

Supplementary Information: Perfluoroalkane Wax Infused Gels for Effective, Regenerating, Anti-Icing Surfaces

Arun Sandhu,^a Oliver J. Walker,^a Andres Nistal,^a Kwang Leong Choy,^{*a} and Adam J. Clancy^{*ab}

a) Institute for Materials Discovery, University College London, London WC1E 7JE, U.K.

b) Department of Chemistry, University College London, WC1E 7JE, U.K

Email: k.choy@ucl.ac.uk

a.clancy@ucl.ac.uk

Contents

1. Chemicals	2
2. Additional SEM Micrographs	2
3. Ice Adhesion Testing	3
4. Environmental testing	5
5. Microscopy	6
6. Contact Angle Testing	6

1. Chemicals

Perfluorononane (99%) and perfluorotetradecane (97%) were purchased from Fischer Scientific UK. Nonadecane (99%) was purchased from Alfa Aesar. Perfluorododecane (95%) were purchased from Apollo Scientific Ltd (UK). Heptadecane (99%) and tetracosane (99%) was purchased from Acros Organics. Sylgard 182 (Dow Corning) was purchased via Onecall. Isopropyl alcohol (99%) was purchased from SLS. Deionised water was purified in-house. Glass slides (soda lime glass) were purchased from SLS Ltd. Steel substrates were purchased from Fays Metals (U.K.)

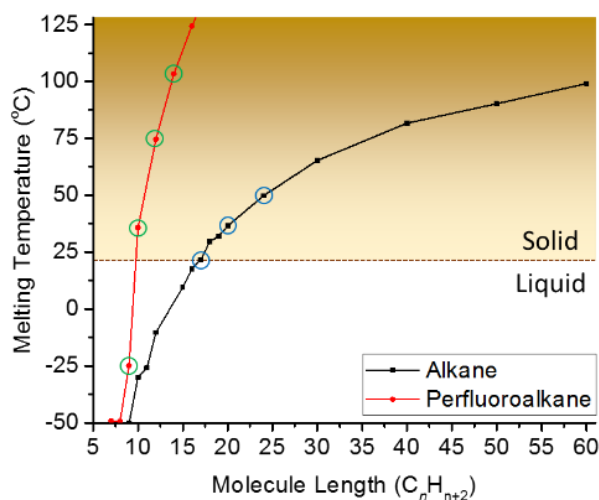


Figure S1. Melting points of linear alkanes and perfluoroalkanes. Values taken from commercial supplier specification sheets. Ringed values indicate chemicals used in this work, dashed line shows room temperature of the laboratory (20 °C).

