

Synthesis and application of fatty acid derived templates for the preparation of mesostructured silica material

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S1. Imidazole-Fatty acid synthesis

A mixture of FAME (1eq), 1-(3-aminopropyl)imidazole (1.5eq) and TBD (2mol%) was stirred in a round-bottom flask at 120°C under reduced pressure (10mbar) for 5h. After completion of the reaction, the excess amine was removed by washing the product with water and subsequently dried under vacuum. The FAME imidazole amides were obtained as viscous liquid products. Products have been analysed by ¹H NMR in CDCl₃.

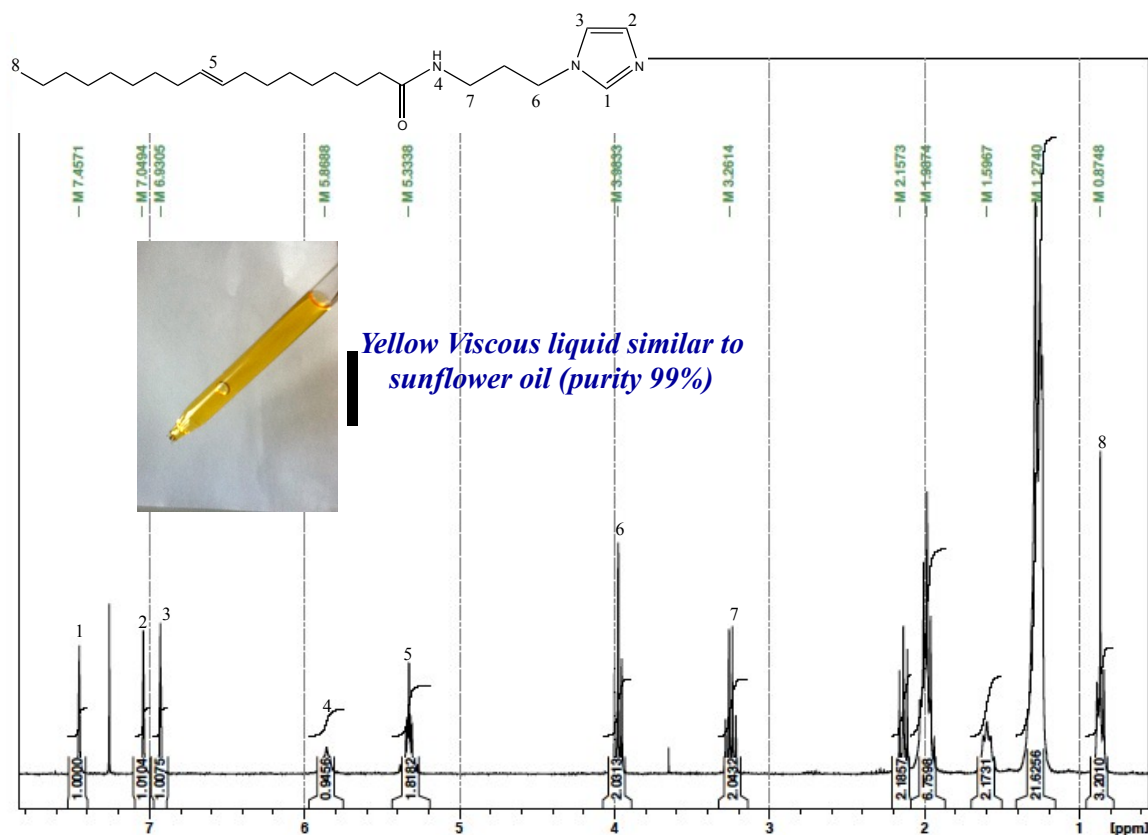


Fig.S1. ^1H NMR of Fatty Oleic Imidazole

- Very High Oleic Sunflower (VHOSO) imidazole amide

Conversion in amide product measured by GC = 94%

$^1\text{H-NMR}$ (400 MHz, CDCl_3 , d): 0.87 (m, $-\text{CH}_2-\text{CH}_3$), 1.27 (m, $-\text{[CH}_2\text{]-}$), 1.6 (m, $-\text{CH}_2-\text{CH}_2-\text{NHOC-}$), 1.99 (m, $-\text{CH}_2-\text{CH=}$), 2.16 (t, $-\text{CH}_2-\text{CONH-}$), 3.26 (q, $-\text{CH}_2-\text{NHCO-}$), 3.98 (t, $-\text{CH}_2-\text{N}<$), 5.33 (m, $-\text{CH=CH-}$), 5.87 (m, $-\text{NH-CO-}$), 6.93 (s, $-\text{CH=CH-N}<$), 7.05 (s, $=\text{N-CH=CH-}$), 7.46 (s, $-\text{N=CH-N}<$)

