Electronic Supplementary Information (ESI)

Ni/Ti layered double hydroxide: Synthesis, characterization and application as a photocatalyst for visible light degradation of aqueous methylene blue

Priyadarshi Roy Chowdhury and Krishna G. Bhattacharyya*
Department of Chemistry, Gauhati University, Guwahati 781014, Assam, India.
**E-mail: kgbhattacharyya@gmail.com*

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S1. Schematic crystal structure of layered double hydroxides (LDHs)

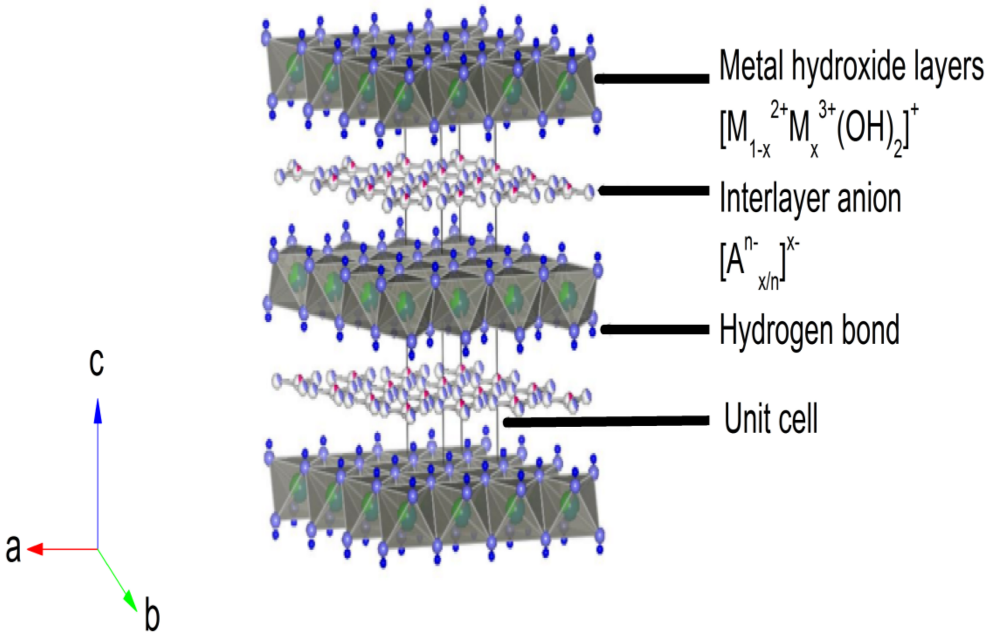

Fig. S1. Schematic crystal structure of layered double hydroxide (rep. from Supplementary Ref. 1)

