

Supplementary Information

Operando Spectroscopy Unravels the Nature of Deactivating Species and their Precursors in Alkene Oligomerization Catalysis

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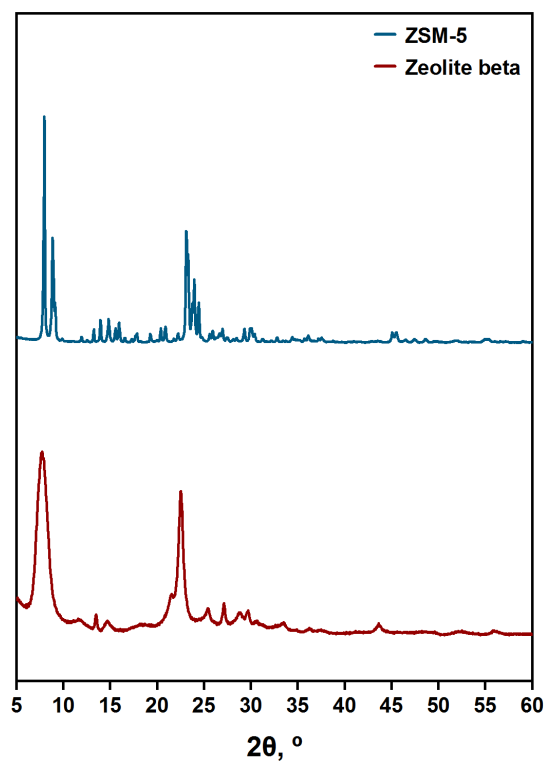


Fig. S1 XRD patterns of ZSM-5 and zeolite beta.

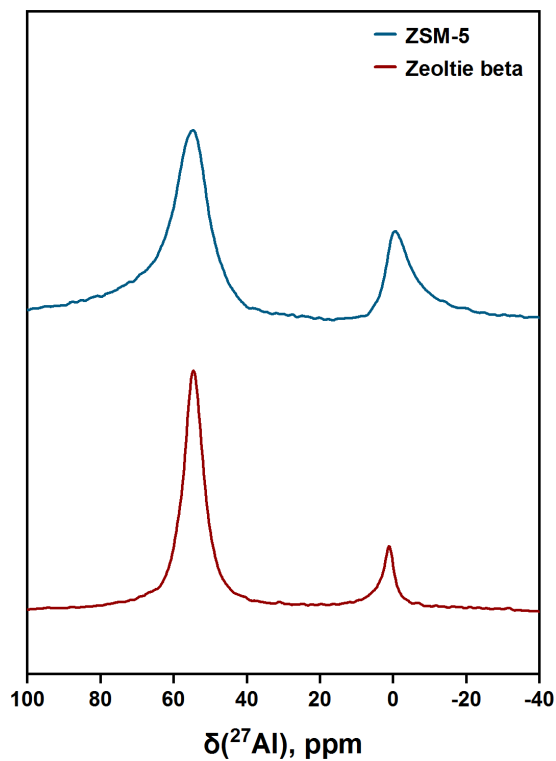


Fig. S2 ^{27}Al MAS NMR spectra of ZSM-5 and zeolite beta.

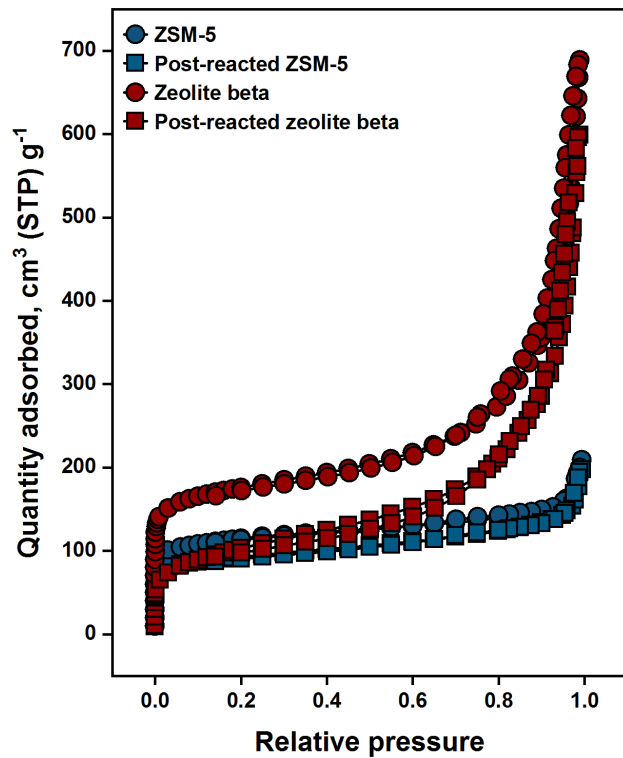


Fig. S3 Nitrogen physisorption isotherms measured on unreacted and post-reacted ZSM-5 and zeolite beta at 77 K.

