Supporting Information

Molybdenum oxide with a varied valency ratio to enable a selective galactose epimerization to talose

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Experimental

Mathematical expressions

$$Galactose\ conversion\ (\%) = \frac{(Intial\ mass\ of\ sugar - Remaining\ mass\ of\ sugar\ after\ completion\ of\ reaction)}{Intial\ mass\ of\ sugar} \times 100$$

---------- (S1)

$$Product\ yield\ (\%) = \frac{Obtained\ mass\ of\ product}{Initial\ mass\ of\ Galactose} \times 100$$

---------- (S2)

$$Product\ selectivity\ (\%) = \frac{Mass\ of\ product}{Converted\ mass\ of\ Galactose} \times 100$$

---------- (S3)

Analytical characterization

$$D = \frac{K\lambda}{\beta\ cos\theta}$$

---------- (S4)

Where, D is crystallite size (nm), K is shape factor (0.9), λ is wavelength of Cu-Kα radiation (1.5406A), β is full width at half maximum (FWHM) intensity, and θ is Bragg's diffraction angle. The crystallite size of MoO_3 and MoO_{3-x} was observed to be ~ 68 nm and ~ 60 nm, respectively.

First-order kinetic rate equation

$$ln\left\{\frac{[Galac]_t}{[Galac]_0}\right\} = -\ k \times time\ (sec)$$

---------- (S5)

Where, $[Galac]_t$ and $[Galac]_0$ represent the final and initial reactant (galactose) concentration at time *t*. *k* is the observed rate constant of reaction (disappearance of galactose).

The temperature dependency of the rate constant, k was determined by using the Arrhenius equation (Eqn. S6):

$$k = A_0 \times e^{\left(-\frac{E_a}{RT}\right)}$$

---------- (S6)

where, A_0 is the frequency factor (or Arrhenius constant), E_a is the activation energy (J/mol), and R is the universal gas constant (8.31 J/mol. K).

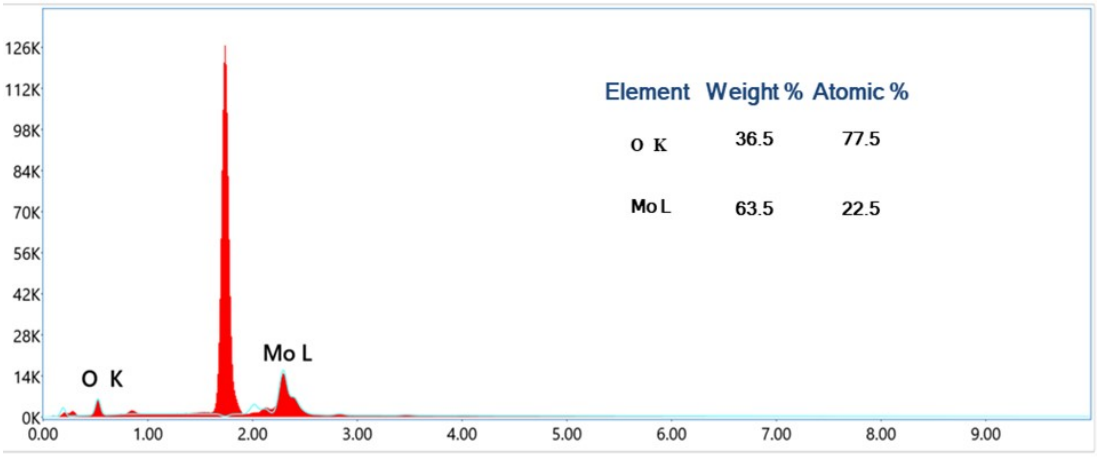

Figure S1. SEM-EDX result of MO-2M catalyst.

