JH Major Project

by James Howe

Submission date: 15-Mar-2024 01:43PM (UTC+0000)
Submission ID: 226769802
File name: 18036_James_Howe_JH_Major_Project_461145_1537704660.docx (11.53M)
Word count: 11111
Character count: 57562



University of Wales Trinity St David
Ordnance, Munitions and Explosives

BA

Academic Year 2019 - 2024

James Howe

Validation of a novel asymmetric RAM vessel to enable the scale-up of mixed in case energetic paste explosives

Supervisor: Gareth Collett
Associate Supervisor: David Holley
January 2024

This thesis is submitted in partial fulfilment of the requirements for the degree of BSc Ordnance, Munitions and Explosives

© UWTSD 2024. All rights reserved. No part of this publication may be reproduced without the written permission of the copyright owner.

Academic integrity declaration

I declare that:

- the thesis submitted has been written by me alone.
- the thesis submitted has not been previously submitted to this university or any other.
- that all content, including primary and/or secondary data, is true to the best of my knowledge.
- that all quotations and references have been duly acknowledged according to the requirements of academic research.

I understand that to knowingly submit work in violation of the above statement will be considered by examiners as academic misconduct.

15/03/2024

X 

James Howe
Student
Signed by: Howe James A

Abstract

In this paper, experiments are conducted to provide confidence in a novel vessel's ability to mix similarly to a pre-established vessel. Resonant Acoustic Mixing (RAM) technology has been utilised in combination with Additive Manufacturing (AM) to mix a range of formulations for the experiments. In commissioning the novel vessel, a six fold increase in material that can be concurrently mixed was achieved. The results provided confidence that the vessel is suitable for use with the Resodyn LabRAM II.

These experiments were dual purpose, also providing insight into the effects of the solids loading and formulation of an energetic material on its extrudability, in the form of an inert simulant. The extrudability of these formulations was analysed using a customised capillary rheometer that was developed to measure paste extrusion. The results of the solids loading investigation present a trend where the material becomes more extrudable with a lower solids loading. However, an ideal extrusion profile was not reached within the test parameters.

A visual aid for assessing mix quality was also developed by stopping and analysing material at intervals during a RAM mix. This led to an investigation into the suitability of a range of dyes. The extrusion characteristics of these formulations were analysed and minimal differences were found between the selected solid dyes. On the contrary, the liquid dye exhibited worse extrusion characteristics and was visibly less incorporated into the material.

Keywords:

Additive Manufacturing

Energetic Materials

Resonant Acoustic Mixing

Formulations

Table of Contents

Academic integrity declaration i
Abstract 2
Acknowledgements 4
List of Figures 5
List of Tables 7
1 Introduction 8
2 Research 9
2.1 Additive Manufacture 9
2.2 Formulations 12
2.3 Resonant Acoustic Mixing (RAM) 13
3 Experimental Methods 15
3.1 MikeBot Material Characterisation 16
3.1.1 Aim 16
3.1.2 Materials and Equipment. 16
3.1.3 Methods 17
3.1.4 Analysis 18
3.2 Hex-Vessel Commissioning 19
3.2.1 Aim 19
3.2.2 Materials and Equipment. 19
3.2.3 Methods 21
3.2.4 Analysis 27
3.3 Print Bed Testing 29
3.3.1 Aim 29
3.3.2 Materials and Equipment. 29
3.3.3 Methods 30
3.3.4 Analysis 31
3.4 Solids Loading Investigation 31
3.4.1 Aim 31
3.4.2 Materials and Equipment. 32
3.4.3 Methods 32
3.4.4 Analysis 32
3.5 Dye Colour Chart 33
3.5.1 Aim 33
3.5.2 Materials and Equipment. 33
3.5.3 Methods 34
3.5.4 Analysis 35
4 Experimental Results 36
4.1 Hex-Vessel Commissioning 36
4.1.1 Empty Vessel Check 36
4.1.2 Two Opposing Syringes 36

4.1.3 Single Asymmetric Syringe 37
4.1.4 Full Capacity 37
4.1.5 Three Asymmetric Syringes 39
4.2 Print Bed Testing 42
4.3 Solids Loading Investigation 49
4.4 Dye Colour Chart 50
5 Discussion 53
6 Conclusion 57
6.1 Recommendations for Further Research 58
6.2 Reflection 59
7 References 60
List of Abbreviations 62
Appendix A MikeBot Nozzle and Cylinder Selection 63
Appendix B RAM Formulations 63

Acknowledgements

I would like to thank my mentors Michael O'Donnell and Hatty Hoskyns at the Defence Science and Technology Laboratory (Dstl) for their support throughout this project.

Thanks must be given to The Falcon Project (TFP) for providing their expertise on the RAM technology and for equipping me with the appropriate knowledge on energetic formulations.

I greatly appreciate the time granted to me by the Future Energetics (FE) work programme to complete this task.

List of Figures

Figure 1 MikeBot Full Assembly 11

Figure 2 MikeBot Diagram [6] 12

Figure 3: Graph showing resonance phenomenon and the relationship between the frequency, amplitude and power [15] 14

Figure 4 Single Syringe Vessel 16

Figure 5 Resodyn LabRAM II 17

Figure 6 Assembled Hex-Vessel 19

Figure 7 Hex-Vessel Exploded Diagram 20

Figure 8 Annotated Hex-Vessel inside the LabRAM II 21

Figure 9 G-Ramp RAM Recipe 22

Figure 10 Two Opposing Syringes Assembly 23

Figure 11 Two Opposing Syringes Assembled 24

Figure 12 Single Asymmetric Syringe Assembly 25

Figure 13 Full Capacity Assembly 26

Figure 14 Three Asymmetric Syringes Assembly 27

Figure 15 Print Bed Example Materials 30

Figure 16 Single Syringe Vessel 33

Figure 17 Post-Mix Hex-Vessel with Opposing Syringes 37

Figure 18 Full Capacity Hex-Vessel MikeBot Analysis 38

Figure 19 Post-Mix Hex-Vessel with Three Asymmetric Syringes 39

Figure 20 Hex-Vessel Lid Post-Mix 40

Figure 21 Three Asymmetric Syringes Hex-Vessel MikeBot Analysis 40

Figure 22 Glass Removal and Debris 43

Figure 23 PEI Powder Coated Spring Steel 43

Figure 24 PEI Powder Coated Spring Steel with Curing 44

Figure 25 PEI Sticker on Spring Steel 45

Figure 26 PEI Sticker on Spring Steel with Curing 45

Figure 27 Card Removal 46

Figure 28 Card Residue 47
Figure 29 Masking Tape on Glass..... 48
Figure 30 Antistatic Bag on Glass 48
Figure 31 Solids Loading MikeBot Analysis..... 49
Figure 32 Red Dye No.2..... 50
Figure 33 Red Dye No.2 Visual Analysis..... 51
Figure 34 Dye Investigation MikeBot Analysis..... 51

List of Tables

Table 1 RAM Formulation Calculator Example [17] 18
Table 2 EPIM RAM Recipe 18
Table 3 Print Quality Grading 30
Table 4 Ease of Removal Grading 31
Table 5 Liquid Dye Formulation Example 34
Table 6 Solid Dye Formulation Example 34
Table 7 Print Bed Testing Results 42
Table 8 PEI Powder Coated Spring Steel Removal 44
Table 9 PEI Sticker on Spring Steel Removal 46

1 Introduction

The Novel Manufacturing work package, where this project is situated, is funded by the Future Energetics Core (FEC) programme that looks at developing novel techniques to improve or enhance the manufacture of energetic materials. Emerging manufacturing techniques, such as Resonant Acoustic Mixing (RAM) and Additive Manufacture (AM), are being explored for use within the energetics industry.

In this project, RAM provides the ability to rapidly prototype and mix formulations for use in a customised paste extrusion 3D printer. RAM is desired in the energetic sector due to its ability to mix materials without blades (eliminating possible initiation stimuli), at faster rates and via remote control (eliminating the risk to operators). AM is desired because of its ability to produce bespoke shaped energetic charges at any time or place. Unfortunately, the downside to AM is that specific material properties are required by the formulation in order to produce a successful print, which can often be a trade-off between energetic loading and print quality. Additionally the number and size of charges produced per print is restricted by the size of the print bed and by the capacity of the material loading vessel.

This project utilised RAM technology to commission a new mixing vessel as part of a scale-up programme that will allow a six fold increase in the amount of material that can be mixed simultaneously. This process adopted the Mixed In Case (MIC) concept where the mixing vessel doubles up as the material casing, eliminating the charge filling step required in traditional methods [1]. Therefore, the ‘Cases’ which held the mixed formulation can be directly placed in to the customised 3D printer for instant extrusion.

A series of activities were carried out. This included; the creation of a dye colour chart, print bed testing, hex vessel commissioning and a solids loading investigation. The aim of these tests was to assess the mix quality of the RAM and to determine the performance of the novel vessel.

2 Research

2.1 Additive Manufacture

3D printing, also known as AM, is a process of creating an object one layer at a time. Comparatively, subtractive manufacturing methods such as machining remove object parts to create a new product [2]. It has become more widely accessible as the cost of the technology has reduced over time and open-source resources have become available [3].

AM has been shown to increase manufacturing efficiency, allow the use of new materials (such as metals and ceramics), revolutionise the production line (by allowing the creation of bespoke products), increase production speed and reduce manufacturing costs. It is used in multiple industries such as construction, pharmaceuticals, agriculture and transport [4] [5]. These industries utilise the following seven AM methods to fit their needs [6] [7]:

- Material extrusion
- Vat photopolymerisation
- Binder jetting
- Material jetting
- Powder bed fusion
- Directed energy deposition
- Sheet lamination

Each method has its own benefits and unique applications [8] [9]. This project focuses on the use of the ‘material extrusion’ technique, which is desired when using energetic materials. Energetic materials are defined as any material that contains a “high amount of chemical energy which can be released through a reaction in order to do some type of work” [6]. The ‘material extrusion’ method is desirable because it does not require as many initiating stimuli for manufacture. For example, it does not subject the system to electrostatic or thermal energy. However, friction is utilised in the extrusion process which deems it suitable for secondary explosives but not primary explosives. The Fused Deposition Modelling (FDM) printers, which utilise ‘material extrusion’, are easier to modify

for energetic materials and cheaper to procure and maintain. This subset of AM has shown great potential for the customisation of energetic material properties and performance [10]. Additionally, another benefit to using AM with energetic materials over some other manufacturing techniques is the ability to conduct the work remotely, removing the risk to personnel.

One shortcoming of AM by paste extrusion is that lots of formulation development is required to create a paste that has desirable solids loadings that can also be extruded with relative ease and safety.

The MikeBot, a type of strain controlled capillary rheometer, has been developed by Dstl specifically to measure paste extrusion. Flow measurement techniques are available such as angle of repose for powders and viscosity for liquids, however, historically there have not been quantitative experiments available to determine the flowability and extrudability of an energetic paste in the context of AM.

The MikeBot (see Figure 1) is comprised of a stepper motor, load cell and a cartridge holder attached to a commercially available Potterbot 3D printer. The motor on top pushes a piston into a cylinder which contains the material. The load cell sits between the motor and the piston and records the force applied by the motor onto the material. There is a nozzle located at the bottom of the cylinder which the material is extruded from. The ratio of diameters between the nozzle and the cylinder is used to calculate the speed at which the print bed of the 3D printer should move.

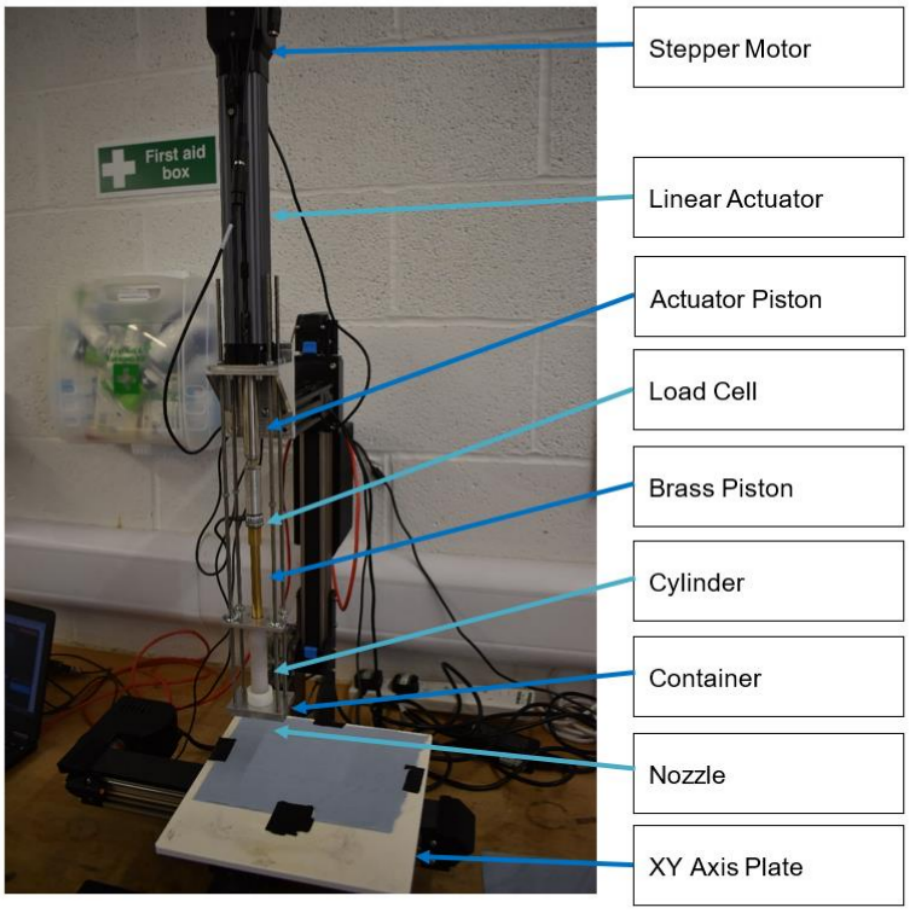


Figure 1 MikeBot Full Assembly

The MikeBot determines flowability by extruding a paste through a specified geometry and measuring the associated force.