2. Additional SEM Micrographs

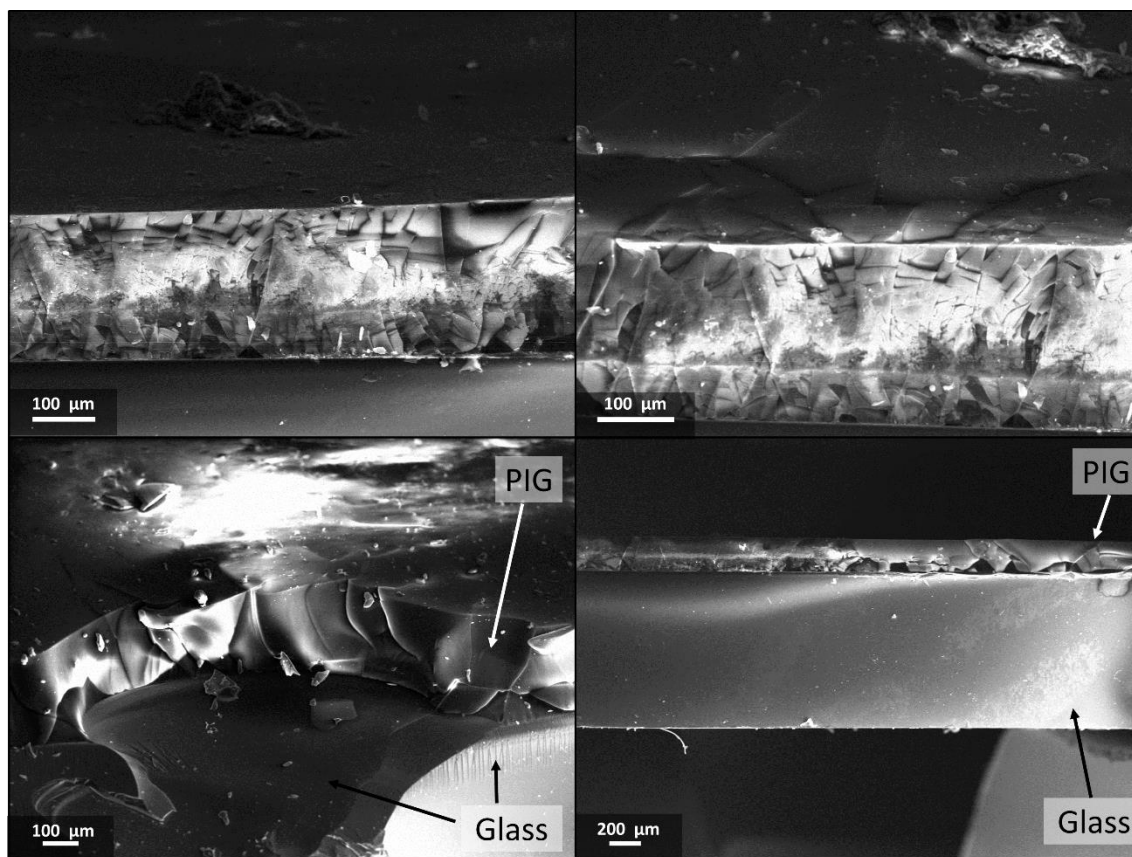


Figure S2. Scanning electron micrographs of C14F PIG cryofracture cross-section. In all images, glass substrate is at the bottom and samples tilted to allow imaging of the top of the infused gel top-surface

3. Ice Adhesion Testing

Ice adhesion was tested using a custom setup. An air cooled thermoelectric cooling stage (Laird Technologies, DA PowerCool Series DA-014-12-02, controlled by a Laird Technologies PWM Programmable PID Controller, PR-59) was placed in a 3D printed (PLA) insulating cage on a stand to improve airflow to the bottom-mounted cooling fan, and adhered to the static frame of a motorized motion stage (“200mm long linear motion stage with 42mm stepper motor” banggood.com). A force transducer (Sauter FK 1K, Force Gauge 1000Hz) was fitted to the stage using a custom 3D printed frame. The test sample was placed on the cooling stage and cooled to $-10\text{ }^{\circ}\text{C}$, and a hollow cuvette (1x1 cm) filled with water placed on the surface and allowed to freeze over 20 min. The transducer on the stage was then moved towards the cuvette at $500\text{ }\mu\text{m s}^{-1}$ (controlled by a Neuftech L298n dual H-bridge DC stepper motor controller drive module) impacting the cuvette 2 mm above the substrate surface. The stage was moved until the ice detached from the surface, with the maximum measured force used to calculate the adhesion strength (strength = Force/ 10^{-4} m^2). Three measurements were taken on separate samples, with the average provided with error bars indicating the standard deviation.

Infused gel samples (PIG and AIG) and unfused PDMS were tested on glass slides as synthesised. Pure wax samples were created from casting melted wax onto steel substrates and cooling. PFAs failed by ice/PFA or PFA cohesive failure (SI Fig. S5a-b), however, as hydrogenated alkane samples failed by steel-wax adhesive failure (SI Fig. S5c-d), additional samples were created from compression of wax into $\sim 4\text{ mm}$ thick, 20 mm diameter disks under 15 tonne of pressure. These disk samples failed by cohesive failure (SI Fig S6).

