

Supplementary Information

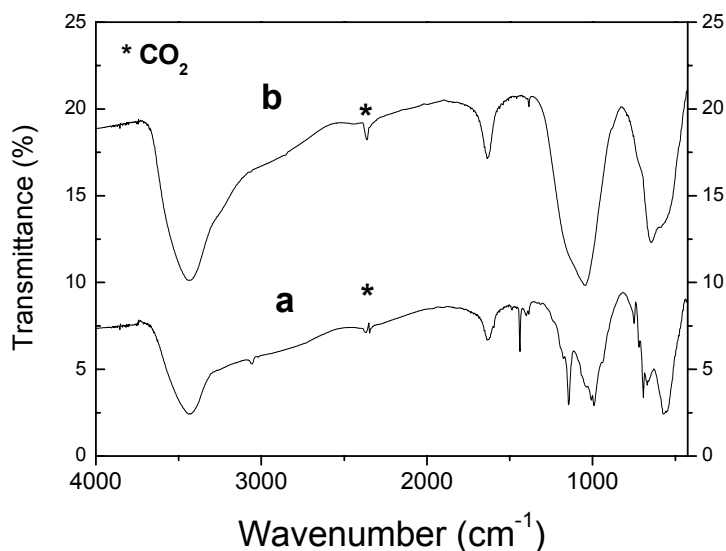


Figure S1. FT-IR spectra of surfactant extracted as-synthesized mesoporous hybrid tin oxophosphates before (a) and after calcination at 823 K (b).

In the above figure, FT-IR spectra of surfactant extracted mesoporous hybrid tin oxophenylphosphate and calcined tin oxophosphate samples are shown. These three bands, 696 and 750 (out of plane), and 728 cm^{-1} serve as an identity of mono-substituted phenyl ring in tin phenylphosphate.^[Ref] Other characteristic bands for phenyl group is at 1437 [$\nu(\text{C}=\text{C})$ aromatic], 3056 cm^{-1} [$\nu(\text{C}-\text{H})$ aromatic], and at 1156, 1145, 1085, 1044, 1011 and 994 cm^{-1} [$\nu(\text{P}-\text{O})$].^[Ref] After calcination, present peaks are at 649 (Sn-O-Sn), 1045 (Sn-O-P), 1636 (H-O-H) and 3440 cm^{-1} (O-H).

[Ref] Mal, N. K.; Fujiwara, M.; Yamada, M.; Kuraoka, K.; Matsukata, M. *J. Ceramic Soc. Japan, Ceramic Lett.* **2003**, *111*, 219, and references are therein.

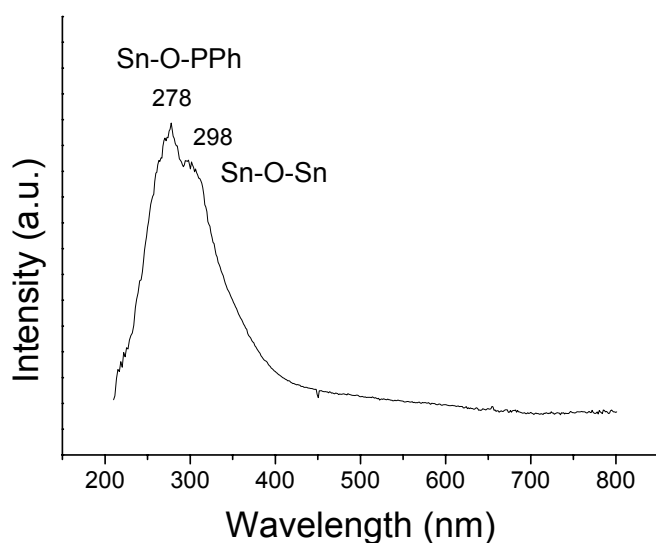


Figure S2. UV-VIS spectrum of mesoporous hybrid tin oxophenylphosphate.

In the above figure, UV-Vis Spectrum of hybrid mesoporous tin oxophenylphosphate is shown. It shows two bands at 278 and 298 nm assigned to octahedral coordination of Sn as Sn-O-PC₆H₅ and Sn-O-Sn, respectively. No band is present at 220 nm confirming the absence of tetrahedral coordinated tin species.^{4f} In case of mesoporous tin phosphate and tin oxide, tetra coordinated Sn-O-P band is present at 220 nm and octahedral coordinated Sn-O-Sn band is present at 303 nm. In case of microporous tin silicate octahedral tin species shows band at 285 to 300 nm.⁸

The determination of molecular formula of materials by comprehensive evaluation using the results of elemental analysis and ion-exchange capacity.