The stepper motor provides a rotational force to extend the linear actuator vertically. The load cell, located between the actuator and the brass piston, outputs data to the laptop that can be interpreted to provide a force read-out in the vertical direction. The piston enters the plastic cylinder that holds the material that's being tested. The container is a set of plates that clamps down on the cylinder to hold it in place as the piston is pushed into it.

The vertical extension of the actuator extrudes the material through the nozzle at the bottom (see Figure 2).

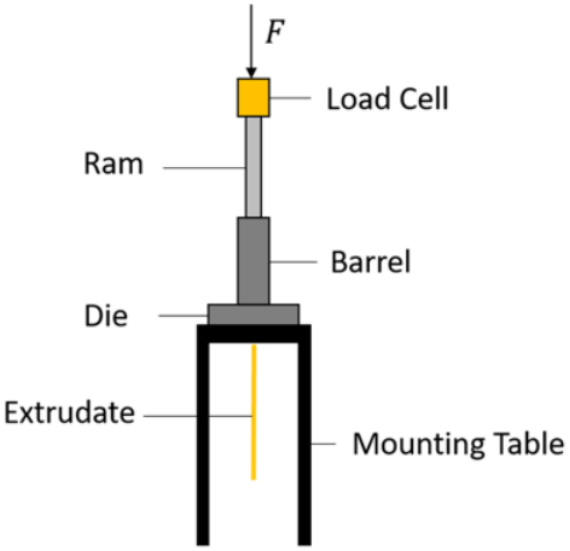


Figure 2 MikeBot Diagram [6]

2.2 Formulations

A formulation is a mixture that is formed by mixing several different substances together in carefully measured quantities to ensure the product has the required properties. Some examples of energetic formulations include Polymer Bonded Explosives (PBXs) and solid rocket propellants. An example PBX might consist of a bimodal distribution of RDX, aluminium and a binder system such as hydroxyl-terminated polybutadiene (HTPB) and isophorone diisocyanate (IPDI) [11]. The most widely used solid rocket propellant formulation consists of Ammonium Perchlorate (70%), aluminium (16%) and binder (14%) [12]. Ammonium perchlorate (AP) is considered “the work-horse oxidizer used in modern rocket propulsion systems” [12].

The formulation is a compromise between the energetic performance and the processability of the material. Maximising the explosive content (solids loading) would be ideal which would minimise the additives, in-turn making the formulation too viscous to carry out processing techniques such as casting, mixing and curing [1]. The smaller the particle size of the energetic material, the larger the overall surface area available and the easier the extrusion [13]. 4–400 μm particles are commonly used, where smaller particles allow for narrower nozzles. It is recommended to have a diameter equal to 10x the size of the largest particle [6].

Within this report a formulation comprising of Dymax and Melamine as an inert representative of Dymax and RDX is investigated. UV curable binders, such as Dymax, are used as a safe curing method for energetics. These utilise light radiation, which is a stimulus to energetics, instead of thermal radiation in the curing process. RDX is relatively safe to handle from an explosive perspective and is commonly used in PBX formulations. Melamine is chosen here as an inert substitute to mimic its extrusion properties and particle size.

Overall, it is an arduous task to develop a desired formulation from its starting materials. Conventional mixing techniques are time consuming and difficult to perfect formulations with [1], therefore exploration in novel techniques is beneficial.

2.3 Resonant Acoustic Mixing (RAM)

RAM is an emerging mixing process that utilises a vibrating platform at around 60 Hz frequency to generate material movement through a sound-induced interaction. This provides more rapid and uniform mixing than conventional mixing techniques within the mix vessel [14]. RAM operates at the resonant condition of its mechanical system. This allows for high amplitude mixing with a reduced energy cost due to a near lossless energy transfer between the mechanical system and the material [1]. Figure 3 shows the phenomenon of resonance allowing for maximised amplitude and minimised power.

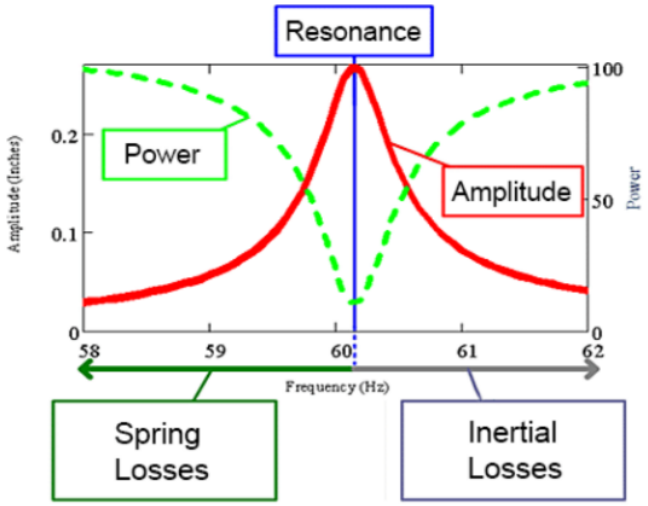


Figure 3: Graph showing resonance phenomenon and the relationship between the frequency, amplitude and power [15]

RAM has seen rapid uptake in a number of industries over the last 20 years including food, pharmaceutical and energetics [16]. Its increased popularity can be attributed to a variety of benefits. For starters, RAM mixing has been shown to dramatically reduce mix times when compared to traditional mixing methods. In some cases this is by orders of magnitude, whilst reproducibly maintaining a precise and uniform mix quality. [14] [16]. RAM can effectively batch mix a range of materials at reproducible scales up to 420kg [13]. A lot of these benefits contribute to a better efficiency and therefore lower running costs and a reduced impact on the environment. This is particularly noticeable when employing the MIC concept as the vessel doubles as the final container of the material, removing the need for material transfer. Finally, it is desired within the energetics industry, in particular, for its safety benefits. It can be controlled remotely, eliminating workers from the energetic area and there are no moving parts inside the vessel. This means that energy is imparted across the material removing the localised areas of high impact and friction. This reduces the explosive risk to process workers and facilities.

3 Experimental Methods

To commission the novel vessel and to characterise the inert material, the following experiments and procedures were developed;

1. A material extrudability assessment using a custom extrusion based characterisation machine, known as MikeBot.
2. A visual guide procedure used to assess mix quality by intermittently pausing a RAM program.
3. A suitability assessment used to commission a novel vessel for the RAM.
4. A procedure for assessing print bed adhesion of materials.
5. An extrudability investigation to evaluate the effect of a formulation's solids loading.

These are detailed in sections 3.1 - 3.5 below.

3.1 MikeBot Material Characterisation

3.1.1 Aim

The aim of this experiment is to use an extrusion apparatus colloquially coined "MikeBot" to analyse the extrudability of various formulations mixed on the RAM, in the form of pressure versus extruded distance graphs.

The solids loading experiments (Section 3.4) and dye experiments (Section 3.5) utilised analysis techniques from the MikeBot so it was important to become familiar with its operation first.

3.1.2 Materials and Equipment

The inert formulations, which from here on out will be referred to as the Extrudable Paste Inert Mimic (EPIM), used Dymax 3069 UV curable binder and Melamine (particle sizes 180-106μm, 106-63μm and <63μm) in varying quantities.

The LabRAM II (manufactured by Resodyn Acoustic Mixers) (see Figure 5) was prepared as per its standard operating procedure (SOP) for mixing the formulation [17]. The single syringe vessel (Figure 4) or the hex-vessel (Figure 6) was assembled based on the number of syringes that were required to be mixed for the experiment.

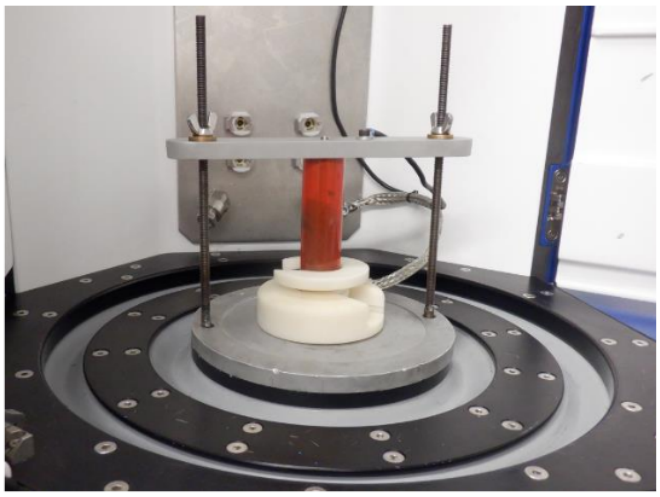


Figure 4 Single Syringe Vessel



Figure 5 Resodyn LabRAM II

The MikeBot was assembled as per Figure 1 and the Dstl Method Statement. The geometry (12.5 mm barrel and 3x20 mm nozzle) was chosen and a speed of 1 mm/s was set, see the MikeBot Nozzle and Cylinder Selection appendix for additional geometry guidance. The cylinder was filled with the RAM mixed formulation up to 1cm from the top (<25 g). This was placed between the metal container plates and tightened. The software was loaded and setup on the laptop.

3.1.3 Methods

The RAM Formulation Calculator (RAM FC) spreadsheet determined the formulation to be tested and the total mass required [18]. The required quantity of each material is shown in column 4 of Table 1 below. Column 5 contains the actual quantity measured on the balance.

Order	Material	Target Percentage	Required (g)	Actual (g)
1	Melamine 106-63μm	37.0	7.4	7.3990
2	Graphite Powder	1.0	0.2	0.2609
3	Dymax 3069	25.0	5.0	5.0529
4	Melamine 180-106μm	37.0	7.4	7.4016

Table 1 RAM Formulation Calculator Example [18]

The following RAM recipe was loaded and run each time (Table 2).

Step	Mix Force (G)	Time (minutes)
1	60	14
2	40	4
3	20	2

Table 2 EPIM RAM Recipe

The vessel was deconstructed and the syringe(s) were removed from the RAM. The required analysis was then conducted on the material through the selected method e.g. visual inspection or MikeBot analysis.

3.1.4 Analysis

After the material was extruded, a MikeBot script generated a text file with the experiment's information, the actuator position and the measured load. This was imported to Microsoft Excel and the extrusion pressure was calculated from the load measurements and the geometry of the cylinder. This data was processed into a graph of extrusion pressure (MPa) against ram (Piston) displacement (mm). Up to 6 series were added to the graph determined by the number of syringes used in an experiment.

3.2 Hex-Vessel Commissioning

3.2.1 Aim

The aim of this experiment was to assess a novel vessel's physical compatibility with the LabRAM II. It was also necessary to build confidence in its use through a material load scale up program and non-standard configurations such as an asymmetric loading.

The vessel will be used to manufacture the material that the proceeding experiments require.

3.2.2 Materials and Equipment

The experiment used the assembled Hex-Vessel mounted to the resonator on the LabRAM II (Figure 6). There were six bungs that the syringes fit onto and the metal lid had 6 corresponding caps to secure the syringes.

Figure 6 shows an overview of the Hex-Vessel inside the LabRAM II whilst it is mounted to the resonator. Figure 7 shows a CAD drawing of the vessel and Figure 8 shows a labelled schematic of the Hex-vessel inside the LabRAM II.

