Supplementary Information

3D printed metal columns for capillary liquid chromatography.

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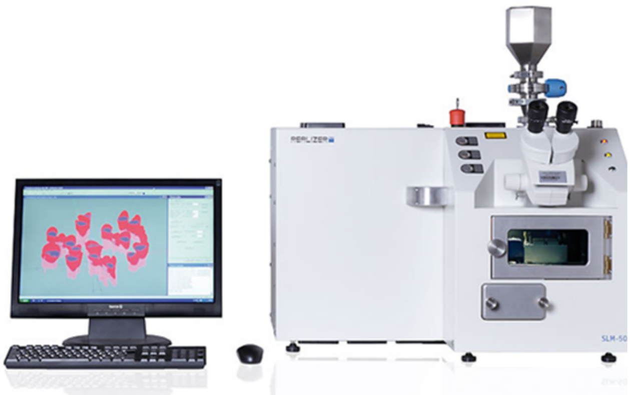
3D printing technology used

Realizer SLM 50 system. Selective Laser Melting (SLM) technology uses a high power laser to incrementally melt and fuse thin layers of powdered metal to form a 3D structure. The Realizer SLM-50 is a bench top system with a cylindrical build volume of Ø80mm x 40mm. Layer thicknesses of 0.025 mm can be achieved. Figure 1 shows the SLM050 and the principle of operation. The system process chamber is an argon environment with O2 content less than 0.1%. The machine catalogue is available at **http://pdf.directindustry.com/pdf/realizer/slm-50-desktop-machine/65956-434985.html**



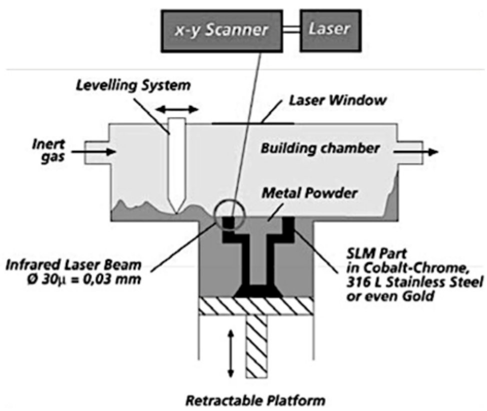
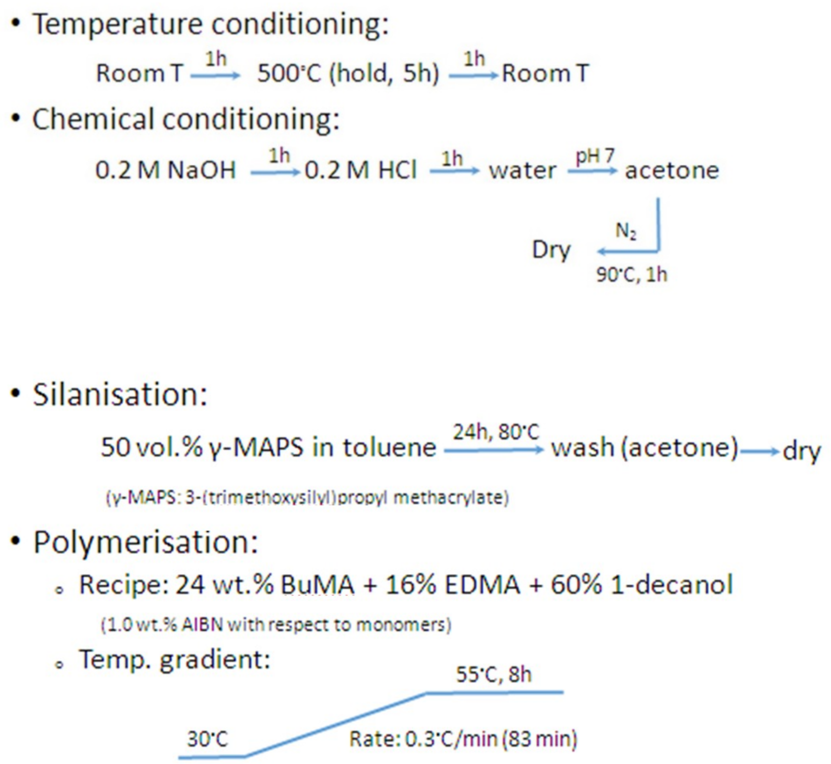