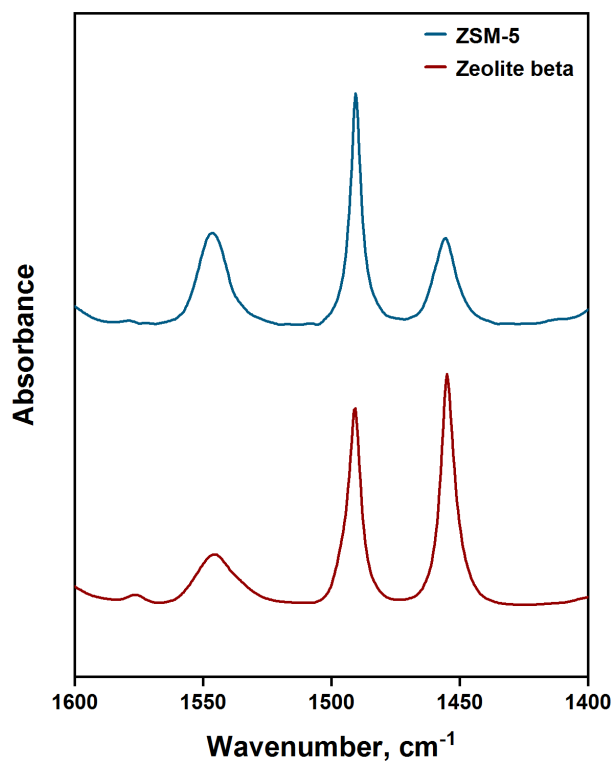


Fig. S4 Pyridine-FTIR spectra measured on ZSM-5 and zeolite beta.

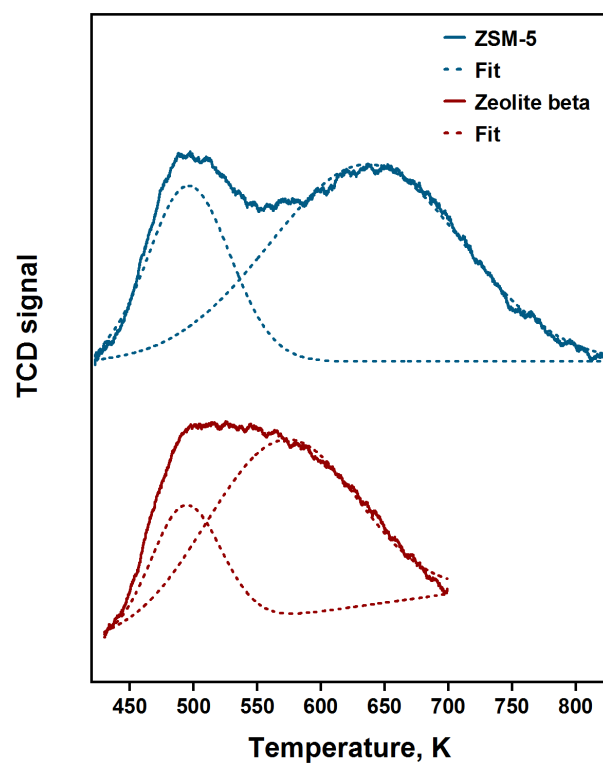


Fig. S5 NH₃-TPD profiles measured on ZSM-5 and zeolite beta.

