

Bio-derived ZnO nanoflower: A highly efficient catalyst for synthesis of chalcones derivatives

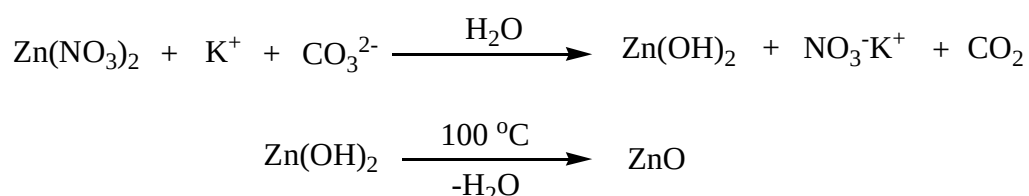
Chandan Tamuly^{a*}, Indranirekha Saikia^a, Moushumi Hazarika^a, Manobjyoti Bordoloi^b,
Najrul Hussain^c, Manash R Das^c

^a Natural Product Chemistry Section, CSIR-North East Institute of Science and Technology. Branch Itanagar, Arunachal Pradesh, India

^b Natural Product Chemistry Division, CSIR-North East Institute of Science and Technology. Jorhat, Assam-785006, India

^c Materials Science Division, CSIR-North East Institute of Science and Technology. Jorhat, Assam-785006, India

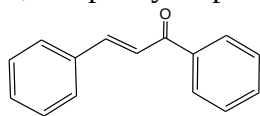
e-mail: c.tamuly@gmail.com,



Scheme 1S: Plausible mechanism in synthesis of ZnO nanoparticles by using peel extract of *Musa balbisiana*

Scheme 2S: Claisen Schmidt Condensation reaction

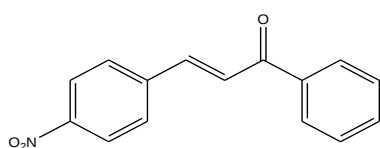
1) 1,3-Diphenyl-1-phenylpropenone



¹H NMR (CDCl₃, 300 MHz) δ 8.04 (dd, *J* = 8.5, 1.9 Hz, 2H), 7.84 (d, *J* = 15.7 Hz, 1H), 7.64 (dd, *J* = 7.7, 3.8 Hz, 2H), 7.59-7.52 (m, 4H), 7.43-7.41 (m, 3H)

¹³C NMR (CDCl₃, 75 MHz) 188.2, 145.3, 137.1, 135.5, 134.2, 129.8, 129.1, 128.6, 128.0, 126.9, 125.3, 121.1

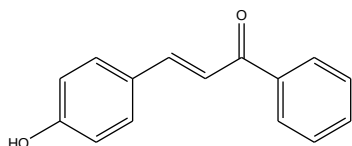
2) 3-(4-nitrophenyl)-1-phenylpropenone



¹H NMR (CDCl₃, 300 MHz) δ 8.14 (d, *J* = 4.5 Hz, 2H), 8.04 (d, *J* = 15.7 Hz, 1H), 7.84 (d, *J* = 3.8 Hz, 1H), 7.59-7.52 (m, 2H), 7.56 (d, *J* = 3.8 Hz, 2H), 7.43-7.41 (m, 3H)

¹³C NMR (75 MHz) δ 190.2, 148.4, 145.2, 141.6, 138.1, 134.1, 130.1, 129.4, 127.4, 121.6, 121.1

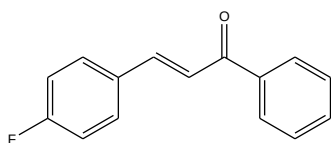
3) 3-(4-hydroxyphenyl)-1-phenylpropenone



¹H NMR (CDCl₃, 300 MHz) δ 7.92 (d, *J* = 4.5 Hz, 1H), 7.54 (d, *J* = 15.7 Hz, 1H), 7.76-7.81 (m, 2H), 7.45-7.54 (m, 3H), 7.14 (d, *J* = 3.8 Hz, 2H), 6.64 (d, *J* = 4.8 Hz, 2H),

¹³C NMR (75 MHz) δ 189.4, 157.3, 145.3, 137.6, 134.5, 130.2, 129.7, 129.4, 127.6, 127.4, 115.1

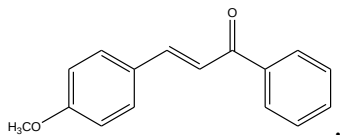
4. 3-(4-fluorophenyl)-1-phenylpropenone



¹H NMR (CDCl₃, 300 MHz) δ 7.91 (d, *J* = 4.2 Hz, 1H), 7.56 (d, *J* = 15.7 Hz, 1H), 7.80 (m, 2H), 7.45-7.54 (m, 3H), 7.24 (d, *J* = 3.2 Hz, 2H), 6.90 (d, *J* = 4.4 Hz, 2H),