- Linseed imidazole amide

Conversion in amide product measured by GC = 93%

$^1\text{H-NMR}$ (400 MHz, CDCl_3 , d): 0.87 (m, $-\text{CH}_2-\text{CH}_3$), 0.95 (m, $=\text{CH-CH}_2-\text{CH}_3$), 1.30 (m, $-\text{[CH}_2\text{]-}$), 1.56 (m, $-\text{CH}_2-\text{CH}_2-\text{NHOC-}$), 1.90 (m, $-\text{CH}_2-\text{CH=}$), 2.10 (t, $-\text{CH}_2-\text{CONH-}$), 2.80 (m, $=\text{CH-CH}_2-\text{CH=}$), 3.20 (q, $-\text{CH}_2-\text{NHCO-}$), 4.00 (t, $-\text{CH}_2-\text{N}<$), 5.20 (m, $-\text{CH=CH-}$), 5.50-6.50 (m, $-\text{CH=CH-CH}_2-\text{CH=CH-}$), 6.00 (m, $-\text{NH-CO-}$), 6.90 (s, $-\text{CH=CH-N}<$), 7.10 (s, $=\text{N-CH=CH-}$), 7.50 (s, $-\text{N=CH-N}<$)

- Castor oil imidazole amide

Conversion in amide product measured by GC = 95%

$^1\text{H-NMR}$ (400 MHz, CDCl_3 , d): 0.87 (m, $-\text{CH}_2-\text{CH}_3$), 1.30 (m, $-\text{[CH}_2\text{]-}$), 1.56 (m, $-\text{CH}_2-\text{CH}_2-\text{NHOC-}$), 1.90 (m, $-\text{CH}_2-\text{CH=}$), 2.10 (t, $-\text{CH}_2-\text{CONH-}$), 2.20 (m, $-\text{CH}_2-\text{CH=}$), 3.20 (q, $-\text{CH}_2-\text{NHCO-}$), 3.60 (m, $-\text{CH}_2-\text{CH-OH}$), 4.00 (t, $-\text{CH}_2-\text{N}<$), 5.30-5.60 (m, $-\text{CH}_2-\text{CH=CH-CH}_2-$), 6.00 (m, $-\text{NH-CO-}$), 6.90 (s, $-\text{CH=CH-N}<$), 7.10 (s, $=\text{N-CH=CH-}$), 7.50 (s, $-\text{N=CH-N}<$)

