

# Electronic Supplementary Information

Comparison of nano-structured transition metal modified tri-metal MgMAl-LDHs ( $M = \text{Fe, Zn, Cu, Ni, Co}$ ) prepared using co-precipitation<sup>†</sup>

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## 1 XRD analysis and crystal data

**Table S 1:** XRD data for the angular position of the primary, secondary and tertiary LDH reflections (003), (006) and (009) with units of  $2\theta$ , the d-spacing calculated with the position of each of these angles, the  $a$ - and average  $c$ -parameter, the  $c$ -parameter based on the (003) reflection only and the crystallite sizes parallel and perpendicular to the platelet diameter. The d-spacing,  $a$ - and  $c$ -parameters and crystallite dimensions are all shown with units of Å.

	(003)	(006)	(009)	(110)	$d_{003}$	$d_{006}$	$d_{009}$	$d_{110}$	$a$	$c$	$c_{(003)}$	$l_{(003)}$	$l_{(110)}$
MgAl-LDH	13.3842	27.0851	40.5305	72.0513	7.676	3.820	2.583	1.521	3.042	23.063	23.028	1190.6	1139.2
MgFeAl-0.5	13.4123	27.1052	40.6377	72.0557	7.660	3.817	2.576	1.521	3.042	23.022	22.980	1285.2	1167
MgFeAl-1	13.3908	27.0935	40.5721	72.05	7.672	3.819	2.580	1.521	3.042	23.050	23.016	1288	1199.5
MgFeAl-5	13.4083	27.0755	40.5672	71.964	7.662	3.821	2.580	1.522	3.045	23.045	22.986	1435.7	1289.7
MgFeAl-10	13.4079	27.0764	40.5301	71.8901	7.662	3.821	2.583	1.524	3.048	23.052	22.987	1440.1	1134.6
MgFeAl-25	13.3566	26.9858	40.4174	71.6527	7.692	3.834	2.589	1.528	3.056	23.127	23.075	1808.3	1434.5
MgCoAl-0.5	13.4583	27.1454	40.6697	72.1591	7.634	3.812	2.574	1.519	3.038	22.979	22.901	1066.8	1179.6
MgCoAl-1	13.4189	27.1348	40.5586	72.0263	7.656	3.813	2.581	1.521	3.043	23.025	22.968	1374	1315.4
MgCoAl-5	13.5154	27.3018	40.5932	72.0872	7.602	3.790	2.579	1.520	3.040	22.918	22.805	1922.9	1558.8
MgCoAl-10	13.4611	27.1775	40.5327	72.0053	7.632	3.807	2.582	1.522	3.043	22.994	22.897	1590.1	1337
MgCoAl-25	13.5242	27.2968	40.5135	71.9083	7.597	3.791	2.584	1.523	3.047	22.929	22.790	2525.4	1639.1
MgNiAl-0.5	13.4145	27.1452	40.6541	71.9928	7.659	3.812	2.575	1.522	3.044	23.007	22.976	1604.2	1279.8
MgNiAl-1	13.3871	27.0342	40.5381	71.9573	7.674	3.827	2.582	1.523	3.045	23.074	23.023	1475.3	1192.1
MgNiAl-5	13.3838	27.0303	40.5571	71.9806	7.676	3.828	2.581	1.522	3.044	23.074	23.028	1520.5	1087.8
MgNiAl-10	13.3661	27.1452	40.6541	72.1287	7.686	3.812	2.575	1.519	3.039	23.034	23.059	996.8	866.5
MgNiAl-25	13.4168	27.113	40.7082	72.1222	7.657	3.816	2.572	1.520	3.039	23.005	22.972	1800.1	875.1
MgCuAl-0.5	13.4055	27.0969	40.5829	72.0256	7.664	3.818	2.579	1.521	3.043	23.038	22.991	1408.4	1233.3
MgCuAl-1	13.3639	27.0694	40.5722	72.0369	7.687	3.822	2.580	1.521	3.042	23.072	23.062	1154.8	1169.1
MgCuAl-5	13.4261	27.1309	40.5588	71.9697	7.652	3.814	2.581	1.522	3.045	23.022	22.956	1705.9	1269.3
MgCuAl-10	13.416	27.1322	40.5892	72.0056	7.658	3.813	2.579	1.522	3.043	23.021	22.973	1264.7	1162.2
MgCuAl-25	13.4599	27.2272	40.6205	71.9281	7.633	3.800	2.577	1.523	3.046	22.965	22.899	1971.2	1467.1
MgZnAl-0.5	13.4215	27.1088	40.5954	72.0709	7.655	3.817	2.579	1.521	3.041	23.024	22.964	1082.3	1102.4
MgZnAl-1	13.4275	27.2869	40.6001	72.0241	7.651	3.792	2.578	1.521	3.043	22.970	22.954	1035.9	968
MgZnAl-5	13.45095	27.0943	40.5684	72.0173	7.638	3.819	2.580	1.521	3.043	23.016	22.914	1314.1	1273.2
MgZnAl-10	13.4202	27.1517	40.5769	71.9917	7.655	3.811	2.580	1.522	3.044	23.016	22.966	1366.8	1252.6
MgZnAl-25	13.4231	27.1748	40.5391	71.8821	7.654	3.808	2.582	1.524	3.048	23.015	22.961	1491	1284.6

