Electronic Supplementary Material (ESI) for Catalysis Science & Technology.

Supporting Information

Efficient method for the cyclopentanone synthesis from furfural: Understanding the role of solvents, solubilities and bimetallic catalytic system

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1.0 Catalytic activity

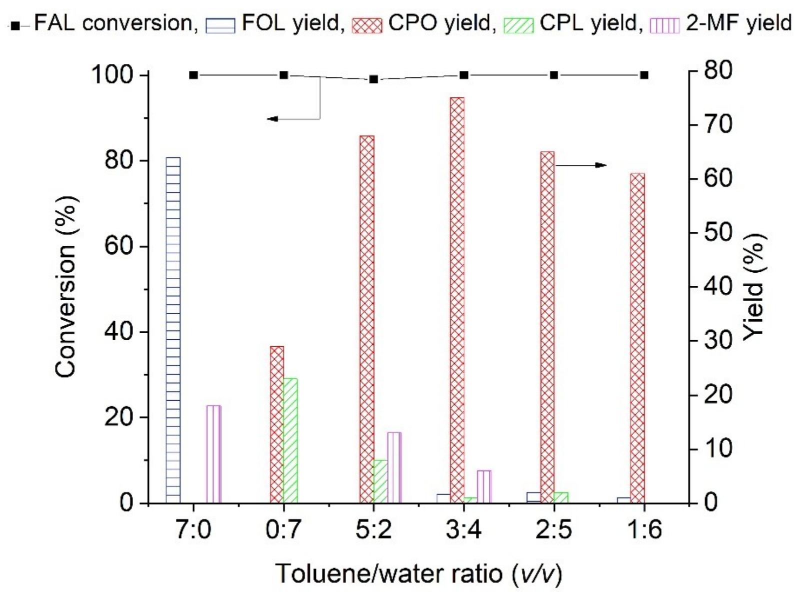

Figure S1. Effect of varying toluene/water ratio (*v/v*) on the conversion of FAL to CPO **Reaction condition**: FAL, 0.35 g; Pt(3)Co(3)/C, 0.078 g; toluene/water ratio (*v/v*), 35 mL; 180 °C; 5 h; 900 rpm; 1 MPa H_2 at room temperature; (FAL: furfural, FOL: Furfuryl alcohol, CPO: cyclopentanone, CPL: cyclopentanol, 2-MF: 2-methylfuran)

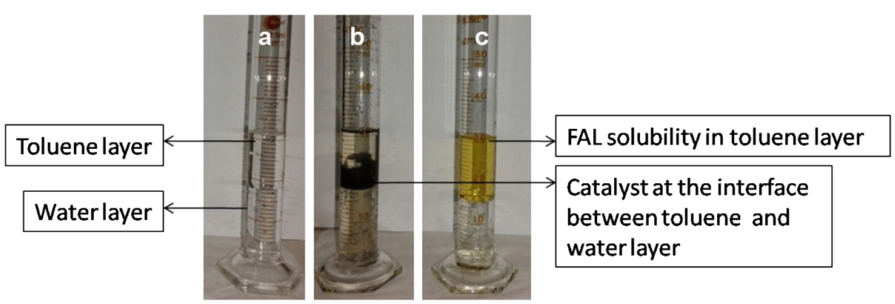

Figure S2. a) distribution of catalyst at the interface between water and toluene layer b) solubility of FAL in toluene layer c) toluene and water biphasic solvent system; (FAL: furfural)

Table S1. Adsorption of substrate and product in biphasic toluene/water 3:4 (v/v), 35 mL solvent system

Chemical	FAL	FOL	CPO	CPL
Adsorption (%)	5	7	3	5

Conditions: Substrate and product 0.35 g,toluene/water 3:4 (*v/v*), 35 mL; 28 °C;600 rpm; 1h;carbon 0.078 g; (FAL: furfural, FOL: furfuryl alcohol, CPO: cyclopentanone, CPL: cyclopentanol)

Table S2. Role of active metal for conversion of cyclopentanol to cyclopentanone

Catalyst	Substrate	CPL/CPO conversion (%)	Yield (%)	
			CPO	CPL
Co(3)/C	CPL	10	2	0
Co(3)/C	CPO	12	0	3
Pt(3)/C	CPL	93	73	0
Pt(3)/C	CPO	15	0	4

Reaction condition: CPO, CPL, 0.35 g; Catalyst, 0.078 g; toluene/water 3:4 *v/v*, 35 mL; 180 °C; 5 h; 900 rpm; 1 MPa H_2, at room temperature

Table S3. Activity of FAL and FOL in water medium

Substrate	FAL/FOL conversion (%)	Yield (%)		
		CPO	CPL	1,2-pentanediol
FAL	100	29	23	0
FOL	100	9	30	5

Reaction condition – FAL, FOL, 0.35 g; Pt(3)Co(3)/C, 0.078 g; water 35 mL; 180 °C; 5 h; 900 rpm; 1 MPa H_2, at room temperature (FAL: furfural, FOL: furfuryl alcohol, CPO: cyclopentanone, CPL: cyclopentanol).

