

## Supplementary material

### Characterisation of oil contaminated soils by comprehensive multiphase NMR spectroscopy

Hashim Farooq,<sup>A</sup> Denis Courtier-Murias,<sup>B</sup> Myrna J. Simpson,<sup>A</sup> Werner E. Maas,<sup>C</sup> Michael Fey,<sup>C</sup> Brian Andrew,<sup>C</sup> Jochem Struppe,<sup>C</sup> Howard Hutchins,<sup>C</sup> Sridevi Krishnamurthy,<sup>C</sup> Rajeev Kumar,<sup>D</sup> Martine Monette,<sup>D</sup> Henry J. Stronks,<sup>C</sup> and André J. Simpson<sup>A,E</sup>

<sup>A</sup>Department of Chemistry, University of Toronto, 1265 Military Trail, Toronto, ON, M1C 1A4, Canada.

<sup>B</sup>Universite Paris-Est, Laboratoire Navier (UMR 8205 IFSTTAR-ENPC-CNRS), Physics of Porous Media group, 2 alle Kepler, F-77420 Champs sur Marne, France.

<sup>C</sup>Bruker Biospin Corporation, 15 Fortune Drive, Billerica, MA 01821-3991, USA.

<sup>D</sup>Bruker Ltd. Canada, 555 Steeles Avenue East, Milton, ON, L9T 1Y6, Canada.

<sup>E</sup>Corresponding author. Email: andre.simpson@utoronto.ca

**Table S1. Time constants with associated  $R^2$  values determined from the cross-polarisation build-up curves measured for various soil–D<sub>2</sub>O mixtures shown in Fig. 2 of the main text**

Dry indicates the soil was run ‘as is’ and wet indicates that it was run with field capacity D<sub>2</sub>O (~33 % by weight)

Soil Type	$T_{CH}$ (ms)	$R^2$	Percentage change in $T_{CH}$ upon wetting
Wettable (dry)	0.120	0.97	30.9
Wettable (wet)	0.0829	0.9886	
Non-wettable (dry)	0.0857	0.9356	12.4
Non-wettable (wet)	0.0751	0.8967	

**Table S2. Time constants and associated  $R^2$  values determined from the cross-polarisation build-up curves measured for various regions of wettable (control) soil (dry or wet) shown in Fig. 3 of the main text**

