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Direct digital manufacturing of products based on high content of stone powder

Master in Product Design Engineering

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Master's Project held under the guidance of Doctor Artur Jorge dos Santos Mateus, Professor of the Higher School of Technology and Management of the Polytechnic Institute of Leiria

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Originality and Copyright

Dedication

To all the great minds
that have augmented collective knowledge via their intellect

To my parents, family, friends and Márcia.

Acknowledgements

To my supervisor, Professor Artur Matheus for the availability and guidance; to Professor Fábio Simões for the interest and availability that facilitated the finalization of this dissertation.

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To all the people I've met throughout life and this Master's Degree, that contributed to my personal and academic evolution.

To my family for the support and last but no least, to Márcia Nakandacare for the support, positive motivation and unconditional affection.

To all, my thanks and appreciation!

Abstract

Residue creation from industrial transformation processes are the focus of circular economy studies with the intent to boost revenue and improve environmental impact. As in the case of stone sludges and powders, residue elimination or re-usage, would bring forth reductions in post processing costs, improve environmental impact, as well as adding new revenue streams with added products to the companies portfolio.

This project aims to develop a technical road-map of materials and processes for stone residues and powders, in which they are incorporated and transformed into new products, via additive manufacturing, using thermosetting polymeric materials as binder and incorporating them in different mixture rates.

Trial and error experimental processes, using Biresin G26, Pavistone 1K and Pavistone 2K resins, incorporating a variety of filler ratios, determine that, usable compounds have filler contents from 30 to 86% wt. Resulting compounds were extruded manually, via syringes, and approved mixtures undergo mechanically assisted extrusions, via an adapted deposition printer. Materials with promising outcomes for additive manufacturing techniques define the material processing windows. Materials in this study were subjected to tensile testing, hardness tests and micro computed tomography scans. Joint analysis of these properties and produced extrusions then define the admissible additive manufacturing technique for industrialization.

This study aims to provide results in the domain of material properties and equipment feasibility. Results show that: filler additions lead to a decrement of the mechanical properties; Losses of properties up to 90% were recorded, with exceptions of minimum gains of 170% for specific binder/filler combinations; filler increase drives increases of porosity; and mixtures with growth in their curing processes are not suitable for additive manufacturing processes that require defined form. The wide range of specifications of these materials and their initial formulations, will provide a wide range of end products that can also be applied in a multitude of applications. Existing technology in conjunction with the concept extruding head of this project, provide a system that is adjustable for a wide latitude of materials and end products.

Keywords: *additive manufacturing, reactive extrusion, stone waste, calcium carbonate powders*

Resumo

O aumento da importância das economias circulares, com o intuito de aumentar as receitas e melhorar o impacto ambiental, despoleta a avaliação dos resíduos criados nos processos de transformação das indústrias. Tal como no caso das lamas e pós de pedra, a eliminação ou a reutilização de resíduos, provocaria reduções nos custos de pós-processamento, melhoraria o impacto ambiental, bem como adicionaria novos produtos e fluxos de receitas às empresas.

O projeto tem como objetivo providenciar uma tabela técnica de materiais e processos em que os resíduos são incorporados e transformados em produtos novos, por processos de fabrico aditivo, usando diferentes materiais poliméricos termoendurecíveis como ligante na incorporação de resíduos de pedra em concentrações diversificadas.

Abordagens de tentativa e erro experimental, utilizando resinas de Biresin G26, Pavistone 1K e Pavistone 2K, com incorporações variadas de resíduos, determinam que compostos utilizáveis terão cargas de 30 to 86% wt. Os materiais resultantes são extrudidos manualmente, através de seringas, que após validação, passam por processos mecanicamente assistidos com recurso a uma impressora de deposição adaptada. Os mais promissores para técnicas de fabrico aditivo definem as janelas de processamento dos materiais. Os materiais deste estudo foram submetidos a testes de tração, de dureza e a micro tomografias computorizadas. A análise conjunta destas propriedades e as extrusões produzidas definem a técnica admissível de fabrico de aditivos para a industrialização.

Este estudo pretende fornecer resultados no domínio das propriedades dos materiais e da viabilidade de produção dum equipamento de fabrico aditivo de polimerização reativa com elevada carga de resíduos. Primeiro, os resultados revelam que: teores de cargas elevados conduzem ao decréscimo das propriedade mecânicas; materiais resultantes apresentam perdas de até 90% das suas propriedades, com algumas exceções com ganhos mínimos de 170%; a porosidade aumenta com o aumento do teor de cargas; e formulações com expansão durante a cura não são adequados para processos de fabrico aditivo que requerem forma definida. Segundo: com a vasta gama de especificações e formulações iniciais destes materiais, obter-se-á uma vasta gama de produtos finais, aplicáveis numa multiplicidade de aplicações e situações; que tecnologia existente, em conjunto com o conceito de extrusão desenvolvido neste projeto, fornece um sistema ajustável a uma ampla latitude de materiais e produtos finais.

Palavras-chave: *fabrico aditivo, extrusão reactiva, resíduos pedra, carbonato de cálcio*

List of Figures

List of Tables

List of acronyms

3D	Three Dimensional
3DP	Three Dimensional printing
aEDP	adapted Extruding Deposition Printer
AM	Additive manufacturing
AMfRP	Additive Manufacturing filler Reactive Polymerization
ASTM	American Society for Testing and Materials
CAD	Computer Aided Design
CE	Common Era
CEO	Chief Executive Officer
CT	Computed Tomography
DED	Directed Energy Deposition
DMLS	Direct metal laser sintering
EBM	Electron beam melting
ESTG	Escola Superior de Tecnologia e Gestão
FDM	Fused Deposition Modeling
HU	Hounsfield units
ISO	International Organization for Standardization
LAB	Laboratory of the Centre for Rapid and Sustainable Product Development
LOM	laminated object manufacturing
Micro-CT	Micro Computed Tomography
PBF	Powder bed fusion
PTFE	Polytetrafluoroethylene
PU	Polyurethane
ROI	Region of interest
SHS	Selective heat sintering
SLIM	Solid/Liquid Injection Manifold machine
SLM	Selective laser melting
SLS	Selective laser sintering
UAM	ultrasonic additive manufacturing
UV	Ultra violet
VOI	Volume of interest

List of symbols

E_F	Extruded form
F_R	Flow resistance

Contents

1 Introduction

The never ending battle of cost reduction, revenue increase, material re-usage and minimal waste has businesses nowadays looking inwards to their production processes and re-evaluating what they consider residues and scrap. The common business model of a core product system is now developing into businesses that re-use their residues and return those into the economic cycle as new products that boost company revenues, reduce their costs and have a positive environmental impact. This project is one of those cases, aiming to turn stone sludges from the transformation process, considered as waste, into new products that simultaneously reduce costs from the post processing of the main product and bring a new revenue stream with added products to the companies portfolio.

1.1 Relevance of this project

The central region of Portugal is heavily populated with quarries that are dedicated not only to the extraction but also to the transformation of stone into several types of products. The transformation processes of these industries produce a considerable amount of sludge residues that have environmental impacts and handling costs. According to recent data, provided by MVC - Portuguese Limestones CEO (chief executive officer), these companies can produce 20 to 80 tons of stone sludges and powders per week being 30% of the content (wt%), water.

Several studies have been carried out in the probable usage of these residues in a manner to reduce deposition costs and favor the re-use of these extracted materials. One case is the ceramics industry, where the incorporation of these residues brings up serious CO_2 release during baking and the other is the cement industry where the incorporation of these residues produce inferior quality products. For this industry there are other products at lower costs that produce better cost-effective materials with better mechanical properties.

In general, all the applications that require the transportation of the stone sludges from their production locations will naturally deem these as unusable from an economic standpoint.

1.2 Goals of this project and materials to be tested

This project is based on experimental research aiming to provide a technical material road map of the resulting materials that mix stone residues to Polyester, Polyurethane and Isocyanates. These are then tested for their shape stability, mechanical strength and surface rigidity. With the gathered information a concept is then developed for a system that can produce these extrusions.

From the universe of available materials several were selected and later some discarded due to the fact that we specified that the materials should be curable at room temperatures. The materials to be mixed and used in this project have been specified and listed as follows.

- **Biresin G26** is a high quality, fast cast, two component (Polyol + Isocyanate) polyurethane casting resin which is widely used for pattern and model making (see Appendix A1);
- **Pavistone 1K** is a Polyisocyanate aggregate binder normally used for pavements (see Appendix A2);
- **Pavistone 2K** is a two component (Polyol + Polyisocyanate) polyurethane binder for construction applications (see Appendix A3).

To characterize each resultant material and to build the materials road-map the following tests have been considered:

- Extrusion with the aid of a syringe to verify when the paste is no longer extrudable and when it starts/stops maintaining its shape;
- Evaluation of viscosity and the highest possible stone sludge content that maintains extrudability for each of the tested materials;
- Subject produced specimens to mechanical testing;
- Subject produced specimens to hardness testing;
- Subject produced specimens to Micro Computed Tomography scanning (micro-CT);
- Evaluation of material storing challenges, dosing and mixing of components (resins, hardeners, reagents, additives, loads, etc...) for an automatic process;
- Design and conceptualize an automatic system that would materialize the above.

1.3 Methods and techniques used

Several types of testing had to be figured out due to the different behaviours of the array of selected materials. For the tests explained in this chapter the following factors were considered: forms of mixing, type of application, viscosity and curing times.

In all of the types of tests performed, the test matrix represented in Table 1.1 will be populated by the selected binder and the residue content in proportions represented by equation 1.1, taking into consideration that an initial test is performed to validate the viscosity of the mixture which could influence the start off factor of equation 1.2. The amount of tests being performed will rely on experimentation and specimen integrity, being so, that in some cases more tests will have been achieved than with others. A minimum of 30% residue has been selected as the minimum proportion to be added to the binder represented by equation 1.2. The subsequent iterations of the equation 1.1 will be the addition of 10% of the residue ratio, to the value of the previous iteration. The number of iterations are shown in equation 1.3, being ***t*** the number of the final iteration that represents the test were the mixed material no longer has psychical properties that are required for the final product.

$$x_n = x_{n-1} + 10 \quad (\%) \tag{1.1}$$

with

$$x_0 = 30 \qquad (\%) \tag{1.2}$$

and

$$n = 1, 2, ..., t \tag{1.3}$$

The goal is to obtain the highest possible residue incorporation, meaning that the residue may have higher volume/mass percentage than the binder being used. The resulting mixes are left to cure for at least 24 hours and will then be cut for internal and external analysis. From the initial battery of tests, valid candidates will be selected to remix and make new test specimens for mechanical testing and further extrusions on an adapted printer for form evaluation. These tests can then be extended if deemed necessary.

Table 1.1: Example table for material experiment setup (Empty table, filled-in in the next chapters)

Test Material	Residue %	Extrusion form	Flow resistance	Approved for 2nd mix
Biresin G26	30 (n+1) max			
Pavistone 1K	30 (n+1) max			
Pavistone 2K	30 (n+1) max			

1.3.1 Manual extrusion based specimens technique

The mixtures will be extruded using a syringe with an applied manual force. The amount of extruded material will be dependent of the mixture viscosity, thus producing the test specimens will rely on experimental sensitivity.

Keeping in mind that the homogeneity of specimens is crucial for the selection process to have any kind of validity, the extrusion, independently of the material viscosity, was produced as a 10 cm long extruded string within approximately 10s.

1.3.2 Syringe extrusion using modified FDM printer (aEDP)

The selected materials from the manual syringe extrusion process will then be re-mixed for testing in a modified Fused Deposition Modelling (FDM) printer, that we will refer from this point on-wards as an adapted Extruding Deposition Printer (aEDP). This printer has its Heating head replaced by a support that holds a syringe which can be emptied out in a controlled manner via an adapted motorized screw. The syringes will be prepared with the mixture to be extruded, placed in the holder and submitted to the program, written in G-code, for this specific task. The program complies of extruding several strings with different machine configurations for evaluation.

1.4 Project Structure

This project is divided into 6 chapters. After this introductory chapter, it will be divided as:

- Chapter 2 describes the *State of the Art* in regards to usage of calcium carbonate in diverse industries, with a brief history for the importance of stone in mankind evolution as a construction material and several examples in a diversity of fields are given for the usage of stone residues. Then several additive manufacturing techniques classified by ISO/ASTM 52900:2015 are listed and explained as well as the new technique that does not fall under any classification in this standard, which will be introduced in this project.

- Chapter 3 describes in detail the experimental methodologies used to obtain specimens needed for a preliminary selection process. It then describes the process and parameters for the manual extrusion experiments of ambient curable resins and the validation of specimens for further testing, on the aEDP, with developed G-Code software for production of basic strings and/or shapes for analysis.

- Chapter 4 is the sample characterization, were several material properties are tested and the data is collected and summarized.
- Chapter 5 describes the concept of an equipment able to provide product based on the technique this project develops.
- Chapter 6 presents the conclusions of the present study and further work.

2 State of the Art

2.1 Introduction

In this chapter an approach to some studies performed on the uses of Calcite, their applications and the existing production techniques will set a base for the comparison of what this project intends to achieve, which is the massive usage of stone residues in products obtained by additive manufacturing. This chapter also includes a summary of the additive manufacturing techniques classified as per ISO/ASTM 52900:2015 as well as the novel process, which is developed in this project and is not classified in this standard. Finally some commercialized systems and their mixing heads are described.

2.2 History of stone usage

Even before time could be recorded, stone has been the election material for building and for memorial material. The oldest remaining structures in the world are made from stone and it is still used in and on new structures. This is not only due to its availability and natural beauty, but also because of its enduring quality. In Figure 2.1 there are some examples of the oldest buildings in the world that have maintained the requirements to be such. The use of natural stone is re-surging, continuing the trend and desire for natural stone that begun centuries ago.

It is a fact that stone industries produce several tons weekly of sludge's and powders [7], thus it only makes sense to try and re-use this material and transform it into other usable products. Stone sludges and powder residues resulting from extraction and transformation processes in the quarries from the central region of Portugal have been studied before and their chemical, physical, mineralogical and morphological results show that these could be used as raw material in several industrial processes with little or no treatment at all [8]. Figure 2.2 has an example of a stone quarry and its cuts.

Portuguese limestones are in majority composed of carbonates: calcite $CaCO_3$ and dolomite $CaMgCO_3$ [9] and we exemplify with some cases were they are used by other industries:

- Calcite or calcium carbonate is a widely used material in industries such as cement, glass and pharmaceutical, it can also be used in the ceramics industry as a whitener and as a humidity corrector;
- The incorporation of marble powder, stone residues from transformation, into ceramic bricks has also been studied with different contents in molded samples and fired in and oven. The conclusions were that in several cases the properties of the final product improved, but the best considered proportion was considered as 15-20% of stone residues in the red ceramic material [10];
- The incorporation of marble powder to improve the performance of problematic soils with the help of cation exchange reactions. The Excess of CA^{2+} is responsible for the improved performance. The mixed soil also shows improved mechanical properties and strength characteristics [11];
- Recent studies have proved that stone sludges can be used as fillers in the tyre industry with potential profit and added environmental value. The incorporation of the

(a) Barnenez, France, 4850 BC. [1]

(b) Tumulus of Bougon, France, 4700 BC. [1]

(c) Anu ziggurat, Iraq, 4000-3800 BC. [2]

(d) Pyramid of Djoser, Egypt, 2667-2648 BC. [3]

(e) Dolmen de Viera, Spain, 2000 BC. [4]

(f) Templo de Hera, Italy, 550 BC. [5]

(g) Colosseum, Italy, 70-80 AD. [6]

Figure 2.1: Some of the oldest known surviving buildings

Figure 2.2: Quarry cuts example [7]

stone residues improved the performance properties of the rubber compounds, with an increase in the elongation capabilities, thus turning a waste into a product [12];

- In the medical and pharmaceutic domains bio-material composites containing calcium carbonate are increasingly suggested as applications for bone replacement material [13] and as drug delivery systems [14].

The goal is to incorporate the biggest amount of residues possible and to avoid processes that during their curing stages release gaseous mixtures containing carbon. For this reason, all processes that involve firing or values of heat that release carbon will not be considered as processes for manufacturing. This fact sets path to alternate manufacturing processes, being the additive manufacturing process the selected one.

2.3 Additive Manufacturing

3DP (Three Dimensional Printing), also known as Additive manufacturing (AM), is a process that, by deposition of material in a layer like fashion, creates an object based on a digital model. Additive manufacturing, in opposition to subtractive manufacturing, creates a final product by addition of parts, while the other forms the final product by cutting away from a block of material. Plastics and metal alloys are the most common materials used for 3D (Three Dimensional) printing but almost all types of materials can be used in this production process from concrete to living tissue. There are several variations of this type of technology, as shown in Figure 2.3, that are described in the standard ISO/ASTM 52900:2015 that have been briefly addressed in this chapter. Considering that the concept developed in this project is not characterized in this standard, a section will be added with a new classification designated by Additive Manufacturing filler Reactive Polymerization (AMfRP). This new approach is very similar to material extrusion with the difference of using thermoset materials instead of thermoplastics.

2.3.1 Material Extrusion

FDM is a common material extrusion process and is trademarked by the company Stratasys. In this process, represented in Figure 2.4, the material is drawn through a heated nozzle and deposited layer by layer on a vertically moving platform. The product is formed from a sliced CAD (Computer Aided Design) model and the material is often added to the machine in spool form and fed through the nozzle under constant pressure and in a continuous stream.

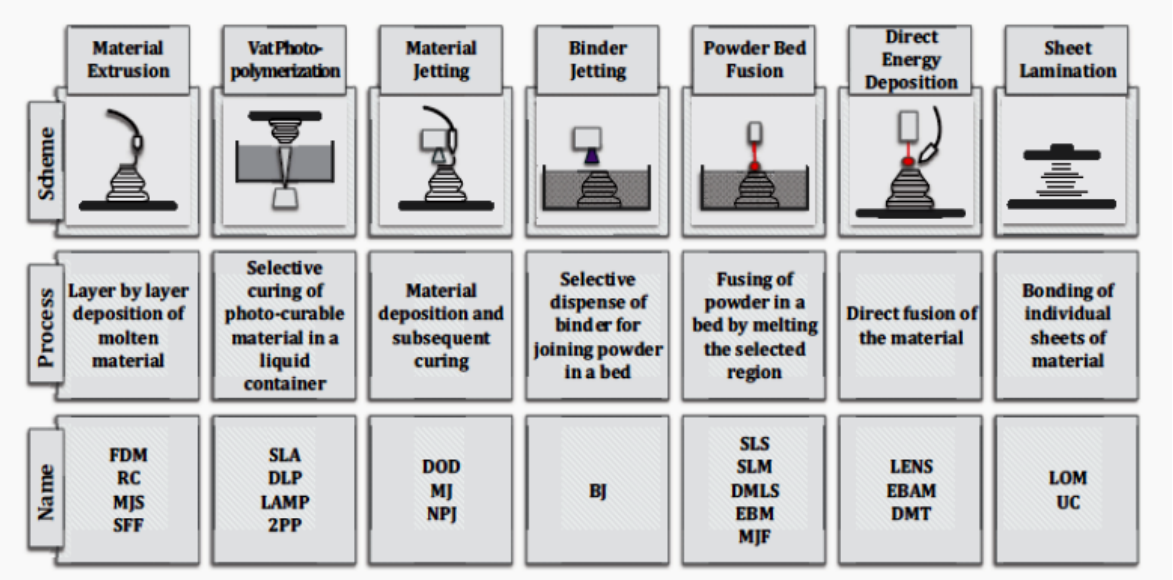


Figure 2.3: ASTM classification for Additive Manufacturing [15]

Although this system seems suitable it uses a heated nozzle and thermoplastic material which makes it an unsuitable for our application.

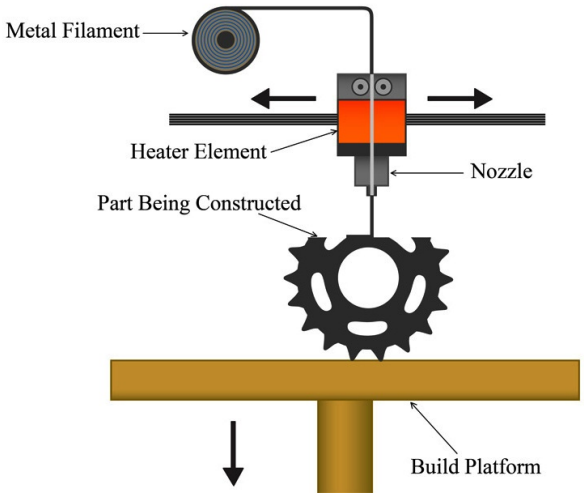


Figure 2.4: Material extrusion process [16]

2.3.2 Vat Photo-polymerization

A vat of a photo-polymer liquid is cured by focused UV (Ultra Violet) light in which the model is constructed layer by layer. During fabrication the platform moves downwards after each layer has been cured. Due to the fact that the object is made in a liquid environment there is no need for structural support and this process produces products with a high-detail surface finish. Figure 2.5 shows two distinct processes being one Stereolithography in which the polymer cures with the focus of a laser in a specific point and the second being Digital Light Processing where UV light is area-projected, curing a complete layer at a time. This process would not fit the requirements of this study, because the polymers in use do not have UV additives required for the curing process during construction of the end product.

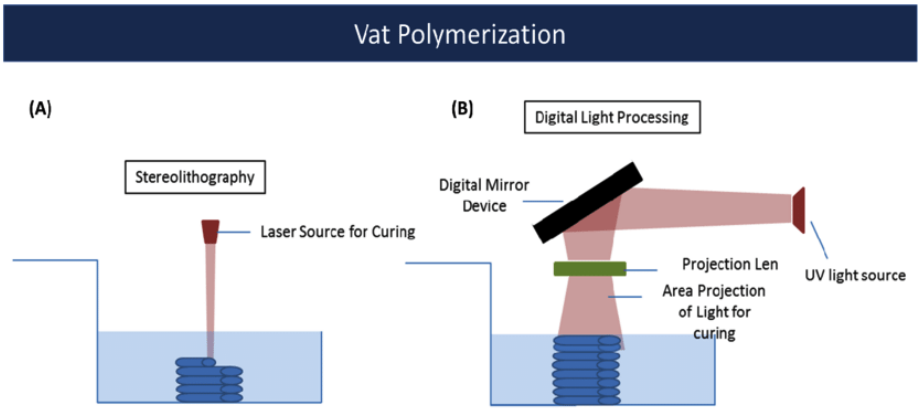


Figure 2.5: Vat-polymerization processes [17]: (A) Stereolithography and (B) Digital Light Processing

2.3.3 Material Jetting

Material jetting creates objects by jetting material onto a build platform, where it solidifies, using either a continuous or drop method. When the product is finalized it is then cured or hardened by UV exposure and it is primarily used where surface finish and form testing is needed. The most common materials used are polymers and waxes due to their viscous nature and ability to form drops. Figure 2.6 exemplifies a material jetting system. The polymer matrix used in this study has no UV curing capabilities and the addition of fillers will also reduce the droplet formation of the mixtures. These facts deem material jetting as a non-usable process in this study.

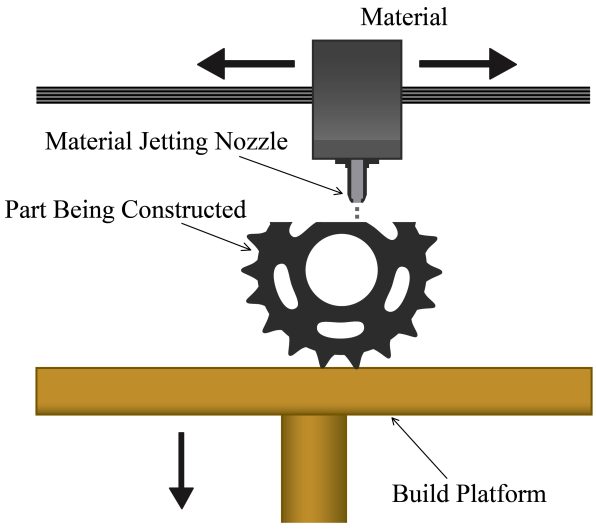


Figure 2.6: Material jetting process [18]

2.3.4 Binder Jetting

Binder jetting uses a powder based material and a binder in a liquid form. A printing head deposits alternating layers of the build material and the binder, which acts as an adhesive between the powder layers. During fabrication the platform, shown in Figure 2.7 item 2, moves downwards and the object being printed is self-supported within the powder bed. This method allows full-color prototype fabrication, requires additional post processing but

due to the material characteristics it may not be suitable for structural parts. The binder jetting process cannot be used as a solution, due to the fact, that fabrication is performed in a platform and powder is not mixed directly into the polymer stream.

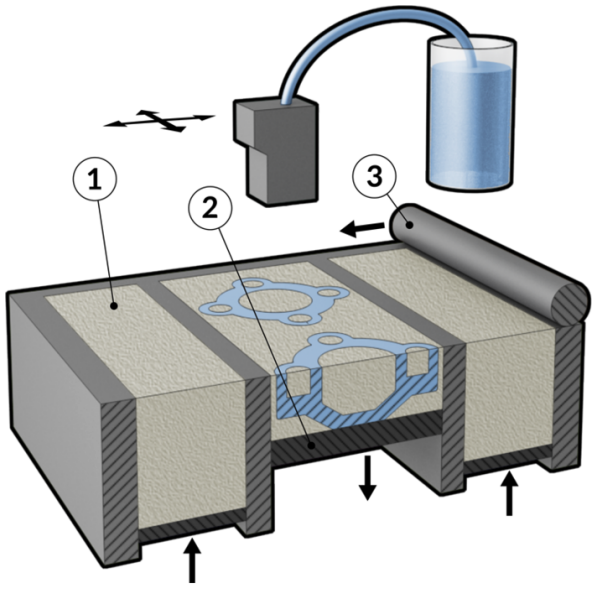


Figure 2.7: Binder jetting process [19]

2.3.5 Powder Bed Fusion

The Powder Bed Fusion process includes the following commonly used printing techniques: Direct metal laser sintering (DMLS), Electron beam melting (EBM), Selective heat sintering (SHS), Selective laser melting (SLM) and Selective laser sintering (SLS).These methods use either a laser or electron beam to melt and fuse material powder together. Electron beam melting (EBM) methods require vacuum but can be used with metals and alloys in the creation of functional parts. They involve the spreading of the powder material over previous layers that can be done either by roller or a blade. A reservoir aside of the bed provides a fresh material supply. Direct metal laser sintering (DMLS) is the same as SLS, but with the use of metals and not plastics. The process sinters the powder, layer by layer. Selective Heat Sintering differs from other processes by way of using a heated thermal print head to fuse powder material together. As before, layers are added with a roller in between fusion of layers. A platform lowers the model accordingly. This is normally used for circuits, structures, and parts. A schematic of a powder bed fusion process is shown in Figure 2.8. The powder bed fusion processes, as the name states, fuses materials together. One of the main requirements in the scope of this study the use of ambient curable processes, which rules out this process automatically.

2.3.6 Directed Energy Deposition

This process repairs or adds to existing components by extruding laser-melted material, commonly metal powders, with a multi-axis nozzle onto the printing surface. It is similar to material extrusion, but the nozzle can move in multiple directions and is not fixed to a specific axis. The material, which can be deposited from any angle due to 4 and 5 axis machines, is melted upon deposition with a laser or electron beam. The process can be used with polymers or ceramics but is typically used with metals, in the form of either powder or wire. In general this process is used in repairing and maintaining structural parts and Figure 2.9 shows a

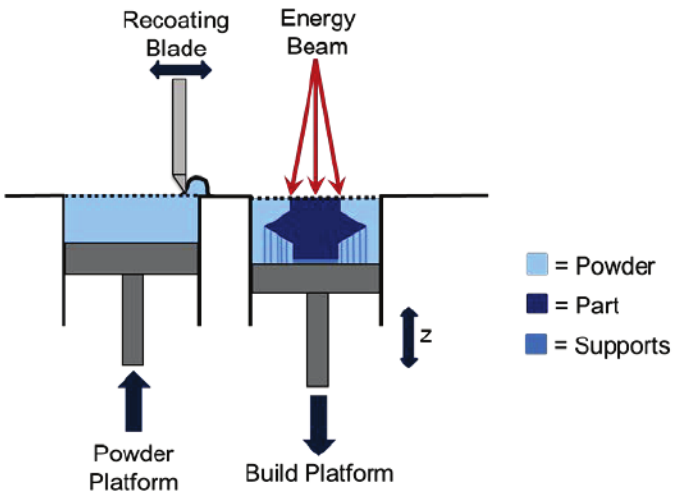


Figure 2.8: Powder bed fusion process [20]

schematic of it. The fusion temperatures involved in theses processes are not applicable to the matrix or materials used in this study.

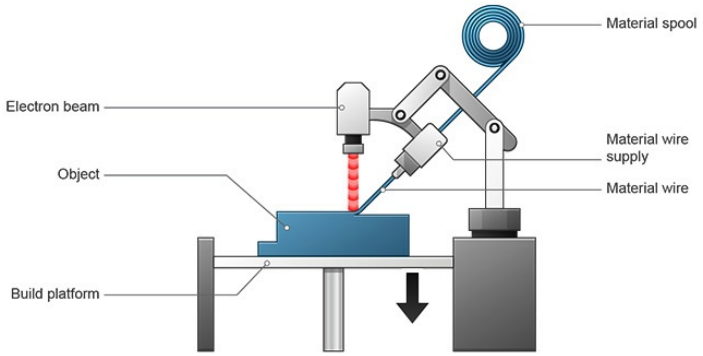


Figure 2.9: Directed energy deposition process [21]

2.3.7 Sheet Lamination

Sheet lamination processes include ultrasonic additive manufacturing (UAM) and laminated object manufacturing (LOM). The Ultrasonic Additive Manufacturing process uses sheets or ribbons of metal, which are bound together using ultrasonic welding. The finished shaping is completed through further material removal processes. Laminated object manufacturing (LOM) uses a similar layer by layer approach but uses paper as material and adhesive instead of welding. The LOM process, exemplified in Figure 2.10, uses a cross hatching method during the printing process to allow for easy removal post build. Laminated objects are often used for aesthetic and visual models and are not suitable for structural use. The process is low temperature and allows for internal geometries to be created. The process can bond different materials and requires relatively little energy, as the metal is not melted. This study intends to mix residues into a polymer matrix and manufacture product for several applications. The lamination process would narrow the product portfolio down to sheet material as well as providing fragile sheets due to the high filler inclusions.

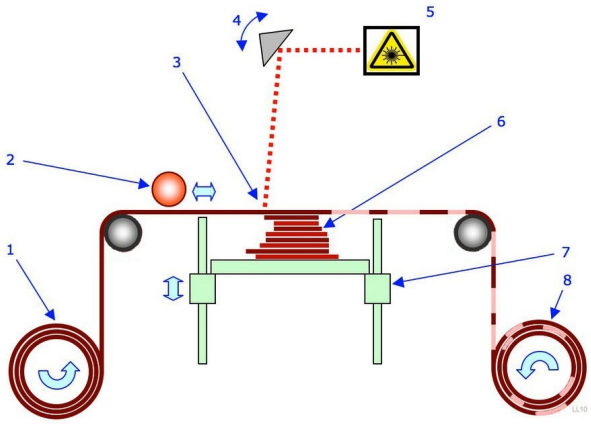


Figure 2.10: Sheet lamination process [22]

2.4 Novi

Novi is a project developed at the Brunel University in London. It consists of a 3D printing mechanism that was designed to produce parts from the extrusion of a limestone mud. The limestone prototype parts presented many challenges. For example, a printed object breaks easily under tension and, in contact with water, disintegrates into mud. Compounds were added to the paste to improve its properties. Initially a Mendel90 3D desktop printer was used, adapted for a 10 milliliter (ml) syringe, then later a robotic arm with tri-axial movements, called Novi, was adapted. Initial tests proved difficult because the extrusion head clogged and for that reason the extrusion nozzle had its diameter incremented to 8mm and to allow larger scale prints the syringes capacity was increased to 200ml. This system can be seen in Figure 2.11. Novi has been included, as it also tests the challenges of printing with limestone mud. Although the matrix is not of a thermoset material, it showcases the advances made in the field of printing with stone residues and for that same reason is not the process considered to fulfill this studies requirements.

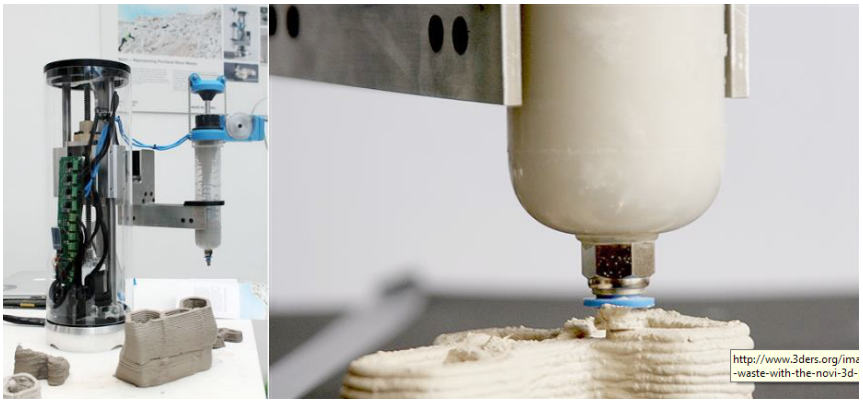


Figure 2.11: Novi 3D printing mechanism [23]

2.5 Additive manufacturing techniques based on reactive polymerization

2.5.1 Introduction

This project has set the goal for massive usage of stone residues in products obtained by additive manufacturing. As stated earlier, the concept developed in this project does not fit under a specific classification of the standard ISO/ASTM 52900:2015. This concept will from now be designated as Additive Manufacturing filler Reactive Polymerization (AMfRP). It is similar to the material extrusion process but uses a thermosetting polymer matrix instead of a thermoplastic one. The advantages of using this type of matrix are: higher additions of fillers; lower viscosity; ambient temperature cure that saves on energy costs; lower feed pressures and lower equipment costs.

This chapter will contain after this introduction a section with a brief exposure of thermoset materials and compounds with high content residues. The third section is related to research of commercial applications and techniques used for systems that require high filler additions in their production methods. The fourth section is a brief explanation of compounding extruders and their function. In the fifth section we list several existing models of mixing heads that can include additional streams of solid fillers and finally in the sixth section a brief introduction is made to side feeders, their functionality and efficiency.

2.5.2 Thermoset and high residue content compounds

A Thermosetting polymer, resin or plastic is composed of a polymeric chain, chemically and irreversibly joined by a curing process. These polymers differentiate themselves from others due to their strong covalent three dimensional network of bonds. Thermosetting polymers are commercially designated as resins and have good chemical resistance, low mass, high resistance to thermal distortion, dimensional stability, high modulus and adaptability to processing methods in comparison to thermoplastic materials. The most used and economically favorable are polyesters, polyurethanes, vinyl ester and phenolic resins; generally to produce glass fibre reinforced composites. Epoxy resins, apart from their higher cost, boast better mechanical and moisture resistance properties and are widely used in aerospace applications. The curing process of resins is a exothermic, promoting a drop in formulation viscosity due to internal heat buildup followed by increases of viscosity and molecular mass. This process can be split into three distinct stages: geltime is the first stage, which is the period from when mixing has started to the time the mixture has a gel-like property and there is a rise in temperature, denominated the exothermic peak; the second stage is the hardening time, which is when the compound obtains a significant part of its mechanical properties; and the third is the maturing stage, which is the period of time necessary for the compound to obtain all of its mechanical properties and dimensional stability. Residues are in general added to compounds with the intent to improve the compound properties, processing capabilities, material usage and cost reduction. High incorporation of residues in compounds normally promotes an increase in hardness and tensile strengths [24].

2.5.3 Mixing processes

To achieve a final product that is homogeneous and has consistent quality the mixing efficiency is critical, thus air incorporation and lumping are factors that need to be eliminated.

Air incorporation

The challenge of mixing powders with liquids is the prevention of incorporation of air which will produce foaming. The whipping action during mixing tends to trap and incorporate air.

The challenge of mixing powders with liquids is the prevention of incorporation of air. The whipping action during mixing tends to trap and incorporate air that may rise and then escape. However, air can stabilize into foam on the products surface or it may dissolve into the product which will later cause foaming in the subsequent process.

Lumping and clogging

When mixing and dissolving powders thoroughly into liquids, the most important challenge is the prevention of lump formation. Lumping reduces the efficiency of the aggregates, meaning that more material has to be added to achieve the same functional result and it also prevents the product from being completely formed. To prevent lump formation a variety of mixing issues have to be considered: the dispersion of the powder; the wetting of solids; shearing and the breakup of agglomerates. So in order to prevent lumping and clogging the powder should be handled correctly, equipment with high shear forces have to be used, the addition of powder to the mix has to be controlled and the right mixing temperature has to be selected. As referred by Paul Edward et Al, "Batch mixers, with an optimal mixing unit inside the mixer tank, are the ideal solution when dealing with difficult powders and highly viscous liquids. Additionally, by dosing powder on the liquid's surface and transporting it quickly towards the mixing head in a controlled vortex, we can wet the powder optimally by ensuring minimum exposure to the liquid medium." [25].

Commercialized mixing processes

Commercially there are several companies specialized in manufacturing devices for powder inclusion in liquid mediums and of the many MGT Industries Ltd is referred to due to their extensive applications and experience, as explained by Eng. Korenblit (M.Sc), Mixing expert at MGT Industries Ltd, specialized in Liquid & Process Systems. To overcome this mixing challenge, various technological solutions have been tested. One such a solution involves a dispersion shear disk. In a second method, a tri-blender utilizes the Venturi effect to create a vacuum that absorbs powder and injects it into a stream of liquids. A third solution involves a direct interior pipe feeder in a rotor-stator homogenizer. These solutions, although different, are effective because powders meet the liquid before entering the shear zone, an area that is prone to lumping. A vacuum is created in the shearing zone that separates particles and allows for better wetting and simultaneously applies shearing power to ensure no floating solids are formed. As per Korenblit, the method that would be most viable and reliable on an industrial scale would be the rotor-stator homogenizer while injecting fine powder into an intensive shearing zone to prevent the formation of powder floating solids.

2.5.4 Compounding extruders

Compounding is a continuous process that is used to mix together several materials into a homogeneous mass. It can be a distributive process, which consists in distributing the components in a uniform ratio without being broken down or dispersive, that consists in mixing by breaking down the agglomerates. Compounding extruders perform several functions: feeding, melting, mixing, venting and developing form via a localized pressure through a die. Several examples exist: single screw; twin screw counter-rotating inter-meshing; twin screw co-rotating intermeshing and twin screw counter-rotating non-intermeshing. Single-screw extruders are generally inadequate to perform dispersive compounding and are designed to maximize pumping uniformity while on the other hand twin screw systems are dedicated to mixing with minimal regard to pumping, performing blending and compounding of particulates into plastics [26]. Twin-screw extruders are available commercially in three modes, see Figure 2.12, co-rotating intermeshing; counter-rotating intermeshing and counter-rotating

non-intermeshing. Each boast attributes for certain applications and the two inter-meshing types are generally better suited for dispersive compounding.

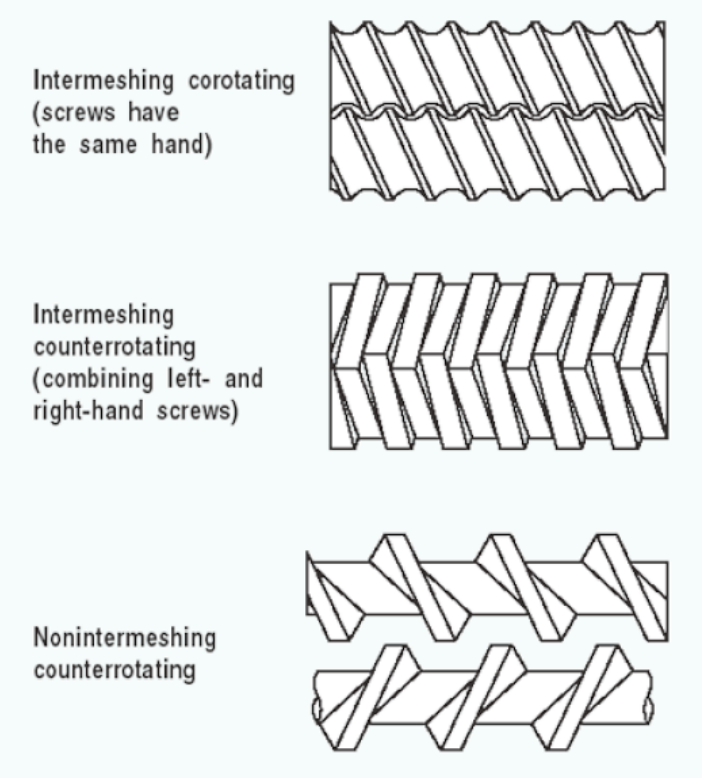


Figure 2.12: Twin-screw extruder types [26]

2.5.5 Mixing Heads

Among the many existing solutions in the market for mixing heads, being them from low to high pressure, from feeding of single to multi component materials, we shall focus on those that have relevance to this project.