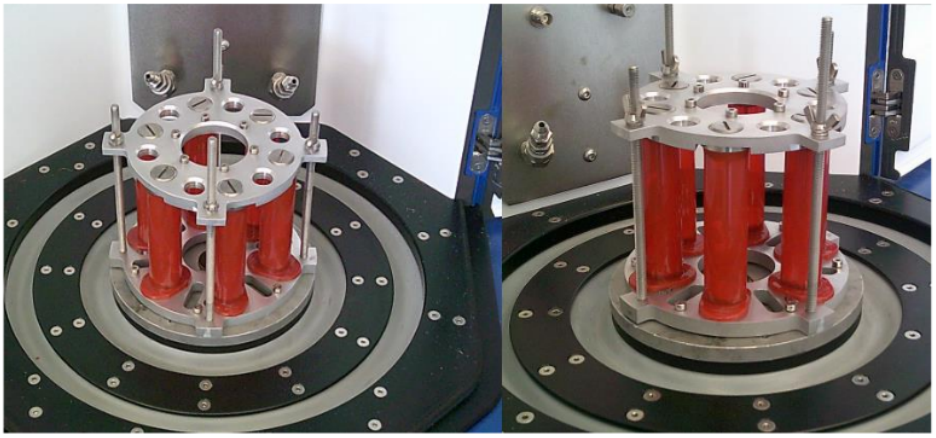


Figure 6 Assembled Hex-Vessel

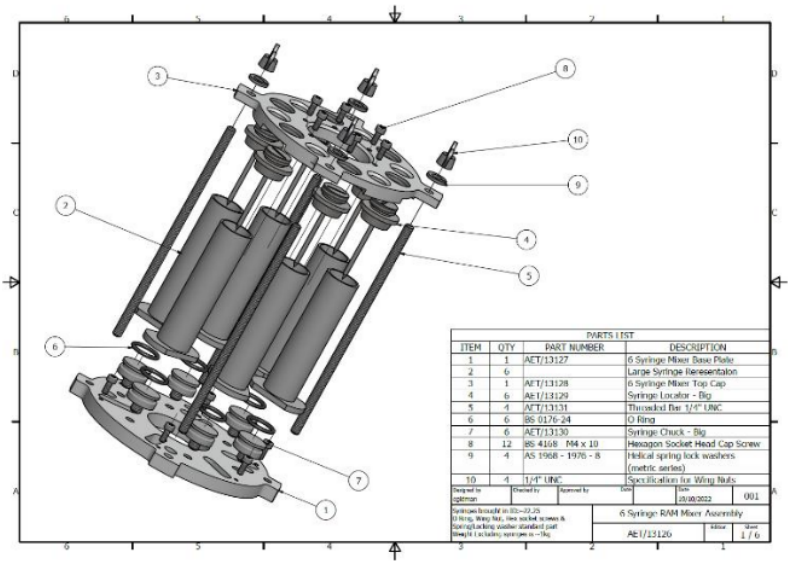


Figure 7 Hex-Vessel Exploded Diagram

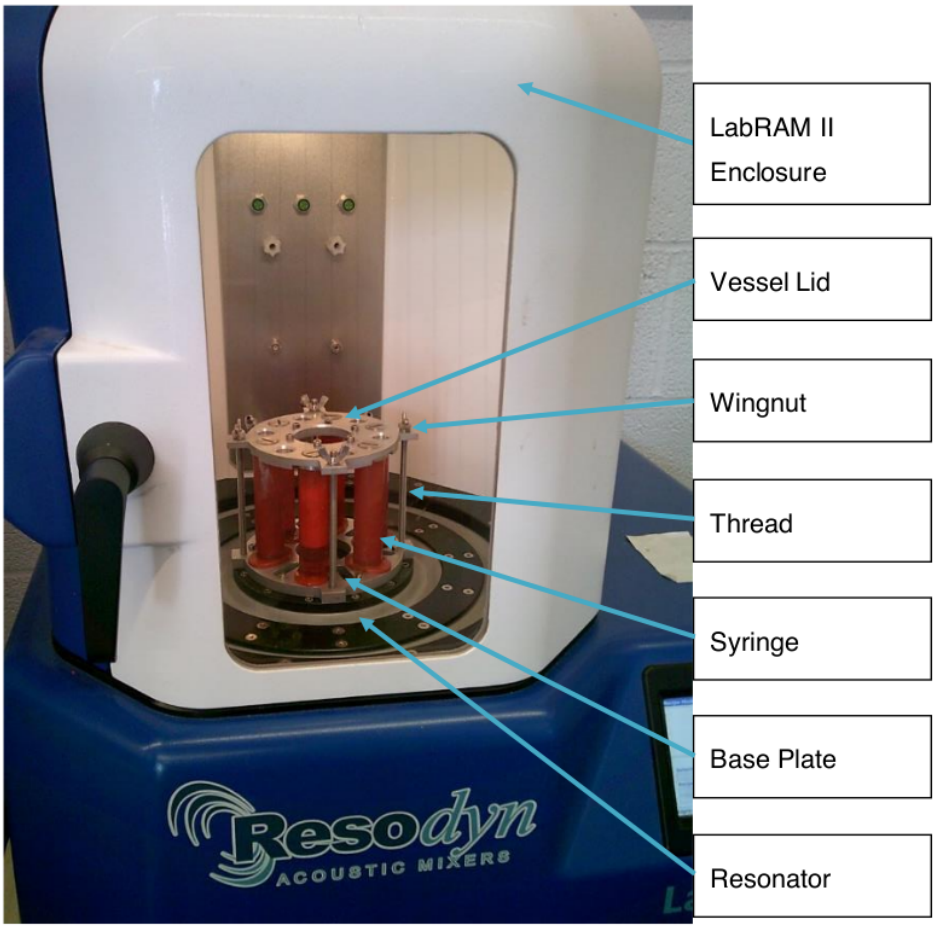


Figure 8 Annotated Hex-Vessel inside the LabRAM II

Syringes were required to provide material to the 3D printer. These were bought commercially (Nordson Orange Syringes, Part No. 7012143) and then modified on-site to remove the tips, providing a purely cylindrical geometry.

3.2.3 Methods

The Hex-Vessel base was secured to the LabRAM II's resonator using 4 radial bolts through the metal base plate. Each time the vessel was to be used, all six syringes would be fitted to the vessel, even if they weren't filled in the experiment, to allow adequate clamping of the vessel lid. The lid was fastened using wing nuts (see Figure 8) on the four outer threads.

Five different experiments were carried out and the setups are described below.

3.2.3.1 Empty Vessel Check

For this experiment, the vessel was assembled and attached to the RAM as described above. No material was used in the syringes.

The 'G-Ramp' RAM recipe was loaded which increased the acceleration at 10 G increments every 10 seconds as shown in Figure 9.

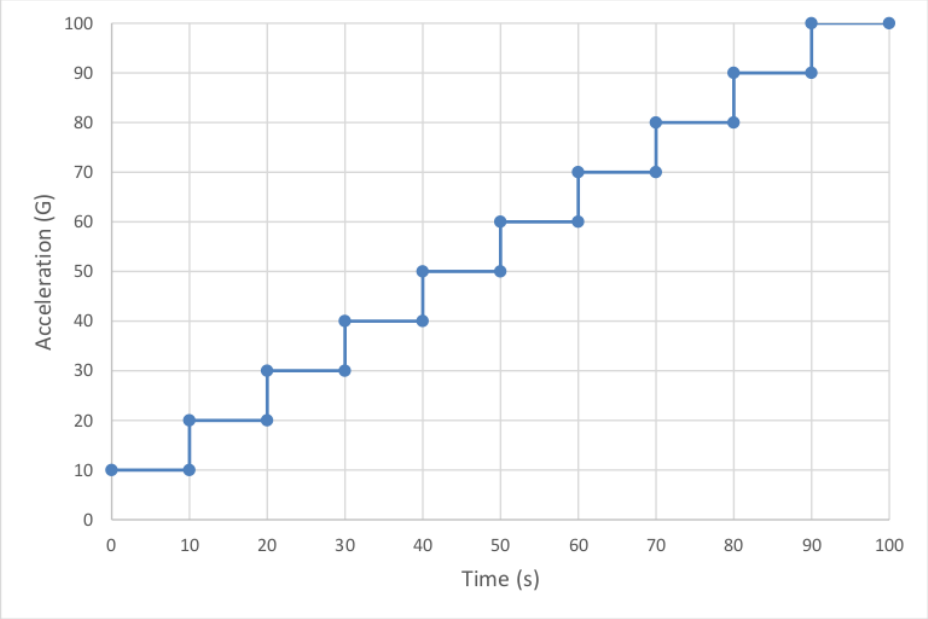


Figure 9 G-Ramp RAM Recipe

After the recipe was run, the LabRAM II's dashboard was monitored for power spikes and anomalies. The enclosure was observed for visible or audible vibrations until it reached 50 G, at which point the recipe was paused.

The enclosure was opened and the wingnuts were re-tightened where necessary. The recipe was resumed until the full 100 G sweep was complete. The tightness of the wingnuts was checked post experiment.

3.2.3.2 Two Opposing Syringes

Two syringes were filled with the chosen formulation, this was recorded using the RAM FC. They were placed opposite each other on the vessel and the remaining four spaces were filled with empty syringes as seen in Figure 10.

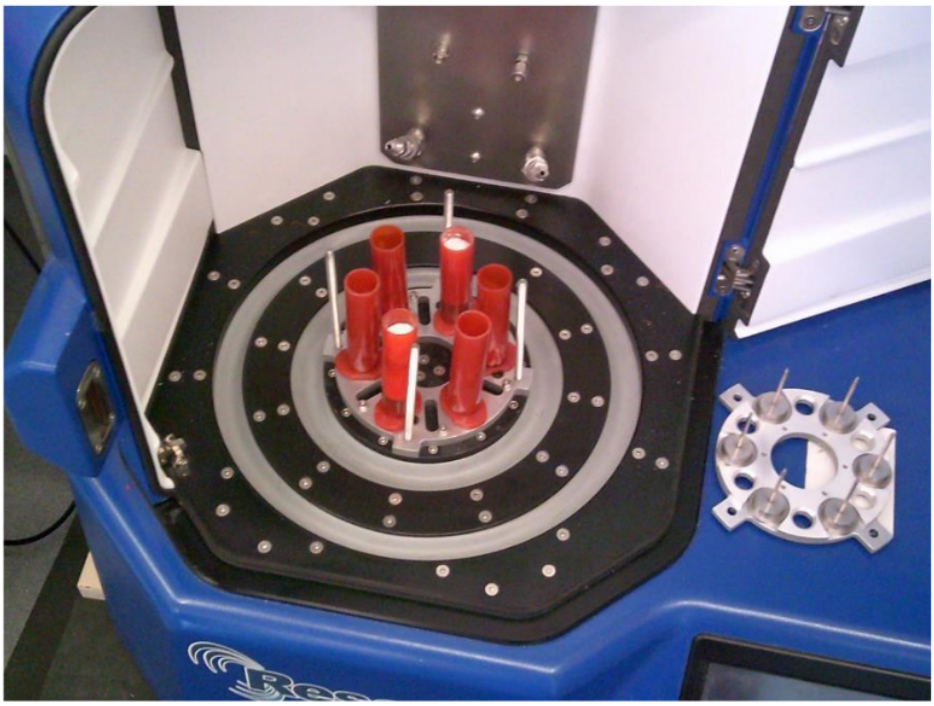


Figure 10 Two Opposing Syringes Assembly

The rest of the vessel was assembled and tightened, shown in Figure 11.

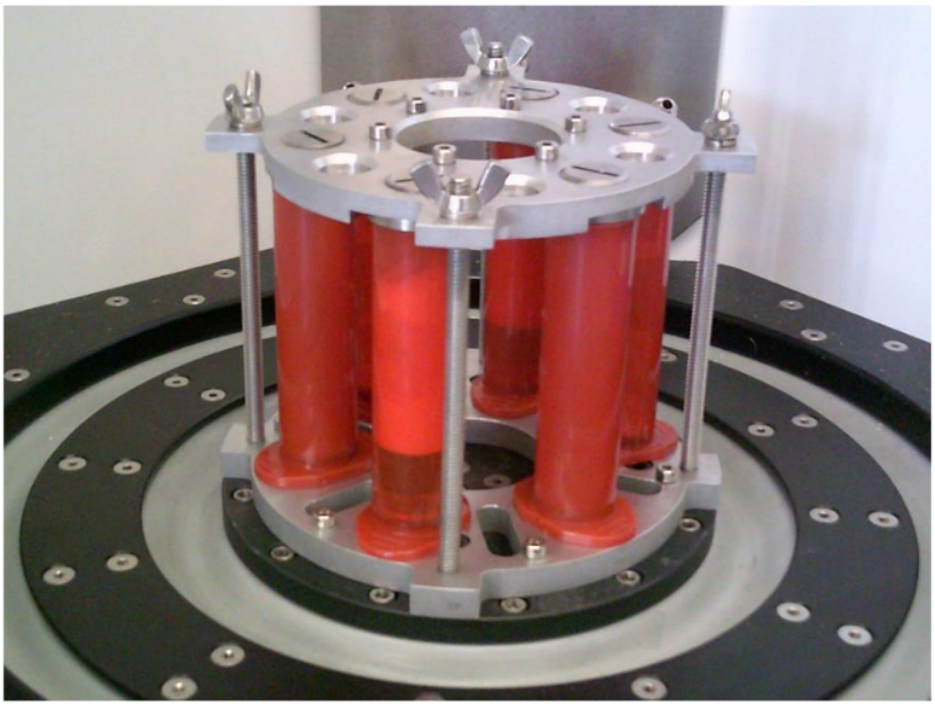


Figure 11 Two Opposing Syringes Assembled

The EPIM RAM recipe was run as per Table 2. Again, the dashboard was monitored for irregularities and the vessel for loose wingnuts.

The material was removed from the syringes, visually inspected and compared.

3.2.3.3 Single Asymmetric Syringe

A single syringe was filled with the chosen formulation, defined by the RAM FC. This was placed onto the vessel as seen in Figure 12 and the remaining 5 spaces were filled by empty syringes.



Figure 12 Single Asymmetric Syringe Assembly

The rest of the vessel was assembled and tightened. The EPIM RAM recipe was run as per Table 2. The dashboard was monitored for irregularities and alarms and the vessel for loose wingnuts.

The material was removed from the syringe, visually inspected and compared to previous results.

3.2.3.4 Full Capacity

Six syringes were filled with the chosen formulation, this was defined by the RAM FC. All syringes were placed onto the bungs on the vessel as seen in Figure 13.



Figure 13 Full Capacity Assembly

The rest of the vessel was assembled and tightened. The EPIM RAM recipe was run as per Table 2. The dashboard was monitored for irregularities and alarms then the vessel for loose wingnuts. The maximum RAM power utilised was also noted as this was the max load for the vessel.

The material was removed from each syringe and loaded onto the MikeBot for extrudability analysis.

3.2.3.5 Three Asymmetric Syringes

Three syringes were filled with the chosen formulation. This was recorded using the RAM FC. The syringes were placed next to each other on one side of the vessel. The other side was filled with empty syringes as seen in Figure 14.

