

**Supplementary information**

**Catalytic Wet Air Oxidation of Organics-Laden Wastewater: *In Situ* Catalyst**

**Regeneration and Process Scale-up**

Rahul Gupta<sup>a</sup>, Kaushik Basak<sup>b</sup>, Nishith Verma<sup>a,c\*</sup>

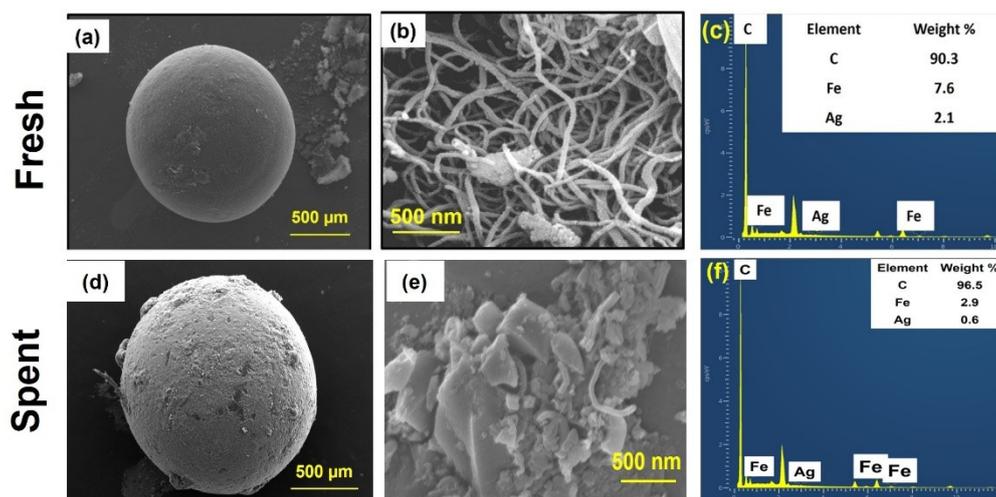
*<sup>a</sup>Department of Chemical Engineering, Indian Institute of Technology Kanpur, Kanpur  
208016, India.*

*<sup>b</sup> Shell Technology Centre Bangalore, Bengaluru, 562149, India.*

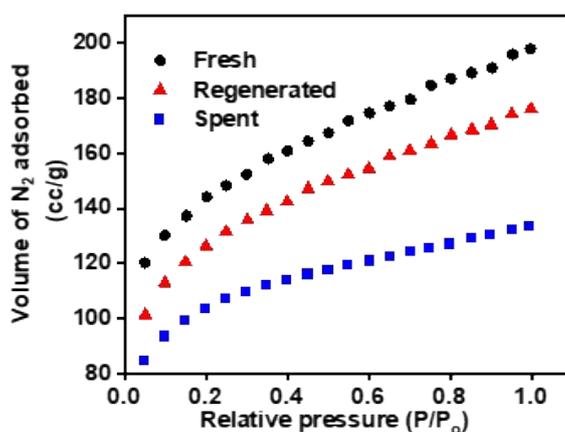
*<sup>c</sup>Center for Environmental Science and Engineering, Indian Institute of Technology  
Kanpur, Kanpur 208016, India.*

\*Corresponding author:

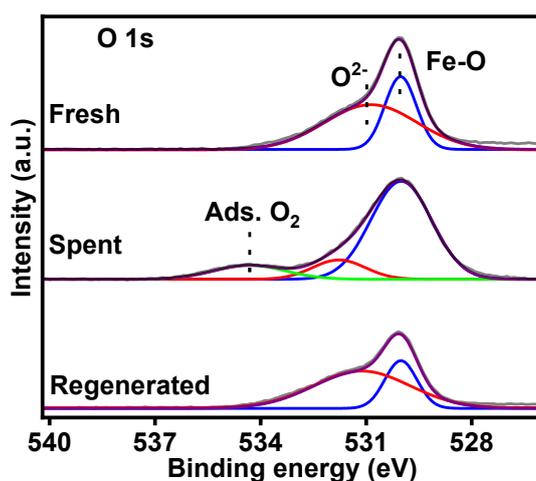
Prof. Nishith Verma (email id: [vermanishith@gmail.com](mailto:vermanishith@gmail.com))



