Supporting Information

Niobium doped Hexagonal Mesoporous Silica (HMS-X) Catalyst for Vapor Phase Beckmann Rearrangement Reaction

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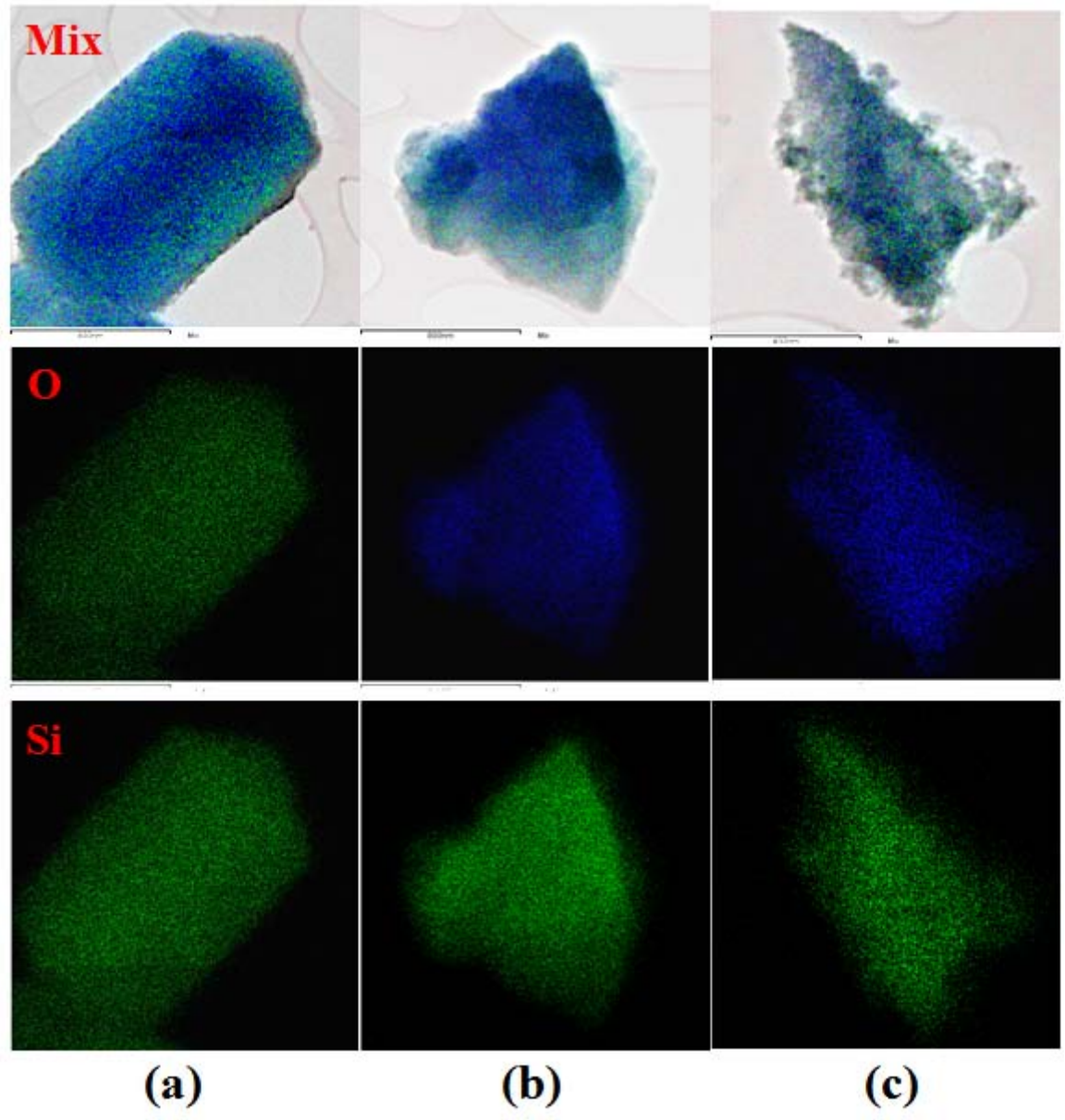

Fig. 1 Elemental mapping obtained from the selected region of the (a) Nb-HMS-X (Si/Nb = 13), (b) Nb-HMS-X (Si/Nb=3) and (c) Nb-HMS-X (Si/Nb=2)

Synthesis of Nb-MCM-41 catalyst:

Nb-MCM-41 material was synthesized by using Cetyl trimethylammonium bromide (CTAB) as a template under basic condition. In this procedure CTAB (Acros) (9.11 g) was dissolved in 162 ml deionized water and stirred the solution for 15 min. Tetraethyl orthosilicate (20.83 g) (TEOS, Acros) was added to the CTAB solution and stirring was continued for 15 min. After that ethanolic solution of Niobium Pentachloride (2.02 g) (Aldrich) was added to this solution and stirring was continued for 1h. The resulting gel composition of the mixture is CTAB : H_2O : TEOS : $NbCl_5$ = 0.025 : 9 : 0.1 : 0.0075 (molar ratio) . After 1 h stirring aqueous NaOH solution was added to the solution until pH attained to 8-8.5. The final mixture was vigorously stirred for 1 h and the autoclaved at 353 K for 3 days. After hydrothermal treatment the material was filtered and washed several time by deionized water and dried in air. After drying the material was calcined at 703 K for 8 h. The final material obtained after calcinations were Nb-MCM-41 and stored in vacuum desicator.