## 2 X-ray fluorescence spectroscopy

**Table S 2:** X-ray fluorescence results showing the gram-amount (percentage) of each compound present in the fused LDH sample at 1000 °C, the loss on ignition (LOI) and the total at 1000 °C.

Sample Name	MgO	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	CaO	Co <sub>3</sub> O <sub>4</sub>	LOI.Bead	TOTAL
MgAl-LDH	32.70	19.60	0.17	0.21	<0.01	47.30	99.98

Sample Name	MgO	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	CaO	SO <sub>3</sub>	LOI.Bead	TOTAL
MgFeAl-0.5	32.10	19.30	0.19	0.33	0.37	0.12	47.50	99.90
MgFeAl-1	33.20	19.30	0.35	0.09	0.24	0.03	46.70	99.91
MgFeAl-5	33.10	18.00	1.60	0.11	0.24	0.04	46.80	99.89
MgFeAl-10	32.30	17.30	3.16	0.24	0.20	0.08	46.60	99.89
MgFeAl-25	29.80	13.20	7.01	0.11	0.13	0.08	49.60	99.94

Sample Name	MgO	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	CaO	Co3O4	LOI.Bead	TOTAL
MgCoAl-0.5	24.80	14.90	0.14	0.41	0.26	59.40	99.91
MgCoAl-1	32.20	19.60	0.13	0.11	0.83	47.00	99.88
MgCoAl-5	30.00	20.50	0.08	0.24	3.73	45.40	99.96
MgCoAl-10	28.10	19.40	0.07	0.08	6.48	45.90	100.03
MgCoAl-25	22.90	17.50	0.06	0.20	15.90	43.40	99.96

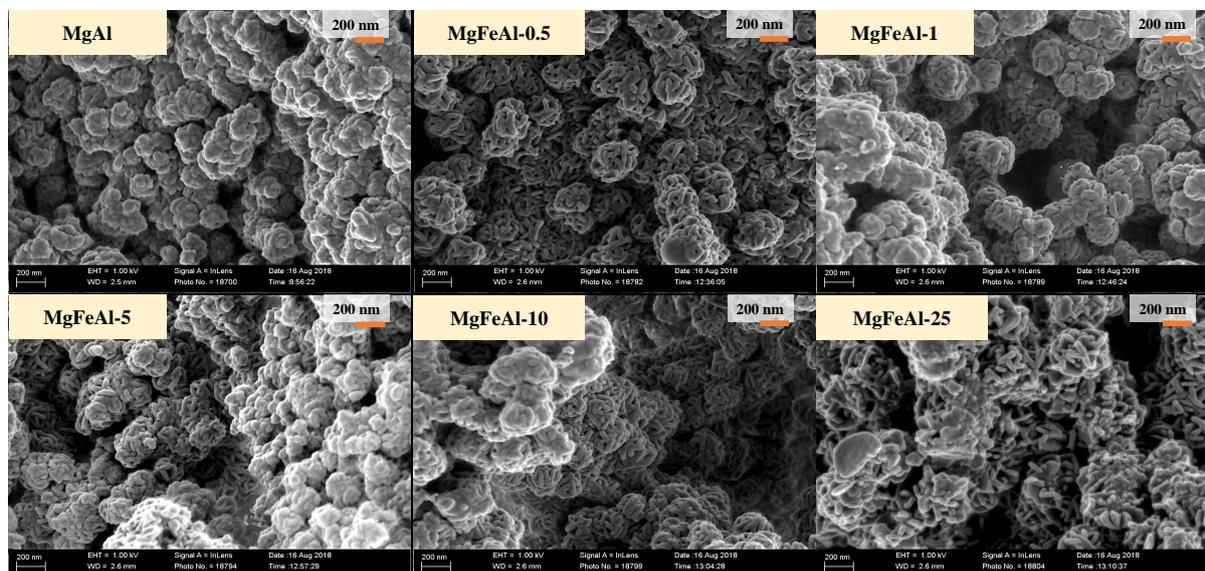
Sample Name	MgO	Al <sub>2</sub> O <sub>3</sub>	NiO	SiO <sub>2</sub>	CaO	LOI.Bead	TOTAL
MgNiAl-0.5	33.50	19.00	0.36	0.11	0.06	46.90	99.93
MgNiAl-1	34.10	17.90	0.51	0.08	0.09	47.30	99.98
MgNiAl-5	31.10	18.40	3.36	0.07	0.05	47.00	99.98
MgNiAl-10	27.50	18.60	6.44	0.01	0.51	46.80	99.86
MgNiAl-25	23.40	16.50	14.40	0.14	0.07	45.50	100.01