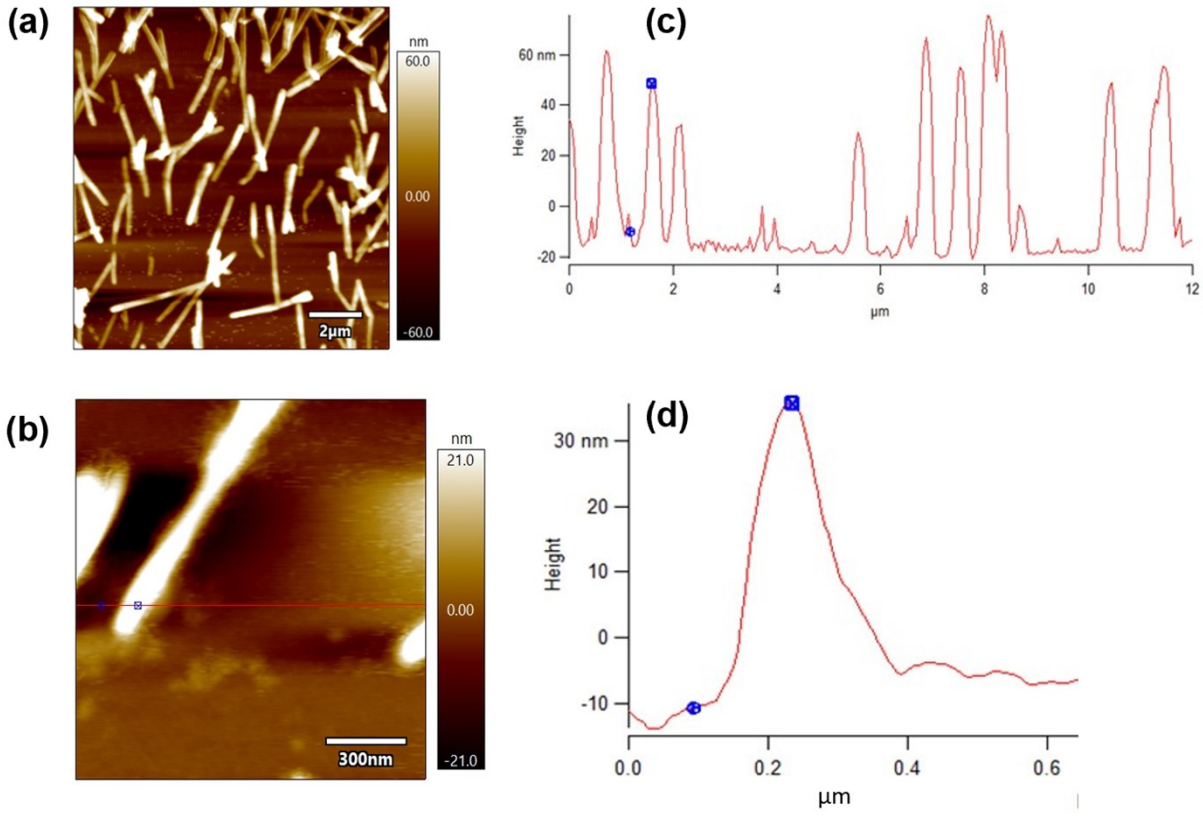

Figure S2. AFM analysis result of MO-2M catalyst: (a) typical AFM image, (b) magnified image of 300 nm, (c) and (d) corresponding height profiles of lines in (a) and (b), respectively.

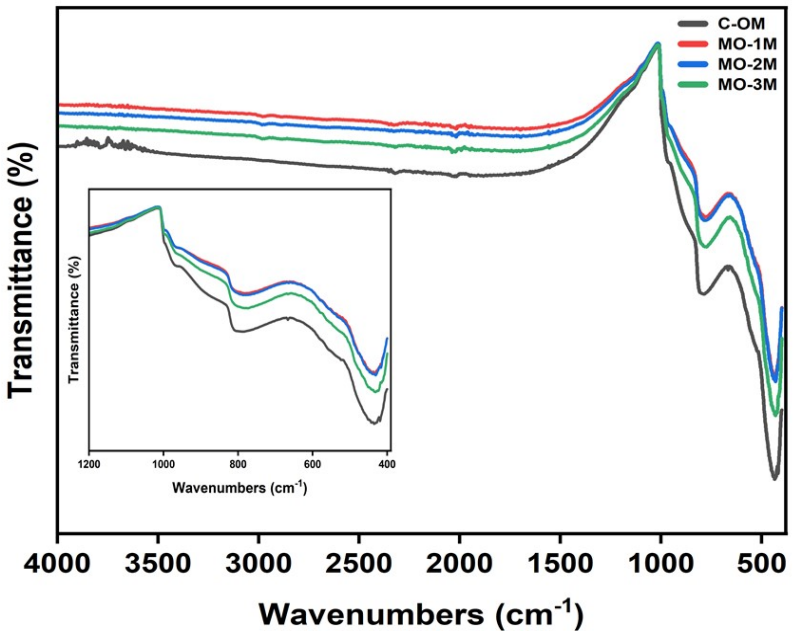

Figure S3. Comparative FTIR analysis result of MoO_3 catalysts (both pristine and acid-treated) in the 4000-400 cm^{-1} range. Inset: scan result of the catalysts in the 1200-400 cm^{-1} absorption range.

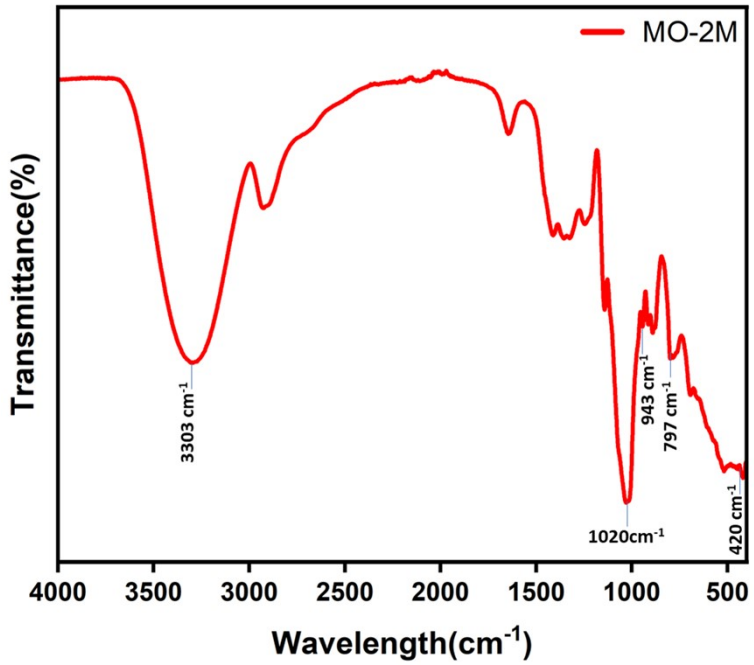

Figure S4. ATR-FTIR result of MO-2M catalyst.