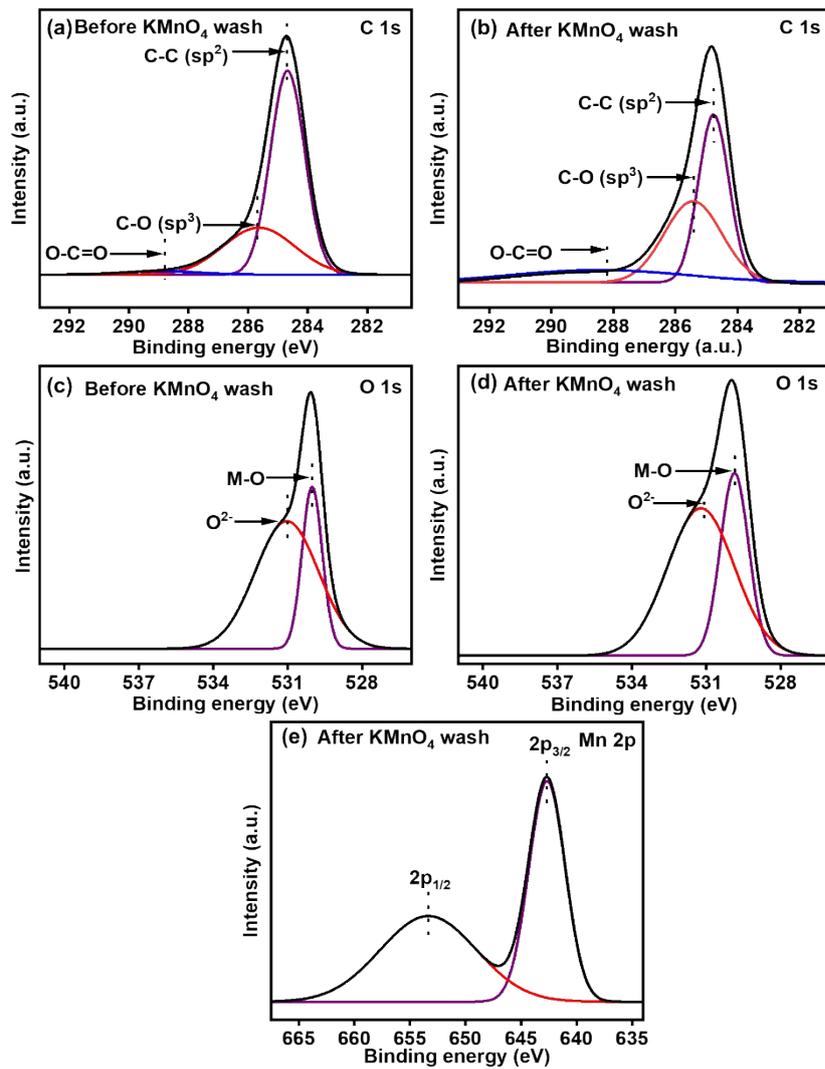
**Fig. S1.** (a and b) SEM images and (c) EDS of the fresh Fe-Ag-CNF/beads; (d and e) SEM images and (f) EDS of the spent Fe-Ag-CNF/beads.



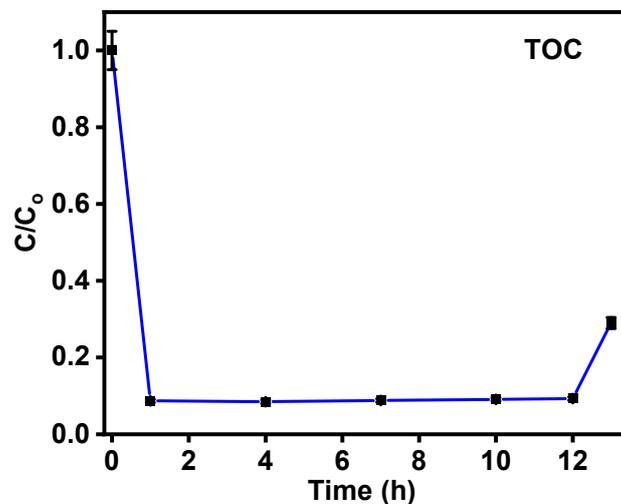
**Fig. S2.**  $N_2$  adsorption isotherm for the fresh, regenerated, and spent catalytic Fe-Ag-CNF/beads.