Sample Name	MgO	Al <sub>2</sub> O <sub>3</sub>	CuO	NiO	SiO <sub>2</sub>	CaO	SO3	Fe <sub>2</sub> O <sub>3</sub>	LOI.Bead	TOTAL
MgCuAl-0.5	32.80	18.60	0.34	0.51	0.12	0.14	0.02	0.12	47.20	99.85
MgCuAl-1	32.20	19.30	0.73	0.01	0.12	0.14	0.02	0.02	47.40	99.94
MgCuAl-5	31.40	18.80	3.43	0.02	0.15	0.29	0.02	0.01	45.80	99.93
MgCuAl-10	28.70	18.40	6.46	<0.01	0.09	0.13	<0.01	0.03	46.10	99.91
MgCuAl-25	22.00	17.30	15.40	0.45	0.29	0.22	0.24	0.13	43.80	99.83

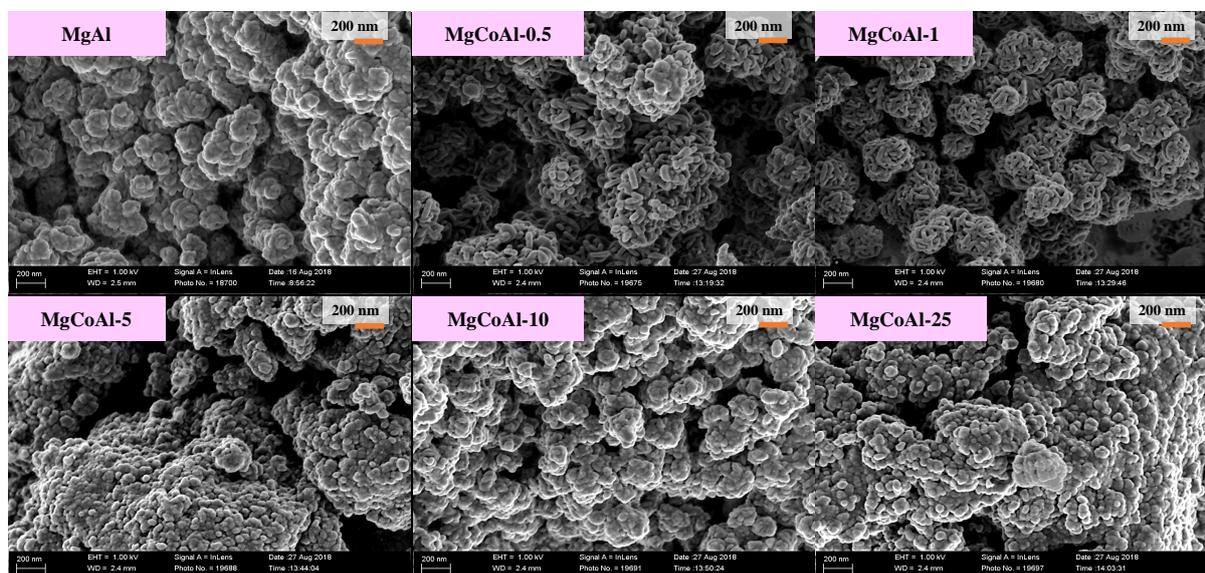
Sample Name	MgO	Al2O3	SiO <sub>2</sub>	CaO	ZnO	LOI.Bead	TOTAL
MgZnAl-0.5	32.80	19.40	0.12	0.46	0.38	46.70	99.86
MgZnAl-1	32.40	19.70	0.08	0.20	0.76	46.80	99.93
MgZnAl-5	30.70	19.30	0.20	0.28	3.48	45.80	99.76
MgZnAl-10	28.60	18.50	0.11	0.21	6.60	45.90	99.91
MgZnAl-25	23.00	17.90	0.17	0.14	15.40	43.20	99.81