1.1 Effect of H_2 pressure

In order to see the effect of H_2 pressure reactions were done with varying pressure from 0.1-1 MPa (Figure S6, ESI). From the results it was found that as the H_2 pressure decrease from 1 to 0.5 MPa the FAL conversion decrease to 64% with 7% CPO, 8% FOL and 7% CPL yield. Further decrease in H_2 pressure to 0.1 MPa, the FAL conversion decreases to 48% with only 1% FOL formation. It shows that lower pressure of H_2 is not sufficient for the conversion of FAL to FOL leading to side reactions.[37] At 1 MPa H_2, conversion of FAL is complete with 75% yield of CPO. It shows that the conversion of FAL and yield of CPO is depending on H_2 pressure because with increase in H_2 pressure solubility of H_2 increases and also interaction between catalyst and substrate also increases which ultimately improve the yield of CPO.

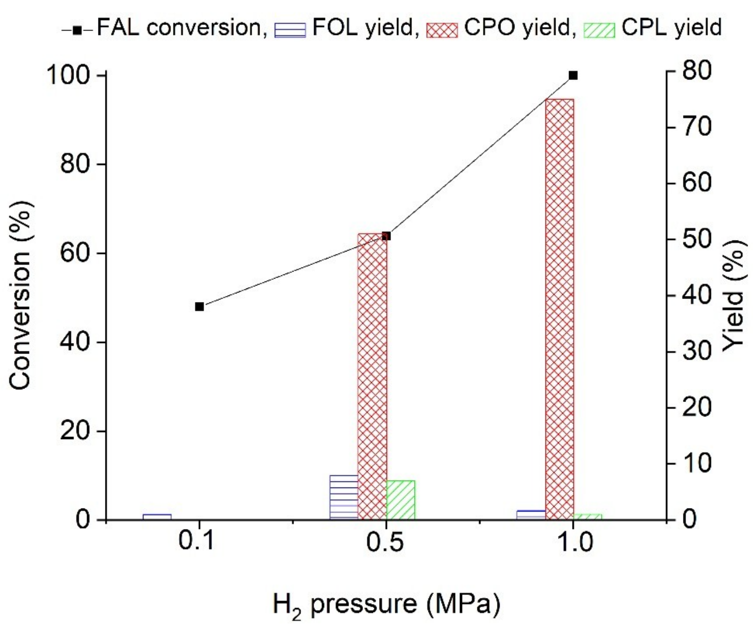

Figure S3 Effect of H_2 pressure

Reaction condition: FAL, 0.35 g; Pt(3)Co(3)/C, 0.078 g; toluene/water 3:4 (*v/v*), 35 mL; 180 °C; 5 h; 900 rpm (FAL: furfural, FOL: furfuryl alcohol, CPO: cyclopentanone, CPL: cyclopentanol).

1.2 Effect of catalyst loading

Effect of catalyst loading was studied to know the optimized loading of catalyst required for achieving maximum yield of CPO. Reactions were carried out with 0.02, 0.04, 0.08 g catalyst loading as shown in Figure S7, (ESI). With 0.02 g loading of catalyst, 80% conversion achieved but yield of FOL is 6% and CPO is 2%. With 0.04 g catalyst loading, the conversion is nearly same to that of earlier loading but the yield of CPO reaches to 22% with 1% FOL formation. When loading of catalyst increases to 0.08 g the yield of CPO achieved was 75% with 2% FOL and 1% CPL yield and complete conversion of FAL was seen. This shows that as the loading of catalyst increases, availability of active metal sites increases for the conversion of FAL to CPO which suppress the side reaction leading to increases in the yield of CPO.