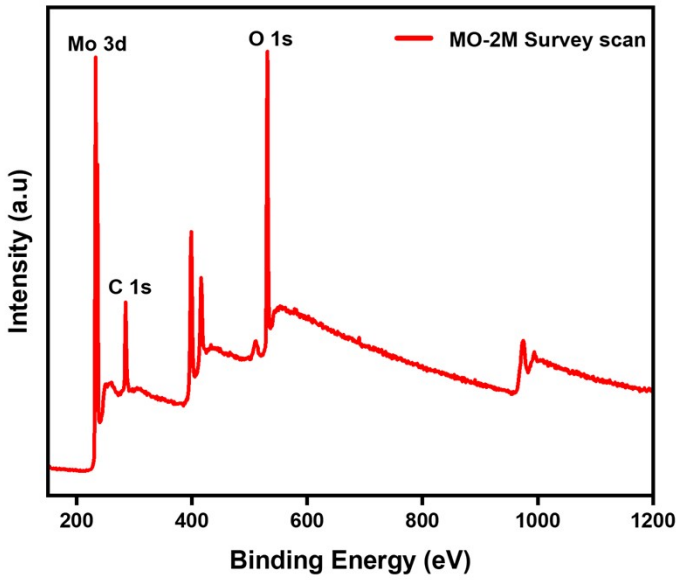

Figure S5. XPS survey spectrum of MO-2M catalyst.

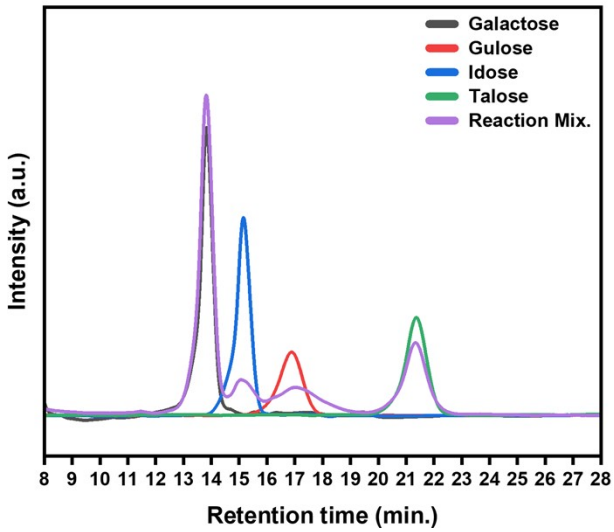

Figure S6. Stacked chromatogram of standard sugars *vs.* post-reaction mixture of galactose epimerization over commercial MoO_3 in a water medium at 15% wt. catalyst load and 120 °C.