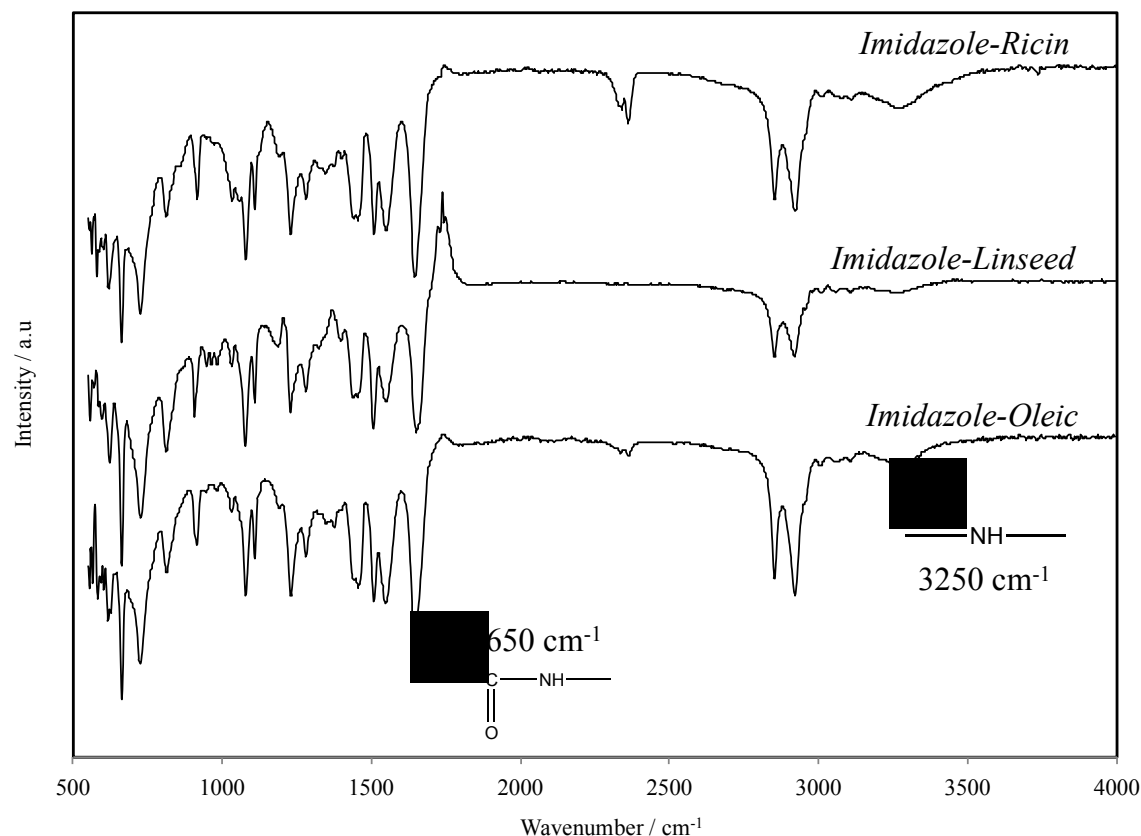


Fig. S2. Infrared spectra of fatty imidazole synthesized

S2. Bio-Organic Salt (BOS) synthesis

3 or 4 g of fatty imidazole are introduced in Teflon autoclave (25ml). Large excess chloropropane is added to ensure complete dissolution of the fatty imidazole. The solution is stirred at 80°C for 24h. After cooling down the solution. The excess of chloropropane is removed by evaporation. Products have been analysed by $^1\text{H NMR}$ in CDCl_3 and show complete conversion of Fatty-imidazole to imidazolium salt. After, evaporation of the excess of chloropropane, the products showed high purity.

Very High Oleic Sunflower imidazolium salt (BOS-Oleic)

$^1\text{H-NMR}$ (400 MHz, CDCl_3 , d): 0.87 (m, $-\text{CH}_2-\text{CH}_3$), 0.97 (t, $-\text{CH}_2-\text{CH}_3$), 1.26 (m, $-\text{[CH}_2\text{]-}$), 1.58 (m, $-\text{CH}_2-\text{CH}_2-\text{NHOC-}$), 1.95 (m, $-\text{CH}_2-\text{CH=}$), 2.15 (t, $-\text{CH}_2-\text{CONH-}$), 3.26 (q, $-\text{CH}_2-\text{NHCO-}$), 4.22 (t, $-\text{CH}_2-\text{N}<$), 4.42 (s, $-\text{CH}_2-\text{CH}_2-\text{N}<$), 5.29 (m, $-\text{CH=CH-}$), 7.27 (s, $-\text{CH=CH-N}<$), 7.66 (s, $=\text{N-CH=CH-}$), 8.28 (m, $-\text{NH-CO-}$), 10.35 (s, $-\text{N=CH-N}<$)