Cannon commercializes a solution for thermoset plastics, represented in Figure 2.13, which includes a third stream that adds recycled powders within the mixing head. This head can dose and mix three individual components through axial jets, which means that polyol no longer carries the solid residues as in other applications. These are fed directly to the mixing head through the port were normally coloring pastes were added [24].

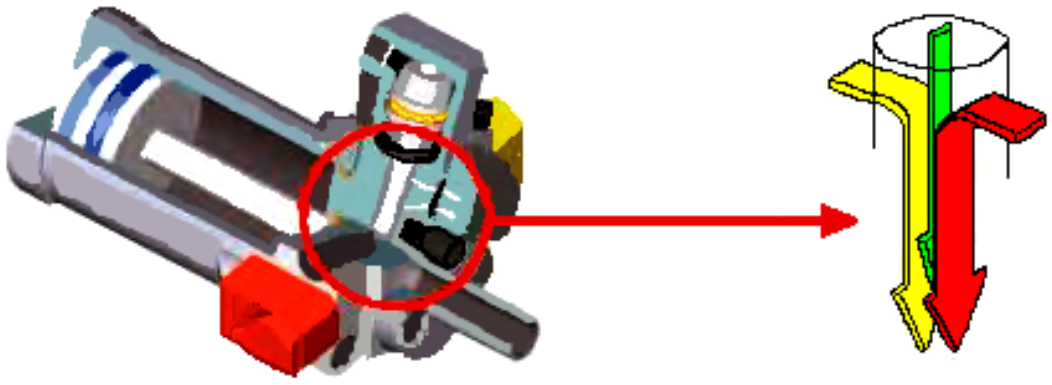


Figure 2.13: Inclusion of third stream in mixing head (Cannon Polyurethane Technology) [24]

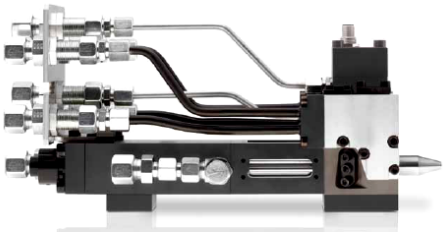


Figure 2.14: Transfer mixing head (KraussMaffei) [27]

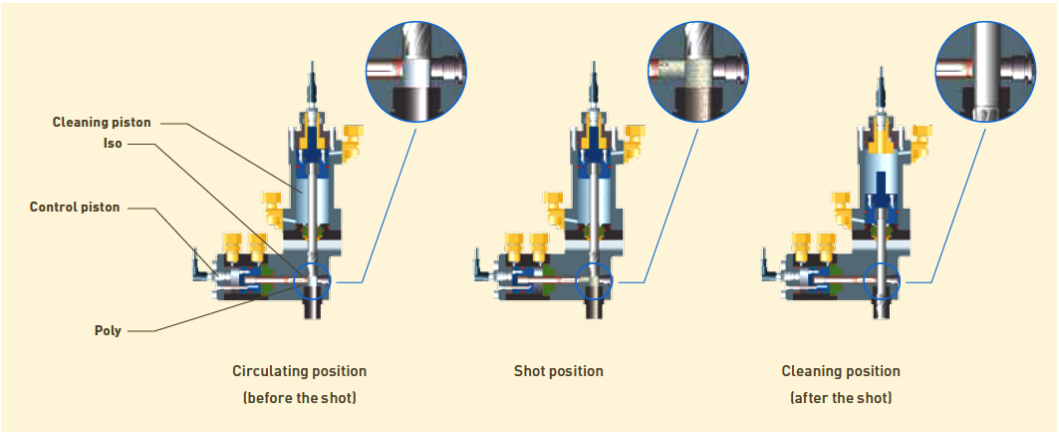


Figure 2.15: Transfer mixing head functional details (KraussMaffei)

KraussMaffei has several mixing heads for multi-component feed of which MK 3.5-5 UL-2KVV could be adapted, see Figure 2.14. This is a high pressure mixing head that contains a cleaning piston that is activated each cycle, avoiding the purge and clean interruption that low-pressure heads require. This head has then self-cleaning capabilities and has flexible processing of different materials. Figure 2.15 shows detail of several of the heads function stages.

Keymix systems also provides low pressure mixing heads capable of mixing 2 to 6 components that have automatic cleaning capabilities, direct metering in the component line, mixing rotor powered by servo motor, automatic pressure regulation, static-Roto static or dynamic mixing systems and possibility of heated versions. An example can be seen in Figure 2.16.

Frimo technologies with its MEL-series low-pressure mix heads with selective or permanent mixing of up to six components, with advanced mix servo-pistons for three components and automatic cleaning capabilities can be seen in Figure 2.17.

2.5.6 Side Feeders

Side feed extruders are one of the methods developed to combine raw materials with additives, such as calcium carbonate, talc, glass, cotton, mica, etc. These extruders are either single or twin screws that positively provide downstream addition with zero compression. They can either be of volumetric or gravimetric feed. A volumetric feeder is composed of a hopper, a discharge device and a controller. In simple terms it supplies a certain volume of material per time unit, while a gravimetric feeder is nothing more that a volumetric feeder with a weighing unit and a new control system. The general function of any of these systems is to supply material via the discharge output that is composed of helical screw(s), fed by material within a hopper that may have an agitator or vibrating cones to maintain flow and prevent bridging. A motor coupled to a gearbox is connected to the discharge screws and the speed is set via a controller. Material characteristics define the type of screw and its pitch: spiral screws with a solid center rod are used to feed powder and pellets; helical screws are used

Figure 2.16: Mixing head (Keymix)

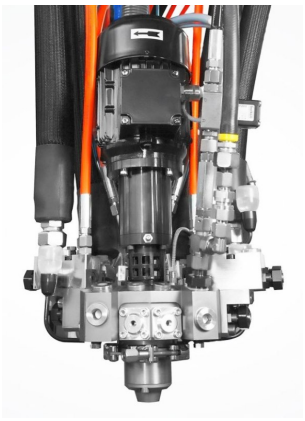


Figure 2.17: Mixing head MEL-series (Frimo)

for low density fluffy materials or fibres. Both designs can have square-pitch or variable-pitch flights. Delivered volume is related to the diameter of the screws, the chute and their relation. This means that feeder output require calibration for each material or product change running through the feeder [28][29].

As per Coperian, a company with over 100 years in feeding technology, "No single feeder design or feeder size can handle all the different material characteristics or throughput requirements". System deliverables, efficiency and operation are dependent on materials chemical and physical characteristics, its flow properties and screw design. Specifications to keep in mind are: particle size and shape; density; compressibility, cohesive strength, moisture, oil or fat content; and behavior under pressure atmospheric and loading conditions [28]. Figures 2.18 and 2.19 show examples of Coperion manufactured equipments that are double and single screw feeders, respectively.

Figure 2.18: Coperion Compact double screw feeder

Figure 2.19: Coperion K-Tron loss-in-weight feeder

3 Methodologies and material experiments

This chapter is divided in five sections after this introduction. The first section compiles details of the experimental setup for each of the manual extrusions and the adapted extrusion printer processes. The second section briefly addresses the outcomes of reactions between isocyanate and water. The third, fourth and fifth sections are the collection of the experimental data and processing windows of each of the selected binders, in a variety of residue contents and additions of agents that alter the thixotropic nature of the tested compounds. The sixth section is a comparison between all the tested materials and the selection of a compound that shows extruding capabilities for mechanical equipment in an industrial scale.

3.1 Experimental setup and methodology

The first experimental approach to this project relied on the notion and behavior of the materials being used. This process consisted of experimentally testing and trying out several formulations of binders with additions of residue in a variety of ratios. The first step was a pure trial and error approach, setting up a variety of mixtures with increasing ratios of residue and verifying which were manually extrudable. During these tests the following parameters were considered: viscosity before and after mixing; potting times before and after mixing; cure times before and after filler addition; and homogeneity.

These factors were important not only to be considered during the mixing and preparation stages but also had to be considered for the extrusion process due to the fact that the syringes selected had a tip diameter of approximately 2mm. This testing phase provided experience on how to maintain stability while producing the extruded samples. This attempts to reach data stability in between materials while confirming as theorized in the equation 1.1 with a starting point shown in equation 1.2 and controlled by the iterations represented in equation 1.3. The experiments performed showed that: residues need to be sifted to obtain homogeneous compounds, see Appendix B for sift details; mixing times of approximately 60s if possible, independently if the mixture is final or intermediate; for bi-component resins and low content of residue, mix residue into the component with the lowest viscosity; for bi-component resins and high content of residue, mix residue into both components; and verify final weight of mixture and adapt data to reflect loss of materials in mixing cups. The diagram in Figure 3.1 represents the complete method of experiments and the approval parameters that leads up to the final selection of the compound.

AM is a layer based process where size and form of the extruded string is critical. With the absence of equipment to quantify these parameters scientifically, methods were devoloped to simply show their relevance. The approval parameters used in methods 1 and 2 for Extrusion form (E_F) and Flow resistance (F_R) were based on the experimental sensitivity, comparing the desired output to a method that could translate them to quantifiable ranges. The form of the extruded compounds would then be classified as: Poor; Fair; and Good with details of this classification represented in Table 3.1.

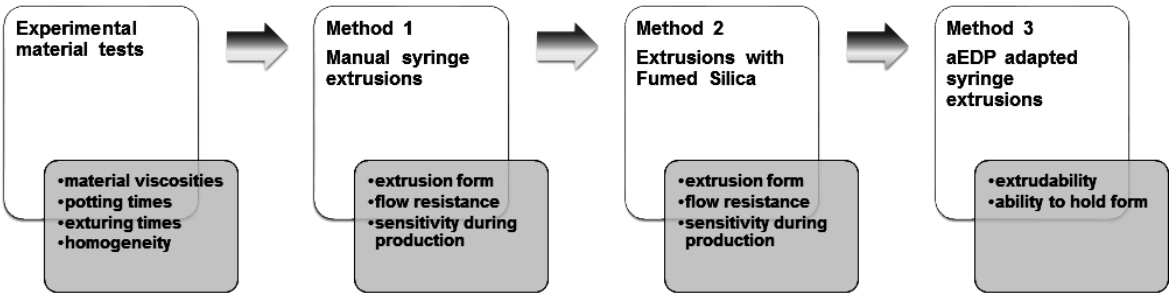


Figure 3.1: Diagram of complete test method

Table 3.1: Qualification parameters for Extrusion form (E_F)

Classification	Width variation	Form factor	Frequency of width variation in 1 cm
Poor	Min to Max > 50%	non-circular	
Fair	Min to Max < 35%	non-circular	> 3 in 1 cm
Good	Min to Max < 35%	circular	< 3 in 1 cm

In relation to the mixtures ability to be extruded, which is normally dependent on its viscosity, the classification would depend on a prototype system built for this purpose. The unit is built of: a support container that will accommodate a syringe, in a vertical position at a height that provides clearance from its base; a second container fixed to this base functioning as a guide; with a third that can be loaded with weight and presses down on the plunger. Figure 3.2 is a representation of this construction.

The compounds undergoing tests are loaded in syringes and placed vertically on the test equipment. A mass of 3 kilograms (kgs) is then applied to the plunger and values of time are recorded as well as the amount of milliliters on the syringe scale. The syring scale ranges from 0 to 5 ml and to obtain stabler results the considered range to read from for calculations was from 3 to 1 ml. Classification of the parameter mentioned in this report as flow resistance (F_R, a perception of viscosity), is then an approximate range and the result of the behavior of the sample in this test. Figure 3.3 represents the scale used to classify them.

Method 1 - Manual syringe extrusions

Each test was prepared by weighing the binders (Biresin G26, Pavistone 1K, Pavistone 2K) and the residues separately with a scale with 1 gram (g) resolution, see Appendix B for scale details. The residue was then incorporated into the cup containing the binder and mixed for approximately 60s with a wooden spatula. For cases were the binders were bi-component the less viscous component was preferably selected for filler addition, but for higher ratios of fillers both components had to incorporate fillers in order to obtain better mixture homogeneity. Weights of the cups/containers and the mixing accessories were accounted for and removed from the data within this project.

The mixed compound was then placed in a syringe (5 ml PiC solution, #21Gx1½") and extruded manually onto a plastic sheet that includes all of the extruded compounds for a specific binder. To be able to have some type of comparability between the extruded materials, the 10 cm extruded string was performed with an approximate target time of 10s. The

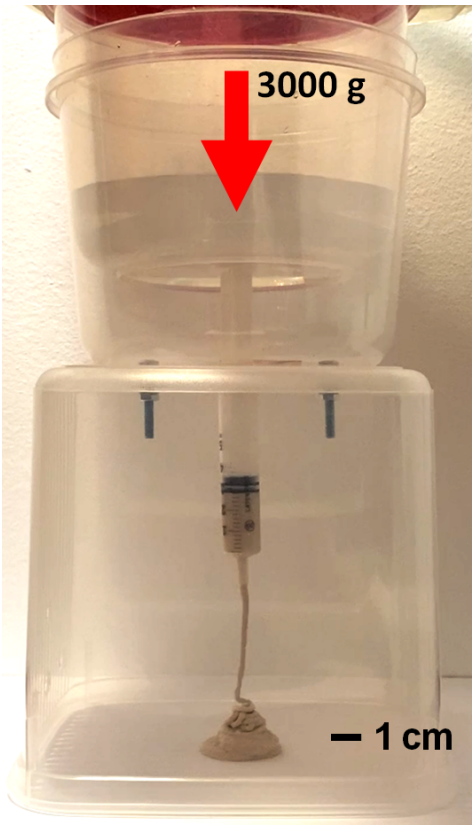


Figure 3.2: Prototype of flow resistance tester

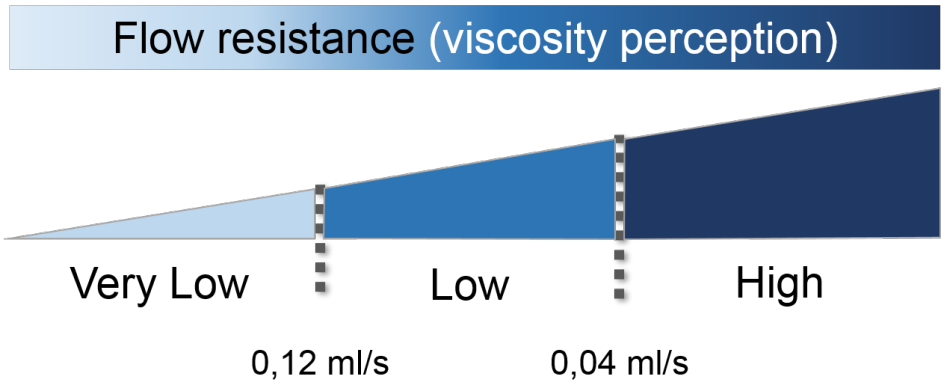


Figure 3.3: Scale of F_R (viscosity perception)

syringe was then refilled and left to cure in order to provide a solid test specimen. Enough material was produced to then still have a considerable amount within the mixing cup that would provide another solid testing specimen, if needed.

The resulting mixtures would then be classified as per Table 3.1 for extrusion form, per Figure 3.3 for flow resistance and the approved specimens would then undergo the extrusion process with the addition of Fumed Silica and the mechanized process using the aEDP.

Method 2 - Extrusions with addition of Fumed Silica

During the experimental phase of the manual extrusions, it was noticeable that increasing amounts of filler had an impact on the extrusions. The gain was the ability to hold form but the downside was the difficulty in mixing and extruding the materials. Homogeneous mixtures

were harder to obtain, and the curing was revealing a cake like outer skin consistency. The addition of an agent was considered and its goal to improve the handling of high viscosity mixtures, needed to be consistent with high loading, improve the caking effect, the blending and the processing. All these desired improvements led to the consideration of the addition of Fumed Silica, which once more would bring a trial and error testing approach.

Due to silicas lightweight characteristics, and the low impact on the weight of formulations, volume was now selected to measure its content for the mixtures. Several tests, with volume ratio variations, were performed and from those two scenarios were selected: for lower filler inclusions, silica would be added in the same volume as of the binder; and for high filler inclusions, silica would be added at half the volume of the binder. Nevertheless it was still weighed, its impact on the content calculated and summarized in Table 3.2.

Table 3.2: Silica weight parameter considerations

Silica volume	**Notation (Binder:Silica)**	**$\approx$ Silica weight (g)**	**Average Silica mixture impact (%)**
Equal to Binder	(1:1)	≈ 1.5	≤ 2.9
Half of Binder	(1:½)	≈ 0.75	≤ 0.9

For silica volumes equal to that of the Binder, the content will average 2.9%, which can be overlooked due to the fact that these mixtures normally contain lower filler content and are not the aim of this study. For silica volumes of half of the Binder, the content will average less than 0.9%, which we consider negligible, thus we no longer consider silicas mass in the formulations but only include it at the volume described so it provides the mixture with the desired characteristics.

The Fumed Silica used is manufactured by Wacker with part code HDK-N20 and its datasheet can be seen in Appendix C. This test consisted in redoing the tests from the previous section, starting with the mixtures with the lowest viscosity and incorporating silica with the same volume amount as of the binder to the mixture. As in the previous test method, the extruded 10 cm string would be performed in approximately 10s. Depending on the outcome of the tests the volume of the silica is then decreased or increased by 50% (in volume) in order to produce extrudable samples for analysis. The resulting mixtures would then be classified as per Table 3.1 for extrusion form, per Figure 3.3 for flow resistance and the approved specimens would then undergo the extrusion process in a mechanized manner in the aEDP.

Method 3 - aEDP adapted syringe extrusions

The aEDP, adapted extrusion process relies on the validation of specimens from the previous processes. The formulations are then remixed and extruded in the aEDP, represented in Figure 3.4. The printer used is a Roland Digital Group, with serial number ZBB5428, modified with a Marlin control unit, a motorized extrusion screw and an adapted syringe holder. The bottom plate had to be re-positioned to the right deposition height by exchanging the spring loaded screws for longer ones. The syringe piston holder was reworked to allow easy insertion while keeping a tight fit and the nut connecting the extruding screw and the piston holder was specifically made for this application. To extrude the strings of material the printer would require a written software, in G-Code, specifically developed for this project.

Several tests were performed, changing speeds, extrusion rates, deposition heights and different types of syringes. As the first step the syringe was selected and the choice fell on a 5 ml syringe from PiC solution with part number 21Gx1½", being the influencing factors for this

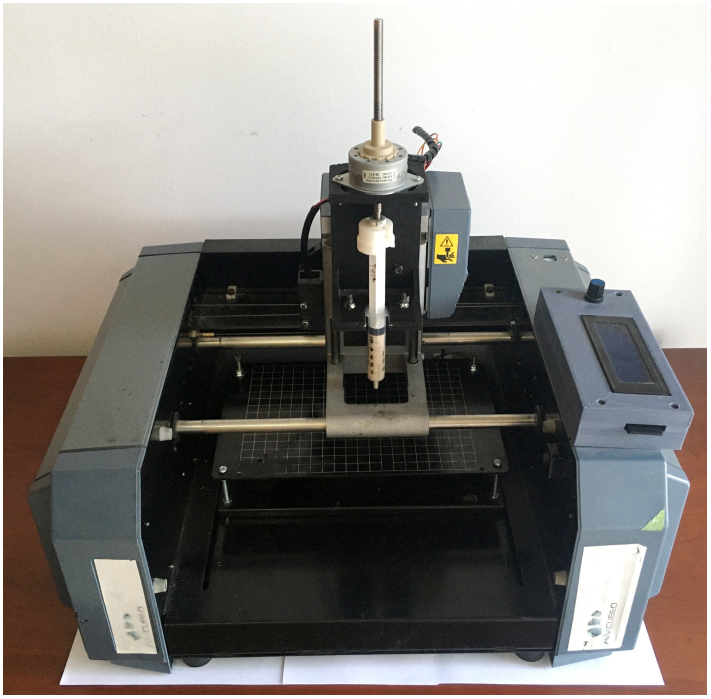


Figure 3.4: FDM adapted printer used for extrusion

selection: easy to source in quantity; its size would hold enough material to do several strings without refilling; fit the printer support; have enough mechanical strength to withstand mild torsion strengths applied to the plunger; tip size, allowing pastier materials to be extruded; and length, which is dependent on bottom plate support height and length of extrusion screw.

The physical height of the bottom support plate was then fixed at a distance of 1.5mm from the syringe tip, when assembled on its support, with the z-axis value at its lowest possible dimension. This would guarantee that even if a mistake happened during extrusion, the unit would not suffer any damage from conflict of moving parts. Note that z-axis dimensions in the software then require the addition of 1.5mm to obtain the real height of extrusion of the strings that were extruded. The amount of pressure applied to the plunger, even when constant, revealed that obtaining an even diameter string was challenging. The extrusion would have excessive material at some point or no material at all. This is mostly due to the curing process, which is expansive in volume and challenges an extrusion in a controlled manner. The gaseous accumulation from the curing, creates an extra parameter that cannot be controlled directly with precision. It was clear that a comprise would have to be considered and the machine code was then written to include a push-pull effect on the extruder to minimize this effect. The speed was selected and the deposition height would then vary from string to string. The extruded strings had now a better form, but still inconsistent, some times not having enough extruded material and in other cases excessive material being extruded due to the inertial accumulation of pressure within the syringe in addition to the stabilized extruding forces applied equally over the complete extrusion process. With this in mind the G-code was then developed, see Appendix D for details, for the printer to provide the required extruded strings, placed together in a sheet of plastic for each binder material, as follows:

- String 1, extruded at a height of 1,5mm with approximately 90mm in length;
- String 2, extruded at a height of 2mm with approximately 90mm in length. Extruded right after the extrusion of string 1;
- String 3, extruded at a height of 2,5mm with approximately 90mm in length. Extruded right after the extrusion of string 2;

- String 4, extruded at a height of 3mm with approximately 90mm in length. Extruded after a 2 minute wait after the extrusion of string 3.

The program starts at (0,y) being y different for each string. The extrusion nozzle is then moved 8mm in the x-axis direction, while priming the extruder and lowering the z-axis with intent to create adhesion of the string to the plastic support sheet. The nozzle is the moved once more another 2mm in the x-axis direction to the start point of the desired string extrusion and the z-axis is set to the desired extrusion height. The extrusion of the string is performed with a push-pull motion of the extruder (syringe plunger) and stops when the x-axis has reached 100mm. The extruder is then retracted to avoid material spill as it shifts sideways 5mm and is then positioned close to the start point of the next string to be extruded. All strings have different programs so they can be timed and started when deemed necessary. They are also sequential and position dependent, meaning that they have to be run in order. The fact that different programs are used, in this experiment, is due to the knowledge drawn in the pre-testing phase with the adapted printer. Handling the mixtures and prepping the samples had a time factor that was different for all materials, thus having separate programs helped during the extruding phases of the experiment.

3.2 Chemical reactions and bubble formations

The reaction involved between isocyanate and water in polyurethane (PU) foam production is represented in Figure 3.5 .

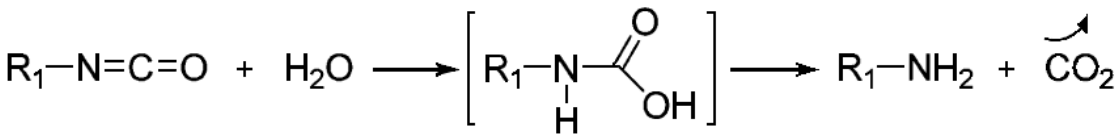


Figure 3.5: Reactions with water used in Polyurethane Chemistry [30]

The reaction with water generates unstable carbamic acid, that decomposes into an amine and gaseous carbon dioxide, quickly producing polyurea segments that result in mixture expansion which in turn provide the foam with its swollen open-cell structure [30] [31].

The release of carbon dioxide and the expansion of mixtures are factors for the unstable production of samples that underwent the sample characterization tests for tensile, hardness and Micro-CT scans, addressed further on in this document.

In flexible polyurethane foams, the fillers promote an increase in density and resistance to compression. However, they reduce the resiliency and contribute to the increase in permanent deformation [32].

3.3 Biresin G26 experiments

Manual extrusions with & without fumed silica incorporation

The mixed compounds were placed in syringes and extruded manually onto a plastic sheet, which includes all of the extruded specimens as can be seen in Figure 3.6. For the compounds extruded with addition of fumed Silica the specimens can be seen in Figure 3.7.

With the analysis of the extruded strings and notes taken during tests, data were then compiled into Table 3.3. The approved mixtures for the aEDP process need as minimum

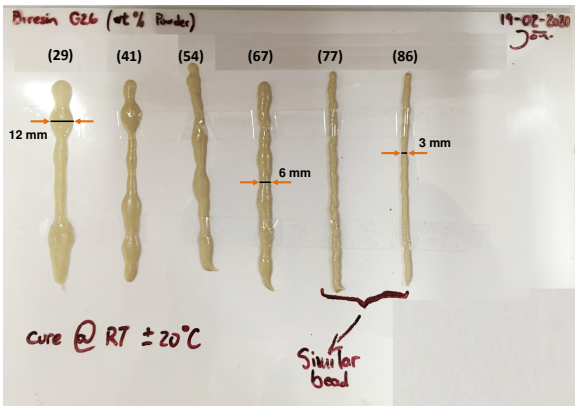


Figure 3.6: Manual Syringe extrusions for Biresin G26

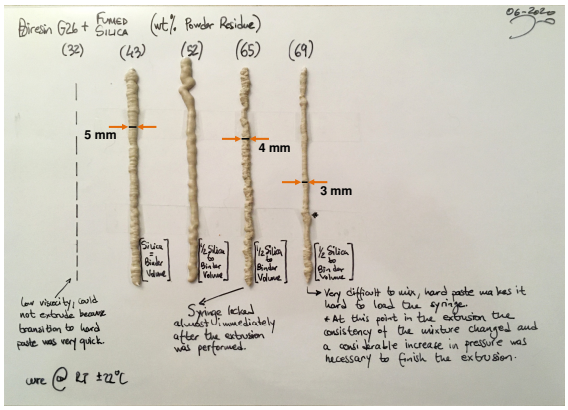


Figure 3.7: Manual Syringe extrusions for Biresin G26 + Fumed Silica

requirements, high flow resistance (F_R), fair extrusion form (E_F) and approved experimental sensibility being the results presented in the last column of this table.

After the curing process was completed, the specimens were then carefully removed from their syringe en-casings and of the plastic containers. Details of these samples can be seen in Appendix E, Figure E.1 and for the samples with addition of fumed Silica in Figure E.2.

aEDP extrusions with & without fumed silica

These extrusions were performed as explained in the experimental setup, apart from the change of the clock start. Due to the short potting time of Biresin G26, the clock would now be started as soon as the mixing of the constituents was initiated. The compiled timed values in Table 3.4 are approximate, they provide information on the mixture behaviors in relation to their potting times, cure times and the changes in extrusion behaviors related to the incorporation of filler content and fumed silica. These time factors will be relevant for residence times within the equipment and are strictly related to the amount of fillers.

For mixtures without the incorporation of fumed silica the viscosity is very low, making it difficult to load and maintain the syringe with material. The continuous loss of material through the tip also makes it difficult to insert it in the extruder and perform a clean string of material on the deposition sheet. During the extrusion process, the mixture continues to cure and the increase in viscosity improves the string form. Although positive, this fact changes the extrusion control, due to the fact that the transition to hard material is quick and the plunger begins to twist under the extruding pressure. The result sheet of the extrusions can be seen in Figure 3.8. No other residue ratios were tested due to the difficulty of performing this test, which would rule out the residue test of 86%. The preparation of higher residue contents are not feasible with the process selected because the mixtures cure before they can be extruded successfully after loading the syringes.

For mixtures with added fumed silica the viscosity is good and during the extrusion process the string form improves, due to material cure, but the transition to hard material is quick and the plunger begins to twist under the extruding pressure. The result sheet of the extrusion can be seen in Figure 3.9. No other residue ratios were tested due to the difficulty of performing this test. This ruled out the residue tests for 43% and 52% because the viscosities are too low. It also ruled out the residue tests for 69% because the viscosity is too high. The preparation of higher residue contents are not feasible with this process because the mixtures cure before they can be extruded successfully after loading them in the syringes. For cases of lower ratio filler contents, mixtures have lower viscosities and as proven earlier those do not provide good test outcomes.

Table 3.3: Biresin G26 experiments and approvals for aEDP

Experiment type	**Binder [A] Silica [B] (ratio [A:B])**	**Residue (%)**	**Extrusion form** (E_F)	**Flow resistance** (F_R)	**aEDP Approved**
Manual Extrusion	–	29	Poor	Very Low	No
		41	Poor	Very Low	No
		54	Poor	Low	No
		67	Poor	Low	No
		77	Fair	High	**Yes**
		86	Good	High	**Yes**
Manual Extrusion	(1:1)	32	–	Very Low	No [a]
		43	Fair	High	**Yes** [b]
		52	Fair	Low	**Yes** [b]
	(1:½)	65	Fair	High	**Yes** [c]
		69	Good	High	**Yes** [c]

Extrusion form (E_F):
- Poor: width (min-max)>50%; non-circular form.
- Fair: width (min-max)<35%; non-circular form; width frequency variation >3/cm.
- Good: width (min-max)<35%; circular form; width frequency variation <3/cm;
Flow resistance (F_R):
- Very low: F_R< 0,12ml/s;
- Low: 0,12ml/s <F_R< 0,04ml/s;
- High: F_R> 0,04ml/s.

[a] Liquid mixture that could not be extruded. Transition to a hardened paste was very sudden and could not be considered for the aEDP test.
[b] The mixture has a pasty consistency that is good for loading the syringes. Preparation must be fast for time is a constraint, there is a fast transition to a very hard paste which reveals extruding issues.
[c] The mixture is a hard to mix paste that is challenging to load the syringes. Preparation must be fast for time is a constraint, there is a fast transition to a very hard paste which reveals extruding challenges.

Table 3.4: Biresin G26 compiled aEDP string START times

Test material [A]	Residue (%wt)	Fumed Silica [B]) (ratio [A:B])	String "x"	Start Time mm:ss
Biresin G26	77	0	string 1	2:58 [a]
			string 2	3:57 [b]
			string 3	lock [c]
	86	–	–	– [d]
Biresin G26	65	(1:½)	string 1	2:27 [e]
			string 2	3:19 [f]
			string 3	3:57 [b]
			string 4	4:38 [g]

[a] Liquid mixture.
[b] Plunger starts to twist due to hardening of mixture.
[c] At 5:00 the plunger was locked, thus no string could be extruded.
[d] Mixture was not achievable in time to perform extrusion.
[e] Good viscosity for deposition of string.
[f] Lack of contact in the beginning of the extrusion. String did not adhere to sheet.
[g] String is just twirling right at the output tip of the syringe. Test stopped at about 20% of the total path with the syringe locked at 5:11.

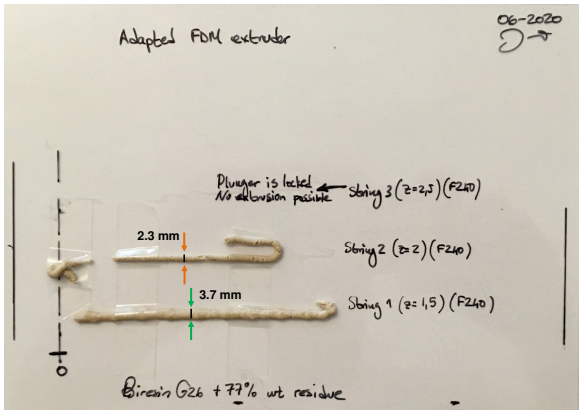


Figure 3.8: aEDP strings for Biresin G26 with Residue @ 77% wt

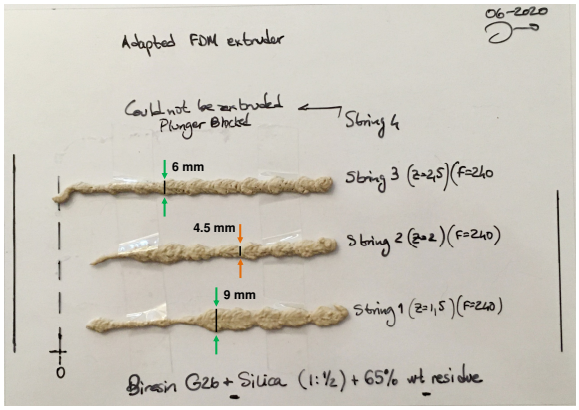


Figure 3.9: aEDP strings for Biresin G26 with Fumed Silica and Residue @ 65% wt

3.3.1 Processing windows for Biresin G26

The data of the extrusions preformed with manual force have been summarized and can be seen in Figure 3.10. The top half of this figure presents the range of mixtures performed, relating them to the content residue on the top axis scale ranging from 29 to 86%. The highlighted section in blue represents the processing window of the approved range of compounds for the next processing method. The bottom half of the figure relates the mixtures to the flow resistance, which only confirms the obvious that higher ratio incorporations of residue produce better form and stable extrusions of these compounds. The data collection sheets populated during the experimental phase can be verified in Appendix H1 for mixtures without incorporation of fumed silica and in Appendix H4 for mixtures with incorporation of fumed silica.

For the case of the strings extruded via the aEDP machine the approach was different

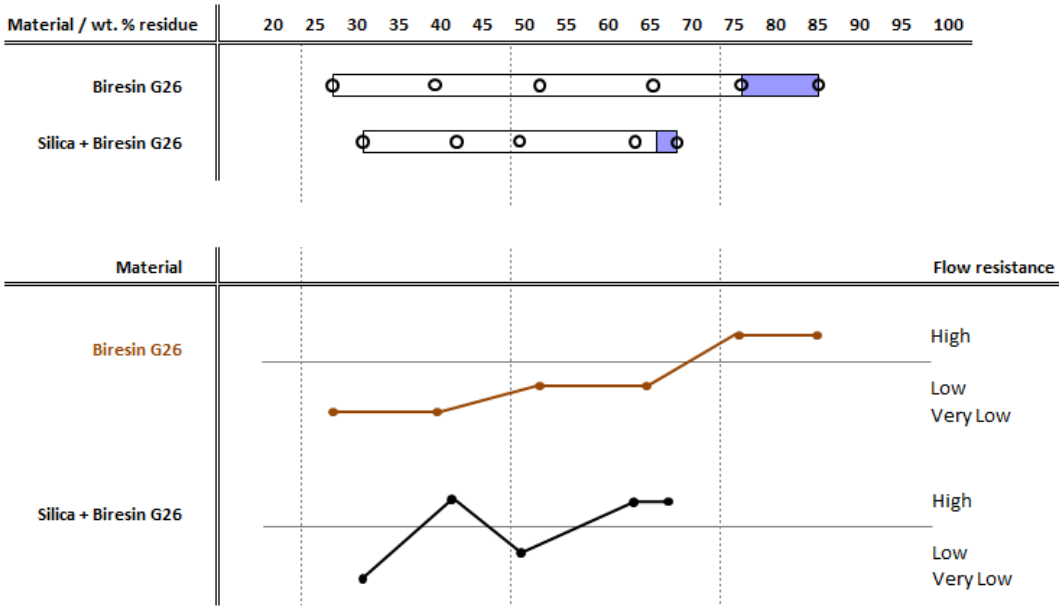


Figure 3.10: Graph of Biresin G26 manual extrusions

due to the diversity of results. Considering that the extruding pressures were kept the same, and keeping in mind that form and extrudability were the goals of this project, the choice of the width of the extruded string and the form on the deposition sheet would be the selecting parameters. We can verify that string 4 could not be extruded for any of the mixtures. For samples with added Silica, widths vary significantly with the exception of the second string of the mixture with 77% wt filler, that presents a good round form. All other extrusions present uneven, flattened or non-existent forms.

3.4 Pavistone 1K experiments

Manual extrusions with & without fumed silica incorporation

The mixed compounds were placed in syringes and extruded manually onto a plastic sheet, which includes all of the extruded specimens as can be seen in Figure 3.11. For the compounds extruded with addition of fumed Silica the specimens can be seen in Figure 3.12.

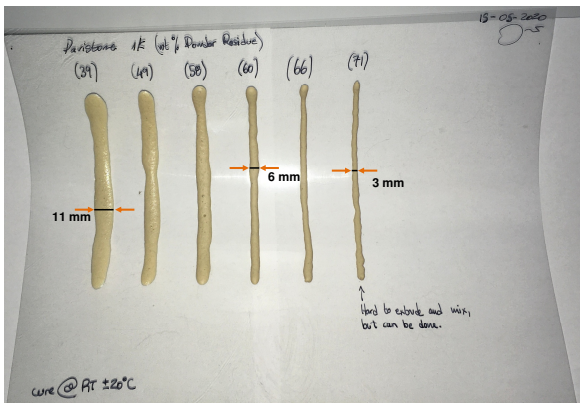


Figure 3.11: Manual Syringe extrusions for Pavistone 1K

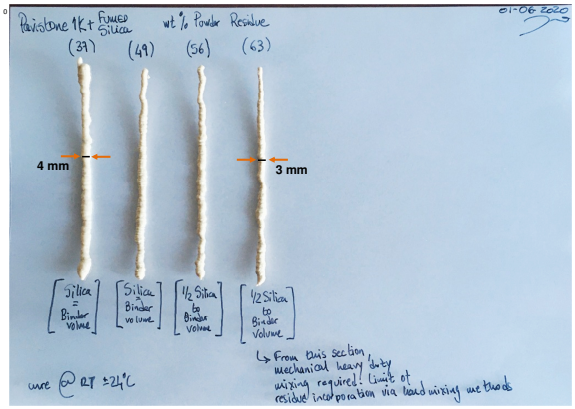


Figure 3.12: Manual Syringe extrusions for Pavistone 1K + Fumed Silica

With the analysis of the extruded strings and notes taken during extrusions, data were

compiled into Table 3.5. The approved mixtures for the aEDP process need as minimum requirements, High flow resistance (F_R), Fair Extrusion form (E_F) and approved experimental sensibility being the results presented in the last column of this table.

Table 3.5: Pavistone 1K experiments and approvals for aEDP

Experiment type	**Binder [A] Silica [B] (ratio [A:B])**	**Residue (%)**	**Extrusion form** (E_F)	**Flow resistance** (F_R)	**aEDP Approved**
Manual Extrusion	–	39	Poor	Very Low	No
		49	Poor	Very Low	No
		58	Poor	Very Low	No
		60	Fair	Low	No
		66	Good	High	**Yes**
		71	Good	High	**Yes**
Manual Extrusion	(1:1)	37	Fair	High	**Yes**
		49	Good	High	**Yes**
	(1:½)	56	Good	High	**Yes**
		63	Good	High	**Yes**

Extruion form (E_F):
- Poor: width (min-max)>50%; non-circular form.
- Fair: width (min-max)<35%; non-circular form; width frequency variation >3/cm.
- Good: width (min-max)<35%; circular form; width frequency variation <3/cm;
Flow resistance (F_R):
- Very low: F_R< 0,12 ml/s;
- Low: 0,12 ml/s <F_R< 0,04 ml/s;
- High: F_R> 0,04 ml/s.

After the curing process was completed, specimens were then carefully removed from their syringe en-casings and of the plastic containers. Details of these samples can be seen in Appendix F, Figure F.1 and for the samples with addition of fumed Silica in Figure F.2.

aEDP extrusions with & without fumed silica

These extrusions were performed as explained in the experimental setup and starting times for the extrusions can be seen in data compiled in Table 3.6. The values in this table intend to add information on the mixture behaviors in relation to their potting times, cure times and the changes in extrusion behaviors related to the incorporation of filler content and fumed silica. These time factors will be relevant for residence times within the equipment and are strictly related to the amount of fillers.

Viscosities of mixtures with and without the incorporation of fumed silica are very high and pasty. This meant that loading the syringes was challenging and it is advisable to use hard recipients for these mixtures. The result sheet for mixtures without additions of fumed silica for 66% wt filler content can be seen in Figure 3.13 and for 70% wt filler in Figure 3.14. High incorporation of fillers improve form factor, but makes it very difficult to press the material into the syringes. Strings produced during the extrusion had a reasonable form, but the growth in volume produced during curing has increased the size of the stings, thus making them spread on the sheet. Another fact verifiable when the cure was finalized is that the plastic

Table 3.6: Pavistone 1K compiled aEDP string START times

Test material [A]	Residue (%wt)	Fumed Silica [B]) (ratio [A:B])	String "x"	Start Time mm:ss
Pavistone 1K	66	–	string 1	3:10 [a]
			string 2	4:05 [a]
			string 3	4:55 [a]
			string 4	8:12 [a]
	71	–	string 1	3:45 [b]
			string 2	4:39 [b]
			string 3	5:35 [b]
			string 4	8:05 [b]
Pavistone 1K	37	(1:1)	string 1	4:40 [b]
			string 2	5:31 [b]
			string 3	6:28 [b]
			string 4	8:48 [b]
	49	(1:1)	string 1	3:40 [b]
			string 2	4:31 [b]
			string 3	5:27 [b]
			string 4	7:45 [b]
	56	(1:½)	string 1	4:14 [c]
			string 2	5:10 [c]
			string 3	6:05 [c]
			string 4	8:17 [c]
	63	(1:½)	string 1	3:24 [d]
			string 2	4:18 [d]
			string 3	4:57 [d]
			string 4	7:28 [d]

[a] Soft paste mixtures that extrude but still don't hold shape.
[b] Hard paste mixture.
[c] Soft paste mixtures that extrude and hold shape.
[d] Hard paste mixture, needs heavy duty mixing.

