

Fig. 8. INS spectra of catalysts reacted with dimethylether for 24 h (1D, (a)), 36 h (2D, (b)) and 75 h (3D, (c)). Reproduced with permission from reference [28].

deactivation time was correspondingly lengthened, and the carbon content after 75 h on stream was 14.6 wt%. In a third experiment, a catalyst was exposed to dimethylether for 24 h at a WHSV of 0.5, showed no loss of activity during this time and gave a carbon content of 8.8 wt%. GC–MS analysis of the trapped liquid products showed predominantly tetramethyl benzenes, trimethyl benzenes and xylenes [28].

INS spectra of all three catalysts were remarkably similar. Fig. 8 shows spectra measured in the 2000–4000 cm^{-1} range with the MERLIN spectrometer.

In all 3 cases the zeolite Si(OH)Al stretching mode at 3600 cm^{-1} is totally absent. The CH stretching band has two clear components, at ~ 2990 and ~ 3100 cm^{-1} . As described above for catalysts used in methanol conversion, these are assigned to aliphatic and olefinic/aromatic species respectively.

For these experiments, spectra in the lower frequency region were measured with an indirect geometry instrument, TOSCA. This

spectrometer provides for better spectroscopic resolution below 1500 cm^{-1} than that achievable with the direct instruments MAPS and MERLIN. Fig. 9 shows TOSCA spectra of the same three catalyst samples reacted with dimethylether for 1, 2 and 3 days. Although broadly speaking the spectra above 800 cm^{-1} are similar to those shown in Fig. 7 from catalysts reacted with methanol, the superior quality of the TOSCA spectra allows better determination of band positions. The TOSCA instrument also shows clearly the presence of multiple bands down to 200 cm^{-1} which were not resolved in the earlier MAPS and MERLIN spectra of zeolite catalysts.

Assigning all of the bands seen here is a challenging exercise. As a first attempt, Zachariou et al. compared the spectra of the used catalysts with those of plausible methylaromatic compounds which are believed to comprise a large component of the hydrocarbon pool. Fig. 10 shows such a comparison. Above 800 cm^{-1} the used catalyst spectrum more closely resembles that of *o*-xylene than durene, and certainly the

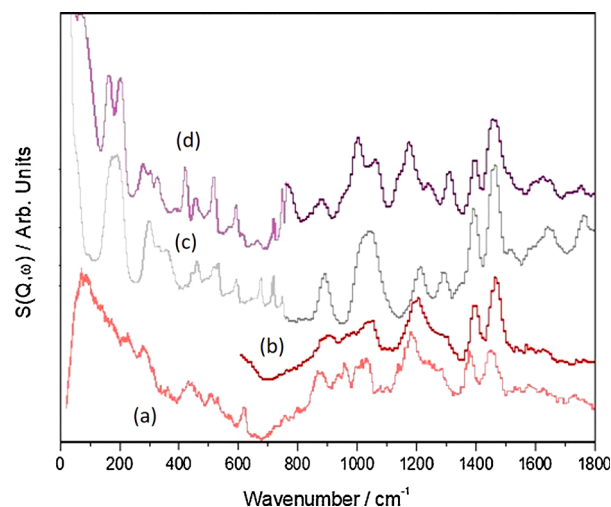


Fig. 10. Comparison of INS spectra of zeolite catalyst reacted with dimethylether for 3 days (TOSCA (a) and MERLIN (b)) with MERLIN spectra of pure durene (c) and ortho-xylene (d). Reproduced with permission from reference [28].

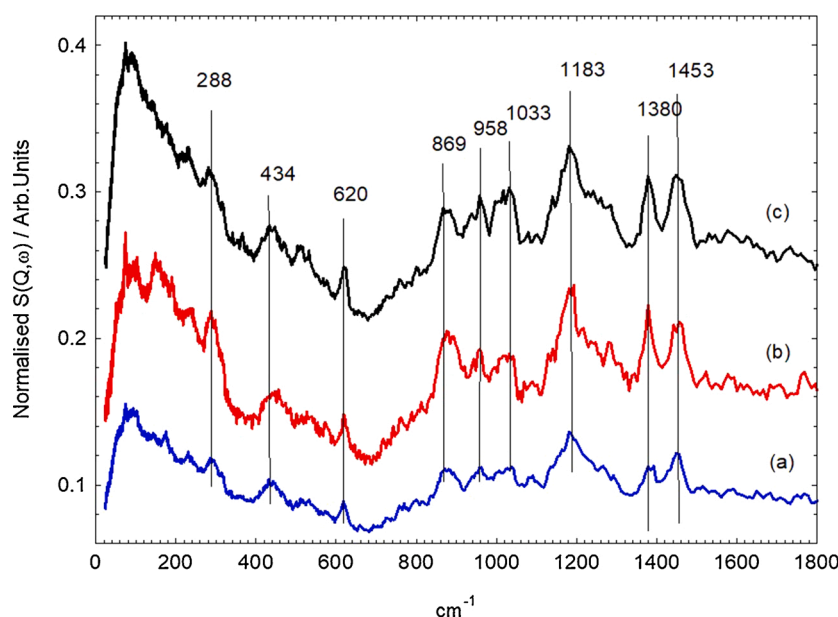


Fig. 9. INS spectra measured on the TOSCA spectrometer of catalysts reacted with dimethylether for 1 day (a), 2 days (b) and 3 days (c). Reproduced with permission from reference [28].