Microscale imaging of leak pathways in FDM 3D printed fluidics

Rowan Leeder,[a] Kathryn E. Rankin,[b] Adrian M. Nightingale[a]

[a] Mechanical Engineering, Faculty of Engineering and Physical Sciences, University of Southampton, SO17 1BJ, UK
[b] µ-VIS X-ray Imaging Centre, Faculty of Engineering and Physical Sciences, University of Southampton, SO17 1BJ, UK

Abstract:

3D printing is a highly attractive method for manufacturing micro- and milli-fluidic devices due to fast fabrication times and low barrier to entry. Of the common 3D printing methods, fused deposition modelling (FDM) is the most accessible but is also susceptible to leakages if printed with default settings. Previous reports have described how specific print parameters are required to ensure reliable leak-free fluidics including increased flow rates, small layer heights, and 100% infill. Here we combine computer tomography (CT) X-ray imaging with bulk leak testing to understand the fundamental structural reasons for the previously reported recommended print parameters. In keeping with previous reports, we see that smaller layer heights (<0.1mm) and increased flow rates (>100 % compared to recommended rate) prevent leakage. We show these two parameters prevent leaks by increasing the quality and integrity of channel walls, though the relevant importance of each parameter depends on whether channels are orientated vertically or horizontally compared to the print bed. In contrast to previous reports, we show that the degree of infill is irrelevant as print bodies are intrinsically porous and it is the integrity of channel walls that determines whether leaking will occur. Consequently channels can be printed with low infills without sacrificing leak integrity and consequently print times and material costs can be greatly reduced (over 50 % time and cost savings for the test pieces used here).

Introduction

3D printing is an increasingly popular tool for fabricating micro- and milli-fluidic systems.[1, 2] While they cannot reach the sub-micron resolution of devices replica-moulded from lithographically fabricated masters, they offer advantages in terms of speed, cost, and accessibility. Fused deposition modelling (FDM) and photocure printing methods (e.g. stereolithographic addition, SLA) are the most common due to the wide availability of low-cost printers. In FDM printing thermoplastics are extruded through a heated nozzle that can move in the x-y plane, such that the nozzle puts down a series of pathways to build up a two-dimensional pattern. If the pattern is on a movable z-stage, two-dimensional layers can be built up, one upon the other, to generate three-dimensional features. While the spatial resolution on FDM printing is slightly inferior to photocure methods,[3] prints can be made in a much wider range of materials, with different mechanical and electrical properties and excellent chemical compatibilities. Furthermore, external items (e.g. electrodes, optics, membranes) can be easily incorporated to expand the functionality of finished printed devices.[4-6] FDM printing has allowed users to design and fabricate a range bespoke flow equipment, including flow reactors with 3D mixing elements,[7] photochemical reactors,[8] redox flow batteries,[9] chromatography columns,[10] and filtration and separation devices.[6]

A challenge with FDM printed fluidics however is they typically leak when fabricated using default print settings. Users can control a range of print setting options to tailor the print and for leak-free fluidics, recent papers have recommended using low layer heights (0.1 mm or less[3, 11-15]), increased flow rates (typically 4-10 % greater than the software-recommended flow rate[8, 12-15]) and 100% infill[3, 8, 11-14] (where infill determines how much material is deposited in the print interior). The recommended print settings have been arrived at empirically, but are hypothesised to prevent leakages by removing small air gaps between neighbouring pathways[13] that can result from the rounded pathway cross sections[16, 17] In this work we use computed tomography (CT) X-ray imaging to find evidence for the leak pathways - linking macroscopic observations of leakage with microscopic observations of the internal structure of printed parts. Our aim was to understand the fundamental basis for the recommended print parameters, how leaking occurs in FDM-printed fluidics, and arrive at an informed understanding of how to best set print parameters.

Experimental

Standardised test pieces were designed with a single channel (1.5 mm diameter, 40 mm length) which was closed at one end and had a female ¼-28” fitting at the other end to connect to external tubing. The channel width was deliberately chosen to be conservatively wide and easily printable to ensure reliable printing. Two variants of this design were implemented to allow the main channel to be positioned vertically (Fig. 1a-c) or horizontally (Fig. 1d-f) whilst ensuring that the ¼-28” fitting was always printed in a vertical orientation- ensuring the screw threads printed well and that the sealing surface (at the bottom of the fitting) was flat and smooth to allow reliable sealing to a flangeless fitting (IDEX). In each model the channels were surrounded by 9.7 mm of solid material, and markings were patterned into the outer surface so that the position of any external leaks could be related back to the print orientation.

