Microwave-assisted single-surfactant templating synthesis of mesoporous zeolites

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EXPERIMENTAL

Characterization and Testing of Zeolitic Materials. The XRD measurements were carried out using CuKα radiation in the 2θ angle ranges of 1 to 7º and 7 to 50º, low-angle and wide-angle, respectively, for the calcined samples using a step of 0.02°, on a PANalytical X'Pert Pro multipurpose diffractometer.

N_2 adsorption-desorption isotherms were measured using a Micromeritics ASAP 2010 and ASAP 2020 volumetric adsorption analyzers at -196 ºC. The samples were outgassed at 200 ºC for 2 hours under vacuum prior to each measurement. The specific surface area (S_{BET}), the total pore volume (V_t) and the pore size distribution (PSD) were calculated using the adsorption branches of isotherms. The specific surface areas were calculated according to the Brunauer–Emmett–Teller (BET) method using adsorption data in the relative pressure P/P_0 range of 0.05–0.2. The total pore volumes (V_t) were estimated from the amount adsorbed at the relative pressure of 0.98. The pore size distributions were calculated from adsorption branches of nitrogen adsorption isotherms using the improved KJS method [1]. The pore widths at the maximum PSDs were defined as the size of ordered mesopores.

CO_2 adsorption isotherms were obtained at 23 ºC using Micromeritics ASAP 2020 volumetric adsorption analyzer. The samples were outgassed at 200 ºC for 2 h under vacuum prior to each measurement.

The Scanning Electron Microscopy (SEM) images were taken using a Hitachi Tabletop Microscope TM-3000 instrument equipped with high-sensitivity semiconductor backscattered

electron detector under 10kV accelerating voltage and 50 Hz frequency. Transmission Electron Microscopy (TEM) images were taken on FEI Tecnai TF20 operated at 200kV. The samples were ultrasonically dispersed in H_2O at a concentration of 1 mg mL^{-1}, and a drop of the suspension was deposited on a carbon copper grid, and then dried at 100 ºC. In addition, the corresponding Fast Fourier Transform (FFT) patterns were obtained.

Microwave-assisted syntheses were performed using a MARS5 Microwave System manufactured by CEM Corporation (Matthews, NC, USA).

Catalytic testing of the MZ samples in the pyrolysis reaction of vacuum gasoil (VGO) was investigated and compared with thermal pyrolysis of VGO. The process was carried out in a micro-reactor at 500 ºC, under helium flowing at 25 mL min^{-1}. The pyrolyzer was coupled with the gas chromatograph equipped with a mass spectrometer detector (GC/MS QP 2010 Series, Shimadzu). The products from a catalytic and thermal pyrolysis of VGO were analyzed using a capillary column UA5-30M-0.25F (30 m x 0.25 mm i.d., 0.25 µm film thickness). VGO is composed of n-paraffins with number of carbons distributed in range from C_{20} to C_{40} and corresponds to the residue obtained in the vacuum distillation of the residue generated at the atmospheric distillation of crude oil [2].

[1] M. Jaroniec, L.A. Solovyov, Langmuir, 2006, 22, 6757.

[2] A. Marcilla, M.R. Hernandez, A.N. Garcia, Appl. Catal. A: Gen., 2008, 341, 181.

Table S1. Textural properties of ZSM-5 zeolite samples and commercial ZSM-5

Sample	V_t	S_{BET}
	Pore volume (cc/g)	(m^2/g)
ZSM-5-MW-22	0.14	297
ZSM-5-MW-24	0.11	288
Commercial ZSM-5	0.10	300

Table S2. CO_2 adsorption capacity of the samples studied

Sample	n_{CO2} (mmol g^{-1})*
MZ-TS-30	2.3
ZSM-5-MW-24	2.0
ZSM-5/AlMCM-41**	1.7
MZ-OS-34	1.5
AlMCM-41 Si/Al = 15	0.8

*CO_2 adsorption capacity at 23 ºC and 760 mmHg
****** one-pot synthesis, dual templating and conventional autoclave heating