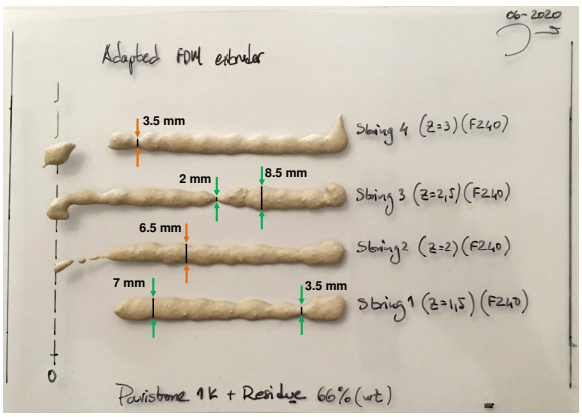


Figure 3.13: aEDP strings for Pavistone 1K with Residue @ 66% wt

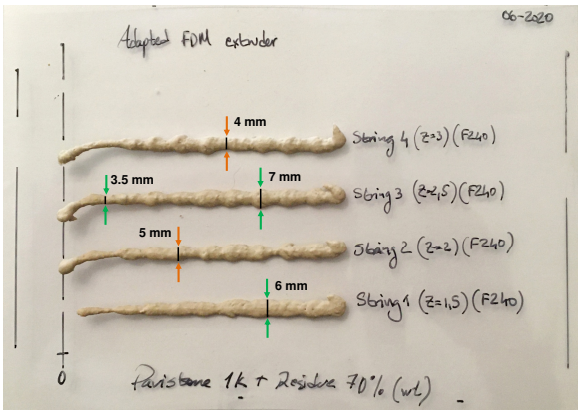


Figure 3.14: aEDP strings for Pavistone 1K with Residue @ 70% wt

sheets have warped, meaning that during the cure process these mixtures have suffered a reduction in volume.

For mixtures with the incorporation of fumed silica the result sheet for the mix with 37% wt filler can be seen in Figure 3.15, in Figure 3.16 for 49% wt filler , in Figure 3.17 for 56% wt filler and in Figure 3.18 for 63% wt filler. The strings produced during the extrusion had a reasonable form, but the growth in volume produced during curing has increased the size of the strings, thus making them spread on the sheet. Another fact verifiable when the cure was finalized is that the plastic sheets have warped, meaning that during curing there has been a reduction in size of the samples.

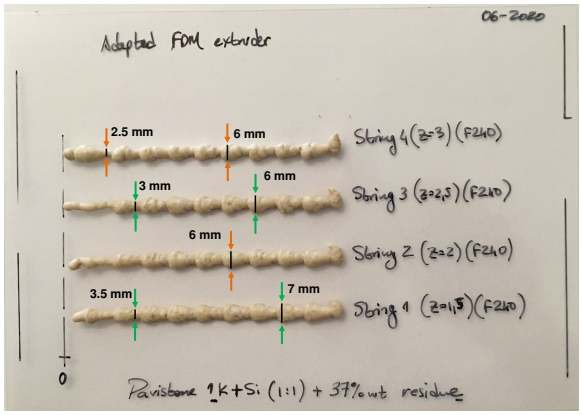


Figure 3.15: aEDP strings for Pavistone 1K with Fumed Silica and Residue @ 37% wt

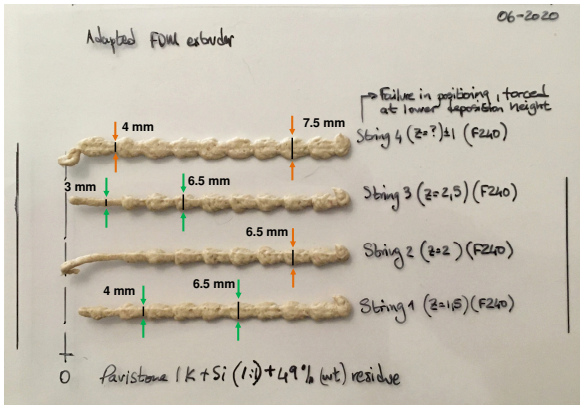


Figure 3.16: aEDP strings for Pavistone 1K with Fumed Silica and Residue @ 49% wt

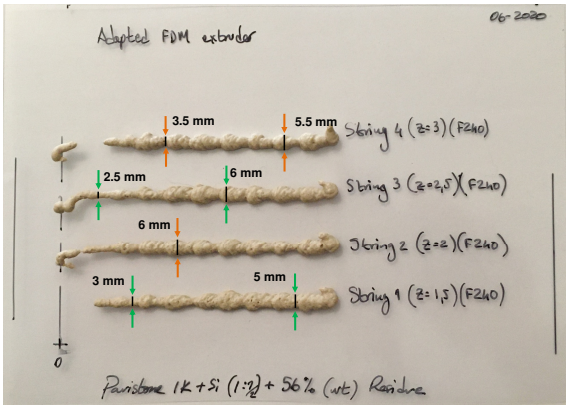


Figure 3.17: aEDP strings for Pavistone 1K with Fumed Silica and Residue @ 56% wt

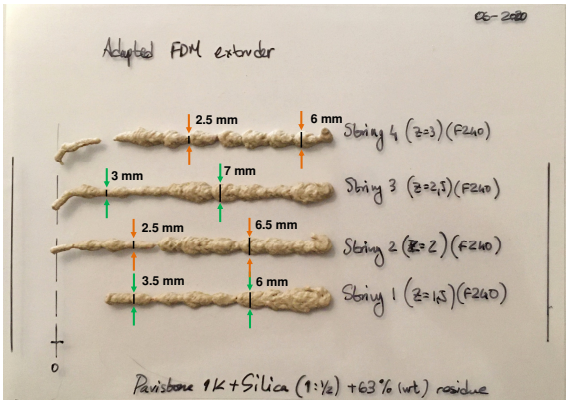


Figure 3.18: aEDP strings for Pavistone 1K with Fumed Silica and Residue @ 63% wt

3.4.1 Processing windows for Pavistone 1K

Data of the extrusions preformed with manual force have been summarized and can be seen in Figure 3.19. The top half of this figure presents the range of mixtures performed, relating them to the content residue on the top axis scale ranging from 37 to 71%. The highlighted section in blue, represents the processing window of the approved range of compounds for the next processing method. The bottom half of the figure relates the mixtures to the flow resistance, which only confirms the obvious that higher ratio incorporation's of fillers produce better form and stable extrusions of these compounds and that fumed Silica did alter the thixotropy of the compound. The data collection sheets populated during the experimental phase can be verified in Appendix H2 for mixtures without incorporation of fumed silica and in Appendix H5 for mixtures with incorporation of fumed silica.

The analysis method is similar to the one performed for Biresin G26, width of the extruded

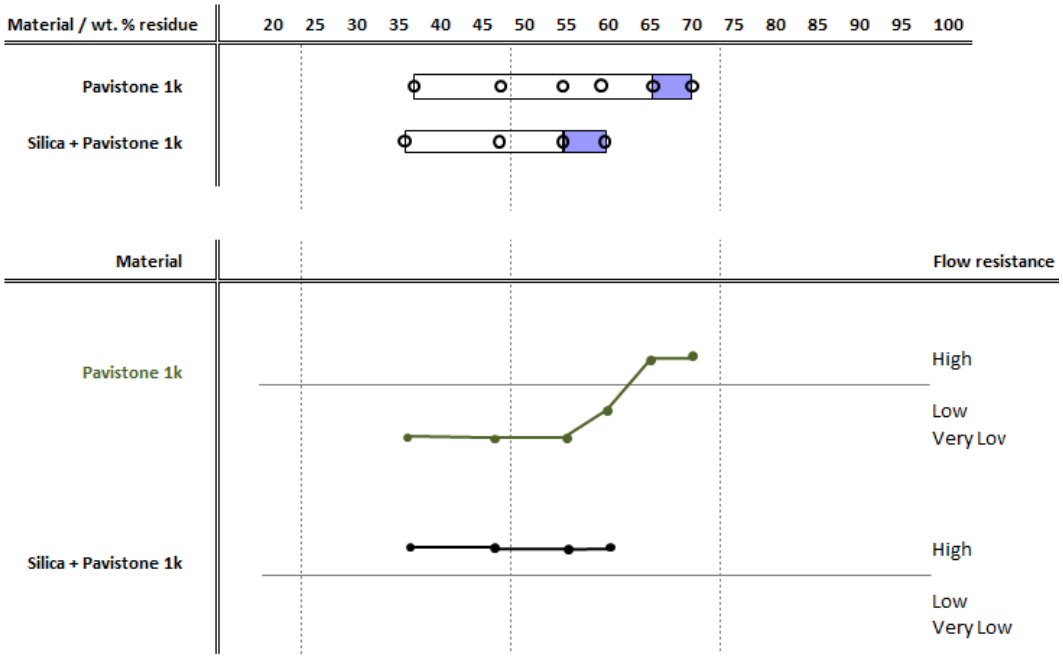


Figure 3.19: Graph of Pavistone 1K manual extrusions

string and form on the deposition sheet would be the selecting parameters. For Pavistone 1K without silica incorporation it is noticeable that were there is width stability there is no form and when there is some sort of form there is a big variation in width along the extrusion. Discarded mixtures due to flattened form have added silica and fillers at 37% wt and 49% wt. Discarded mixtures due to flattened form and inconsistent widths have added silica and fillers at 56% wt and 63% wt. String 4 was not extrudable for any of the mixtures. For cases of Silica incorporation widths vary significantly. The only string that presents a round form is string 2 which has fillers of 77% wt. All other extrusions present uneven, flattened or non-existent forms.

3.5 Pavistone 2K experiments

The mixed compounds were placed in syringes and extruded manually onto a plastic sheet, which includes all of the extruded specimens as can be seen in Figure 3.20. For the compounds extruded with addition of fumed Silica the specimens can be seen in Figure 3.21.

With the analysis of the extruded strings and notes taken during the extrusions, data were compiled into Table 3.7. Approved mixtures for the aEDP process need as minimum requirements, High flow resistance (F_R), Fair Extrusion form (E_F) and approved experimental sensibility being the results presented in the last column of this table.

After the curing process was completed, the specimens were then carefully removed from their plastic containers but no actions were taken on the syringes due to the fact that most would not provide usable specimens after removal of the syringe casing. Details of these samples can be seen in Appendix G, Figure G.1 and for the samples with addition of fumed Silica in Figure G.2.

aEDP extrusions with & without fumed silica

These extrusions were performed as explained in the experimental setup and the starting times for the extrusions can be seen in the data compiled in Table 3.8. The values in this

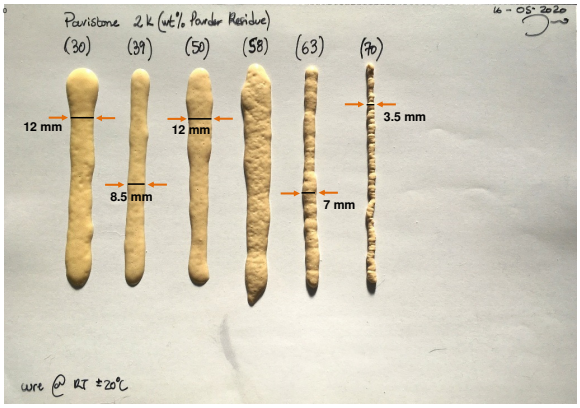


Figure 3.20: Manual Syringe extrusions for Pavistone 2K

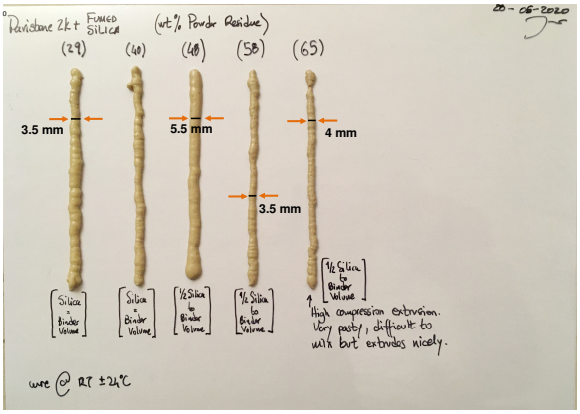


Figure 3.21: Manual Syringe extrusions for Pavistone 2K + Fumed Silica

Table 3.7: Pavistone 2K experiments and approvals for aEDP

Experiment type	**Binder [A] Silica [B] (ratio [A:B])**	**Residue (%)**	**Extrusion form** (E_F)	**Flow resistance** (F_R)	**aEDP Approved**
Manual Extrusion	–	30	Poor	Very Low	No
		39	Poor	Very Low	No
		50	Poor	Very Low	No
		58	Poor	High	No
		63	Fair	High	**Yes**
		70	Good	High	**Yes**
Manual Extrusion	(1:1)	29	Fair	High	**Yes**
		40	Fair	High	**Yes**
	(1:½)	48	Fair	Very Low	**Yes** [a]
		58	Fair	High	**Yes**
		65	Good	High	**Yes**

Extruion form (E_F):
- Poor: width (min-max)>50%; non-circular form.
- Fair: width (min-max)<35%; non-circular form; width frequency variation >3/cm.
- Good: width (min-max)<35%; circular form; width frequency variation <3/cm;
Flow resistance (F_R):
- Very low: F_R< 0,12 ml/s;
- Low: 0,12 ml/s <F_R< 0,04 ml/s;
- High: F_R> 0,04 ml/s.

[a] Although the mixture has very low flow resistance it may still produce a good extruded string and for that reason it was considered for the aEDP tests.

table intend to add information on the mixture behaviors in relation to their potting times, cure times and the changes in extrusion behaviors related to the incorporation of filler and fumed silica content. These time factors will be relevant for residence times within the equipment and are strictly related to the amount of fillers.

Mixture viscosity without incorporation of fumed silica was high and pasty, meaning that loading the syringes was challenging. The results of these extrusions can be seen in Figure 3.22 for a 63% wt filler and in Figure 3.23 for a 70% wt filler. Note that the figures show string

Table 3.8: Pavistone 2K compiled aEDP string START times

Test material [A]	Fumed Silica [B] (ratio [A:B])	Residue (%wt)	String "x"	Start Time mm:ss
Pavistone 2K	–	63	string 1	3:37
			string 2	4:32
			string 3	5:15
			string 4	7:22
		70	string 1	3:46 [b]
			string 2	4:46 [b]
			string 3	5:27 [b]
			string 4	7:58 [b]
Pavistone 2K	(1:1)	29	string 1	4:50 [a]
			string 2	5:35 [a]
			string 3	6:30 [a]
			string 4	8:30 [a]
		40	string 1	3:15 [a]
			string 2	4:10 [a]
			string 3	5:10 [a]
			string 4	7:25 [a]
	(1:½)	48	string 1	3:25 [a]
			string 2	4:10 [a]
			string 3	5:00 [a]
			string 4	7:00 [a]
		58	string 1	3:15
			string 2	3:57
			string 3	4:34
			string 4	7:10
		65	string 1	2:43 [b]
			string 2	3:37 [b]
			string 3	4:23 [b]
			string 4	6:56 [b]

[a] Liquid mixtures.
[b] Hard paste mixture.

forms that do not comply with the acceptable string forms stated earlier, from the experimental phase, and that is explained by the loss in form, due to the volume growth while curing after deposition, thus producing the results shown. For mixtures with incorporation of fumed silica, viscosity of mixtures increased with fillers at higher ratios and hard recipients for mixing are recommended. Results of these extrusions can be seen in Figure 3.24 for 29% wt filler, in Figure 3.25 for 40% wt filler, in Figure 3.26 for 48% wt filler, in Figure 3.27 for 58% wt filler and in Figure 3.28 for a 65% wt filler.

3.5.1 Processing windows for Pavistone 2K

Data of the extrusions preformed with manual force have been summarized and represented in Figure 3.29. The top half of this figure presents the range of mixtures performed, relating them to the content residue on the top axis scale ranging from 29 to 70%. The highlighted section in blue, represents the processing window of the approved range of compounds for

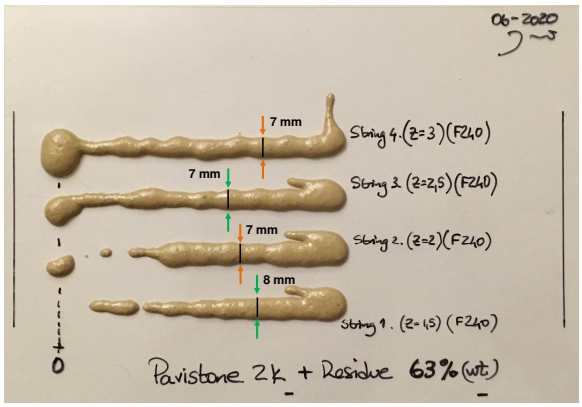


Figure 3.22: aEDP strings for Pavistone 2K with Residue @ 63% wt

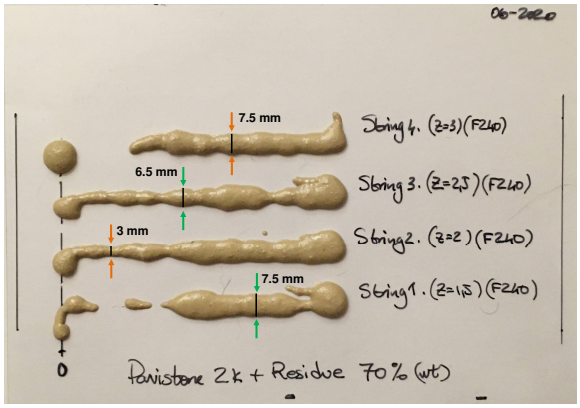


Figure 3.23: aEDP strings for Pavistone 2K with Residue @ 70% wt

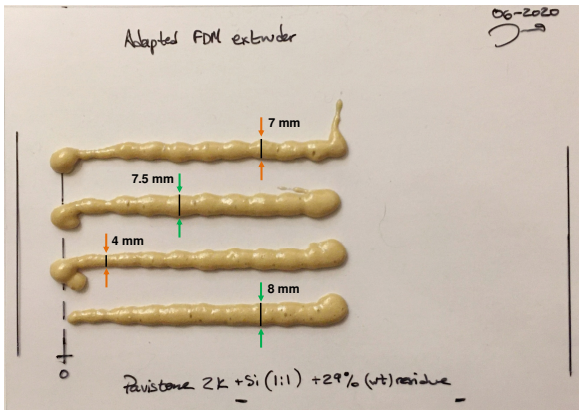


Figure 3.24: aEDP strings for Pavistone 2K with Fumed Silica and Residue @ 29% wt

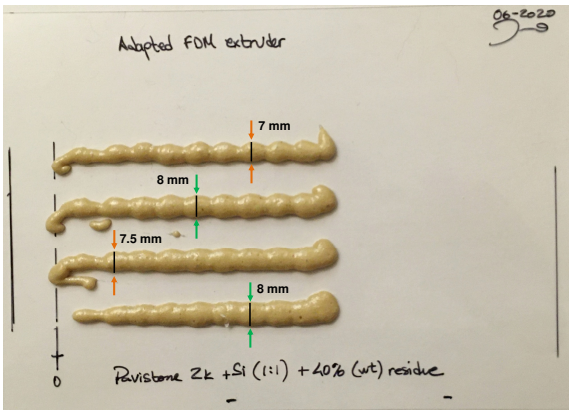


Figure 3.25: aEDP strings for Pavistone 2K with Fumed Silica and Residue @ 40% wt

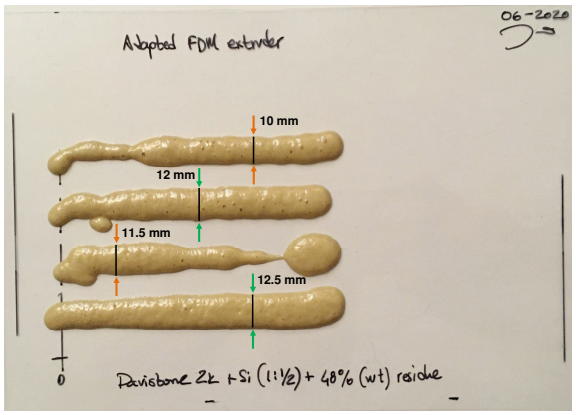


Figure 3.26: aEDP strings for Pavistone 2K with Fumed Silica and Residue @ 48% wt

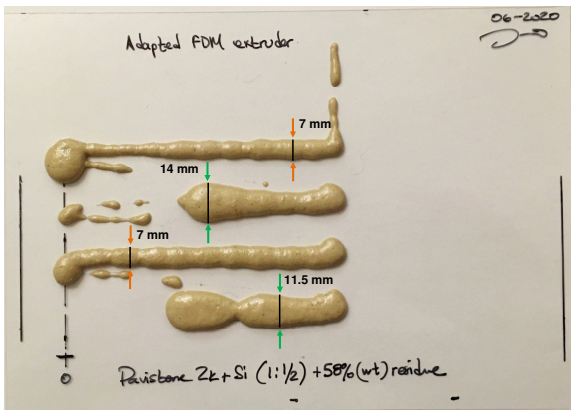


Figure 3.27: aEDP strings for Pavistone 2K with Fumed Silica and Residue @ 58% wt

the next processing method. The bottom half of the figure relates the mixtures to the flow resistance, which only confirms the obvious that higher ratio incorporation's of filler produce better form and stable extrusions of these compounds. Data collection sheets populated during the experimental phase can be verified in Appendix H3 for mixtures without incorporation of fumed silica and in Appendix H6 for mixtures with incorporation of fumed silica.

The analysis method is similar to the one performed for Pavistone 1K, width of the extruded string and form on the deposition sheet would be the selecting parameters.

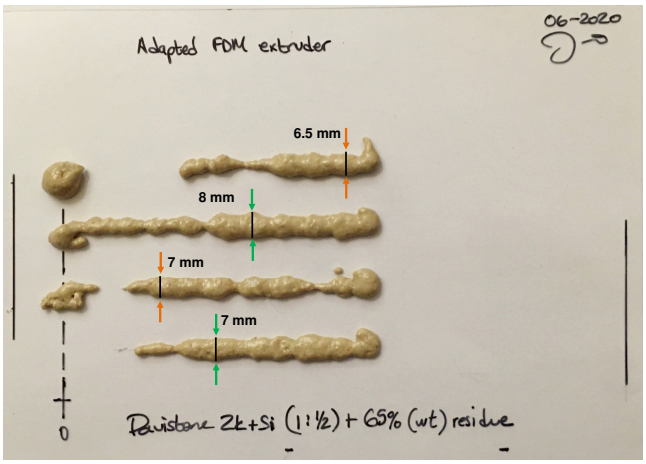


Figure 3.28: aEDP strings for Pavistone 2K with Fumed Silica and Residue @ 65% wt

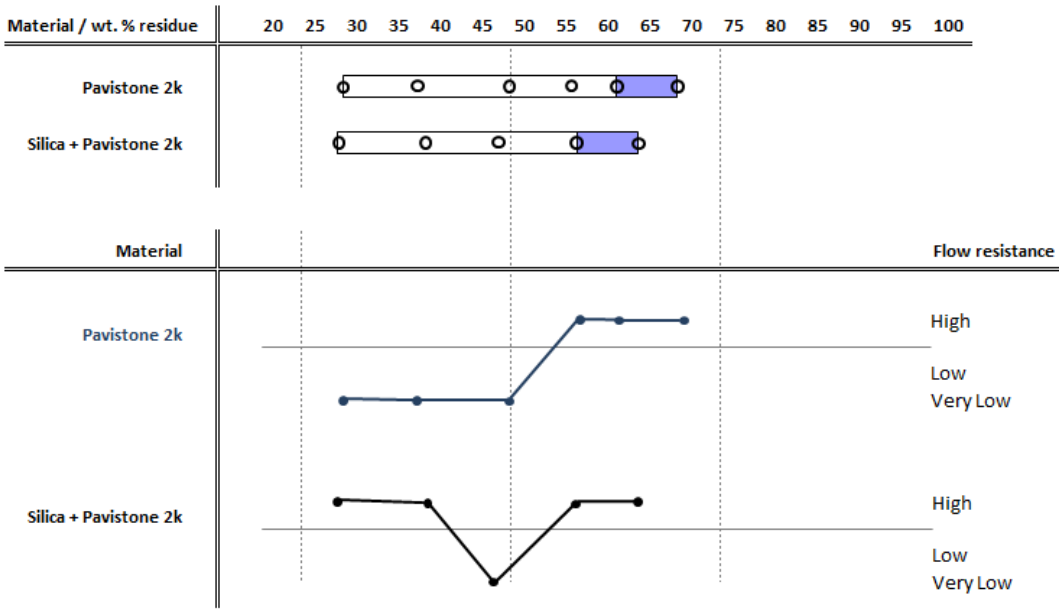


Figure 3.29: Graph of Pavistone 2K manual extrusions

All mixtures without addition of silica have been discarded due to inconsistent form. Although the mixture with 63% wt filler has a stable form throughout its extrusion it is not round.

All mixtures with added silica have been discarded due to inconsistent widths.

3.6 Summary and material selection

Analysis of the results and experience gained from the execution of these experiments reveal that low potting times and pastier mixtures provide good extrusions with acceptable form. Comparative values can be verified in Appendix I which is a summary of the manual extrusion experiments relating filler incorporation to the flow resistance. This also provides processing windows of the mixed materials where the highlighted blue sections are the windows of approved mixtures that deliver the necessary outcome for approval for a next stage test.

Low potting times do however present challenges and require extrusion precision and mixing efficiency to be able to provide a homogeneous output. High viscosity mixtures are in fact positive because very high filler content is one of this projects main goals, but this

also requires efficient mixing systems. All materials that have expansion during curing are not suitable for outcomes that require defined extrusion forms but do however present excellent possibilities as candidates for material functions such as fillers, gasket type materials or porous materials that require absorption or being light-weight as a material function.

With all of this in mind, we can say that Pavistone 1K and Pavistone 2K are not suitable for additive manufacturing processes that require defined form. This is due to their growth in volume which is a consequence of their cure processes and because of their long potting time windows.

Biresin G26 presents promising results for form maintenance but only with high filler content. This does however present mixing challenges which can be overcome with powerful mechanical mixing designs. The shorter potting time windows is also an advantage and speed control can be fine tuned to guarantee that mixtures only cure after the extrusion. In general the addition of Fumed silica lowered the incorporation of residue in approximately 10% but improved considerably the mixtures flow rate, thus making extrusions more consistent and easier to perform. Biresin G26 is an epoxy resin especially used to produce reinforced plastic parts, being used by several companies to produce several parts of horizontal and vertical wind turbines due to its high performance, excellent properties and flexibility.

We can finalize stating that for the application intended, Biresin G26 has promising results and provides processing windows above 75% to 86% wt filler. Additions above these values need mechanical mixing and are probably possible but could not be tested in this work. Additions of Silica will improve mixture handling but will reduce filler inclusions of approximately 10% wt filler, a trade off that may be advantageous depending on the desired end product.

4 Sample characterization

To determine the behavior of the composites produced during the experimental phases, the mixtures were redone and specimens were produced in accordance with the mechanical test standards that are characterized in the following sections of this chapter. Specimens were produced by pouring or filling the mixtures into silicone molds which were allowed to cure at room temperature with free sample growth and no shape constraint. There are however some specimens that were produced under shape constraint. The intention is to have comparative tests for process evaluation on the differences that shape constraint may produce on the mechanical properties of a specific mixture. After the curing process, the specimens had to be cut down to size and light abrasion was used to produce an homogeneous surface finish. These specimens were then submitted to tensile, hardness and imaging tests to reveal their properties with more detail.

4.1 Tensile test (ISO 527-2)

Produced specimens with a reference length of 60 mm were subjected to tests in accordance to the ISO standard 527-2.

Tests were performed using a universal testing machine from Instron, model 4505 equipped with a 5 kN load cell. For flexible composites a speed of 5 mm/min was used and for harder composites 0,5 mm/min.

The samples for each test came from the same production batch and the molds were filled right after mixing. Mixture times were in line with the 60 seconds stated for the experimental phase, except for mixtures with low potting time windows, where mixing was performed as quickly as possible, producing samples immediately and all within a 30 second time window. Samples were produced by pouring/spreading the compound into an empty mold, filling it up to the brim, then scrapping the excess off with a spatula. This process would repeat itself until all mold slots were used up or the mixture was no longer manageable. For compounds with very high viscosities there were several difficulties producing the samples, so a fixed number of samples wasn't obtained, at least three was examined to a maximum of eight.

Data collected from the tensile tests, were then plotted into curves. Their analysis would provide elongation at break, Young's modulus, yield and ultimate tensile strengths. Determining the exact yield stress point was challenging in many cases, even using the typical value of 0.2% to find the offset yield stress did not provide results. Experimentation provided a good fit with a line drawn from slope E at a strain offset of 0.05%, which would then be used for all the stress-strain curves in this study. Figure 4.1 is an example of data collection for the elongation at break and Young's modulus of Biresin G26, with added silica and filler at 65% wt.

Test sample data of each compound was combined to produce an average stress-strain curve. The following pages present a large number of these average stress versus strain graphs that have been placed in this manner to allow a comparative analysis. Each page presents graphs of the same binder material, being Biresin G26 binder tests represented in Figure 4.4 , Pavistone 1K in Figure 4.5 and Pavistone 2K in Figure 4.6 . Joint analysis of these graphs: the average values of Youngs modulus, ultimate tensile strength, yield strength and standard deviation compiled in Figure 4.2; average data of elongation at break and its standard deviation in Figure 4.3; and the binders mechanical proprieties for tensile strength and elongation at break, when in pure state, represented in Table 4.1 , may provide insight

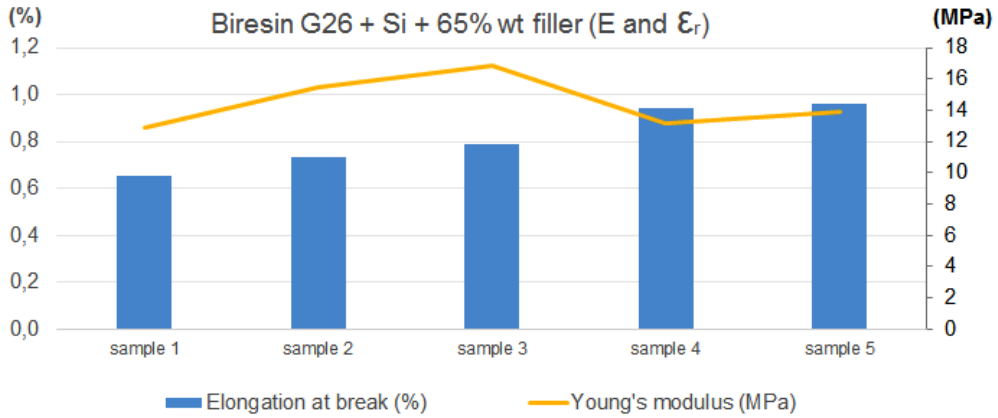


Figure 4.1: Elongation at break and Young's modulus for Biresin G26 +65%wt residue + fumed Silica

into the compounds trends and properties.

Table 4.1: Binder physical data (approx. values)

Binder	Tensile strength (MPa)	Elongation at break (%)
Biresin G26	30	3
Pavistone 1K	24	30
Pavistone 2K	12	<10

For Biresin G26 the analysis of Figures 4.4 , 4.2 and 4.3 present two distinct results: the first is that in relation to pure binder mixtures, the resulting compounds present losses of tensile strength, elongation at break and Youngs modulus; the second is that for compounds with added fillers, their increase will produce increases of Young's modulus and decreases in strain.

For Pavistone 1K the analysis of Figures 4.5 , 4.2 and 4.3 present two distinct results: the first is that in relation to pure binder mixtures, the resulting compounds present losses of tensile strength and elongation at break; the second is that filler additions have a significant negative impact on the ability to withstand stress.

For Pavistone 2K the analysis of Figures 4.6 , 4.2 and 4.3 present three distinct results: the first is that in relation to pure binder mixtures, the resulting compounds present losses of tensile strength; the second is that for filler additions under 40% wt the original materials elongation at break is exceeded; and the third is that filler additions above 63% invert the decreasing tendency of the elongation at break and Youngs modulus of the resulting materials.

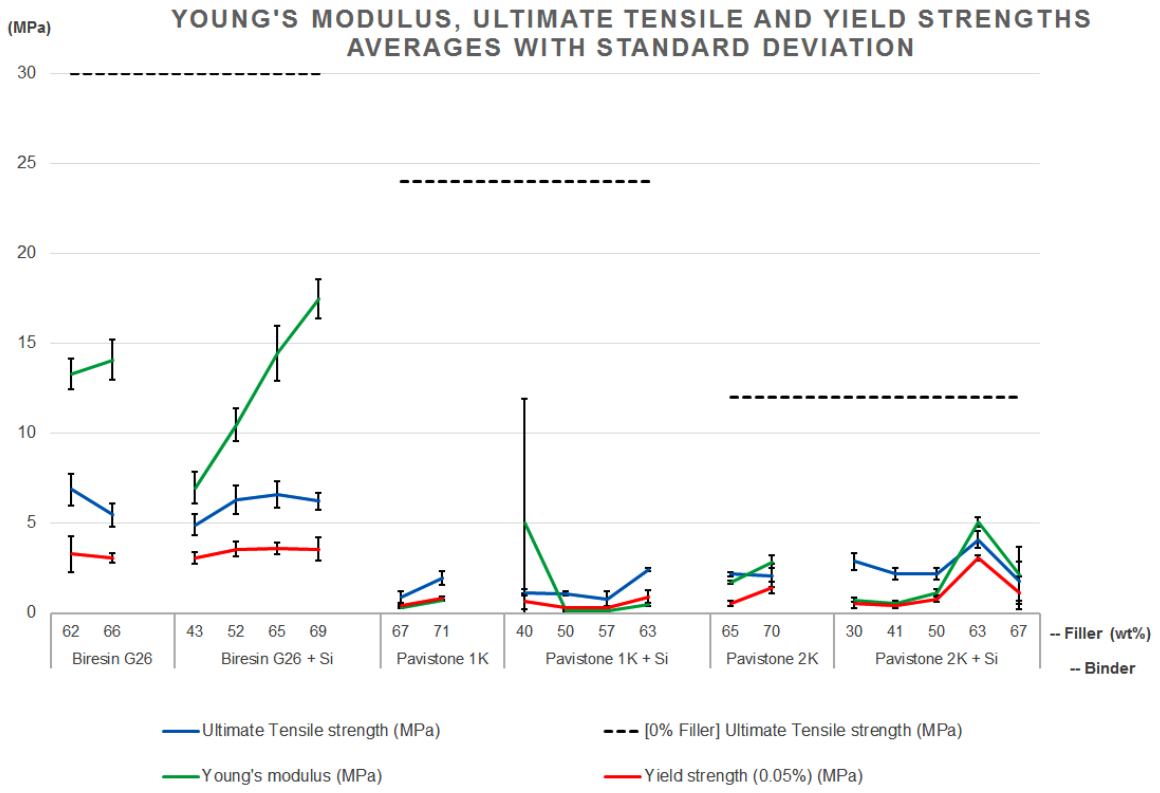


Figure 4.2: Graph of averages of Youngs modulus, ultimate tensile strength, yield strength and standard deviation versus pure binder formulation values

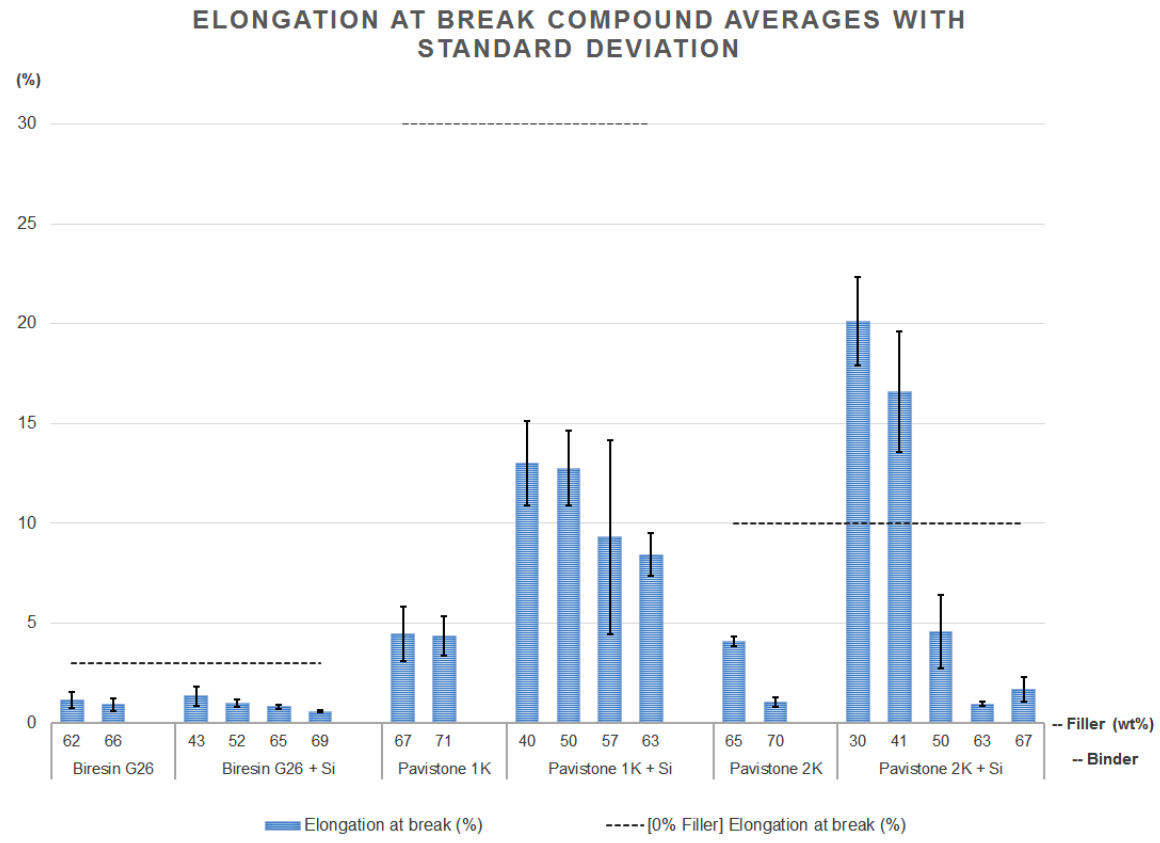
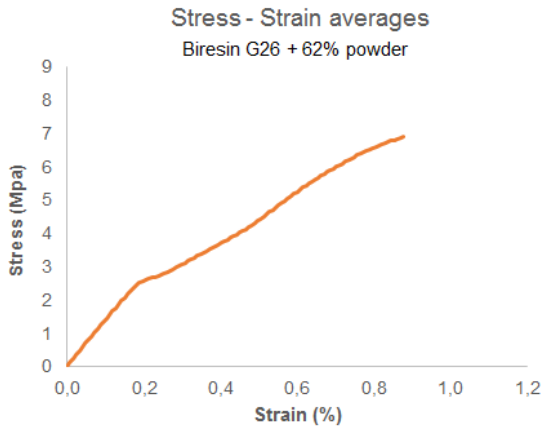
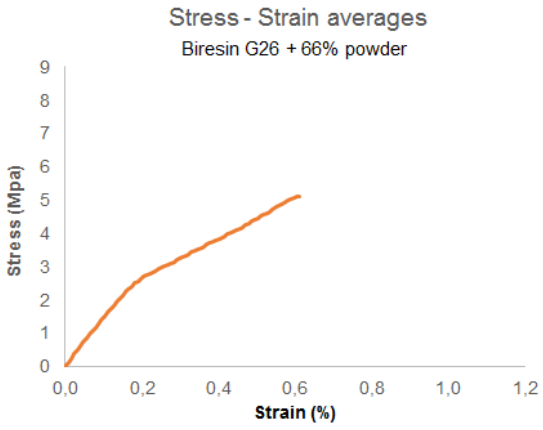


