

SUPPORTING INFORMATION

Hierarchical macroporous-mesoporous SBA-15 sulfonic acid catalysts for biodiesel synthesis

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1. Characterisation of PS beads

SEM of the PS beads (**Figure S1**), reveals a narrow size distribution with an average diameter of ~320 nm (**Figure S2**). Thermal analysis shows that complete decomposition/combustion of the beads, and thus template removal, can be achieved at 550 °C (**Figure S3**).

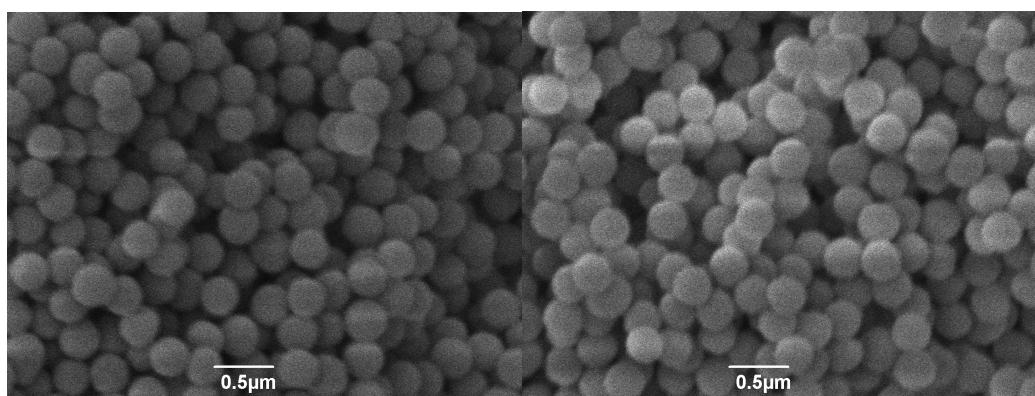


Figure S1. SEM images of 320 nm polystyrene beads.

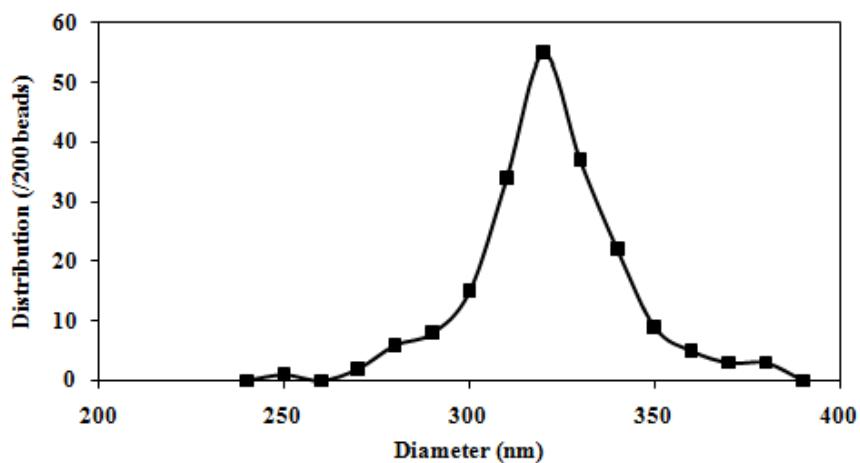


Figure S2. Particle size distribution of PS beads.

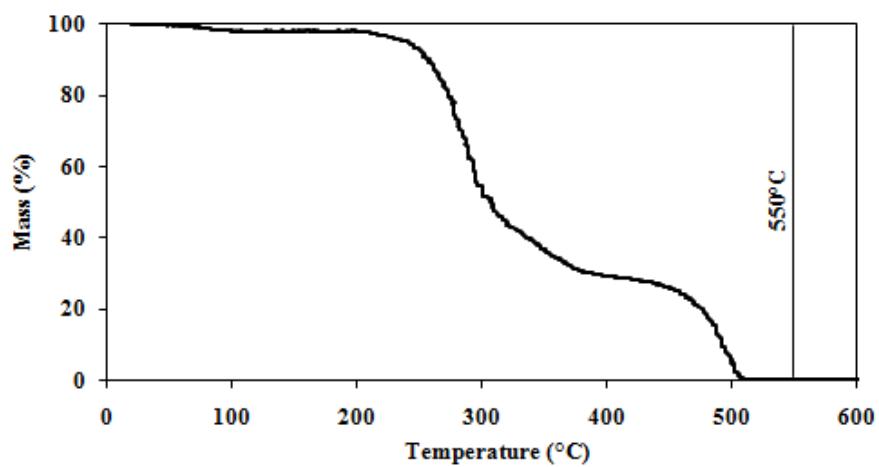


Figure S3. TGA of PS beads.