^{13}C NMR (75 MHz) δ 189.1, 162.3, 145.3, 138.6, 134.5, 130.2, 129.7, 129.4, 128.1, 127.4, 115.6

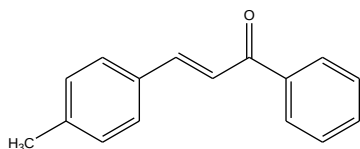
5. 3-(4-methoxyphenyl)-1-phenylpropenone



^1H NMR (CDCl_3 , 300 MHz) δ 7.91 (d, J = 4.2 Hz, 1H), 7.56 (d, J = 12.7 Hz, 1H), 7.80 (m, 2H), 7.45-7.54 (m, 3H), 7.18 (d, J = 3.2 Hz, 2H), 6.70 (d, J = 4.4 Hz, 2H), 3.5(s, 3H)

^{13}C NMR (75 MHz) δ 189.1, 160.1, 145.1, 138.1, 134.5, 130.2, 129.7, 129.4, 128.1, 127.4, 114.6, 60.1

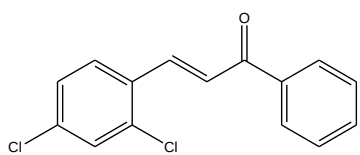
6. 3-(4-methylphenyl)-1-phenylpropenone



^1H NMR (CDCl_3 , 300 MHz) δ 7.88 (d, J = 4.6 Hz, 1H), 7.56 (d, J = 10.8 Hz, 1H), 7.80 (m, 2H), 7.45-7.54 (m, 3H), 7.18 (d, J = 4.2 Hz, 2H), 7.02 (d, J = 6.4 Hz, 2H), 2.4(s, 3H)

^{13}C NMR (75 MHz) δ 189.1, 145.3, 137.6, 137.1, 134.5, 130.1, 129.7, 129.4, 128.1, 127.4, 114.6, 25.1

7. 3-(2, 4-chlorophenyl)-1-phenylpropenone



^1H NMR (CDCl_3 , 300 MHz) δ 7.88 (d, J = 4.2 Hz, 1H), 7.56 (d, J = 12.8 Hz, 1H), 7.80 (m, 2H), 7.45-7.54 (m, 3H), 7.22 (s, 1H), 7.16 (d, J = 4.4 Hz, 1H), 7.10(d, J =3.4 Hz, 1H)

^{13}C NMR (75 MHz) δ 189.4, 145.1, 137.8, 137.1, 134.9, 134.5, 132.4, 131.1, 130.3, 129.8, 129.4, 129.1, 127.4