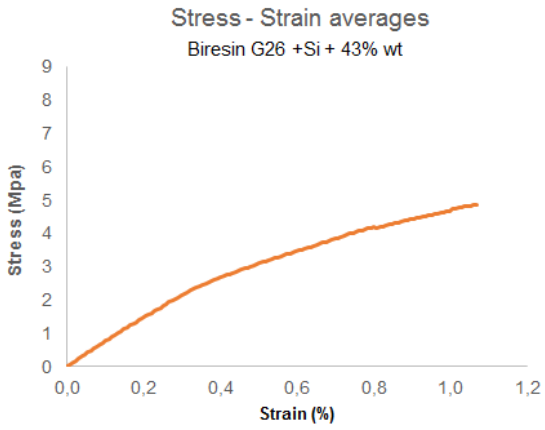
Figure 4.3: Graph of average elongation at break and standard deviation



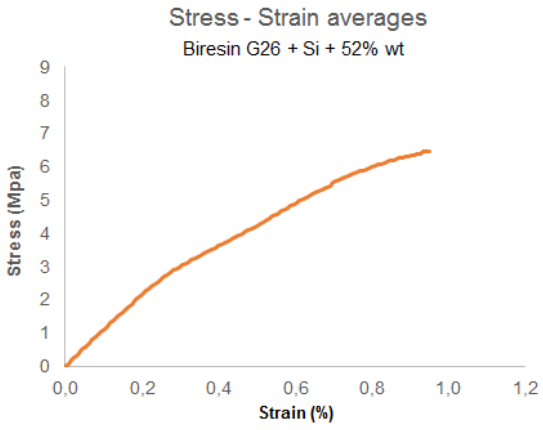
(a) Biresin G26 +62%wt



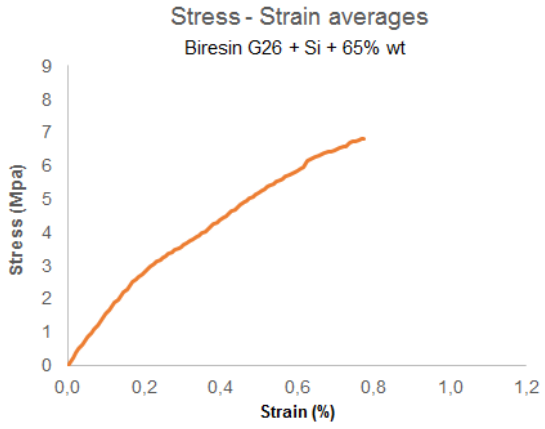
(b) Biresin G26 +66%wt



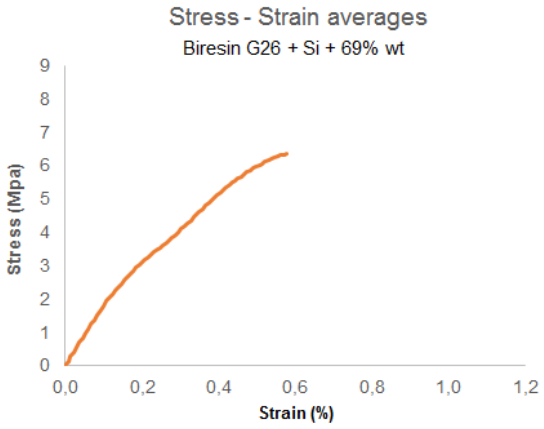
(c) Biresin G26 +Si +43%wt



(d) Biresin G26 +Si +52%wt

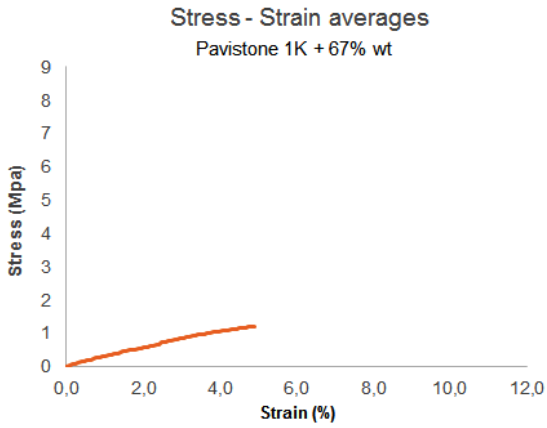


(e) Biresin G26 +Si +65%wt

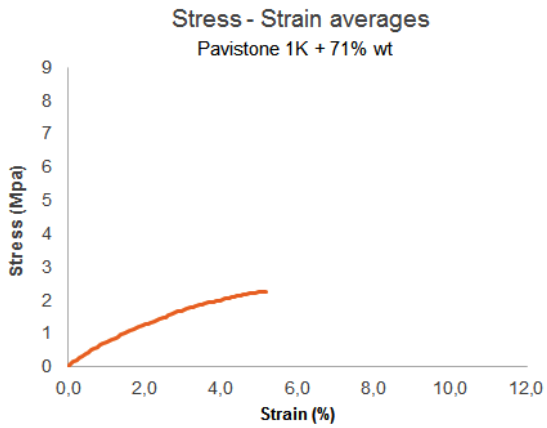


(f) Biresin G26 +Si +69%wt

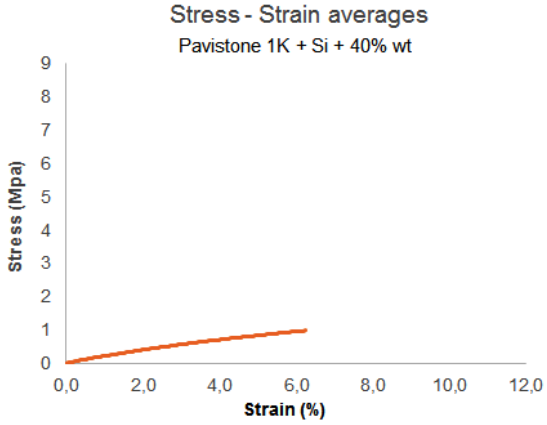
Figure 4.4: Average Stress - Strain compiled graphs for Biresin G26



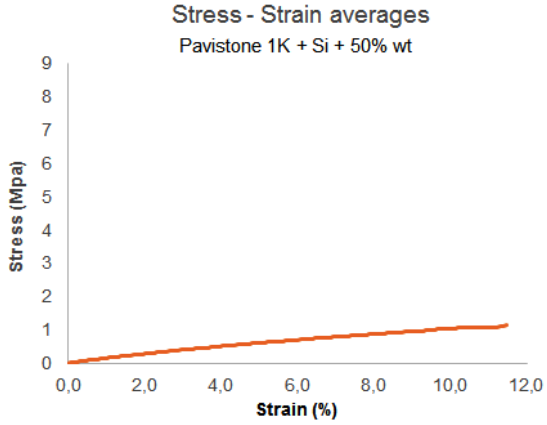
(a) Pavistone 1K +66%wt



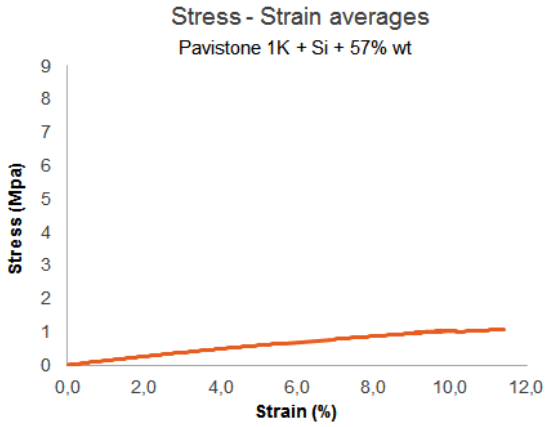
(b) Pavistone 1K +71%wt



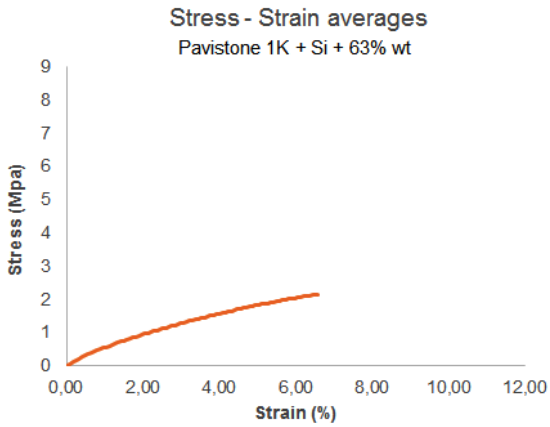
(c) Pavistone 1K +Si +37%wt



(d) Pavistone 1K +Si +49%wt

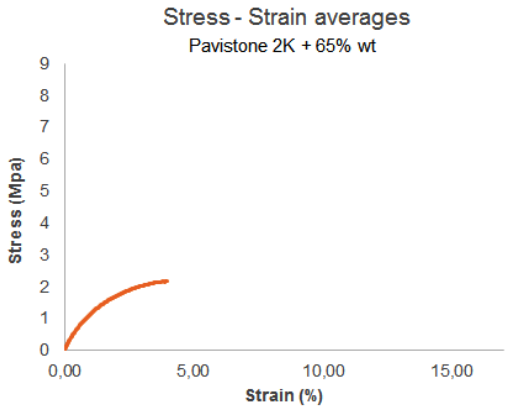


(e) Pavistone 1K +Si +56%wt

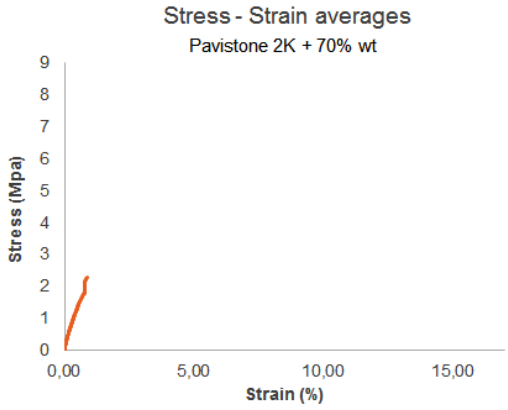


(f) Pavistone 1K +Si +63%wt

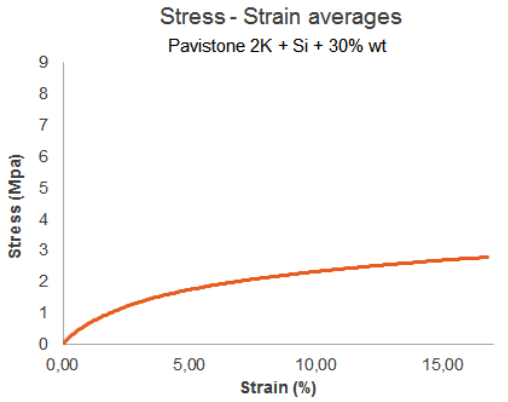
Figure 4.5: Average Stress - Strain compiled graphs for Pavistone 1K



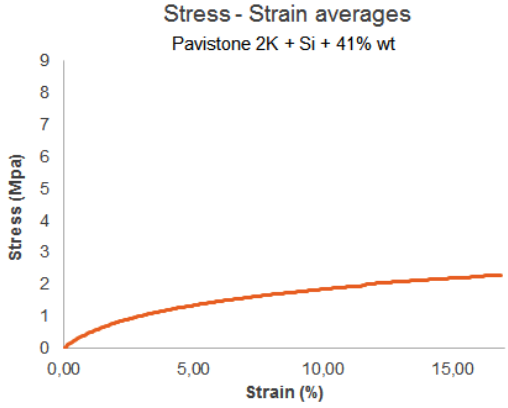
(a) Pavistone 2K +63%wt



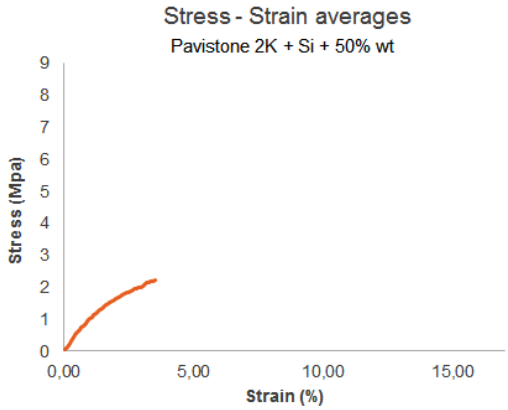
(b) Pavistone 2K +70%wt



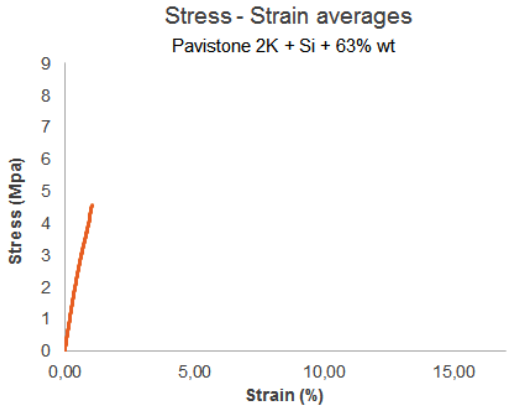
(c) Pavistone 2K +Si +29%wt



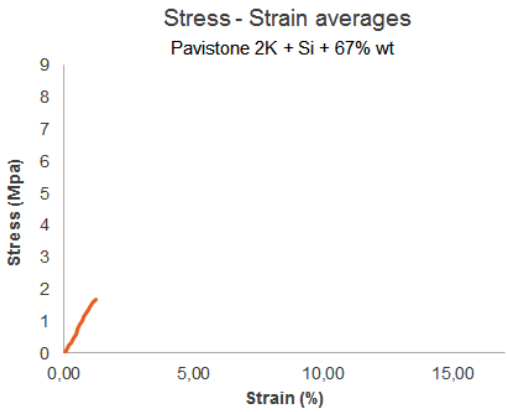
(d) Pavistone 2K +Si +40%wt



(e) Pavistone 2K +Si +48%wt



(f) Pavistone 2K +Si +58%wt



(g) Pavistone 2K +Si +65%wt

Figure 4.6: Average Stress - Strain compiled graphs for Pavistone 2K

Figures 4.7 , 4.8 and 4.9 have photographs of some samples and cross sections that have undergone the tensile test. Existence of bubbles in the composite are the main factor of the early failure exhibited for several of the tested samples. This accounts for the diverse values obtained during the testing and explains some of the high values of standard deviation.

Figure 4.7: Image of sample of tensile test, Biresin G26 +Si +69%wt

Figure 4.8: Image of sample of tensile test, Pavistone 1K +Si +63%wt

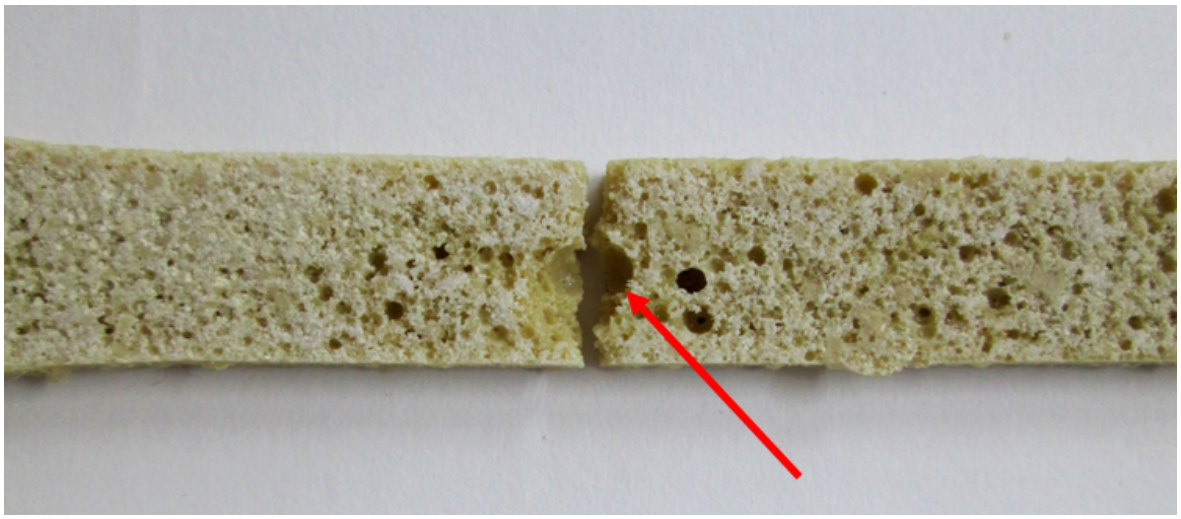


Figure 4.9: Image of sample of tensile test, Pavistone 2K +Si +58%wt

Summary and result analysis of Tensile tests

We initiate this summary with two peculiar singularities that deserve further investigation: the first is for formulations of Pavistone 2k with filler additions below 41% wt, that produce resulting materials that exceed pure binder formulations in at least 170%; the second is also for Pavistone 2K above 63% wt filler that invert the decreasing tendency of the elongation at break with the increase of filler additions.

The overall conclusion, for compounds within this test, with filler inclusions, is that filler additions decrease elongation at break and tensile strengths. Losses produced by increasing filler inclusions produce two distinct tendencies: decrease of the elongation at break; and increase of the tensile strength. Results of Biresin G26 and Pavistone 1K data present a continuous tendency in contrast with Pavistone 2K that produces inflection points in the data that are filler ratio dependent. Addition of fumed silica has impact on the material properties, improving mixtures thixotropy, but lowers incorporation of fillers.

Filler addition in Biresin G26 produces a minimum decrease of 55% for the elongation at break and 77% for tensile strength. In Pavistone 1K filler additions produces a minimum decrease of 57% for the elongation at break and 90% for the tensile strength. Pavistone 2k, with 63% wt filler: the elongation at break maximizes its loss with 90%, from this point there is an increasing tendency of the elongation at break independently of the filler ratio; and the tensile strength minimizes with 66% loss, from this point there is a decreasing tendency of the tensile strength independently of the filler ratio.

Specific high standard deviations can be explained by non-homogeneous composition of samples which is a consequence of the existence of air pockets. The analysis of samples considers a fabrication time window and the results have averages and standard deviations of a specific produced compound, as being all produced within a specific time point.

No other manner could data be obtained without making several hundreds of iterations to provide materials produced within that exact moment. The factors with contributions to this are: mixing by hand several components in a preparation phase before the final mixing could occur; re-mixing all the components to be able to obtain the final composite; the amount of force required to incorporate fillers in high content that challenged the mixing processes, due to the loss of viscosity; and gel as well as potting times of the binders that challenged the mixing stages.

4.2 Hardness tests

To measure the solidity of the specimens, the resistance to indentation was performed according to the procedure described in ASTM D2240 [33] and ISO 868. Due to the diverse hard and soft nature of the specimens, tests were performed initially using the Shore A scale, selecting the lowest and highest content of fillers for each group of specimens. The analysis of the results would then determine which Shore test to perform on each group of samples. Shore A tests were performed on a Instron automatic operating stand, model 903; and Shore D tests were performed on a Bareiss-Prüfgeräte, model BS 61. Machine calibrations were verified with elastomer blocks of different hardness before testing. The compiled data with averages and standard deviation can be verified in Table 4.2.

Table 4.2: Hardness averages, standard deviation and hardness percentage variation for tested samles

Material	**Shore D**	σ_D	**Shore A**	σ_A	**Hardness % variation** [a]
Biresin G26 +62%wt	61	5.9	97	1,2	-13%
Biresin G26 +66%wt	71	3,7			-2%
Biresin G26 +43%wt +Si	51	8,2			-28%
Biresin G26 +52%wt +Si	65	6,8			-7%
Biresin G26 +65%wt +Si	97	3,7			38%
Biresin G26 +69%wt +Si	72	3,9	96	1,1	3%
Pavistone 1K +67%wt	42	4,0	87	3,2	-15%
Pavistone 1K +71%wt	53	2,8			5%
Pavistone 1K +40%wt +Si	29	2,2			-42%
Pavistone 1K +50%wt +Si	38	2,8			-24%
Pavistone 1K +57%wt +Si	28	2,0			-44%
Pavistone 1K +63%wt +Si	43	2,5	89	2,2	-13%
Pavistone 2K +65%wt	38	2,4	89	5,3	-42%
Pavistone 2K +70%wt	39	4,1			-40%
Pavistone 2K +30%wt +Si	27	3,9			-58%
Pavistone 2K +41%wt +Si	26	5,0			-61%
Pavistone 2K +50%wt +Si	30	6,2			-54%
Pavistone 2K +63%wt +Si	52	7,1			-20%
Pavistone 2K +67%wt +Si	47	8,7	93	2,7	-28%
Pavistone 1K +67%wt	36	3,2	87	3,6	-29%
Pavistone 2K +65%wt	53	4,2			-19%
Pavistone 2K +70%wt	50	5,2	95	2,8	-23%

[a] Compound Hardness with no filler, Shore D:
Biresin G26: 70D;
Pavistone 1K: 50D;
Pavistone 2K: 65-70D.

Durometer readings less than 10 are not reported because they are inexact, readings lower than 20 and greater than 90 are not considered reliable for either the Shore A or Shore D instruments [34], so for that reason, no analysis will be made on the recorded reading for the Shore A instrument. All specimens were tested against the Shore D scale, the values were plotted in Figure 4.10 with the average hardness values in columns and their standard

deviation in the error bars. The graph is categorized per material group, with and without the addition of fumed Silica and the fillers increase from left to right.

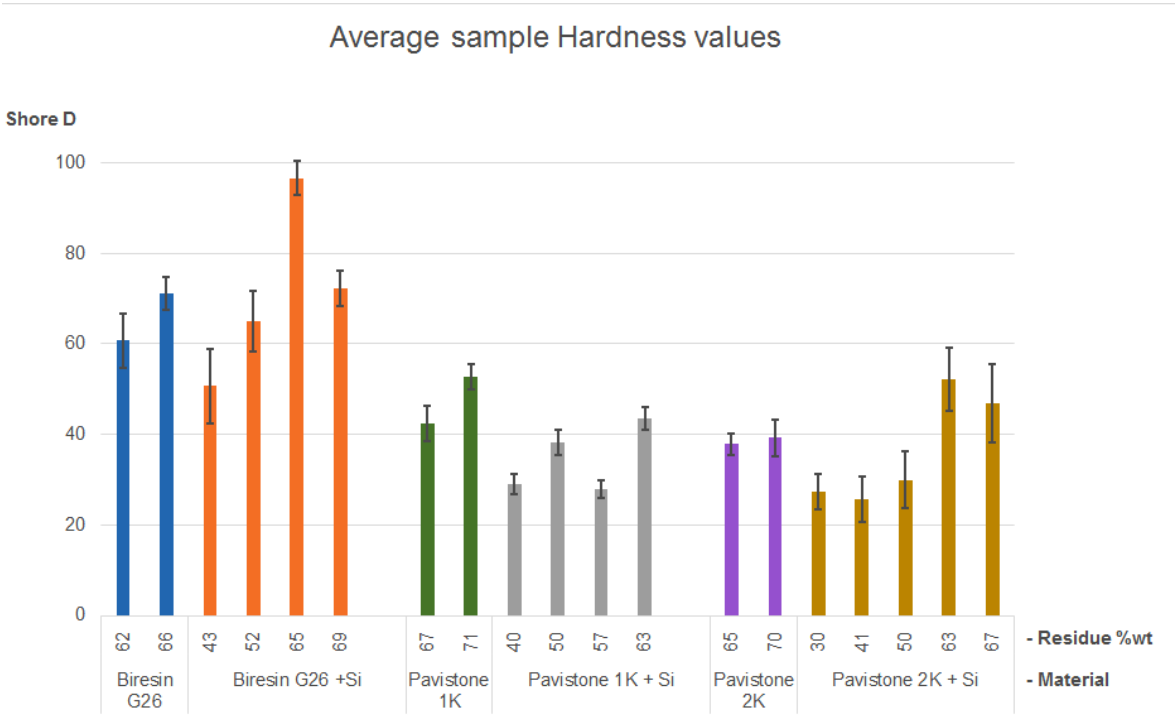


Figure 4.10: Graph of average hardness for tested specimens (Shore D)

Samples of Biresin G26 with fillers that underwent the hardness test are displayed in Figures 4.11 to 4.13. Sections that seemed relevant at the naked eye were then examined by optical microscope and a photo with a x4 factor captured the details of those specimens. The indenter mark is precise and clearly visible which confirms the higher values of hardness plotted in Figure 4.10.

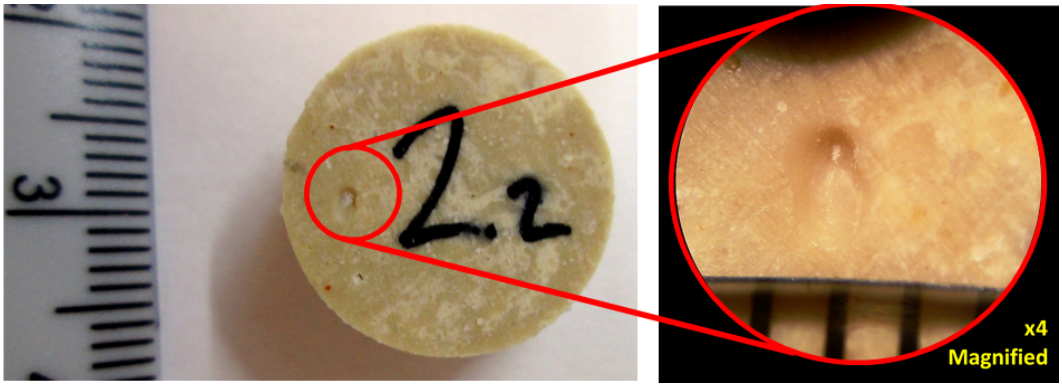


Figure 4.11: Sample details of Biresin G26 +66%wt

The same procedure was applied to samples of Pavistone 1K, Figures 4.14 to 4.16, but the magnification factor was adjusted to one that provided better detail, thus different magnification factors exist for the samples shown in these figures. The fact that powder is visible in the samples is not proof of lumping, but mostly due to the grinding produced to level the samples down to the size referred to the standard.

Figures 4.17 to 4.19 display the samples for Pavistone 2K and the magnification factor x2 presented good details. As in samples of Pavistone 1K the existence of powder is due to the grinding of the samples.

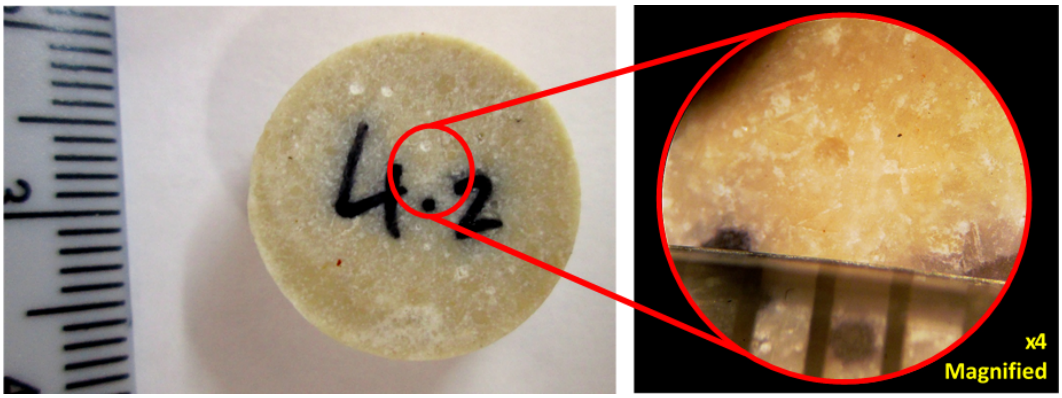


Figure 4.12: Sample details of Biresin G26 +Si +52%wt

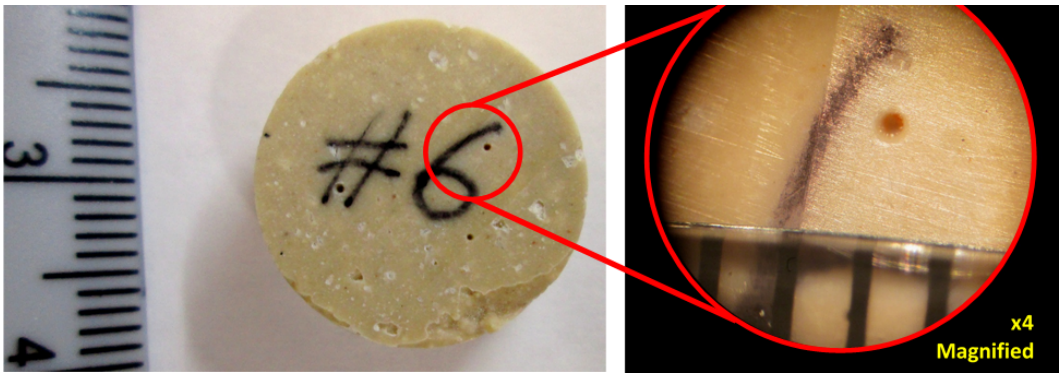


Figure 4.13: Sample details of Biresin G26 +Si +69%wt

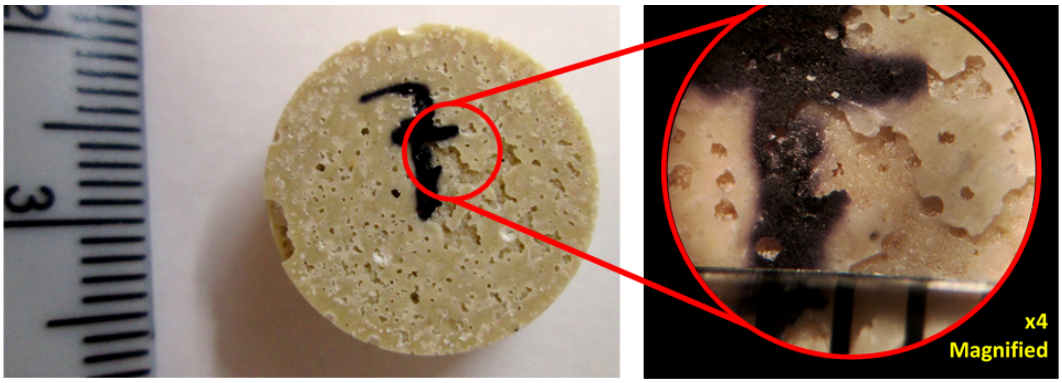


Figure 4.14: Sample details of Pavistone 1K +66%wt

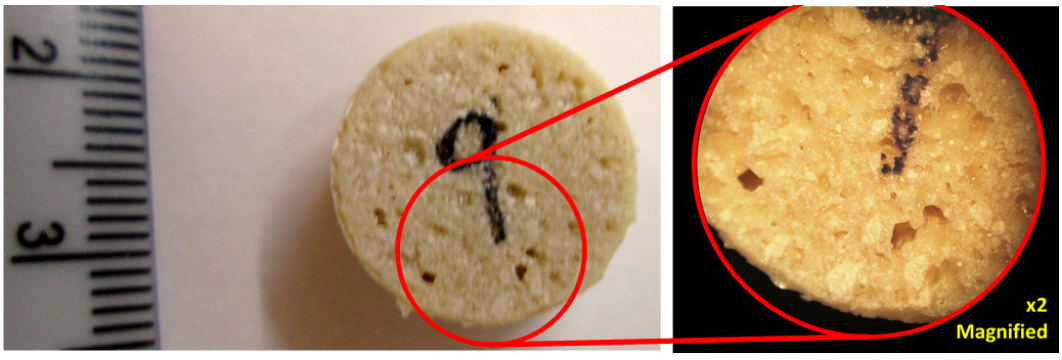


Figure 4.15: Sample details of Pavistone 1K +Si +37%wt

Figure 4.16: Sample details of Pavistone 1K +Si +56%wt

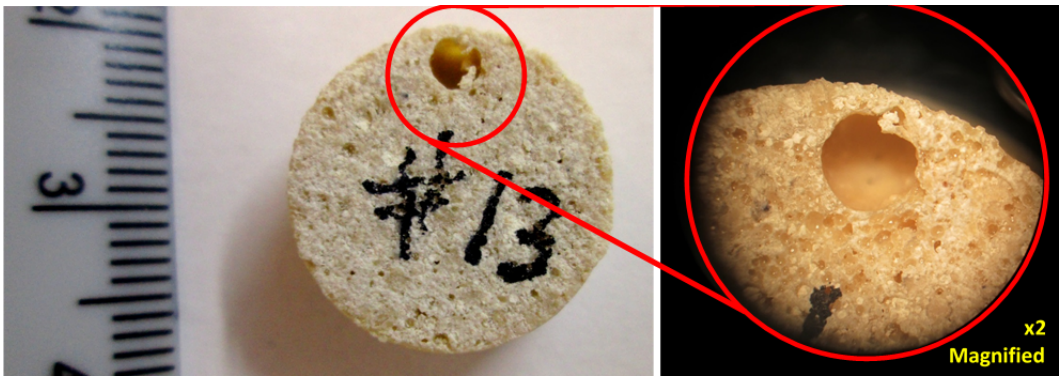


Figure 4.17: Sample details of Pavistone 2K +63%wt

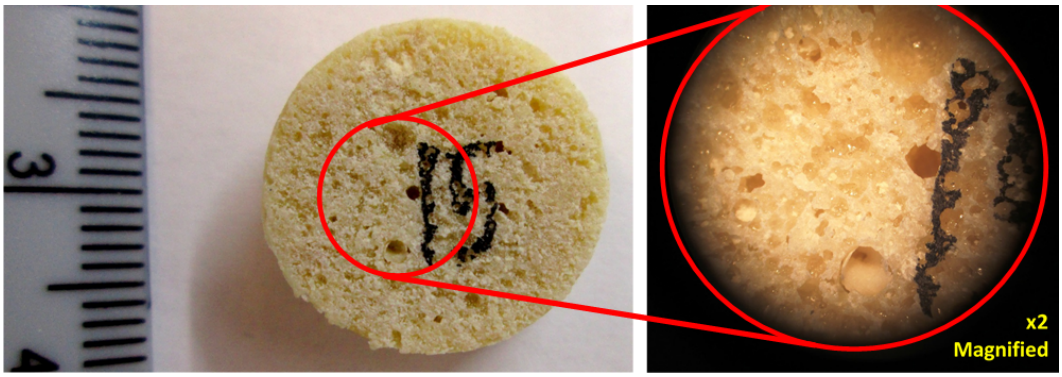


Figure 4.18: Sample details of Pavistone 2K +Si +29%wt

Summary and result analysis of Hardness tests

The analysis of Figure 4.10 reveals that there is a clear increase in hardness with the increase of filler addition to the compounds. Note that this increase in hardness is relative to compounds within the tests and not to the binders cured without fillers. Filler addition generally leads to a decrement of the mechanical properties than that of a pure polymer sample. This fact is valid for compounds with and without the addition of fumed Silica being that the amount of Silica mixed, influences the mixtures thixotropy and lowers the amount of includable fillers. The standard deviation is high denoting that there is no stability in the tests and produced samples. This may be explained by the existence of air pockets in the composites, clearly visible in any of the Figures 4.14 to 4.19, lowering the density in that region and providing an escape for the forces when the indenter falls close to them, which in turn lowers

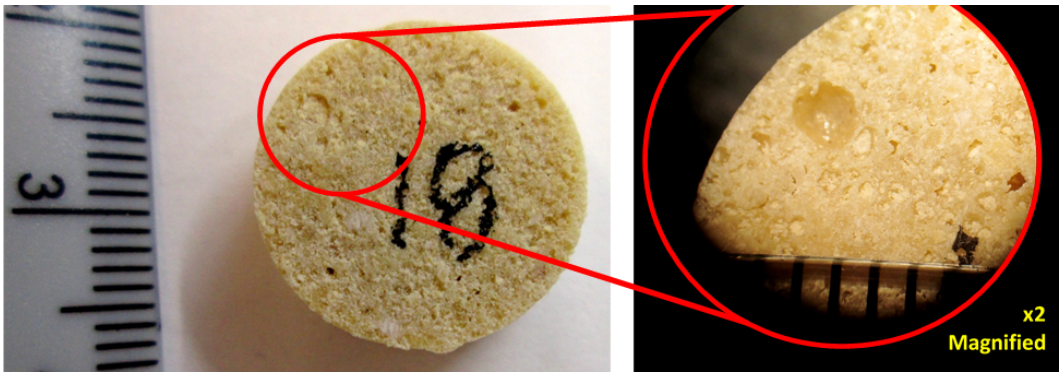


Figure 4.19: Sample details of Pavistone 2K +Si +58%wt

the outcome on the durometer scale. This variance phenomena is most surely a result of non-homogeneous samples resultant from the curing process and the production stages of the compounds. It can also be corroborated with the hardness values (shore) stated for the binders, cured without fillers in their data-sheets: 70D for Biresin G26; 50D for Pavistone 1K; and 65-70D for Pavistone 2K. For most of the cases there is a loss in compound hardness visible in the right column of Table 4.2 with losses up to: -28% for Biresin G26; -44% for Pavistone 1K; and -61% for Pavistone 2K.

Although Shore A readings were not used for analysis they do provide confirmation of a correct testing procedure and calibration of the machines used for the tests. With the use of the values from Table 4.2 and the chart in Figure 4.20, the results from Shore A scale do confirm the Shore D values of the tests.

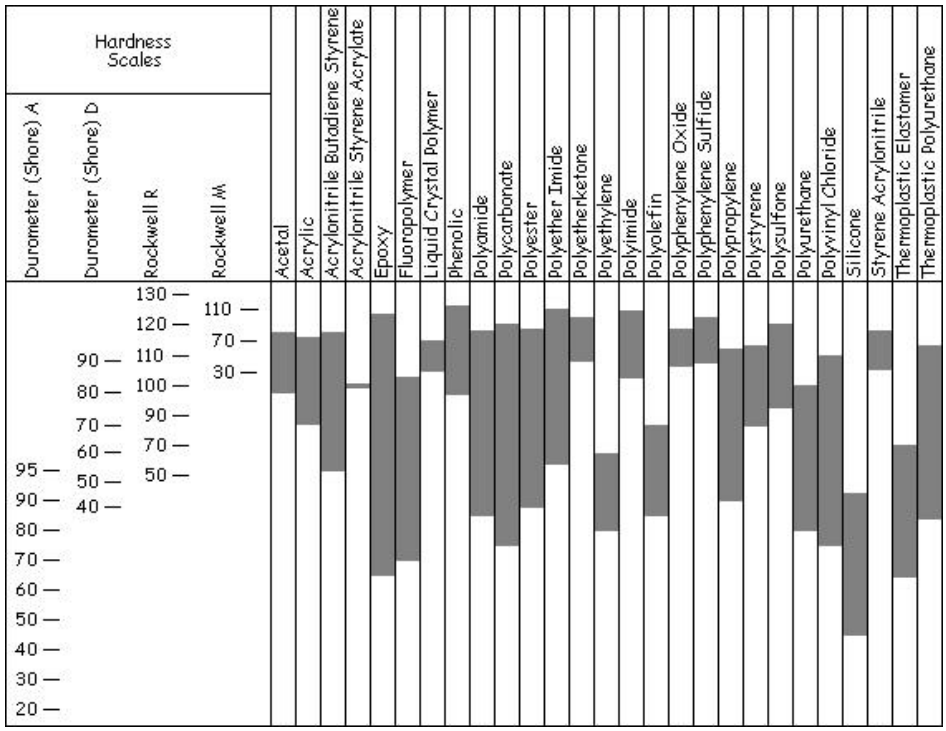


Figure 4.20: Hardness conversion chart [33]

4.3 Micro computed tomography scan (Micro-CT)

Micro-CT scans were performed to determine the specimen's detailed internal structure and the density of a volume of interest (VOI) using the Hounsfield unit (HU) as a reference, a dimensionless unit universally used in X-ray Micro Computed Tomography. These are obtained from a linear transformation of the measured attenuation coefficients which are based on the arbitrarily-assigned densities of air and pure water [35]. Hounsfield units are generally used in clinical applications and the results are a scale running from -1000 HU for air to + 2000 HU for very dense bone and over 3000 for metals [36]. The reported values from these tests are calibrated with a water reference scanned phantom.

The images were acquired on a Bruker SkyScan 1174 (ver2.A) with the following parameters: Voltage: 50 kV; Current: 800 µA; Exposure time: 5500 ms; Rotation Step: 0.50°; with 360°rotation; Image pixel size: 16.7 µm; frame averaging of 2 and a 0.25 Al filter. The scans had a duration of 2h20 min. Images were enhanced with ring reduction: 4 and smoothing: 3 providing sufficient smoothing and removal of excess background noise. An example of an acquired image is visible in Figure 4.21 and the remaining images can be seen in Appendix J.