2. Characterisation of macroporous-mesporous silicas

HRTEM images of as-prepared SBA-15 and MM-SBA15-4 are shown in **Figure S4**.

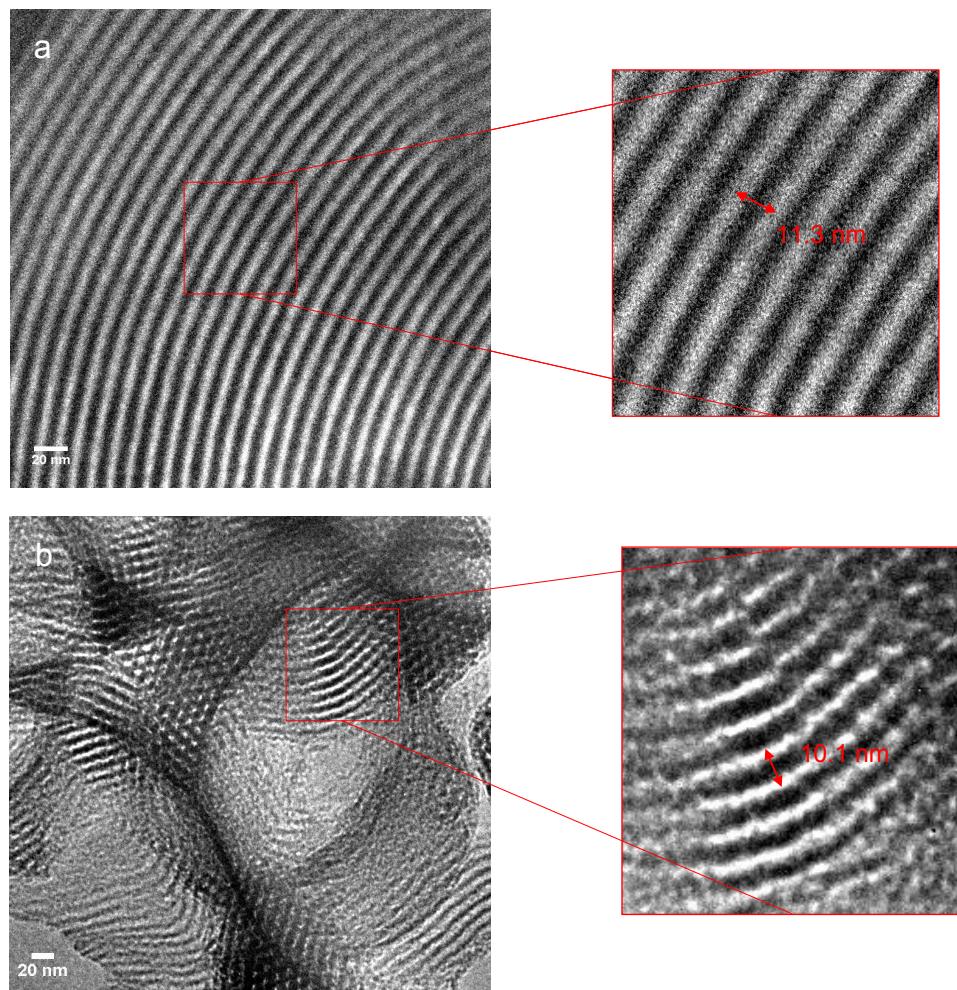


Figure S4. HRTEM micrographs of (a) SBA-15 and (b) MM-SBA15-4.

3. Functionalization of macroporous-mesoporous silicas

Fitted S 2p_{1/2} (blue) and S 2p_{3/2} (red) XP spectra of sulfonic acid functionalized materials are shown in **Figure S5**.

