

**Electronic Supplementary Information**

**Synthesis of high surface area mixed metal oxide from NiMgAl LDH  
precursor for Nitro-aldol condensation reaction**

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### ***XRD Analysis:***

The diffraction peaks in case of NiMgAl mixed oxide (Fig 1 (ii)) were observed at  $2\theta = 16.4$  which can be attributed to (001) reflection of  $\theta$ - $\text{Al}_2\text{O}_3$  (JCPDS card No. 86-1410), 21.85, 55.05 and 64.95 are attributed to (200), (422) and (440) reflections, respectively of  $\text{MgAl}_2\text{O}_4$  (JCPDS card No. 77-1193 and 82-2424), 26.9 is attributed to (111) reflection of AlO (JCPDS card No. 75-0278), 30.9 is attributed to (220) reflection of  $(\text{Ni}_{0.198}\text{Al}_{0.802})(\text{Al}_{1.198}\text{Ni}_{0.802})\text{O}_4$  (JCPDS card No. 81-0718), 36.85 and 62.1 are attributed to (111) and (220) reflections of MgO (JCPDS card No. 89-7746 and 79-0612), 44.1 is attributed to (012) reflection of NiO (JCPDS card No. 89-7390), 48.85 is attributed to (133) reflection of  $\kappa$ - $\text{Al}_2\text{O}_3$  (JCPDS card No. 88-0107) and 52.7 is attributed to (511) reflection of  $\text{Mg}_{0.36}\text{Al}_{2.44}\text{O}_4$  (JCPDS card No. 77-0729). The diffraction peaks in case of CoMgAl mixed oxide (Fig 1 (ii)) were observed at  $2\theta = 12$  is attributed to (110) reflection of  $\text{Al}_{12}\text{Mg}_{17}$  (JCPDS card No. 73-1148), 22.2 and 43.45 are due to  $\text{Co}_6\text{Al}_2\text{O}_{11}$  (JCPDS card No. 51-0041), 25.1 is attributed to (012) reflection of  $\text{Al}_2\text{O}_3$  (JCPDS card No. 89-3072), 35.55 is attributed to (222) reflection of  $\text{Mg}_{0.36}\text{Al}_{2.44}\text{O}_4$  (JCPDS card No. 77-0729) and 62.6 is attributed to (112) reflection of CoO (JCPDS card No. 65-5474).

**Table S1**

Textural properties of mixed oxides.

Entry	Catalysts	$S_{\text{BET}}$ (m <sup>2</sup> /g)	Pore Volume (cm <sup>3</sup> /g)	Pore Diameter (Å)
1	MgAl (O)	561	0.974	88
2	NiMgAl (O)	753	1.279	49.6
3	CoMgAl (O)	401	0.937	107

**Table S2**

Effect of calcination temperature on Nitro-aldol condensation reaction of 4-nitrobenzaldehyde and nitromethane catalyzed by NiMgAl mixed oxide <sup>a</sup>

Entry	Calcination Temperature (°C)	Time (h)	Conversion (%) <sup>b</sup>
1	350	6	84
2	450	2	99
3	550	8	86

<sup>a</sup>Reactions were carried out with 1:10 molar ratio in a 1 mmol scale of 4-nitrobenzaldehyde and nitromethane using 10 mg catalyst

<sup>b</sup>Determined from <sup>1</sup>H NMR data of crude mixture

**Table S3**  
**Recyclability of the catalyst**

Entry	No. of cycle	Time(h)	Conversion(%) <sup>a</sup>
1	1 <sup>st</sup> run	2	96
2	2 <sup>nd</sup> run	3	95
3	3 <sup>rd</sup> run	4	92