### 3 Scanning electron microscopy

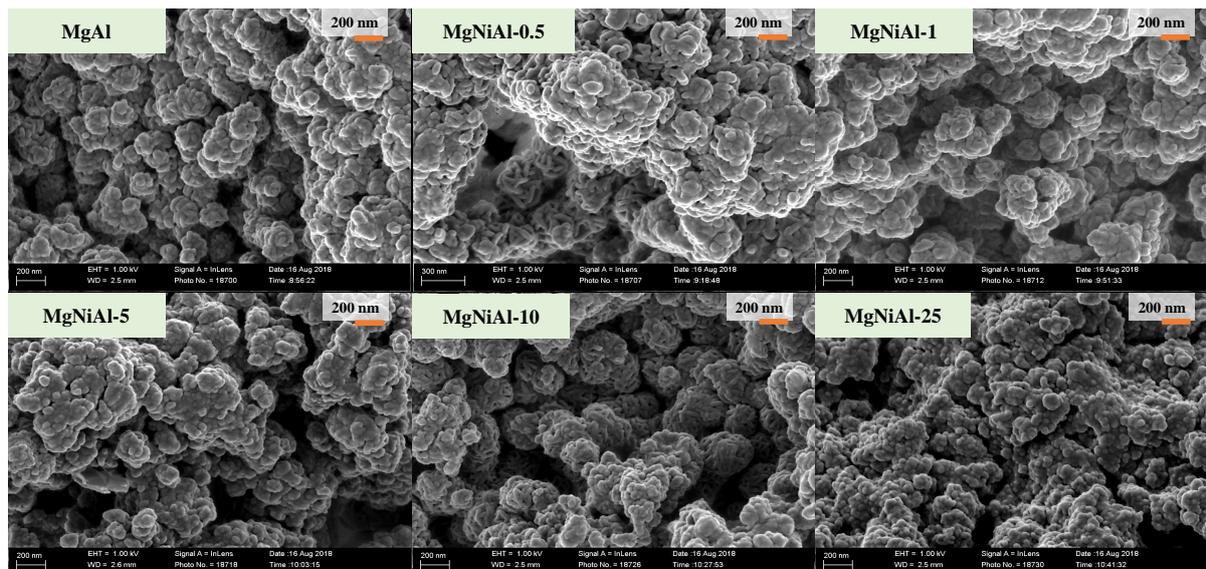
Figure S 1 to Figure S 5 below show larger, full text figures of those provided in the paper.



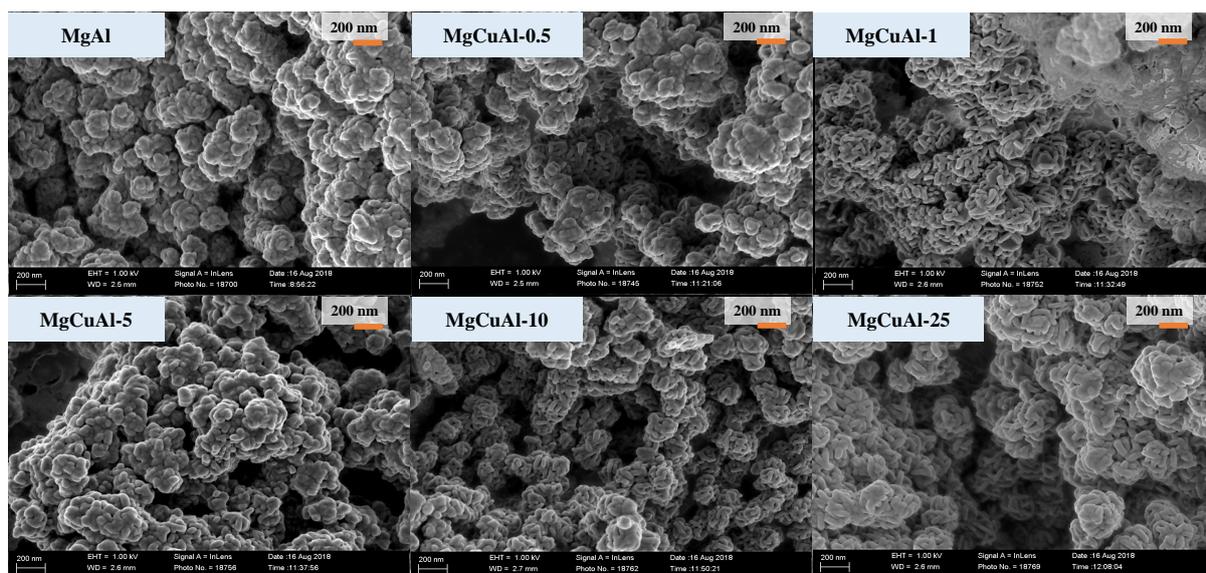
**Figure S 1:** SEM micrographs of the series of MgFeAl-LDHs with for substitutions of 0.5 %, 1 %, 5 %, 10 % and 25 % compared against MgAl-LDH