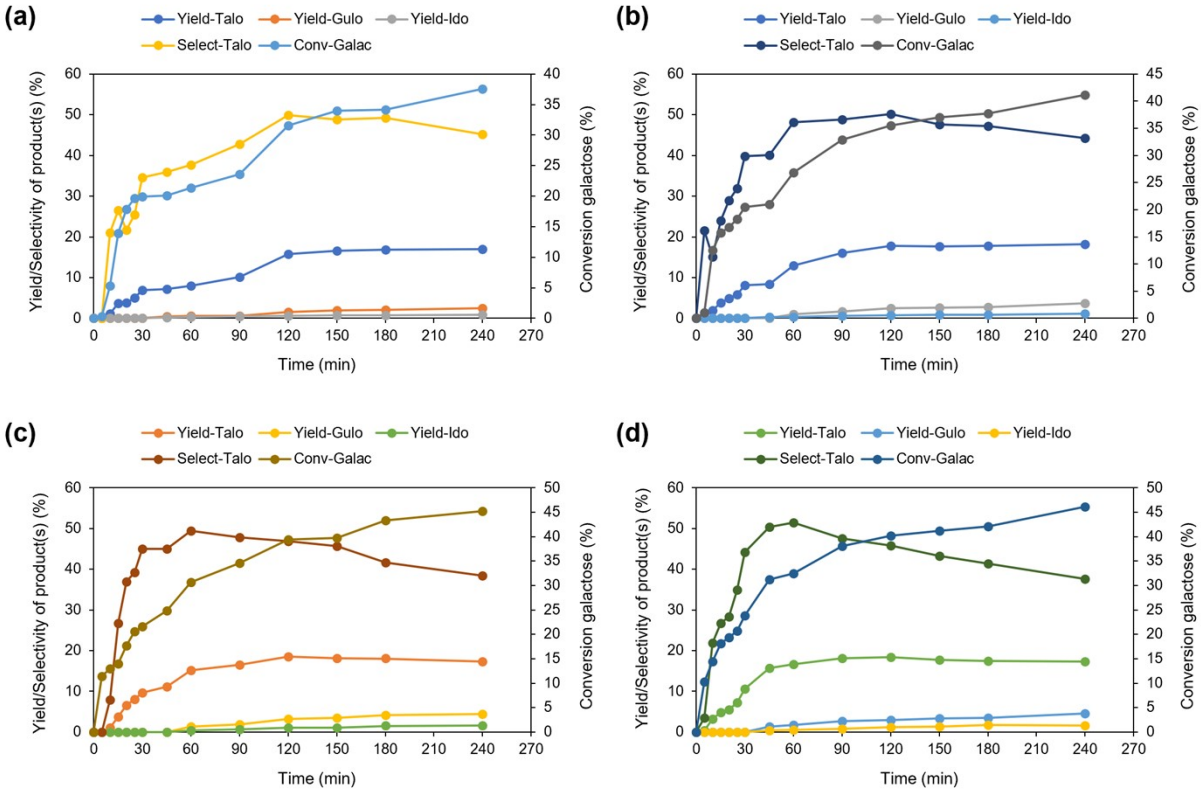

Figure S7. Galactose epimerization profile over commercial MoO_3 in a water medium under different catalyst loadings (w/w ratio of MoO_3 to galactose) at 90 °C: (a) 5%, (b) 10%, (c) 15% and (d) 20%.

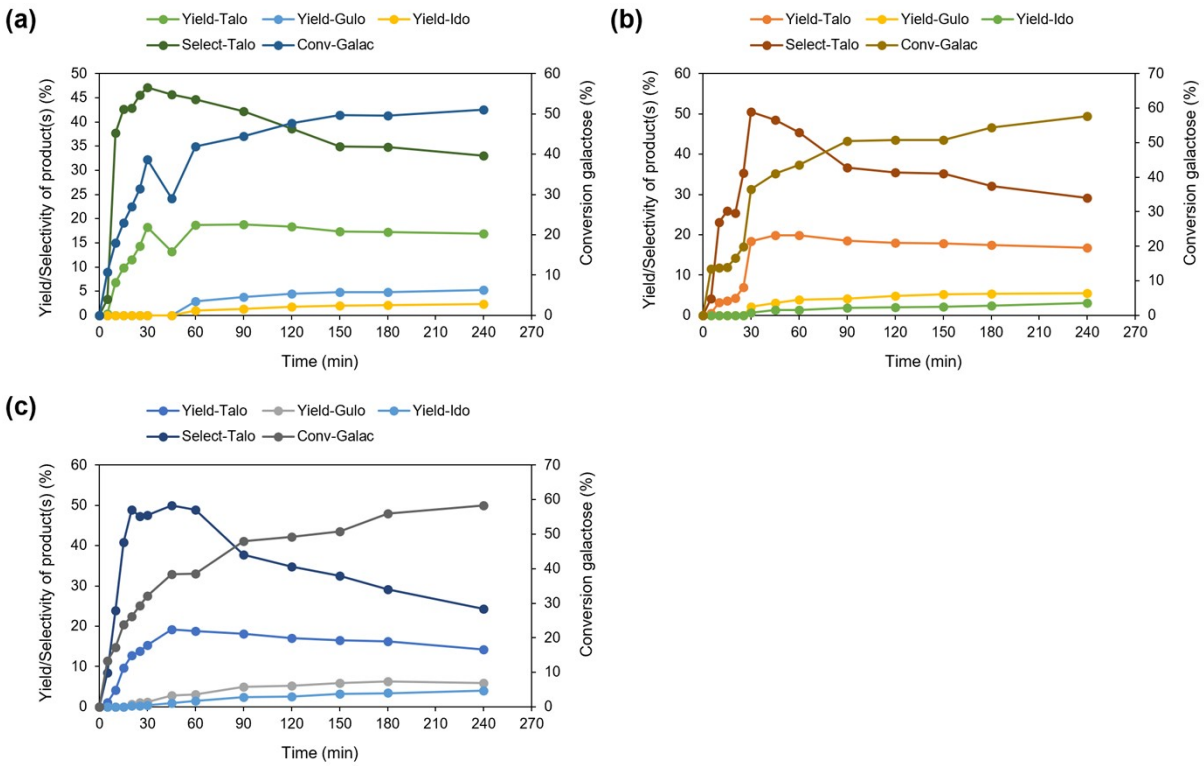

Figure S8. Galactose epimerization profile over commercial MoO_3 in a water medium under different catalyst loadings (w/w ratio of MoO_3 to galactose) at 100 °C: (a) 10%, (b) 15%, and (c) 20%.