<sup>a</sup> Determined from <sup>1</sup>H NMR data of crude mixture

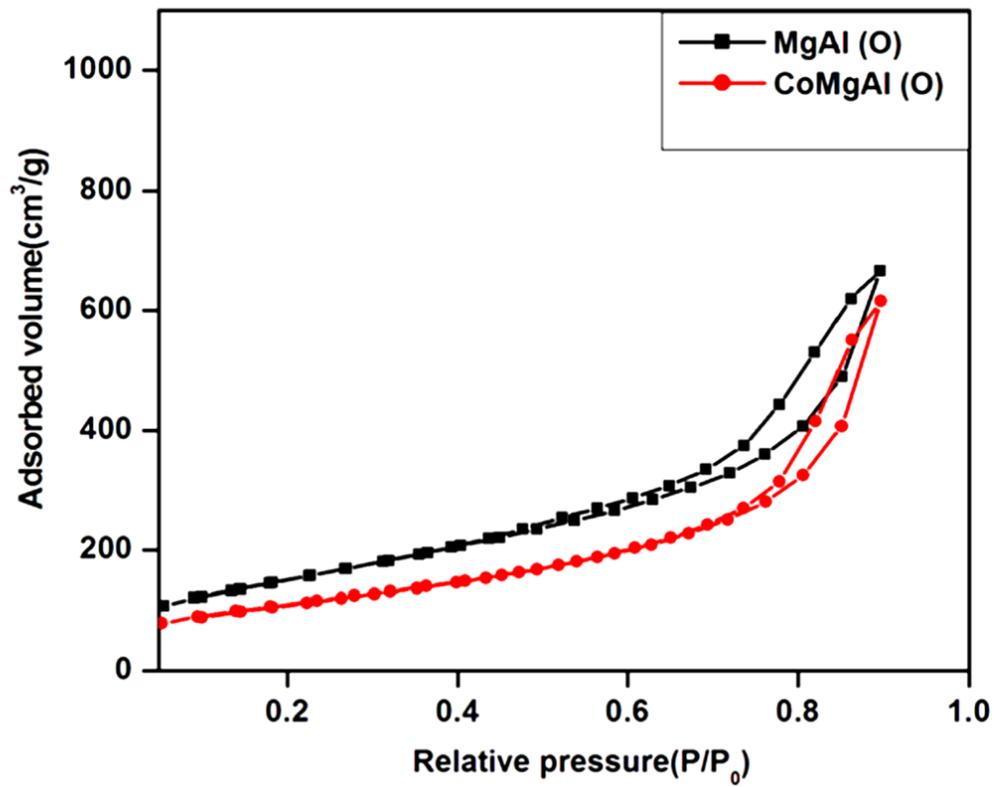


Fig. S1 Nitrogen-sorption isotherms of MgAl and CoMgAl mixed oxides.

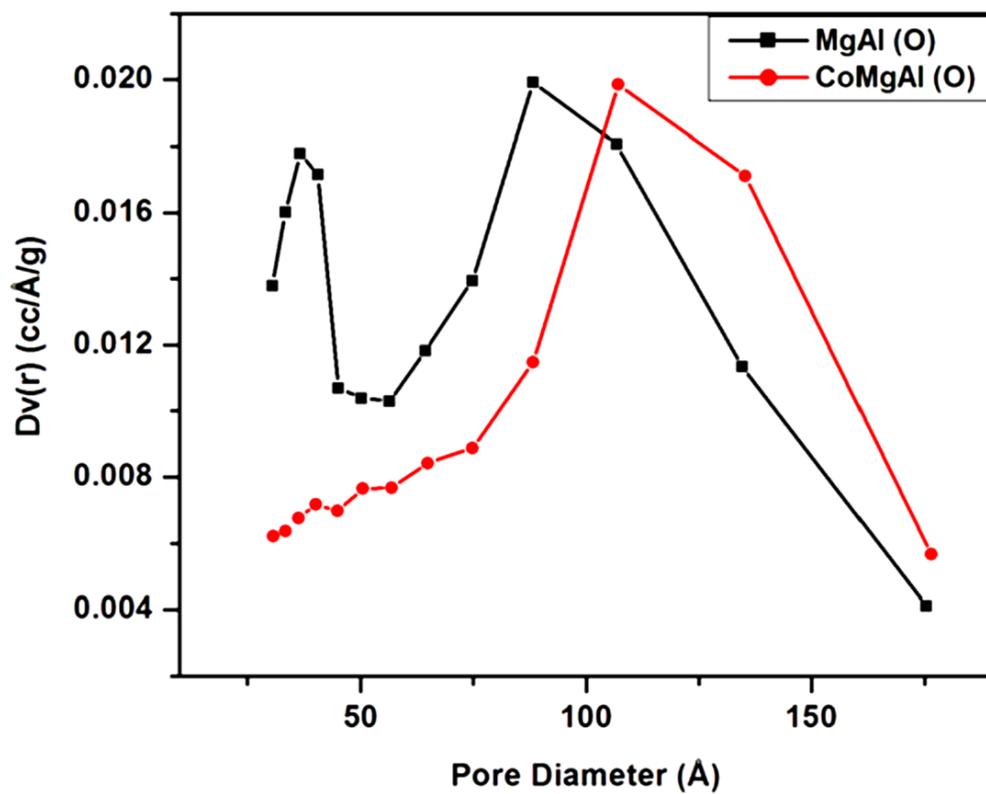


Fig. S2 Pore size distribution curve of MgAl LDH and CoMgAl mixed oxides.