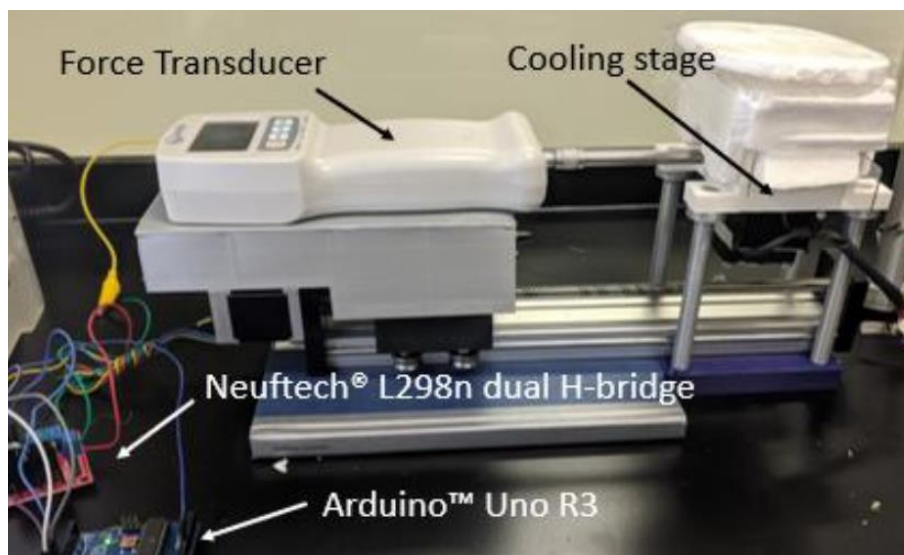
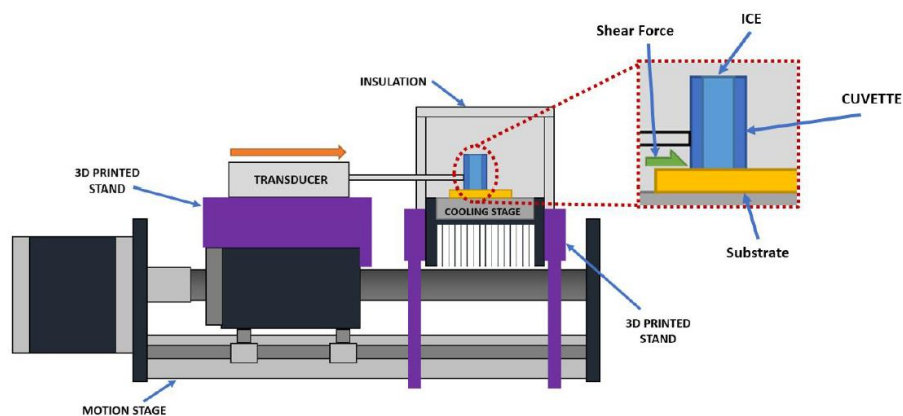


Figure S3. Schematic (top) and digital picture (bottom) of ice adhesion testing setup