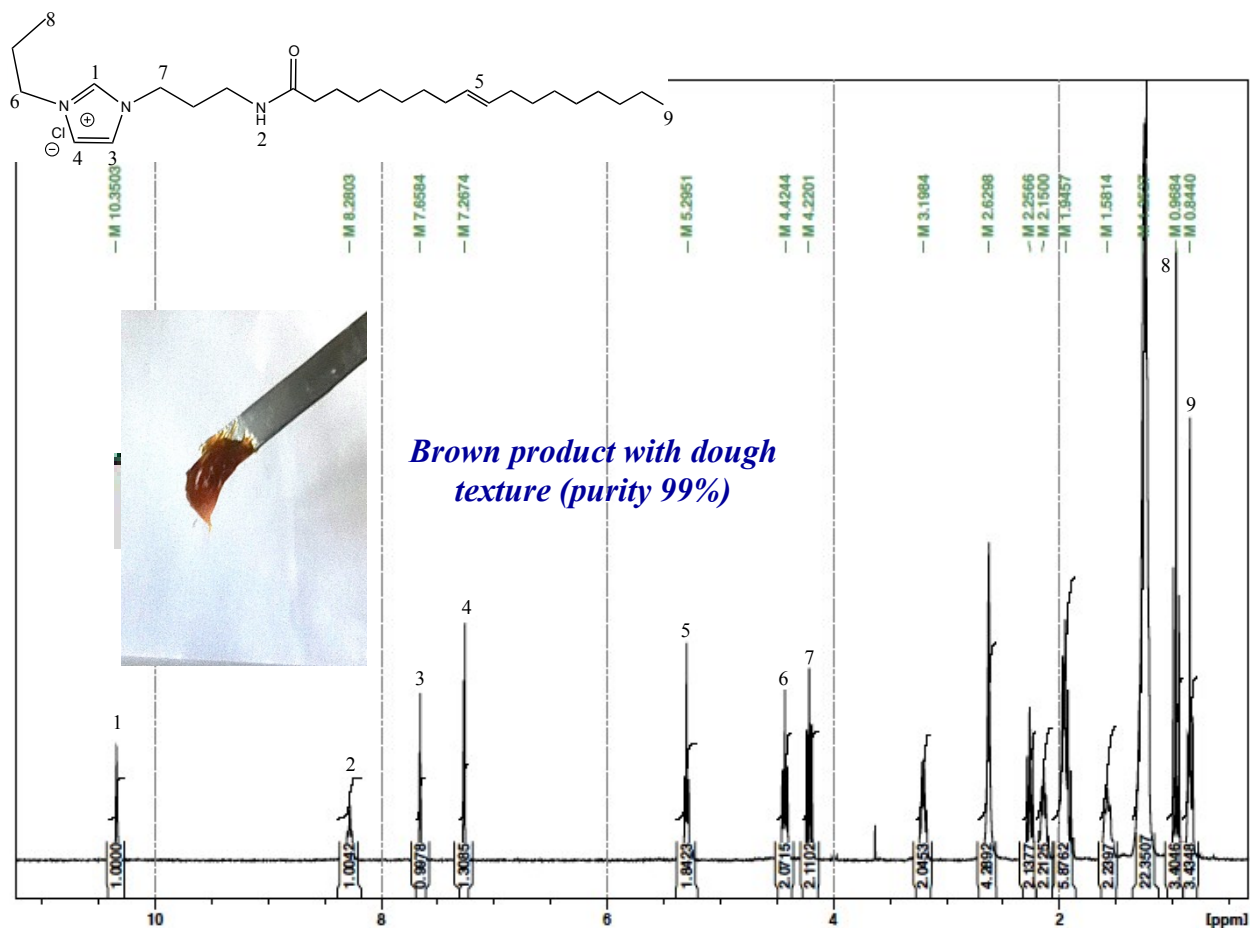


Fig.S3. ^1H NMR of BOS-Oleic

Linseed imidazolium salt (BOS-Linseed)

$^1\text{H-NMR}$ (400 MHz, CDCl_3 , d): 0.85 (m, $-\text{CH}_2-\text{CH}_3$), 0.97 (m, $-\text{CH}_2-\text{CH}_3$ / $=\text{CH}-\text{CH}_2-\text{CH}_3$), 1.27 (m, $-\text{[CH}_2\text{]-}$), 1.58 (m, $-\text{CH}_2-\text{CH}_2-\text{NHOC-}$), 1.95 (m, $-\text{CH}_2-\text{CH=}$), 2.10 (t, $-\text{CH}_2-\text{CONH-}$), 2.77 (m, $=\text{CH}-\text{CH}_2-\text{CH=}$), 3.18 (q, $-\text{CH}_2-\text{NHCO-}$), 4.2 (t, $-\text{CH}_2-\text{N}<$), 4.42 (s, $-\text{CH}_2-\text{CH}_2-\text{N}<$), 5.30 (m, $-\text{CH=CH-}$), 5.50-6.50 (m, $-\text{CH=CH-CH}_2-\text{CH=CH-}$), 7.28 (s, $-\text{CH=CH-N}<$), 7.66 (s, $=\text{N-CH=CH-}$), 8.47 (m, $-\text{NH-CO-}$), 10.56 (s, $-\text{N=CH-N}<$)

Castor oil imidazolium salt (BOS-Ricin)

$^1\text{H-NMR}$ (400 MHz, CDCl_3 , d): 0.84 (m, $-\text{CH}_2-\text{CH}_3$), 0.96 (m, $-\text{CH}_2-\text{CH}_3$), 1.28 (m, $-\text{[CH}_2\text{]-}$), 1.43 (m, $-\text{CH}_2-\text{CH}_2-\text{NHOC-}$), 1.57 (m, $-\text{CH}_2-\text{CH=}$), 1.94 (t, $-\text{CH}_2-\text{CONH-}$), 2.26 (m, $-\text{CH}_2-\text{CH=}$), 3.17 (q, $-\text{CH}_2-\text{NHCO-}$), 3.57 (m, $-\text{CH}_2-\text{CH-OH}$), 4.21 (t, $-\text{CH}_2-\text{N}<$), 4.18 (s, $-\text{CH}_2-\text{CH}_2-\text{N}<$), 5.42 (m, $-\text{CH}_2-\text{CH=CH-CH}_2-$), 7.29 (s, $-\text{CH=CH-N}<$), 7.68 (s, $=\text{N-CH=CH-}$), 8.42 (m, $-\text{NH-CO-}$), 10.35 (s, $-\text{N=CH-N}<$)

S3. Silica material synthesis using BOS as biotemplate

In a Teflon autoclave, Xg of Bio-Organic Salt was dissolved in 17.4ml of distillate water. Then, after complete dissolution of the template, 2.6 ml of NH_3 (26wt%) and 2.2 ml of TetraEthoxyOrthoSilica (TEOS, silica precursor) was added in the solution. The mixture is stirred for 24h at room temperature. The materials were filtered and dried at room temperature for one night. All the materials were calcined at $550^\circ\text{C} - 1^\circ\text{C}/\text{min} - 6\text{h}$.