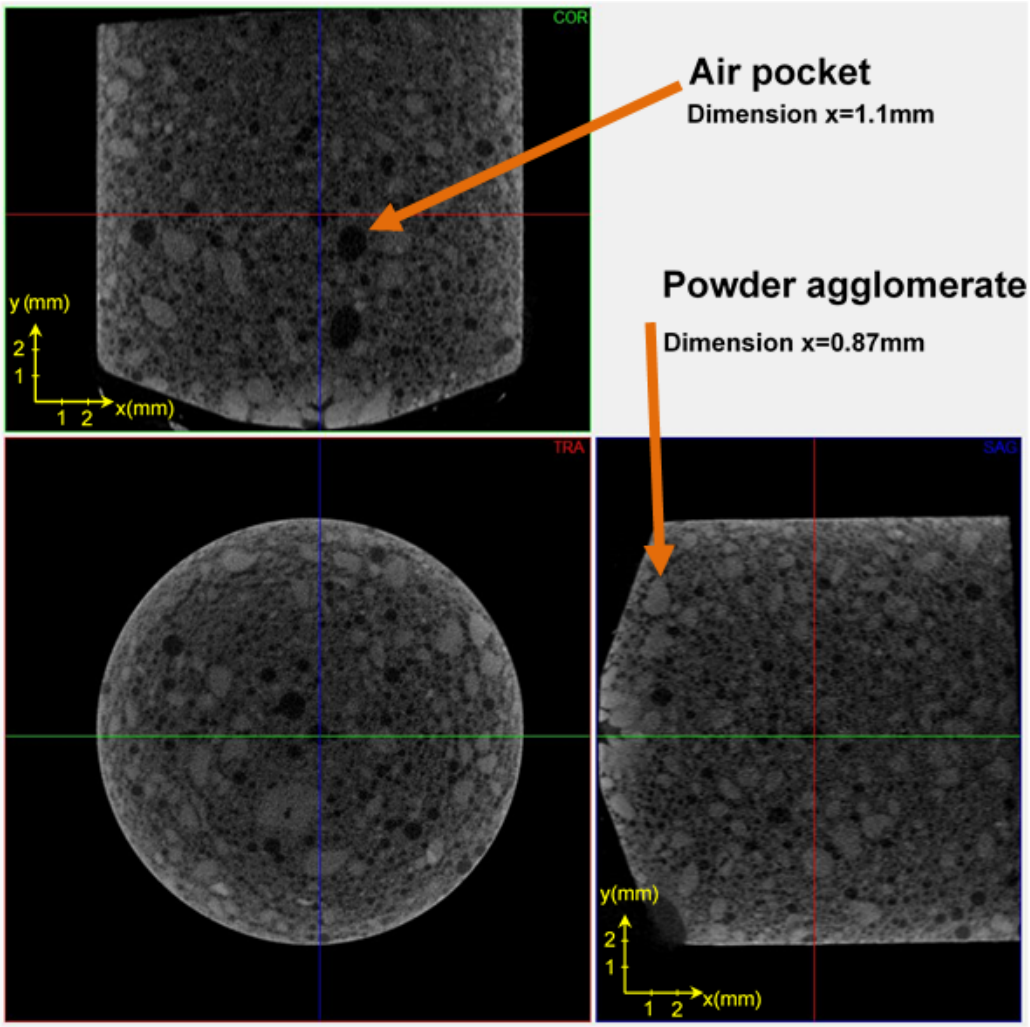


Figure 4.21: Three plane images of Micro-CT from specimen Pavistone 2k +63%wt

Internal verification of sample composition was considered important and for that reason a few samples were cracked open with hard impact. The fractured samples were then placed under an optical microscope and evidence of powder was found in several pockets within a variety of them, as well as voids that confirm existence of air pockets. The internal composition could then be identified as lighter gray colors for agglomerates of powder and the black voids as air pockets. Figure 4.22 shows clearly the material differences and confirms the existence of powder within one of the samples tested.

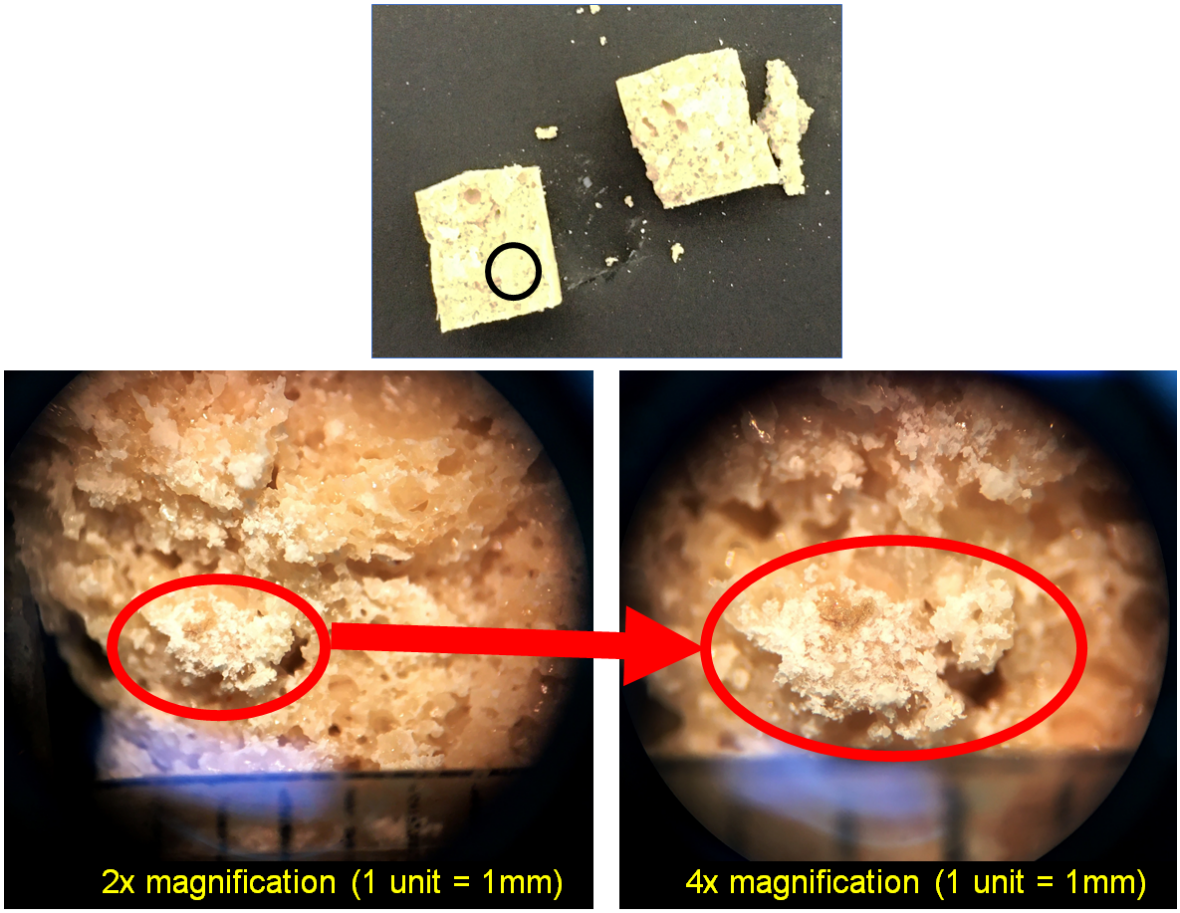
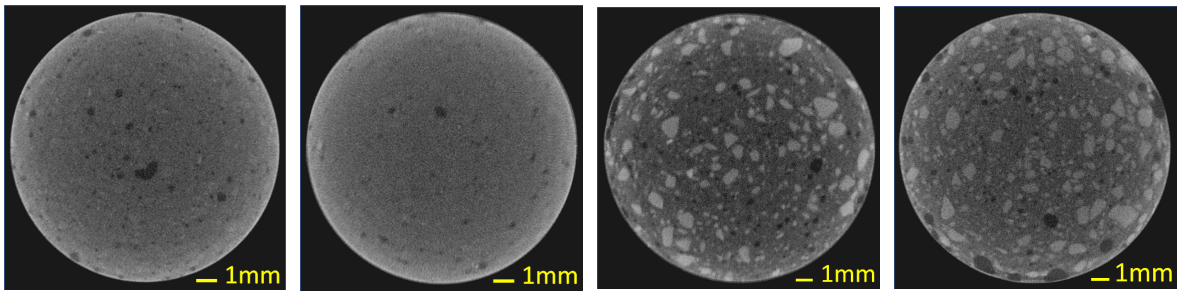
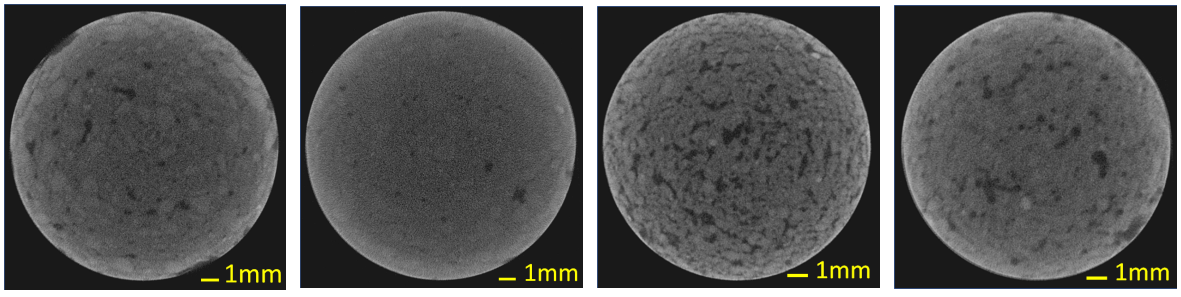


Figure 4.22: Cracked sample Pavistone 2k + Si +40%wt, visible white powder

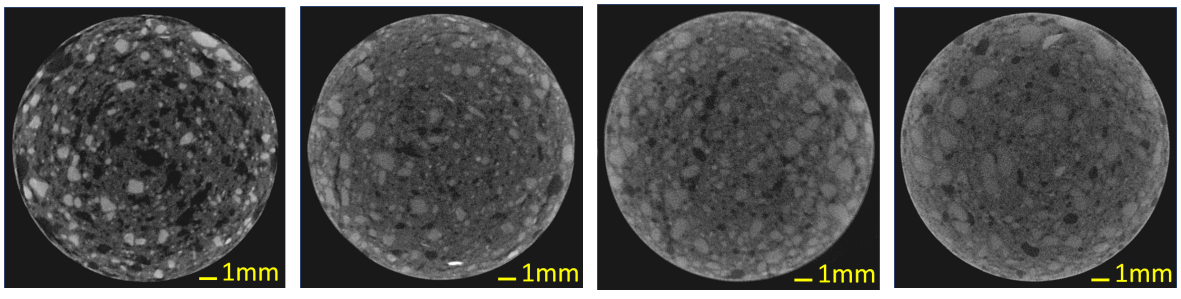
Figure 4.23 displays all specimens tested, showing slice 400, an inner slice which was arbitrarily been selected to represent the internal structure of the sample viewed. This view provides information relevant to the internal composition of the composite but also showcases the porosity represented by all the black voids within the diameter of the sample.



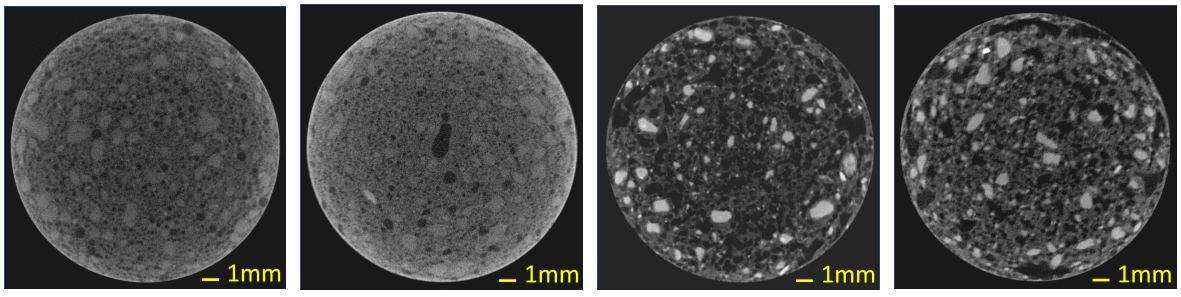
(a) Biresin G26 +77%wt **(b)** Biresin G26 +86%wt **(c)** Biresin G26 +Si +43%wt **(d)** Biresin G26 +Si +52%wt



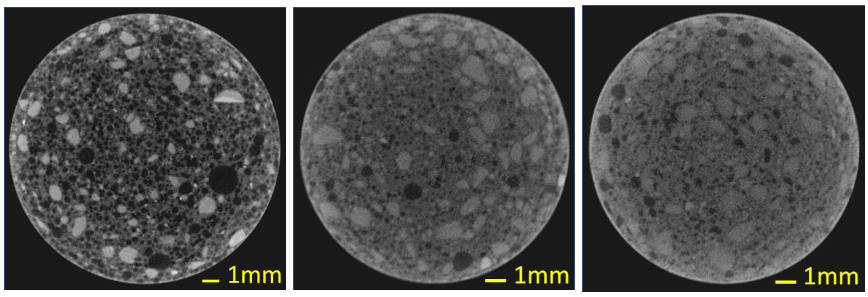
(e) Biresin G26 +Si +65%wt **(f)** Biresin G26 +Si +69%wt **(g)** Pavistone 1K +66%wt **(h)** Pavistone 1K +71%wt



(i) Pavistone 1K +Si +37%wt **(j)** Pavistone 1K +Si +49%wt **(k)** Pavistone 1K +Si +56%wt **(l)** Pavistone 1K +Si +63%wt



(m) Pavistone 2K +63%wt **(n)** Pavistone 2K +70%wt **(o)** Pavistone 2K +Si +29%wt **(p)** Pavistone 2K +Si +40%wt



(q) Pavistone 2K +Si +48%wt **(r)** Pavistone 2K +Si +58%wt **(s)** Pavistone 2K +Si +65%wt

Figure 4.23: Slice 400 of CT-scanned samples

The scanned data used the same ROI for all samples and density calibration was performed with a water phantom, being it a polyethylene syringe cut to the samples size and filled with distilled water. The reconstruction settings were identical to all samples and calibration procedures. The sample density's were calculated considering the same VOI based on the initial ROI of each sample and phantom. The samples originated from cutting slices of the syringes from the initial manual string tests and the results can be seen in Table 4.3. This data was also plotted in Figure 4.24.

Table 4.3: Micro-CT values for tested composite materials

Material	VOI (μm^3)	Object volume (μm^3)	Total porosity (%)	Object (%)	Density (HU)
Biresin G26 +77%wt	9,19x10^{11}	8,98x10^{11}	2,25	97,70	219,01
Biresin G26 +86%wt	9,19x10^{11}	9,12x10^{11}	0,70	99,30	266,80
Biresin G26 +43%wt +Si	9,19x10^{11}	8,59x10^{11}	6,48	93,52	29,70
Biresin G26 +52%wt +Si	9,19x10^{11}	8,89x10^{11}	3,19	96,81	44,50
Biresin G26 +65%wt +Si	9,19x10^{11}	8,31x10^{11}	9,54	90,46	219,50
Biresin G26 +69%wt +Si	9,19x10^{11}	7,76x10^{11}	15,54	84,46	-278,80
Pavistone 1K +66%wt	9,19x10^{11}	8,47x10^{11}	7,80	92,20	189,50
Pavistone 1K +71%wt	9,19x10^{11}	8,85x10^{11}	3,65	96,35	231,30
Pavistone 1K +37%wt +Si	9,19x10^{11}	6,96x10^{11}	24,20	75,80	-179,20
Pavistone 1K +49%wt +Si	9,19x10^{11}	8,41x10^{11}	8,42	91,58	-11,41
Pavistone 1K +56%wt +Si	9,19x10^{11}	8,57x10^{11}	6,75	93,25	69,00
Pavistone 1K +63%wt +Si	9,19x10^{11}	8,71x10^{11}	5,21	94,79	116,90
Pavistone 2K +63%wt	9,19x10^{11}	7,78x10^{11}	15,28	84,72	13,62
Pavistone 2K +70%wt	9,19x10^{11}	8,61x10^{11}	6,30	93,70	34,80
Pavistone 2K +29%wt +Si	9,17x10^{11}	6,20x10^{11}	32,37	67,63	-398,00
Pavistone 2K +40%wt +Si	9,19x10^{11}	7,65x10^{11}	16,73	83,27	-466,30
Pavistone 2K +48%wt +Si	9,19x10^{11}	8,00x10^{11}	12,88	87,13	-245,90
Pavistone 2K +58%wt +Si	9,19x10^{11}	8,12x10^{11}	11,56	88,44	51,80
Pavistone 2K +65%wt +Si	9,19x10^{11}	8,60x10^{11}	6,44	93,56	148,50

Summary and result analysis of Micro-CT scan

In general we can state that increased additions of powder residues increase the density of composites, which in turn lower porosity within the composite. In exception for Biresin G26 for formulations with added silica, the increase of residues will decrease the composite density, thus increasing significantly the porosity. These statements can easily be verified in Figure 4.25 where the total porosity in percentage has been plotted against the sample and the addition of fillers %wt. The addition of fumed silica lowers the composite density, decreases the amount of residue that can be added and improves the mixtures viscosity, it also improves the lumping effect but shortens the gel-time.

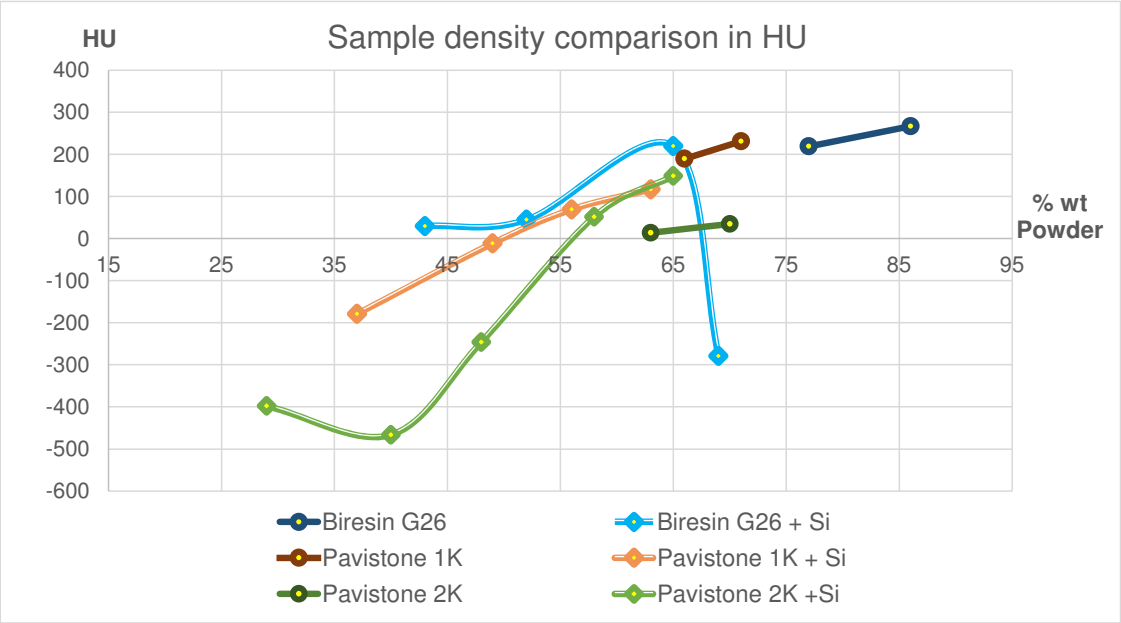


Figure 4.24: Plotted values for material density's in HU

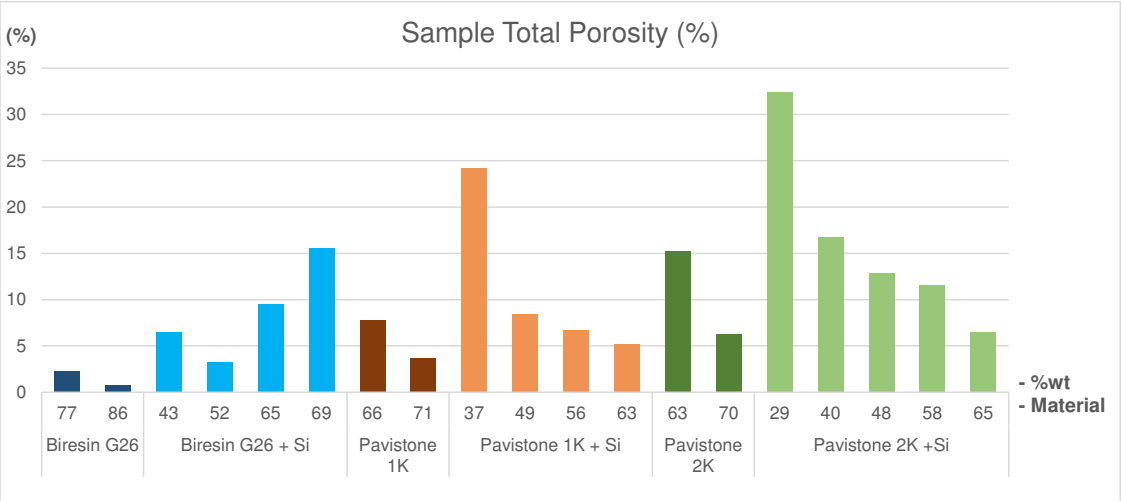


Figure 4.25: Samples Total Porosity in percentage

4.4 Volumetric restricted cure comparison

Additive manufacturing processes produce components without the need for geometric constriction. During the experimental phase variations were observed as well as the difficulty in producing samples, in accordance to the requirements of the standards, due to the growth in volume of the mixtures, which challenged the production of samples with precise dimensions. Taking into consideration that this may not be the best manner to compare samples produced with and without volumetric restriction, the volumetric restriction was considered as a test that could provide valuable data, that could show if the variations of the results were due to malformations in the samples during the curing process.

The creation of gas during cure creates voids within the compounds that can compromise mechanical properties of the produced material. So with the intent of understanding the differences of curing with and without physical restriction, several samples were placed in silicone molds and covered in Polytetrafluoroethylene (PTFE) sheets. The volumetric restriction was setup with escape vents on the silicone molds: For the tensile test samples 2 mm escape vents on each extremity; and for the Hardness test samples a side escape vent was performed for each sample. When the molds had been filled the sheet of PTFE was placed and then loaded with a 2 kg mass to make sure pressure was placed on the samples and the growth of material would escape from the vents and not lift the PTFE sheet.

Figure 4.26 compiles average results of Young's modulus and elongation at break for tests performed on formulations with volumetric restricted cure: Pavistone 1K with 67%wt filler; Pavistone 2K with 65%wt filler and Pavistone 2K with 70%wt filler. It displays the comparison values for the same formulation without volumetric restricted cure (column on the left) and for volumetric restricted cure (column on the right).

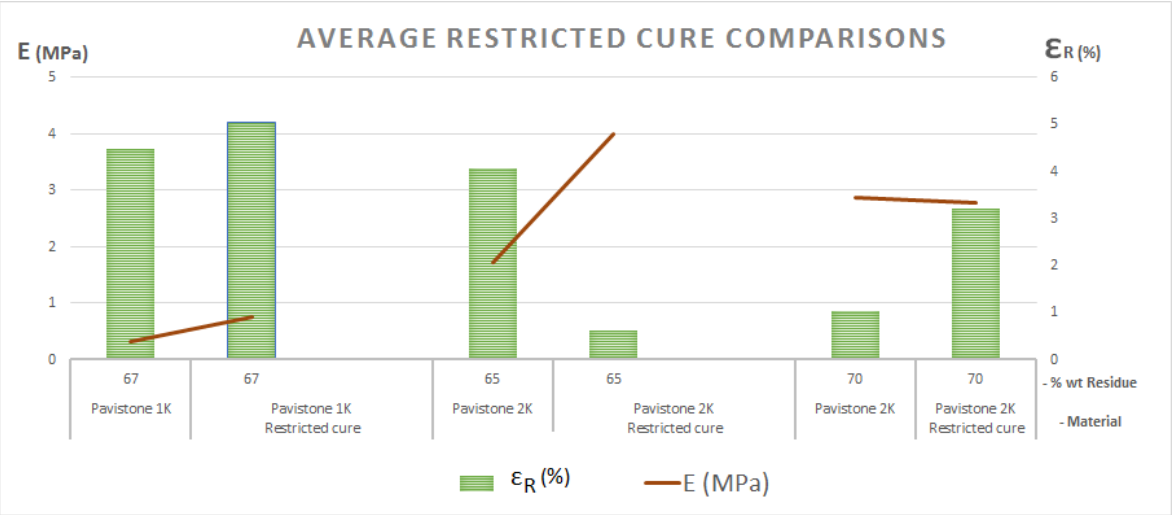


Figure 4.26: Restricted cure comparison Graph of average Young's modulus and elongation at break

Apart from the visible difference of the compact and polished surface finish of the restricted cure samples, there are no apparent general differences across the board, provided by the characterization techniques, that would provide proof that restricted cure would make materials perform in a specific manner. So with a separate analysis of the compounds we can conclude that: for Pavistone 1K with 67%wt there is a gain of 13% for the elongation at break and a gain of 139% for Young's modulus; for Pavistone 2K with 65%wt there is a loss of 85% for the elongation at break and a gain of 132% for Young's modulus; for Pavistone 2K with 70%wt there is a gain of 207% for the elongation at break and a loss of 3% for Young's

modulus.

In Figure 4.27 the Hardness values of the samples are displayed, being the columns to the right of each group the samples that have undergone physical restricted cure.

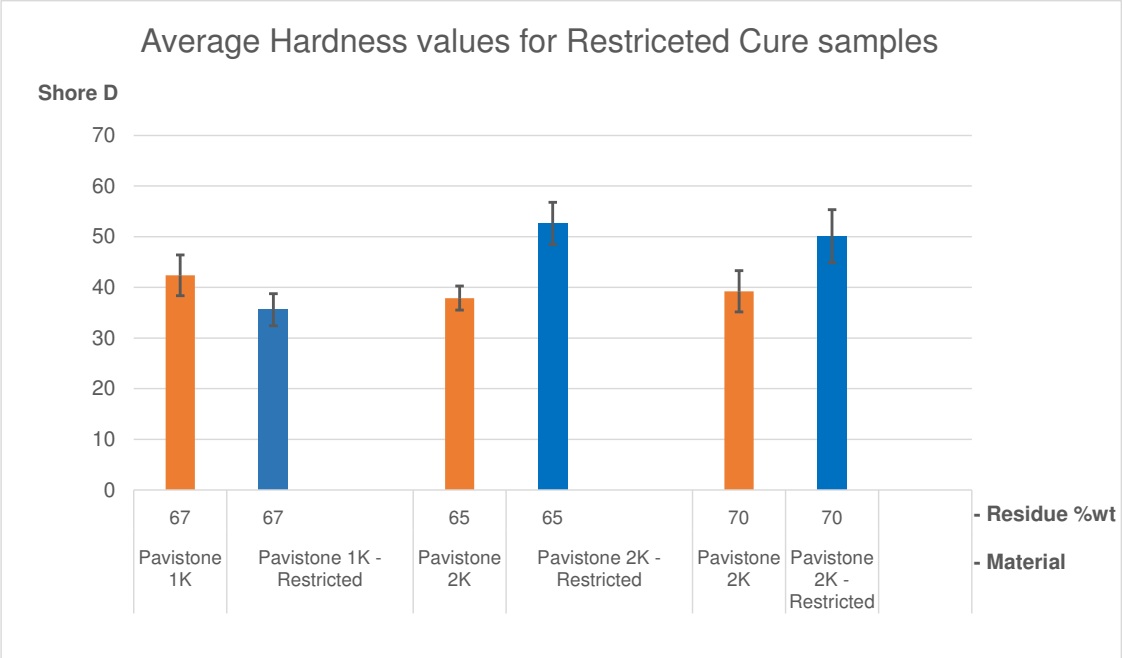


Figure 4.27: Restricted cure comparison Graph of average Young's modulus and elongation at break

For Pavistone 2K we can state that there is an increase of hardness with the increase of residue incorporation. For formulations with 65%wt residue there is an increase in hardness of 39% and for formulations with 70%wt residue there is an increase in hardness of 28%. In the case of Pavistone 1K there is a loss of 16% in hardness for formulations with incorporation of 67%.

4.5 Summary of sample characterization

The analysis of performed tests revealed correlations between addition of residues to the mechanical properties of the materials. The mixtures need to have the correct stochiometric proportions in order to produce viable specimens. The addition of fumed silica improves mixture viscosity, lowers the ability to add fillers as well as the gel time.

The results obtained at this date are that in general filler additions lead to a decrease of the mechanical properties (lower stress, elongation at break and hardness than pure binder formulations).

Overall we can state that materials with filler inclusions generally suffer from a decrease of their elongation at break, tensile strength and hardness in comparison to their pure binder formulations. There are however exceptions and for that reason they have been summarized in Table 4.4. For situations where the compounds were subjected to physical restriction during the curing process the conclusions, as stated above, could not be generalized and are summarized in Table 4.5.

Table 4.4: Results of sample characterization

Characterization technique	Binder + %wt residue	Compound properties
Density (CT-Scan)	63 to 86%wt No Silica	All compounds show an increase in density and a decrease in porosity with the increase of residue incorporation.
	29 to 86%wt + Silica	With the increase in residue incorporation all compounds have a decrease in porosity except for Biresin G26 where there is an increase in porosity for additions of fillers over 52%wt. The densities also increase with the increased incorporation of residue except for Pavistone 2K under 40%wt and for Biresin G26 from 65%wt that has an abrupt decrease of density.
Hardness tests	29 to 86%wt No Silica	Hardness of the compounds increase with higher incorporation's of residue.
	Biresin G26 + Silica	Hardness of compound decreases from 65%wt.
	Pavistone 1K + Silica	Hardness increases with a decreasing inflection at 57%wt.
	Pavistone 2K + Silica	Hardness is more or less stable up-to 50%wt, then increases up-to 63%wt from where it starts to decrease.
Tensile tests	29 to 86%wt	Increasing additions of fillers produce materials that withstand higher tensile strengths except for Pavistone 1K up to 50%wt and Pavistone 2K for incorporation's over 63%wt. There is in general a decrease in the ability to withstand strain with higher additions of fillers, except for Pavistone 2K with added fillers lower than 41%wt.

Table 4.5: Form restricted cure results of sample characterization

Characterization technique	Binder + %wt residue	Compound properties
Hardness tests	Pavistone 1K + 67%wt	Hardness loss of 16%.
	Pavistone 2K + 65%wt	Hardness gain of 85%.
	Pavistone 2K + 70%wt	Hardness gain of 28%.
Tensile tests	Pavistone 1K + 67%wt	Gain of 13% for ε and gain of over 139% for E.
	Pavistone 2K + 65%wt	Loss of 85% for ε and gain of approximately 132% for E.
	Pavistone 2K + 70%wt	Gain of 207% for ε and a loss of 5% for E.

5 Equipment concept (AMfRP)

The quality of a final product is dependent of the quality of its raw materials and its processing stages. From the experience of manually mixing and combining the components of these mixtures, it was noticeable that the mixing efficiency is critical to achieve a smooth, homogeneous product with consistent quality. The mixing stages should then be controlled to avoid air incorporation and lumping. As the materials being handled are thermoset polymers (resins) that during their curing stages produce chemical and mechanical irreversible changes in their formulations, the equipments required to handle these components and mixtures will require re-usage and self cleaning capabilities. The mixing stages have to be carefully figured out with special regard to minimizing operation and maintenance costs.

5.1 Concept

The existence of several commercial systems that optimize many stages of the process are in fact options to be considered. For that reason the new processing system will be a collection of experience and existing technology assembled and modified to meet the requirement of this project.

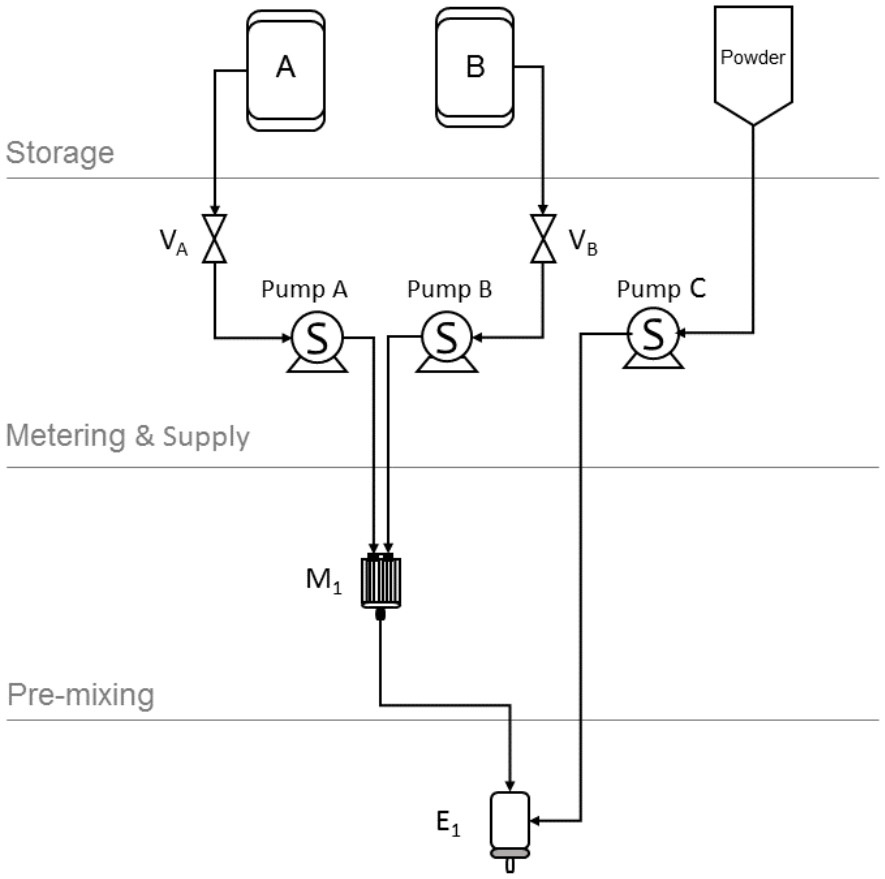
The AMfRP process flow concept, represented in Figure 5.1 is composed of four stages: storage of the materials; metering and supply; pre-mixer for liquids; and mixing head that is an extruder that has self-cleaning capabilities with a side feeder for powder inclusions. The liquid components are stored in tanks and gate valves will control the feed of the system; electric pumps will meter the necessary amount of liquid reagents to the pre-mixing head with jet impact at 90º, this mixture is then almost immediately injected into the double co-rotating screw extruder, where a side feeder includes powders into the stream. This system will have self-cleaning capabilities due to the fact that when the extruders inputs are turned off, the continuous running of the co-rotating screws will deplete all materials from within. Residence time is the main parameter to look out for in order to avoid blockages and expensive system clean-ups.

5.2 Storage of materials

There are many suppliers in the market place that can fulfill this need, being them standardized or custom built. In this case we have preferred that the storage units be part of the metering or delivery system to be purchased. This has only been represented in this manner to better understand the different stages of the process. Storage tanks A and B, with a capacity of 250 liters each, will be part of the closed system produced by Hennecke model Topline HT80; and the powder bin will be part of the system produced by Zamack model RES-2P/16A Explorer.

5.3 Metering and supply

Two different types of materials will need metering in this process: resins in the liquid state; and powder that has been previously dried and sifted. As in the previous section, the universe of applicable equipment's is vast and for this application the selection for the liquid materials is a system produced by Hennecke model Topline HT80, see Figure 5.2 for similar model



V_A	-	Component A, valve
V_B	-	Component B, valve
Pump A	-	Electronically controlled metering pump, stream A
Pump B	-	Electronically controlled metering pump, stream B
Pump C	-	Side feeder system controlled pump
M_1	-	Liquid mixing head
E_1	-	Mixing head/ Extruder – co-rotating double shafts

Figure 5.1: AMfRP Process flow diagram

HT500. This is a high-precision metering tandem plunger pump, that is wear resistant with output capabilities of 4.8 l/min in continuous operation, it handles high-viscosity raw materials, recycled polyols and abrasive fillers. It boasts advantages such as: adjustment to numerous requirements; continuous feed; discontinuous operation (single metering stroke); processing at high temperatures if needed; and processing of corrosive components. For this case specifically only the liquid feed and metering system will be in use. Machine specifications can be seen in Appendix K.

The selected system to feed the powder is a Zamack model RES-2P/16A Explorer, represented in Figure 5.3. Its a modular and portable system which can then be tailored with the supplier to be used on a suspension arm that will include both the side feeder and the extruder head. Due to its limited hopper size a wand supply system can be incorporated to

provide longer production cycles. Its data-sheet can be verified in Appendix L.

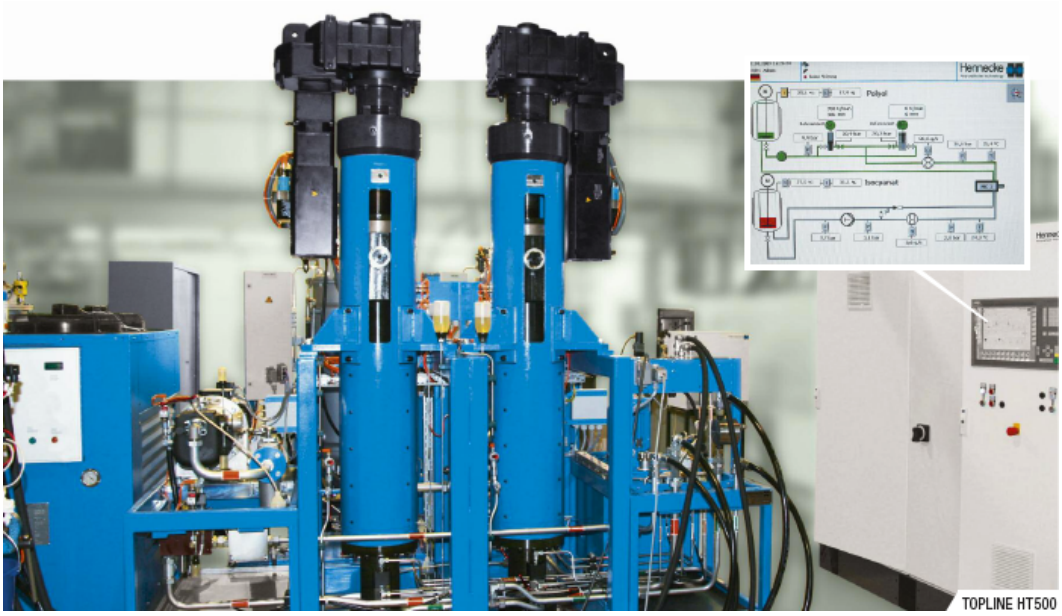


Figure 5.2: Hennecke Topline HT500 example

Figure 5.3: Zamack RES-2P/16A Explorer

5.4 Pre-mixing head for liquids

This section is a pre-mixing stage composed of a Hennecke MT 18 high-efficiency deflection mix-head, whose data-sheet is in Appendix M. It receives the metered liquid components and mixes them correctly and injects the result almost immediately into the next mixing head/extruder. This mixing head has a robust design that can manage output changes without injector adjustment, has the ability for small to large outputs (125 - 600 cm^3/s) and is fed via high-efficiency injectors. The reaction mix deflects into a 90° offset outlet pipe that supports the mixing process by calming the mixtures and ensuring a smooth discharge. Configuration of inlets at 90º were selected because they provide higher turbulence in the chamber, thus improving mixing [37]. MT mix-heads, shown in Figure 5.4 , are also particularly maintenance-friendly and noted for their extremely attractive life-cycle costs.

Figure 5.4: Example of Hennecke MT mix-head

5.5 Mixing/extrusion head

As Tiago Nunes concluded in his investigative work of Equipment development for thermoset and residue injection: twin inter-meshing co-rotating screws with self-cleaning abilities are the most adequate to provide homogeneous composite mixtures due to the ease of feeding and the high shear forces developed during function [24].

The mixing head will be a high pressure system in cooperation with co-rotational screws, preceded by a mixing chamber that receives the mixed liquids from the Pre-mixing head and incorporates the powders being delivered from the side feeder. The mixing chamber can be closed by applying hydraulic or pneumatic pressure to the cleaning fitting. When the fitting is activated it will reduce the chamber size up to the co-rotating screws, by sealing the inlet from the pre-mixer head and the side feeder. The control system of the cleaning fitting will function as follows when activated: first the side feeder is disabled; then the isocyanate feeding valve is cutoff to the pre-mixer; poloyol is then flushed through the pre-mixer and extruding head, being cutoff after a few seconds; then the cleaning fitting is engaged, blocking all input ports to the mixing chamber; and the extruder is then starve fed, kept running for enough time to deplete all material from within the barrels.

This process will avoid excessive pressure build up in the system and guarantee cleaning of the whole system by first cleaning the pre-mixer, then the mixing chamber and finally the screw barrels. Figure 5.5 is a sketch of the mixing/extrusion head.