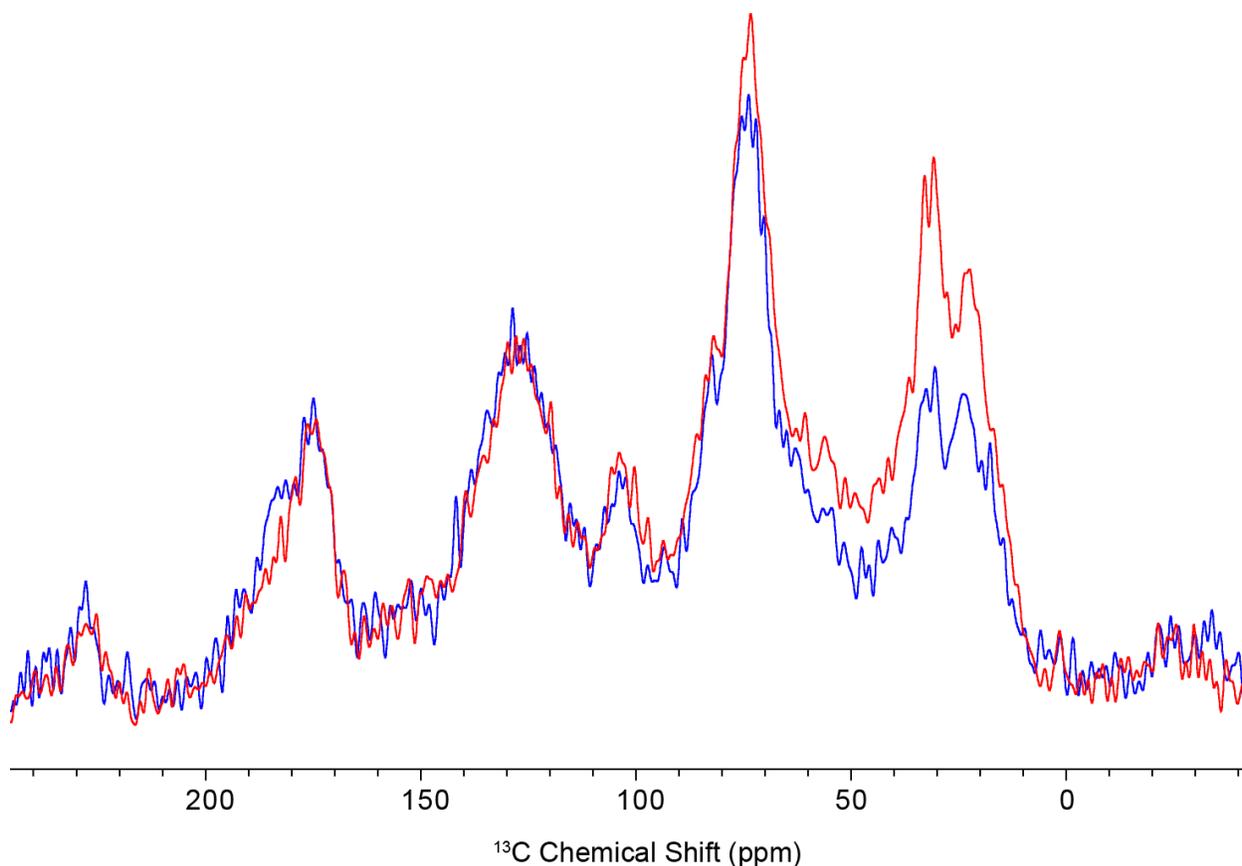
Dry indicates the soil was run ‘as is’ and wet indicates that it was run with field capacity D<sub>2</sub>O (~33 % by weight). The value for the carboxy group could not be determined (CNBT) because of the poor signal to noise ratio for this region after swelling. The poor signal-to-noise after swelling indicates the carbonyl groups interact with the water efficiently. NA, not applicable

Region	Dry soil		Wet soil		Percentage decrease from dry to wet
	$T_{CH}$ (s)	$R^2$	$T_{CH}$ (s)	$R^2$	
Aliphatic	$7.97 \times 10^{-4}$	0.927	$7.12 \times 10^{-5}$	0.984	91.07
O-alkyl	$8.37 \times 10^{-4}$	0.920	$4.39 \times 10^{-5}$	0.996	94.76
Aromatic	$1.07 \times 10^{-3}$	0.908	$5.16 \times 10^{-5}$	0.885	95.16

**Table S3.** Time constants and associated  $R^2$  values determined from the cross-polarisation build-up curves measured for various regions of non-wettable (contaminated) soil (dry or wet) shown in Fig. 3 of the main text

Dry indicates the soil was run 'as is' and wet indicates that it was run with field capacity  $D_2O$  (~33 % by weight)

Region	Dry soil		Wet soil		Percentage decrease from dry to wet
	$T_{CH}$ (s)	$R^2$	$T_{CH}$ (s)	$R^2$	
Aliphatic	$8.91 \times 10^{-5}$	0.955	$2.92 \times 10^{-5}$	0.989	67.23
O-alkyl	$4.29 \times 10^{-5}$	0.958	$2.68 \times 10^{-9}$	0.989	99.99
Aromatic	$4.21 \times 10^{-5}$	0.946	$1.76 \times 10^{-11}$	0.940	99.99
Carboxy	$6.57 \times 10^{-5}$	0.921	$1.60 \times 10^{-8}$	0.846	99.98



**Fig. S1.**  $^{13}C$  spectra of the wettable (blue) and non-wettable (red) soil shown with an expanded spectroscopic region in the dry state. Spinning side bands (SSBs) arising from the aliphatic and the carboxy regions are seen between below 0 ppm and above 200 ppm, respectively. We can see that the SSBs in both samples for both regions are within error of each other.