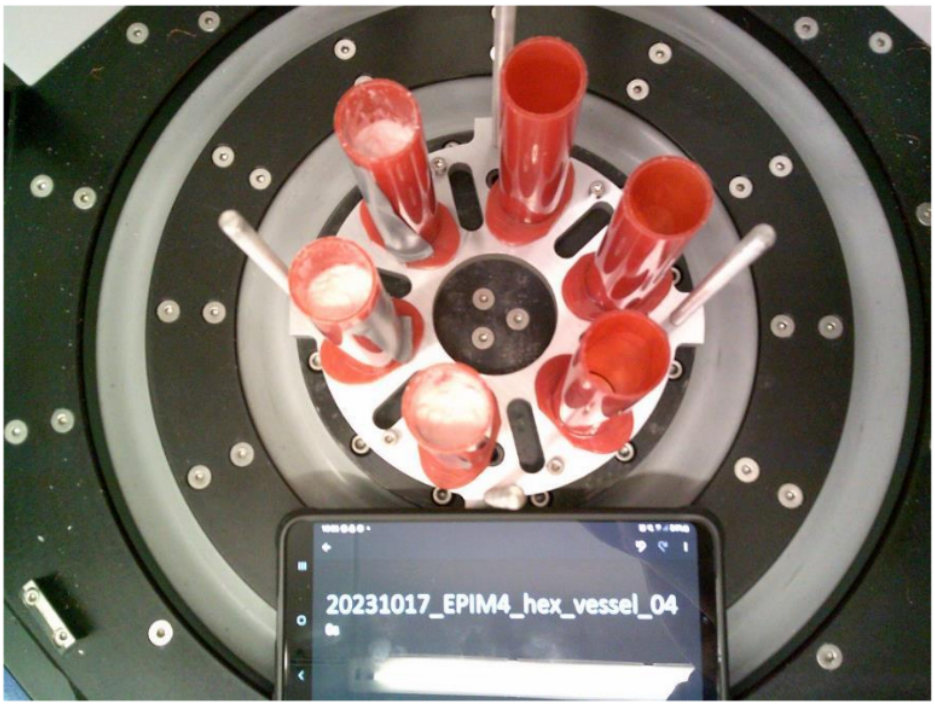


Figure 14 Three Asymmetric Syringes Assembly

The rest of the vessel was assembled and tightened. The EPIM RAM recipe was run as per Table 2. The dashboard was monitored for irregularities and alarms and the vessel for loose wingnuts.

The material was removed from each syringe and loaded onto the MikeBot for extrudability analysis.

3.2.4 Analysis

The Hex-Vessel was monitored for audible vibrations and loose wingnuts during all of the above methods.

After completing the mixes in 3.2.3.2 and 3.2.3.3, the material was removed from the syringe and visually inspected for surface sheen, binder rich areas, agglomerates and discontinuities.

Material from mixes 3.2.3.4 and 3.2.3.5 received visual inspection as well as extrudability testing on the MikeBot (equipment and procedure located in 3.1

MikeBot Material Characterisation). The RAM power usage was also monitored as these tests contained the highest material loading and therefore the highest total vessel mass.

3.3 Print Bed Testing

3.3.1 Aim

The aim of this experiment was to identify the best candidate for the print bed of a 3D printer from a selection of materials.

3.3.2 Materials and Equipment

The following materials were chosen and tested:

- Glass
- Polyetherimide (PEI) powder coated spring steel
- PEI sticker on spring steel
- PEI sticker on glass
- Card
- Masking tape
- Antistatic bag

These materials are shown in Figure 15. Glass was used as it came as standard with the 3D printer. PEI coatings are also commonly used and as such coated spring steel and a sticker were tested. Card and masking tape were investigated as disposable print bed solutions. Antistatic bags were tested as they are commonly used with explosives and compatibility testing had already been completed.

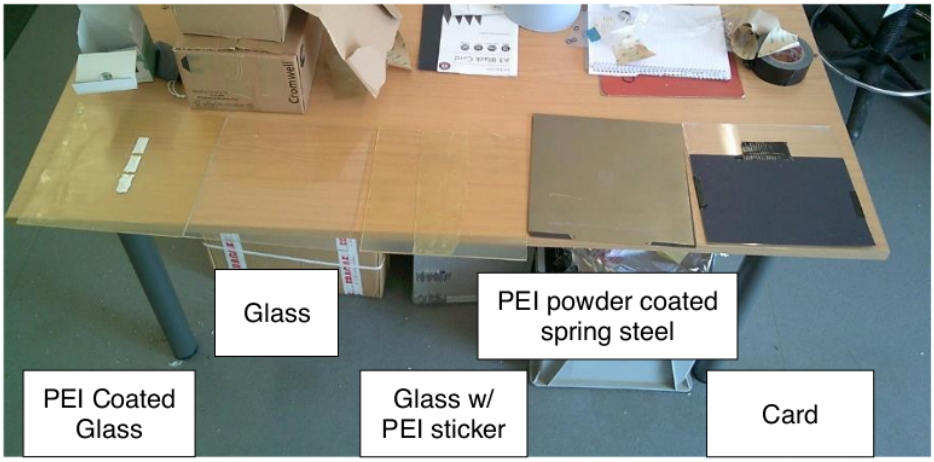


Figure 15 Print Bed Example Materials

3.3.3 Methods

The 3D printer was loaded with a RAM mixed EPIM syringe and a new print bed material was selected for each experiment. The 3D printer deposited the material on to the print bed that moved in the X and Y axis to create 3 rectangles. One with one layer, the next with two layers and the final with 3 layers of material.

For each print bed material, the print quality and the ease of removal were assessed as per Table 3 and Table 4 below. A grading system was developed for these properties to allow the materials to be compared quantitatively.

Grading for print quality	Comment
1	Does not stick at all
2	Sticks but pulls at nozzle. Easy to dislodge
3	Sticks fully

Table 3 Print Quality Grading

Grading for ease of removal	Comment
1	Very difficult to remove with all objects
2	Difficult to remove with metal spatula
3	Fairly easy to remove with metal spatula. Cannot be removed with plastic spatula
4	Difficult to remove with plastic spatula
5	Easy to remove with plastic spatula

Table 4 Ease of Removal Grading

The print quality assessed how well the extruded material stuck to the print bed during printing so that an accurate structure can be formed.

The ease of removal assessed the effort and tools required to remove the material from the print bed after the printing. If a material is easy to remove the risk of initiation can be minimised by reducing the energy imparted on the material through impact (force required) and electrostatic stimuli (possible use of conducting tools). This could allow more sensitive materials to be printed. The ease of removal score has been weighted more heavily than the print score.

3.3.4 Analysis

A table was completed with the print bed material, the print quality score and the ease of removal score. These were added together for a total score where a higher number depicts a better overall print bed material.

The table was consulted for the highest combined scores to determine the most suitable print bed material.

3.4 Solids Loading Investigation

3.4.1 Aim

The aim of this experiment was to explore the effect that the solids loading of a formulation has on its extrudability. Additionally, it aimed to find the optimum solids loading fraction for EPIM for extrudability.

3.4.2 Materials and Equipment

This experiment required the following:

- LabRAM II (Figure 5)
- Hex-Vessel (Figure 6)
- Syringes (Figure 8)
- MikeBot (Figure 1)
- RAM Formulation Calculator (Table 1)

3.4.3 Methods

Six syringes were prepared and, using the RAM FC, six formulations were planned where the Dymax quantity increased from 22.0 % to 24.5 % in 0.5 % increments.

The Hex-Vessel was assembled as per 3.2.3.4 and the EPIM RAM recipe in Table 2 was run. Although not the aim of the investigation, the vessel was still monitored for vibrations and the power graph for anomalies to ensure it was run safely.

Once the mix was complete, one syringe was removed at a time. The material from the syringe was removed where a brief visual inspection was conducted and afterwards loaded into the MikeBot container. An extrudability test was completed and this was repeated for each syringe.

3.4.4 Analysis

MikeBot extrudability analysis was conducted and the results of the six syringes were plotted on a graph of ram displacement against pressure. This procedure can be seen in 3.1.4.

The effect of the solids loading of the formulation was analysed by the trend of the graph. The extrudability curves were compared to previous results and the optimum extrusion profile.

3.5 Dye Colour Chart

3.5.1 Aim

The aim of this experiment was to create a visual aid for assessing the quality and completeness of a mix conducted on the RAM. A range of dyes were also added to the formulation to see how they mixed, the clarity they provided and if they effected the extrudability of the material.

3.5.2 Materials and Equipment

A range of dyes were selected to be added to the standard formulation:

- Red food colouring (liquid)
- Iron Oxide (powder)
- Graphite (powder)
- Red tracer dye (small pellet)
- Blue tracer dye (small pellet)

A LabRAM II was used to mix the formulations and utilised both the single syringe vessel, Figure 16, and the Hex-Vessel, setup described in section 3.2, to mix up to six syringes at once.

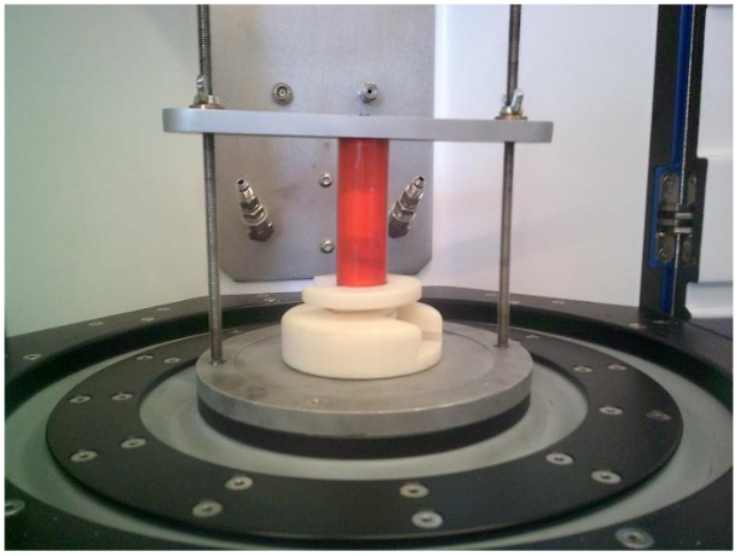


Figure 16 Single Syringe Vessel

The dye formulations were again based on EPIM, however where a liquid dye was added, mass was taken away from the Dymax (Table 5) and where a powder dye was added, mass was taken away from the Melamine (Table 6).

Order	Material	Percentage	Required (g)
1	Melamine 106-63μm	37	7.4
2	Dymax	25	5
3	Red Dye	1.00	0.2
4	Melamine 180-106μm	37	7.4

Table 5 Liquid Dye Formulation Example

Order	Material	Percentage	Required (g)
1	Melamine 106-63μm	37.25	7.45
2	Iron Oxide	0.5	0.1
3	Dymax	25.00	5
4	Melamine 180-106μm	37.25	7.45

Table 6 Solid Dye Formulation Example

The MikeBot, setup described in Section 3.1.2, was used to assess the extrudability of the formulations with the different dyes.

3.5.3 Methods

Initially the single syringe vessel was used with the red food dye to gather initial thoughts on mix quality. The following steps describe the initial investigation:

1. The RAM FC was completed for the red dye formulation.
2. The materials were weighed out into a syringe.
3. The syringe was mounted to the LabRAM II and the vessel's lid was tightened.
4. The RAM recipe was chosen and run.
5. The RAM was paused at intervals into the run and the syringe was removed from the vessel.
6. The material was visually inspected.
7. Steps 3-6 were repeated until the recipe had finished in its entirety.

The final material was analysed and a longer recipe was implemented until the 20 minute mark was reached. After the analysis of this material and the commissioning of the Hex-Vessel, multiple syringes were mixed at once and eventually the liquid dye was replaced with a range of solid dyes.

The following steps were taken to complete the dye comparison:

1. A dye was chosen and the RAM FC was completed.
2. A syringe was prepared and labelled.
3. The materials were weighed out into the syringe and the masses recorded in the RAM FC.
4. The syringe was added to the Hex-Vessel on the LabRAM II.
5. Steps 1-4 were repeated until six syringes had been made up and then the RAM was started using the recipe in Table 2.
6. The RAM was stopped at 6, 13 and 20 minutes into the mix and at each interval, two of the red food dye syringes were removed.
7. MikeBot analysis was conducted on each of the six syringes to assess the extrudability of the red food dye.
8. Steps 1-5 were repeated after the red food colouring was analysed and new dyes were chosen.
9. The hex-vessel was loaded with two syringes with the iron oxide dye, two with the graphite dye and one of each of the red and blue drain dyes.
10. The RAM was run with the EPIM recipe.
11. The samples were analysed visually and their extrusion characteristics were assessed using the MikeBot.

3.5.4 Analysis

Visual analysis of the syringes were conducted at the set intervals for the red food dye and at the end of the experiment for the remaining dyes. Pictures were taken of each syringe and any unexpected results.

Each syringe with the solid dye formulation was loaded into the MikeBot to analyse its extrusion characteristics.

4 Experimental Results

4.1 Hex-Vessel Commissioning

The experimental methods used for the hex-vessel commissioning are discussed in 3.2 Hex-Vessel Commissioning and the results of these experiments are discussed below.

4.1.1 Empty Vessel Check

During the initial acceleration sweep with the empty Hex-Vessel, excessive vibrations accompanied by a loud noise were evident at as low as 20 G. The testing was therefore discontinued and the cause of the vibrations was investigated. In the course of this investigation, it was found that the studding used to fasten the Hex-Vessel to the resonator used metric screw threads instead of the imperial threads required by the LabRAM II.