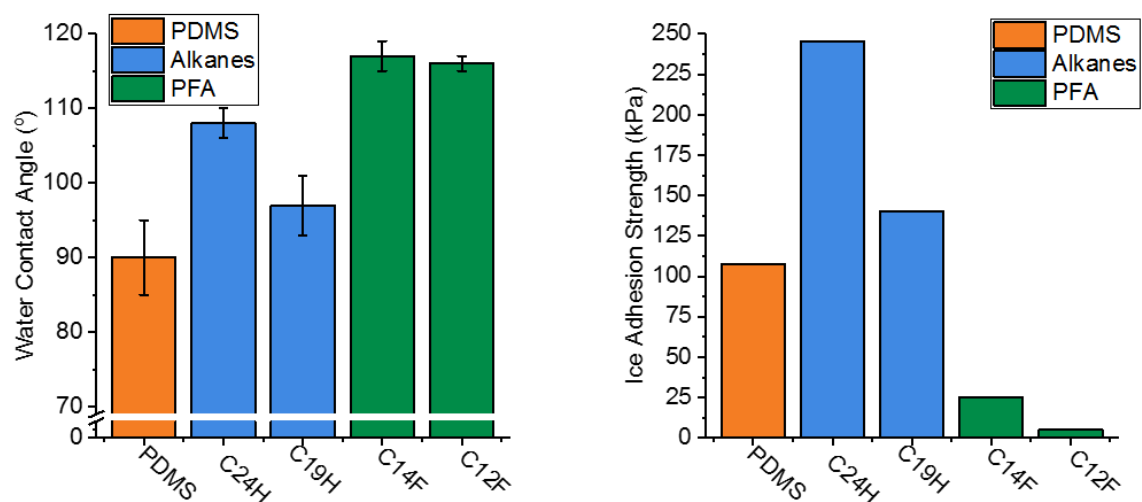


Figure S4. Water Contact Angle (left) and IAS (right) of constituent materials.

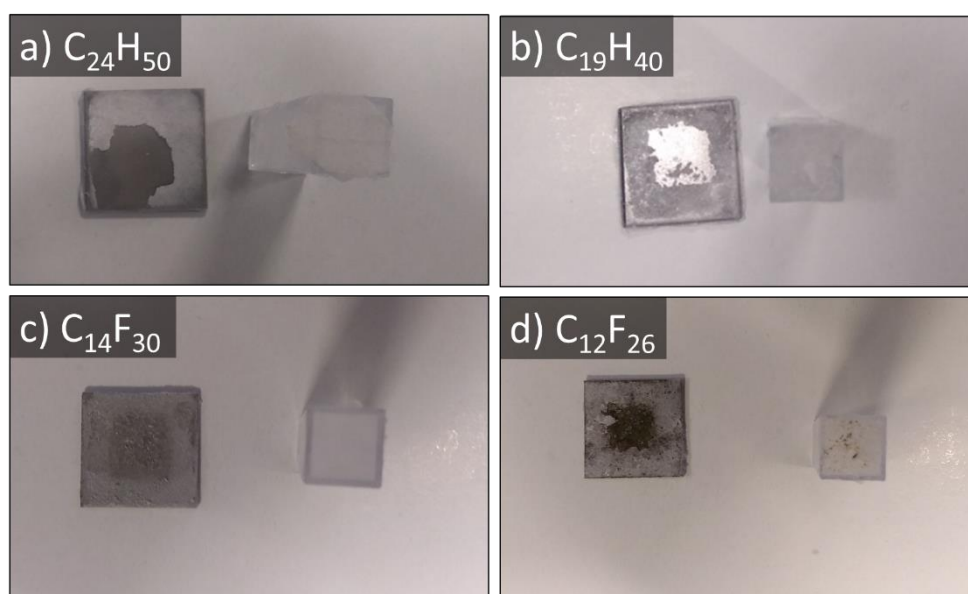


Figure S5. Coated steel (2.5 x 2.5 cm) and cuvettes after ice adhesion tests of pure waxes. Note complete cleavage of alkanes from steel (a,b) due to steel/alkane adhesive failure at 2.5N (~25 kPa assuming 1 cm² area). Perfluoroalkane samples showed alkane on both the steel and the ice. N.B. The white outline seen is due to atmospheric water freezing around the cuvette during the hold.

