft

Personal Project Brief - IDE Master Graduation



Personal Project Brief - IDE Master Graduation

PROBLEM DEFINITION **

imit and define the scope and solution space of your project to one that is manageable within one Master Graduation Project of 30. IC = 20 full time weeks or 100 working days) and clearly indicate what issue(s) should be addressed in this project

It is redundant to explain the issue of climate change and our need for new technology to reduce environmental impact, so I assume that you as a reader are aware. My thesis focuses on additive manufacturing, specifically on PLA FDM printers used for example for prototypes and hobby projects. Conventional 3D printers have two main factors where their impact lies. Material consumption of printed parts and energy consumption during printing. Paste printing addresses both of these issues, by using a paste that is made from a 'waste' stream, most commonly pecan shells, and by requiring less heat (due to water evaporation in an ambient environment) and thus significantly less energy. One of the major issues with current objects printed in paste is their mechanical strength (Faludi, 2018), among others such as water resistance and aesthetic appeal.

My ultimate goal is to "make paste printing a true competitor, in the sense of strength, of conventional FDM printing of PLA". Important to note here is that significant progress towards this goal is already an amazing achievement. Additionally, I mention PLA here because it very common but ABS is also interesting and widely used so I am going to include both in my analysis.

In order to achieve that I am going to investigate three different aspects of paste printing; recipe, printer parameters, post production. As a start, I will first look into current research to strengthen my understanding of the process. For the recipe I am using current knowledge on best practises as demonstrated by graduating student Henssen (estimated september 2023) and others. Thus I will most likely use pecan shells as a base, other options include various kinds of wood flour and gypsum. For printer parameters the effects I will investigate are speed, layer thickness, temperature, pressure, and printing conditions. Keep in mind that this may be subject to change depending on findings during research. Lastly post production, namely conditions after printing such as humidity, temperature and UV will also be parameters. Depending on the effects of each parameter a focus will be chosen in order to achieve the strength equivalent to PLA.

ASSIGNMENT**

State in 2 or 3 sentences what you are going to research, design, create and / or generate, that will solve (part of) the issue(s) pointed out in "problem definition". Then illustrate this assignment by indicating what kind of solution you expect and / or aim to deliver, for instance: a product, a product-service combination, a strategy illustrated through product or product-service combination ideas, In case of a Specialisation and/or Annotation, make sure the assignment reflects this/these.

In order to "make paste printing a true competitor, in the sense of strength, of conventional FDM printing of PLA" I am going to make adjustments to recipe, printer parameters and post production of paste printed parts to achieve equivalent strength as compared to PLA FDM printed parts.

In order to determine the strength objects will be tested on various behaviours; tensile, 3 points bending and compression. Other options include; torsion, fatigue cycling, impact, and creep. These options are interesting but the focus lies on the most fundamental tests because next to mechanical strength reprintability is an important aspect, especially for circularity. The ability of the material to be recycled, ideally back into a printable paste is important and appropriate tests will have to be conducted. Again, the first three tests are the focus and any tests regarding life-cycle will be determined once I have acquired sufficient knowledge. Other tests such as water resistance, durability in UV and hardness tests are out of scope for this project. As well as in depth analysis of mechanical behaviour by the other options as described above. However parts should be able to maintain shape and structural integrity in an ambient environment for proper time. What this exactly is will be determined based on literature research.

In order to determine the progress towards my goal, characteristics of PLA and ABS will serve as a benchmark. The tensile strength is 64 MPa and compression is 76 MPa (Granta, 2022). In order to prevent the testing equipment from having a too large impact on results FDM printed samples of PLA and ABS will also be tested with the same equipment.

For these tests I aim to use ISO 527 (for tensile tests), ISO 178 (for 3 point bending) and ISO 604 (for compression) to adhere to internationally recognized methodology. Testing equipment in the Applied labs will be necessary to conduct these tests as well as guidance from staff.

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Initials & Name	M.C.	Coster		Student number	4663020	
Title of Project	Improvi	ng the mech	anical strength of bio material pas	e printing		

Personal Project Brief - IDE Master Graduation

PLANNING AND APPROACH **

Include a Gantt Chart (replace the example below - more examples can be found in Manual 2) that shows the different phases of your project, deliverables you have in mind, meetings, and how you plan to spend your time. Please note that all activities should fit within the given net time of 30 EC = 20 full time weeks or 100 working days, and your planning should include a kick-off meeting, mid-term meeting, green light meeting and graduation ceremony. Illustrate your Gantt Chart by, for instance, explaining your approach, and please indicate periods of part-time activities and/or periods of not spending time on your graduation project, if any, for instance because of holidays or parallel activities.



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Initials & Name	M.C. Coster	Student number 4663020	
Title of Project	Improving the mechanical strength of bio m	naterial paste printing	

Personal Project Brief - IDE Master Graduation

MOTIVATION AND PERSONAL AMBITIONS

Explain why you set up this project, what competences you want to prove and learn. For example: acquired competences from your MSc programme, the elective semester, extra-curricular activities (etc.) and point out the competences you have yet developed. Optionally, describe which personal learning ambitions you explicitly want to address in this project, on top of the learning objectives of the Graduation Project, such as: in depth knowledge a on specific subject, broadening your competences or experimenting with a specific tool and/or methodology, Stick to no more than five ambitions.

My entire masters program I have focused on sustainability, with every project I had the opportunity to choose (solar surplus in neighbourhoods & circular bicycle helmet), with both of the extracurricular summer programs I did (circular economy in Finland and design for disassembly in Italy) and most of all with my internship (product & test designer of circular solar panels). During my thesis I want to contribute to sustainability in a broad sense, while also acknowledging the limitations of a thesis. During the pandemic I got my 3D printer and over the years I've enjoyed working with it and experiencing the opportunities it gives. For my thesis to revolve around making something I enjoy so much more sustainable is a great combination. It is not realistic to expect to make paste printing the perfect alternative for conventional 3D printing within one thesis project. By focusing on mechanical strength I have a topic that is doable within a hundred days. During this project I have the opportunity to showcase my problem solving skills, my ability to think creatively within a set of boundaries but not be limited by them and experience with an iterative workflow. In addition, by using the sprint system described earlier, I can learn more about working agile and gain first hand experience on the benefits and drawbacks of the method. Vital lessons that can aid me when I start working at a company.

FINAL COMMENTS

ments, please add any information you think is relevant.

I have had severe RSI (previously discussed with the academic counselors) and to prevent the issue from flaring up again I will work four days a week, taking one day a week to rest my wrist. This entire project will be done four days a week, resulting in 25 weeks (see Gantt Chart). Throughout the project I have planned two holidays with friends & family, week 52 and week 6 & 7.

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Initials & Name	M.C. Coster		Student number	4663020	
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