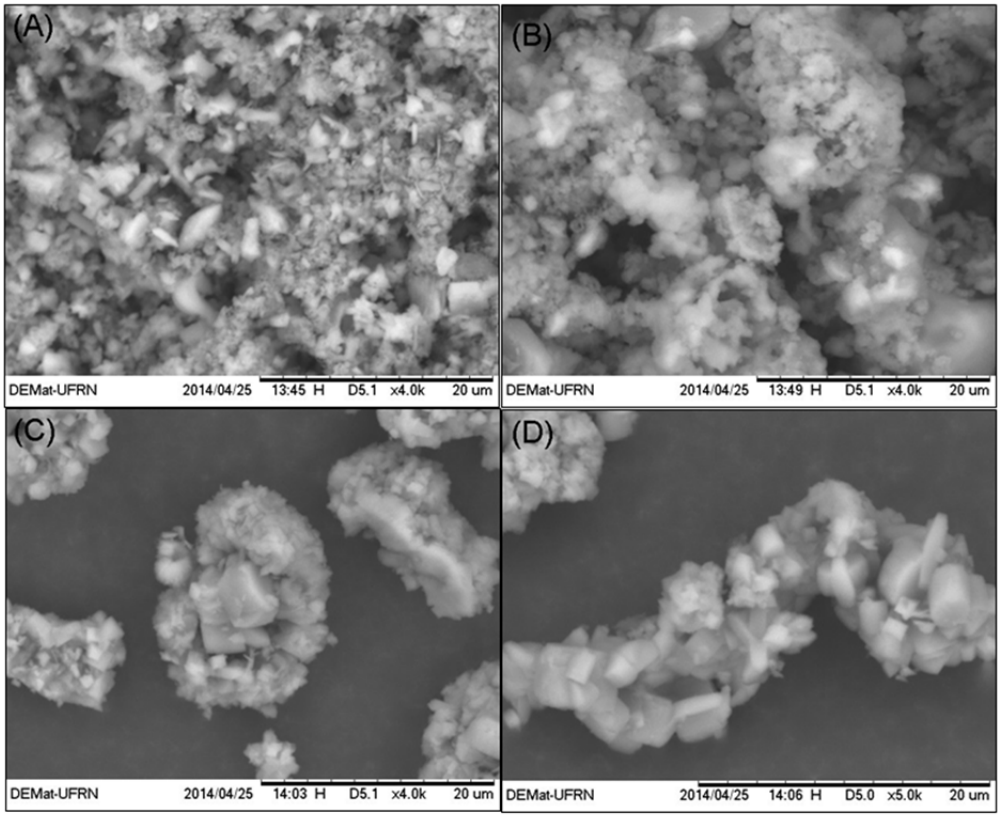
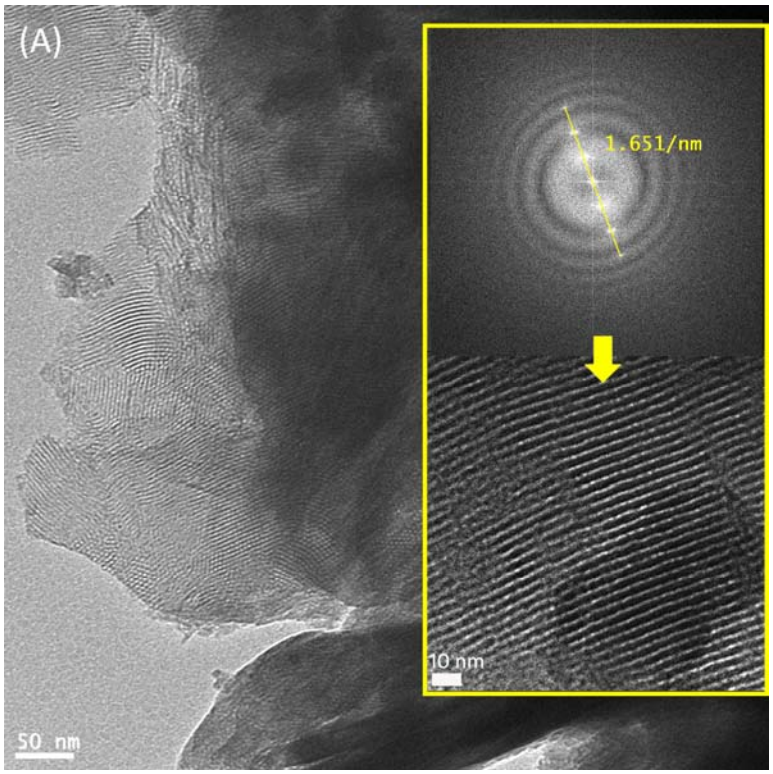
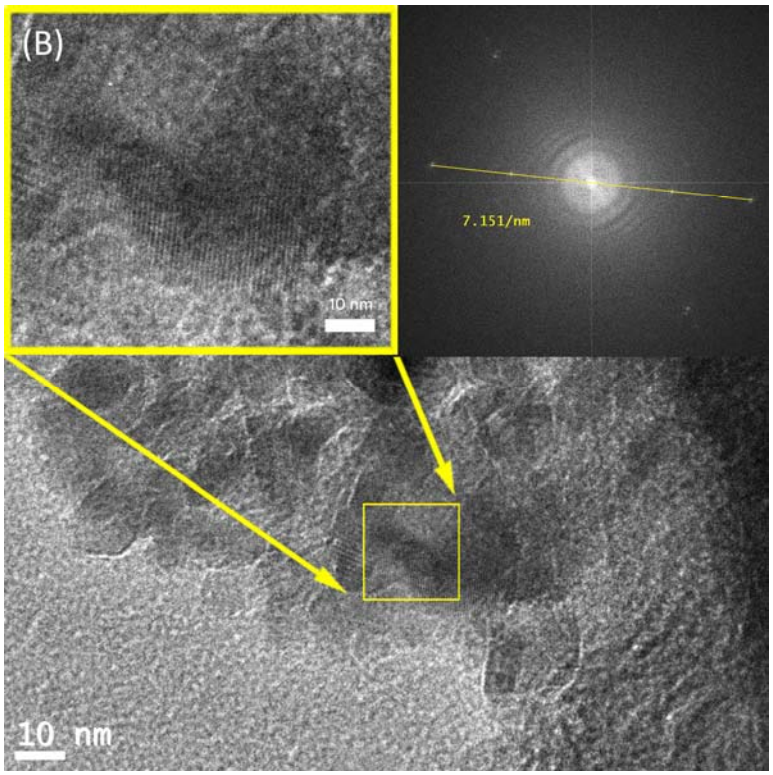