Figure ESI1: 3D metal printing, Selective Laser Melting (SLM)

Formation of polymeric frits in FSC:

Polymeric column frits (5 mm L) were prepared in fused silica capillaries (250 µm i.d., 360 µm o.d.). Firstly, the capillary wall was functionalised with 3-trimethoxysilylpropyl methacrylate (MAPS, 5% v/v), rinsed with MeOH and acetone and left overnight at room temperature. 10 cm sections of capillary were then dipped into a polymerisation mixture containing: 8 mg azobisisobutyronitrile (AIBN), 500 mg divinylbenzene, 300 mg styrene, 250 mg toluene and 950 mg dodecanol. When the polymerisation mixture entered the capillary (through capillary action) to a height of 5 mm, the latter was sealed and transferred to a water bath at 60 ^oC to allow thermal polymerisation. The capillary was then placed in a polytetrafluoroethylene (PTFE) sleeve and fitted in a Rheflex 1/16”, 10-32 fitting and connected to the stainless steel or titanium printed columns.

Formation of polymer monoliths in titanium printed columns:



Column heating system (version 1)

A direct contact heating system was developed using peltier thermoelectric heater / coolers allowing heating or cooling in either single sided or double sided configuration. Figure ESI2 shows the double sided set-up. On each side of the column are 30 x 30 mm peltier elements (37.4 Watt max, 3.9 Amp max, 15.7 Volt max, Δt max 73K) with heat-sinks and fans. The temperature was controlled by means of a PT1000 RTD in contact with the column and a Shinko MCS-100 temperature controller in On/Off mode. The system can control column temperature between 10 °C and 90 °C with an accuracy of 2 °C.

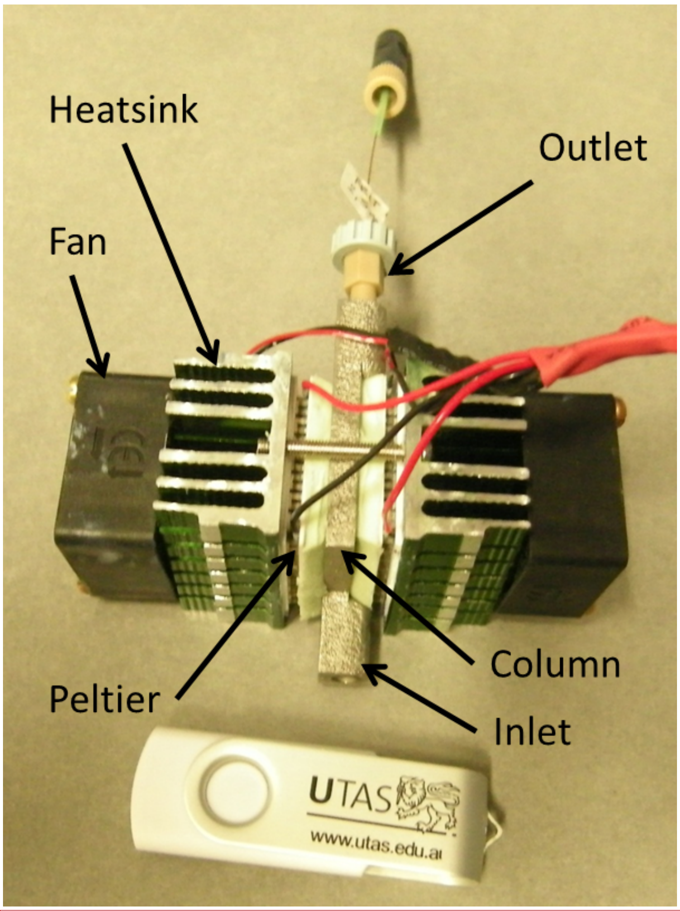


Figure ESI2: Printed column heating system (version 1), control system and power supply not shown. Here fitted with SS printed column.