The issue was discussed with Dstl Engineering and it was decided that the mounting method would be changed to use a different set of locating holes on the resonator. These adjustments were made and the test was repeated.

In the repeated test the full acceleration sweep up to 100 G was completed without excessive vibration of the vessel. The LabRAM II equipment provides a live plot of machine power against time for each mix carried out and this was monitored during the test.

4.1.2 Two Opposing Syringes

The formulations mixed in opposing syringes were removed from the vessel at the end of the mix. The materials were photographed and removed from the syringe for further investigation. A photograph of two syringes following mixing is shown below in Figure 17.

The materials were observed to be of a reliable paste-like consistency without evident solid agglomerates. Qualitatively, they appeared to be well mixed and similar to each other.



Figure 17 Post-Mix Hex-Vessel with Opposing Syringes

4.1.3 Single Asymmetric Syringe

The vessel and the LabRAM II's dashboard were constantly monitored during the mix of the first asymmetric load. No off-axis oscillation or horizontal movement was visually detectable and no warning alarms from the internal displacement sensors were triggered.

The materials were observed to be a regular paste-like consistency and there were no visual indicators that the material had mixed poorly or differently to each other.

4.1.4 Full Capacity

The 6 syringes from the full capacity test of the Hex-Vessel were removed following mixing. The mixed materials were photographed and removed from the syringe for further investigation.

Qualitatively, the materials were all similar to each other and of a paste-like consistency. Each material was a similar light red colour indicating the dye had been evenly distributed. A notable exception to this was close to the bung at the bottom of the syringe where the material had remained white.

The materials were then analysed using the MikeBot equipment according to the method stated in 3.1.3 Methods. This analysis produced plots of measured pressure against ram displacement which are shown in Figure 18.

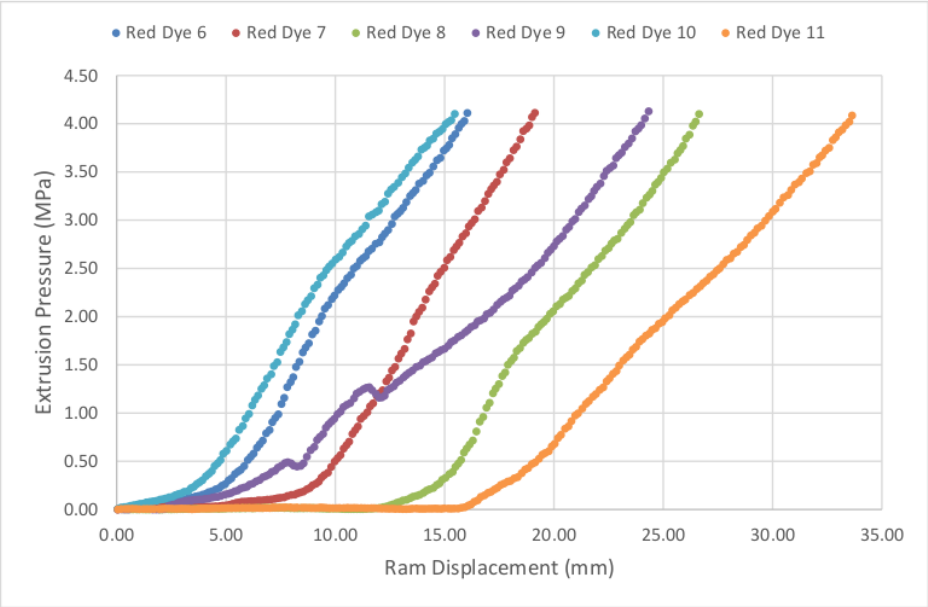


Figure 18 Full Capacity Hex-Vessel MikeBot Analysis

An ideal extrusion curve would initially exhibit a pressure of around 0, followed by a rapid rise in pressure as the ram makes contact with the material under test. The pressure would then level out to a steady state value until the ram piston meets the static material close to the nozzle entrance.

The curves shown in Figure 18 all exhibit a similar trend which includes an initial rapid rise in pressure followed by an inflection point. The pressure then continues to rise until it reaches the maximum possible for this equipment which is about

4.1 MPa. This result generally indicates that the material does not reach steady state extrusion under the test conditions.

It is important to note that the plots in Figure 18 are displaced along the x-axis by different amounts although they all exhibit similar shapes and gradients. This is not significant as the initial ram position in each experiment is arbitrary as it's not fixed.

4.1.5 Three Asymmetric Syringes

The three syringes that were arranged asymmetrically were removed from the Hex-Vessel at the end of the mix. Photographs were taken, as seen in Figure 19 and the material was removed from the syringes.

After qualitative observation, each appeared to be well mixed with a paste-like consistency and they were similar to each other.



Figure 19 Post-Mix Hex-Vessel with Three Asymmetric Syringes

Figure 20 displays some of the residual material left on the syringe caps on the vessel lid. The three right side caps are clean as they held empty syringes.

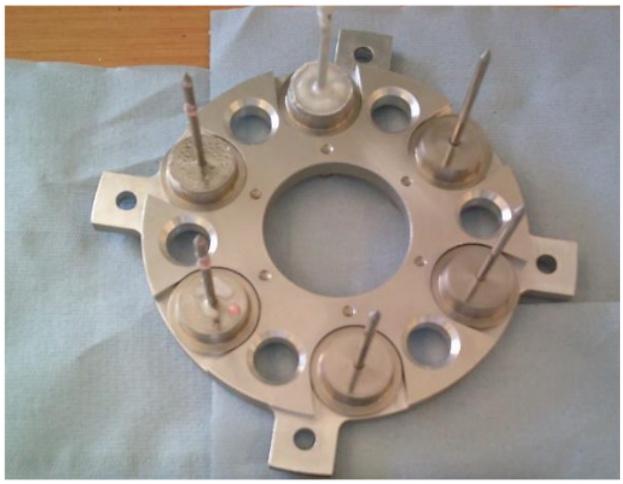


Figure 20 Hex-Vessel Lid Post-Mix

The materials were analysed on the MikeBot equipment, the plot of extrusion pressure against ram displacement is shown in Figure 21.

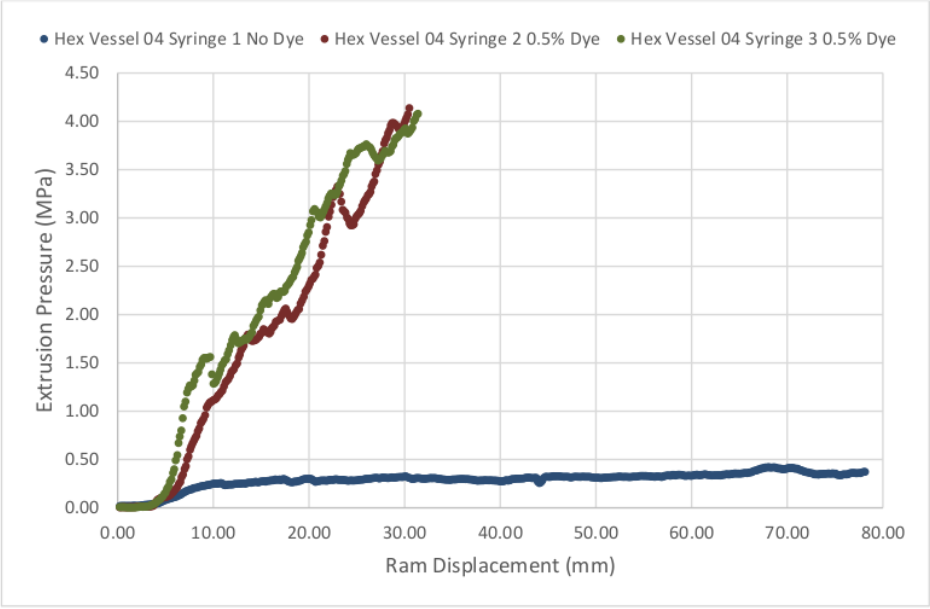


Figure 21 Three Asymmetric Syringes Hex-Vessel MikeBot Analysis

Syringe 2 and 3 exhibit a similar trend to those seen in Figure 18 where the maximum extrusion pressure is reached rapidly. It is important to note that the x-

axis on this graph is extended and a higher ram displacement is actually achieved at ~30mm compared to 10-25mm in the previous test.

Syringe 1 exhibits a near ideal extrusion curve where the pressure remains mostly consistent and minimal throughout the extension of the ram. This material reached steady state extrusion under the test conditions.

4.2 Print Bed Testing

The experimental methods used in the print bed testing are outlined in 3.3 Print Bed Testing and the results of the experiments are discussed below.

Table 7 was created that combined the recorded print score and removal score. Each material was assessed based on its combined score out of 8 where a higher score is more suitable.

Material	Print score	Removal score	Total score
Glass	3	1	4
PEI powder coated spring steel	3	5	8
PEI sticker on spring steel	3	4	7
PEI sticker on glass	3	3	6
Card	2	5	7
Masking tape	3	2	5
Antistatic bag	3	5	8

Table 7 Print Bed Testing Results

PEI powder coated spring steel and antistatic bags both received 8/8 scores where the material stuck to the print bed during the print whilst also being easy to remove with a plastic spatula.

Figure 22 shows the 3 layer print stuck to the glass print bed. Extreme force was required to remove the material which created debris as it was chipped away.



Figure 22 Glass Removal and Debris

Figure 23 displays a small amount of material left behind after the prints were removed from the PEI powder coated spring steel.



Figure 23 PEI Powder Coated Spring Steel

The underside of the prints which were left to cure for 10 minutes are shown in Figure 24 after removal from the print bed. Table 8 describes how difficult removing the material was and if it was cured.

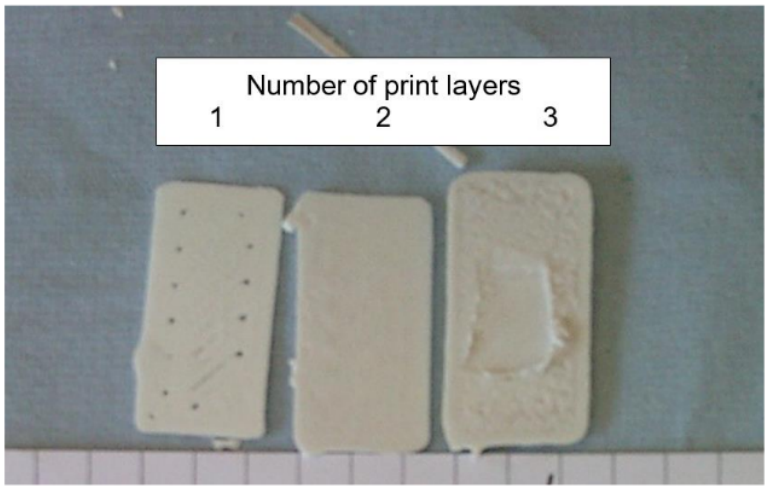


Figure 24 PEI Powder Coated Spring Steel with Curing

Layer	Removal	Curing
1	Difficult but no destruction	Cured
2	Easy	Cured
3	Easy	Not cured

Table 8 PEI Powder Coated Spring Steel Removal

A small amount of material was left behind on the PEI sticker on spring steel in Figure 25. Only the single layer experienced some damage upon removal. The single layer (Table 9) proved slightly more difficult to remove than the powder coated PEI.



Figure 25 PEI Sticker on Spring Steel

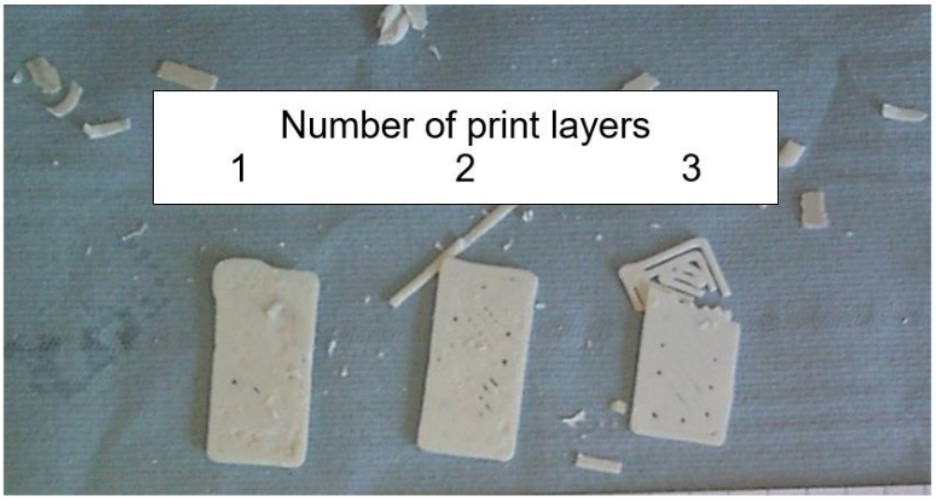


Figure 26 PEI Sticker on Spring Steel with Curing

Layer	Removal	Curing
1	Minor difficulty	Cured
2	Easy	Cured
3	Easy	Cured

Table 9 PEI Sticker on Spring Steel Removal

Figure 27 and Figure 28 show the removal of the prints from the card print bed. The removal was easy but a residue was left by each print.

