Electronic Supplementary Material (ESI)

Synthesis and characterization of Co/Ti layered double hydroxide and its application as a photocatalyst for degradation of aqueous Congo Red†

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S1. X-ray diffraction parameters

The diffraction peaks the LDH could be indexed in a hexagonal lattice. For LDH, the lattice parameter 'c' depends on factors like anion size, hydratation and interlayer anion. The lattice parameter 'a' can be correlated with the cation–cation distance within the brucite-like layer.

Table S1: X-ray diffraction parameters of 2:1 Co/Ti LDH

h	k	l	2θ (deg)	FWHM (deg)	d-spacing (nm)	Relative intensity
0	0	3	11.49	0.846	0.763	65.45
0	0	6	22.98	0.214	0.424	29.32
0	0	9	34.47	0.235	0.274	28.31
1	1	0	28.35	0.267	0.146	33.24
1	0	0	32.31	0.425	0.156	27.23
1	0	1	37.44	0.335	0.263	43.28
0	1	8	47.12	0.248	0.267	29.24
1	1	$\underline{11}$	53.35	0.285	0.315	28.15
1	1	3	67.61	0.904	0.151	18.24
1	0	$\underline{13}$	76.58	0.915	0.186	15.43

The lattice parameters 'a' and 'c' have been calculated using the following relations-
$a=2d_{110}$, c [$= (3d_{003}+6d_{006}+9d_{009})/3$] and $c=3c'$.

Interlayer thickness= (c′ — brucite-like sheet thickness); c′= 0.811 nm and
Brucite like sheet thickness= 0.42 nm. (calculated using cross-sectional TEM and AFM measurements)

Table S2: Lattice parameters

Parameters of XRD analysis	2:1Co/Ti LDH
Lattice parameter a (nm)	0.292
Lattice parameter c (nm)	2.433
Lattice parameter c′ (nm)	0.811
Interlayer thickness (nm)	0.391
(003/006) peak height ratio	2.24