Column heating system (version 2)

A computer controlled heating/cooling system was developed to allow bidirectional temperature control of the printed column. The system consists of three Peltier elements stacked to achieve a Δt max of 200K. These are attached to a heat-sink with a fan inside a metal enclosure as shown in Figure ESI3. The Peltier elements used are 30 x 30 mm (37.4 Watt max, 3.9 Amp max, 15.7 Volt max, Δt max 73K).

The system is controlled by a Texas instruments CC2511F32 micro-controller based development board called Wixel. This is programmed in a variant of C via USB comms. The board offers features including 3.3 V regulator, USB, low power radio, 12 bit ADC. The system communicates via USB to ExtraPutty terminal on a PC allowing input of desired temperature in 1 °C increments. The system is powered from a 12 Volt, 6 Amp mains adaptor power supply.

Temperature control is by means of a PT1000 RTD in contact with the column. This is read by the microcontroller which implements pseudo PID temperature control. The Peltier elements are driven using a pwm controlled motor driver to give a high degree of control and efficiency. The system can rapidly control temperature between 0 °C and 150 °C with an accuracy of ± 1 °C. A temperature ramp 0 °C to 100 °C takes approximately 1 minute depending on the thermal mass of the column.

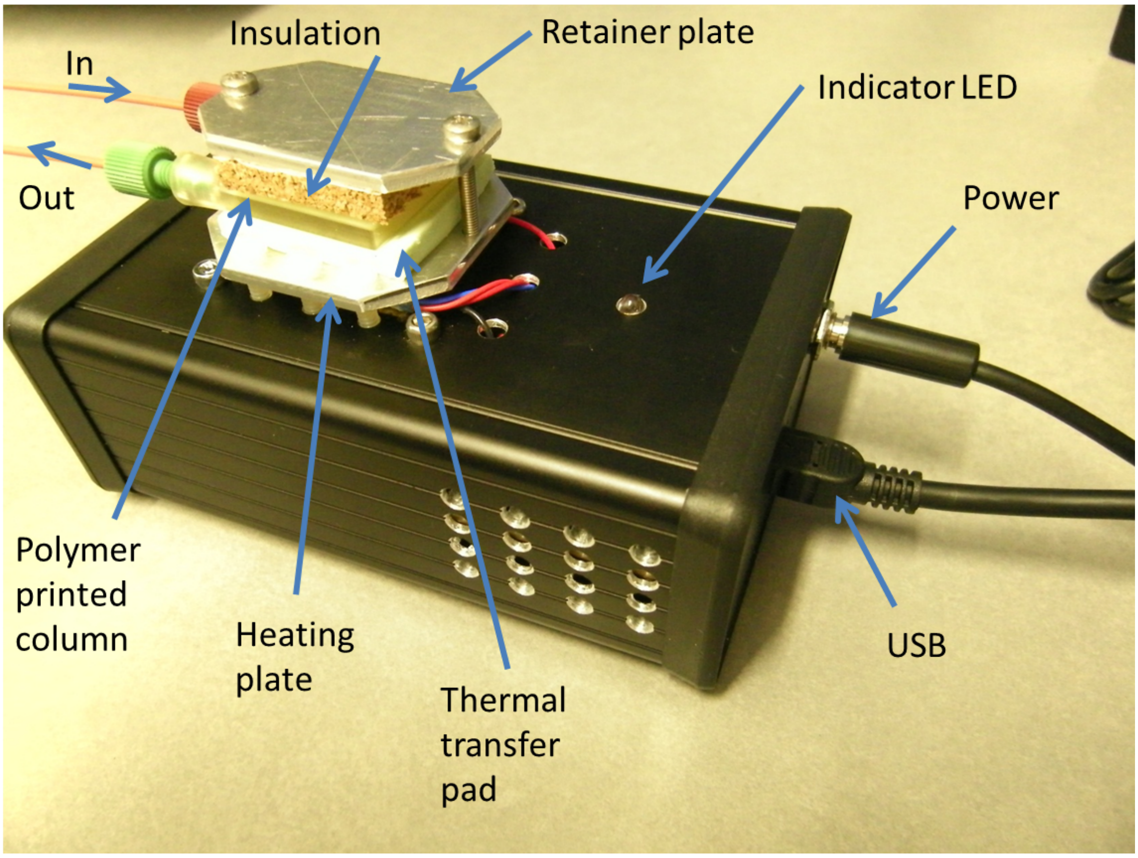


Figure ESI3: Printed column heating system (version 2), here fitted with polymer printed column.

van Deemter plots for particle packed SS printed columns

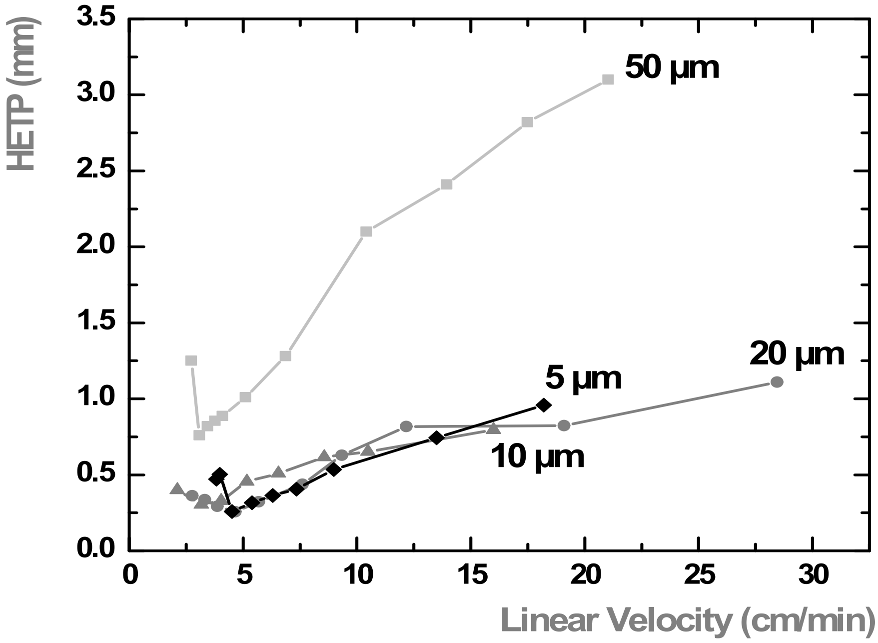


Figure ESI4. Van Deemter plot for stainless steel column packed with 5, 10, 20 and 50 µm particles. (b) Temperature effect on the efficiency of column packed with 5 µm particles.