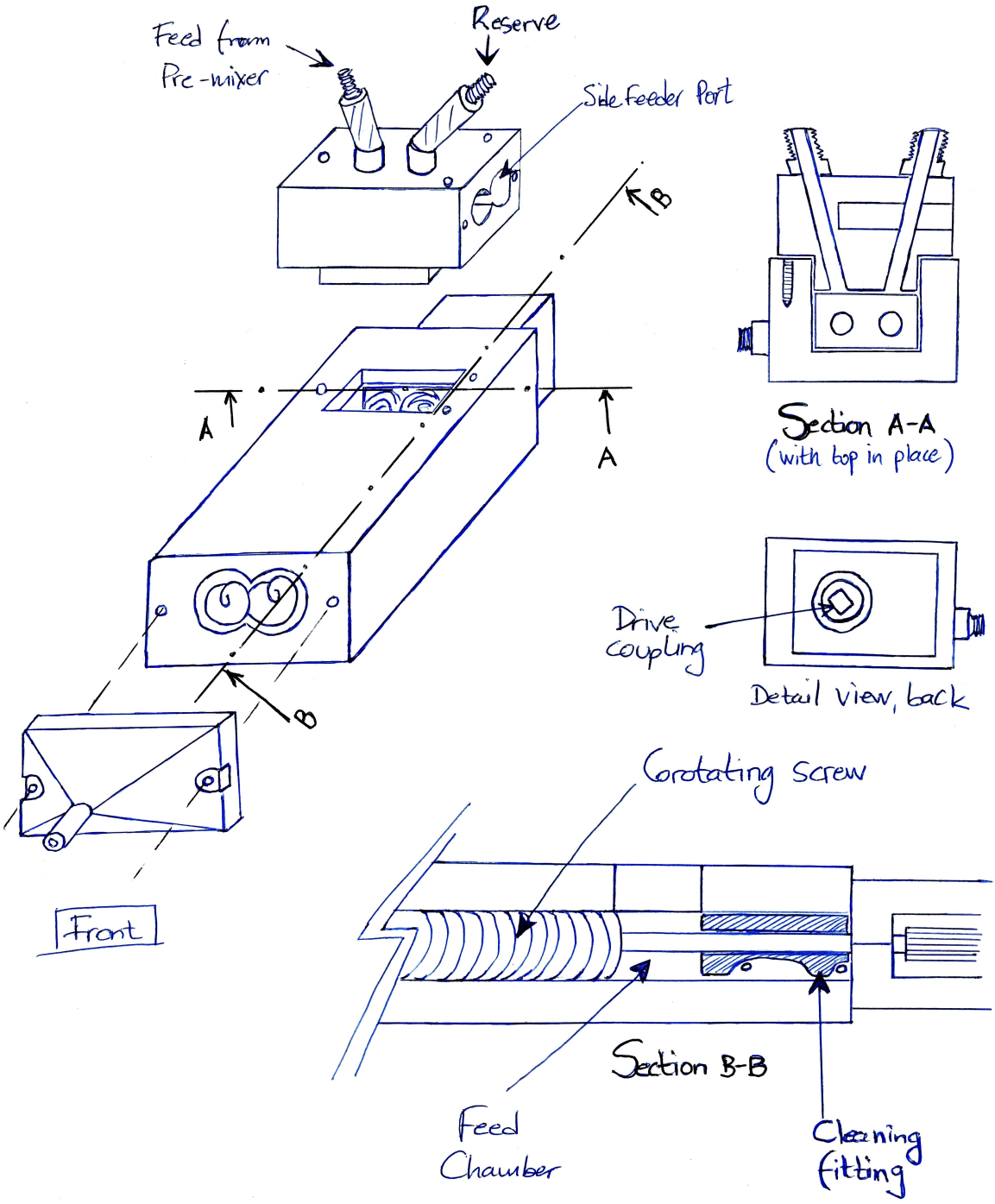


Figure 5.5: AMfRP extruder concept

6 Conclusions and further work

The initial scope of this research was to provide insight into the possibility of using additive manufacturing techniques with thermoset materials, with fillers at the highest possible concentrations. This document provides proof of that possibility, if material and equipment constraints are met. Among the diverse universe of reactive polymers, brand names and types, a selection fell on three that were available for testing. The results of this study show that, from the wide range of specifications of these materials and their initial formulations, a wide range of end products will be obtainable and that these can also be applied in a multitude of applications and situations.

It becomes clear that in conjunction with the developed extruding head, based on an existing concept, that the manufacturing technology is in existence, scattered in usage over several applications, that can be assembled in a manner to provide an equipment, the AMfRP, which is adjustable for a wide latitude of materials so that processing obtains the desired product output.

The results obtained at this date, are that in general filler additions lead to a decrement of the mechanical properties (lower stress, elongation at break and hardness than pure binder formulations). Losses of up to 61% were recorded for material hardness with few exceptions were binder/filler dependencies produce gains of 38%. Resulting materials present increases in porosity with higher filler content. Binders that have growth in their curing processes are not suitable for additive manufacturing processes that require defined form but do however present excellent possibilities as candidates for material functions such as fillers, gasket type materials or porous materials that require absorption or being light-weight as a material function.

This research apart from general conclusions also provide specific outcomes that have been summarized below:

- Biresin G26 presents promising results for AM and high filler inclusions. It maintains low viscosity after its components have been mixed, providing potlife windows of approximately 200 seconds for powder wetting and mixing prior to extruding. These potlife windows decrease with the increase of filler additions in the compound. Application of alternate hardeners will slice these cure times in half, providing possibility for higher speeds in processing equipment;

- Biresin G26 provides processing windows above 75% to 86% wt filler. Additions above these values need mechanical mixing and are probably possible but could not be tested in this work;

- Binders Pavistone 1K and Pavistone 2K are not suitable for AM processes that require defined form, due to their long potting time windows and because of their growth during curing;

- Pavistone 2k with filler additions below 41% wt, produce resulting materials that exceed pure binder formulations in at least 170%;

- Long post curing is required for Pavistone 1K and Pavistone 2K and the resultant products present an integral type skin composition;

- Addition of Fumed silica lowers filler incorporation approximately 10% but improves

considerably the mixtures thixotropy and caking effect. This trade-off may be advantageous depending on the desired end product;

- Fumed Silica improves compounds hardness, for mixtures of the same filler content. Silica seems to work like a binder;
- Biresin G26 compounds with 65% wt filler and added silica improved hardness by 38%.

Further work & Possible Applications

As this has been exploratory work, foundations are set for further study that may include: study of the cure rate with Reaction Calorimetry, layer adhesion and composition degradation due to water and/or salt water exposure. Other polymer substances as photopolymers are of great interest as they can provide almost instantaneous curing features with no impact on mixing. The equipment would need the addition of an Ultra-violet light source at its extruding tip, providing AM capabilities and precision manufacturing to the system.

In depth study for Pavistone 2k with filler additions below 41% wt, producing resulting materials that exceed pure binder formulations in at least 170%.

The high content of air in several of the composites and their good hardness resistance makes them good candidates for usage as a buoyancy components. For situations like the novel floating housing systems appearing in the Netherlands, Ijburg neighborhood, where giant concrete foundations are produced and then filled with styrofoam. These composites could be a replacement for the styrofoam, with the advantage of being able to produce these in a modular manner and with minimal usage of binder. Being encapsulated in a laminar concrete housing these would resist corrosion from water attack and provide a larger footprint of sustainability as wastes are re-used and other polymer non-environmental friendly materials are no longer applied.

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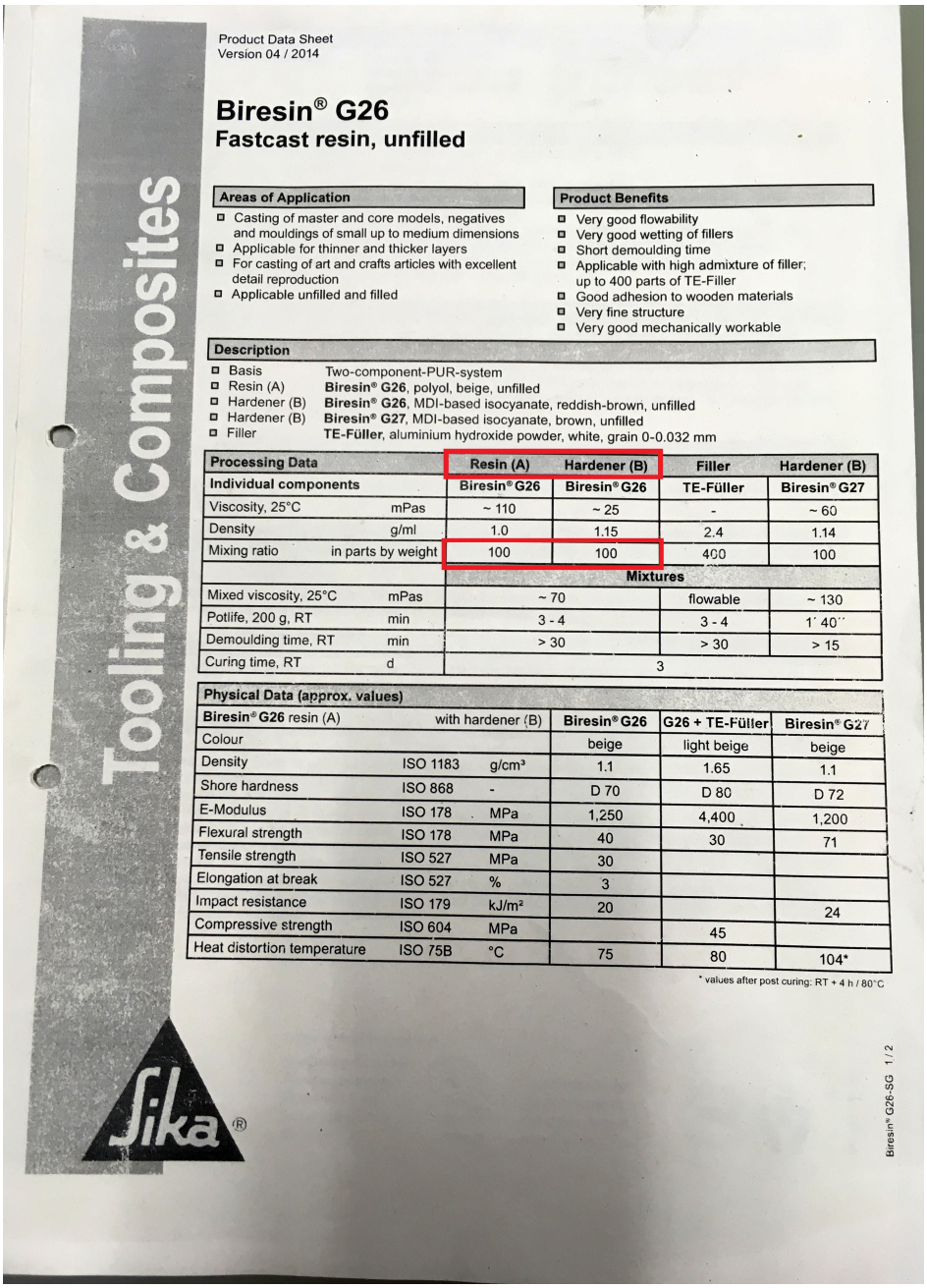
Appendix A: Data sheets of materials

A.1 Biresin G26 data sheet



Tooling & Composites

Product Data Sheet
Version 04 / 2014

Biresin® G26
Fastcast resin, unfilled

Areas of Application
- Casting of master and core models, negatives and mouldings of small up to medium dimensions
- Applicable for thinner and thicker layers
- For casting of art and crafts articles with excellent detail reproduction
- Applicable unfilled and filled

Product Benefits
- Very good flowability
- Very good wetting of fillers
- Short demoulding time
- Applicable with high admixture of filler; up to 400 parts of TE-Filler
- Good adhesion to wooden materials
- Very fine structure
- Very good mechanically workable

Description

Basis	Two-component-PUR-system
Resin (A)	**Biresin® G26**, polyol, beige, unfilled
Hardener (B)	**Biresin® G26**, MDI-based isocyanate, reddish-brown, unfilled
Hardener (B)	**Biresin® G27**, MDI-based isocyanate, brown, unfilled
Filler	**TE-Füller**, aluminium hydroxide powder, white, grain 0-0.032 mm

Processing Data		Resin (A)	Hardener (B)	Filler	Hardener (B)
Individual components		Biresin® G26	Biresin® G26	TE-Füller	Biresin® G27
Viscosity, 25°C	mPas	~ 110	~ 25	-	~ 60
Density	g/ml	1.0	1.15	2.4	1.14
Mixing ratio	in parts by weight	100	100	400	100
		Mixtures			
Mixed viscosity, 25°C	mPas	~ 70		flowable	~ 130
Potlife, 200 g, RT	min	3 - 4		3 - 4	1´ 40´´
Demoulding time, RT	min	> 30		> 30	> 15
Curing time, RT	d	3			

Physical Data (approx. values)					
Biresin® G26 resin (A) with hardener (B)			Biresin® G26	G26 + TE-Füller	Biresin® G27
Colour			beige	light beige	beige
Density	ISO 1183	g/cm³	1.1	1.65	1.1
Shore hardness	ISO 868	-	D 70	D 80	D 72
E-Modulus	ISO 178	MPa	1,250	4,400	1,200
Flexural strength	ISO 178	MPa	40	30	71
Tensile strength	ISO 527	MPa	30		
Elongation at break	ISO 527	%	3		
Impact resistance	ISO 179	kJ/m²	20		24
Compressive strength	ISO 604	MPa		45	
Heat distortion temperature	ISO 75B	°C	75	80	104*

* values after post curing: RT + 4 h / 80°C

Sika®

Biresin® G26-SG 1 / 2

A.2 Pavistone 1K data sheet

PAVISTONE 1K

Aliphatic polyurethane binder

DESCRIPTION AND APPLICATIONS

Pavistone 1K is a polyurethane aggregate binder for pavements that gives for a smooth floor, modern, tough, low maintenance, porous or semi porous finish, depending on the type of aggregates used.
The surface finish is a seamless, flexible and resistant to cracking floor.
Product is colourless.

APPLICATIONS

- Paths
- Parking decks
- Bike lanes
- Fences
- Ramps
- Pedestrian areas
- Parks
- Commercial areas
- Roads
- Footbridges
- Residential areas

TECHNICAL DATA

INFORMATION ON THE PRODUCT BEFORE APPLICATION	
Chemical description	Solventless aliphatic polyisocyanate
Physical state	Liquid
Packaging	Metal container 5kg 25 kg
Non-volatile content (%)	100%
Flash point	100ºC
Colour	Whitish
Density	

Temp (ºC)	Density (g/cm3)
23	1.12

Viscosity

approximate Brookfield

Temp (ºC)	Viscosity (mPa.s)
15	2680
20	1940
25	1500

Pot life approximate

Conditions (100g filter+5g resin)	Pot life(min)
20ºC, 40% hr	90-120

Storage	Keep between 10º y 30ºC, protected from moisture.
Use before	12 months after manufacturing date.

INFORMATION ON THE FINAL PRODUCT	
Final state	Elastomeric solid binder
Colour	Colourless
Solid density	1,10-1,15 g/cm3 g/cm3
Hardness (shore)	50D
Mechanical properties	Elongation at break: 30% Tensile strength: 24 Mpa
UV resistance	Colour stable under sunlight
Water absorption	Very low (6 days, 20ºC)
Chemical resistance	Surface contact (24 hours, room temperatur, 5=ok, 0= not recommended)

Chemical	Result
White Spirit	5
Coffee	5
Isopropyl alcohol	5
Methoxypropyl acetate	5
Petrol/gasoline	5
Xylene	5
Sodium hydroxide (saturate)	5
Ethanol	4
Bleach	5
Trichloroisocyanuric acid	5
Formaline	5
Lubricant oil	5
Hydrogen peroxide	4
Acetic acid (10%)	2
Sulphuric Acid (30%)	1
Skydrol	5
Ammonia (3%)	5
Diesel	5

SUPPORT REQUIREMENTS

In order to achieve a good bonding, support must be:
1. Flat and levelled
2. Compact and cohesive (pull off test must show a minimum resistance of 1,5 N/mm2).
3. Even and regular surface
4. Free from cracks and fissures. If any, they must be previously repaired.
5. Clean and dry, free of dust, loose particles, oils, organic residues or laitance.

Asphalt supports must be clean and dry. For more information on treatment of critical spots, consult our technical service.
Edges of the application can be finished with brick, stone, concrete, for a high quality finish.

RECOMMENDED AMBIENTAL CONDITIONS

Support temperature should be between 10ºC and 25ºC. At higher temperatures, specific precautionary measures must be taken. At lower temperatures, curing is very slow. Please follow manufacturer advice.
Support moisture should be less than 4%.
High temperature and moisture conditions can lead to bubbling/foaming.
Preferred air conditions are 10-30ºC and 30-80% rh

RECOMMENDED COMBINATIONS

Aggregate/Pavistone 1K ratio is as follws

Aggregate type	Pavistone % (A+B)
Regular, smooth, big stone	3 to 5%
Small particles, porous, irregular sizes	5 to 7%

An advisable practice is to seal the upper surface with a thin coat of pure Pavistone resin in order to prevent surface wearing off

APPLICATION

Homogeneize completely by gentle stirring before use.
After mixing, Pavistone 1k is added to the aggregate mass, using a sutable mechanical mixer. Mix for 2 minutes and spread immediately on the application site. It is important to wet throughly all the solids for the same length of time each batch in order to prevent colour differences. See pot life data for details.

Spread evenly at the desired thickness on the surface using a flat spreader and press gently to obtain a smooth and compact surface.

Use the following table as a guide for consumption estimations.

Agregate	Desired pavement thickness (mm)	Pavistone + stone consumption kg/m2
6 to 10 mm	15	30
	20	40
	25	50
	30	60

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A.3 Pavistone 2K data sheet

PAVISTONE 2K

Two component polyurethane binder for construction applications

DESCRIPTION

2-component, clear polyurethane resin, suitable for use as a aggregate stone binder. Designed for use as a binder for pavement applications that allows for a smooth floor, modern, tough, low maintenance, porous or semi-porous finish, depending on the type of aggregates used. The surface finish is a seamless, hard and resistant.

TECHNICAL DATA

PRODUCT INFORMATION BEFORE APPLICATION

	Component A	Component B
Chemical description	Polyol mixture	Solventless polyisocyanate
Physical State	Liquid	Liquid
Presentation	Metal container 192kg x 3 4.9 kg	Metal container 250kg 2.1 kg
Non volatile content (%)	approx 100	100
Flash point	>100ºC	>100ºC
Colour	Yellow	dark brown

Density

Temperature (ºC)	*Density (g/cm³)*	*Temperature (ºC)*	*Density (g/cm³)*
25	1,01	25	1,23

Viscosity Approximate values, Brookfield

Temperature (ºC)	*Viscosity (mPa.s)*	*Temperature (ºC)*	*Viscosity (mPa.s)*
15	6000	25	200
25	2800		
35	800		

A/B mixing ratio	A=100, B=43,5 by weight A=100, B= 36.6 by volume
Initial mixture properties	Viscosity: 2000 mPa.s (25ºC) Colour: milky yellow
Time of processing At 25ºC	20 minutes

Pot life

Conditions (100g)	*Pot life (min)*
22ºC,40% rh	35

Storage	Keep between 10ºC and 30ºC protected from moisture.
Use before	12 months after manufacturing date, in its unopened container

INFORMATION ON THE FINAL PRODUCT

Final State	Solid Polyurethane
Colour	Clear yellow
Hardness (Shore)	65-70D
Mechanical Properties	Maximum elongation: <10% Maximum tensile strength: 12 MPa
UV Resistance	Pavistone 2k changes colour upon sunlight without impairment of its mechanical properties.
Temperatures of use	Stable between -15ºC and 80ºC.

SUPPORT REQUIREMENTS

In order to achieve a good bonding, support must be:
1. Flat and leveled
2. Compact and cohesive (pull off test must show a minimum resistance of 1,5 N/mm2).
3. Even and regular surface
4. Free from cracks and fissures. If any, they must be previously repaired.
5. Clean and dry, free of dust, loose particles, oils, organic residues or laitance.

Asphalt supports must be clean and dry. For more information on treatment of critical spots, consult our technical service.
Edges of the application can be finished with brick, stone, concrete, for a high quality finish.

RECOMMENDED ENVIRONMENTAL CONDITIONS

Support temperature should be between 10ºC and 25ºC. At higher temperatures, specific precautionary measures must be taken. At lower temperatures, curing is very slow. Please follow manufacturer advice.

Support moisture should be less than 4%.

High temperature and moisture conditions can lead to bubbling/foaming. Preferred air conditions are 10-30ºC and 30-80% rh

RECOMMENDED COMBINATIONS

Aggregate/Pavistone 2k ratio is as follws

Aggregate type	*Pavistone % (A+B)*
Regular, smooth, big stone	3 to 5 %
Small particles, porous, irregular sizes	5 to 7%

An advisable practice is to seal the upper surface with a thin coat of pure Pavistone2k resin in order to prevent surface wearing off.

APPLICATION GUIDELINES

Homogeneize completely by gentle stirring before use.

After mixing, Pavistone 2k is added to the aggregate mass, using a sutable mechanical mixer. Mix for 2 minutes and spread immediately on the application site. It is important to wet throughly all the solids for the same length of time each batch in order to prevent colour differences. See pot life data for details.

Spread evenly at the desired thickness on the surface using a flat spreader and press gently to obtain a smooth and compact surface.

Use the following table as a guide for consumption estimations.

Aggregate size	*Desired pavement thickness (mm)*	*Pavistone+stone consumption kg/m2*
6 to 10 mm	15	30
	20	40
	25	50
	30	60

Some aggregates contain a certain proportion of finer sands that impair adhesion of the main components. Use clean materials with suitable particle distribution.

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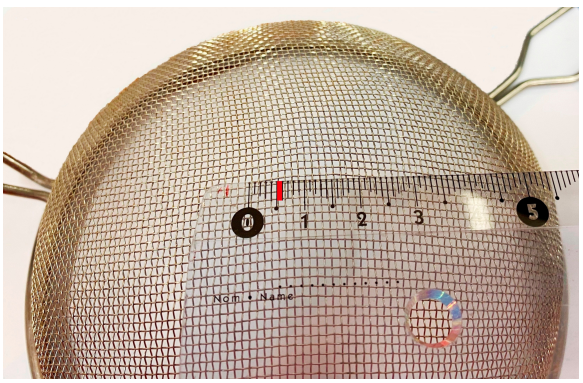
Appendix B: Scale and Sift used for tests



Appendix C: Data sheet of Fumed Silica

WACKER

CREATING TOMORROW'S SOLUTIONS

HDK® N20

HDK®

Pyrogenic Silica

Synthetic, hydrophilic, amorphous silica, produced via flame hydrolysis. Standard product for industrial applications.

INCI Silica

Properties

White colloidal powder of high purity.

Technical data

Specification

Property	Condition	Value	Method
BET surface	-	175 - 225 m^2/g	DIN ISO 9277 DIN 66132
Tamped density	-	approx. 40 g/l	DIN EN ISO 787-11
pH	-	3.8 - 4.3	DIN EN ISO 787-9
Sieve residue[1]	-	< 0.03 %	DIN EN ISO 787-18
Loss on drying[2]	-	< 1.5 %	DIN EN ISO 787-2

[1] acc. to Mocker > 40 µm
[2] ex works (2 h at 105 °C)

General Characteristics

Property	Condition	Value	Method
Density[1]	20 °C	approx. 2.2 g/cm^3	DIN 51757
INCI name	-	Silica	-
Loss of weight[2]	-	< 2 %	DIN EN ISO 3262-19
Refraction index	-	1.46	-
SiO_2 content[3]	-	> 99.8 %	DIN EN ISO 3262-19
Silanol group density	-	2 SiOH/nm^2	-

[1]SiO2
[2]at 1000 °C / 2h (based on the substance dried at 105 °C for 2 h)
[3]based on the substance heated at 1000 °C for 2 h

These figures are only intended as a guide and should not be used in preparing specifications.

Applications

- Toners
- Insulation Materials
- Architectural Coatings
- Batteries
- Industrial Coatings
- Composites & Molding
- Personal Care
- Offset Printing

Application details

HDK® N20 is applied as a thickening and thixotropic agent in many organic systems, e.g. in unsaturated polyesters, coatings, printing inks, adhesives, cosmetics and others.
HDK® N20 is used as a reinforcing filler in elastomers, mainly silicone-elastomers.
HDK® N20 acts as a free flow additive in the production of technical powders.
HDK® N20 is not suitable for pharmaceuticals, food and feed.
A good dispersion of HDK® N20 is a must to assure optimum performance.
More detailed information about the application and processing ofHDK® N20 is available in our HDK-brochures and on the WACKER web site.

Appendix D: G-Code's for aEDP printer

D.1 G-Code for 1st extruded String

```
;FLAVOR:Ma3rlin
M82 ;absolute extrusion mode

G21 ;mm format
G92 E0 ;Prime the extruder
G28 ;Machine Home

;String1 Prep
G0 F3000 X0 Y20 Z5
G1 F300 X5 Y20 Z1 E1
G1 F300 X8 Y20 Z2,5 E2
G1 F300 X9 Y20 Z2 E2

;String1 extrusion
G92 E0 ;Prime the extruder
G1 F240 X10,0 Y20 Z1,5 E0,0
G1 F240 X11,0 Y20 Z1,5 E0,1
G1 F240 X11,5 Y20 Z1,5 E0,0
G1 F240 X12,0 Y20 Z1,5 E0,2
G1 F240 X12,5 Y20 Z1,5 E0,1
G1 F240 X13,0 Y20 Z1,5 E0,3
G1 F240 X13,5 Y20 Z1,5 E0,2
G1 F240 X14,0 Y20 Z1,5 E0,4
G1 F240 X14,5 Y20 Z1,5 E0,3
G1 F240 X15,0 Y20 Z1,5 E0,5
G1 F240 X15,5 Y20 Z1,5 E0,4
G1 F240 X16,0 Y20 Z1,5 E0,6
G1 F240 X16,5 Y20 Z1,5 E0,5
G1 F240 X17,0 Y20 Z1,5 E0,7
G1 F240 X17,5 Y20 Z1,5 E0,6
G1 F240 X18,0 Y20 Z1,5 E0,8
G1 F240 X18,5 Y20 Z1,5 E0,7
G1 F240 X19,0 Y20 Z1,5 E0,9
G1 F240 X19,5 Y20 Z1,5 E0,8
G1 F240 X20,0 Y20 Z1,5 E1,0
G1 F240 X20,5 Y20 Z1,5 E0,9
G1 F240 X21,0 Y20 Z1,5 E1,1
G1 F240 X21,5 Y20 Z1,5 E1,0
G1 F240 X22,0 Y20 Z1,5 E1,2
G1 F240 X22,5 Y20 Z1,5 E1,1
G1 F240 X23,0 Y20 Z1,5 E1,3
G1 F240 X23,5 Y20 Z1,5 E1,2
G1 F240 X24,0 Y20 Z1,5 E1,4
G1 F240 X24,5 Y20 Z1,5 E1,3
G1 F240 X25,0 Y20 Z1,5 E1,5
G1 F240 X25,5 Y20 Z1,5 E1,4
G1 F240 X26,0 Y20 Z1,5 E1,6
G1 F240 X26,5 Y20 Z1,5 E1,5
G1 F240 X27,0 Y20 Z1,5 E1,7
G1 F240 X27,5 Y20 Z1,5 E1,6
G1 F240 X28,0 Y20 Z1,5 E1,8
G1 F240 X28,5 Y20 Z1,5 E1,7
G1 F240 X29,0 Y20 Z1,5 E1,9
G1 F240 X29,5 Y20 Z1,5 E1,8
G1 F240 X30,0 Y20 Z1,5 E2,0
G1 F240 X30,5 Y20 Z1,5 E1,9
G1 F240 X31,0 Y20 Z1,5 E2,1
G1 F240 X31,5 Y20 Z1,5 E2,0
G1 F240 X32,0 Y20 Z1,5 E2,2
G1 F240 X32,5 Y20 Z1,5 E2,1
G1 F240 X33,0 Y20 Z1,5 E2,3
G1 F240 X33,5 Y20 Z1,5 E2,2
G1 F240 X34,0 Y20 Z1,5 E2,4
G1 F240 X34,5 Y20 Z1,5 E2,3
G1 F240 X35,0 Y20 Z1,5 E2,5
G1 F240 X35,5 Y20 Z1,5 E2,4
G1 F240 X36,0 Y20 Z1,5 E2,6
G1 F240 X36,5 Y20 Z1,5 E2,5
G1 F240 X37,0 Y20 Z1,5 E2,7
G1 F240 X37,5 Y20 Z1,5 E2,6
G1 F240 X38,0 Y20 Z1,5 E2,8
G1 F240 X38,5 Y20 Z1,5 E2,7
G1 F240 X39,0 Y20 Z1,5 E2,9
G1 F240 X39,5 Y20 Z1,5 E2,8
G1 F240 X40,0 Y20 Z1,5 E3,0
G1 F240 X40,5 Y20 Z1,5 E2,9
G1 F240 X41,0 Y20 Z1,5 E3,1
G1 F240 X41,5 Y20 Z1,5 E3,0
G1 F240 X42,0 Y20 Z1,5 E3,2
G1 F240 X42,5 Y20 Z1,5 E3,1
G1 F240 X43,0 Y20 Z1,5 E3,3
G1 F240 X43,5 Y20 Z1,5 E3,2
G1 F240 X44,0 Y20 Z1,5 E3,4
G1 F240 X44,5 Y20 Z1,5 E3,3
G1 F240 X45,0 Y20 Z1,5 E3,5
G1 F240 X45,5 Y20 Z1,5 E3,4
G1 F240 X46,0 Y20 Z1,5 E3,6
G1 F240 X46,5 Y20 Z1,5 E3,5
G1 F240 X47,0 Y20 Z1,5 E3,7
G1 F240 X47,5 Y20 Z1,5 E3,6
G1 F240 X48,0 Y20 Z1,5 E3,8
G1 F240 X48,5 Y20 Z1,5 E3,7
G1 F240 X49,0 Y20 Z1,5 E3,9
G1 F240 X49,5 Y20 Z1,5 E3,8
G1 F240 X50,0 Y20 Z1,5 E4,0
G1 F240 X50,5 Y20 Z1,5 E3,9
G1 F240 X51,0 Y20 Z1,5 E4,1
G1 F240 X51,5 Y20 Z1,5 E4,0
G1 F240 X52,0 Y20 Z1,5 E4,2
G1 F240 X52,5 Y20 Z1,5 E4,1
G1 F240 X53,0 Y20 Z1,5 E4,3
G1 F240 X53,5 Y20 Z1,5 E4,2
G1 F240 X54,0 Y20 Z1,5 E4,4
G1 F240 X54,5 Y20 Z1,5 E4,3
G1 F240 X55,0 Y20 Z1,5 E4,5
G1 F240 X55,5 Y20 Z1,5 E4,4
G1 F240 X56,0 Y20 Z1,5 E4,6
G1 F240 X56,5 Y20 Z1,5 E4,5
G1 F240 X57,0 Y20 Z1,5 E4,7
G1 F240 X57,5 Y20 Z1,5 E4,6
G1 F240 X58,0 Y20 Z1,5 E4,8
G1 F240 X58,5 Y20 Z1,5 E4,7
G1 F240 X59,0 Y20 Z1,5 E4,9
G1 F240 X59,5 Y20 Z1,5 E4,8
G1 F240 X60,0 Y20 Z1,5 E5,0
G1 F240 X60,5 Y20 Z1,5 E4,9
G1 F240 X61,0 Y20 Z1,5 E5,1
G1 F240 X61,5 Y20 Z1,5 E5,0
G1 F240 X62,0 Y20 Z1,5 E5,2
G1 F240 X62,5 Y20 Z1,5 E5,1
G1 F240 X63,0 Y20 Z1,5 E5,3
G1 F240 X63,5 Y20 Z1,5 E5,2
G1 F240 X64,0 Y20 Z1,5 E5,4
G1 F240 X64,5 Y20 Z1,5 E5,3
G1 F240 X65,0 Y20 Z1,5 E5,5
G1 F240 X65,5 Y20 Z1,5 E5,4
G1 F240 X66,0 Y20 Z1,5 E5,6
G1 F240 X66,5 Y20 Z1,5 E5,5
G1 F240 X67,0 Y20 Z1,5 E5,7
G1 F240 X67,5 Y20 Z1,5 E5,6
G1 F240 X68,0 Y20 Z1,5 E5,8
G1 F240 X68,5 Y20 Z1,5 E5,7
G1 F240 X69,0 Y20 Z1,5 E5,9
G1 F240 X69,5 Y20 Z1,5 E5,8
G1 F240 X70,0 Y20 Z1,5 E6,0
G1 F240 X70,5 Y20 Z1,5 E5,9
G1 F240 X71,0 Y20 Z1,5 E6,1
G1 F240 X71,5 Y20 Z1,5 E6,0
G1 F240 X72,0 Y20 Z1,5 E6,2
G1 F240 X72,5 Y20 Z1,5 E6,1
G1 F240 X73,0 Y20 Z1,5 E6,3
G1 F240 X73,5 Y20 Z1,5 E6,2
G1 F240 X74,0 Y20 Z1,5 E6,4
G1 F240 X74,5 Y20 Z1,5 E6,3
G1 F240 X75,0 Y20 Z1,5 E6,5
G1 F240 X75,5 Y20 Z1,5 E6,4
G1 F240 X76,0 Y20 Z1,5 E6,6
G1 F240 X76,5 Y20 Z1,5 E6,5
G1 F240 X77,0 Y20 Z1,5 E6,7
G1 F240 X77,5 Y20 Z1,5 E6,6
G1 F240 X78,0 Y20 Z1,5 E6,8
G1 F240 X78,5 Y20 Z1,5 E6,7
G1 F240 X79,0 Y20 Z1,5 E6,9
G1 F240 X79,5 Y20 Z1,5 E6,8
G1 F240 X80,0 Y20 Z1,5 E7,0
G1 F240 X80,5 Y20 Z1,5 E6,9
G1 F240 X81,0 Y20 Z1,5 E7,1
G1 F240 X81,5 Y20 Z1,5 E7,0
G1 F240 X82,0 Y20 Z1,5 E7,2
G1 F240 X82,5 Y20 Z1,5 E7,1
G1 F240 X83,0 Y20 Z1,5 E7,3
G1 F240 X83,5 Y20 Z1,5 E7,2
G1 F240 X84,0 Y20 Z1,5 E7,4
G1 F240 X84,5 Y20 Z1,5 E7,3
G1 F240 X85,0 Y20 Z1,5 E7,5
G1 F240 X85,5 Y20 Z1,5 E7,4
G1 F240 X86,0 Y20 Z1,5 E7,6
G1 F240 X86,5 Y20 Z1,5 E7,5
G1 F240 X87,0 Y20 Z1,5 E7,7
G1 F240 X87,5 Y20 Z1,5 E7,6
G1 F240 X88,0 Y20 Z1,5 E7,8
G1 F240 X88,5 Y20 Z1,5 E7,7
G1 F240 X89,0 Y20 Z1,5 E7,9
G1 F240 X89,5 Y20 Z1,5 E7,8
G1 F240 X90,0 Y20 Z1,5 E8,0
G1 F240 X90,5 Y20 Z1,5 E7,9
G1 F240 X91,0 Y20 Z1,5 E8,1
G1 F240 X91,5 Y20 Z1,5 E8,0
G1 F240 X92,0 Y20 Z1,5 E8,2
G1 F240 X92,5 Y20 Z1,5 E8,1
G1 F240 X93,0 Y20 Z1,5 E8,3
G1 F240 X93,5 Y20 Z1,5 E8,2
G1 F240 X94,0 Y20 Z1,5 E8,4
G1 F240 X94,5 Y20 Z1,5 E8,3
G1 F240 X95,0 Y20 Z1,5 E8,5
G1 F240 X95,5 Y20 Z1,5 E8,4
G1 F240 X96,0 Y20 Z1,5 E8,6
G1 F240 X96,5 Y20 Z1,5 E8,5
G1 F240 X97,0 Y20 Z1,5 E8,7
G1 F240 X97,5 Y20 Z1,5 E8,6
G1 F240 X98,0 Y20 Z1,5 E8,8
G1 F240 X98,5 Y20 Z1,5 E8,7
G1 F240 X99,0 Y20 Z1,5 E8,9
G1 F240 X99,5 Y20 Z1,5 E8,8
G1 F240 X100 Y20 Z1,5 E9

;Stop Process
G1 F240 X100 Y25 Z1,5 E7

;Prep for string2
G0 F3000 X0 Y35 Z5

M82 ;absolute extrusion mode
;End of Gcode
```