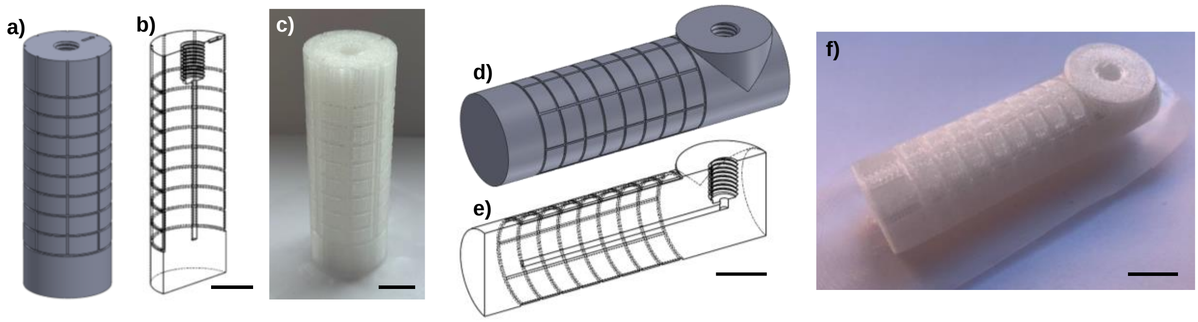

Figure 1: Images showing the test pieces printed with vertical channels (a-c) and horizontal channels (d-f). In each case the design is shown as a solid device (a,d), in cross-section (b,e) and as printed (c,f). All scale bars represent 1 cm.

All test pieces were printed on an Ultimaker 3 printer in polypropylene (Ultimaker). Polypropylene was used as this is the most chemically compatible of the commonly available filament materials and hence best suited to fluidic applications. It has been widely used in studies of FDM printed fluidics.[6, 12-14, 16] and more generally in 3D printed reactors.[5, 8, 18-20] Pieces were designed in Solidworks, exported as .stl files, then imported into Ultimaker Cura to prepare print settings. The default settings for polypropylene were used, with the exception of layer height, flow rate, and infill which were adjusted as later described.

Leak testing was done in two ways - qualitive manual testing and a quantitative approach. For the manual testing a 10 ml disposable syringe (BD Plastipak) was connected to the test piece and then pressure applied by hand (giving gauge pressures > 160 kPa). The quantitative approach involved exposing the test piece to a pressurised system and then tracking what happened to the systems pressure after exposure: Each test piece was connected in series to a) an open/close manual valve (Idex P-782), b) a T-junction (Idex P-713) with pressure sensor (NXP, MPX4250A) used to quantify the system pressure and c) a syringe pump (KD Scientific KDS 100, using a 10 ml BD Plastipak syringe) used to pressurise the system. Throughout, ¼-28" flangeless fittings were used (IDEX) and PTFE tubing (2 mm inner diameter). The pressure sensor was connected to an Arduino Nano microcontroller which in turn fed readings to a desktop computer running a Labview script (developed in-house) to continually record the system pressure. During testing, the valve was initially closed and the syringe pump run (1 ml/min nominal flow rate) until the gauge pressure increased to approximately 150 kPa. When the required pressure had been achieved, the syringe pump was stopped, and the pressure reading left to stabilise (~30 s). The valve was then opened to expose the test piece to the pressurised side of the system and the ensuing pressure trend recorded.

For CT imaging, test pieces were imaged using a Nikon XTEK XTH 225 kVp micro-focus CT system. For each piece 1501 radiographs were taken as the bed was rotated through 360° around a single axis, using the scan parameters as follows: 100 kVp peak voltage, 238 µA current, 250 ms exposure time, 24 dB analogue gain, 4 frames per projection. The projection data was reconstructed to produce a volume with a spatial resolution of 0.025 mm.