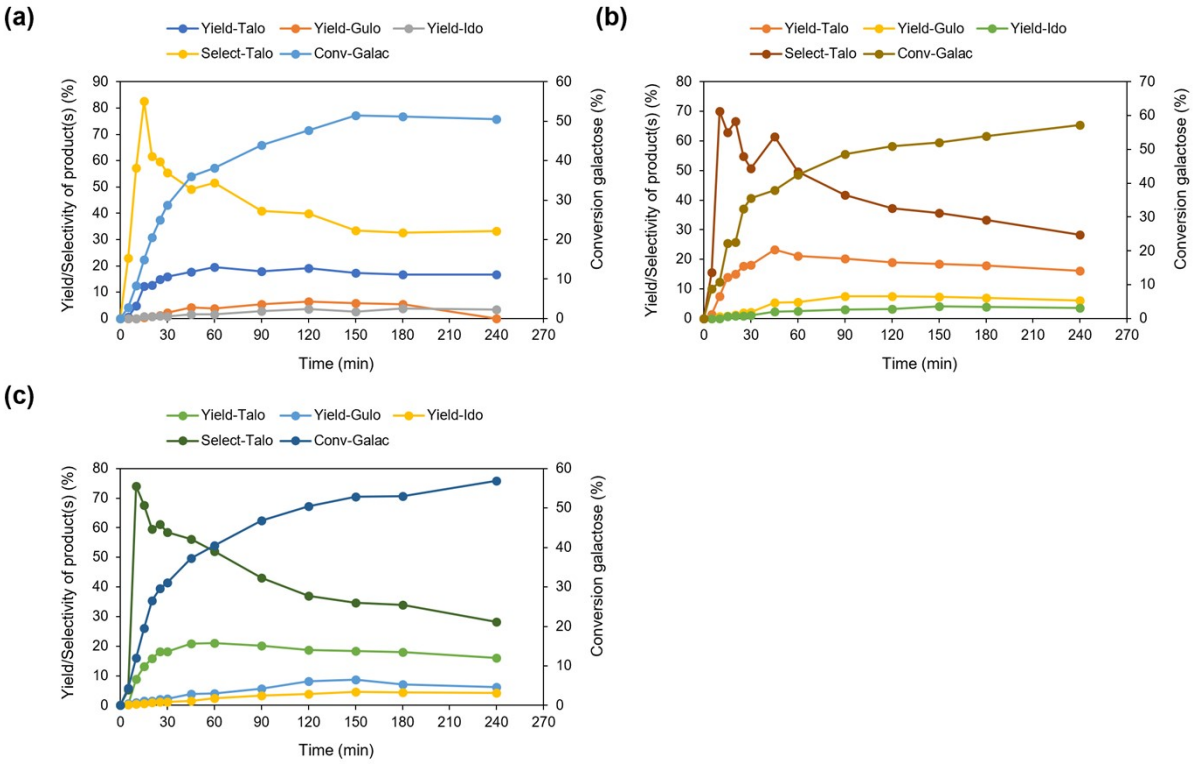

Figure S9. Galactose epimerization profile over commercial MoO_3 in a water medium under different catalyst loadings (w/w ratio of MoO_3 to galactose) at 110 °C: (a) 10%, (b) 15%, and (c) 20%.

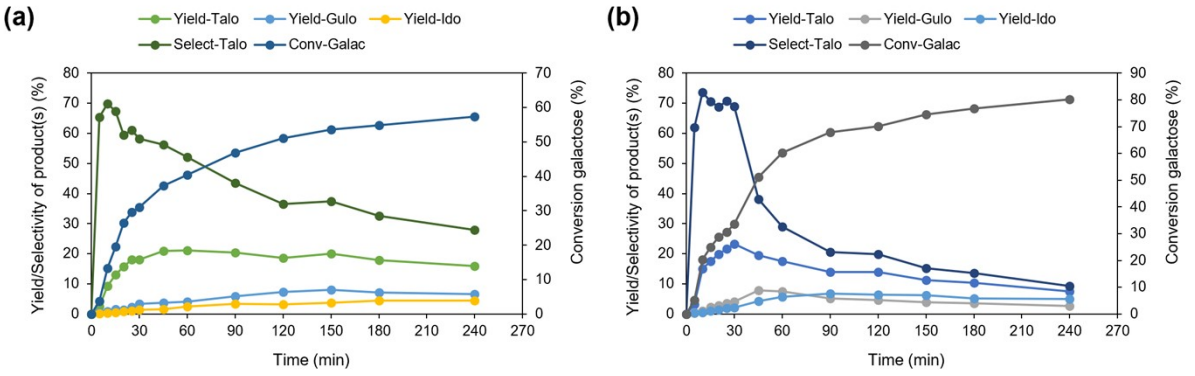

Figure S10. Galactose epimerization profile over commercial MoO_3 in a water medium at 15% wt. catalyst load on substrate under different temperature conditions: (a) 120 °C and (b) 130 °C.

