# Summary of the PHA feasibility tests for FDM Printing

## 1. Main processes

Sample preparation → Compounding → Filamenting → Printing → Testing

## 2. Sample preparation

Provided sample material (flakes in heterogenic particle size) were crushed by hand so that the particle size was under 4 mm and then the material was dried in 50C over night.

## 3. Compounding

Blending materials → Extruding → Cooling in waterbath → Granulating

Compounding was carried out with a co-rotating twin extruder. The sample materials were blended 20wt% to PBS polymer (grade FZ91PB from PTT MCC Biochem Company Limited) using following temperature phase profile:

130-140-140-150-150-140-140C (nozzle)

Screw speed: 200rpm, melt temperature: 132C

## 4. Filamenting

Compounded granulate was dried over night in 50C. Filamenting was carried out with a 3Devo Composer350 filament winder using following profiles:

### 4.1 PBS:

Heating: 125-130-130-125C (nozzle)

Screw: 4.0rpm

Cooling fans: 25%

Filament diameter: 2,85mm

### 4.2 Brewery PHA 20wt% in PBS

Heating: 130-150-150-140C (nozzle)

Screw: 4.0rpm

Cooling fans: 50%

Filament diameter: 2,85mm

### 4.3 Juice PHA 20wt% in PBS

Heating: 150-160-150-140C (nozzle)

Screw: 5.0rpm

Cooling fans: 100%

Filament diameter: 2,85mm

# 5. Printing

Printing of  the tensile test specimens was carried out with a Ultimaker S3 FDM printer. Main specifications were following:

## 5.1 PBS:

Nozzle temperature: 190C

Build plate temperature: 95C with Magigoo glue

Nozzle diameter (core AA): 0.8mm

Layer height: 0.4mm

Cooling fans: 0%

## 5.2 Brewery PHA 20wt% in PBS

Nozzle temperature: 170C

Build plate temperature: 95C with Magigoo glue

Nozzle diameter (core AA): 0.8mm

Layer height: 0.4mm

Cooling fans: 0%

## 5.3 Juice PHA 20wt% in PBS

Nozzle temperature: 170C

Build plate temperature: 95C with Magigoo glue

Nozzle diameter (core AA): 0.8mm

Layer height: 0.4mm

Cooling fans: 0%

Printing specifications were modified in the Ultimaker Cura slicing software (version 5.4.0) from a generic polypropylene material profile for the printer. The same print file was used for the reference and both sample materials. For the sample materials, the printing temperature and material flow were slightly tuned in the printer to achieve as similar output as possible. Temperatures were lowered 190→170C from the reference and the material flow was increased 100%→110%. Further details of the slicing and printing can be found from the provided Cura project file (.3mf) and print file (.ufp).

# 6. Testing

Tensile strength test were carried out with a Wance testing machine (model: TSE10104B, software: TestPilot_X10A v2.1.1016) using ISO 527 standard method. Results in the following table:

| | **PBS (reference)** | **Brewery PHA 20% in PBS** | **Juice PHA 20% in PBS** |
|---|---|---|---|
| Modulus of elasticity [MPa] | 807,6 | 692,9 | 576,0 |
| Tensile strength [MPa] | 38,5 | 21,9 | 18,4 |
| Tensile strain at break [%] | 25,7 | 14,6 | 12,6 |
| Tensile strain at tensile strength [%] | 22,1 | 12,9 | 12,4 |
| Tensile strain at yield [%] | 14,6 | 8,0 | 7,2 |
| Tensile strength at yield [MPa] | 37,3 | 21,4 | 17,4 |

# 7. Feasibility discussion

## 7.1 Overview

This feasibility test proved that PHA materials can be blended with PBS polymer and the blend can be processed to filament for FDM printing. However, according to these tests, adding PHA material decreases the tensile strength and strain. Decreasing strain can be useful in some applications but in most cases it would be desirable not to decrease the tensile strength at the same time. In these results, the tensile strenght still stayed in a usable range for some use cases.

## 7.2 Compounding

In this test there was not major issues in the compounding phase. Further test could include other blend ratios and/or with other polymer blends. First it might be reasonable to study the properties of the material itself and investigate the usable temperature range where material can be processed without degrading and losing mechanical properties. This way it would be easier to evaluate suitable other polymers to blend with. Test might be best to carry out from injection molded test specimens to eliminate effects of the printing process. In this phase there was not enough sample material to carry out these kind of tests.

## 7.3 Filamenting

Filamenting of the PHA material is a bit challenging. It's notable that filamenting of the PBS is also quite challenging. PBS is very prone to warping if cooled down too fast and/or unevenly leading to oval filament. PBS is very rubbery like when it's melted and it hardens very slowly when it cools down. PHA has somewhat similar caracteristics. In this test though, it was not possible to produce 100% PHA filament due to the small sample amount. Also, even with blended with the PBS, it might not been possible to blend in much more than 20wt% of PHA since it started to decrease the melt viscosity/strenght so that cooling capacity was not sufficient in the used machine. Slowing down speed didn't help either since the melt couldn't stand much of its own weight without straining and breaking before the winding rolls. Even if the cooling was sufficient, the material might be too brittle for winding but that cannot be confirmed with these tests. Using cooling fans at full speed also made the filament very heavily oval shaped which was not desirable but it was possible to get some printable filament. Longer cooling line or different kind of filamenting method might help to get better quality filament. Modifying (chain extending, crosslinking, fiber reinforcing etc.) material itself might help too.

## 7.4 Printing

Mainly the same properties that made filamenting challenging caused similar challenges in printing. Due to the warping/shrinking the adhesion to the buildplate is not very easily achieved. In this test the build plate temperature had to be raised to 95C so that the specimen wasn't detached before the print was ready. Printing temperature had to be quite high too and cooling fans was not used for the same reason. Otherwise the materials could be extruded in a lower temperatures too. These parameters were suitable to get the tensile test specimens printed successfully but might not apply to different shaped objects, especially to tall objects. Compared to the reference PBS, the PHA containing samples printed cleaner/easier since the melt viscosity was lower and material was not that rubbery/stringy which causes challenges in tight direction changes in printing. All the prints were basically in melt form to the end of the print and then cooled quite slowly on the build plate. This was a good thing for the layer adhesion but of course wouldn't be possible to achieve very tall objects this way since object is soft and starts to bend easily when printed more layers. Material detached itself from the build plate right after it started to cool down. At least closed printing chamber is highly recommended and more preferably temperature controlled chamber which could help a lot to limit the warping tendency. Printing PBS/PHA-blend could be described somewhat similar to printing polypropylene. It is completely possible but not very easy without good control and optimization of the chamber temperature.

## 7.5 Testing

There was not issues with testing the specimens. All the samples snapped in the right region, about middle of the sample. The break points were surprisingly sharp and clean for a printed specimens. Basically no visible detached strings or delamination of the layers but there was some small cavities probably due to variation of the material flow in printing caused by small changes in the filament diameter. Most surprising was the significant difference in the reference PBS tensile strain at break that was much lower than material technical data sheet indicated. It is quite common that strains of the printed specimens are quite a bit lower and varies more than TDS or relative injection molded samples but in this case the difference was huge (26% vs. >300%). There must be some unoptimal crystallisation and tensions happening. Tensile strenght was basically the same as it stated in TDS, indicating good accuracy of the results and feasible material processing and printing parameters.