S2. Zeta potential (ξ) curve of Co/Ti LDH

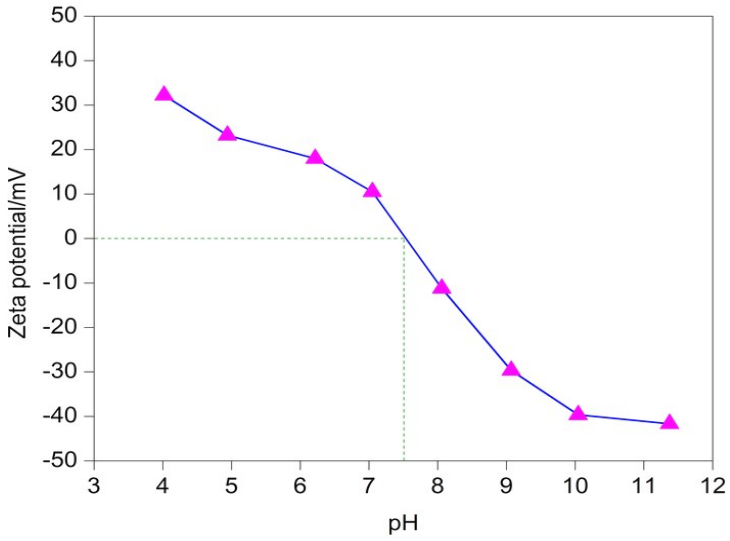

Fig. S1. Zeta potential (ξ) of 2:1 Co/Ti LDH

S3. BET isotherm and pore diameter curve of Co/Ti LDH

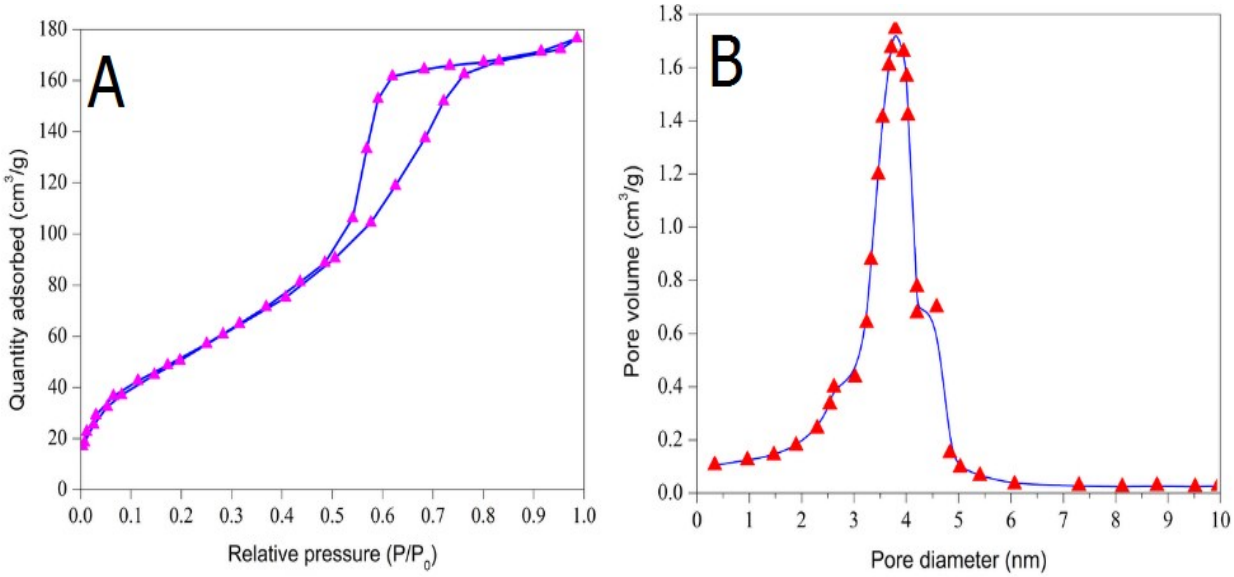

Fig. S2 (A). BET isotherm (B) Pore diameter curve of 2:1 Co/Ti LDH

S4. FT-IR spectrum of Co/Ti LDH

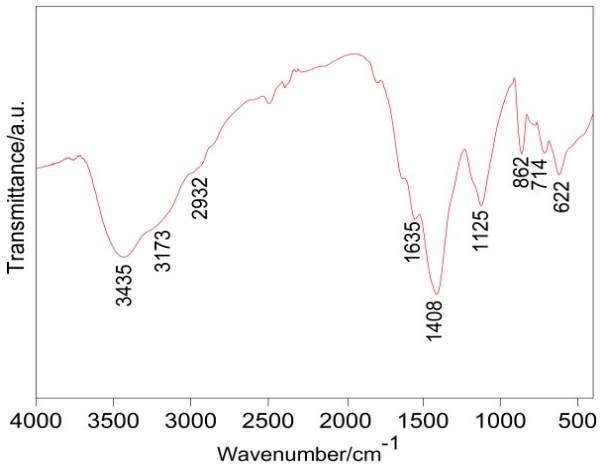

Fig. S3. FT-IR spectra of 2:1 Co/Ti LDH

S5. TG/DTG curve of 2:1 Co/Ti LDH

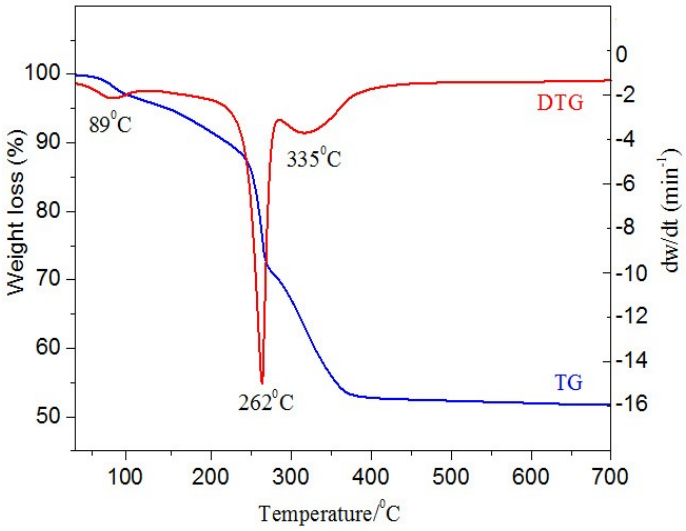

Fig. S4. TG/DTG curve of 2:1 Co/Ti LDH

S6. Cross-sectional TEM image

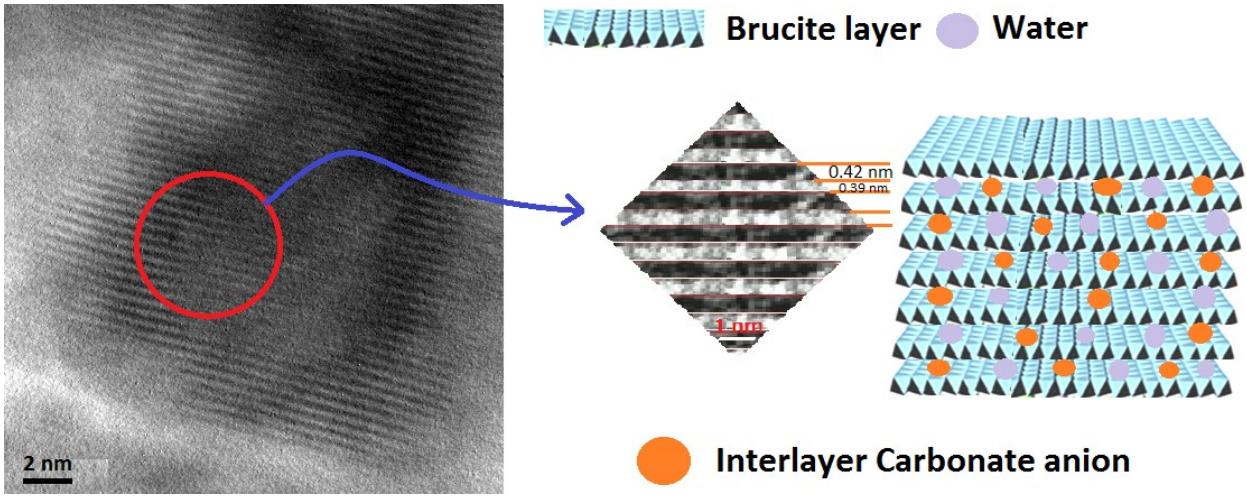

Fig. S5. Cross sectional TEM image of 2:1 Co/Ti LDH and its co-relation with idealized LDH structure

S7. Adsorption equilibrium between 2:1 Co/Ti LDH and Congo Red dye

During the 30 min dark period prior to the irradiation by visible light, the reaction mixture was analysed by taking out aliquots at every 5 minutes by UV-vis spectrophotometer **(Fig. S6; ESI†).** The plot of C/C_0 vs irradiation time shows a straight line parallel to X-axis without any observable deviation in the C/C_0 values, validates the establishment of adsorption equilibrium between 2:1 Co/Ti LDH and Congo Red dye.