Figure S1. SEM images of MZ-TS-30 (A), MZ-OS-34 (B) and ZSM-5-MW-24 zeolite (C, D).


(A)
1.651/nm
10 nm
50 nm


(B)
10 nm
7.151/nm
10 nm

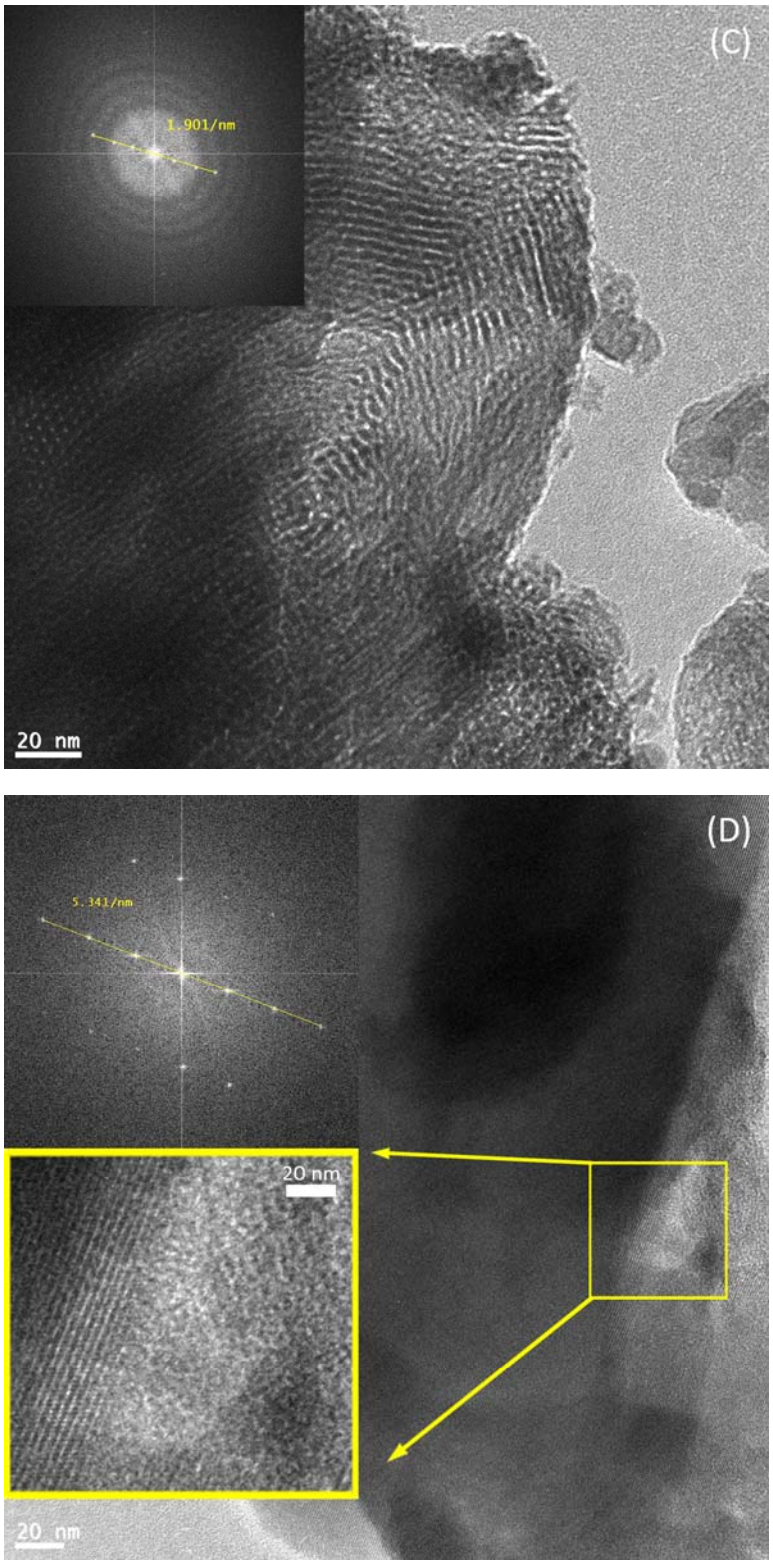


Figure S2. TEM and FTT (insets) images obtained at different foci to visualize the mesoporosity and crystallinity of the MZ-OS-34 (A and B) and MZ-TS-30 samples (C and D).