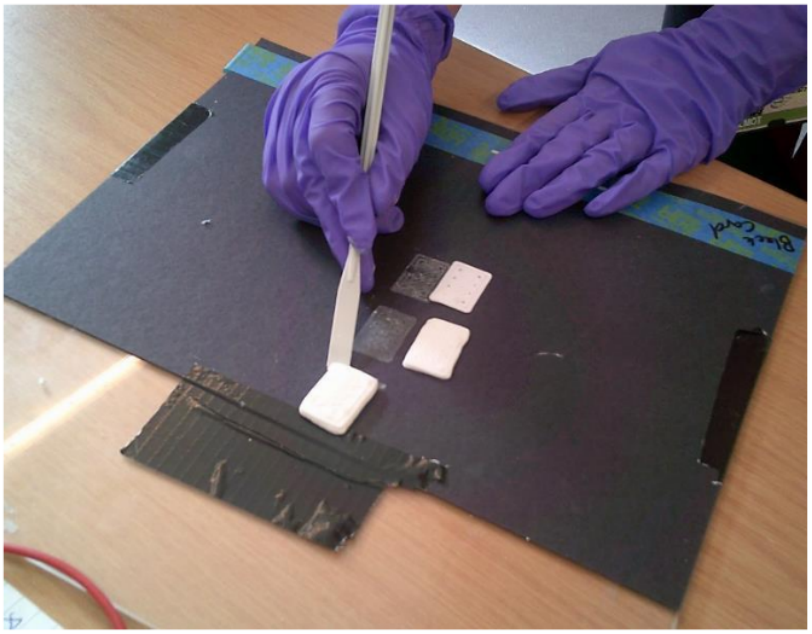


Figure 27 Card Removal

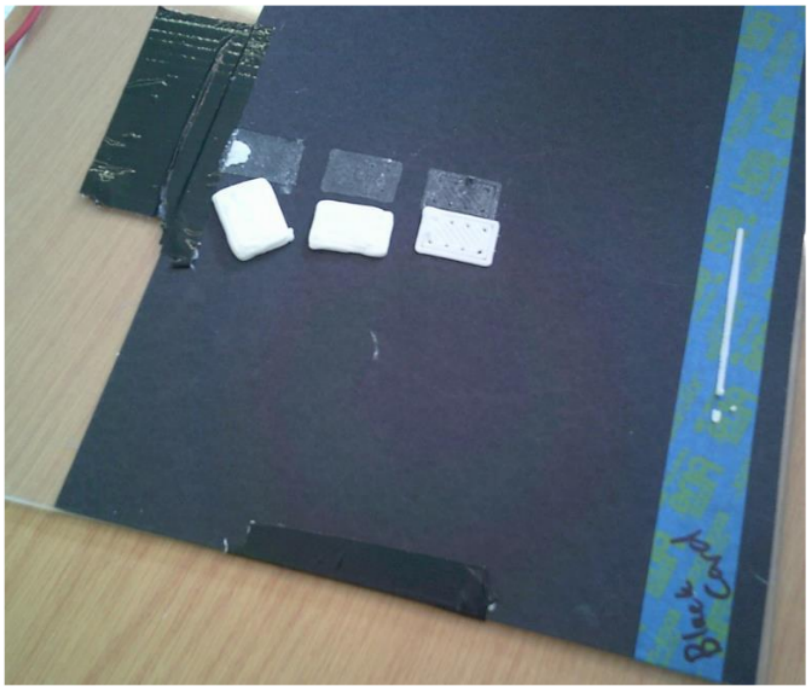


Figure 28 Card Residue

Figure 29 shows the prints attached to the masking tape print bed and Figure 30 shows the prints after they had been removed from the antistatic bag.



Figure 29 Masking Tape on Glass



Figure 30 Antistatic Bag on Glass

4.3 Solids Loading Investigation

The output from the MikeBot analysis of the solids loading investigation is located below in Figure 31. Here each syringe contains a range of binder between 22.0 % and 24.5 % which results in a range of gradients as the ram piston travels. It is important to note that the fine particle size of the melamine was changed from <63 μm to 106 - 68 μm due to supply issues. This could cause this set of material to have a decreased particle packing and a worse extrusion profile.

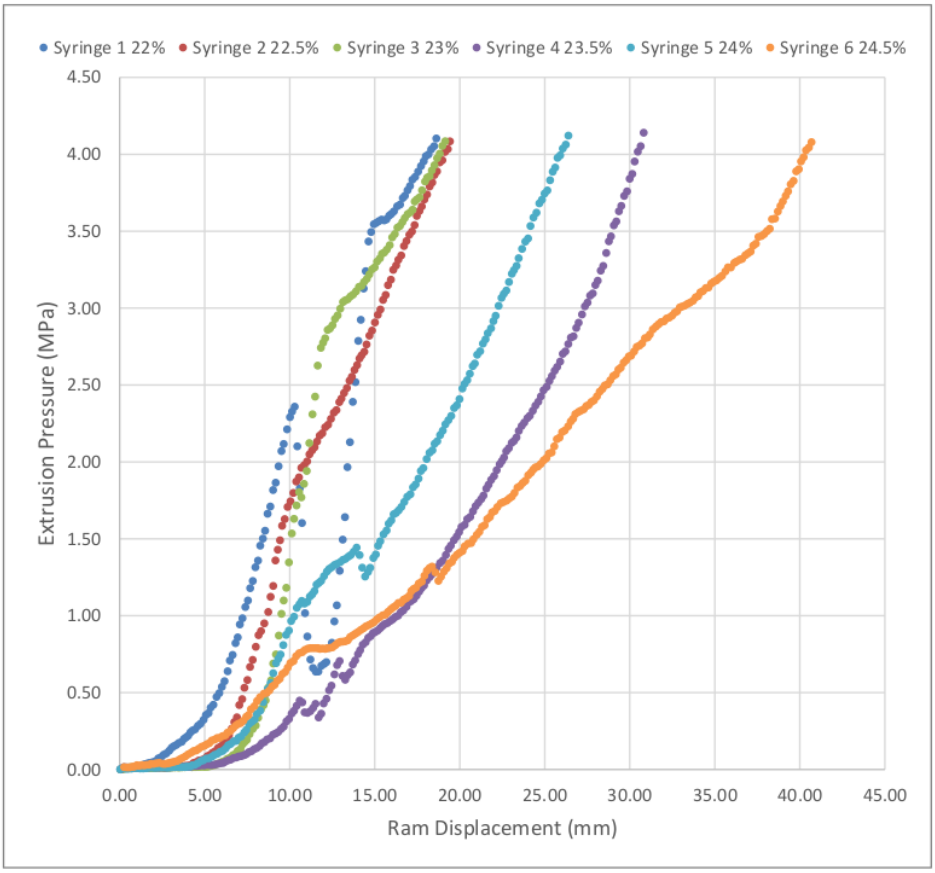


Figure 31 Solids Loading MikeBot Analysis

Syringe 1 exhibits a large drop in extrusion pressure at 10 mm displacement which is likely due to an air bubble in the material. However, it quickly returns to its previous trajectory.

Each of the syringes reach the maximum extrusion pressure at 4.1 MPa causing the test to be stopped. All series exhibit an inflection point where the gradient begins to shallow before becoming steep again.

4.4 Dye Colour Chart

Figure 32 shows the mix “Red Dye 2” at five different intervals in the mix: 30, 90, 120, 240 and 330 s. The formulation included 0.2 % (0.04 g) red food colouring.

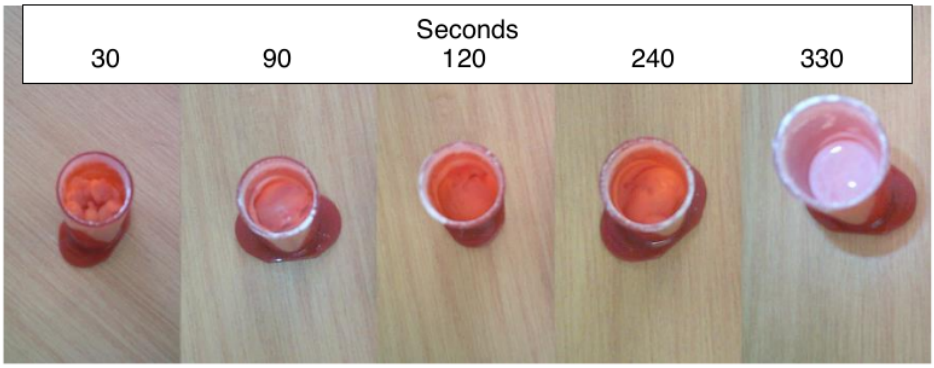


Figure 32 Red Dye No.2

At 30 s the material is clearly clumped into small balls and the dye is very spotty throughout. At 90 s the material started to consolidate into a single mass and the dye started to streak through the material. At 120 and 240 s the material developed a surface sheen and dye continued to streak. At 330 s a flat surface developed with a shiny surface sheen and spots of dye were still visible.

The post mix analysis and the MikeBot extrusion is displayed in Figure 33. The post mix material had a light pink colouring the whole way through however dark deposits of localised dye could be seen and overall it was very streaky. The extruded material appeared more uniform in colour although it continued to be streaky.

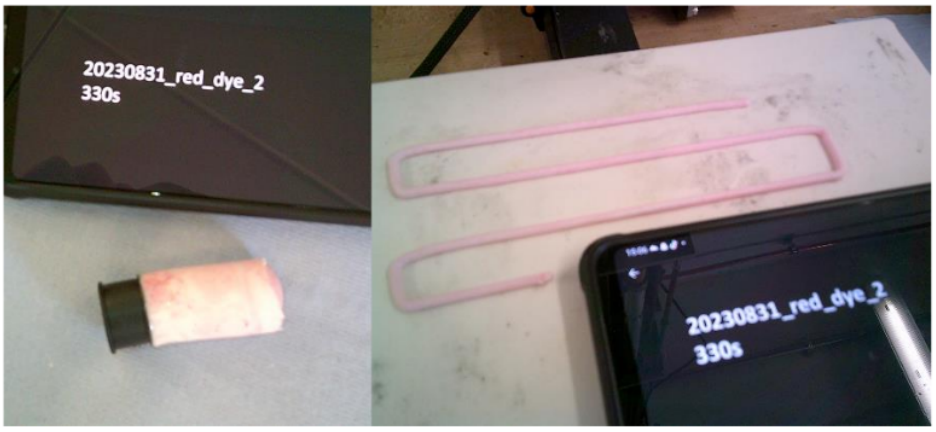


Figure 33 Red Dye No.2 Visual Analysis

The MikeBot analysis results of the dye investigation outlined in the second method of 3.5.3 Methods is graphed in Figure 34. Qualitatively, the materials all behaved similar to each other, especially the respective repeats.

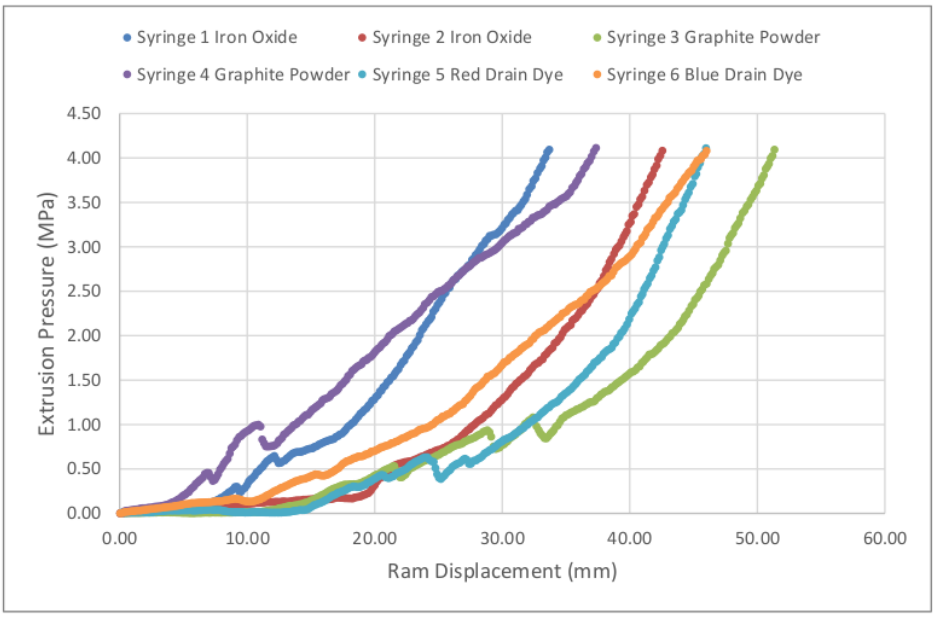


Figure 34 Dye Investigation MikeBot Analysis

All of the dyes exhibit a similar trend where the pressure builds as the ram travels until the pressure limit of the system is reached at 4.1 MPa. Some of the dyes

appear to change the rate at which the pressure increases for example, the iron oxide formulations display steeper gradients than the graphite formulations.

Up to twice as much extrusion (33 – 51 mm) was seen compared to the food colouring mixes shown previously in Figure 18 (15 – 33 mm). It is important to note again that the recorded displacement could be altered by how much material was added to the MikeBot's cylinder container.

Jagged lines near the start of the test could be caused by small air bubbles in the material which would cause the pressure to dip however, the gradient quickly stabilises after extruding the air.

5 Discussion

After resolving the vibration problem from the first acceleration sweep a successful acceleration sweep demonstrated the Hex-Vessel could be considered safe to use up to 100 G. Although no vibrations could be seen or heard during this test, it is still advised that the RAM is monitored at all time when mixes are carried out.

Subsequently, the two opposing syringe test proved that the vessel could hold and mix material where it remained intact and connected to the resonator. The material in the two syringes appeared similar to each other signifying the vessel provides the same level of mixing to two syringes as the single syringe vessel does to one. Only 8% of the RAM's power was used during this mix, suggesting it could handle a fully loaded mix with ease.