S3.1. Characterization

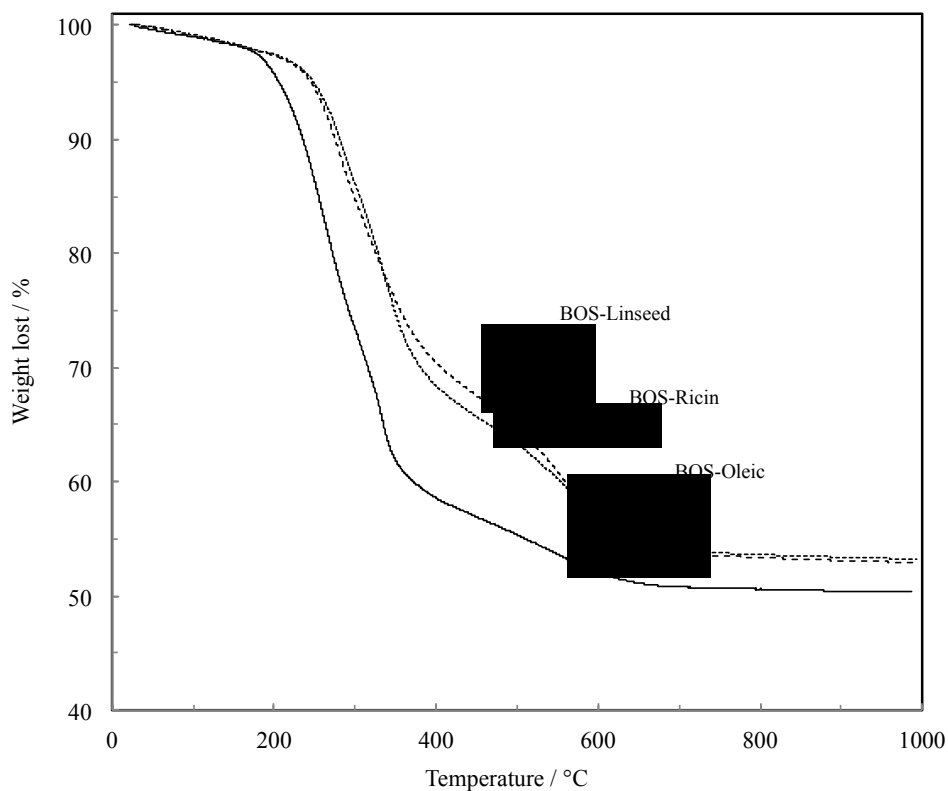


Fig.S4. Thermogravimetry analysis of Meso-BOS silica materials uncalcined

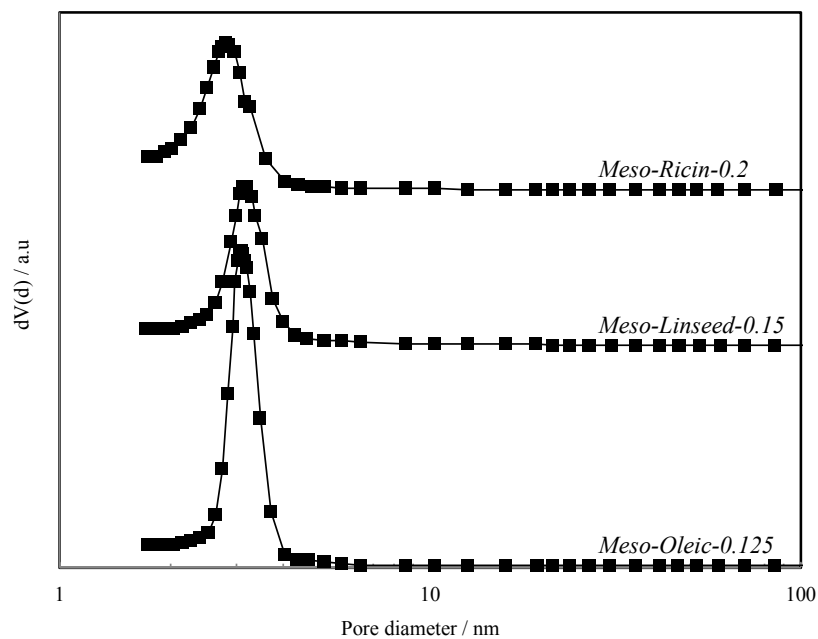