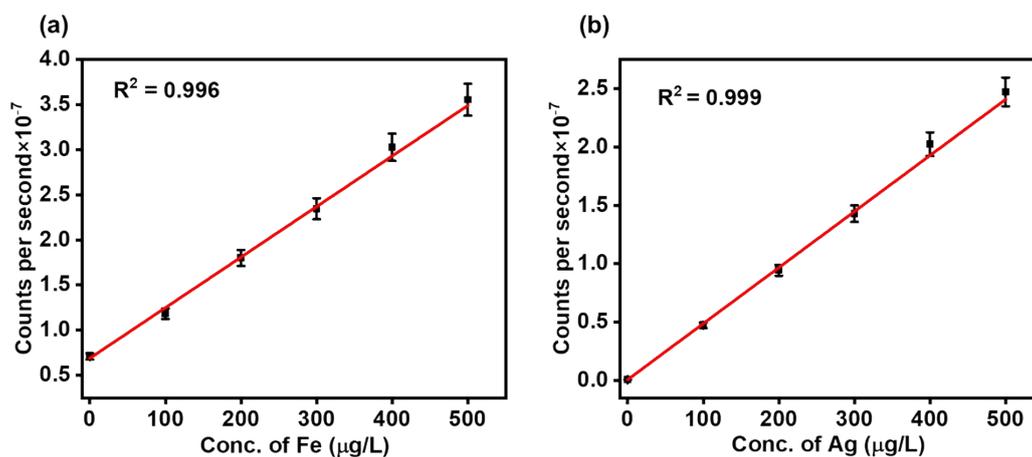
**Fig. S3.** O 1s deconvoluted XPS spectra of the fresh, regenerated, and spent catalytic Fe-Ag-CNF/beads.



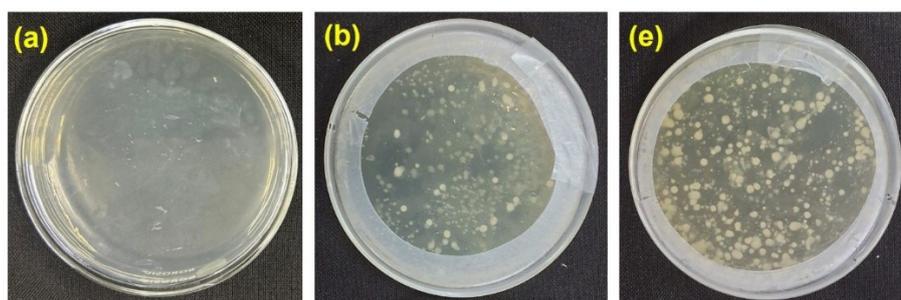
**Fig. S4.** XPS of C 1s (a) before and (b) after; O 1s (c) before and (d) after, and (e) Mn 2p after  $\text{KMnO}_4$  washing.



**Fig. S5.** TOC measurements of the samples collected during cWAO reaction of organics-laden wastewater during continuous operation at the flow rate of 4 cc/min.



**Fig. S6.** ICP-MS calibration curves for (a) Fe and (b) Ag.



**Fig. S7.** Digital photograph of the (*Escherichia Coli*) bacterium growth in (a) organics-laden wastewater, (b) treated wastewater, and (c) control.

**Table S1.**  $S_{BET}$  and pore volumes of the synthesized catalytic beads.

Beads	$S_{BET}$ ( $m^2/g$ )	$V_{total}$ ( $cc/g$ )	$V_{micro}$ ( $cc/g$ )	Avg. pore dia (nm)
Fresh	454.8	0.325	0.217	2.79
Spent	398.9	0.273	0.192	2.74
Regenerated	447.1	0.307	0.213	2.75

**Table S2a.** Consumables cost analysis for Fe-Ag phenolic beads.

S.No.	Reagent name	Unit size	Cost per unit (INR)	Amount required per batch	Cost (INR)
1	Phenol (mL)	500.0	600.0	50.0	60.0
2	FA (mL)	500.0	250.0	63.0	31.5
3	TEA (mL)	500.0	800.0	1.5	2.4
4	Distilled water (mL)	1000.0	10.0	240.0	2.4
5	HMTA (g)	1000.0	350.0	6.0	2.1
6	PVA (g)	500.0	1200.0	3.4	8.2