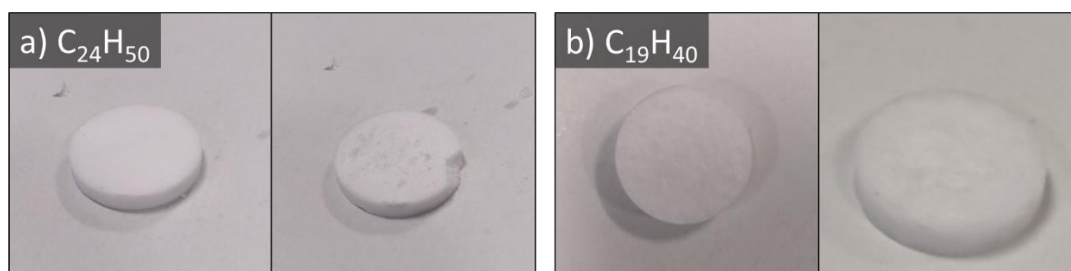


Figure S6. Compressed alkane disks of a) C₂₄H₅₀ and b) C₁₉H₄₀, used for pure-wax ice adhesion tests both before (left) and after (right) testing. The rough surface of the post-testing samples indicates cohesive failure for both samples (i.e. ice/wax adhesive strength > cohesive strength). As the measured cohesive strength (Fig S4) is greater than AIG IAS (manuscript, Fig 3), ice adhesion for AIGs are attributed to PDMS/wax failure not present in the pure-wax disk measurements.

4. Environmental testing.

Using previously established ratios (Kester *et al.*, *Limnology and Oceanography*, 1967, 12, 176), synthetic sea water was created by mixing deionised water (100 mL), sodium chloride (23.93 g), sodium sulphate (4.01 g), and potassium chloride (0.68 g). A 15 × 15 mm organogel was submerged in a stirred bath of 50 mL of synthetic water for 24 h. The sample was then transferred to a fresh batch of synthetic seawater for a further 24 h; the process was repeated for a total of 5 days. The contaminated water was mixed with 10.00 mL of propan-2-ol and heated to 10 °C above the organic melting point to ensure solubility of any displaced or precipitated solid organic and to act as quantitative calibration for gas chromatography-mass spectrometry (GCMS). 1 µL of each sample was injected into a GCMS (Perkin Elmer SQ8 GCMS) with helium flowing at 1sccm at 250 °C For comparison and peak-identification, 1 µg mL⁻¹ solutions of the organics in 10:1 water/isopropyl alcohol were mixed and used as standards unless stated.