Fig. S5. Pore size distribution of Meso-BOS-X from adsorption branch

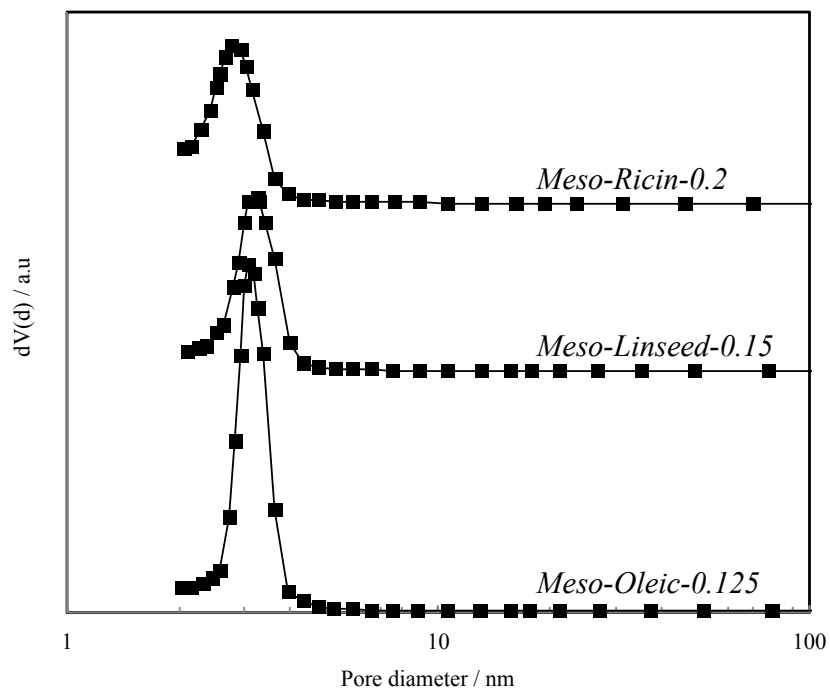


Fig. S6. Pore size distribution of Meso-BOS-X from desorption branch