D.2 G-Code for 2nd extruded String

```
;FLAVOR:Ma3rlin
M82 ;absolute extrusion mode

G21 ;mm format
G92 X0 Y35 Z5 E0 ;Prime the
extruder

;String2 Prep
G0 F3000 X0 Y40 Z5
G1 F300 X5 Y40 Z1 E1
G1 F300 X8 Y40 Z3 E2
G1 F300 X9 Y40 Z2,5 E2

;String2 extrusion (2mm)
G92 E0 ;Prime the extruder
G1 F240 X10,0 Y40 Z2 E0,0
G1 F240 X11,0 Y40 Z2 E0,1
G1 F240 X11,5 Y40 Z2 E0,0
G1 F240 X12,0 Y40 Z2 E0,2
G1 F240 X12,5 Y40 Z2 E0,1
G1 F240 X13,0 Y40 Z2 E0,3
G1 F240 X13,5 Y40 Z2 E0,2
G1 F240 X14,0 Y40 Z2 E0,4
G1 F240 X14,5 Y40 Z2 E0,3
G1 F240 X15,0 Y40 Z2 E0,5
G1 F240 X15,5 Y40 Z2 E0,4
G1 F240 X16,0 Y40 Z2 E0,6
G1 F240 X16,5 Y40 Z2 E0,5
G1 F240 X17,0 Y40 Z2 E0,7
G1 F240 X17,5 Y40 Z2 E0,6
G1 F240 X18,0 Y40 Z2 E0,8
G1 F240 X18,5 Y40 Z2 E0,7
G1 F240 X19,0 Y40 Z2 E0,9
G1 F240 X19,5 Y40 Z2 E0,8
G1 F240 X20,0 Y40 Z2 E1,0
G1 F240 X20,5 Y40 Z2 E0,9
G1 F240 X21,0 Y40 Z2 E1,1
G1 F240 X21,5 Y40 Z2 E1,0
G1 F240 X22,0 Y40 Z2 E1,2
G1 F240 X22,5 Y40 Z2 E1,1
G1 F240 X23,0 Y40 Z2 E1,3
G1 F240 X23,5 Y40 Z2 E1,2
G1 F240 X24,0 Y40 Z2 E1,4
G1 F240 X24,5 Y40 Z2 E1,3
G1 F240 X25,0 Y40 Z2 E1,5
G1 F240 X25,5 Y40 Z2 E1,4
G1 F240 X26,0 Y40 Z2 E1,6
G1 F240 X26,5 Y40 Z2 E1,5
G1 F240 X27,0 Y40 Z2 E1,7
G1 F240 X27,5 Y40 Z2 E1,6
G1 F240 X28,0 Y40 Z2 E1,8
G1 F240 X28,5 Y40 Z2 E1,7
G1 F240 X29,0 Y40 Z2 E1,9
G1 F240 X29,5 Y40 Z2 E1,8
G1 F240 X30,0 Y40 Z2 E2,0
G1 F240 X30,5 Y40 Z2 E1,9
G1 F240 X31,0 Y40 Z2 E2,1
G1 F240 X31,5 Y40 Z2 E2,0
G1 F240 X32,0 Y40 Z2 E2,2
G1 F240 X32,5 Y40 Z2 E2,1
G1 F240 X33,0 Y40 Z2 E2,3
G1 F240 X33,5 Y40 Z2 E2,2
G1 F240 X34,0 Y40 Z2 E2,4
G1 F240 X34,5 Y40 Z2 E2,3
G1 F240 X35,0 Y40 Z2 E2,5
G1 F240 X35,5 Y40 Z2 E2,4
G1 F240 X36,0 Y40 Z2 E2,6
G1 F240 X36,5 Y40 Z2 E2,5
G1 F240 X37,0 Y40 Z2 E2,7
G1 F240 X37,5 Y40 Z2 E2,6
G1 F240 X38,0 Y40 Z2 E2,8
G1 F240 X38,5 Y40 Z2 E2,7
G1 F240 X39,0 Y40 Z2 E2,9
G1 F240 X39,5 Y40 Z2 E2,8
G1 F240 X40,0 Y40 Z2 E3,0
G1 F240 X40,5 Y40 Z2 E2,9
G1 F240 X41,0 Y40 Z2 E3,1
G1 F240 X41,5 Y40 Z2 E3,0
G1 F240 X42,0 Y40 Z2 E3,2
G1 F240 X42,5 Y40 Z2 E3,1
G1 F240 X43,0 Y40 Z2 E3,3
G1 F240 X43,5 Y40 Z2 E3,2
G1 F240 X44,0 Y40 Z2 E3,4
G1 F240 X44,5 Y40 Z2 E3,3
G1 F240 X45,0 Y40 Z2 E3,5
G1 F240 X45,5 Y40 Z2 E3,4
G1 F240 X46,0 Y40 Z2 E3,6
G1 F240 X46,5 Y40 Z2 E3,5
G1 F240 X47,0 Y40 Z2 E3,7
G1 F240 X47,5 Y40 Z2 E3,6
G1 F240 X48,0 Y40 Z2 E3,8
G1 F240 X48,5 Y40 Z2 E3,7
G1 F240 X49,0 Y40 Z2 E3,9
G1 F240 X49,5 Y40 Z2 E3,8
G1 F240 X50,0 Y40 Z2 E4,0
G1 F240 X50,5 Y40 Z2 E3,9
G1 F240 X51,0 Y40 Z2 E4,1
G1 F240 X51,5 Y40 Z2 E4,0
G1 F240 X52,0 Y40 Z2 E4,2
G1 F240 X52,5 Y40 Z2 E4,1
G1 F240 X53,0 Y40 Z2 E4,3
G1 F240 X53,5 Y40 Z2 E4,2
G1 F240 X54,0 Y40 Z2 E4,4
G1 F240 X54,5 Y40 Z2 E4,3
G1 F240 X55,0 Y40 Z2 E4,5
G1 F240 X55,5 Y40 Z2 E4,4
G1 F240 X56,0 Y40 Z2 E4,6
G1 F240 X56,5 Y40 Z2 E4,5
G1 F240 X57,0 Y40 Z2 E4,7
G1 F240 X57,5 Y40 Z2 E4,6
G1 F240 X58,0 Y40 Z2 E4,8
G1 F240 X58,5 Y40 Z2 E4,7
G1 F240 X59,0 Y40 Z2 E4,9
G1 F240 X59,5 Y40 Z2 E4,8
G1 F240 X60,0 Y40 Z2 E5,0
G1 F240 X60,5 Y40 Z2 E4,9
G1 F240 X61,0 Y40 Z2 E5,1
G1 F240 X61,5 Y40 Z2 E5,0
G1 F240 X62,0 Y40 Z2 E5,2
G1 F240 X62,5 Y40 Z2 E5,1
G1 F240 X63,0 Y40 Z2 E5,3
G1 F240 X63,5 Y40 Z2 E5,2
G1 F240 X64,0 Y40 Z2 E5,4
G1 F240 X64,5 Y40 Z2 E5,3
G1 F240 X65,0 Y40 Z2 E5,5
G1 F240 X65,5 Y40 Z2 E5,4
G1 F240 X66,0 Y40 Z2 E5,6
G1 F240 X66,5 Y40 Z2 E5,5
G1 F240 X67,0 Y40 Z2 E5,7
G1 F240 X67,5 Y40 Z2 E5,6
G1 F240 X68,0 Y40 Z2 E5,8
G1 F240 X68,5 Y40 Z2 E5,7
G1 F240 X69,0 Y40 Z2 E5,9
G1 F240 X69,5 Y40 Z2 E5,8
G1 F240 X70,0 Y40 Z2 E6,0
G1 F240 X70,5 Y40 Z2 E5,9
G1 F240 X71,0 Y40 Z2 E6,1
G1 F240 X71,5 Y40 Z2 E6,0
G1 F240 X72,0 Y40 Z2 E6,2
G1 F240 X72,5 Y40 Z2 E6,1
G1 F240 X73,0 Y40 Z2 E6,3
G1 F240 X73,5 Y40 Z2 E6,2
G1 F240 X74,0 Y40 Z2 E6,4
G1 F240 X74,5 Y40 Z2 E6,3
G1 F240 X75,0 Y40 Z2 E6,5
G1 F240 X75,5 Y40 Z2 E6,4
G1 F240 X76,0 Y40 Z2 E6,6
G1 F240 X76,5 Y40 Z2 E6,5
G1 F240 X77,0 Y40 Z2 E6,7
G1 F240 X77,5 Y40 Z2 E6,6
G1 F240 X78,0 Y40 Z2 E6,8
G1 F240 X78,5 Y40 Z2 E6,7
G1 F240 X79,0 Y40 Z2 E6,9
G1 F240 X79,5 Y40 Z2 E6,8
G1 F240 X80,0 Y40 Z2 E7,0
G1 F240 X80,5 Y40 Z2 E6,9
G1 F240 X81,0 Y40 Z2 E7,1
G1 F240 X81,5 Y40 Z2 E7,0
G1 F240 X82,0 Y40 Z2 E7,2
G1 F240 X82,5 Y40 Z2 E7,1
G1 F240 X83,0 Y40 Z2 E7,3
G1 F240 X83,5 Y40 Z2 E7,2
G1 F240 X84,0 Y40 Z2 E7,4
G1 F240 X84,5 Y40 Z2 E7,3
G1 F240 X85,0 Y40 Z2 E7,5
G1 F240 X85,5 Y40 Z2 E7,4
G1 F240 X86,0 Y40 Z2 E7,6
G1 F240 X86,5 Y40 Z2 E7,5
G1 F240 X87,0 Y40 Z2 E7,7
G1 F240 X87,5 Y40 Z2 E7,6
G1 F240 X88,0 Y40 Z2 E7,8
G1 F240 X88,5 Y40 Z2 E7,7
G1 F240 X89,0 Y40 Z2 E7,9
G1 F240 X89,5 Y40 Z2 E7,8
G1 F240 X90,0 Y40 Z2 E8,0
G1 F240 X90,5 Y40 Z2 E7,9
G1 F240 X91,0 Y40 Z2 E8,1
G1 F240 X91,5 Y40 Z2 E8,0
G1 F240 X92,0 Y40 Z2 E8,2
G1 F240 X92,5 Y40 Z2 E8,1
G1 F240 X93,0 Y40 Z2 E8,3
G1 F240 X93,5 Y40 Z2 E8,2
G1 F240 X94,0 Y40 Z2 E8,4
G1 F240 X94,5 Y40 Z2 E8,3
G1 F240 X95,0 Y40 Z2 E8,5
G1 F240 X95,5 Y40 Z2 E8,4
G1 F240 X96,0 Y40 Z2 E8,6
G1 F240 X96,5 Y40 Z2 E8,5
G1 F240 X97,0 Y40 Z2 E8,7
G1 F240 X97,5 Y40 Z2 E8,6
G1 F240 X98,0 Y40 Z2 E8,8
G1 F240 X98,5 Y40 Z2 E8,7
G1 F240 X99,0 Y40 Z2 E8,9
G1 F240 X99,5 Y40 Z2 E8,8
G1 F240 X100 Y40 Z2 E9

;Stop Process
G1 F240 X100 Y45 Z2 E7

;Prep for string3
G0 F3000 X0 Y55 Z5

M82 ;absolute extrusion mode
;End of Gcode
```

D.3 G-Code for 3rd extruded String

```
;FLAVOR:Ma3rlin
M82 ;absolute extrusion mode

G21 ;mm format
G92 X0 Y55 Z5 E0 ;Prime the
extruder

;String3 Prep
G0 F3000 X0 Y60 Z5
G1 F300 X5 Y60 Z1 E1
G1 F300 X8 Y60 Z3 E2
G1 F300 X9 Y60 Z2,5 E2

;String3 extrusion (2,5mm)
G92 E0 ;Prime the extruder
G1 F240 X10,0 Y60 Z2,5 E0,0
G1 F240 X11,0 Y60 Z2,5 E0,1
G1 F240 X11,5 Y60 Z2,5 E0,0
G1 F240 X12,0 Y60 Z2,5 E0,2
G1 F240 X12,5 Y60 Z2,5 E0,1
G1 F240 X13,0 Y60 Z2,5 E0,3
G1 F240 X13,5 Y60 Z2,5 E0,2
G1 F240 X14,0 Y60 Z2,5 E0,4
G1 F240 X14,5 Y60 Z2,5 E0,3
G1 F240 X15,0 Y60 Z2,5 E0,5
G1 F240 X15,5 Y60 Z2,5 E0,4
G1 F240 X16,0 Y60 Z2,5 E0,6
G1 F240 X16,5 Y60 Z2,5 E0,5
G1 F240 X17,0 Y60 Z2,5 E0,7
G1 F240 X17,5 Y60 Z2,5 E0,6
G1 F240 X18,0 Y60 Z2,5 E0,8
G1 F240 X18,5 Y60 Z2,5 E0,7
G1 F240 X19,0 Y60 Z2,5 E0,9
G1 F240 X19,5 Y60 Z2,5 E0,8
G1 F240 X20,0 Y60 Z2,5 E1,0
G1 F240 X20,5 Y60 Z2,5 E0,9
G1 F240 X21,0 Y60 Z2,5 E1,1
G1 F240 X21,5 Y60 Z2,5 E1,0
G1 F240 X22,0 Y60 Z2,5 E1,2
G1 F240 X22,5 Y60 Z2,5 E1,1
G1 F240 X23,0 Y60 Z2,5 E1,3
G1 F240 X23,5 Y60 Z2,5 E1,2
G1 F240 X24,0 Y60 Z2,5 E1,4
G1 F240 X24,5 Y60 Z2,5 E1,3
G1 F240 X25,0 Y60 Z2,5 E1,5
G1 F240 X25,5 Y60 Z2,5 E1,4
G1 F240 X26,0 Y60 Z2,5 E1,6
G1 F240 X26,5 Y60 Z2,5 E1,5
G1 F240 X27,0 Y60 Z2,5 E1,7
G1 F240 X27,5 Y60 Z2,5 E1,6
G1 F240 X28,0 Y60 Z2,5 E1,8
G1 F240 X28,5 Y60 Z2,5 E1,7
G1 F240 X29,0 Y60 Z2,5 E1,9
G1 F240 X29,5 Y60 Z2,5 E1,8
G1 F240 X30,0 Y60 Z2,5 E2,0
G1 F240 X30,5 Y60 Z2,5 E1,9
G1 F240 X31,0 Y60 Z2,5 E2,1
G1 F240 X31,5 Y60 Z2,5 E2,0
G1 F240 X32,0 Y60 Z2,5 E2,2
G1 F240 X32,5 Y60 Z2,5 E2,1
G1 F240 X33,0 Y60 Z2,5 E2,3
G1 F240 X33,5 Y60 Z2,5 E2,2
G1 F240 X34,0 Y60 Z2,5 E2,4
G1 F240 X34,5 Y60 Z2,5 E2,3
G1 F240 X35,0 Y60 Z2,5 E2,5
G1 F240 X35,5 Y60 Z2,5 E2,4
G1 F240 X36,0 Y60 Z2,5 E2,6
G1 F240 X36,5 Y60 Z2,5 E2,5
G1 F240 X37,0 Y60 Z2,5 E2,7
G1 F240 X37,5 Y60 Z2,5 E2,6
G1 F240 X38,0 Y60 Z2,5 E2,8
G1 F240 X38,5 Y60 Z2,5 E2,7
G1 F240 X39,0 Y60 Z2,5 E2,9
G1 F240 X39,5 Y60 Z2,5 E2,8
G1 F240 X40,0 Y60 Z2,5 E3,0
G1 F240 X40,5 Y60 Z2,5 E2,9
G1 F240 X41,0 Y60 Z2,5 E3,1
G1 F240 X41,5 Y60 Z2,5 E3,0
G1 F240 X42,0 Y60 Z2,5 E3,2
G1 F240 X42,5 Y60 Z2,5 E3,1
G1 F240 X43,0 Y60 Z2,5 E3,3
G1 F240 X43,5 Y60 Z2,5 E3,2
G1 F240 X44,0 Y60 Z2,5 E3,4
G1 F240 X44,5 Y60 Z2,5 E3,3
G1 F240 X45,0 Y60 Z2,5 E3,5
G1 F240 X45,5 Y60 Z2,5 E3,4
G1 F240 X46,0 Y60 Z2,5 E3,6
G1 F240 X46,5 Y60 Z2,5 E3,5
G1 F240 X47,0 Y60 Z2,5 E3,7
G1 F240 X47,5 Y60 Z2,5 E3,6
G1 F240 X48,0 Y60 Z2,5 E3,8
G1 F240 X48,5 Y60 Z2,5 E3,7
G1 F240 X49,0 Y60 Z2,5 E3,9
G1 F240 X49,5 Y60 Z2,5 E3,8
G1 F240 X50,0 Y60 Z2,5 E4,0
G1 F240 X50,5 Y60 Z2,5 E3,9
G1 F240 X51,0 Y60 Z2,5 E4,1
G1 F240 X51,5 Y60 Z2,5 E4,0
G1 F240 X52,0 Y60 Z2,5 E4,2
G1 F240 X52,5 Y60 Z2,5 E4,1
G1 F240 X53,0 Y60 Z2,5 E4,3
G1 F240 X53,5 Y60 Z2,5 E4,2
G1 F240 X54,0 Y60 Z2,5 E4,4
G1 F240 X54,5 Y60 Z2,5 E4,3
G1 F240 X55,0 Y60 Z2,5 E4,5
G1 F240 X55,5 Y60 Z2,5 E4,4
G1 F240 X56,0 Y60 Z2,5 E4,6
G1 F240 X56,5 Y60 Z2,5 E4,5
G1 F240 X57,0 Y60 Z2,5 E4,7
G1 F240 X57,5 Y60 Z2,5 E4,6
G1 F240 X58,0 Y60 Z2,5 E4,8
G1 F240 X58,5 Y60 Z2,5 E4,7
G1 F240 X59,0 Y60 Z2,5 E4,9
G1 F240 X59,5 Y60 Z2,5 E4,8
G1 F240 X60,0 Y60 Z2,5 E5,0
G1 F240 X60,5 Y60 Z2,5 E4,9
G1 F240 X61,0 Y60 Z2,5 E5,1
G1 F240 X61,5 Y60 Z2,5 E5,0
G1 F240 X62,0 Y60 Z2,5 E5,2
G1 F240 X62,5 Y60 Z2,5 E5,1
G1 F240 X63,0 Y60 Z2,5 E5,3
G1 F240 X63,5 Y60 Z2,5 E5,2
G1 F240 X64,0 Y60 Z2,5 E5,4
G1 F240 X64,5 Y60 Z2,5 E5,3
G1 F240 X65,0 Y60 Z2,5 E5,5
G1 F240 X65,5 Y60 Z2,5 E5,4
G1 F240 X66,0 Y60 Z2,5 E5,6
G1 F240 X66,5 Y60 Z2,5 E5,5
G1 F240 X67,0 Y60 Z2,5 E5,7
G1 F240 X67,5 Y60 Z2,5 E5,6
G1 F240 X68,0 Y60 Z2,5 E5,8
G1 F240 X68,5 Y60 Z2,5 E5,7
G1 F240 X69,0 Y60 Z2,5 E5,9
G1 F240 X69,5 Y60 Z2,5 E5,8
G1 F240 X70,0 Y60 Z2,5 E6,0
G1 F240 X70,5 Y60 Z2,5 E5,9
G1 F240 X71,0 Y60 Z2,5 E6,1
G1 F240 X71,5 Y60 Z2,5 E6,0
G1 F240 X72,0 Y60 Z2,5 E6,2
G1 F240 X72,5 Y60 Z2,5 E6,1
G1 F240 X73,0 Y60 Z2,5 E6,3
G1 F240 X73,5 Y60 Z2,5 E6,2
G1 F240 X74,0 Y60 Z2,5 E6,4
G1 F240 X74,5 Y60 Z2,5 E6,3
G1 F240 X75,0 Y60 Z2,5 E6,5
G1 F240 X75,5 Y60 Z2,5 E6,4
G1 F240 X76,0 Y60 Z2,5 E6,6
G1 F240 X76,5 Y60 Z2,5 E6,5
G1 F240 X77,0 Y60 Z2,5 E6,7
G1 F240 X77,5 Y60 Z2,5 E6,6
G1 F240 X78,0 Y60 Z2,5 E6,8
G1 F240 X78,5 Y60 Z2,5 E6,7
G1 F240 X79,0 Y60 Z2,5 E6,9
G1 F240 X79,5 Y60 Z2,5 E6,8
G1 F240 X80,0 Y60 Z2,5 E7,0
G1 F240 X80,5 Y60 Z2,5 E6,9
G1 F240 X81,0 Y60 Z2,5 E7,1
G1 F240 X81,5 Y60 Z2,5 E7,0
G1 F240 X82,0 Y60 Z2,5 E7,2
G1 F240 X82,5 Y60 Z2,5 E7,1
G1 F240 X83,0 Y60 Z2,5 E7,3
G1 F240 X83,5 Y60 Z2,5 E7,2
G1 F240 X84,0 Y60 Z2,5 E7,4
G1 F240 X84,5 Y60 Z2,5 E7,3
G1 F240 X85,0 Y60 Z2,5 E7,5
G1 F240 X85,5 Y60 Z2,5 E7,4
G1 F240 X86,0 Y60 Z2,5 E7,6
G1 F240 X86,5 Y60 Z2,5 E7,5
G1 F240 X87,0 Y60 Z2,5 E7,7
G1 F240 X87,5 Y60 Z2,5 E7,6
G1 F240 X88,0 Y60 Z2,5 E7,8
G1 F240 X88,5 Y60 Z2,5 E7,7
G1 F240 X89,0 Y60 Z2,5 E7,9
G1 F240 X89,5 Y60 Z2,5 E7,8
G1 F240 X90,0 Y60 Z2,5 E8,0
G1 F240 X90,5 Y60 Z2,5 E7,9
G1 F240 X91,0 Y60 Z2,5 E8,1
G1 F240 X91,5 Y60 Z2,5 E8,0
G1 F240 X92,0 Y60 Z2,5 E8,2
G1 F240 X92,5 Y60 Z2,5 E8,1
G1 F240 X93,0 Y60 Z2,5 E8,3
G1 F240 X93,5 Y60 Z2,5 E8,2
G1 F240 X94,0 Y60 Z2,5 E8,4
G1 F240 X94,5 Y60 Z2,5 E8,3
G1 F240 X95,0 Y60 Z2,5 E8,5
G1 F240 X95,5 Y60 Z2,5 E8,4
G1 F240 X96,0 Y60 Z2,5 E8,6
G1 F240 X96,5 Y60 Z2,5 E8,5
G1 F240 X97,0 Y60 Z2,5 E8,7
G1 F240 X97,5 Y60 Z2,5 E8,6
G1 F240 X98,0 Y60 Z2,5 E8,8
G1 F240 X98,5 Y60 Z2,5 E8,7
G1 F240 X99,0 Y60 Z2,5 E8,9
G1 F240 X99,5 Y60 Z2,5 E8,8
G1 F240 X100 Y60 Z2,5 E9

;Stop Process
G1 F240 X100 Y65 Z2,5 E7

;Prep for string4
G0 F3000 X0 Y75 Z5

M82 ;absolute extrusion mode
;End of Gcode
```

D.4 G-Code for 4^{th} extruded String

```
;FLAVOR:Ma3rlin
M82 ;absolute extrusion mode

G21 ;mm format
G92 X0 Y75 Z5 E0 ;Prime the
extruder

;String4 Prep
G0 F3000 X0 Y80 Z5
G1 F300 X5 Y80 Z2 E1
G1 F300 X8 Y80 Z3,5 E2
G1 F300 X9 Y80 Z3 E2

;String4 extrusion (3mm)
G92 E0 ;Prime the extruder
G1 F240 X10,0 Y80 Z3 E0,0
G1 F240 X11,0 Y80 Z3 E0,1
G1 F240 X11,5 Y80 Z3 E0,0
G1 F240 X12,0 Y80 Z3 E0,2
G1 F240 X12,5 Y80 Z3 E0,1
G1 F240 X13,0 Y80 Z3 E0,3
G1 F240 X13,5 Y80 Z3 E0,2
G1 F240 X14,0 Y80 Z3 E0,4
G1 F240 X14,5 Y80 Z3 E0,3
G1 F240 X15,0 Y80 Z3 E0,5
G1 F240 X15,5 Y80 Z3 E0,4
G1 F240 X16,0 Y80 Z3 E0,6
G1 F240 X16,5 Y80 Z3 E0,5
G1 F240 X17,0 Y80 Z3 E0,7
G1 F240 X17,5 Y80 Z3 E0,6
G1 F240 X18,0 Y80 Z3 E0,8
G1 F240 X18,5 Y80 Z3 E0,7
G1 F240 X19,0 Y80 Z3 E0,9
G1 F240 X19,5 Y80 Z3 E0,8
G1 F240 X20,0 Y80 Z3 E1,0
G1 F240 X20,5 Y80 Z3 E0,9
G1 F240 X21,0 Y80 Z3 E1,1
G1 F240 X21,5 Y80 Z3 E1,0
G1 F240 X22,0 Y80 Z3 E1,2
G1 F240 X22,5 Y80 Z3 E1,1
G1 F240 X23,0 Y80 Z3 E1,3
G1 F240 X23,5 Y80 Z3 E1,2
G1 F240 X24,0 Y80 Z3 E1,4
G1 F240 X24,5 Y80 Z3 E1,3
G1 F240 X25,0 Y80 Z3 E1,5
G1 F240 X25,5 Y80 Z3 E1,4
G1 F240 X26,0 Y80 Z3 E1,6
G1 F240 X26,5 Y80 Z3 E1,5
G1 F240 X27,0 Y80 Z3 E1,7
G1 F240 X27,5 Y80 Z3 E1,6
G1 F240 X28,0 Y80 Z3 E1,8
G1 F240 X28,5 Y80 Z3 E1,7
G1 F240 X29,0 Y80 Z3 E1,9
G1 F240 X29,5 Y80 Z3 E1,8
G1 F240 X30,0 Y80 Z3 E2,0
G1 F240 X30,5 Y80 Z3 E1,9
G1 F240 X31,0 Y80 Z3 E2,1
G1 F240 X31,5 Y80 Z3 E2,0
G1 F240 X32,0 Y80 Z3 E2,2
G1 F240 X32,5 Y80 Z3 E2,1
G1 F240 X33,0 Y80 Z3 E2,3
G1 F240 X33,5 Y80 Z3 E2,2
G1 F240 X34,0 Y80 Z3 E2,4
G1 F240 X34,5 Y80 Z3 E2,3
G1 F240 X35,0 Y80 Z3 E2,5
G1 F240 X35,5 Y80 Z3 E2,4
G1 F240 X36,0 Y80 Z3 E2,6
G1 F240 X36,5 Y80 Z3 E2,5
G1 F240 X37,0 Y80 Z3 E2,7
G1 F240 X37,5 Y80 Z3 E2,6
G1 F240 X38,0 Y80 Z3 E2,8
G1 F240 X38,5 Y80 Z3 E2,7
G1 F240 X39,0 Y80 Z3 E2,9
G1 F240 X39,5 Y80 Z3 E2,8
G1 F240 X40,0 Y80 Z3 E3,0
G1 F240 X40,5 Y80 Z3 E2,9
G1 F240 X41,0 Y80 Z3 E3,1
G1 F240 X41,5 Y80 Z3 E3,0
G1 F240 X42,0 Y80 Z3 E3,2
G1 F240 X42,5 Y80 Z3 E3,1
G1 F240 X43,0 Y80 Z3 E3,3
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G1 F240 X47,0 Y80 Z3 E3,7
G1 F240 X47,5 Y80 Z3 E3,6
G1 F240 X48,0 Y80 Z3 E3,8
G1 F240 X48,5 Y80 Z3 E3,7
G1 F240 X49,0 Y80 Z3 E3,9
G1 F240 X49,5 Y80 Z3 E3,8
G1 F240 X50,0 Y80 Z3 E4,0
G1 F240 X50,5 Y80 Z3 E3,9
G1 F240 X51,0 Y80 Z3 E4,1
G1 F240 X51,5 Y80 Z3 E4,0
G1 F240 X52,0 Y80 Z3 E4,2
G1 F240 X52,5 Y80 Z3 E4,1
G1 F240 X53,0 Y80 Z3 E4,3
G1 F240 X53,5 Y80 Z3 E4,2
G1 F240 X54,0 Y80 Z3 E4,4
G1 F240 X54,5 Y80 Z3 E4,3
G1 F240 X55,0 Y80 Z3 E4,5
G1 F240 X55,5 Y80 Z3 E4,4
G1 F240 X56,0 Y80 Z3 E4,6
G1 F240 X56,5 Y80 Z3 E4,5
G1 F240 X57,0 Y80 Z3 E4,7
G1 F240 X57,5 Y80 Z3 E4,6
G1 F240 X58,0 Y80 Z3 E4,8
G1 F240 X58,5 Y80 Z3 E4,7
G1 F240 X59,0 Y80 Z3 E4,9
G1 F240 X59,5 Y80 Z3 E4,8
G1 F240 X60,0 Y80 Z3 E5,0
G1 F240 X60,5 Y80 Z3 E4,9
G1 F240 X61,0 Y80 Z3 E5,1
G1 F240 X61,5 Y80 Z3 E5,0
G1 F240 X62,0 Y80 Z3 E5,2
G1 F240 X62,5 Y80 Z3 E5,1
G1 F240 X63,0 Y80 Z3 E5,3
G1 F240 X63,5 Y80 Z3 E5,2
G1 F240 X64,0 Y80 Z3 E5,4
G1 F240 X64,5 Y80 Z3 E5,3
G1 F240 X65,0 Y80 Z3 E5,5
G1 F240 X65,5 Y80 Z3 E5,4
G1 F240 X66,0 Y80 Z3 E5,6
G1 F240 X66,5 Y80 Z3 E5,5
G1 F240 X67,0 Y80 Z3 E5,7
G1 F240 X67,5 Y80 Z3 E5,6
G1 F240 X68,0 Y80 Z3 E5,8
G1 F240 X68,5 Y80 Z3 E5,7
G1 F240 X69,0 Y80 Z3 E5,9
G1 F240 X69,5 Y80 Z3 E5,8
G1 F240 X70,0 Y80 Z3 E6,0
G1 F240 X70,5 Y80 Z3 E5,9
G1 F240 X71,0 Y80 Z3 E6,1
G1 F240 X71,5 Y80 Z3 E6,0
G1 F240 X72,0 Y80 Z3 E6,2
G1 F240 X72,5 Y80 Z3 E6,1
G1 F240 X73,0 Y80 Z3 E6,3
G1 F240 X73,5 Y80 Z3 E6,2
G1 F240 X74,0 Y80 Z3 E6,4
G1 F240 X74,5 Y80 Z3 E6,3
G1 F240 X75,0 Y80 Z3 E6,5
G1 F240 X75,5 Y80 Z3 E6,4
G1 F240 X76,0 Y80 Z3 E6,6
G1 F240 X76,5 Y80 Z3 E6,5
G1 F240 X77,0 Y80 Z3 E6,7
G1 F240 X77,5 Y80 Z3 E6,6
G1 F240 X78,0 Y80 Z3 E6,8
G1 F240 X78,5 Y80 Z3 E6,7
G1 F240 X79,0 Y80 Z3 E6,9
G1 F240 X79,5 Y80 Z3 E6,8
G1 F240 X80,0 Y80 Z3 E7,0
G1 F240 X80,5 Y80 Z3 E6,9
G1 F240 X81,0 Y80 Z3 E7,1
G1 F240 X81,5 Y80 Z3 E7,0
G1 F240 X82,0 Y80 Z3 E7,2
G1 F240 X82,5 Y80 Z3 E7,1
G1 F240 X83,0 Y80 Z3 E7,3
G1 F240 X83,5 Y80 Z3 E7,2
G1 F240 X84,0 Y80 Z3 E7,4
G1 F240 X84,5 Y80 Z3 E7,3
G1 F240 X85,0 Y80 Z3 E7,5
G1 F240 X85,5 Y80 Z3 E7,4
G1 F240 X86,0 Y80 Z3 E7,6
G1 F240 X86,5 Y80 Z3 E7,5
G1 F240 X87,0 Y80 Z3 E7,7
G1 F240 X87,5 Y80 Z3 E7,6
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G1 F240 X89,5 Y80 Z3 E7,8
G1 F240 X90,0 Y80 Z3 E8,0
G1 F240 X90,5 Y80 Z3 E7,9
G1 F240 X91,0 Y80 Z3 E8,1
G1 F240 X91,5 Y80 Z3 E8,0
G1 F240 X92,0 Y80 Z3 E8,2
G1 F240 X92,5 Y80 Z3 E8,1
G1 F240 X93,0 Y80 Z3 E8,3
G1 F240 X93,5 Y80 Z3 E8,2
G1 F240 X94,0 Y80 Z3 E8,4
G1 F240 X94,5 Y80 Z3 E8,3
G1 F240 X95,0 Y80 Z3 E8,5
G1 F240 X95,5 Y80 Z3 E8,4
G1 F240 X96,0 Y80 Z3 E8,6
G1 F240 X96,5 Y80 Z3 E8,5
G1 F240 X97,0 Y80 Z3 E8,7
G1 F240 X97,5 Y80 Z3 E8,6
G1 F240 X98,0 Y80 Z3 E8,8
G1 F240 X98,5 Y80 Z3 E8,7
G1 F240 X99,0 Y80 Z3 E8,9
G1 F240 X99,5 Y80 Z3 E8,8
G1 F240 X100 Y80 Z3 E9

;Stop Process
G1 F240 X100 Y85 Z3 E7

;Finish
G0 F3000 X100 Y110 Z90 E4

M82 ;absolute extrusion mode
;End of Gcode
```

Appendix E: Biresin G26 Cup and Syringe samples

Figure E.1: Cup and Syringe samples for Biresin G26

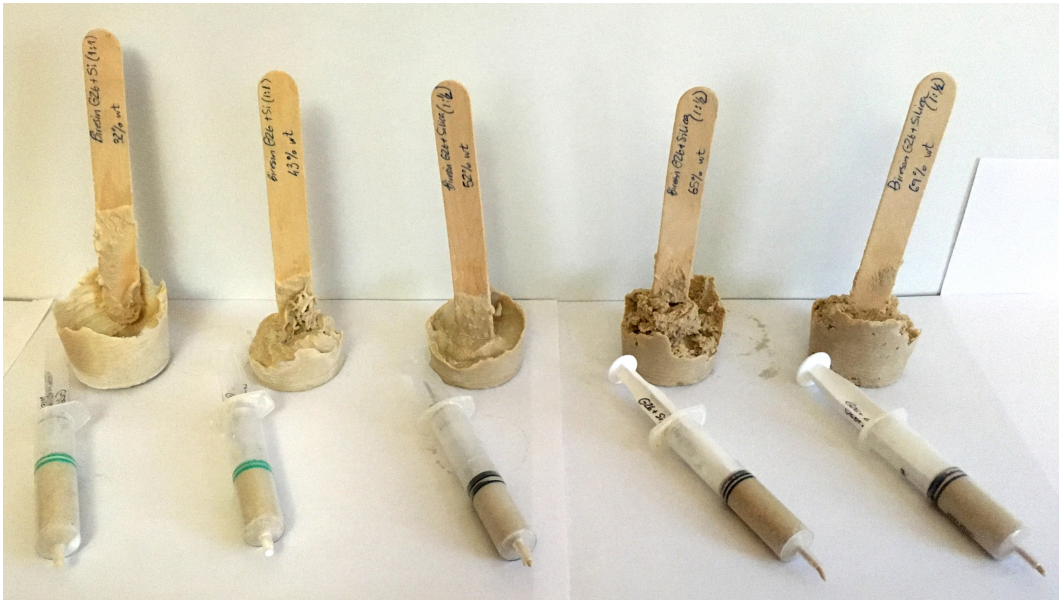

Figure E.2: Cup and Syringe samples for Biresin G26 + Fumed Silica

Appendix F: Pavistone 1K Cup and Syringe samples

Figure F.1: Cup and Syringe samples for Pavistone 1K

Figure F.2: Cup and Syringe samples for Pavistone 1K + Fumed Silica

Appendix G: Pavistone 2K Cup and Syringe samples

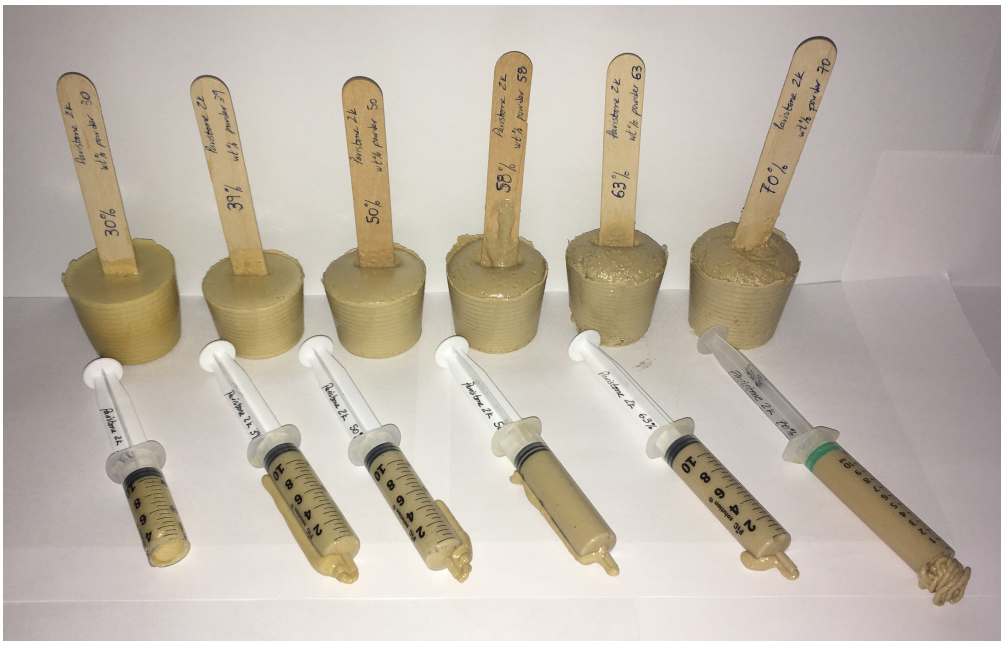

Figure G.1: Cup and Syringe samples for Pavistone 2K

Figure G.2: Cup and Syringe samples for Pavistone 2K + Fumed Silica

Appendix H: Experiment data files

H.1 Biresin G26 Data file

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Mixing Experiments - Direct Digital Manufacturing

Materials & Test Conditions

Test date: 19-02-2020

Binder Material: Biresin 26 (A+B) A - Resin 100% B - Hardener 100%

$CaCO_2$ Residue Material: Powder ~~Sludge~~

Pot Life: 3-4 min Cure Time: 3 days

Ambiente temperature (ºC): 20

Notes: For Items 1 to 3 the Residue is mixed into component B for 1 minute, then added to component A and mixed for 1.5 minutes until homogeneous. For items 4 to 6 the residue is split between Binder A & B, then mixed together for 1.5 minutes until homogeneous. The mixtures are then placed inside a syringe and extruded along 10cms for approximately 10s. The aim was to extrude the bead within 1.5 minutes from mixing.

Item	Iteration mix of Residue content (wt%)	≈ Mass measurements (grs)						Notes
		Binder A	Binder B	Binder Total	Residue	Theoretical Total weight	Measured Total weight after mixing	
1	29	15	15	30	12	42	41	Low viscosity, doesn't hold form.
2	41	15	15	30	21	51	51	Low viscosity, doesn't hold form.
3	54	15	15	30	30	60	56	Low viscosity, doesn't hold form. Hardens and losses fluid state quicker than above.
4	67	15	15	30	39	69	58	Low viscosity, doesn't hold form. Hardens and losses fluid state quicker than above.
5	77	15	15	30	48	78	62	Very pasty. Hard to place in syringe.
6	86	15	15	30	57	87	66	Very pasty. Could not add all of the residue, 10grs couldn't be added. Hard to place in syringe.

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Mixing Experiments - Direct Digital Manufacturing

Materials & Test Conditions

Test date: 15-05-2020

Binder Material: Pavistone 1K (A) A - Polyisocyanate 100%

$CaCO_2$ Residue Material: Powder ~~Sludge~~

Pot Life: 90-120 min Cure Time: 5-24 hours

Ambiente temperature (ºC): 20

Notes: Residue and binder mixed for 1.5 minutes until the mixture is homogeneous. This is then placed inside a syringe then extruded along 10cms for approximately 10s. The aim was to extrude the bead within 1.5 minutes from mixing but for item 6 in the list that was not possible.

Item	Iteration mix of Residue content (wt%)	≈ Mass measurements (grs)					Results
		Binder A	Binder Total	Residue	Theoretical Total weight	Measured Total weight after mixing	
1	39	30	30	20	50	51	Low viscosity, doesn't hold form.
2	49	30	30	30	60	61	Low viscosity, doesn't hold form.
3	58	30	30	40	70	69	Low viscosity, doesn't hold form.
4	60	30	30	50	80	83	Low viscosity, doesn't hold form.
5	66	30	30	60	90	91	Viscosity improves but still has issues holding form. Harder recipient needed to perform mixtures of this content or higher.
6	71	30	30	74	104	104	Very pasty. Hard to place in syringe. Not good for small diameter nozzles (syringe has 2mm nozzle).

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Mixing Experiments - Direct Digital Manufacturing

Materials & Test Conditions

Test date: 16-05-2020

Binder Material: Pavistone 2K

(A+B) A - Polyol 100%

B - Polyisocyanate 43.5%

$CaCO_2$ Residue Material: Powder ~~Sludge~~

Pot Life: 35 min Cure Time: --

Ambiente temperature (ºC): 20

Notes: For Items 1 to 3 the Residue is mixed into component B for 1 minute, then added to component A and mixed for 1.5 minutes until homogeneous. For Items 4 to 6The mixtures are then placed inside a syringe then extruded along 10cms for approximately 10s. The aim was to extrude the bead within 1.5mintes from finishing the mixing.

Item	Iteration mix of Residue content (wt%)	≈ Mass measurements (grs)						Results
		Binder A	Binder B	Binder Total	Residue	Theoretical Total weight	Measured Total weight after mixing	
1	30	23	10	33	14	47	47	Low viscosity, doesn't hold form.
2	39	23	11	34	22	56	57	Low viscosity, doesn't hold form.
3	50	23	10	33	33	66	66	Low viscosity, doesn't hold form.
4	58	23	10	33	49	82	84	Loss of liquid state. Material was hard to extrude which doesn't make sense compared to the next test.
5	63	23	10	33	60	93	95	Good extrusion; Bead held its form after 1min from extrusion. From here a hard recipient is necessary for mixing.
6	70	23	10	33	76	109	109	Hard to mix; Good extrusion but requires considerable pressure; Bead held its form after 4min from extrusion.

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Mixing Experiments - Direct Digital Manufacturing

Materials & Test Conditions

Test date: 08-06-2020

Binder Material: Biresin G26 + Si

A - Resin 100%

B - Hardener 100%

$CaCO_2$ Residue Material: Powder ~~Sludge~~

Pot Life: 35 min Cure Time: --

Ambiente temperature (ºC): 24

Notes: Fumed Silica added to the mix in approximaterly the volume content values shown in table, as of the Binder being used. Mixing times are no longer relevant. Silica is first incorporated into the binder and only then the residue. This is then placed inside a syringe and extruded along 10cms for approximately 10s.

Item	Iteration mix of Residue content (wt%)	≈ Mass measurements (grs)							Results
		Binder A + B		Ratio Binder : Silica	Binder Total	Residue	Theoretical Total weight	Measured Total weight after mixing	
1	32	15	15	(1:1)	30	14	44	44	Very low viscosity, locked up before extrusion could be obtained. Si + residue mixed into both components.
2	43	15	15	(1:1)	30	23	53	53	Medium viscosity, holds form but expands. Si + residue mixed into comp. A and B.
3	52	15	15	(1: ½)	30	33	63	63	Low viscosity, temporarily holds form but expands. Si + residue mixed into both components.
4	65	15	15	(1: ½)	30	55	85	85	Very pasty peanut butter consistency, holds form. Si + residue mixed into both components. Hard recipient neded.
5	69	15	15	(1: ½)	30	68	98	98	Very Pasty, extruds and holds its form. Si + residue mixed into both components. Hard recipient neded.

POLITÉCNICO DE LEIRIA | ESCOLA SUPERIOR DE TECNOLOGIA E GESTÃO 30

Mixing Experiments - Direct Digital Manufacturing

Materials & Test Conditions

Test date: 01-06-2020

Binder Material: Pavistone 1K + Si (A - Polyisocyanate 100%

$CaCO_2$ Residue Material: Powder ~~Sludge~~

Pot Life: 90-120 min Cure Time: 5-24 hours

Ambiente temperature (ºC): 24

Notes: Fumed Silica added to the mix in approximaterly the content values, shown in table, as of the Binder being used. Mixing times are no longer relevant. Silica is first incorporated into the binder and only then the residue. This is then placed inside a syringe and extruded along 10cms for approximately 10s.

Item	Iteration mix of Residue content (wt%)	≈ Mass measurements (grs)						Results
		Binder A	Ratio Binder : Silica	Binder Total	Residue	Theoretical Total weight	Measured Total weight after mixing	
1	37	32	(1:1)	32	20	52	54	Very pasty. Hard to place in syringe but extrudes holding form.
2	49	30	(1:1)	30	30	60	61	Very pasty. Hard to place in syringe but extrudes holding form. Harder recipient recommended for mixture.
3	56	30	(1: ½)	30	40	70	72	Pasty. Hard to place in syringe but extrudes with ease and holds form. Harder recipient recommended for mixture.
4	63	30	(1: ½)	30	50	80	80	Very Pasty. Limit of residue incorporation via hand mixing methods. Need hard recipient for mixing. From here heavy duty mechanical mixing methods are required.