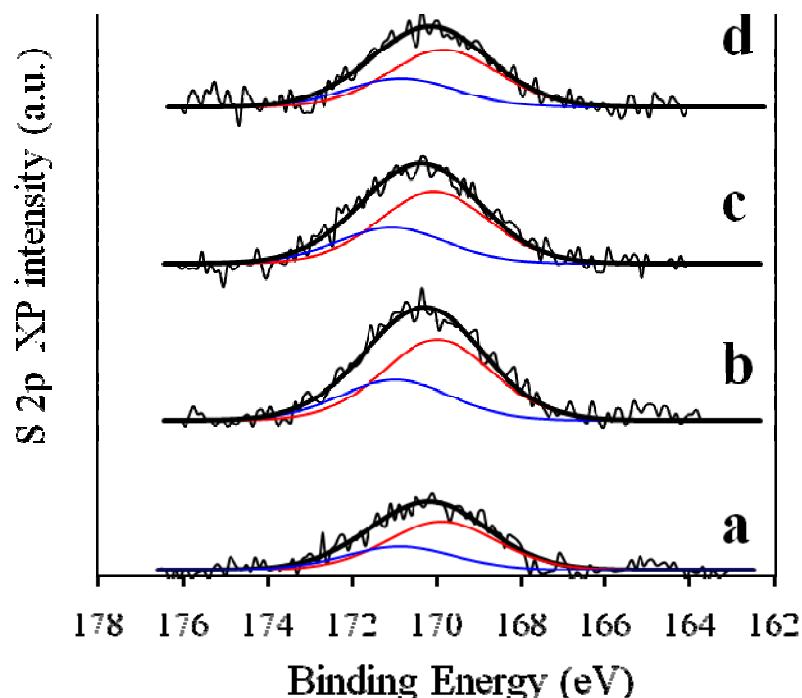


Figure S5. S 2p XPS of sulfonic acid functionalised (a) SBA-15, (b) MM-SBA15-1, (c) MM-SBA15-2 and (d) MM-SBA15-4.

Table S1. Carbon content of pre- and post-sulfonic acid functionalized macroporous-mesoporous silicas

Sample	Fresh C content ^a / wt%	Functionalised C ^b content / wt%
Meso SBA-15	1.28	4.92
MM-SBA-15-1	2.11	4.32
MM-SBA-15 -2	2.73	3.69
MM-SBA-15-4	3.78	3.59

^aICP analysis. ^bXPS analysis.

Porosimetry (**Figure S6a-b**) and low angle XRD (**Figure S7**) of as-prepared and sulphonated macroporous-mesoporous SBA-15 silicas shows preservation of mesopore structure.

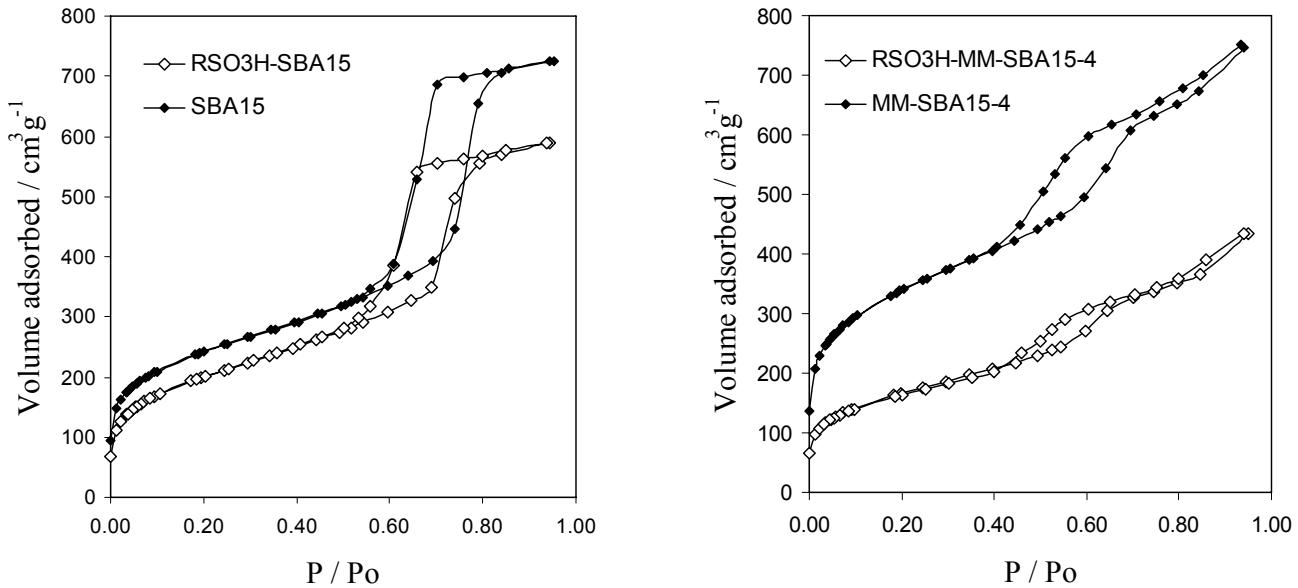


Figure S6a. Adsorption-desorption isotherms of SBA-15 and MM-SBA15-4 before and after functionalisation with sulfonic acid groups.