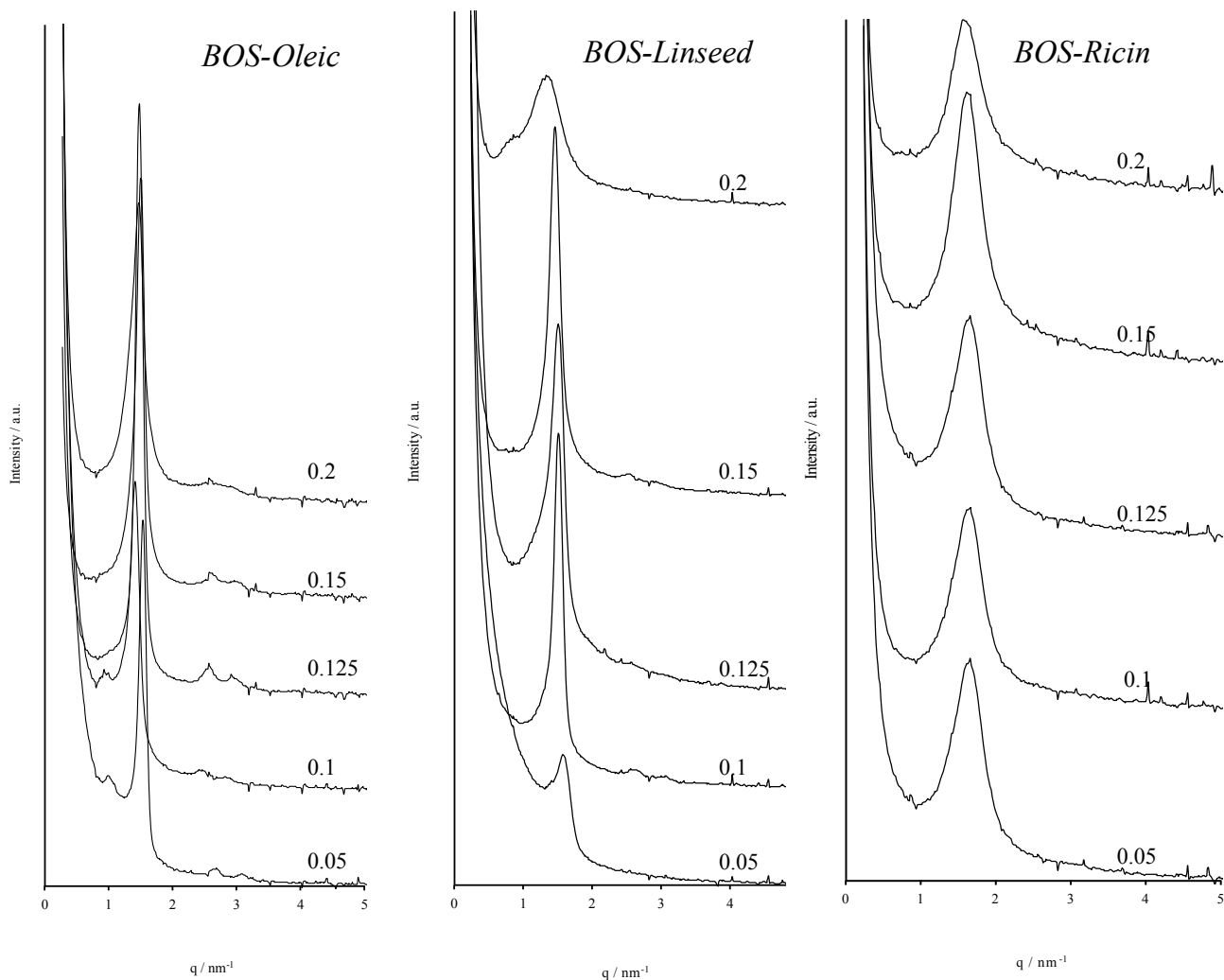
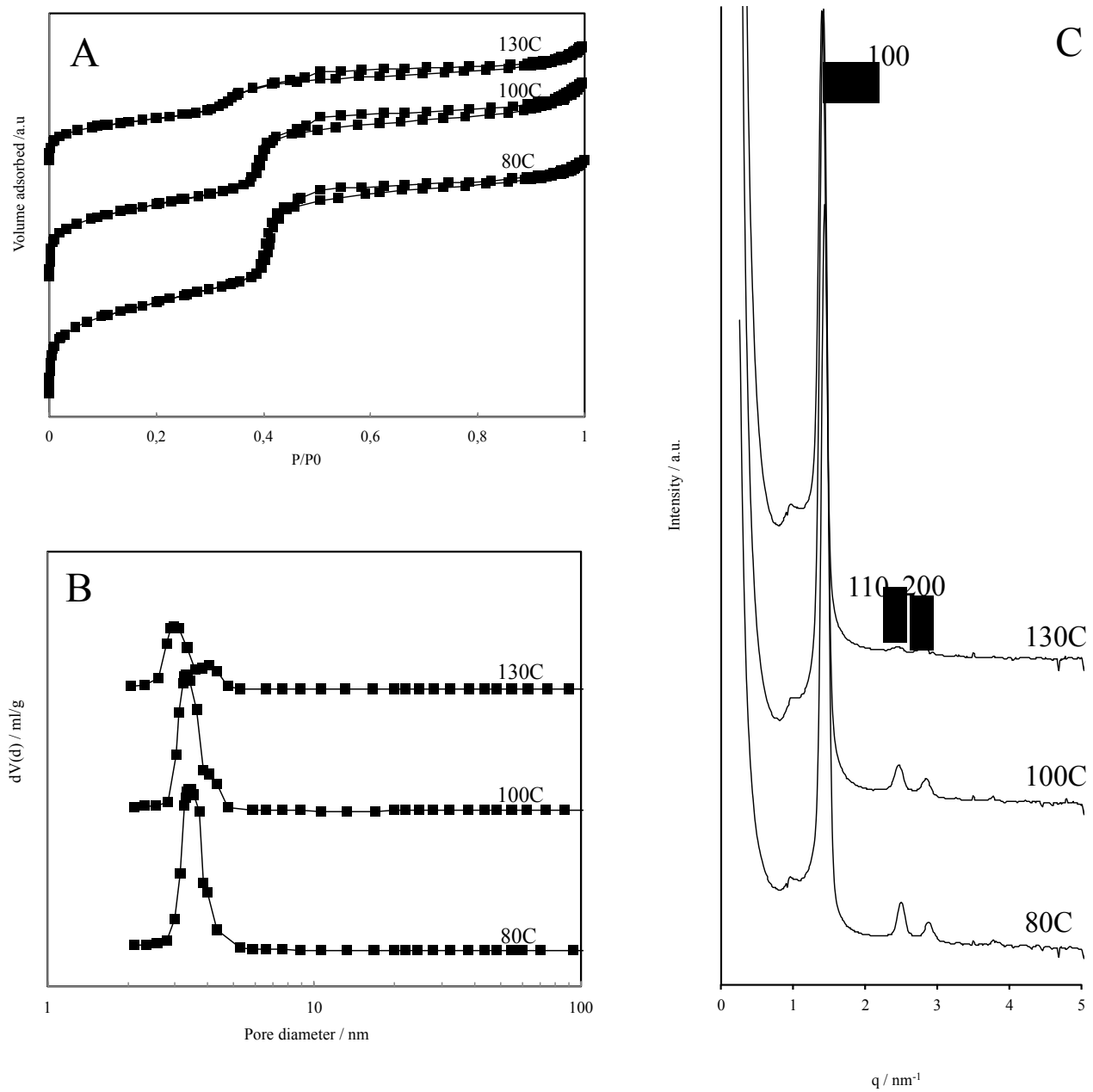


Fig.S7. SAXS Intensity profiles of Meso-BOS-X in varying BOS/TEOS molar ratio (X)