POLITÉCNICO DE LEIRIA | ESCOLA SUPERIOR DE TECNOLOGIA E GESTÃO

Mixing Experiments - Direct Digital Manufacturing

Materials & Test Conditions

Test date: 28-05-2020

Binder Material: Pavistone 2K + Si (A+B)

A - Polyol 100%

B - Polyisocyanate 43.5%

$CaCO_2$ Residue Material: Powder ~~Sludge~~

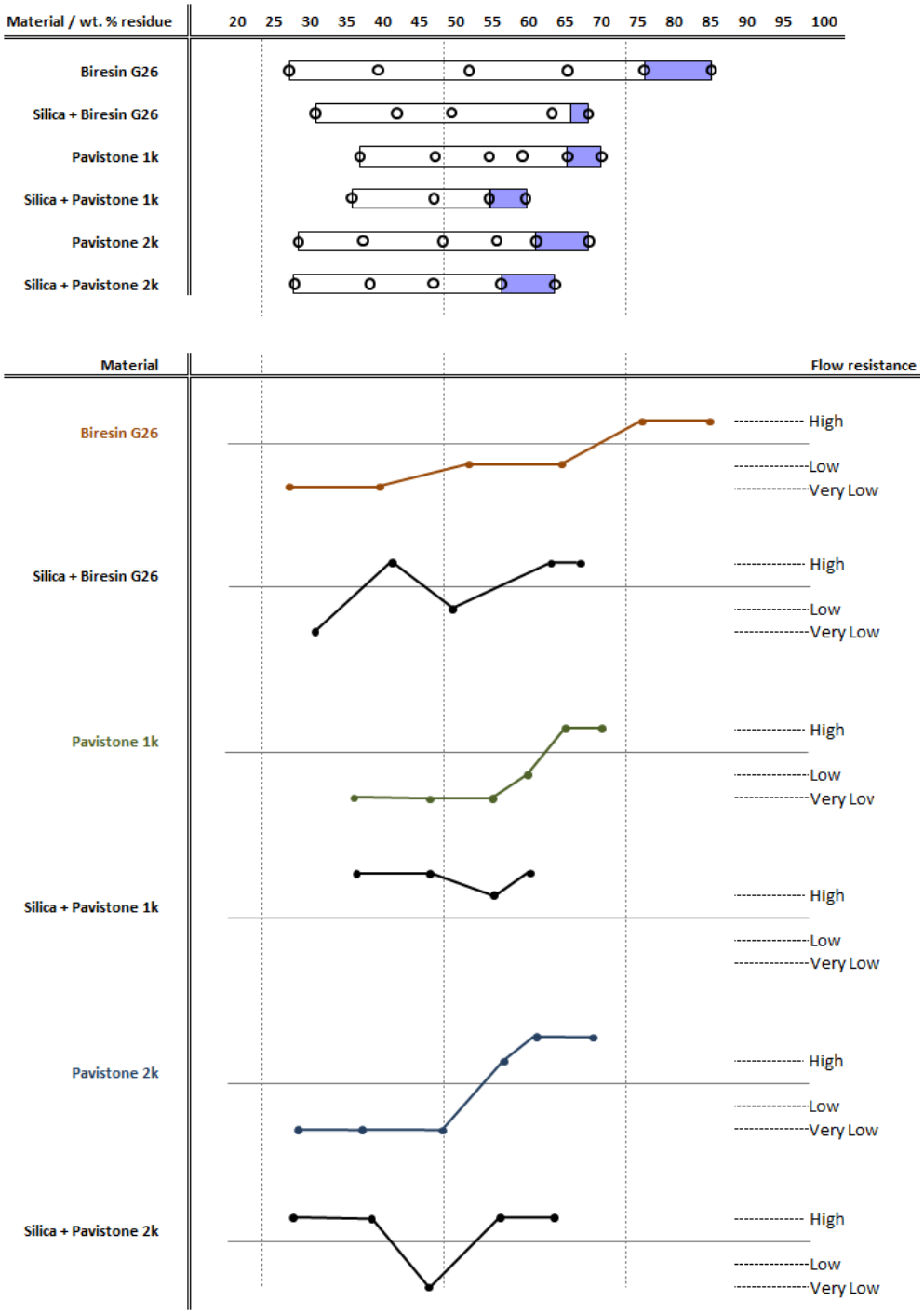
Pot Life: 35 min Cure Time: --

Ambiente temperature (ºC): 24

Notes: Fumed Silica added to the mix in approximaterly the volume content values shown in table, as of the Binder being used. Mixing times are no longer relevant. Silica is first incorporated into the binder and only then the residue. This is then placed inside a syringe and extruded along 10cms for approximately 10s.

Item	Iteration mix of Residue content (wt%)	≈ Mass measurements (grs)							Results
		Binder A + B		Ratio Binder : Silica	Binder Total	Residue	Theoretical Total weight	Measured Total weight after mixing	
1	29	23	12	(1:1)	35	14	49	49	Medium viscosity, holds form but expands. Si + residue mixed into comp. A.
2	40	24	11	(1:1)	35	23	58	58	Medium viscosity, holds form but expands. Si + residue mixed into comp. A and B.
3	48	25	12	(1: ½)	37	33	70	69	Low viscosity, temporarily holds form but expands. Si + residue mixed into comp. A.
4	58	26	14	(1: ½)	40	55	95	95	Medium viscosity, holds form but expands. Si + residue mixed into comp. A and B. Needs hard recipient for mixing.
5	65	24	13	(1: ½)	37	68	105	105	Very Pasty, extruds and holds its form. Si + residue mixed into comp. A and B. Needs hard recipient for mixing.

Appendix I: Summary of relations between fillers & flow resistance



Appendix J: Micro-CT images of composite samples

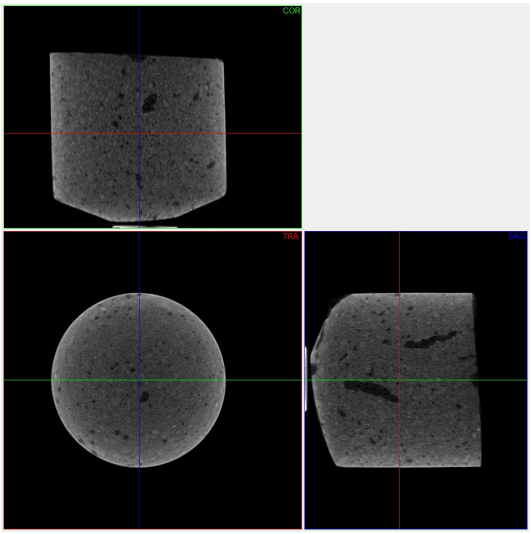


Figure J.1: Micro-CT of Biresin G26 + 77%wt Residue

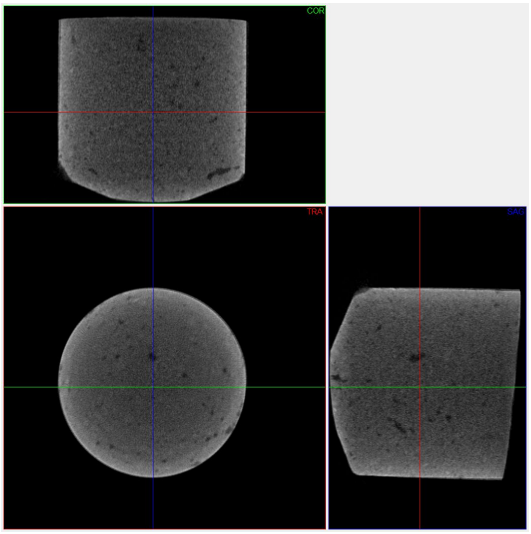


Figure J.2: Micro-CT of Biresin G26 + 86%wt Residue

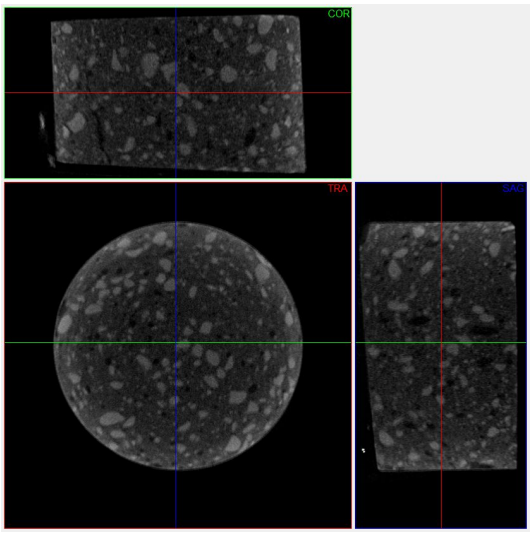


Figure J.3: Micro-CT of Biresin G26 + 43%wt Residue + Silica

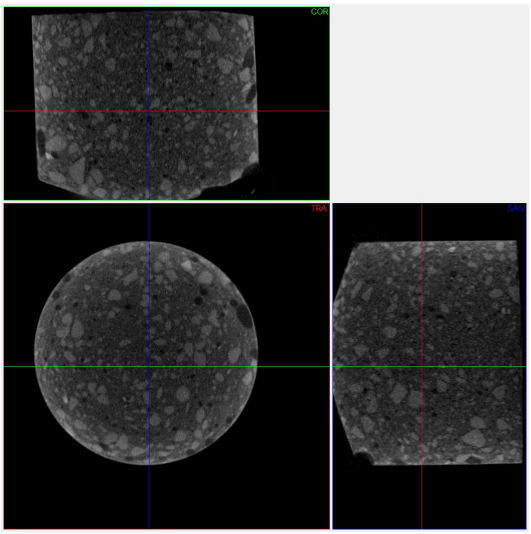


Figure J.4: Micro-CT of Biresin G26 + 52%wt Residue + Silica

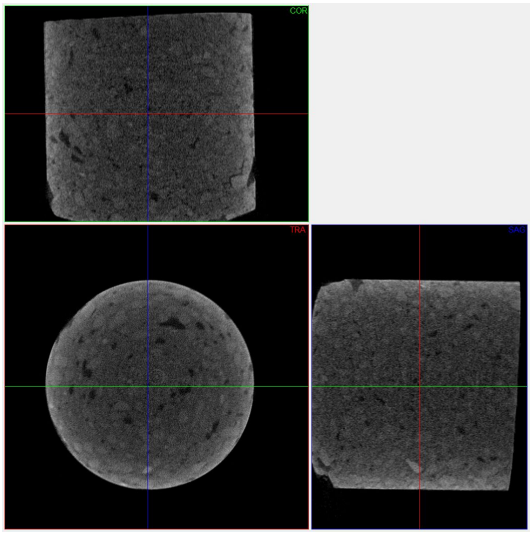


Figure J.5: Micro-CT of Biresin G26 + 65%wt Residue + Silica

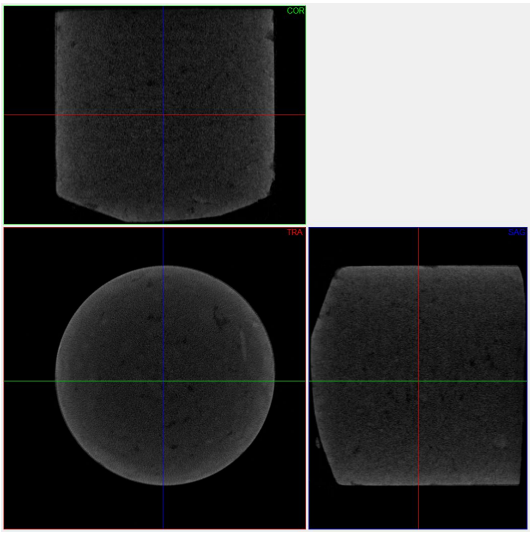


Figure J.6: Micro-CT of Biresin G26 + 69%wt Residue + Silica

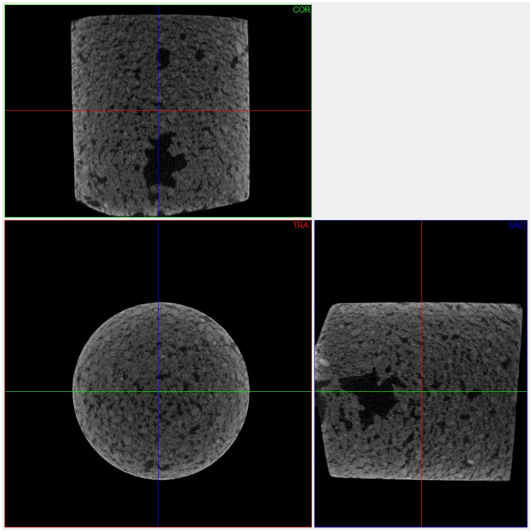


Figure J.7: Micro-CT of Pavistone 1K + 66%wt Residue

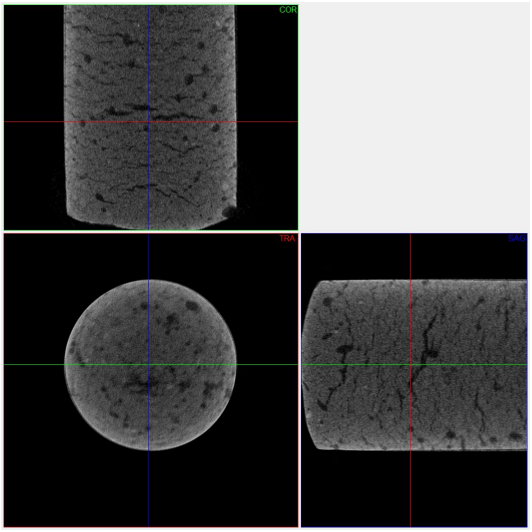


Figure J.8: Micro-CT of Pavistone 1K + 71%wt Residue

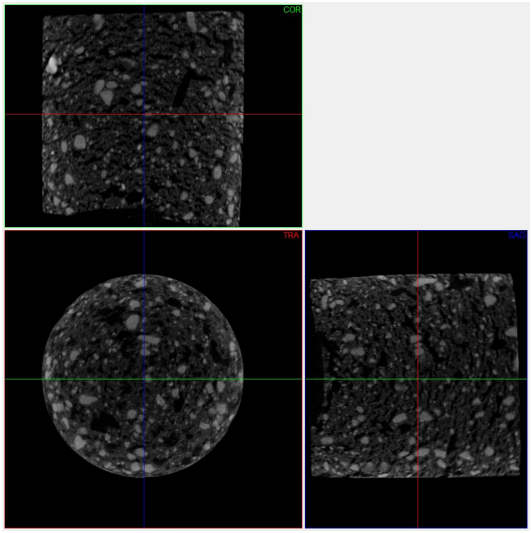


Figure J.9: Micro-CT of Pavistone 1K + 37%wt Residue + Silica

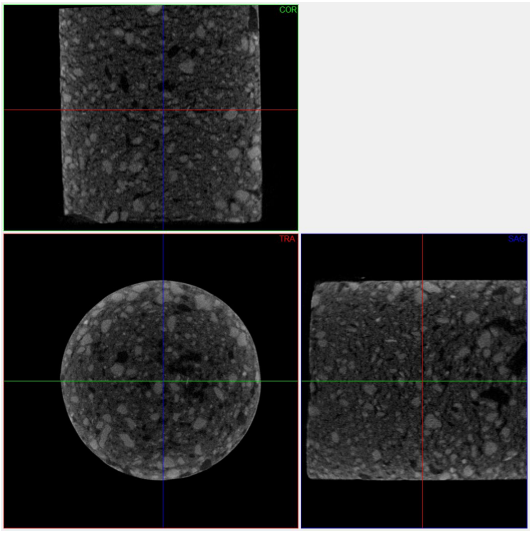


Figure J.10: Micro-CT of Pavistone 1K + 49%wt Residue + Silica

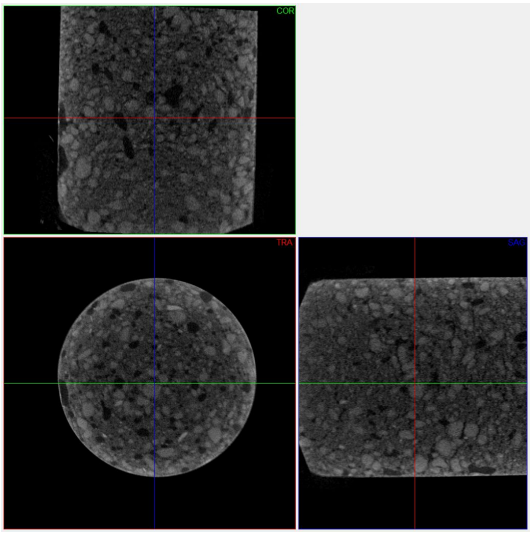


Figure J.11: Micro-CT of Pavistone 1K + 56%wt Residue + Silica

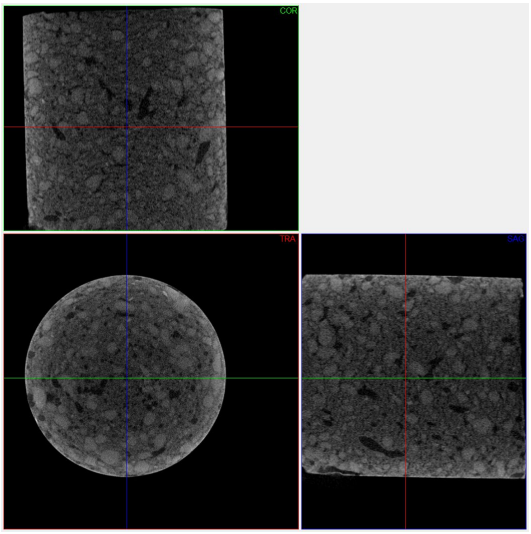


Figure J.12: Micro-CT of Pavistone 1K + 63%wt Residue + Silica

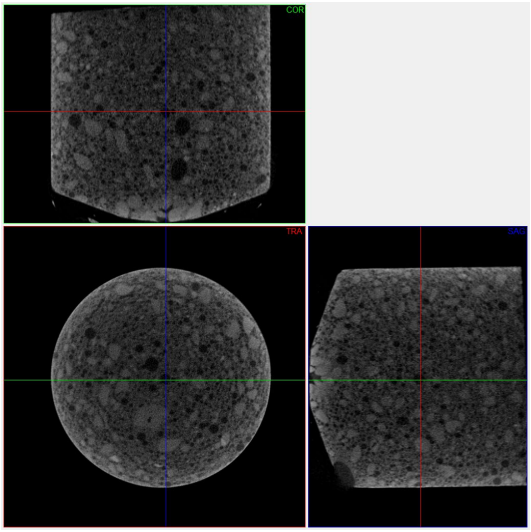


Figure J.13: Micro-CT of Pavistone 2K + 63%wt Residue

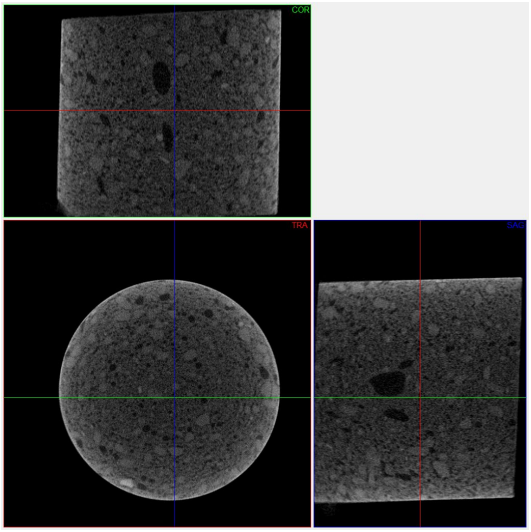


Figure J.14: Micro-CT of Pavistone 2K + 70%wt Residue

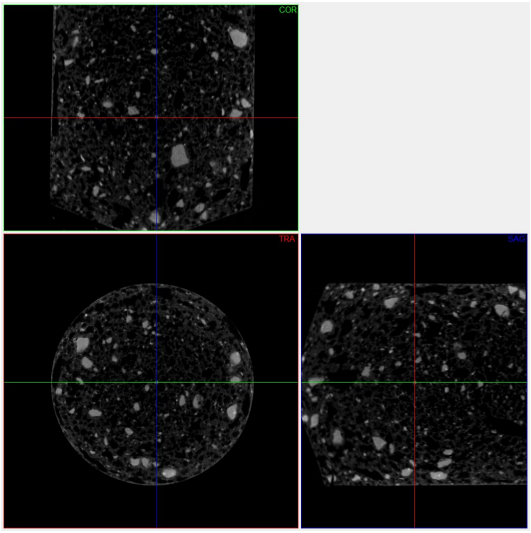


Figure J.15: Micro-CT of Pavistone 2K + 29%wt Residue + Silica

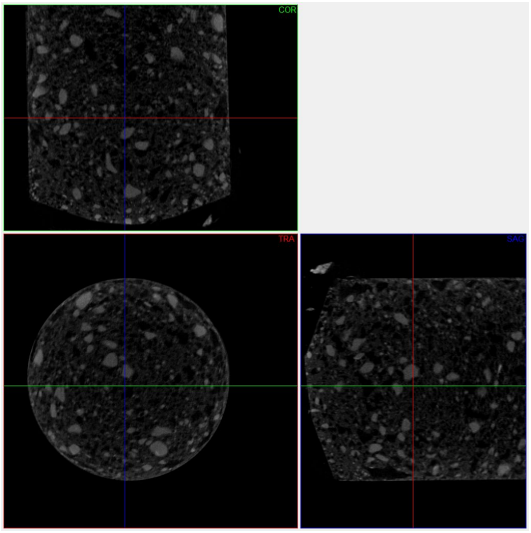


Figure J.16: Micro-CT of Pavistone 2K + 40%wt Residue + Silica

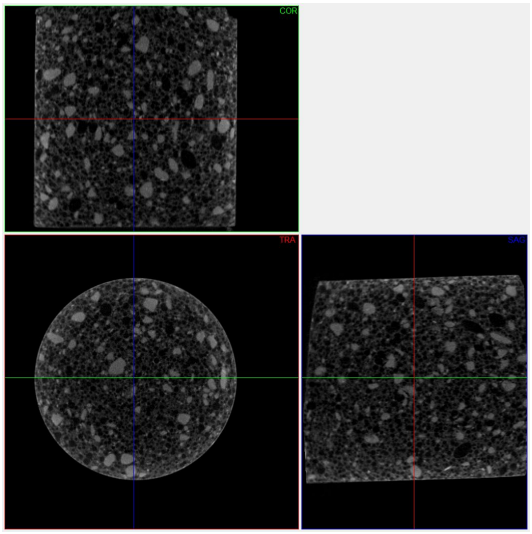


Figure J.17: Micro-CT of Pavistone 2K + 48%wt Residue + Silica

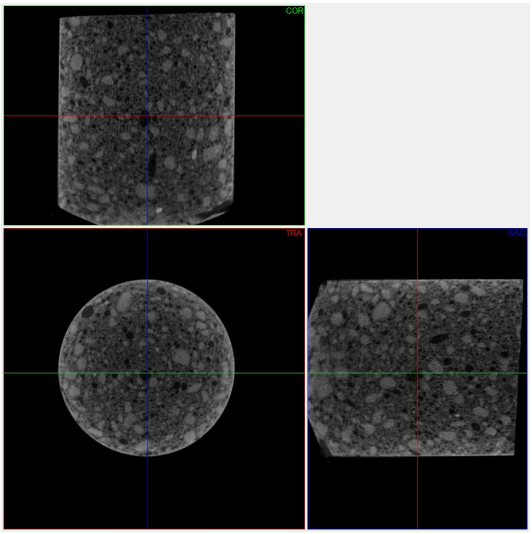


Figure J.18: Micro-CT of Pavistone 2K + 58%wt Residue + Silica

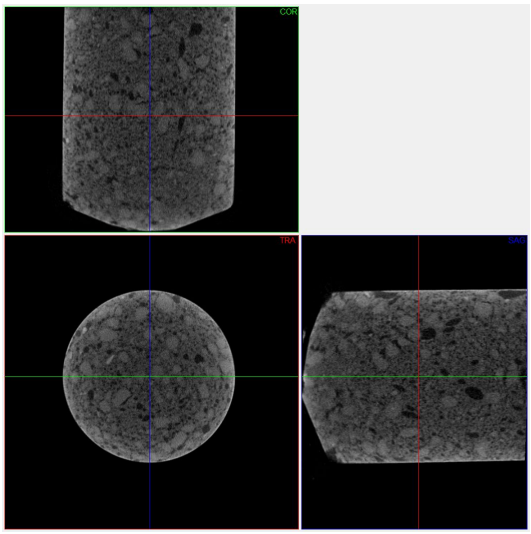
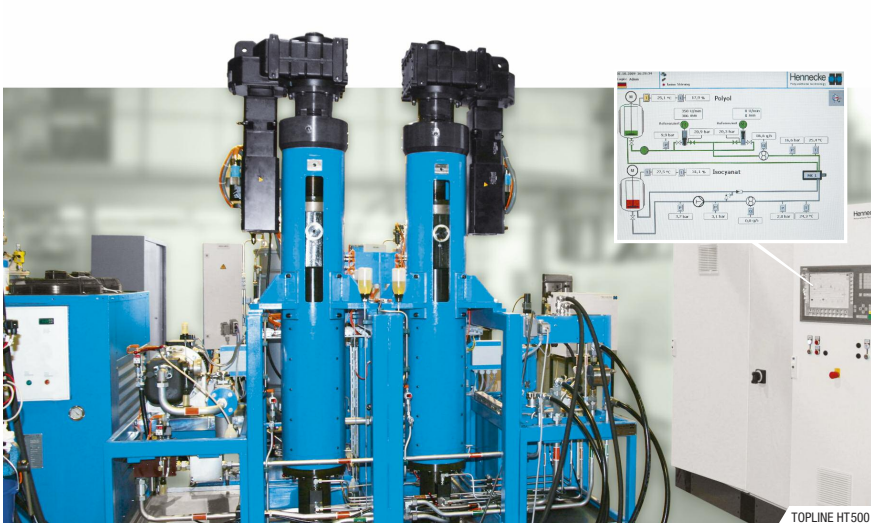


Figure J.19: Micro-CT of Pavistone 2K + 65%wt Residue + Silica

Appendix K: Data sheet of Topline HT80

TOPLINE HT



TOPLINE HT500

Einsatzspektrum

Das verschleißfeste und rückwirkungsfreie Dosiersystem der TOPLINE HT80 / HT180 mit Tandem-Tauchkolbenpumpen ist auf extreme Anforderungen im unteren bis mittleren Leistungsbereich zugeschnitten.

Mit der Baugröße HT500 steht zudem eine Dosiermaschine zur Verfügung, mit der auch Austragsleistungen pro Komponente von 500 cm^3/s (Kontibetrieb) und 1.000 cm^3/s (Hubbetrieb) realisiert werden können.

Es kann sowohl ungefüllte als auch gefüllte Komponenten verarbeiten: absolut rezepturgetreu, im Schuss- und Kontibetrieb.
HT-Maschinen eignen sich besonders zur Verarbeitung von:

- Polyolen aus nachwachsenden Rohstoffen
- Recyclat-Polyolen
- Polyolen mit Füllstoffen wie PUR-Regrind oder abrasiven Füllstoffen wie Glasfasern, Kreide, Melamin, Schwerspat etc.
- Abrasiven Farbpigmenten
- Hochviskosen Rohstoffsystemen
- Polyolen mit festen Flammschutzmitteln wie z.B. Ammoniumpolyphosphat und Blähgraphit

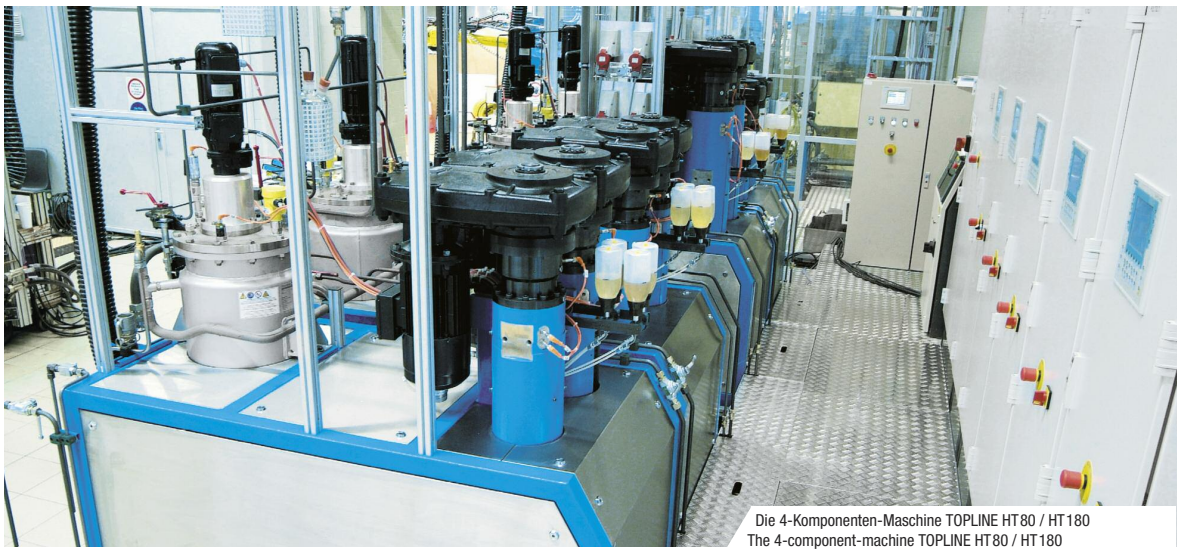
Fields of application

The wear-resistant, reactionless TOPLINE HT80 / HT180 metering system with tandem plunger pumps is suitable for extreme demands in the lower to medium output range.

The HT500 type of metering machine also enables outputs of 500 cm^3/s (continuous operation) and 1.000 cm^3/s (stroke operation) per component to be achieved.

Not only filled but also unfilled components can be processed in absolute conformity with the formulation both in a discontinuous and continuous operation.
HT machines are especially suitable for the processing of:

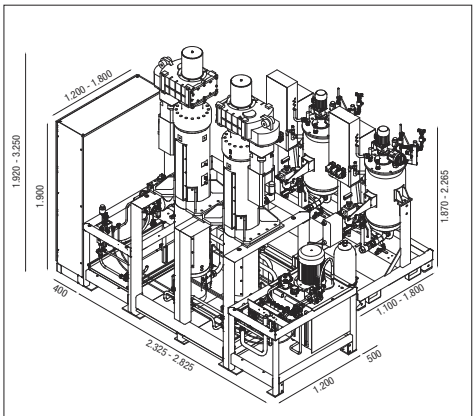
- Polyols made from regrowing raw materials
- Recycled Polyols
- Polyols with fillers such as PUR-regrind or abrasive fillers like glass fibres, calcium carbonate, melamine, barium sulphate, etc.
- Abrasive coloured pigments
- High-viscosity raw material systems
- Polyols with solid fire retardants such as ammonium phosphate and expanded graphite



Die 4-Komponenten-Maschine TOPLINE HT80 / HT180
The 4-component-machine TOPLINE HT80 / HT180

Technische Daten:
Technical data:

		HT 80	HT 180	HT 500
Einzelkomponentenausstoß bei kontinuierlichem Betrieb Single-component output in continuous operation	[cm³/s]	80	180	500
Bei Hubbetrieb At stroke operation	[cm³/s]	160	360	1.000
Verarbeitungstemperaturen Processing temperatures	[°C]	20 – 50°		
Absicherungsdruck Safety pressure	[bar]	300	300	320
Mischkopftypen Mixhead types		MN, MT18 /F		
Behältergrößen Tank sizes	[L]	60 – 250 (500)		
Gewicht Weight	[kg]	1.705	ca. 2.200	4.600



Bemaßung TOPLINE HT500 (in mm)
Dimensions TOPLINE HT500 (in mm)

500 DHE 01/15 RER.C / / SG 12.1

Hennecke GmbH · Birlinghovener Str. 30 · 53757 Sankt Augustin (Germany)
Email: info@hennecke.com · See www.hennecke.com for worldwide locations

Appendix L: Data sheet of Zamak feeder

SIDE FEEDERS

SINGLE-SCREW EXTRUDERS |FOR PLASTIC MATERIALS|
TWIN-SCREW EXTRUDERS |FOR PLASTIC MATERIALS|
SINGLE-SCREW EXTRUDERS |FOR RUBBER AND SILICONE|
ROLLING MILLS
MICROINJECTION MOULDING MACHINES
TEST STAND FOR DURABILITY TESTS
PRODUCTION LINES
SERVICES AND TESTS
INJECTION MOULDING MACHINES
DOWNLOAD OUR NEW CALATOG >>
SINGLE-SCREW EXTRUDERS |FOR PLASTIC MATERIALS|
TWIN-SCREW EXTRUDERS |FOR PLASTIC MATERIALS|
SINGLE-SCREW EXTRUDERS |FOR RUBBER AND SILICONE|
SERVICES AND TESTS

Production R&D

Our side dispensers include co-rotating and counter-rotating models. The equipment is suitable for all ZAMAK MERCATOR extruders. The feeder is an advanced feeding device for extrudate-processing sections, additives, and fillers in the form of granules or powder, as well as cut fibres. Our side-feeding systems allow us to increase performance and stabilise our production process, as well as improve product quality. These devices are very efficient and compact, so they don't need much space to fulfil their function.

Technical specifications

Side dispensers

Side dispensers		2 x 12mm	2 x 16mm	2 x 20mm	2 x 24mm	2 x 32mm
Number of screws	[pcs]	2				
Screw diameters	[mm]	12	16	20	24	32
Screw design		Monolithic				
Screw length L/D		8				
Maximum drive power	[kW]	Selected individually, depending on the diameter of the screws, the number of dispensers, the mutual distance of the dosing ports, and the power range of the used gear motors 0.55 - 075 - 1.1 - 1.5 - 2.3 - 3 - 4kW.				
Maximum crew speed	[rpm]	Customised Depends on the drive power and maximum screw torque.				
Maximum torque		Customised Depends on the drive power and maximum screw speed.				
Torque measurement	[Nm]	Motor-torque measurement				
Torque-measurement accuracy	[%]	< ±3%				
Torque-measurement resolution		0,1				
Max. operating pressure	[bar]	200				
Type of cylinder internal surface		Monolithic nitrided / hardened / HIP inserts / other coatings				
Direction of screw rotation		Co-rotation (C) Counter-rotation (B)				
Hopper system		Yes (installation of gravimetric or volumetric dispensers possible)				
Control system		Ethernet PowerlInk or CANopen integrated with the extruder				

SINGLE-SCREW EXTRUDERS |FOR PLASTIC MATERIALS|
TWIN-SCREW EXTRUDERS |FOR PLASTIC MATERIALS|
SINGLE-SCREW EXTRUDERS |FOR RUBBER AND SILICONE|
ROLLING MILLS
MICROINJECTION MOULDING MACHINES
TEST STAND FOR DURABILITY TESTS
PRODUCTION LINES
Filament production line for 3D printers
Rubber-production line
Medical-tubing-production line
Pelletisation line
Granulation line
Optical-fibre-coating line
Fibre-(plastic)stretching line
SERVICES AND TESTS

Zamak Mercator Sp. z o.o.
ul. J. Piłsudskiego 63
32-050 Skawina
NIP: 679-248-81-95
Administrative Office
tel.: (12) 276 07 20
tel.: (12) 276 84 69
biuro@zamakmercator.pl

Appendix M: Hennecke MT Mix-heads

Auswahl Kriterien / Selection of criteria Mischkopftypen / Types of Mixhead	Leistungsdaten / Performance data			Abmessungen / Dimensions*				Eintrags-Art / Type of injection				Anwendung / Application						Accessories
	Anzahl Komponenten Number of components	Laminare Austrag-leistung bei Eintrag in offene Form [cm³/s] Laminar output with injection into open mould [cm³/s]	Austragsleistung bei Anbau an Form [cm³/s] Mixture attachment to mould [cm³/s]	Höhe [mm] Height [mm]	Länge [mm] Length [mm]	Breite [mm] Width [mm]	Gewicht ca. [kg] Weight approx. [kg]	Eintrag in offene Form Injection into open mould	Anbau an Werkzeug Attachment to mould	Sprühen Spraying	Conti-Schäumen Continuous foaming	Hart Schäum Rigid foam	Weich Schäum Flexible foam	Integral Schäum Integral skin foam	Pentan Pentane	Direkt für Fässpfe Direkt valve for fillers	Spezial Systeme Special systems	Gleich-druckdüsen Constant pressure injectors
Umlenkmischköpfe MT: rutengesteuert / MT: deflection mixheads, groove-controlled																		
MT 3	2	3 - 20	20	215	100	90	4	+	+			■	+	+	■		+	
MT 6	2	8 - 50	50	290	195	80	8	+	+			+	+	+	+			✓
MT 8	2	25 - 150	300	395	170	130	17	+	+			+	+	+	+		■	✓
MT 12	2	50 - 300	600	400	175	130	17	+	+			+	+	+	+		■	✓
MT 12-4	4	50 - 300	600	410	205	185	24	+	+			+	+	+			■	✓
MT 18	2	125 - 600	1.200	450	200	140	24	+	+			+	+		+	✓	■	✓
MT 18-4	4	125 - 600	-	450	215	185	31	+				+	+		+		■	✓
MT 22	2	250 - 950	2.000	570	256	160	38,7	+	+			+	+		+			✓
MT 22-6	6	200 - 750	-	600	330	220	46	+	+				+		+		■	✓
MT 26	2	300 - 1.300	2.600	570/660	260	185	48/50	+	+			+	+		+		■	✓
MT 36	2	500 - 2.500	5.000	865	343	262	100	+				+	+		+		■	✓
Linearmischköpfe MN: rutengesteuert / MN: linear mixheads, groove-controlled																		
MN 6-3 RTM	3	-	6 - 80	65	155	65	4		+								+	
MN 6-3 CSM	3	6 - 40	-	65	155	65	4			+							+	
MN 8 CSM	2	20 - 160	-	90	20	165	7			+							+	✓
MN 10 CSM	2	15 - 250	-	175	400	175	14		+	+							+	✓
MN 10 RTM	2	-	15 - 250	130	378,5	180	18	+[2]	+								+	✓
MN 10-3 RTM	3	-	15 - 250	130	378,5	180	18	+[2]	+								+	✓
MN 10-4 CSM	4	30 - 250	-	175	300	175	13			+						✓	+	✓
MN 14	2	50 - 300	-	125	335	190	14				+				+		+	✓
MN 16-4	4	-	500 - 3000	180	480	180	27		+			+				✓	+	
MN 20-4	4	-	1.000 - 5.000	210	485	210	39		+							✓	+	
MN 30-4	4	-	2.000 - 10.000	225	545	225	48		+							✓	+	
Koaxialmischköpfe MX: rutengesteuert / MX: coaxial mixheads, groove-controlled																		
MX 8	2	25 - 150	300	370	200	135	21	+	+			■	■	+	+		+	✓
MX 12	2	50 - 300	600	375	220	140	24	+	+			■	■	+	+		+	✓
MX 12-3	3	50 - 300	600	375	220	140	24	+	+			■	■	+	+		+	✓
MX 18	2	125 - 600	1000	415	240	140	31	+	+			■	■		+		+	✓
Mischköpfe MD: druckgesteuert / MD: mixheads, pressure-controlled																		
MD 11	2	250 - 1.000	-	55	105	220	2				+	+			+			✓
MD 16	2	70 - 1.000	-	55	105	220	2				+	+			+			✓
Mischköpfe luftgereinigt / Air-cleaned mixheads																		
ML 12	2	150 - 600	-	175	150	100	7	+				+	+	+	+		■	
ML 18	2	1.000 - 3.500	-	200	160	160	9	+				+	+		+		■	
ML 25-4	4	2.000 - 9.000	-	225	150	225	20	+				+	+		+		■	
MXL 14	2	100 - 1.500	-	240	225	180	12	+				+	+		+		■	✓
MXL 25	2	750 - 5.000	-	300	285	235	21	+				+	+		+		■	✓
Rührermischköpfe / Stirrermixheads																		
MEL 6C	4 (+ 2)	7 - 500	-	830	320	350	86	+									+[1]	
MEL 8C	4 (+ 4)	7 - 260	-	540	340	175	30	+									+[1]	
MSL	variabel/variable	80 - 10.000	-	350 - 1.550	115 - 180	115 - 180	25 - 185	+			+		+			✓	+	✓
MNR 42	2	1.500 - 7.500	-	1.570	560	300	270	+									+	

* Je nach technischer Ausstattung abweichend
Different depending on technical equipment

✚ Beste Eignung / Perfectly suitable
■ Geeignet / Suitable
✔ Verfügbar / Available

GSM Composite Spray Moulding
RTM Resin Transfer Moulding

[1] Für Elastomer-Anwendungen
For elastomer applications

[2] Mit Breitschlitzdüse
With wide slot nozzle