Results

Previous literature reports of leak-free fluidics recommend using 100% infill, low layer heights (0.1 mm or lower), and increased flow rates.[3, 8, 11-15] Of these parameters, the infill makes the most intuitive sense as it should leave no spaces in the bulk of the print for fluid to leak into. Hence we began by keeping infill constant at 100 % and investigating the role of over-extrusion (increased flow rate) and layer height. Over extrusion is the most notable of these parameters as it is not normally used for standard (non-fluidic) printing applications and the option to control this parameter is not easily accessible within slicing software, in contrast to layer height or infill. By increasing the flow of the plastic without increasing the distance between layer paths or layer height, the width of the extruded pathway (i.e. the width of the molten plastic trail put down by the moving nozzle) increases, such that it should better contact (and hence better bond with) neighbouring pathways in each 2D print layer. An inherent disadvantage of over-extrusion, however, is that it will drive printed dimensions away from their nominal sizes (increasing dimensions of positive features in the x-y plane, decreasing dimensions of negative features), increasing the need for empirical dimension optimisation, and hence over-extrusion should be avoided if possible.

a)

Flow rate (%)	Layer height (mm)			
	0.06	0.10	0.15	0.20
95	No	Yes *	Yes	Yes
100	No	No	Yes	Yes
110	No	No	No	Yes

b)

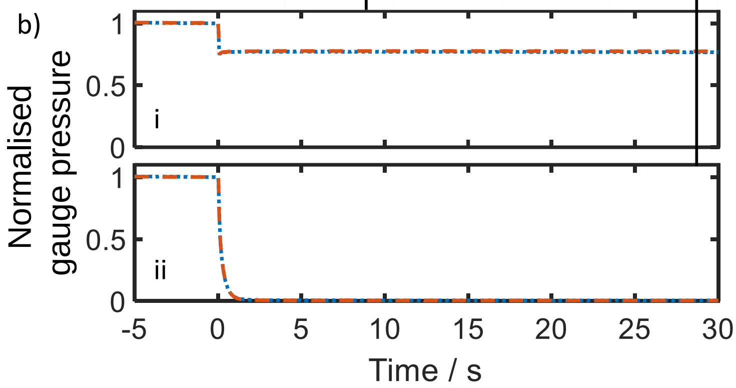

Figure 2: a) Manual test results for test pieces with a vertical main channel, printed with varying layer heights and flow rates, showing whether leaking was observed. For the 0.10 mm layer height, 95 % flow rate sample (result marked "") leaking was only observed at elevated pressures. Example results for two quantitative tests are shown below: b)i - a leak-free test piece printed with 100 % flow rate, 0.06 mm layer height, and b)ii - a leaking test piece printed with 100 % flow rate, 0.20 mm layer height. In both cases, the valve exposing the test piece to the elevated pressure was opened at t = 0. For both quantitative tests two separate measurements of the same device are shown (red dashed and blue dotted lines). In each case the lines overlay each other showing the measurements to be repeatable.*

Testing began with the test pieces with vertical channels (Fig. 1a-c). Multiple versions were printed, with layer heights ranging between 0.06 and 0.2 mm (the standard range suggested by the slicing software for this printer and material) and extrusion rates of 95, 100 and 110 %. Fig. 2a shows the results from manual leak testing. At the smallest layer height, no leaks were detected in any of the test pieces, even when the plastic was under-extruded at 95 %. Extrusion rate also had a positive effect, most notably at the higher layer heights - for example the 0.15 mm layer height print was only leak-free at 110 % extrusion. For all pieces that leaked, the observed external position of the leaks varied randomly and could not be linked to print orientation.

The manual results were subsequently checked with quantitative testing in which test pieces were exposed to a pre-pressurised fluidic manifold and the drop in pressure monitored. All testing confirmed the findings from manual testing, as shown by the representative results in Fig. 2b. When a leak-free test piece was tested (Fig. 2b.i) the pressure drop on exposure was finite, consistent with the pressure dissipating across an increased volume but immediately stabilising due to the absence of leaks. By contrast, when a leaky test piece was exposed to the pressurised system (Fig. 2b.ii), the pressure continuously dropped until the system was completely depressurised - consistent with fluid being freely lost. These findings correlate well with previous reports that emphasise the positive effect of over-extrusion (high flow rates) and low layer height, however it is notable that test pieces printed with the lowest layer heights (≤ 0.1 mm) did not require over-extrusion. The avoidance of over-extrusion where possible would be allow printed dimensions to more closely match the nominal dimensions as defined in the computer model.