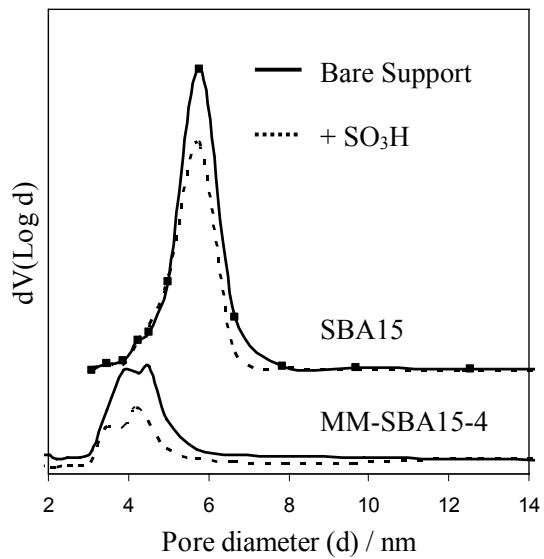


Figure S6b. BJH pore size distributions for SBA-15 and MM-SBA15-4 before and after functionalisation with sulfonic acid groups.

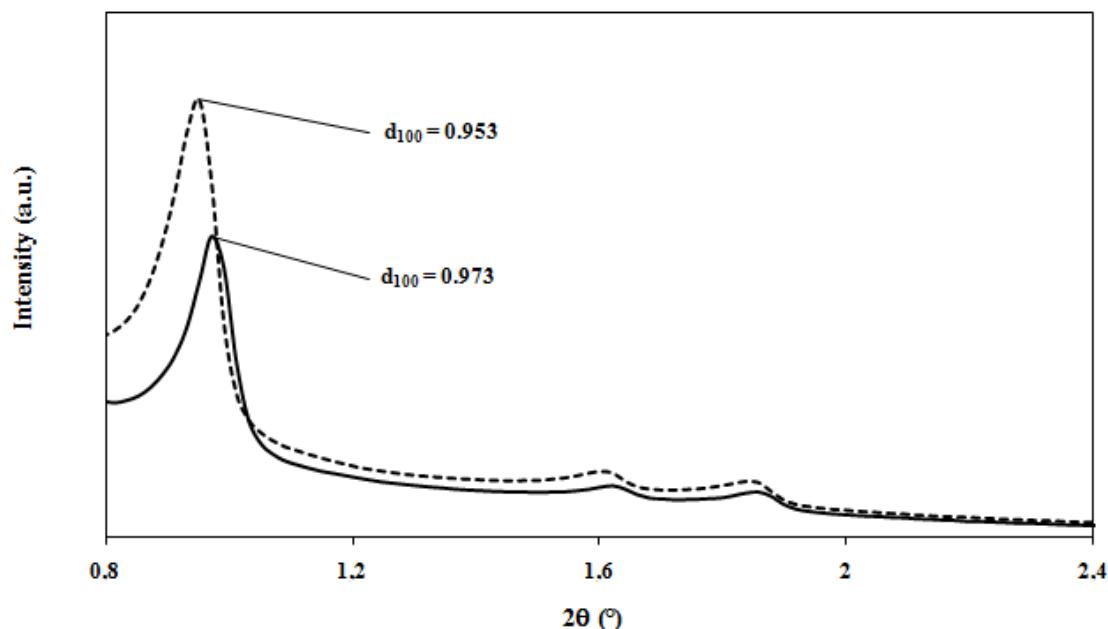


Figure S7. Low angle XRD of mesoporous SBA-15 before (---) and after functionalization (—).

Table S2. Textural properties of sulfonic acid functionalized macroporous-mesoporous silicas

Sample	Mesopore diameter ^a /nm	Mesopore volume ^{a,b} /cm ³ g ⁻¹
SBA-15	5.8	1.08 (1.18)
RSO ₃ H-SBA-15	5.7	0.81 (0.91)
MM-SBA15-1	5.0	0.82 (1.07)
RSO ₃ H-MM-SBA15-1	5.0	0.62 (0.71)
MM-SBA15-2	4.5	0.87 (0.99)
RSO ₃ H-MM-SBA15-2	4.4	0.54 (0.65)
MM-SBA15-4	3.9	0.84 (1.15)
RSO ₃ H-MM-SBA15-4	4.1	0.56 (0.67)

^aBHJ mesopore volumes and diameters from the desorption isotherm. ^bTotal pore volume shown in brackets.