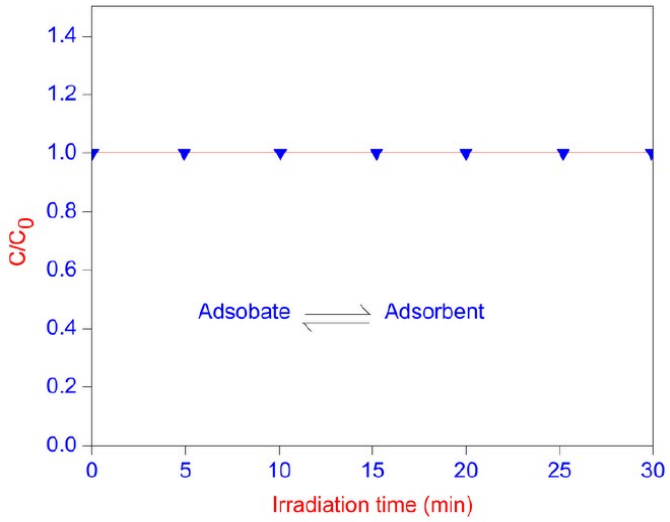

Fig. S6. Plot of C/C_0 versus irradiation time (during 30 mins of vigorous stirring in dark, prior to visible light irradiation) establishing the LDH-dye equilibrium.

S8. Synthesis of 1:1, 3:1 and 4:1 Co/Ti LDHs

The synthetic procedure employed for 2:1 Co/Ti LDH was also followed for preparation of 1:1, 2:1 and 3:1 Co/Ti LDHs. In the typical synthesis, 5.89 g of $Co(NO_3)_2.6H_2O$, 1.1 ml $TiCl_4$ and 3.0 g of urea, were dissolved in 100 ml de-ionized water at room temperature (~30 ^{0}C) and the contents were vigorously stirred for 3 h. The mixture was aged in Teflon lined autoclave at 140 ^{0}C for 36 h and the dark pink crystalline product was extracted by centrifugation, washed several times with de-ionized water and dried at 100 ^{0}C for 4 h. The product so obtained was designated as 1:1 Co/Ti LDH.

The 3:1 and 4:1 Co/Ti LDHs were synthesized by varying the molar ratio of Co during hydrothermal synthesis, keeping others constant. Herein, 17.67 g and 23.56 g of $Co(NO_3)_2.6H_2O$ were used during the synthesis of 3:1 and 4:1 Co/Ti LDHs respectively.