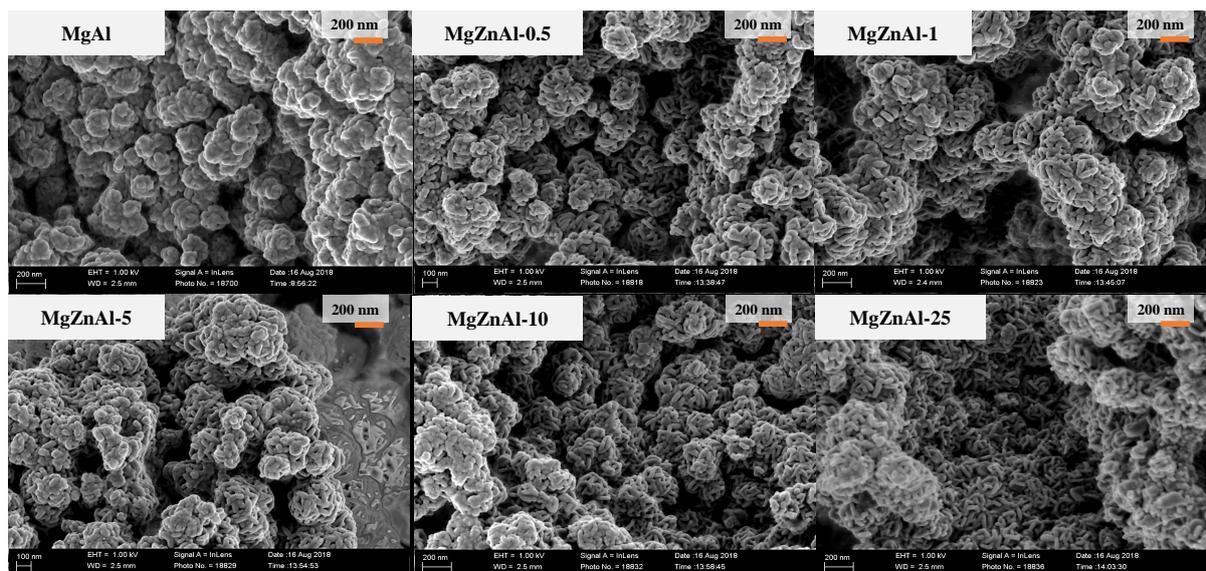
**Figure S 2:** SEM micrographs of the series of MgCoAl-LDHs with for substitutions of 0.5 %, 1 %, 5 %, 10 % and 25 % compared against MgAl-LDH



**Figure S 3:** SEM micrographs of the series of MgNiAl-LDHs with for substitutions of 0.5 %, 1 %, 5 %, 10 % and 25 % compared against MgAl-LDH



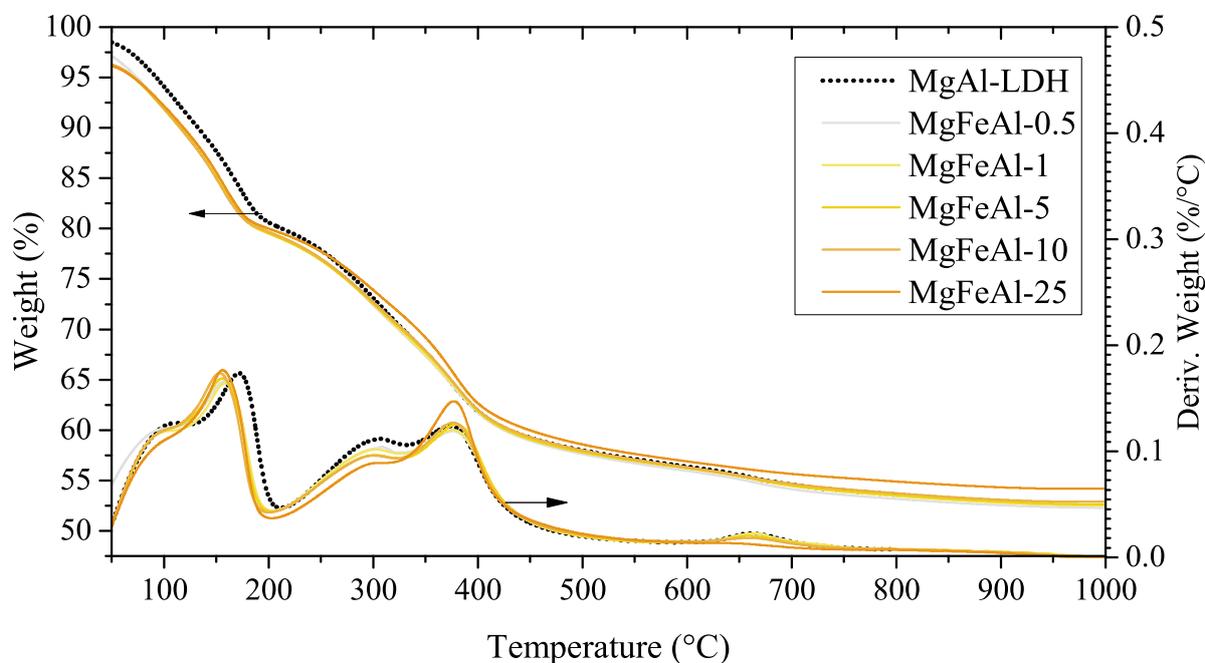
**Figure S 4:** SEM micrographs of the series of MgCuAl-LDHs with for substitutions of 0.5 %, 1 %, 5 %, 10 % and 25 % compared against MgAl-LDH