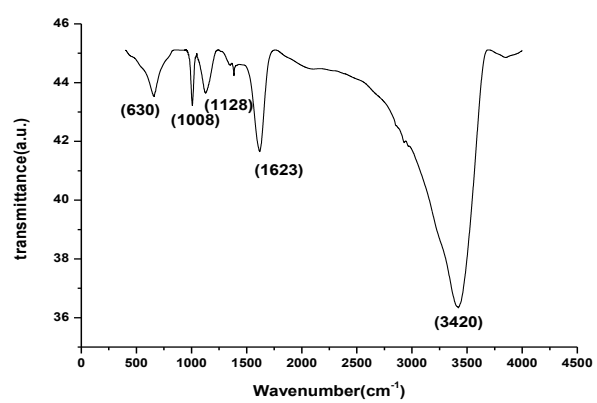


Figure 1S: FTIR spectra of ZnO nanoparticles

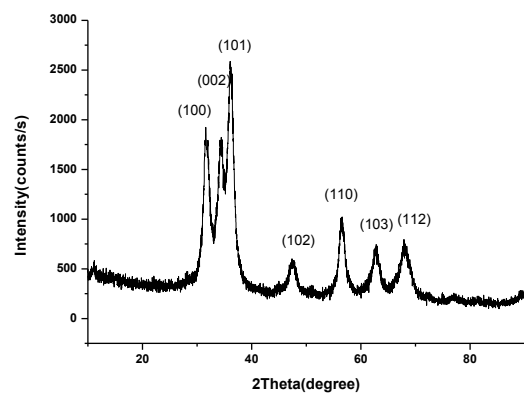


Figure 2S: XRD spectra of ZnO nanoparticles synthesized by K_2CO_3

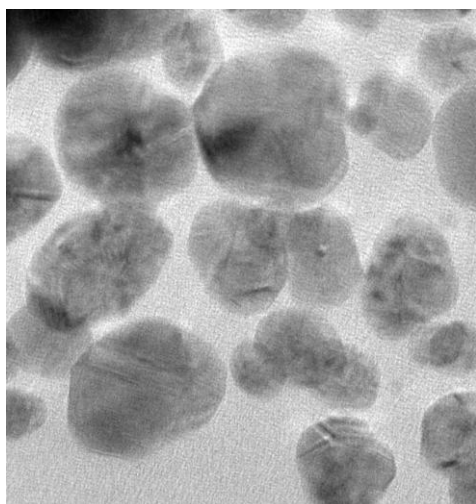


Figure 3S: TEM image of ZnO nanoparticles synthesized by K_2CO_3

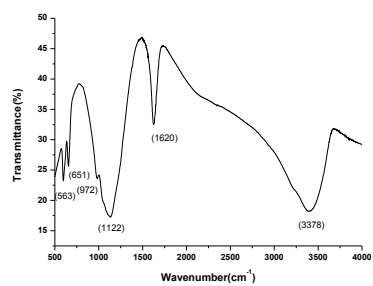


Figure 4S: FTIR spectra of ZnO nanoparticles synthesized by K_2CO_3

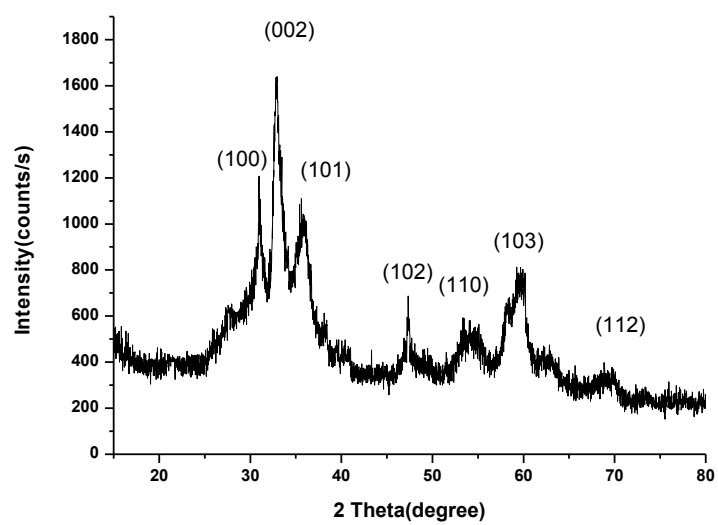


Figure 5S: XRD spectrum of ZnO catalyst after 5 recycle

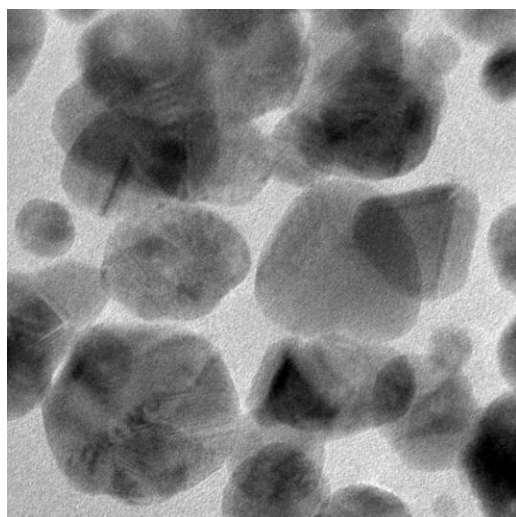


Figure 6S: TEM image after 5 recycle of ZnO nanocatalyst

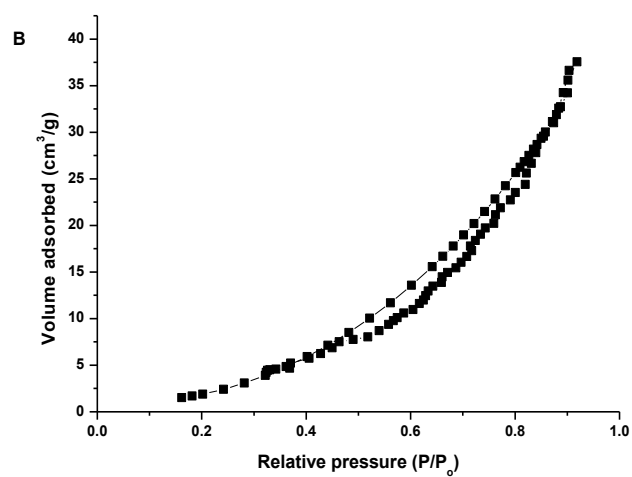
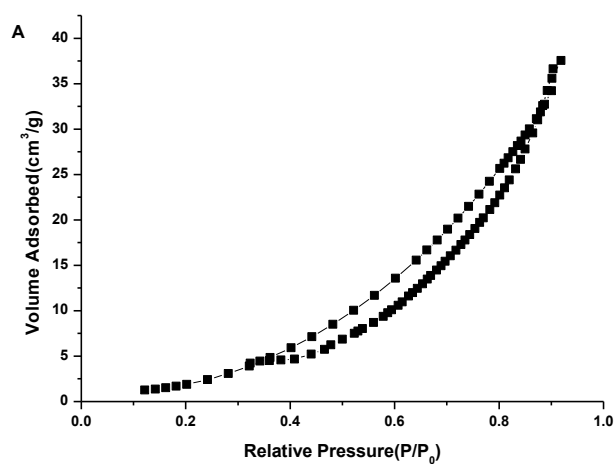