After constantly monitoring the LabRAM II's dashboard, confidence was built in the vessel's ability to be asymmetrically loaded as no lateral movement was visible and no alarms were triggered during the test. The material appearing similar to other mixes in the single syringe vessel proved that asymmetric loading did not change the mix quality the RAM was able to provide.

Further confidence was provided on the Hex-Vessel's ability to deal with asymmetric loads after it was loaded with three syringes on one side. Again no visible lateral movement was detected nor any alarms raised. Similarly, the three syringes produced material consistent of that of other tests and vessels. This configuration provides the highest asymmetric loading possible for the Hex-Vessel and as such, the data indicates that the loading order and configuration of the vessel does not affect the mix quality that can be provided.

An extra 0.5 % more Dymax was added to Syringe 1 and the red food dye was missed accidentally during material addition. This resulted in a near ideal extrusion profile where the ram piston was able to travel the full distance without exceeding the pressure limits of the equipment, something no other mix was able to do. It was theorised that this could be due to the increased binder quantity and the lack of dye. This led to the 'Solids Loading Investigation' being developed to

identify the cause of this result. There is no reason to suggest that the lack of dye in the mix was conducive to this outcome as many other tests had been completed with similar binder quantities (23 %) that did not yield near ideal extrusion profiles. It is possible that the length of time selected for the mix (20 minutes) is not adequate for every formulation or that the material was loaded into the MikeBot container differently as this process is completed by hand it is not identical every time. If it was packed less densely into the container, there could be less friction as the material collates onto the ram piston as it travels.

The full capacity experiment explored the LabRAM II's capability to conduct a mix where the material mass would be the highest (180 g). Low power usage (<15 %) was displayed on the RAM's dashboard during the mix which provided confidence that a full load could be managed.

Final confidence was provided to the vessel after each syringe output consistent paste-like material upon visual inspection. This was further supported by the MikeBot analysis as the graphs were similar to previous experiments.

All syringes contained the same formulation and the MikeBot analysis shows that they were relatively similar to each other as they have very similar gradients which is the primary measure of a material's extrudability. The pressure limit was reached in all tests before the ram piston could extrude the full amount of material.

The print bed testing experiments output two suitable candidates for future 3D printing endeavours; PEI powder coated spring steel and antistatic bags. Both scored 3/3 on the print score meaning the first layer deposited by the 3D printer stuck to the surface, allowing the second layer to be placed accurately on top. Both also scored 5/5 on the removal score where both could be easily removed with a plastic tool. The spring steel's ability to be bent meant the material would pop off of the surface for easy removal and the antistatic bags could be removed from the print bed entirely. Antistatic bags are often used to store energetic materials as they reduce the static energy available, as such they are a good candidate when 3D printing energetic materials. This is particularly true if novel formulations are to be printed to provide the material to conduct Small Scale

Hazard Testing as the hazard properties may be unknown. Testing card as a print bed material provided insight into the importance of the porosity of a material as binder leaching occurred in the first layer that was printed. This layer would cure differently as it would have a different formulation.

Due to a supply shortage, the fine particle size, <63 μm, of melamine was not available for this test. The lack of fine material makes the extrusion profile worse overall and this MikeBot analysis harder to compare to previous runs as the particle packing would be decreased. This would increase the internal friction as the binder is distributed less efficiently and the large particles cannot slide over each other as easily.

Each material causes the equipment to reach the maximum operational pressure before the ram piston is able to travel the full distance. None of the formulations provided an ideal extrusion profile. However, each series does have an inflection point where the gradient decreases momentarily before rising sharply again. This is more prominent in the formulations with a higher Dymax quantity and the highest quantity, Series 6, had the highest ram displacement. The graph shows that increasing the Dymax quantity within a formulation further than 24.5% improves the extrudability of the material with each gradient getting progressively shallower. As the solids loading decreases, the inflection point occurs at a lower pressure. This alludes to an ideal extrusion profile, where the inflection occurs early and then a stable pressure is maintained, being possible at lower solids loading.

Syringe 1 experiences a notable drop in pressure followed by a sharp increase moments later. This is hypothesised to be caused by an air bubble in the material providing little resistance to the ram piston. When the material starts extruding again, the pressure returns to the original trajectory, meaning it is unlikely to be a true inflection point.

The mixes conducted under the ‘Dye Colour Chart’ experiment originally had mix times of 330 seconds. In this timeframe a clear progression is visible between clumped unmixed materials to a material with a paste-like consistency. However, the dye was not distributed throughout the material, leaving dark spots and white

streaks. To address this, mixes were conducted with longer and longer mix times, eventually reaching 20 minutes or 1200 seconds. These provided a material with a much more consistent colour and less deposits of dye. Future mixes can be compared to the photos taken during this experiment, which were combined to create the dye colour chart in Figure 32, to assess the mix quality of their material. If the material looks similar to an image on the left of the chart, it would be likely that further mixing is required until a consistent material with a surface sheen has been developed.

The dye colour chart was developed alongside the other experiments and as such the solids loading also changed as the other experiments recommended lower solids loading. Also, the addition order of the formulation was changed to allow for better mixing by adding the dye after the fine material and before the binder. This visually improved the quality of the mix.

Even with 20 minutes of mixing the red food dye was not incorporating fully into the mix. Inert propellants use iron oxide to add colour to the mix and as such, a variety of powder dyes were tested. Upon visual inspection, it was immediately clear that these were able to mix more consistently than the food dye.

The formulations with the new dyes were analysed on the MikeBot and similarly to the previous materials they exceeded the pressure limit. However, the new dyes were capable of up to twice as much extrusion. Overall they had shallower gradients than the food dye, this was particularly noticeable in the material with the graphite dye. This could be attributed to graphite's use as a natural lubricant which could have reduced the friction during extrusion. Additionally, the mass of the solid dye included was taken away from the melamine instead of the Dymax which reduced the viscosity of these formulations.

The powder dye formulations used a solids loading of 75 % and did not produce an ideal extrusion profile. This profile could be achieved by reducing the solids loading which is supported by the outcome of the Solids Loading Investigation, not necessarily by choosing a different dye.

6 Conclusion

The mechanical aims of this project were to assess the suitability of a novel asymmetric vessel's use on the LabRAM II and to investigate if asymmetric loading effected mix quality. The formulation aims were to develop a comparison chart for mix quality, to determine suitable dyes for inert formulations and to analyse a range of solids loadings to identify the effect on the extrudability of the material.

A series of scale-up tests were conducted on the Hex-Vessel. These allowed confidence to be built in both its compatibility with the LabRAM II and its ability to provide the same mix quality as the single syringe vessel, whilst delivering a six fold increase in capacity. The asymmetric loading investigations determined that there was no effect on the mix quality or its connectedness to the RAM's resonator plate based on the configuration of the vessel.

A "dye colour chart" was developed as a visual aid to assess the mix quality of material at different time intervals through a RAM mix. The dye investigation has recommended that solid dyes are used in future formulations as they were incorporated into the mix better and were more useful as a visual aid. The solids loading investigation developed a formulation with a better extrusion profile, however continued development is required.

Overall, the aims of the project have been achieved and a novel vessel has been commissioned that will allow the further development of the RAM MIC 3D printing technology.

6.1 Recommendations for Further Research

Some recommendations for additional research are categorised and listed below:

Novel vessel development

- Investigate the effect of the geometry of the vessel on its mix quality such as the height-to-width ratio.
- Develop MIC RAM mixing by allowing different size syringes or other containers to fit on to a vessel for a wider range of applications.
- Integrate additional features into the vessels such as a water jacket for heating and cooling or the ability to pull a vacuum during the mix to improve mix quality.

Formulations

- A trend where the extrudability increased as the Dymax quantity increased was developed in the solids loading investigation but the near ideal extrusion profile was never reached. Continue to decrease the solids loading to explore the continuation of the trend.
- Additional dyes could be tested, both solid and liquid. The amount of dye in the formulation should also be considered. Investigate effect on viscosity.
- The mix quality could be analysed further using x-ray Computed Tomography (CT) by obtaining detailed images of the structure of the mix. This would provide both qualitative data from visualisation and quantitative data from the analysis of void volumes or areas of streaking.

6.2 Reflection

During this project I learnt the importance of planning and note taking whilst conducting the extensive list of mixes and the respective analysis. I believe I set out reasonable goals, conducted experiments and collected data to provide confidence in my findings. I gained important knowledge on PBX and propellant formulations whilst manufacturing the inert simulations and developed my understanding of the importance of flow mechanics in AM applications.

To improve my project, a better camera could have been used as many of the photos taken were blurry or lacked detail. This was not conducive to gathering usable qualitative images for analysis. Additionally, sorting and labelling all of the pictures after the experiments was time consuming and should've been done as the photos were taken.

7 References

[1] A. Claydon, “Resonant acoustic mixing of polymer bonded explosives,” Cranfield University, 2021.

[2] Rapid Direct, “Comparing Additive Manufacturing Vs Subtractive Manufacturing: What Are the Differences,” 10 February 2022. [Online]. Available: https://www.rapiddirect.com/blog/additive-vs-subtractive-manufacturing/. [Accessed 11 March 2024].

[3] UltiMaker, “Thingiverse - Digital Designs for Physical Objects,” [Online]. Available: www.thingiverse.com. [Accessed 23 February 2024].

[4] N. Shahrubudin, “An Overview on 3D Printing Technology: Technological, Materials, and Applications,” *Procedia Manufacturing,* vol. XXXV, pp. 1286-1296, 2019.

[5] M. O'Donnell, “Flow Visualization in the Extrusion of Highly Filled Polymer Composites for Additive Manufacturing,” *Thermomechanics & Infrared Imaging, Inverse Problem Methodologies and Mechanics of Additive & Advanced Manufactured Materials,* vol. VI, pp. 61-67, 2023.

[6] M. O'Donnell, “Thermo-mechanical characterisation of energetic materials for additive manufacturing,” Queen's College, University of Cambridge, Cambridge, 2024.

[7] H. E. Hoskyns, “Advances in additive manufacture of energetic materials,” Dstl, 2022.

[8] A. Wolff, “Additive Manufacturing Processes Applied to Energetic Materials,” in *16th Rapid Design Prototyping & Manufacturing Conference*, London, 2019.

[9] N. Muravyev and K. Monogarov, “Progress in Additive Manufacturing of Energetic Materials: Creating the Reactive Microstructures with High Potential of Applications,” *Propellants, Explosives, Pyrotechnics,* vol. XLIV, pp. 1-30, 2019.

[10] A. Wolff, “Additive Manufacturing Processes Applied to Energetic Materials,” in *16th Rapid Design Prototyping & Manufacturing Conference*, London, 2019.

[11] G. Bocksteiner, “The Effect of Ageing on PBXW-115, PBXN-103 and PBXN-105,” DSTO Aeronautical and Maritime Research Laboratory, 1995.

[12] M. Talawar, “Environmentally compatible next generation green energetic materials (GEMs),” *Journal of Hazardous Material,* vol. CLXI, no. 2-3, pp. 589-607, 2009.

[13] C. J. Wright, “Is Resonant Acoustic Mixing® (RAM) a Game Changer for Manufacturing Solid Composite Rocket Propellants?,” *Propellants, Explosives, Pyrotechnics,* vol. XLVII, no. 1, 2021.

[14] Resodyn, “What is Resonant Acoustic Mixing,” 30 December 2020. [Online]. Available: https://resodynmixers.com/2020/12/30/what-is-resonantacoustic-mixing/. [Accessed 14 February 2024].

[15] Resodyn, “How RAM Mixes,” [Online]. Available: https://resodynmixers.com/how-ram-mixes/. [Accessed 14 February 2024].

[16] M. Andrews, “Resonant Acoustic® Mixing: Processing and Safety,” *Propellants, Explosives, Pyrotechnics,* vol. XLV, no. 1, pp. 77-86, 2019.

[17] E. Rose, “Dstl/DOC133225 - Use of the Resodyn LabRAM II Resonant Acoustic Mixer in the Energetics Enclosure,” 1 September 2021. [Online]. Available: "\\Rnet\org\DIV_CTS\Standard\215_WWShef\5.ETG\Risk Assessments \20210623-LabRAMII _in_EE_SOP-DOC133225-v1.0-O.pdf".

[18] J. Howe, “RAM Formulation Calculator,” 22 June 2023. [Online]. Available: "\\RNET\Tech\Project_Technical\Standard\PJ100818\TOP_TASKS\FEC\WP002 - Novel Manufacturing\05_Technical_WIP\ RAM\ RAM_Formulation_Calculator.xlsx".

[19] T. J. Fleck, A. K. Murray and I. E. Gunduz, “Additive Manufacturing of Multifunctional Reactive Materials,” *Additive Manufacturing ,* vol. 17, pp. 176-182, 2017.

List of Abbreviations

AM	Additive Manufacturing
AP	Ammonium perchlorate
CT	Computed Tomography
Dstl	Defence science technology laboratory
FEC	Future Energetics Core
HTPB	Hydroxyl-Terminated Polybutadiene
IPDI	Isophorone Diisocyanate
PEI	Polyetherimide
PBX	Polymer Bonded eXplosives
RAM FC	RAM Formulation Calculator
RAM	Resonant Acoustic Mixing/Mixer
SOP	Standard Operating Procedure
TFP	The Falcon Project

Appendix A MikeBot Nozzle and Cylinder Selection

A larger cylinder diameter spreads the load over a larger area which lowers the pressure whereas a smaller cylinder requires less material to fill to the required level. Novel formulations could require less material to be used in initial mixes, so having the option is beneficial. A larger nozzle diameter again reduces the pressure but also decreases the print resolution. A longer nozzle provides a longer flow development which can lead to a more stable print, however it will increase the pressure required.

