

# Mo-LDH-GO Hybrid Catalysts for Indigo Carmine Advanced Oxidation

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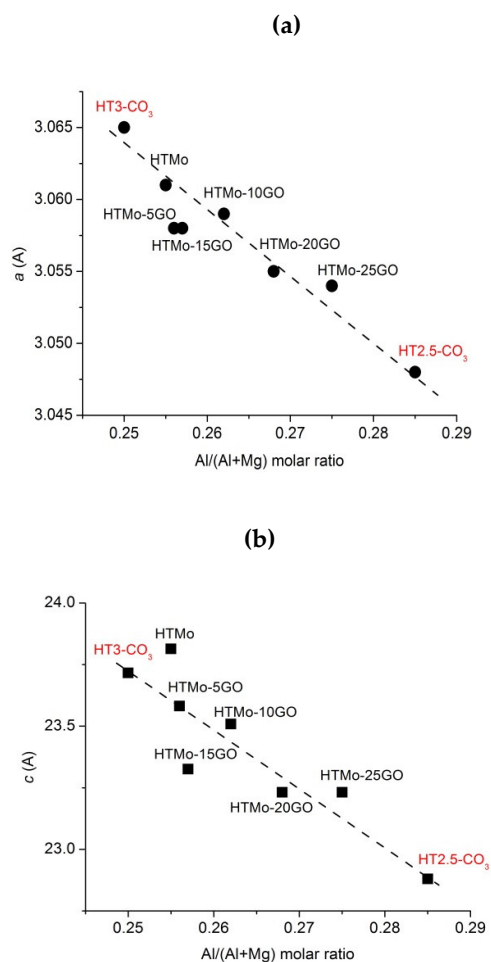
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## S1. The preparation of the GO suspension

The method applied for the preparation of graphene oxide is based on the technique developed by Hummers in 1952 [4, 24]. Thus, graphite powder (325 mesh, from Aldrich, Schnelldorf, Germany), sodium nitrate ( $\text{NaNO}_3$ ) and potassium permanganate ( $\text{KMnO}_4$ , chemical purity, from Merck, Darmstadt, Germany), sulfuric acid ( $\text{H}_2\text{SO}_4$ , 98%, from Merck, Darmstadt, Germany), hydrochloric acid ( $\text{HCl}$ , 37%, from Merck), and hydrogen peroxide ( $\text{H}_2\text{O}_2$ , 30%, from Chimreactiv, Bucharest, Romania) were utilized. In brief, 23 mL concentrated sulfuric acid and 1 g of graphite were mixed in an Erlenmeyer flask maintained in an ice bath at 0 °C. Then, 0.5 g of sodium nitrate and 3.0 g of potassium permanganate were slowly added to the formed solution which was kept under continuous stirring for 30 min. Further, the paste was diluted with 46 mL of distilled water causing a strong exothermic effect accompanied by bubbling, and the resulting mixture was stirred for one hour at 90 °C. Moreover, to remove the residual permanganate, the whole solution was mixed with 71 mL of warm distilled water and 5 mL  $\text{H}_2\text{O}_2$  and kept for one hour under stirring. For the removal of the remaining metal ions, 125 mL  $\text{HCl}$  0.1 N was added, and the solution was stirred for another hour. The resulting mixture was centrifuged and washed with warm distilled water until the conductivity was below 100  $\mu\text{S}/\text{cm}$ . The concentration of GO in the suspension was determined using the gravimetric method, by weighing 3 liquid samples of 100 mL in Petri dishes before and after the evaporation of water under vacuum at 60 °C for 24 h. The amount of solid recovered from the 3 samples was 0.4001, 0.4005, and 0.3998 g, which gave an average value of the GO concentration in the suspension of ca. 4 g/L.