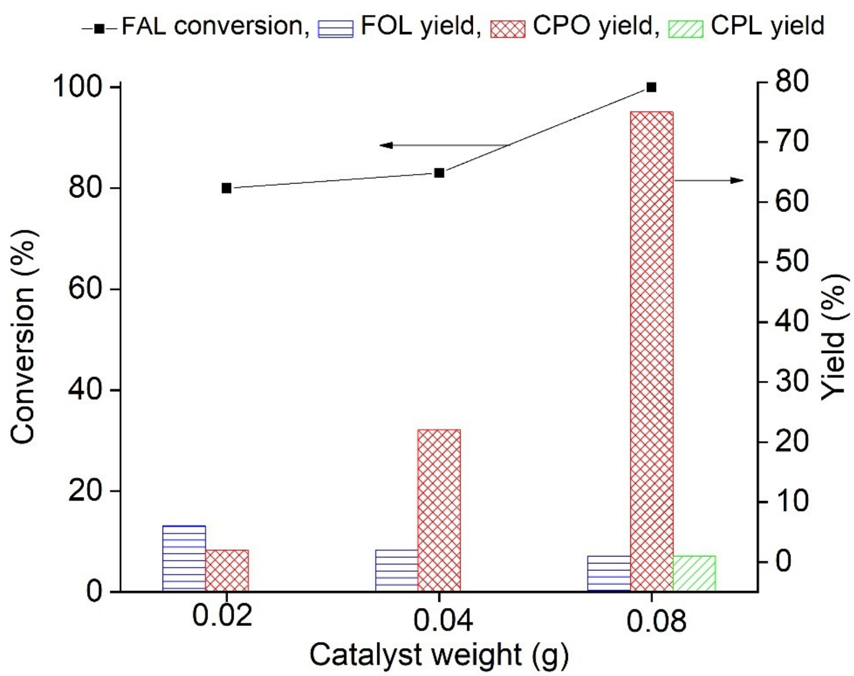

Figure S4. Effect of catalyst loading
Reaction condition: FAL, 0.35 g; Pt(3)Co(3)/C; toluene/water 3:4 *v/v*, 35 mL; 180 °C; 5 h; 900 rpm; 1 MPa H_2, at room temperature (FAL: furfural, FOL: furfuryl alcohol, CPO: cylclopentanone, CPL: cyclopentanol).

1.3 Effect of stirring speed

To investigate the effect of stirring speed under reaction condition, reactions were carried out with varying stirring speed (Figure S8, ESI). It was found out that at 300 rpm; conversion of FAL is 91% with 20% CPO and 10% FOL yield. At 600 rpm, 95% conversion with 36% yield of CPO and at 900 rpm, 100% conversion with 75% yield of CPO was obtained. Increase in stirring speed from 300 to 900 rpm the interaction between substrate, catalyst and diffusion of H_2 from gas to liquid phase increases which suppress the side reactions hence improve the yield of CPO.

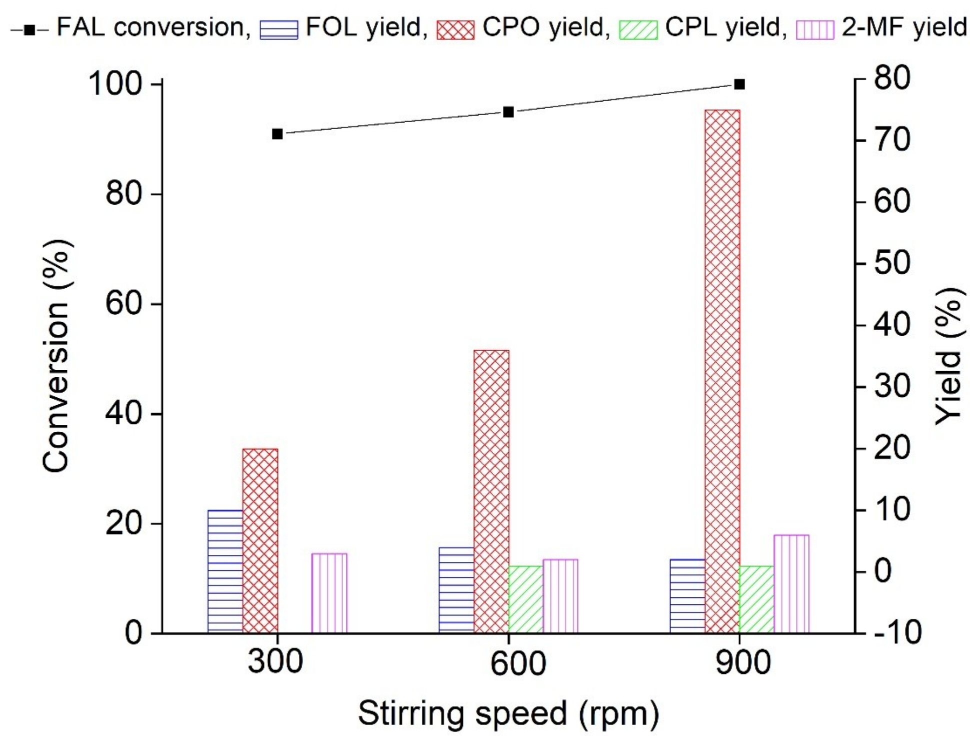

Figure S5. Effect of stirring speed

Reaction condition: FAL, 0.35 g; Catalyst, 0.078 g; toluene/water 3:4 *v/v*, 35 mL; 180 °C; 5 h; 1 MPa H_2 at room temperature (FAL: furfural, FOL: furfuryl alcohol, CPO: cyclopentanone, CPL: cyclopentanol, 2-MF: 2-methylfuran).