S2. HR-TEM image of 2:1 Ni/Ti LDH

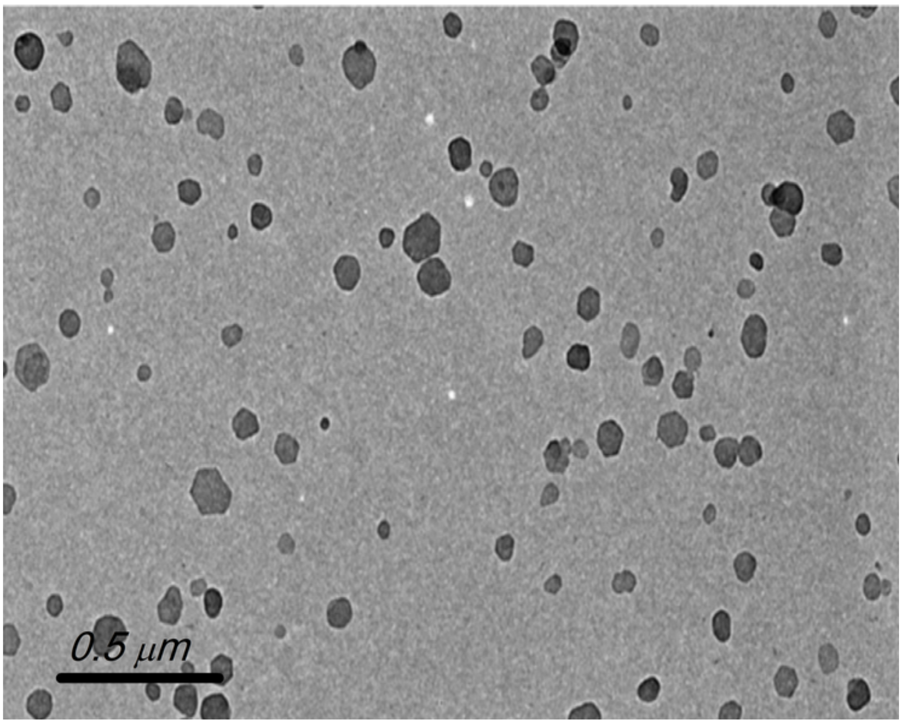

Figure S2. HR-TEM image of 2:1 Ni/Ti LDH

S3. XRD parameters of the 2:1 Ni/Ti LDH

h	k	l	2θ(deg)	FWHM	d-spacing	Relative intensity
0	0	3	13.69	0.844	0.763	100
0	0	6	27.38	0.237	0.448	12.32
1	1	0	27.31	0.254	0.153	16.14
1	0	1	37.81	0.193	0.168	12.04
0	0	9	41.07	0.241	0.297	15.31

S4. Lattice parameters of 2:1 Ni/Ti LDH

Parameters of P-XRD analysis	2:1 Ni/Ti LDH
Lattice parameter a	0.306
Lattice parameter c	2.550
Lattice parameter c´	0.85
Interlayer thickness[a]	0.416
(003/006) peak height ratio	~3.97

The diffraction peaks of the hydrotalcites can be indexed in a hexagonal lattice. It is known that, for hydrotalcites, the lattice parameter c depends on several factors like anion size, hydratation and amount of interlayer anion. The lattice parameter a of the LDHs can be correlated with the cation–cation distance within the brucite-like layer. The values of the lattice parameters a and c have been calculated using the following relations [3, 6]

$a=2d_{110}$, $c\ [= (3d_{003}+6d_{006}+9d_{009})/3]$ and $c=3c'$.

Interlayer thickness= (c' — brucite-like sheet thickness); c'= 0.85 and

Brucite like sheet thickness= 0.434.

S5. Differential Thermal Analysis (DTA) of 2:1 Ni/Ti LDH

The differential thermal analysis (DTA) was performed with 2:1 Ni/Ti LDH in order to validate the results of thermogravimetric analysis (TG). The DTA measurements with yielded results (Fig. S5) similar to those of TG observations. It was found that the weight loss processes of DTA were associated with three endothermic transformations, [2] the first one appearing at 93°C, the second one at 293°C and the third at 372°C corresponding to removal of physisorbed as well as interlayer water, dehydration of the brucite like layers and decomposition of carbonate ions in the interlayer respectively. Any additional DTA peak were not observed up to 780°C indicating that the material was quite stable in this temperature range. These observations are comparable with previously reported results for LDH. [2, 3]