**Table S1.** Compositions of the solutions A and B used in the syntheses of HTMo-xGO hybrids.

Samples	Solution A					Solution B		
	Volume H <sub>2</sub> O (mL)	Volume GO suspension (mL)	Mg(NO <sub>3</sub> ) <sub>2</sub> · 6H <sub>2</sub> O g/mMoles	Al(NO <sub>3</sub> ) <sub>3</sub> · 9H <sub>2</sub> O g/mMoles	GO (g)	Volume H <sub>2</sub> O (mL)	NaOH g/mMoles	Na <sub>2</sub> MoO <sub>4</sub> · 2H <sub>2</sub> O g/mMoles
HTMo	847	0	22.72/88.6	11.08/29.5	0	845	10.4/260	3.6/14.9
HTMo-5GO	679.5	125	21.59/84.2	10.54/28.1	0.5	817.6	9.9/247.5	3.4/14.1
HTMo-10GO	519	250	20.49/79.9	10/26.7	1	782	9.4/235	3.24/13.4
HTMo-15GO	308.5	375	19.29/75.3	9.4/25.1	1.5	695	8.84/221	3.05/12.6
HTMo-20GO	150.6	500	18/70.2	8.79/23.4	2	661.6	8.24/206	2.84/11.7
HTMo-25GO	25	625	17/66.3	8.3/22.1	2.5	660	7.8/195	2.7/11.2



**Figure S1.** The linear variation of the lattice parameters determined by XRD depending on the 60 chemical composition of the brucite type layer: (a) variation of  $a$  parameter, (b) variation of  $c$  parameter. The reference samples for the  $\text{Mg}_x\text{Al-CO}_{32}$  ( $x=3$  and  $2.5$ , labeled HT3-CO<sub>3</sub> and HT2.5, respectively) were extracted from our previous works [33, 34, 56].

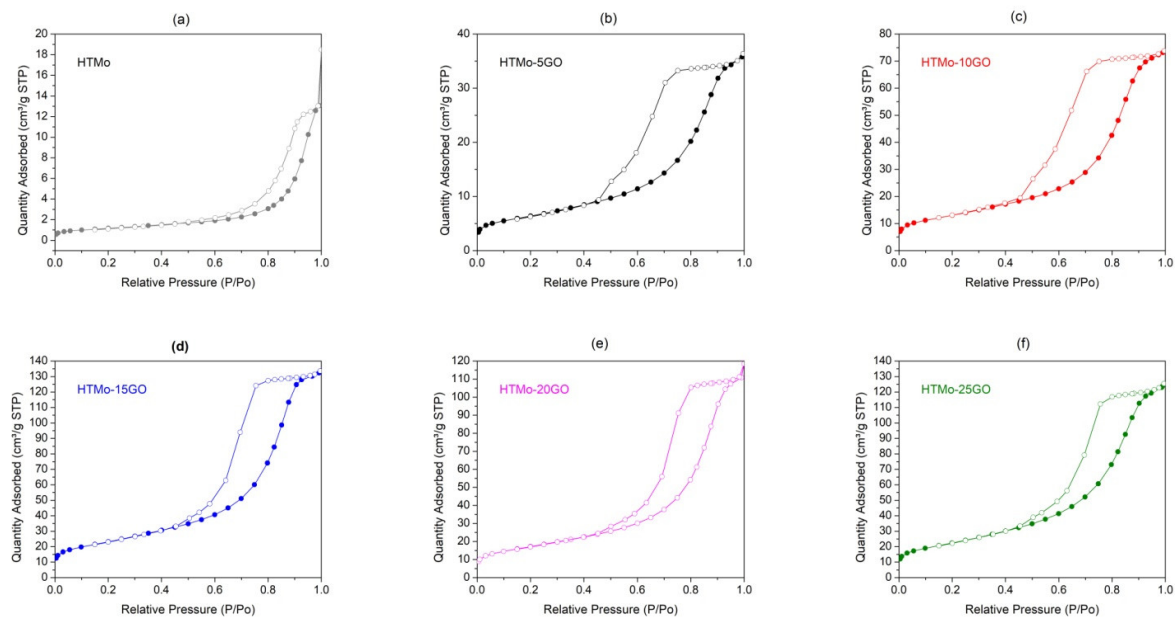


Figure S2. BET isotherms of the investigated samples.