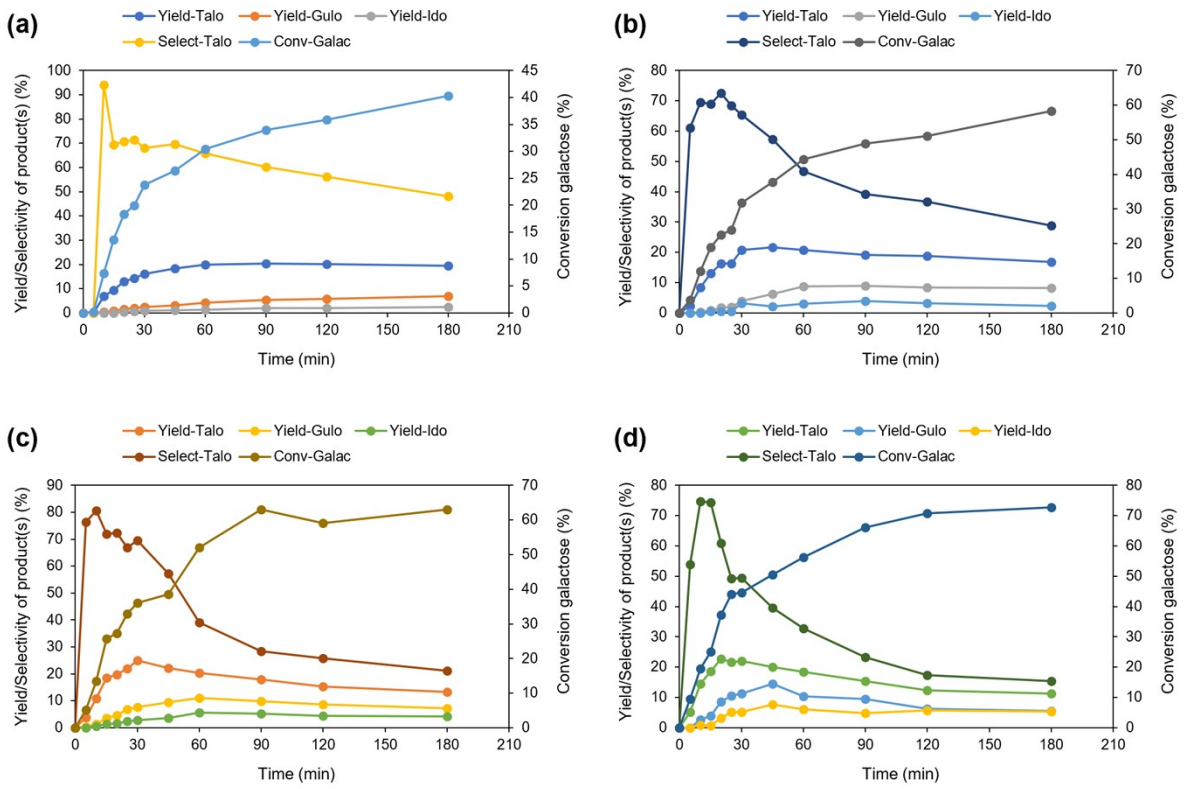

Figure S11. Galactose epimerization profile over acid-treated MoO_3 (MO-2M) in a water medium at 15% wt. catalyst load on substrate under different temperature conditions: (a) 100 °C, (b) 110 °C, (c) 120 °C and (d) 130 °C.

Table S1. Data of product(s) selectivity, yield and carbon balance of MO-2M catalyst under different temperature conditions.

Reaction Temperature. (°C)	S_{Tal} (%)	Y_{Gul} (%)	Y_{Ido} (%)	*TON*	*TOF*	*CB* (%)
100	68.1	2.4	0.7	1.0330	0.000573	82.2
110	65.3	3.9	2.1	1.4966	0.000831	88.1
120	69.5	7.5	2.7	1.8847	0.001048	98.0
130	49.3	11.2	5.2	2.0456	0.001136	86.0

S-selectivity, Y-yield, Tal-talose, Gul-gulose, Ido-idose and CB-carbon balance. TON-turn over number is calculated as moles of reactant consumed per mole of catalyst. TOF-turn over frequency is calculated as moles of reactant consumed per mole of catalyst per unit time.

Table S2. Comparative data of product(s) selectivity, yield and carbon balance of MoO_3 catalysts (both pristine and acid-treated) under optimum conditions.

Catalysts (MoO_3)	S_{Tal} (%)	Y_{Gul} (%)	Y_{Ido} (%)	*TON*	*TOF*	*CB* (%)
CMO	58.2	3.4	1.3	1.22063	0.000113	73.6
MO-1M	60.4	5.5	2.2	1.48016	0.000137	83.8
MO-2M	69.4	7.5	2.7	1.88472	0.000174	98.0
MO-3M	57.4	6.3	2.8	1.61417	0.000149	82.7

S-selectivity, Y-yield, Tal-talose, Gul-gulose, Ido-idose and CB-carbon balance. TON-turn over number is calculated as moles of reactant consumed per mole of catalyst. TOF-turn over frequency is calculated as moles of reactant consumed per mole of catalyst per unit time.

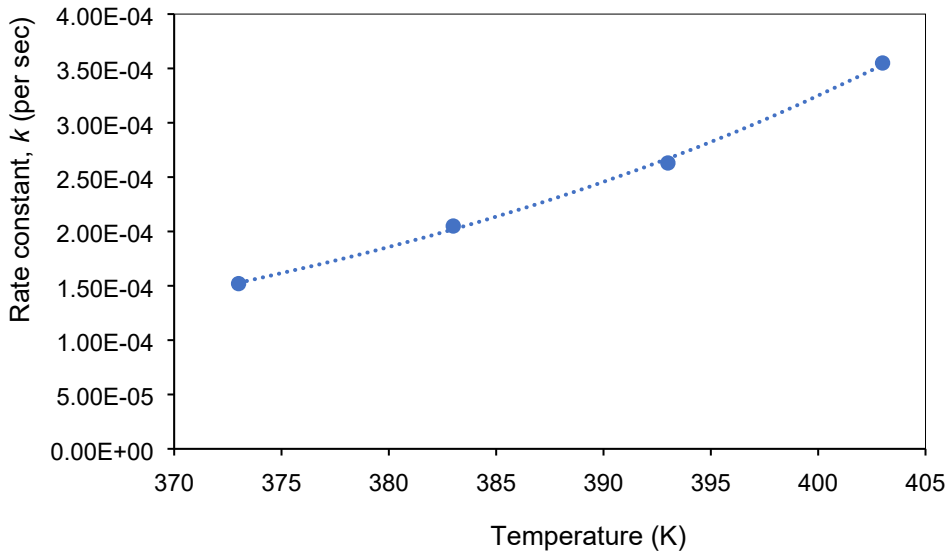

Figure S12. Correlation plot between first-order rate constant of galactose disappearance and temperature over acid treated MoO_3 (MO-2M) in a water medium at 15% wt. catalyst load on substrate.