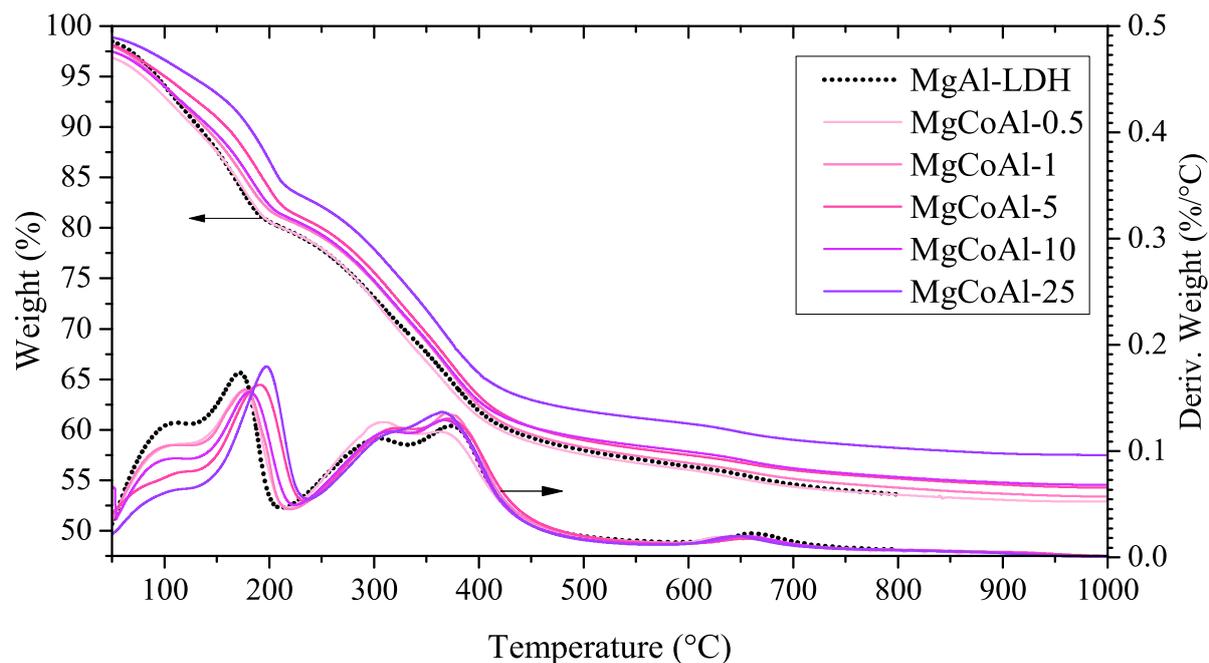
**Figure S 5:** SEM micrographs of the series of MgZnAl-LDHs with for substitutions of 0.5 %, 1 %, 5 %, 10 % and 25 % compared against MgAl-LDH