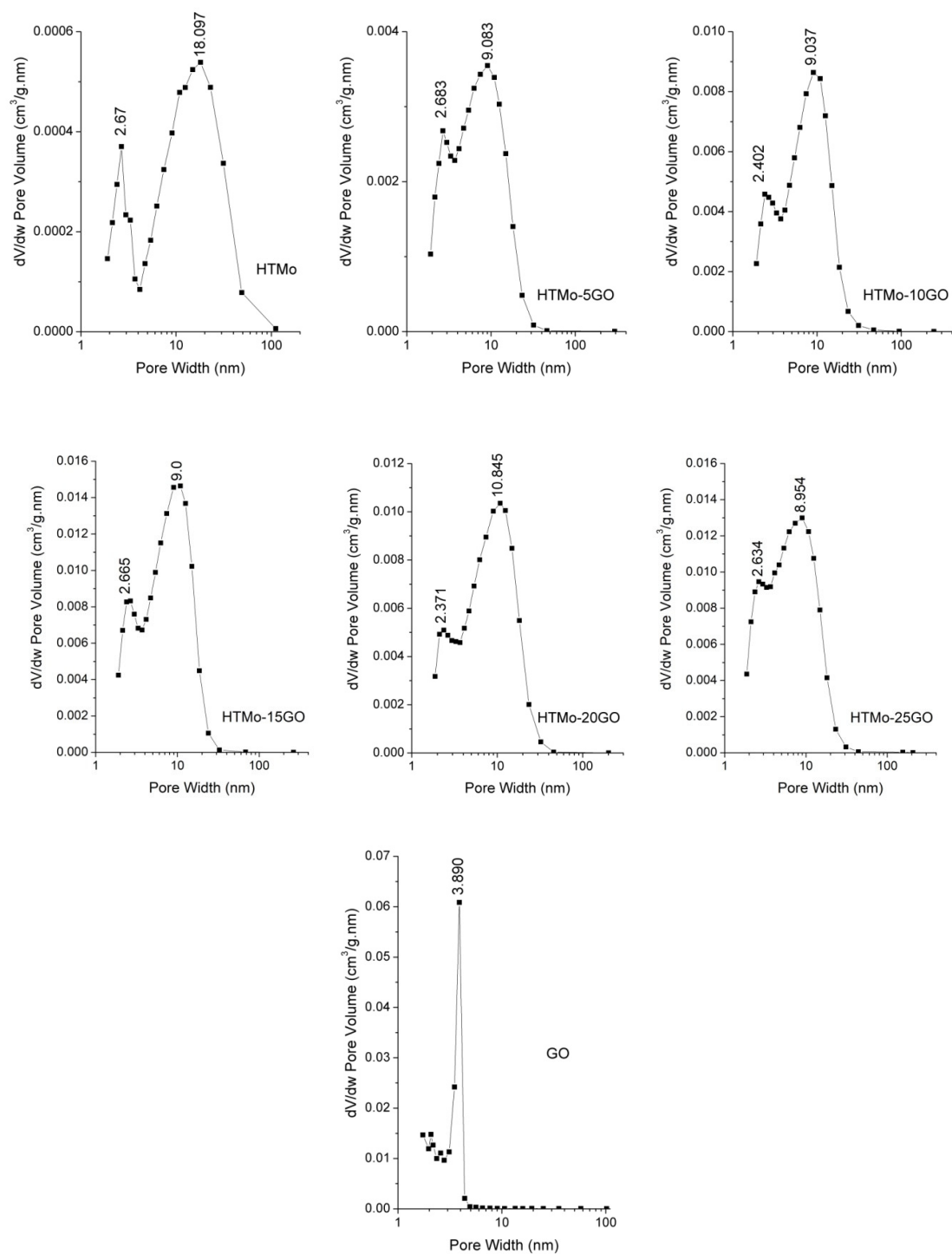
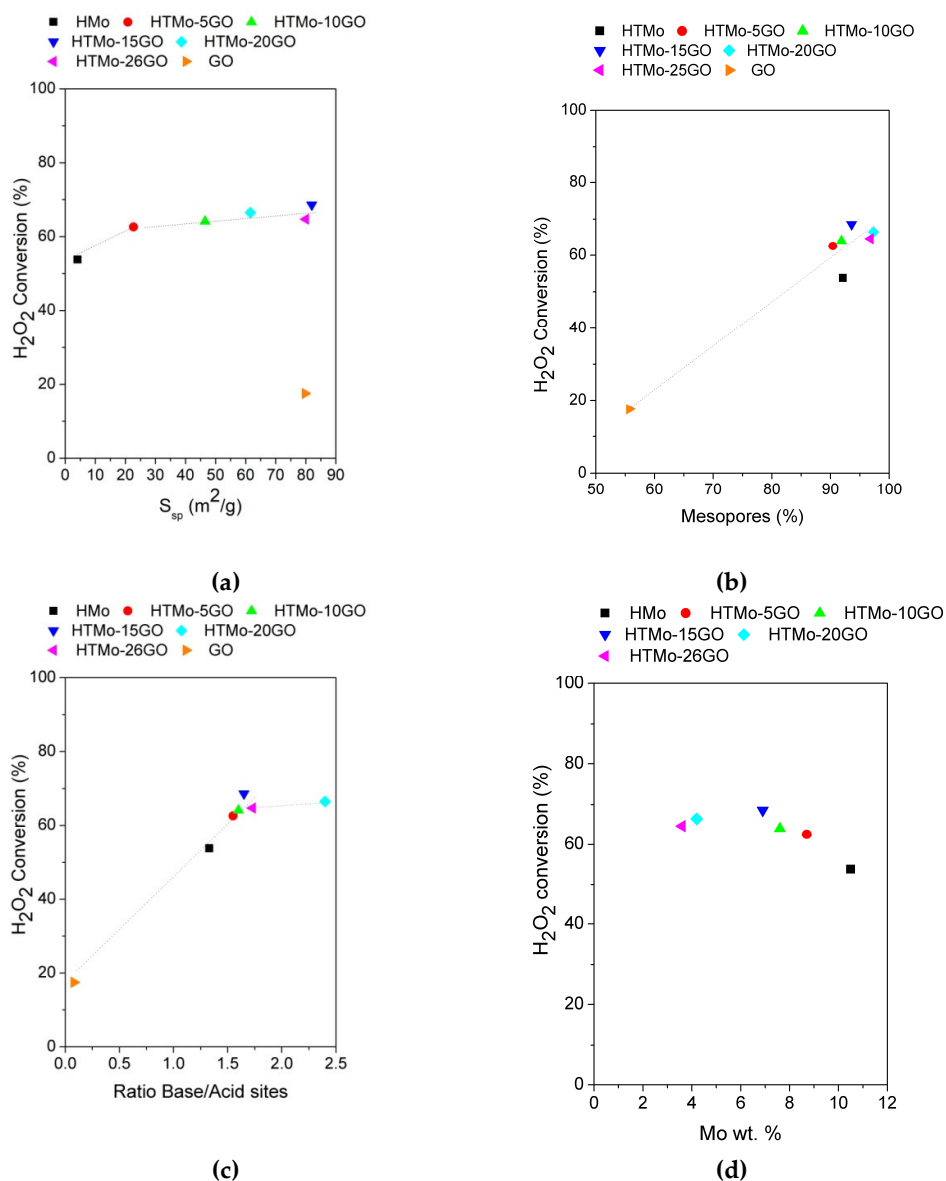