Figure 7S: The stacking pattern of N_2 adsorption desorption curves (A) fresh catalyst (B) after 5th cycle

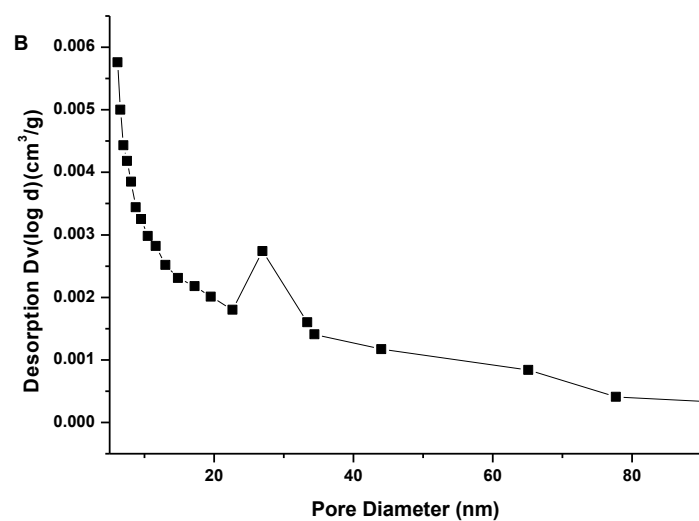
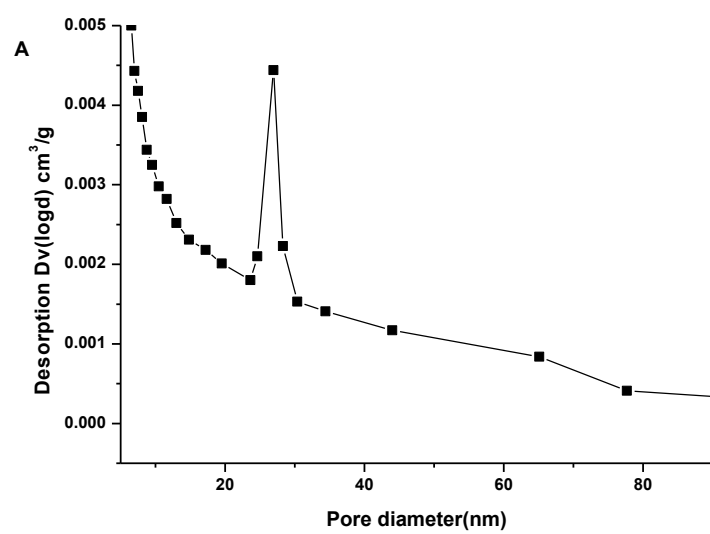


Figure 8S: BJH pore distribution curves of ZnO nanocatalyst (A) Fresh catalyst (B) After 5th cycle

Table 1S: Claisen Schmidt Condensation reaction conditions

Entry	Catalyst	Concentration	Temp (°C)	Time ^a (min)	Yields ^b (%)	TON	TOF(h ⁻¹)
1	-----	-----	80	30	0	0	0
2	-----	-----	100	60	0	0	0
3	ZnSO ₄	10 mol%	100	20	40	0.4	1.21
4	ZnSO ₄	5 mol%	100	15	58	1.16	4.64
5	Zn(NO ₃) ₂	10 mol%	100	15	40	0.40	1.6
6	Zn(NO ₃) ₂	5 mol%	100	10	48	0.96	5.78
7	ZnCl ₂	10 mol%	100	20	56	1.12	3.36
8	ZnCl ₂	5 mol%	100	10	60	1.20	7.22
9	ZnO nanopowder (Commercial)	10 mol%	100	10	70	0.70	4.20
10	ZnO nanopowder (Commercial)	5 mol%	100	8	72	1.42	10.65
11	ZnO nanocatalyst	10 mol%	100	5	84	0.9	10.84
12	ZnO nanocatalyst	5 mol%	100	0.8	98	1.96	147.36
13	ZnO nanocatalyst	3 mol%	100	4	90	3.0	50.4
14	ZnO nanocatalyst	1 mol%	100	5	86	8.6	103.6

^a Reactions performed at 80°C & 100°C and monitored using TLC until all the aldehyde and acetophenone was found to have been consumed. ^b Isolated yield after column chromatography with 2% standard deviation.