## 4 Thermogravimetric analysis

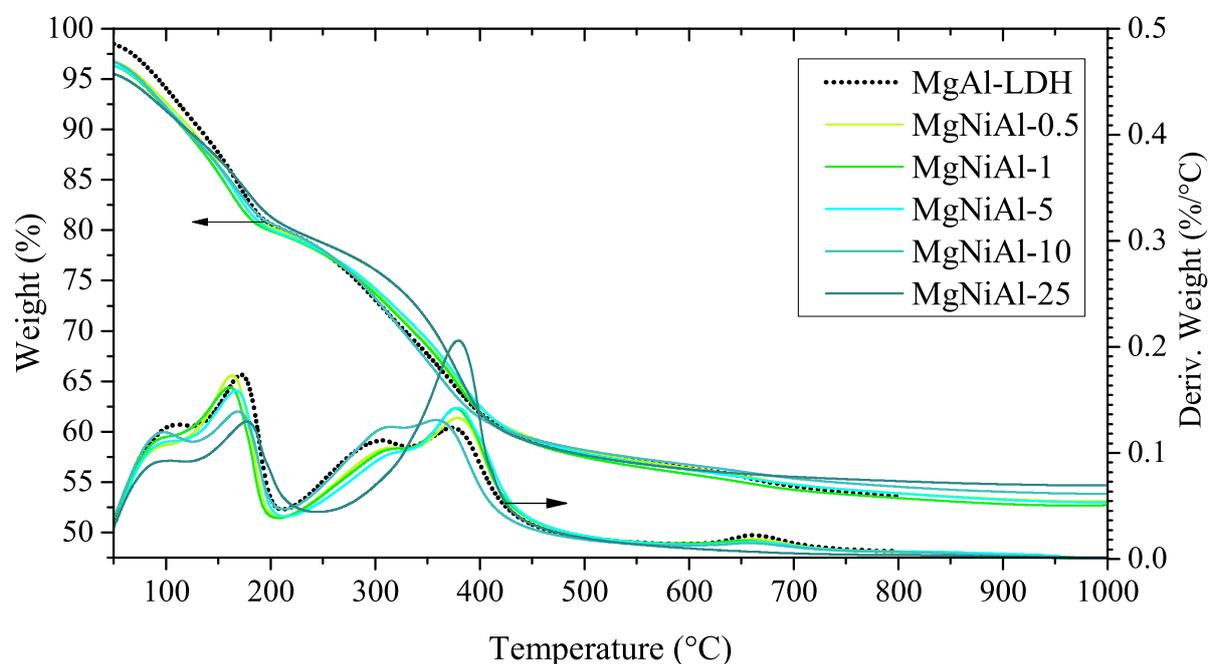
Figure S 6 to Figure S 10 below show the TGA results obtained for the series of MgMAl-LDHs with  $M = \text{Fe, Co, Ni, Cu, and Zn}$ . The mass loss and derivative mass loss are plotted on the same figure.



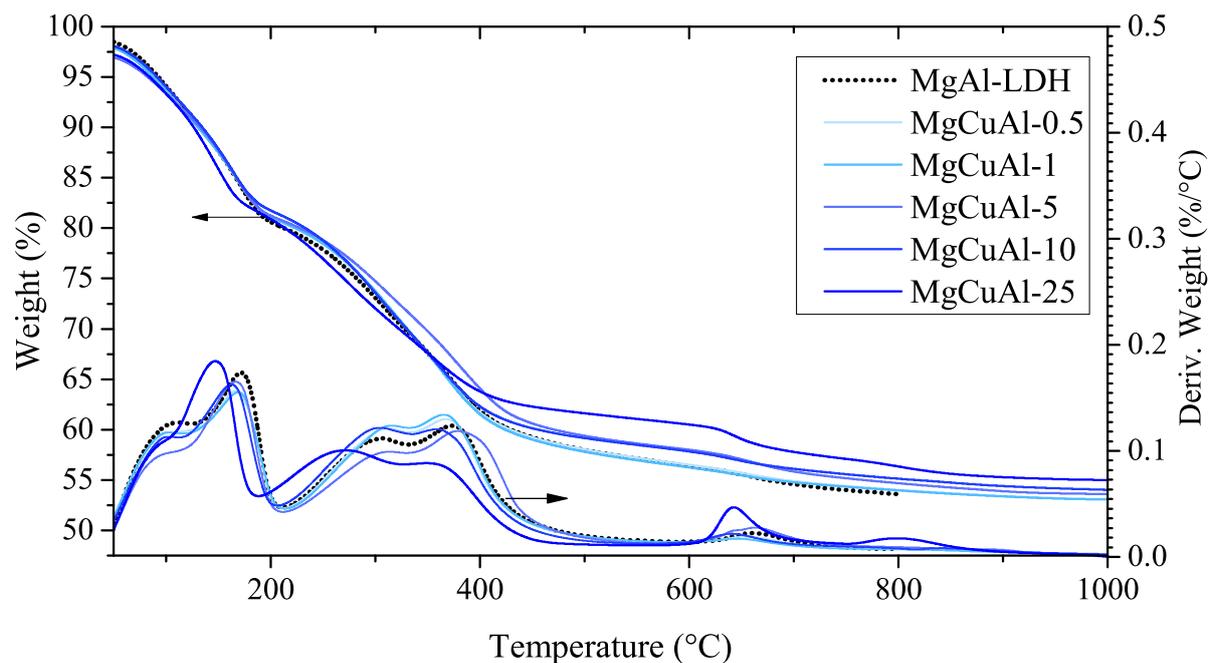
**Figure S 6:** TGA results of the series of MgFeAl-LDHs with for substitutions of 0.5 %, 1 %, 5 %, 10 % and 25 % compared against MgAl-LDH. The mass loss profile (left axis) and the derivative mass loss profile (right axis) are both indicated.