<u>% ACN in mobile phase – ODS packed SS coiled column</u>

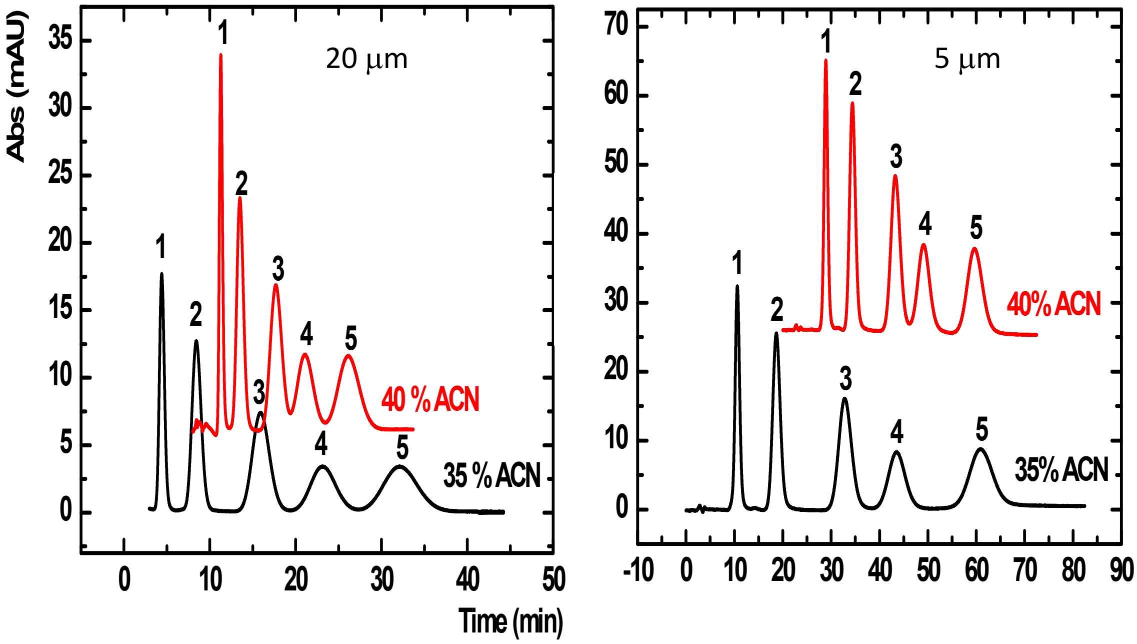


Figure ESI5. Separations of phenone mixture upon the SS printed column packed with 5 and 20 μm ODS particles with either 35 or 40% ACN containing mobile phases (25 μL/min).

Loading capacity – ODS packed SS coiled column

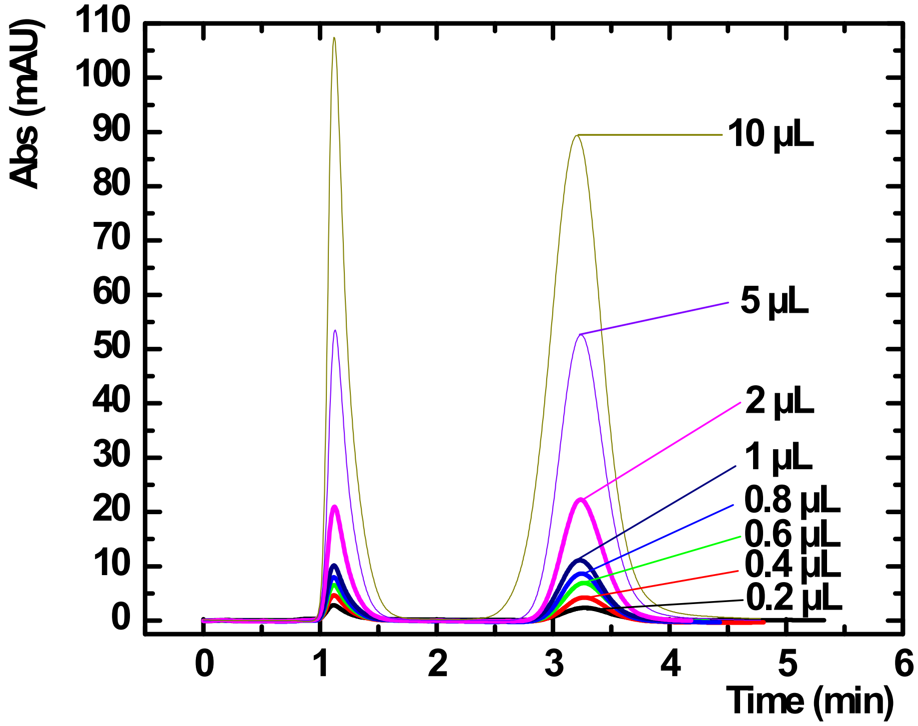


Figure ESI6. Loading capacity (injection volume) for ODS packed SS printed coiled column (Flow rate: 0.3 mL/min, Mobile Phase: 35 % ACN, Temperature: 65 °C, Peaks = uracil and acetophenone.