We then imaged identical (but untested) test pieces with vertical channels to investigate the structural cause of the leaks. Fig. 3 shows CT scans of test pieces printed with differing layer heights (a-d) but the same extrusion flow rate (100 %), shown as vertical (i) and horizontal (ii-v) cross sections. The horizontal cross sections (on a parallel plane to the print bed) clearly show the two-dimensional printing pathways taken by the printhead as it lay down each layer. It shows the test piece exterior and the fluidic channel were clearly defined by "wall" pathways with infill in between, put down as parallel lines. Despite the 100 % infill, the body of each print is far from being a solid monolithic piece. Airgaps are visible within the interior of all test pieces irrespective of layer height setting. The porousness of the test pieces indicate that the main print body will have minimal to no effect on preventing leakage and hence it is the integrity of the walls, and in particular the walls of the fluidic channel, that will determine whether a piece leaks or not.

The print quality of the channel walls correlated well with the corresponding manual leak testing: At low layer height (e.g. 0.06 mm layer height, Fig. 3a) the cross sections show the channel walls to be consistently well printed, with no visible gaps, and a good connection between walls and infill. As the layer height increases (Fig. 3b-d), the quality of the channel walls visibly deteriorate. At the extreme of 0.2 mm gaps are clearly visible (Fig. 3d.ii-iv), with clear pathways from the channel into the infill area (Fig. 3a.v). The high quality of the low layer height prints is consistent with the bulk leak testing (where over extrusion was not required) and shows how leak prevention is consistent with the quality of the channel walls. This correlates well with a previous report that found increasing wall size had a positive effect on leak prevention,[21] though in our own testing we found wall size had no impact on leakage (data not shown).

The importance of layer height here is likely due to the vertical orientation of the channels during printing, where connection between layers (rather that between pathways within layers) is of optimum importance. Small layer heights will generate a cross-section with a higher aspect ratio, which will lead to an increased contact area between layers.

