Electronic Supplementary Information (ESI)

To

Clean synthesis of furfural oxime through liquid-phase ammoximation of furfural over titanosilicate catalysts

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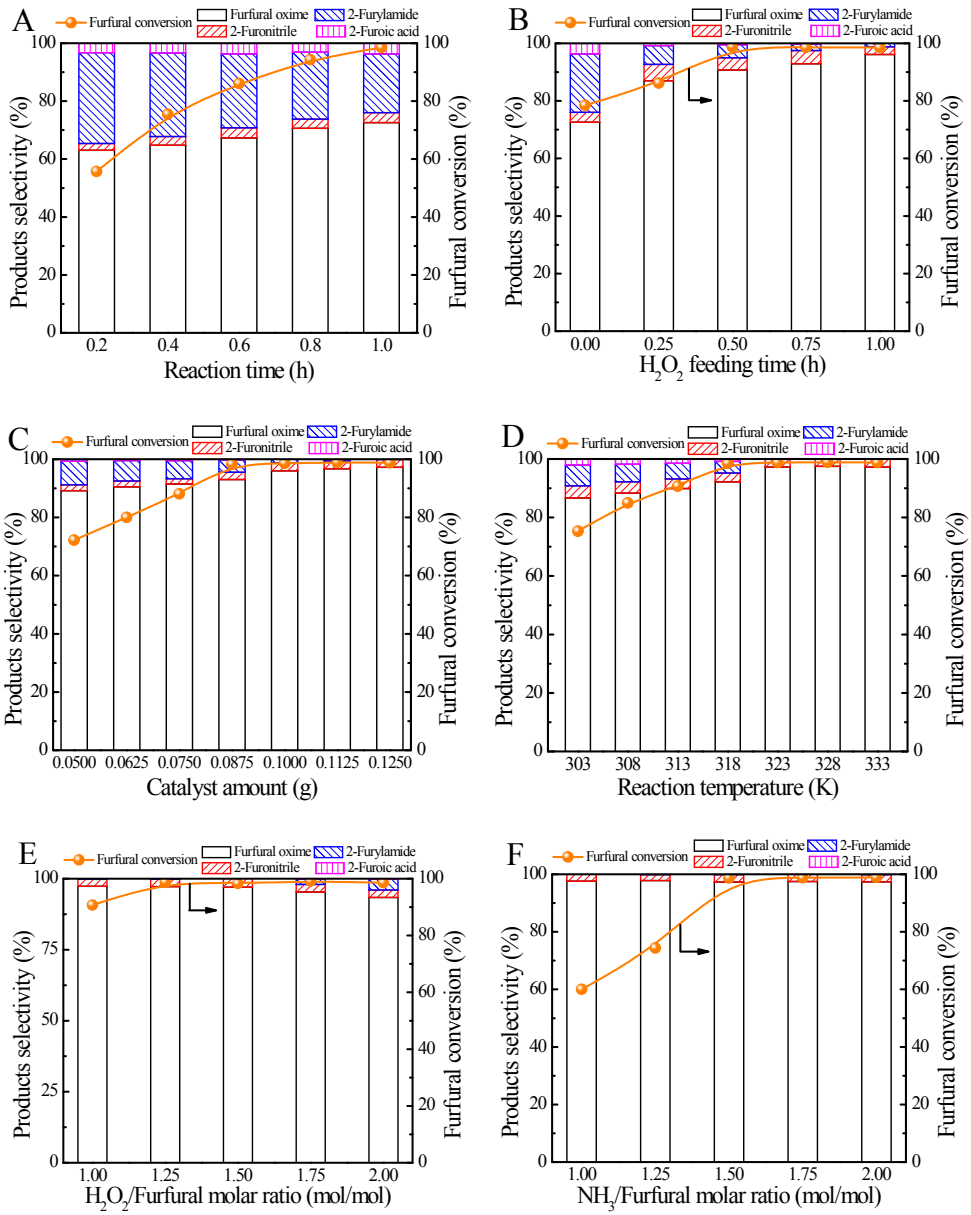

Fig. S1. Effect of reaction time (A), feeding time of H_2O_2 (B), catalyst amount (C), reaction temperature (D), H_2O_2/furfural molar ratio (E) and NH_3/furfural molar ratio (F) on furfural ammoximation over Ti-MOR. Reaction conditions: Ti-MOR, 0.1 g (A, B), 0.125 g (D, E, F); H_2O, 12 g; furfural, 20 mmol; H_2O_2 (31 wt. %), 30 mmol (A), 25 mmol (B, C, D, F); NH_3 (25 wt. %), 30 mmol; temp., 323 K; total time, 1 h. H_2O_2 was fed at once (A); The feeding time of 0 h stands for that H_2O_2 was fed at once, while the feeding time of 0.25, 0.50, 0.75 and 1.00 h stand for that H_2O_2 was dropwise added at different feeding rate of 9.6, 4.8, 3.2 and 2.4 mL h^{-1}, respectively (B); H_2O_2 was dropwise added within 1 h (C, D, E, F).