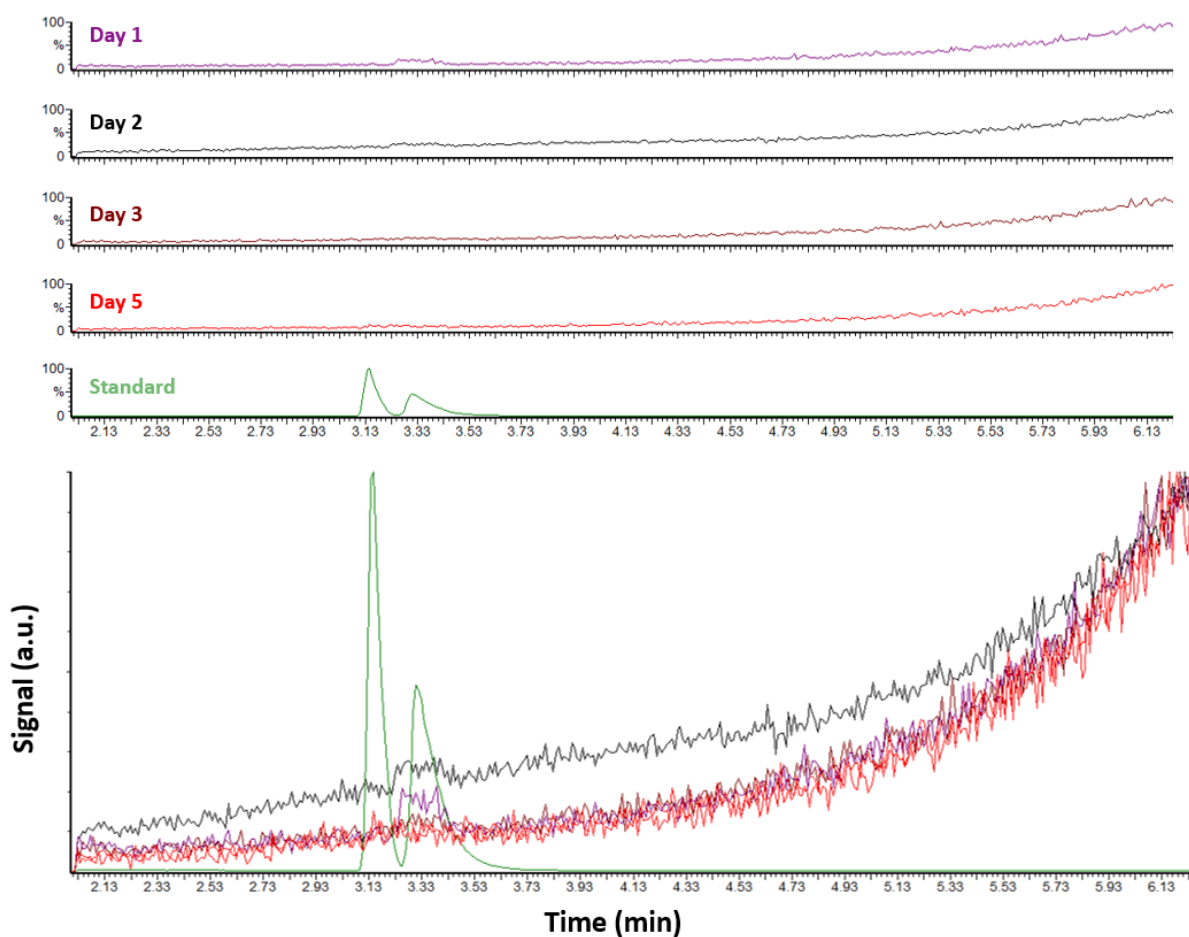


Figure S7. GC-MS (electron ionization mode) signal of aliquots of washing from C₁₉H AIG soaked in water, from top to bottom: Day 1 aliquot (purple); day 2 aliquot (black); day 3 aliquot (brown); day 5 aliquot (red); and C₁₉H₄₀ control (green), higher magnification of all overlaid signals. Deviations from drift on days 1 and 2 were supported by MS signals characteristic of C₁₉H₄₀.