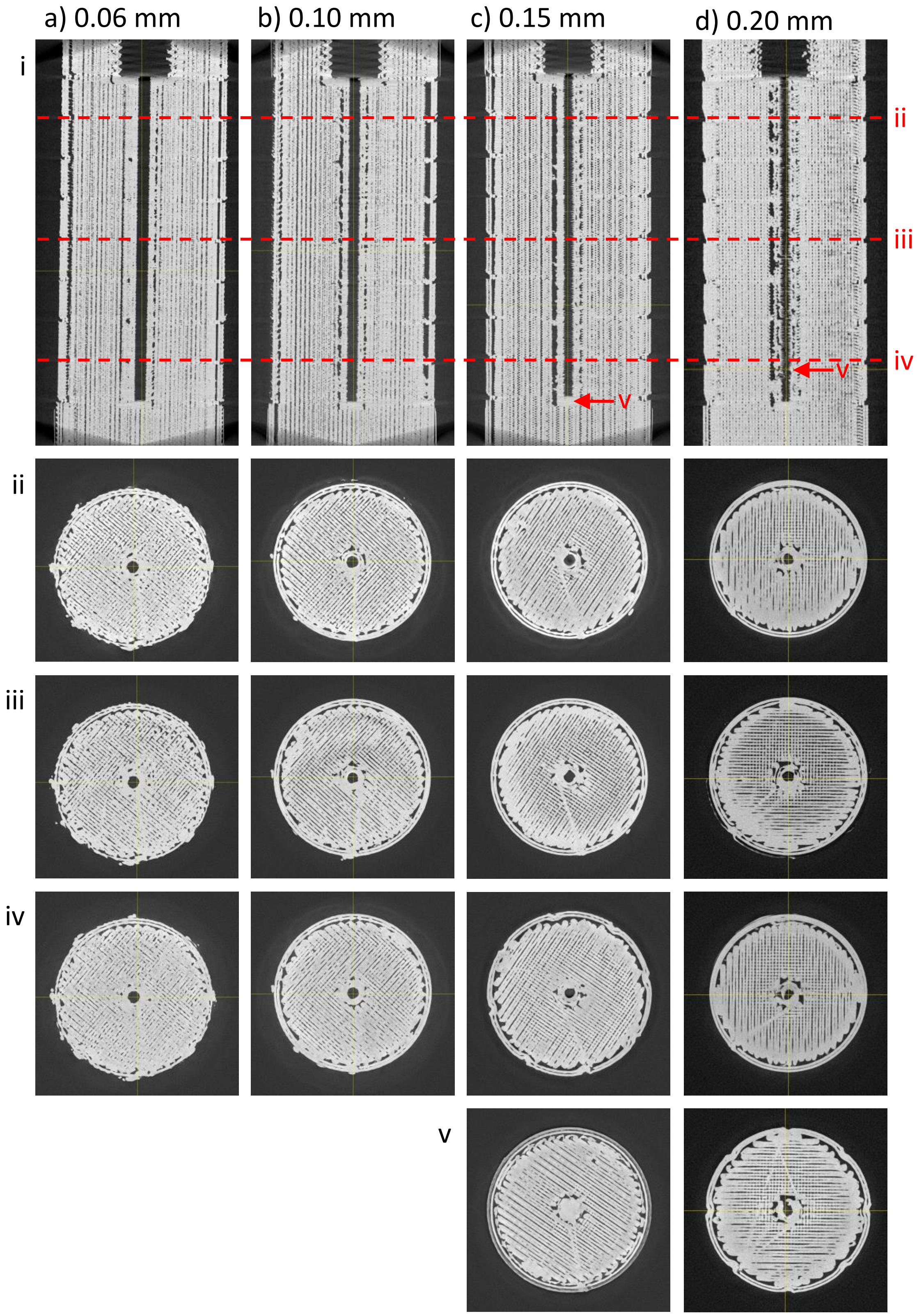

Figure 3: CT scan images of test pieces with a vertical channel printed at 100 % flow rate and layer heights of a) 0.06 mm, b) 0.10 mm, c) 0.15 mm, and d) 0.20 mm. Each are shown in vertical profile (i) and below in horizontal cross-section (ii, iii, and iv) at the positions indicated by the red dotted lines in (i). Additional cross-sections (v, marked by red arrows in i) show the intact base of the channel for the 0.15 mm test piece (c), and a very clear break in the channel wall for the 0.20 mm test piece (d).

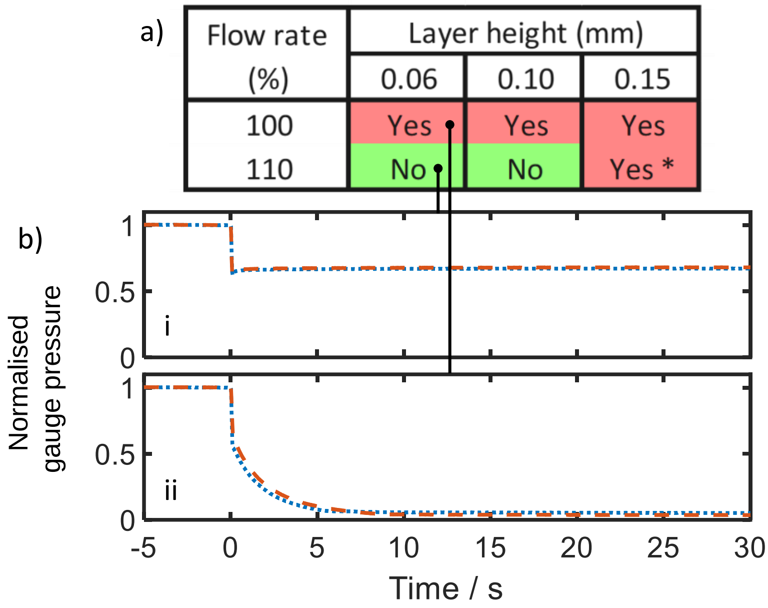

Figure 4: a) Manual test results for test pieces with a horizontal main channel, printed with varying layer heights and flow rates, showing whether leaking was observed. For the 0.15 mm layer height, 110 % flow rate sample (marked "") leaking was only observed at elevated pressures. The results of two quantitative tests are shown below: b)i - a leak-free test piece printed with 110 % flow rate, 0.06 mm layer height, and b)ii - a leaking test piece printed with 100 % flow rate, 0.06 mm layer height, where t = 0 represents the moment the pressurised system was exposed to the test pieces. For both quantitative tests two separate measurements of the same device are shown (red dashed and blue dotted lines) and in each case the lines overlay each other showing the measurements to be repeatable.*

To ascertain whether the findings for the vertical channel test pieces were more generally true, we then examined test pieces printed with horizontal channels (Fig. 1d-f). As was the case for the vertical channel test pieces, the test pieces with horizontal channels were printed with different layer heights and flow rates, whilst keeping the infill constant at 100 %. As before, leaks could be clearly identified from manual testing and this was corroborated by quantitative testing (Fig. 4). Again, the position of all external leaks varied randomly and could not be linked to print orientation.