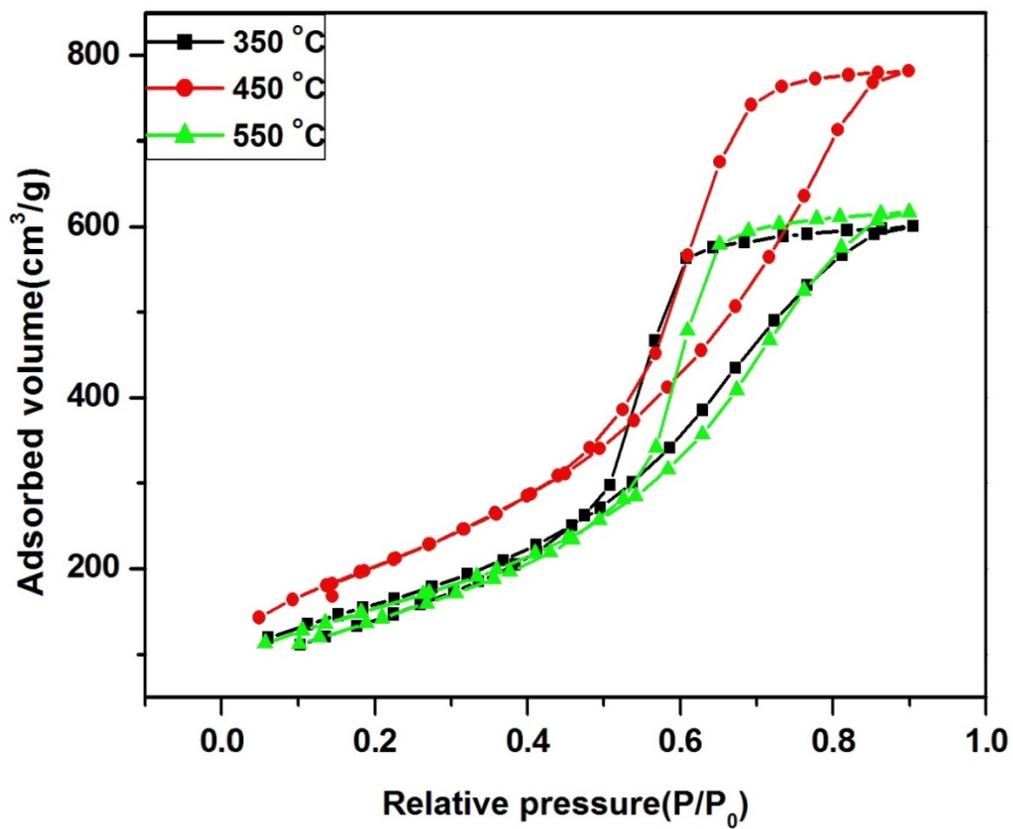


Fig. S3 N<sub>2</sub> adsorption-desorption graphs of NiMgAl (O) at different calcination temperature.

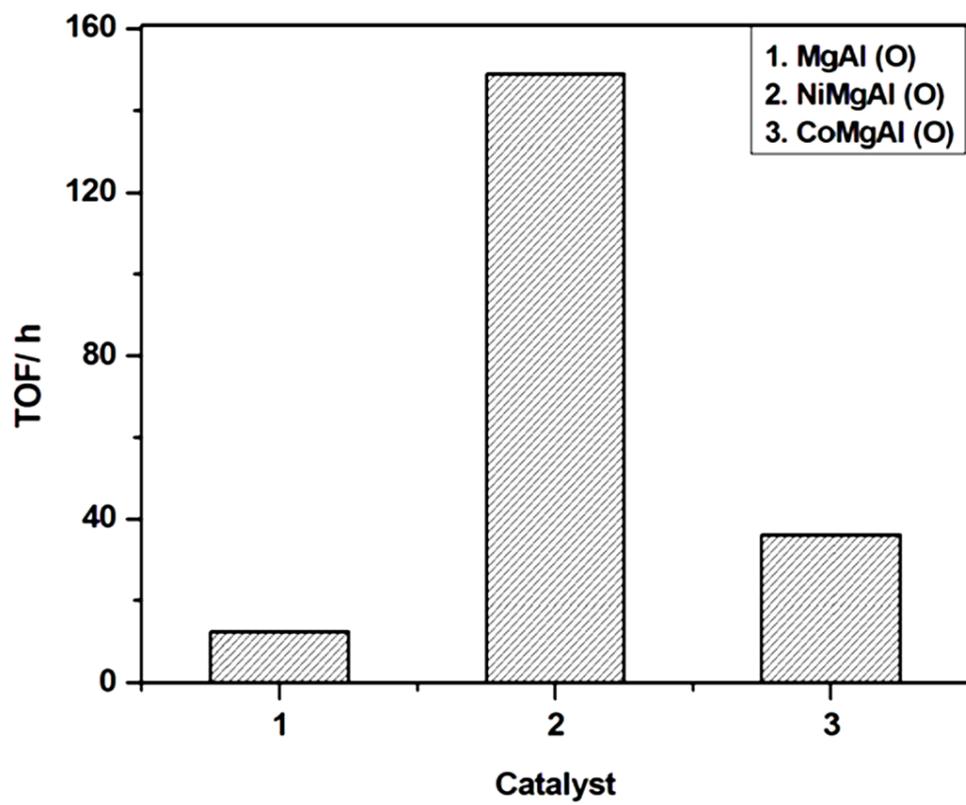


Fig. S4 Turnover frequency (TOF) of mixed metal oxides.



