

In-situ studies on vapor phase catalytic decomposition of Dimethyl Oxalate

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Supplementary information:

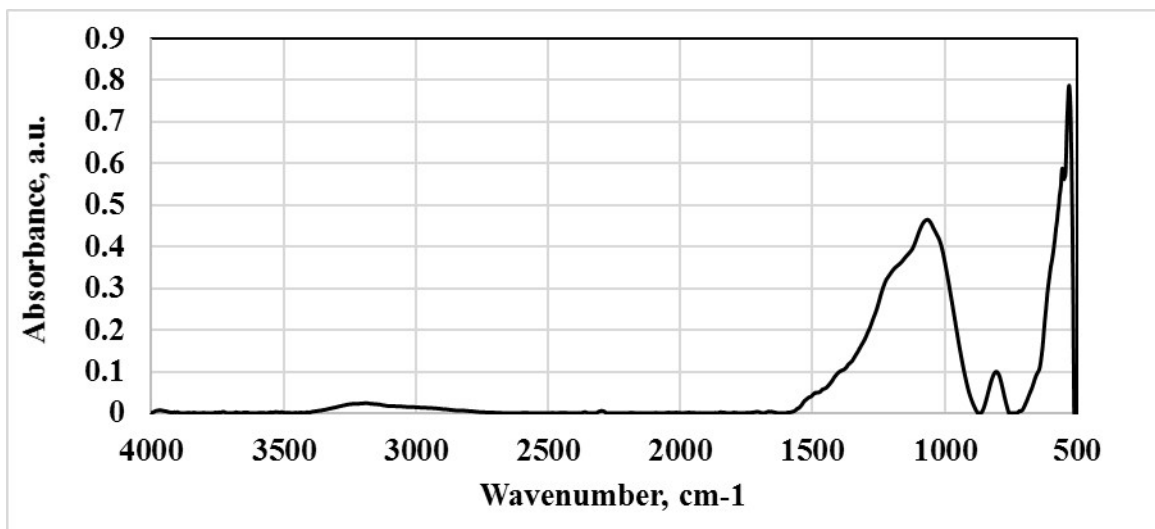


Figure A1: IR spectrum of α -Alumina – Presence of surface hydroxyl groups

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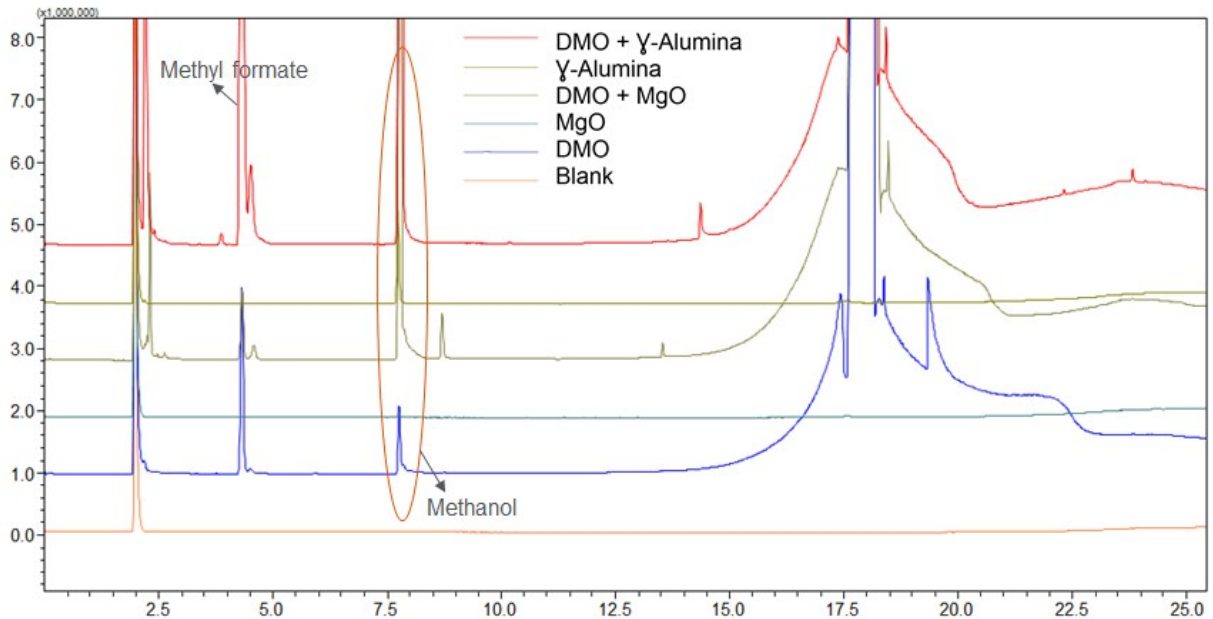


Figure A2: DMO on catalysts- MgO and γ -alumina

The TIC spectra of gaseous product resulting from DMO on two catalysts, i.e., MgO and γ -alumina, were compared in Fig. 1. In short, the procedure involves addition of 0.1 g of DMO and 0.5 g of either MgO or γ -alumina in a HS vial under nitrogen atmosphere and the vial was shaken rigorously on vortex before placing on headspace platen at 200 °C for 20 minute before analyzing on GCMS. In order to exclude any extraneous peaks, GCMS spectra for blank vial, neat DMO, MgO and γ -alumina were also recorded (Fig. 1).

The overlay of TIC spectra showed difference in product profile between DMO on MgO and DMO on γ -alumina. However, since the product identification is based only on the library match the peaks were not assign. Trend understanding from this experiment are as follows-

- a. Oxalic acid (vapor product) was not found when DMO is on both MgO and γ -alumina. DMO may not be decomposing to oxalic acid on MgO as it was also not found on the surface of MgO (by IR study) whereas oxalic acid must be anchoring on hydroxyl group of γ -alumina (supported by IR study).
- b. DMO decomposes to methyl formate and methanol on both the catalyst. However, the amount of methylformate is lower in case of DMO on MgO because we found that methylformate decomposes to methanol on MgO
- c. Permanent gaseous products such as CO and CO₂ were also detected at retention time less than 3 min, supporting the DMO decomposition to methylformate or oxalic acid.