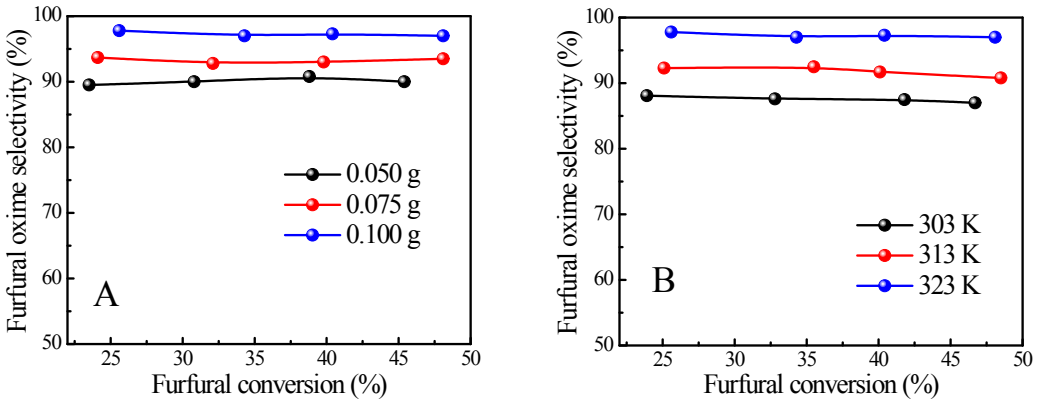

Fig. S2. Effect of catalyst amount (A) and reaction temperature (B) on the furfural oxime selectivity. Reaction conditions: Ti-MOR, 0.100 g (B); H_2O, 12 g; furfural, 20 mmol; NH_3 (25 wt. %), 30 mmol; temp., 323 K (A). H_2O_2 was fed at a constant rate of 0.5 mmol min^{-1}.

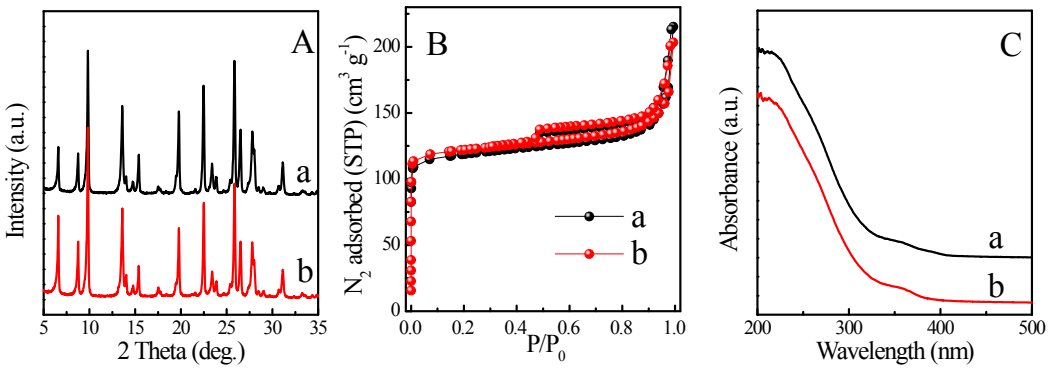

Fig. S3. XRD patterns (A), Nitrogen adsorption desorption isotherms at 77 K (B) and UV-vis spectra (C) of fresh Ti-MOR (a) and regenerated Ti-MOR (b) after five recycles of reaction-regeneration in the furfural ammoximation.