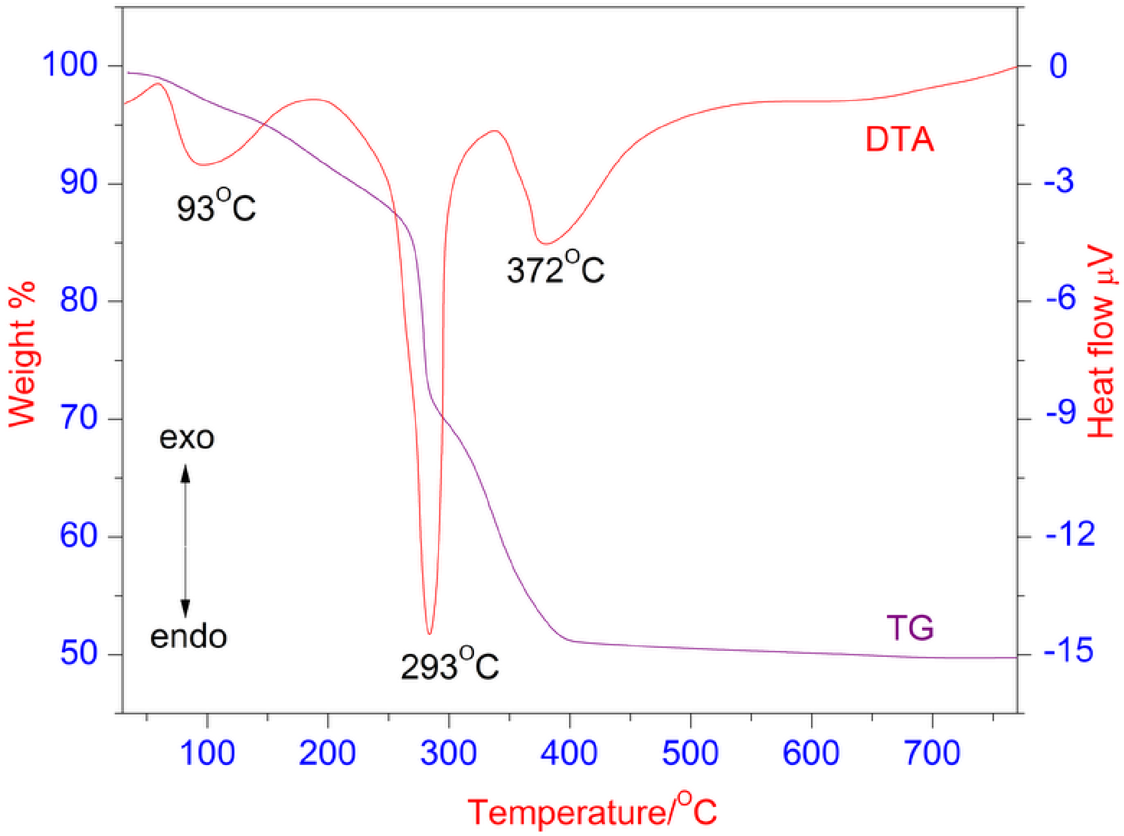

Fig. S5. TG/DTA curve of 2:1 Ni/Ti LDH

S6. Differential Scanning Calorimetry (DSC) analysis of 2:1 Ni/Ti LDH

The TG/DSC curve of 2:1 Ni/Ti LDH (Fig. S6) measured under nitrogen gas flow showed three inflection points involving three transitions typical of the LDH materials. [4] The results of the DSC measurements are found to be in good agreement with those of DTA. The first transition at 94°C corresponds to the endothermic removal of water physisorbed on the external surface of the Ni/Ti LDH as well as the water in the interlayer galleries.[5] The sharp endothermic peak at around 295°C involved dehydration from the brucite like layers and the third broad endothermic event around 372°C represented the decomposition of the interlayer CO_3^{2-} anions to CO_2 which leads to the collapse of the layered structure.[6, 7] Weight loss above 550°C was not observed showing that the residual carbonaceous content in the synthesized LDH was very small.