Appendix B RAM Formulations

All of the formulations mixed in this project are detailed below under their relevant sections.

Hex-Vessel Commissioning:

Two Opposing, Syringe 1:

Order	Material	Percentage	Required (g)	Actual (g)
1	Melamine <63μm	38.75	7.75	7.7525
2	Dymax	22.50	4.50	4.5357
3	Melamine 180-106μm	38.75	7.75	7.7512

Two Opposing, Syringe 2:

Order	Material	Percentage	Required (g)	Actual (g)
1	Melamine <63μm	38.75	7.75	7.7519
2	Dymax	22.50	4.50	4.522
3	Melamine 180-106μm	38.75	7.75	7.7526

Single Asymmetric, Syringe 1:

Order	Material	Percentage	Required (g)	Actual (g)
1	Melamine <63μm	38.75	7.75	7.7493
2	Dymax	22.50	4.50	4.4972
3	Melamine 180-106μm	38.75	7.75	7.7485

Full Capacity, Syringe 1:

Order	Material	Percentage	Required (g)	Actual (g)
1	Melamine <63μm	38.75	7.75	7.7594
2	Dymax	22.50	4.50	4.5032
3	Melamine 180-106μm	38.75	7.75	7.7551

Full Capacity, Syringe 2:

Order	Material	Percentage	Required (g)	Actual (g)
1	Melamine <63μm	38.75	7.75	7.7523
2	Dymax	22.50	4.50	4.5079
3	Melamine 180-106μm	38.75	7.75	7.754

Full Capacity, Syringe 3:

Order	Material	Percentage	Required (g)	Actual (g)
1	Melamine <63μm	38.75	7.75	7.7491
2	Dymax	22.50	4.50	4.4956
3	Melamine 180-106μm	38.75	7.75	7.7519

Full Capacity, Syringe 4:

Order	Material	Percentage	Required (g)	Actual (g)
1	Melamine <63μm	38.75	7.75	7.7499
2	Dymax	22.50	4.50	4.4968
3	Melamine 180-106μm	38.75	7.75	7.7551

Full Capacity, Syringe 5:

Order	Material	Percentage	Required (g)	Actual (g)
1	Melamine <63μm	38.75	7.75	7.7573
2	Dymax	22.50	4.50	4.4948
3	Melamine 180-106μm	38.75	7.75	7.7562

Full Capacity, Syringe 6:

Order	Material	Percentage	Required (g)	Actual (g)
1	Melamine <63μm	38.75	7.75	7.7516
2	Dymax	22.50	4.50	4.5004
3	Melamine 180-106μm	38.75	7.75	7.7596

Three Asymmetric, Syringe 1:

Order	Material	Percentage	Required (g)	Actual (g)
1	Melamine <63μm	38.50	7.70	7.7015
2	Dymax	23.00	4.60	4.5862
3	Melamine 180-106μm	38.50	7.70	7.7009

Three Asymmetric, Syringe 2:

Order	Material	Percentage	Required (g)	Actual (g)
1	Melamine <63μm	38.25	7.65	7.648
2	Dymax	22.50	4.50	4.6045
3	Red Dye	0.50	0.10	0.0945
4	Melamine 180-106μm	38.25	7.65	7.6496

Three Asymmetric, Syringe 3:

Order	Material	Percentage	Required (g)	Actual (g)
1	Melamine <63μm	38.50	7.70	7.708
2	Dymax	22.50	4.50	4.4854
3	Red Dye	0.50	0.10	0.0816
4	Melamine 180-106μm	38.50	7.70	7.703

Print Bed Testing:

Syringe 1:

Order	Material	Percentage	Required (g)	Actual (g)
1	Melamine <63μm	38.75	7.75	7.7512
2	Dymax	22.50	4.5	4.537
3	Melamine 180-106μm	38.75	7.75	7.7522

Syringe 2:

Order	Material	Percentage	Required (g)	Actual (g)
1	Melamine 106-68μm	38.75	7.75	7.7503
2	Dymax	22.50	4.5	4.5115
3	Melamine 180-106μm	38.75	7.75	7.7566

Syringe 3:

Order	Material	Percentage	Required (g)	Actual (g)
1	Melamine 106-68μm	38.75	9.6875	9.6851
2	Dymax	22.50	5.625	5.6396
3	Melamine 180-106μm	38.75	9.6875	9.6957

Syringe 4:

Order	Material	Percentage	Required (g)	Actual (g)
1	Melamine 106-68μm	38.75	9.6875	9.6875
2	Dymax	22.50	5.625	5.6594
3	Melamine 180-106μm	38.75	9.6875	9.6887

Syringe 5:

Order	Material	Percentage	Required (g)	Actual (g)
1	Melamine 106-68μm	38.75	7.75	7.7531
2	Dymax	22.50	4.5	4.5263
3	Melamine 180-106μm	38.75	7.75	7.7503

Syringe 6:

Order	Material	Percentage	Required (g)	Actual (g)
1	Melamine 106-68μm	38.75	7.75	7.7472
2	Dymax	22.50	4.5	4.4916
3	Melamine 180-106μm	38.75	7.75	7.7482

Solids loading Investigation:

Syringe 1 (22.0% Dymax)

Order	Material	Percentage	Required (g)	Actual (g)
1	Melamine 106-68μm	39.00	7.80	7.7998
2	Dymax	22.00	4.40	4.4458
3	Melamine 180-106μm	39.00	7.80	7.8017

Syringe 2 (22.5% Dymax)

Order	Material	Percentage	Required (g)	Actual (g)
1	Melamine 106-68μm	38.75	7.75	7.748
2	Dymax	22.50	4.50	4.5037
3	Melamine 180-106μm	38.75	7.75	7.7515

Syringe 3 (23.0% Dymax)

Order	Material	Percentage	Required (g)	Actual (g)
1	Melamine 106-68μm	38.50	7.70	7.7027
2	Dymax	23.00	4.60	4.6007

3	Melamine 180-106μm	38.50	7.70	7.7021

Syringe 4 (23.5% Dymax)

Order	Material	Percentage	Required (g)	Actual (g)
1	Melamine 106-68μm	38.25	7.65	7.6480
2	Dymax	23.50	4.70	4.7040
3	Melamine 180-106μm	38.25	7.65	7.6450

Syringe 5 (24.0% Dymax)

Order	Material	Percentage	Required (g)	Actual (g)
1	Melamine 106-68μm	38.00	7.60	7.5990
2	Dymax	24.00	4.80	4.7964
3	Melamine 180-106μm	38.00	7.60	7.5987

Syringe 6 (24.5% Dymax)

Order	Material	Percentage	Required (g)	Actual (g)
1	Melamine 106-68μm	37.75	7.55	7.5514
2	Dymax	24.50	4.90	4.8985
3	Melamine 180-106μm	37.75	7.55	7.5500

Dye Colour Chart:

Red dye No.1:

Order	Material	Percentage	Required (g)	Actual (g)
1	Melamine 180-106μm	38.75	7.75	7.750
2	Dymax	22.4	4.48	4.509
3	Melamine <63μm	38.75	7.75	7.760
4	Red Dye	0.10	0.02	0.030

Red dye No.2:

Order	Material	Percentage	Required (g)	Actual (g)
1	Melamine 180-106μm	38.75	7.75	7.7526
2	Dymax	22.30	4.46	4.4668
3	Red Dye	0.20	0.04	0.0390
4	Melamine <63μm	38.75	7.75	7.7568

Red dye No.3:

Order	Material	Percentage	Required (g)	Actual (g)
1	Melamine 180-106μm	38.75	7.75	7.7493
2	Dymax	22.30	4.46	4.4635
3	Red Dye	0.20	0.04	0.0468
4	Melamine <63μm	38.75	7.75	7.752

Red dye No.4:

Order	Material	Percentage	Required (g)	Actual (g)
1	Melamine 180-106μm	38.75	7.75	7.7514
2	Dymax	22.30	4.46	4.4911
3	Red Dye	0.20	0.04	0.0409
4	Melamine <63μm	38.75	7.75	7.7544

Red dye No.5:

Order	Material	Percentage	Required (g)	Actual (g)
1	Melamine <63μm	38.75	7.75	7.752
2	Dymax	22.30	4.46	4.6034
3	Red Dye	0.20	0.04	0.0422
4	Melamine 180-106μm	38.75	7.75	7.7812

Red dye No.6:

Order	Material	Percentage	Required (g)	Actual (g)
1	Melamine 106-63μm	37	7.4	7.3999
2	Dymax	25.00	5	4.969
3	Red Dye	1.00	0.2	0.1916
4	Melamine 180-106μm	37	7.4	7.3987

Red dye No.7:

Order	Material	Percentage	Required (g)	Actual (g)
1	Melamine 106-63μm	37	7.4	7.3999
2	Dymax	25.00	5	4.969
3	Red Dye	1.00	0.2	0.1916
4	Melamine 180-106μm	37	7.4	7.3987

Red dye No.8:

Order	Material	Percentage	Required (g)	Actual (g)
1	Melamine 106-63μm	37	7.4	7.4052
2	Dymax	25.00	5	4.9765
3	Red Dye	1.00	0.2	0.1946
4	Melamine 180-106μm	37	7.4	7.4065

Red dye No.9:

Order	Material	Percentage	Required (g)	Actual (g)
1	Melamine 106-63μm	37	7.4	7.4013
2	Dymax	25.00	5	4.9807
3	Red Dye	1.00	0.2	0.2067
4	Melamine 180-106μm	37	7.4	7.4048

Red dye No.10:

Order	Material	Percentage	Required (g)	Actual (g)
1	Melamine 106-63μm	37	7.4	7.4019
2	Dymax	25.00	5	4.987
3	Red Dye	1.00	0.2	0.2266
4	Melamine 180-106μm	37	7.4	7.4015

Red dye No.11:

Order	Material	Percentage	Required (g)	Actual (g)
1	Melamine 106-63μm	37	7.4	7.3983
2	Dymax	25.00	5	4.9843
3	Red Dye	1.00	0.2	0.1898
4	Melamine 180-106μm	37	7.4	7.3994

Dye Investigation:

Syringe 1:

Order	Material	Percentage	Required (g)	Actual (g)
1	Melamine 106-63μm	37.25	7.45	7.4478
2	Iron Oxide	0.5	0.1	0.1018
3	Dymax	25.00	5	5.0224
4	Melamine 180-106μm	37.25	7.45	7.4517

Syringe 2:

Order	Material	Percentage	Required (g)	Actual (g)
1	Melamine 106-63μm	37.25	7.45	7.4521
2	Iron Oxide	0.5	0.1	0.0994
3	Dymax	25.00	5	5.0128
4	Melamine 180-106μm	37.25	7.45	7.4513

Syringe 3:

Order	Material	Percentage	Required (g)	Actual (g)
1	Melamine 106-63μm	37	7.4	7.3994
2	Graphite powder	1	0.2	0.2148
3	Dymax	25.00	5	5.0004
4	Melamine 180-106μm	37	7.4	7.3979

Syringe 4:

Order	Material	Percentage	Required (g)	Actual (g)
1	Melamine 106-63μm	37	7.4	7.4024
2	Graphite powder	1	0.2	0.1995
3	Dymax	25.00	5	4.9945
4	Melamine 180-106μm	37	7.4	7.4038

Syringe 5:

Order	Material	Percentage	Required (g)	Actual (g)
1	Melamine 106-63μm	37	7.4	7.4034
2	Red drain dye	1	0.2	0.1968
3	Dymax	25.00	5	5.0291
4	Melamine 180-106μm	37	7.4	7.4017

Syringe 6

Order	Material	Percentage	Required (g)	Actual (g)
1	Melamine 106-63μm	37	7.4	7.399
2	Blue drain dye	1	0.2	0.2609
3	Dymax	25.00	5	5.0529
4	Melamine 180-106μm	37	7.4	7.4016

JH Major Project

GRADEMARK REPORT

FINAL GRADE

79/100

GENERAL COMMENTS

Please ask Jenni Steen for your marking sheet for feedback.

PAGE 1

PAGE 2

PAGE 3

PAGE 4

PAGE 5

PAGE 6

PAGE 7

PAGE 8

PAGE 9

PAGE 10

PAGE 11

PAGE 12

PAGE 13

PAGE 14

PAGE 15

PAGE 16

PAGE 17

PAGE 18

PAGE 19

PAGE 20

PAGE 21

PAGE 22

PAGE 23

PAGE 24

PAGE 25

PAGE 26

PAGE 27

PAGE 28

PAGE 29

PAGE 30

PAGE 31

PAGE 32

PAGE 33

PAGE 34

PAGE 35

PAGE 36

PAGE 37

PAGE 38

PAGE 39

PAGE 40

PAGE 41

PAGE 42

PAGE 43

PAGE 44

PAGE 45

PAGE 46

PAGE 47

PAGE 48

PAGE 49

PAGE 50

PAGE 51

PAGE 52

PAGE 53

PAGE 54

PAGE 55

PAGE 56

PAGE 57

PAGE 58

PAGE 59

PAGE 60

PAGE 61

PAGE 62

PAGE 63

PAGE 64

PAGE 65

PAGE 66

PAGE 67

PAGE 68

PAGE 69

PAGE 70

PAGE 71

PAGE 72

PAGE 73

PAGE 74

PAGE 75

PAGE 76