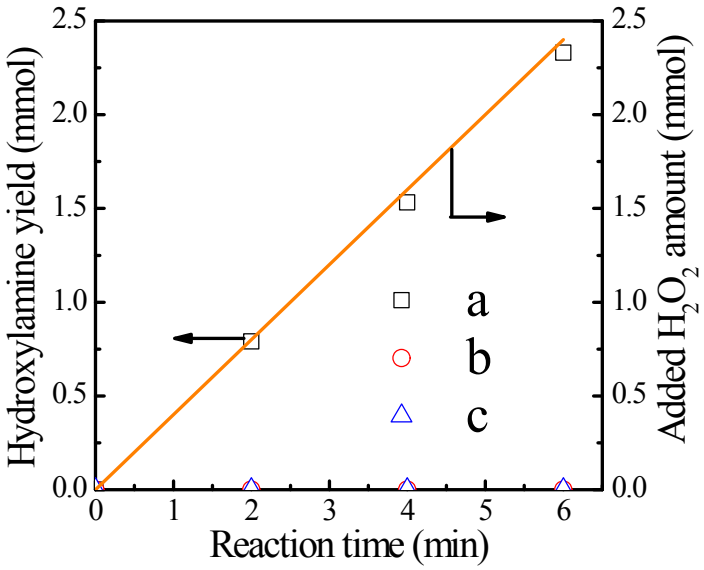

Fig. S4. Kinetic of hydroxylamine formation over Ti-MOR (a, □), TS-1 (b, ○) and Ti-MWW (c, Δ). Reaction conditions: cat., 0.1 g; NH_3, 30 mmol; H_2O_2, 2.4 mmol; H_2O, 12 g; temp., 323 K. H_2O_2 was dropwise added at a constant rate within 6 min. The change of the amount of H_2O_2 added with time is also shown.

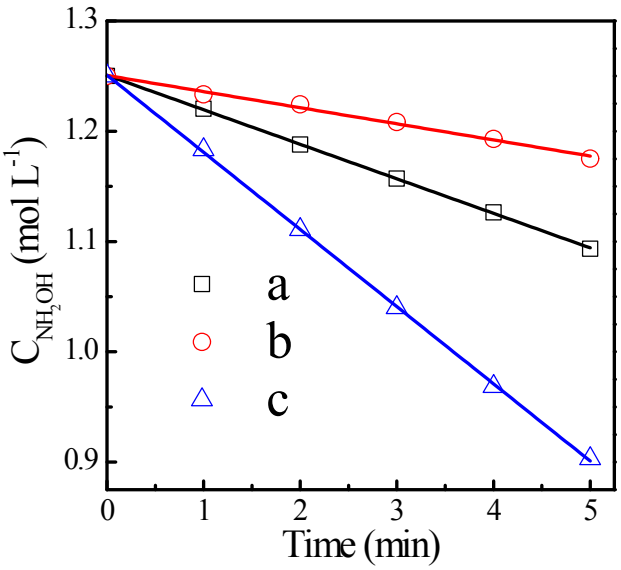

Fig. S5. Kinetic of hydroxylamine decomposition over Ti-MOR (a), TS-1 (b) and Ti-MWW (c). Reaction conditions: cat., 0.1 g; $NH_2OH \cdot HCl$, 20 mmol; H_2O_2, 2.4 mmol; H_2O, 12 g; temp., 323 K. H_2O_2 was dropwise added at a constant rate within 6 min.

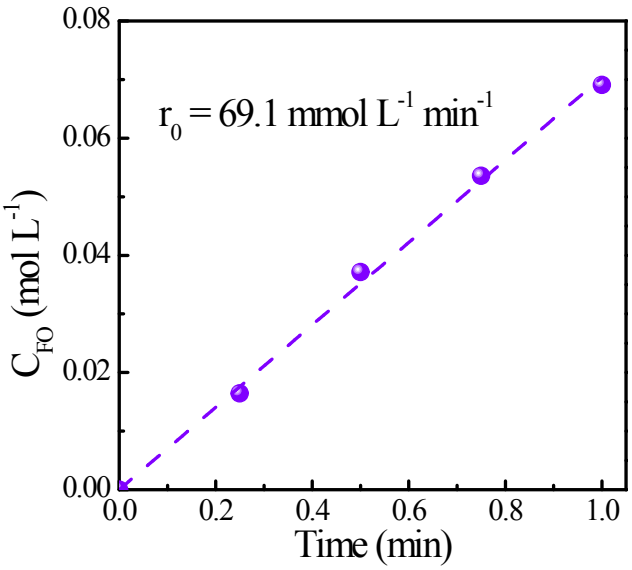

Fig. S6. Kinetic of the furfural oximation with hydroxylamine. Reaction conditions: $NH_2OH{\cdot}HCl$, 20 mmol; H_2O, 12 g; temp., 323 K.