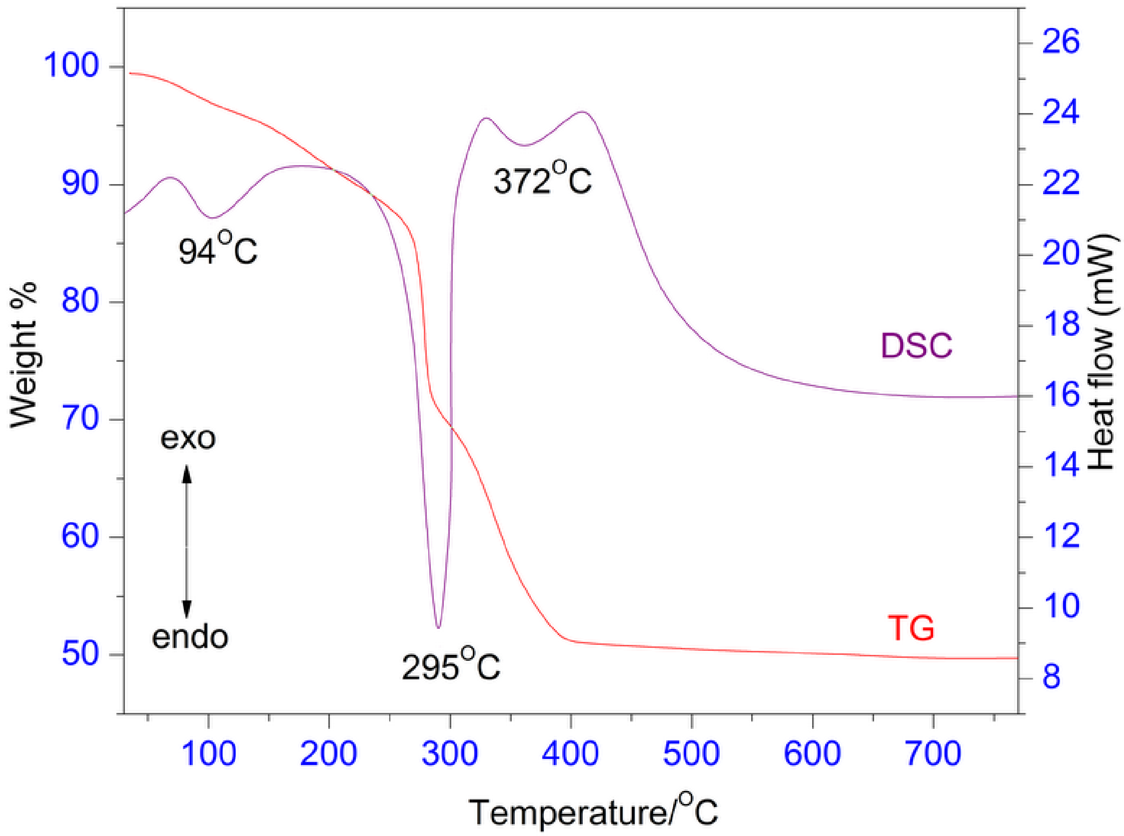

Fig. S6. TG/DSC curve of 2:1 Ni/Ti LDH

S7. Adsorption equilibrium between catalyst and MB

The reaction mixture was also analysed by taking out aliquots at 5 minute intervals (during the 30 min period in the dark, with vigorous stirring of the reaction mixture) and thereby analyzing it by UV-vis spectrophotometer (Fig. S7). The plot of C/C_0 Vs irradiation time shows a straight line parallel to X-axis without any change in the C/C_0 values indicating clearly that adsorption was not actually possible, thereby establishing the adsorption equilibrium between the catalyst and MB.

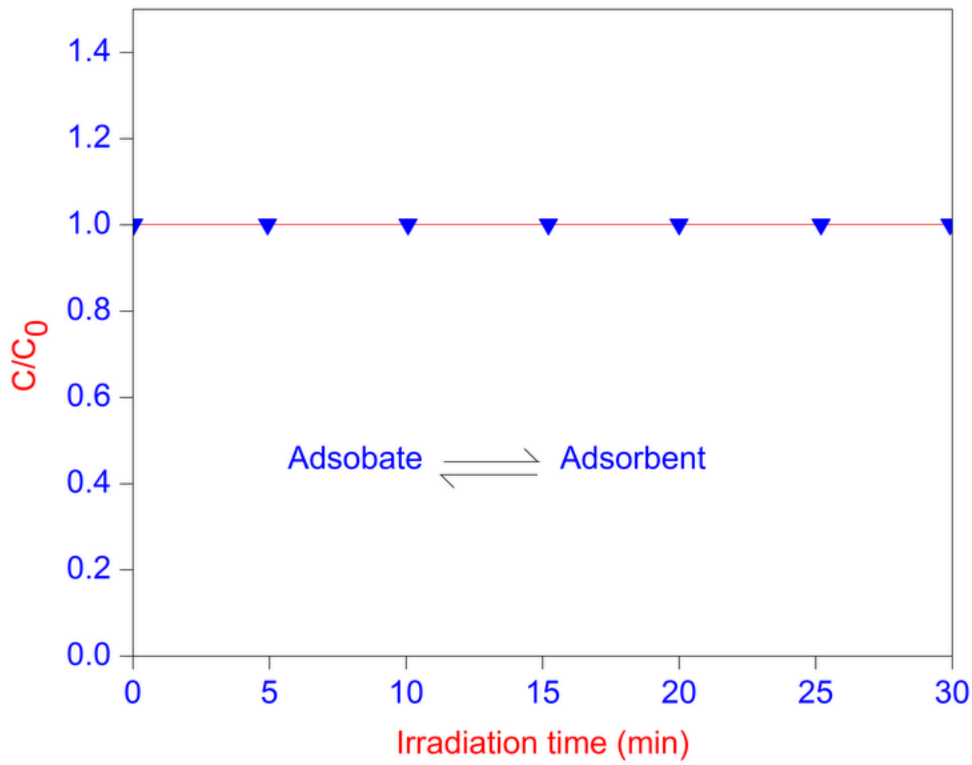

Fig. S7. Plot of C/C_0 versus irradiation time (30 mins of vigorous stirring before irradiation by visible light) establishing the adsorbate-adsorbent equilibrium.

References

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