Synthesis of Nb-HMS-3 catalyst:

Nb-HMS-3 material was synthesized by using Cetyl trimethylammonium bromide (CTAB) as a template under basic condition. In this procedure CTAB (Acros) (15.3 g) was added to a mixture of water (729 g) and ethanol (306.6 g), and then pH of the solution was maintained at 12.4 by adding aqueous NH_3 solution. Then the solution was stirred at 277 K for 30 min. Tetraethyl orthosilicate (20.83 g) (TEOS, Acros) and ethanolic solution of Niobium Pentachloride (2.02 g) (Aldrich) was added to the CTAB solution and stirring was continued for another 3 h at 277 K temperature. The resulting gel composition of the mixture is CTAB : H_2O : EtOH : TEOS : $NbCl_5$ = 0.042 : 40.89 : 6.66 : 0.01 : 0.0075 (molar ratio). The resulting slurry was refrigerated for 16 h and the precipitated material was collected by filtration, and washed several time by deionized water. After washing the material was calcined at 698 K

for 8 h. The final material obtained after calcinations were Nb-HMS-3 and stored in vacuum desicator.

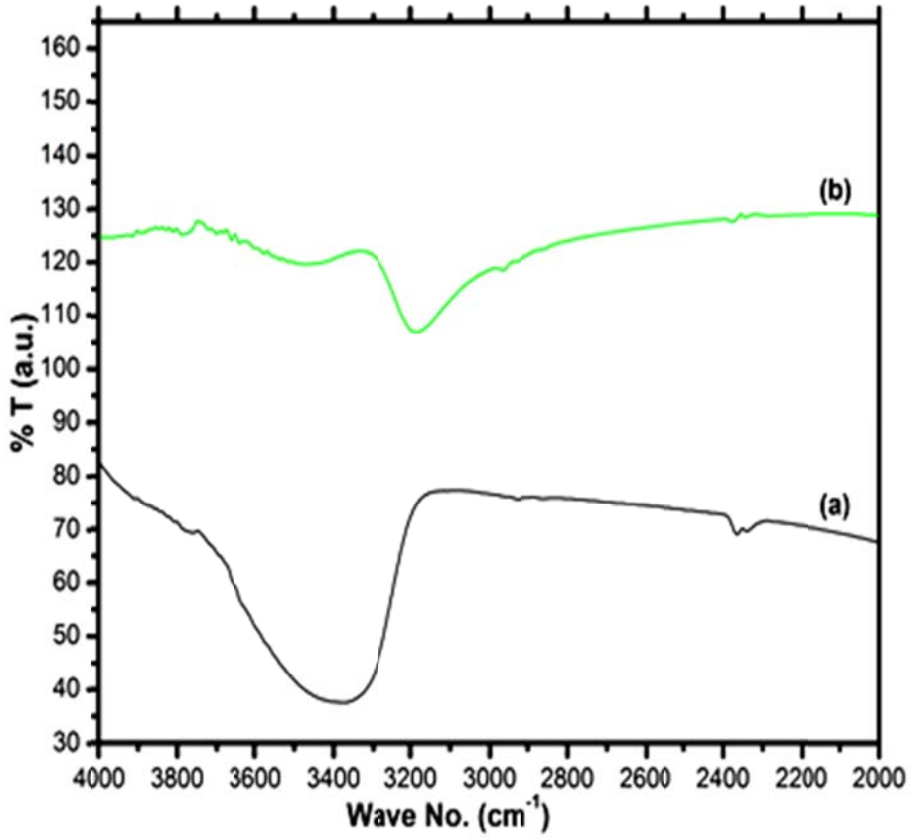

Fig. 2 FT-IR spectra of (a) Nb-HMS-X (Si/Nb = 13), (b) Silylated Nb-HMS-X (Si/Nb = 13) catalyst

Table S1 The gel composition and thermal treatment of different catalysts

Catalyst	Gel composition (mol %)	Hydro thermal temp. (^{0}C)	Drying temp. (^{0}C)	Calcination temp. (^{0}C)
	P123 : H_2O : HCl : n-Butanol : TEOS : $NbCl_5$			
KIT-6	0.017 : 200 : 5.4 : 1.325 : 1	100	100	540
Nb-KIT-6 (Si/Nb=20)	0.017 : 200 : 5.4 : 1.325 : 1 : 0.050	100	100	540
Nb-KIT-6 (Si/Nb=13)	0.017 : 200 : 5.4 : 1.325 : 1 : 0.075	100	100	540
Nb-KIT-6 (Si/Nb=3)	0.017 : 200 : 5.4 : 1.325 : 1 : 0.3	100	100	540
Nb-KIT-6 (Si/Nb=2)	0.017 : 200 : 5.4 : 1.325 : 1 : 0.45	100	100	540
	CTAB : H_2O : EtOH : TEOS : $NbCl_5$			
Nb-HMS-3 (Si/Nb=13)	0.042 : 40.89 : 6.66 : 0.01 : 0.0075	-	-	425
	CTAB : H_2O : TEOS : $NbCl_5$			
Nb-MCM-41 (Si/Nb=13)	0.025 : 9 : 0.1 : 0.0075	80	100	430