The relative importance of the different print parameters were notably different compared to the vertical channel test pieces. Here flow rate was the most important parameter, with over-extrusion a requirement for leak free test pieces, irrespective of layer height (Fig. 4a) The reason behind this can be seen by looking at the interior structure. Fig. 5 shows three test pieces printed with differing print parameters, where a) and c) leaked, and b) was leak free. Again it can be seen that the bulk interior of all test piece bodies was porous, though this was markedly less pronounced when over-extruding at 110 % flow rate (Fig. 5b), consistent with previous reports.[9] The internal porosity of the prints again shows that if prints are to be leak-free, fluid must be contained by the channel walls and hence these must be printed without gaps or breakages. Fig 5aiii-v shows several cross sections and shows how the channel walls were constructed. Across the middle of the channel (Fig. 5b,iv) filament has been put down around the perimeter of the channels (similar to the cross section of the vertical channels shown in Fig. 3), however the top (Fig. 5b,iii) and bottom (Fig. 5b,v) of the channels are capped with 2D flat plates, constructed by putting down filament in a zig-zag pattern, similar to that used to infill the bulk of the test pieces. It is in those two-dimensional top and bottom pieces that imperfections in the channel wall are visible in the leaky test pieces (Fig. 5a,ii and Fig. 5c,ii). A good seal between neighbouring pathways in the 2D print plane is therefore key to having a water-tight seal and explains why over-extrusion is more important for these horizontal channels than for the test pieces with vertical channels – over extrusion increases the width of the bead (whilst maintaining the same height), increases the contact between neighbouring paths, and hence reduces chances of gaps in the plate structures that cap the top and bottom of the channels.