By the elemental analyses (wt%) of Sn, P, S, Na, C and H elements in the samples, we the molecular formulas were deduce. The weights of oxygen were calculated by hundred (100) minus weight of all these elements. In addition, the ion-exchange capacity measurement for Na⁺ ions was applied to confirm its presence outside the framework. For example, in case of mesoporous hybrid tin oxophenylphosphate, the observed wt% of Sn, P, C, H and Na found to be 40.71, 7.69, 17.88, 1.24 and 10.06 wt%, respectively. Rest amount (22.42 wt%) was considered as weight of oxygen. When these values of wt% were divided by their respective atomic weights and normalized (Sn + P = 1), the observed formula was Sn_{0.58}P_{0.42}C_{2.52}O_{2.37}H_{2.1}Na_{0.74}. Which was re-arranged to deduce a comprehensive molecular formula, [(SnO₃)_{0.37} Sn_{0.21}(O₃PC₆H₅)_{0.42}]_{0.74}Na. In this case sodium ion exchange capacity observed was 0.115 g Na per g sample (5.00 mmol/g), which is equivalent to 0.86 Na in proposed formula, but we took the elemental analysis result. In the case of calcined tin oxophosphate observed (experimental) weights for each elements, Sn, P and Na atoms were found to be 49.12, 9.28 and 12.14 wt%, respectively. Rest 29.46 wt% was considered as weight of oxygen. The finally deduce formula was Sn_{0.58}P_{0.42}O_{2.58}Na_{0.74}. Which was re-arranged to deduce a comprehensive molecular formula, [(SnO₃)_{0.30} Sn_{0.28}(O₄P)_{0.42}]_{0.74}Na. When the sodium ion exchange capacity was independently measured, the value observed was 0.133 g Na per g sample (5.79 mmol/g), which is equivalent to 0.86 Na in proposed formula. However, we took the elemental analysis results in reported ones. This ion exchange capacity measurement confirms that all the Na⁺ ions is present outside the framework.. Similarly, Absence of sulfur in our surfactant extracted sample also confirms that all the surfactant was removed during extraction processes.

Determination of acid sites (Titration curve)

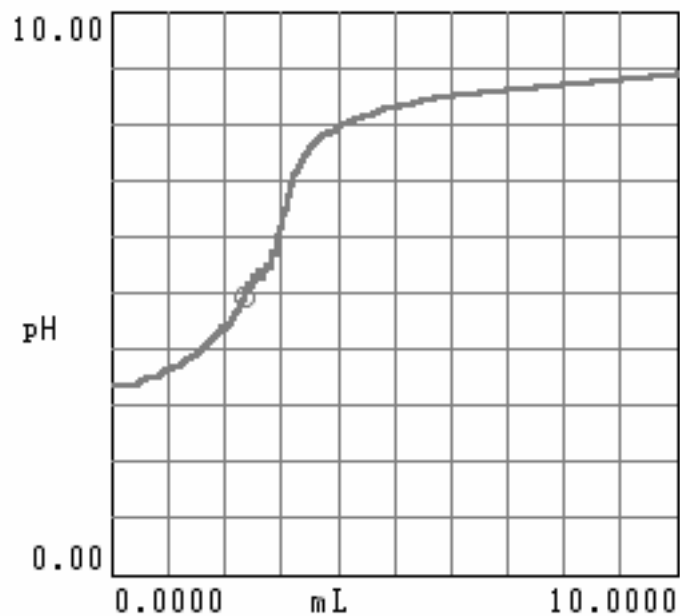


Figure S3: Titration of 20 mg of sulfonated hybrid mesoporous Sn oxophenylphosphate sample in 50 g of H₂O against 0.01 M NaOH solution at 25 °C.

Analytical Procedures

Elemental analyses of Sn, P and Na of samples were carried out using ICP (Shimadzu ICPV-1017). C, H and N analyses were also done. Cation exchange capacity of the mesoporous hybrid and calcined tin oxophosphate was measured by using ICP (Shimadzu ICPV-1017). The samples were characterized using XRD (Cu-K α radiation, λ = 0.15406 nm, Shimadzu XRD-6000), TEM (Hitachi H-9000NA, 300 kV), and N₂ sorption at 77 K using a Bellsorp 28 instrument. Prior to N₂ adsorption, samples were degassed for 2 h at 353 K. For the Fourier transform infrared (FT IR) measurement, a NICOLET MAGNA IR 750 was used. ¹H-¹³C CP/MAS NMR and ³¹P MAS NMR spectra were recorded on a (JEOL CMX-400) machine at 100.54 MHz for ¹H-¹³C and 161.84 MHz for ³¹P with a spinning rate of 8 kHz, pulse time (P1) of 3.0 μ s and a repetition time (D1) of 30 s for ³¹P NMR and 5.0 s for ¹H-¹³C CP/MAS NMR. The total numbers of scan were 1248 times for ¹H-¹³C NMR and 16 times for ³¹P NMR. Chemical shifts for ¹³C and ³¹P were measured with reference to hexamethylbenzene and triphenylphosphine, respectively.