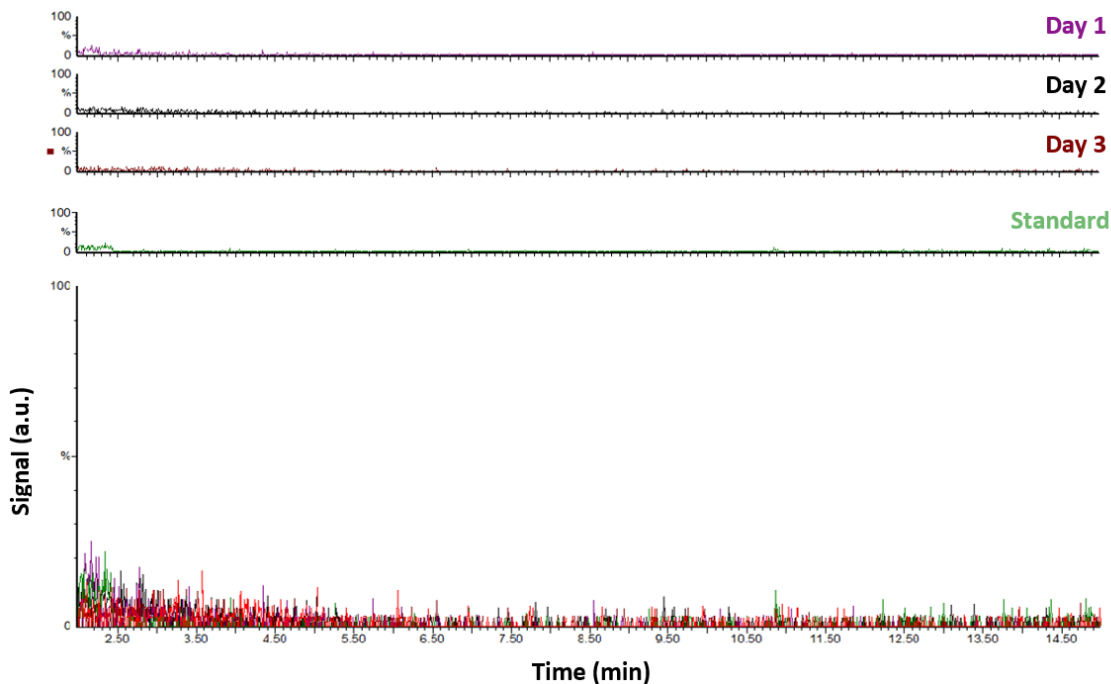


Figure S8. GC-MS (electron ionization mode) signal of aliquots of washing from C14F PIG soaked in water, from top to bottom, Day 1 aliquot (purple), day 2 aliquot (black), day 3 aliquot (red), C₁₄F₃₀ control (green), higher magnification of all overlaid signals. No clear peak can be seen in any samples due to insolubility of perfluoroalkane.

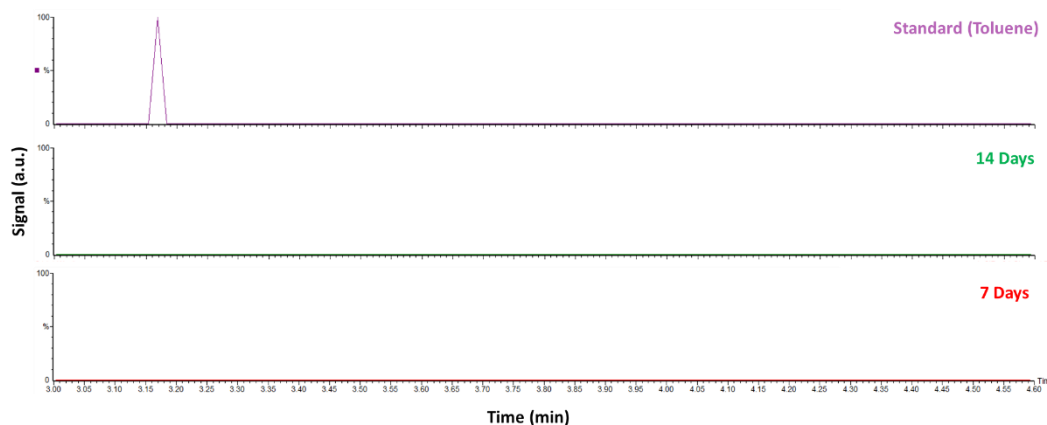


Figure S9. GC-MS signal of aliquots of washing from C14F PIG soaked in water, run in selected ion monitoring (SIM) mode to maximize PFA detection. Standard dissolved in toluene ($1 \mu\text{g mL}^{-1}$) to ensure good signal seen for GC time value.

5. Microscopy

SEM was performed on a Zeiss EVO LS15, with samples mounted to aluminium stubs (Agar Scientific) by carbon tabs (Agar) and sputtered with 10 nm Cobalt. Images were taken with an accelerating voltage of 10 keV. EDS measurements were taken using an Oxford Instruments Xmax20 with an accelerating voltage of 20 keV.

Optical Microscopy was performed on a Zeiss Axio Lab A1 in reflectance mode, fitted with an Axiocam 105.

6. Contact Angle Testing.

Sessile contact angles were measured on a 'Biolin Specific One Attention® Theta Contact Angle Optical Tensiometer' goniometer, dropping $\sim 3 \mu\text{L}$ of deionised water onto a substrate. Baseline and angles were measured automatically, with the average of left and right contact angles used as the measured value. Averages of 5 measurements at different regions of the samples were taken with the standard deviation provided as error bars.