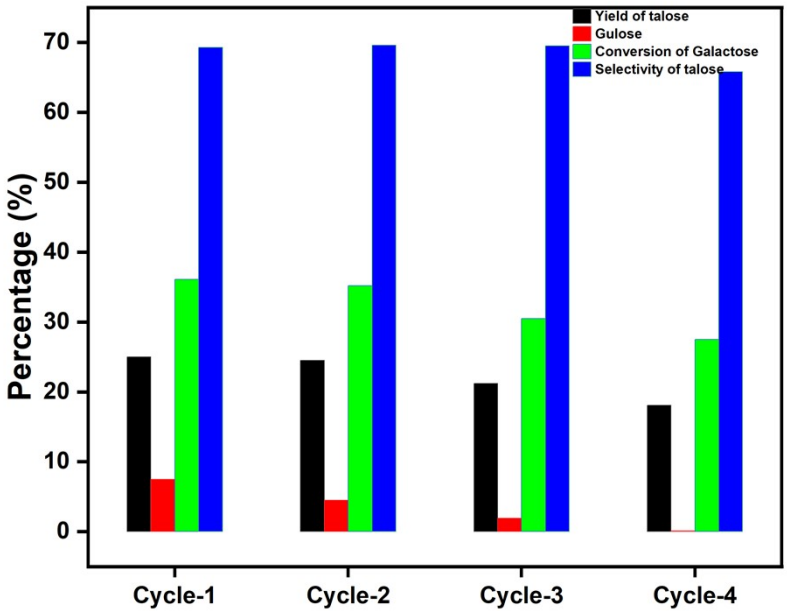

Figure S13. Recyclability result of MO-2M catalyst under optimum conditions in a water medium.

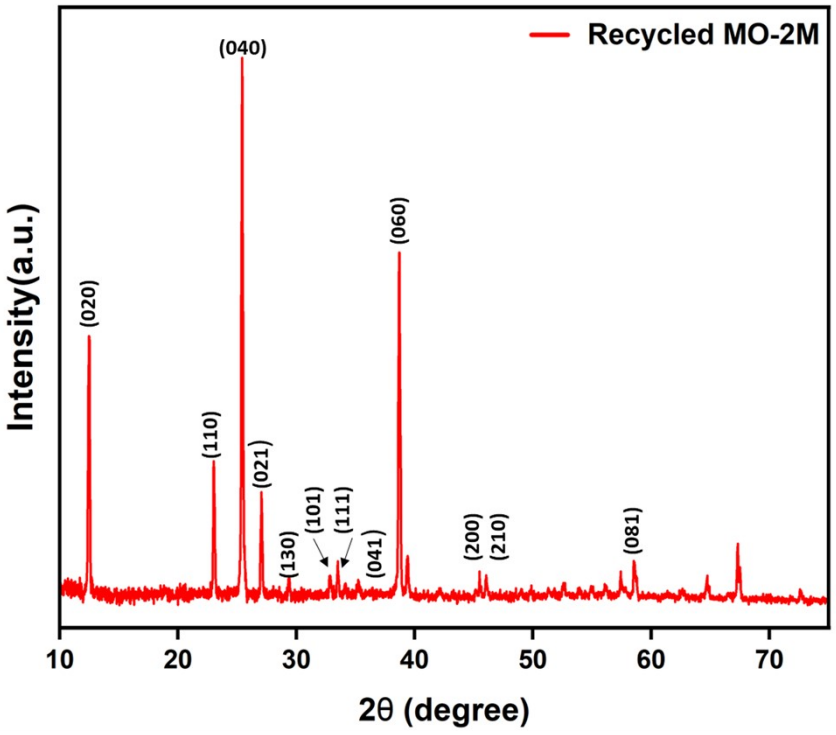

Figure S14. XRD pattern of recycled MO-2M catalyst after 4 runs.