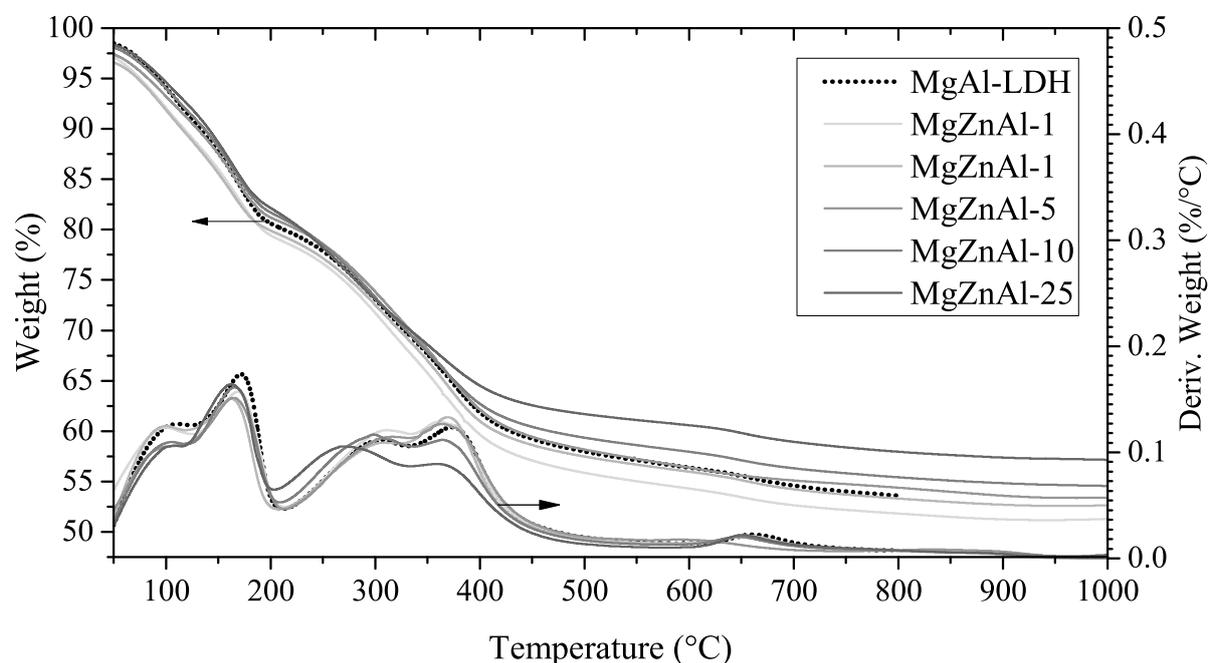
**Figure S 7:** TGA results of the series of MgCoAl-LDHs with for substitutions of 0.5 %, 1 %, 5 %, 10 % and 25 % compared against MgAl-LDH. The mass loss profile (left axis) and the derivative mass loss profile (right axis) are both indicated.



**Figure S 8:** TGA results of the series of MgNiAl-LDHs with for substitutions of 0.5 %, 1 %, 5 %, 10 % and 25 % compared against MgAl-LDH. The mass loss profile (left axis) and the derivative mass loss profile (right axis) are both indicated.



**Figure S 9:** TGA results of the series of MgCuAl-LDHs with for substitutions of 0.5 %, 1 %, 5 %, 10 % and 25 % compared against MgAl-LDH. The mass loss profile (left axis) and the derivative mass loss profile (right axis) are both indicated.



**Figure S 10:** TGA results of the series of MgZnAl-LDHs with for substitutions of 0.5 %, 1 %, 5 %, 10 % and 25 % compared against MgAl-LDH. The mass loss profile (left axis) and the derivative mass loss profile (right axis) are both indicated.