7	Fe nitrate nonahydrate (g)	500.0	450.0	4.5	4.1
8	Ag nitrate (g)	1000.0	30000.0	1.5	45.0
Total cost of consumables per batch (~25 g beads) in INR					155.6

**Table S2b.** Cost of gases for thermal treatment of the synthesized beads.

S.No.	Gas	Unit size (m <sup>3</sup> )	Cost per unit (INR)	Flow rate (cc/min)	Process time (h)	Cost (INR)
1	N <sub>2</sub> (m <sup>3</sup> )	7.0	2100.0	150.0	8.0	21.6
2	H <sub>2</sub> (m <sup>3</sup> )	7.0	2800.0	150.0	2.0	7.2
3	C <sub>2</sub> H <sub>2</sub> (m <sup>3</sup> )	5.0	3000.0	30.0	1.0	1.1
Total cost of gases per batch (~ 25 g beads) in INR						29.9

**Table S2c.** Cost of energy required for thermal treatment of synthesized beads (cost of 1 kWh is taken to be INR 7.5).

S.No.	Thermal steps	Temperature (°C)	Treatment time (h)	Energy consumption (kWh)	Cost (INR)
1	Carbonization and steam activation	900	6.0	9.0	67.5
2	Hydrogen reduction	650	2.0	3.0	22.5
3	Chemical vapor deposition	650	1.0	1.5	11.2
Total cost of gases per batch (~25 g beads) in INR					101.2

**Table S2d.** Total cost of the Fe-Ag-CNF/beads catalyst.

S. No.	Amount of catalyst (kg)	Raw material (INR)	Gases (INR)	Energy (INR)	Total cost (INR)
1	0.025	311.2	59.8	202.4	573.4
2	0.050	622.4	119.5	404.8	1146.8
3	0.100	1244.9	239.0	809.6	2293.6
4	0.500	6224.4	1195.2	4048.0	11468.0
5	1.000	12448.8	2390.4	8096.0	22936.0

**Table S3a.** Chemicals cost analysis for *in situ* regeneration of 25 g catalyst.

S.No.	Reagent name	Unit size	Cost per unit (INR)	Quantity of reagents required	Cost (INR)
1	Acetone (mL)	1000.0	85.0	540.0	45.9
2	Distilled water (mL)	1000.0	10.0	1680.0	16.8
3	KMnO <sub>4</sub> (g)	500.0	700.0	3.4	4.8
Consumables cost for single regeneration of 25 g catalyst in INR					67.5

**Table S3b.** Cost of gases for *in situ* regeneration of catalyst.

S.No.	Gas	Unit size	Cost per unit (INR)	Flow rate (cc/min)	Process time (h)	Cost (INR)
1	N <sub>2</sub> (m <sup>3</sup> )	7.00	2100.00	150.00	2.00	5.4
2	H <sub>2</sub> (m <sup>3</sup> )	7.00	2800.00	150.00	3.00	10.8
Gases cost for single regeneration of 25 g catalyst in INR						16.2

**Table S3c.** Cost of energy required during regeneration (cost of 1 kWh is taken to be INR 7.5).

S.No.	Thermal step	Temperature (°C)	Treatment time (h)	Energy consumption (kWh)	Cost (INR)
1	<i>In situ</i> hydrogen reduction	650	5	7.5	56.25

**Table S4.** Cost of gases and energy required during cWAO reaction.

S.No.	Process	Temperature (°C)	Treatment time (h)	Energy consumption (kWh)		Cost (INR)
1	cWAO reaction	244	15	7.5		56.25
	<b>O<sub>2</sub> gas (Initial pressurization of 27 bar and constant flow for 15 h)</b>	<b>Flow rate (cc/min)</b>	<b>Process time (h)</b>	<b>Unit size (m<sup>3</sup>)</b>	<b>Cost per unit (INR)</b>	<b>Cost (INR)</b>
2	cWAO reaction	100	15	7	2100.0	27.03
Total cost of processing cWAO of wastewater at 1 cc/min flow rate for 15 h in INR						83.28