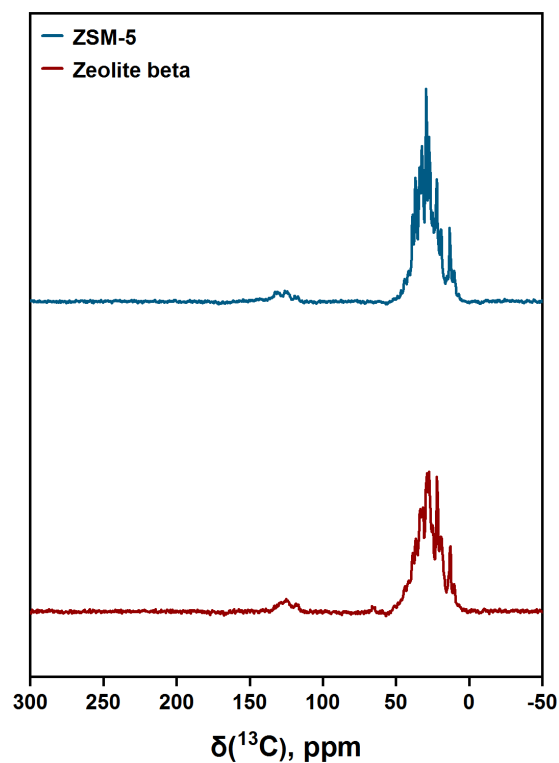


Fig. S6 INEPT-based solid-state ^{13}C NMR spectra of post-reacted ZSM-5 and zeolite beta. [523 K; 100 kPa propene pressure; 10-15 min TOS]. The spectra denote features of methyl and methylene groups (10-40 ppm) and olefinic groups (120-135 ppm).

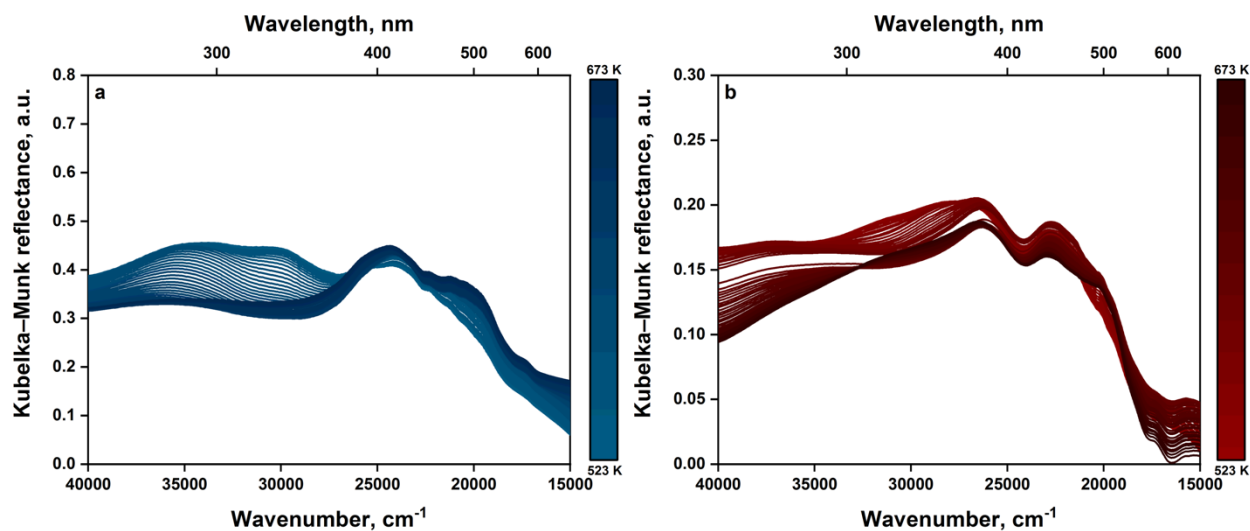


Fig. S7 Kubelka-Munk reflectance of the UV-Vis bands measured during temperature-programmed experiments on (a) ZSM-5 and (b) zeolite beta.

Table S1 Nitrogen physisorption measurements conducted on unreacted and post-reacted zeolites.

| Material | $S_{\text{Micropore}},^a$ $\text{m}^2 \text{g}^{-1}$ | $S_{\text{Mesopore}},^b$ $\text{m}^2 \text{g}^{-1}$ | $S_{\text{BET}},^c$ $\text{m}^2 \text{g}^{-1}$ | $V_{\text{Micropore}},^d$ $\text{cm}^3 \text{g}^{-1}$ | $V_{\text{Mesopore}},^e$ $\text{cm}^3 \text{g}^{-1}$ | $V_{\text{Total}},^f$ $\text{cm}^3 \text{g}^{-1}$ |
|---------------------------|---|--|---|--|---|--|
| ZSM-5 | 269 | 156 | 425 | 0.11 | 0.14 | 0.25 |
| Post-reacted ZSM-5 | 204 | 136 | 340 | 0.084 | 0.15 | 0.23 |
| Zeolite beta | 357 | 281 | 638 | 0.15 | 0.74 | 0.89 |
| Post-reacted zeolite beta | 37 | 318 | 355 | 0.021 | 0.62 | 0.64 |

^a Micropore area. ^b Mesopore area. ^c BET surface area. ^d Micropore volume. ^e Mesopore volume. ^f Total pore volume measured at *ca.* 0.95 of relative pressure.