Figure S3. Pore size distribution of the investigated samples (BJH, Halsey-Faas correction).

**Table S2.** The chemical oxygen demand (COD) and total organic carbon content (TOC) of the water samples after the catalytic tests performed with concentrated solution of IC ( $IC_0=90 \times 10^{-3} M$ ; COD initial 574.7 mg  $O_2/L$ ; TOC initial -174.2 mg C/L) at a molar ratio  $H_2O_2/IC=48/1$ .

Catalysts	IC conversion (%)	COD	TOC
HTMo	68.7	180	55
HTMo-5GO	81.2	108	u.d.l.
HTMo-10GO	84.7	88	u.d.l.
HTMo-15GO	85.4	84	u.d.l.
HTMo-20GO	95.4	27	u.d.l.
HTMo-25GO	88.3	67	u.d.l.

u.d.l. = under detection limit



**Figure S4.** The influence of the physico-chemical characteristics of the investigated catalysts on  $H_2O_2$  conversion: (a) specific surface areas; (b) the proportion of mesopores; (c) the basicity expressed as the ratio between base and acid sites; (d) Mo concentration; (Reaction conditions:  $IC_0=30 \times 10^{-3}$  M,  $H_2O_2/IC=48$  catalysts concentration 1 wt. %, 150 rpm, 2 h, 25 °C).

## References

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