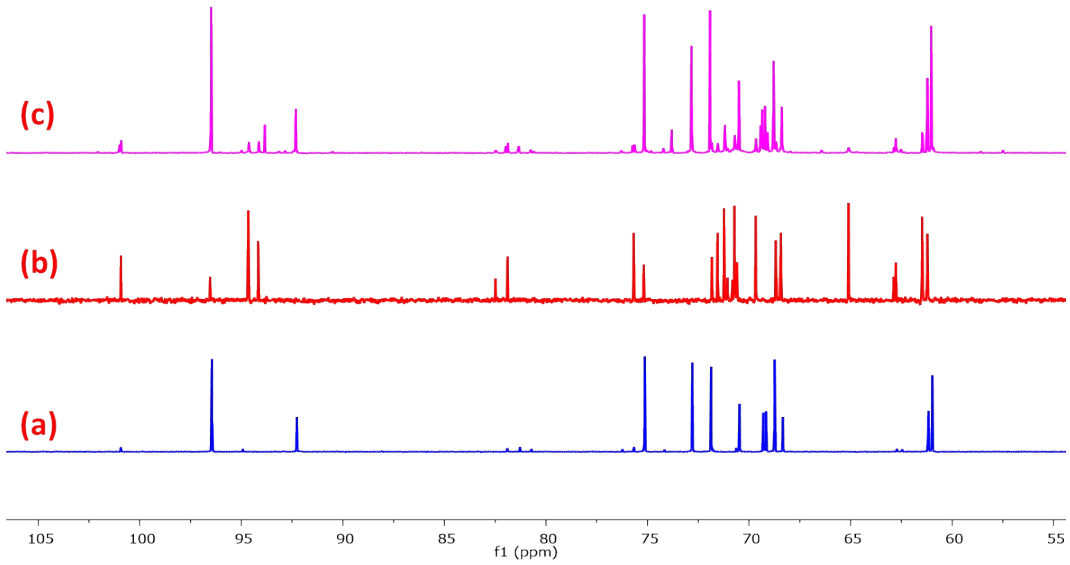

Figure S15. ^{13}C-NMR characterization of galactose epimerization over MO-2M catalyst: (a) standard galactose, (b) standard talose and (c) post reaction mixture obtained using D-galactose as a substrate under optimum conditions.

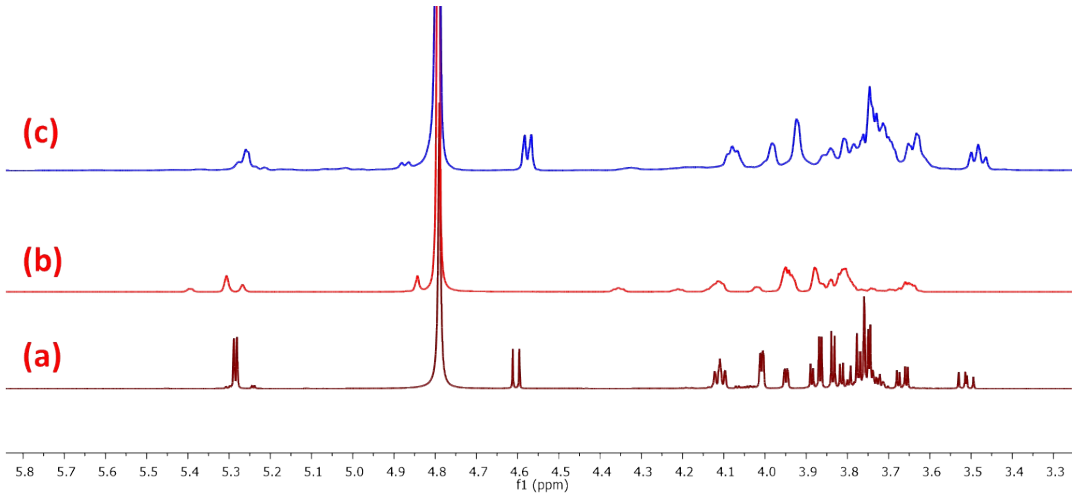

Figure S16. ^{1}H-NMR characterization of galactose epimerization over MO-2M catalyst: (a) standard galactose, (b) standard talose and (c) post reaction mixture obtained using D-galactose as a substrate under optimum conditions.

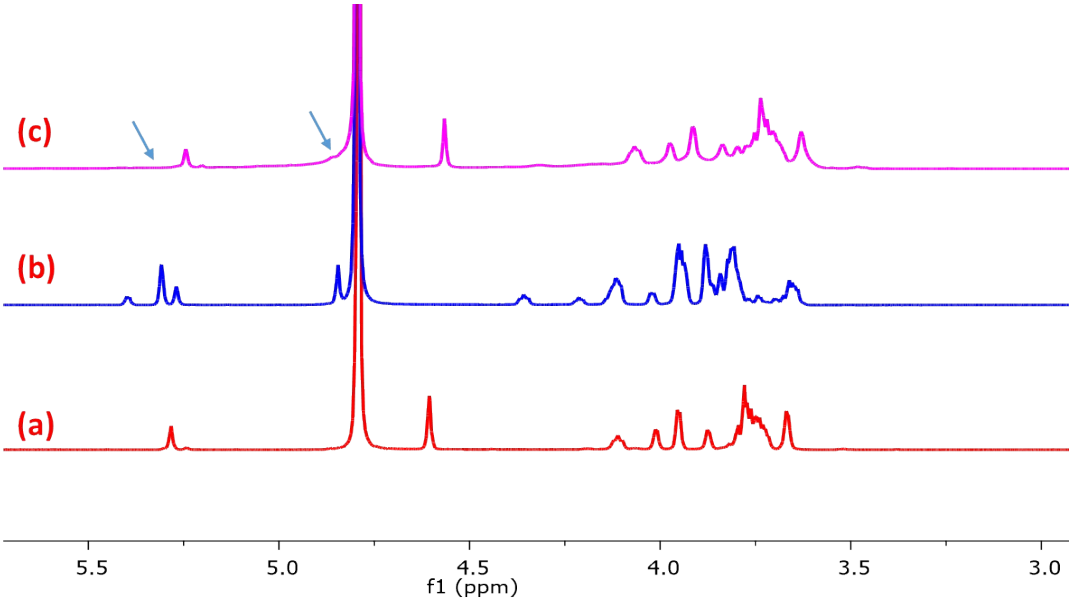

Figure S17. ^{1}H-NMR characterization of galactose epimerization over MO-2M catalyst; (a) standard D-(d^2)-galactose, (b) standard talose and (c) post reaction mixture obtained using D-(d^2)-galactose as a substrate under optimum conditions.