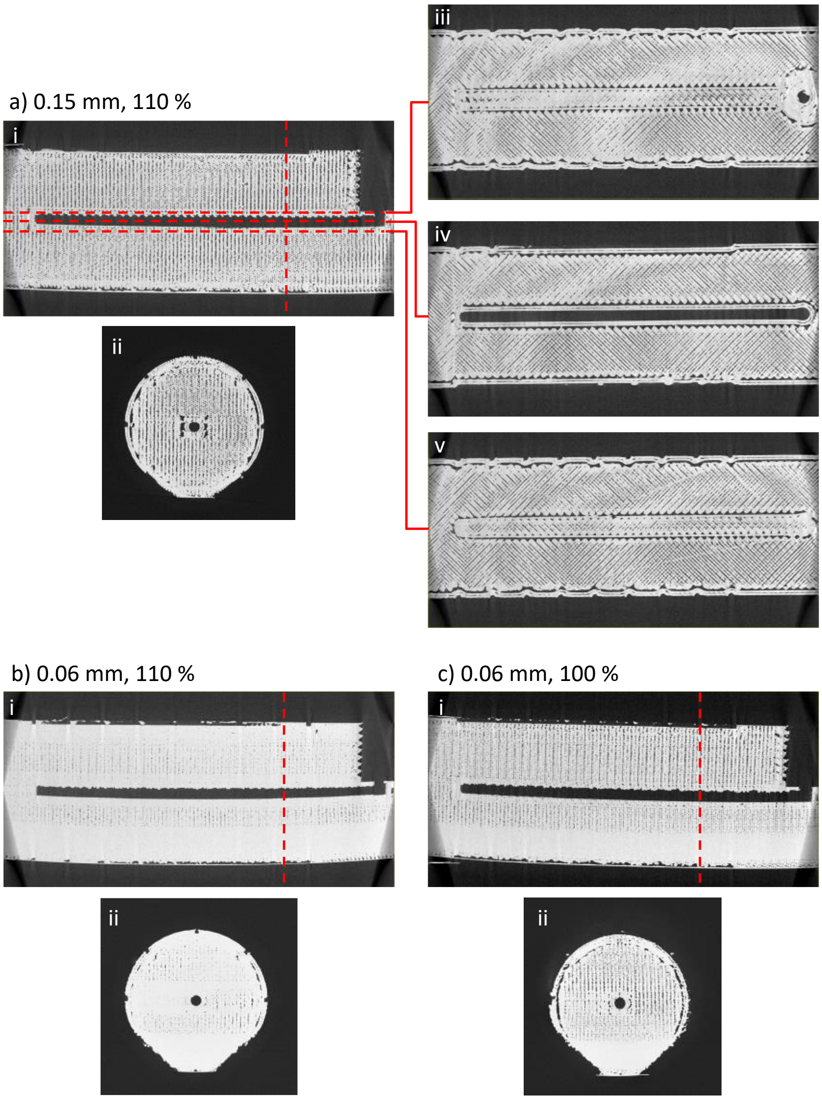

Figure 5: CT scan images of test pieces with a horizontal channel printed with a) 0.15 mm layer height and 100 % flow rate, b) 0.06 mm layer height and 110 % flow rate, and c) 0.06 mm layer height and 100 % flow rate. For a), the internal structure of the piece is shown as a vertical cross-section along the length of the piece (i), a vertical cross-section across the width (ii) and three horizontal cross sections positioned immediately above (iii), at the same height as (iv), and immediately below (v) the channel. The dashed red lines in (i) correspond to the position of the cross sections in (ii)-(v). For b) and c) the internal structure is shown via a vertical cross-section along the length of the piece (i) and a vertical cross-section across the width (ii). The dashed red lines in each (i) correspond to the position of the cross section shown in (ii).

Interestingly, the porousness of the test piece bodies and the importance of channel wall integrity suggests that the amount of infill should make no difference to whether a piece leaks or not. To test this we printed a vertical-channel test piece with 20 % infill, a layer height of 0.06 mm and 110 % flow rate (Fig 6). The resulting test piece had well defined contiguous channel walls (Fig. 6a,b) and was consequently leak free (Fig. 6c). The ability to reduce infill without compromising leak integrity is significant as reducing the infill reduces print time (e.g. 4.75 hours *vs* 10.75 hours for the vertical test piece here) and material use (9 g vs 20 g here).

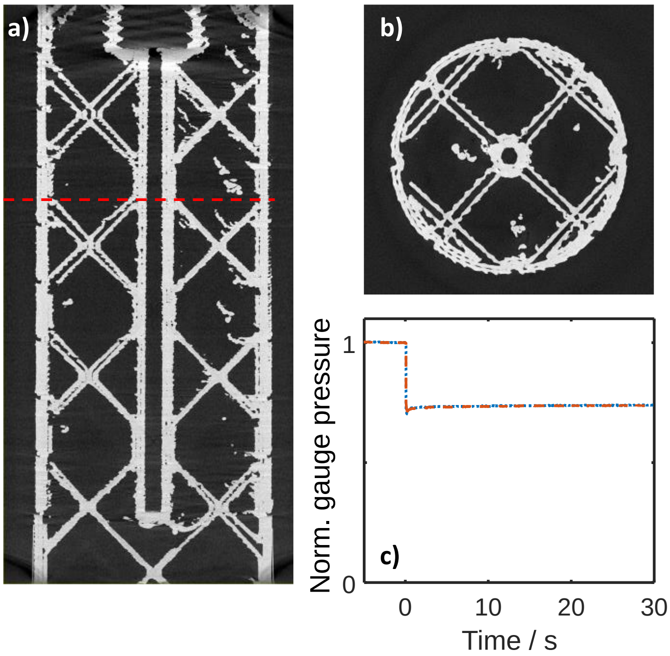

Figure 6: a)&b) CT scan images of test pieces with a vertical channel printed with 0.06 mm layer height and 100 % flow rate, shown as a vertical horizontal cross section (a) with a red dashed line showing the location of a corresponding horizontal cross section (b). Quantitative testing results of the same test piece are shown in (c). Two separate measurements of the same device are shown (red dashed and blue dotted lines) which overlay each other, showing the measurements to be repeatable.