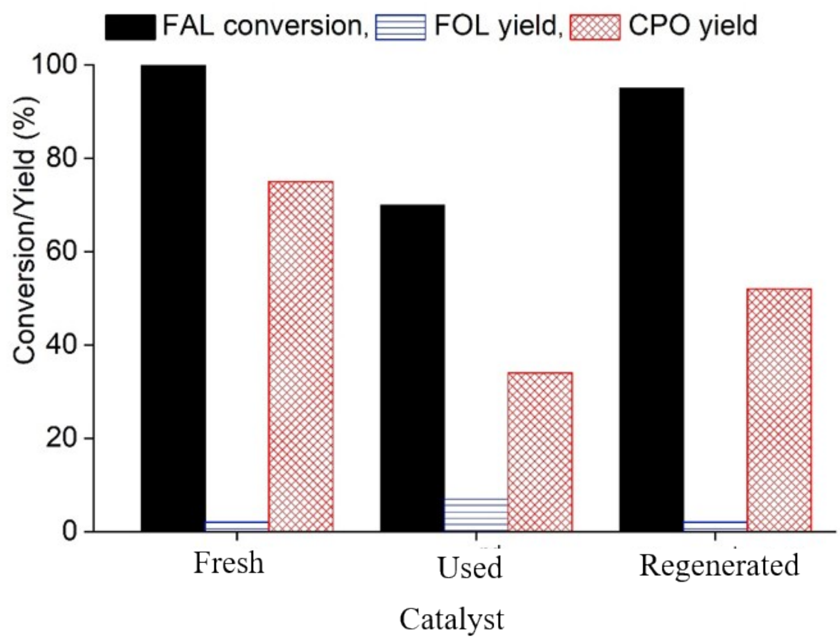

Figure S6 Recycle study of catalyst

Reaction condition: FAL, 0.35 g; Catalyst, 0.078 g; toluene/water 3:4 (*v/v*), 35 mL; 180 °C; 5 h; 900 rpm; 1 MPa H_2 at room temperature (FAL: furfural, FOL: furfuryl alcohol, CPO: cyclopentanone)

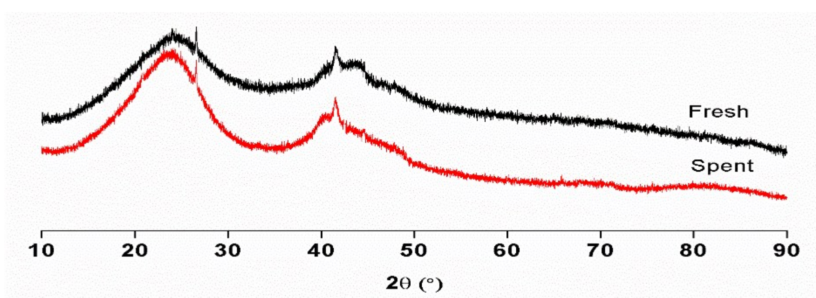

Figure S7. XRD of fresh and spent catalyst

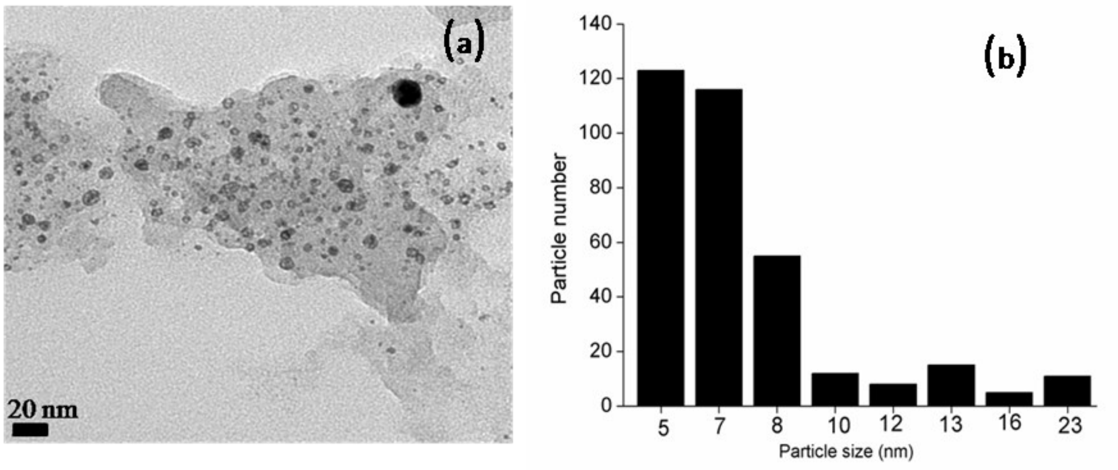

Figure S8. a) TEM image of spent catalyst, b) particle size distribution of spent catalyst