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g. S8. Textural and structural analysis of Meso-Oleic-0.125 synthesized with different hydrothermal temperature; A- N₂-Adsorption-desorption, B- Pore size distribution (desorption branch), C- SAXS patterns

Table S1. Textural and structural properties of the mesostructured silica material prepared with Bio-Oleic as template for different hydrothermal temperatures

Hydrothermal temperature / °C	BET / m ² .g ⁻¹	V _p / cm ³ .g ⁻¹	W _{BJH} / nm	d spacing / nm	Unit cell parameter / nm	Wall thickness / nm
80	952	1,01	3,7	4,40	5,10	1,70
100	823	0,89	3,8	4,40	5,10	1,30
130	549	0,58	3,7	4,50	5,10	1,40

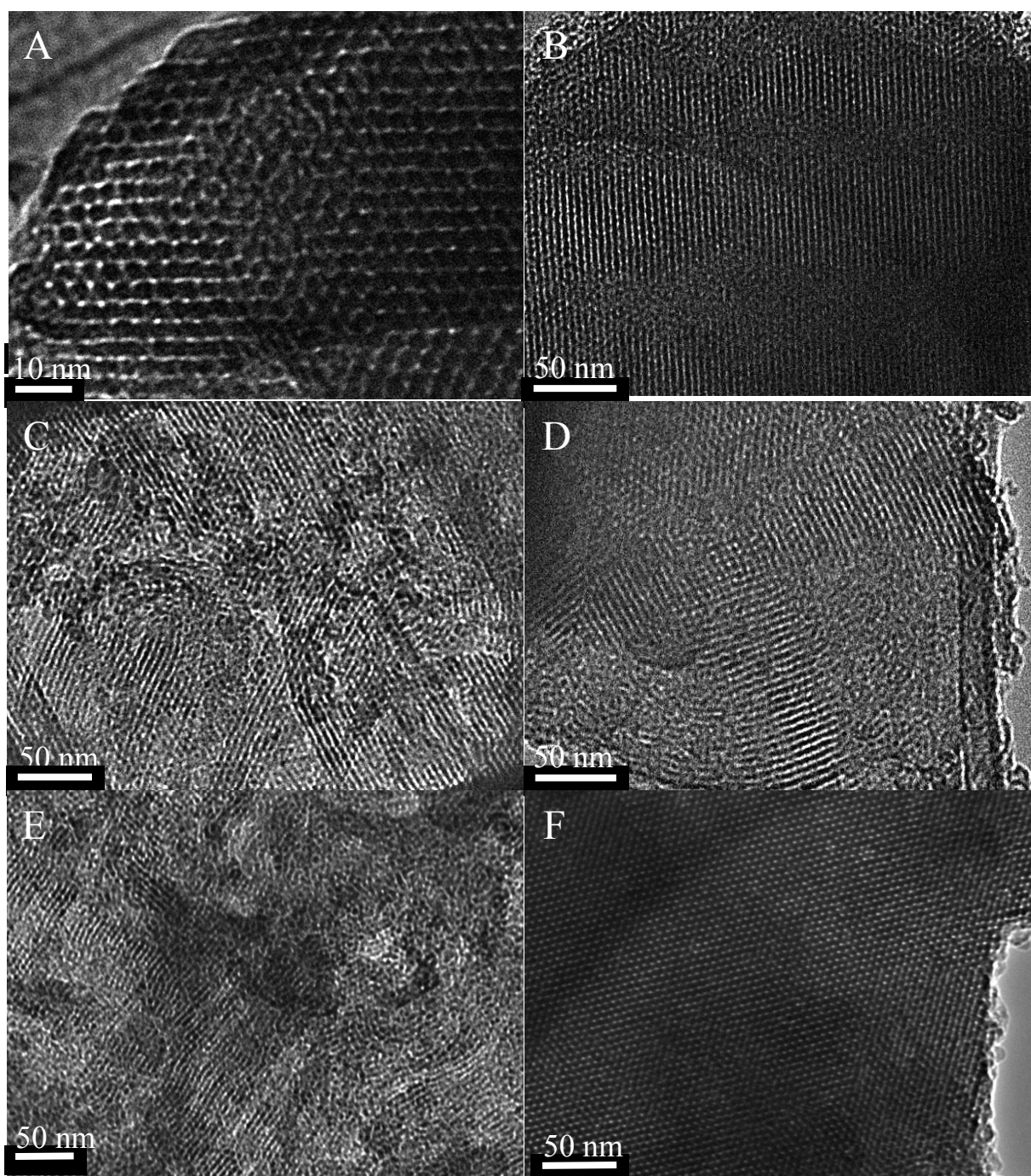


Fig.S9. TEM pictures of the Meso-Oleic-125 synthesized in basic media with different hydrothermal temperature (HT) for one day; A/B-HT 80C, C/D HT 100C, E/F-HT 130C