Taken as a whole, these results confirm previous reports that low layer heights and increased flowrates lead to leak-free devices but show that these print parameters are of different importance depending on whether channels are vertical or horizontal. Moreover CT scans show the underlying reason for these parameter choices – ensuring channel walls are well formed. For vertical channels low layer height (≤0.1 mm) is most important to ensure channel wall integrity, while over-extrusion (>100%) is more important for horizontal channels. Hence both are recommended for most prints where channels can be in a mixture of orientations. The importance of channel walls also means that, with optimised layer height and flow rate settings, infills can be set much lower to greatly decrease print times and material usage – in contrast to previous literature recommendations of 100 % infill. Finally we note that this testing was done using polypropylene. While printing behaviour might differ slightly between materials, most common print materials have similar properties and printing behaviours and the findings described here are expected to be applicable for most common print materials.

References

1. R. Su, F. Wang and M. C. McAlpine, *Lab on a Chip*, 2023, **23**, 1279-1299.
2. M. B. Montaner and S. T. Hilton, *Current Opinion in Green and Sustainable Chemistry*, 2024, **47**.
3. N. P. Macdonald, J. M. Cabot, P. Smejkal, R. M. Guijt, B. Paull and M. C. Breadmore, *Analytical Chemistry*, 2017, **89**, 3858-3866.
4. F. Li, N. P. Macdonald, R. M. Guijt and M. C. Breadmore, *Lab on a Chip*, 2019, **19**, 35-49.
5. A. du Preez, R. Meijboom and E. Smit, *Food Analytical Methods*, 2022, **15**, 1816-1825.
6. M. J. Clark, T. Garg, K. E. Rankin, D. Bradshaw and A. M. Nightingale, *Reaction Chemistry & Engineering*, 2024, **9**, 251-259.
7. M. J. Harding, S. Brady, H. O'Connor, R. Lopez-Rodriguez, M. D. Edwards, S. Tracy, D. Dowling, G. Gibson, K. P. Girard and S. Ferguson, *Reaction Chemistry & Engineering*, 2020, **5**, 728-735.
8. M. R. Penny and S. T. Hilton, *Journal of Flow Chemistry*, 2023, **13**, 435-442.
9. H. O'Connor, J. J. Bailey, O. M. Istrate, P. A. A. Klusener, R. Watson, S. Glover, F. Iacoviello, D. J. L. Brett, P. R. Shearing and P. Nockemann, *Sustainable Energy & Fuels*, 2022, **6**, 1529-1540.
10. N. Abdulhussain, S. Nawada, S. Currivan, M. Passamonti and P. Schoenmakers, *Journal of Chromatography A*, 2020, **1623**, 461159.
11. M. Tiboni, M. Tiboni, A. Pierro, M. Del Papa, S. Sparaventi, M. Cespi and L. Casettari, *International Journal of Pharmaceutics*, 2021, **599**.
12. Z. X. Rao, B. Patel, A. Monaco, Z. J. Cao, M. Barniol-Xicota, E. Pichon, M. Ladlow and S. T. Hilton, *European Journal of Organic Chemistry*, 2017, **2017**, 6499-6504.
13. R. F. Quero, G. D. da Silveira, J. A. F. da Silva and D. P. de Jesus, *Lab on a Chip*, 2021, **21**, 3715-3729.
14. A. J. N. Price, A. J. Capel, R. J. Lee, P. Pradel and S. D. R. Christie, *Journal of Flow Chemistry*, 2021, **11**, 37-51.
15. S. Hapke, G. A. Luinstra and K. M. Zentel, *Journal of Flow Chemistry*, 2021, **11**, 539-552.
16. O. S. Carneiro, A. F. Silva and R. Gomes, *Materials & Design*, 2015, **83**, 768-776.
17. L. Wu, S. Beirne, J. M. Cabot, B. Paull, G. G. Wallace and P. C. Innis, *Lab on a Chip*, 2021, **21**, 3338-3351.
18. C. Malatini, C. Carbajales, M. Luna, O. Beltrán, M. Amorfn, C. F. Masaguer, J. M. Blanco, S. Barbosa, P. Taboada and A. Coelho, *Pharmaceuticals*, 2023, **16**.
19. P. J. Kitson, M. D. Symes, V. Dragone and L. Cronin, *Chemical Science*, 2013, **4**, 3099-3103.
20. P. J. Kitson, G. Marie, J. P. Francoia, S. S. Zalesskiy, R. C. Sigerson, J. S. Mathieson and L. Cronin, *Science*, 2018, **359**, 314-319.
21. G. I. J. Salentijn, P. E. Oomen, M. Grajewski and E. Verpoorte, *Analytical Chemistry*, 2017, **89**, 7053-7061.