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Supplementary Material

Effect of Chain Length and Functional Group of Organic Anions on the Retention Ability of Mg,Al-Layered Double Hydroxides for Chlorinated Organic Solvents

Karen Maria Dietmann, Tobias Linke, Raquel Trujillano, and Vicente Rives

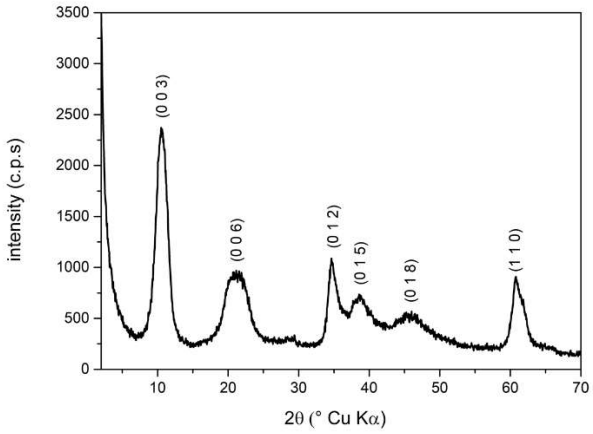

Figure S1. Powder X-Ray diffraction pattern of sample MgAl-5COO.

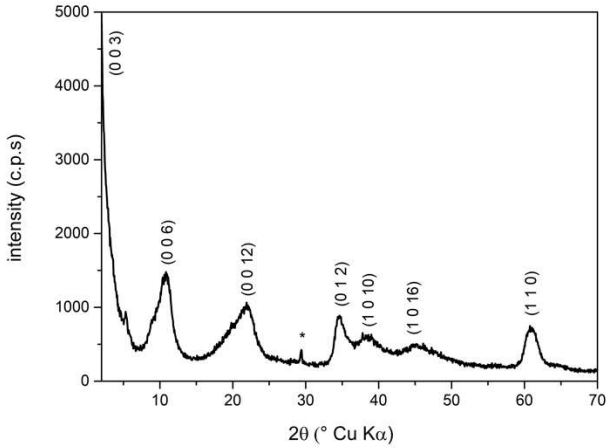

Figure S2. Powder X-Ray diffraction pattern of sample MgAl-7COO. (*) Sodium nitrate impurity.

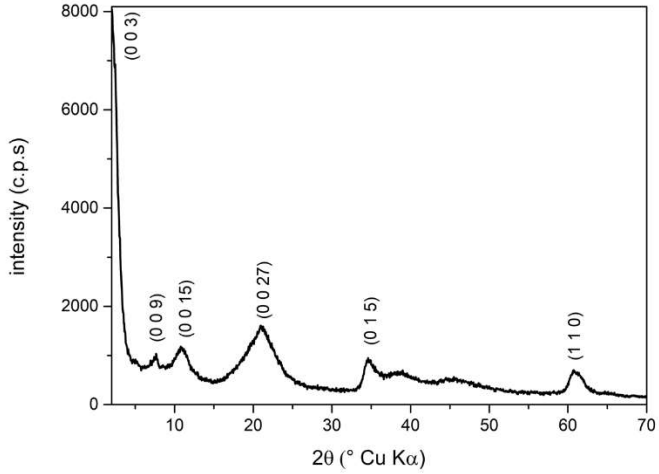

Figure S3. Powder X-Ray diffraction pattern of sample MgAl-11COO.

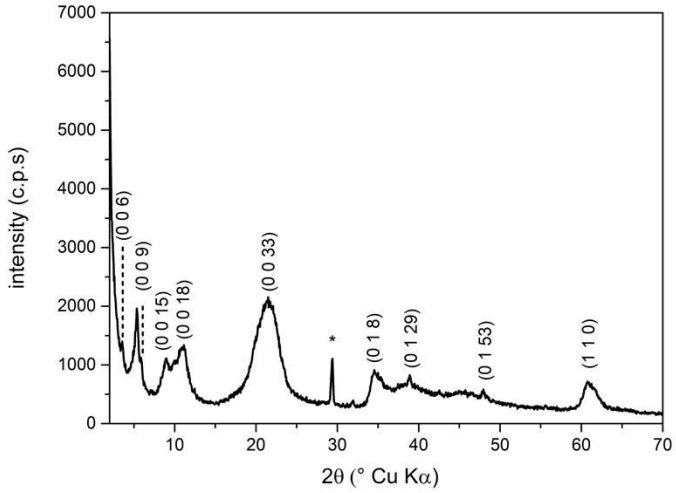

Figure S4. Powder X-Ray diffraction pattern of sample MgAl-17COO. (*) Sodium nitrate impurity.

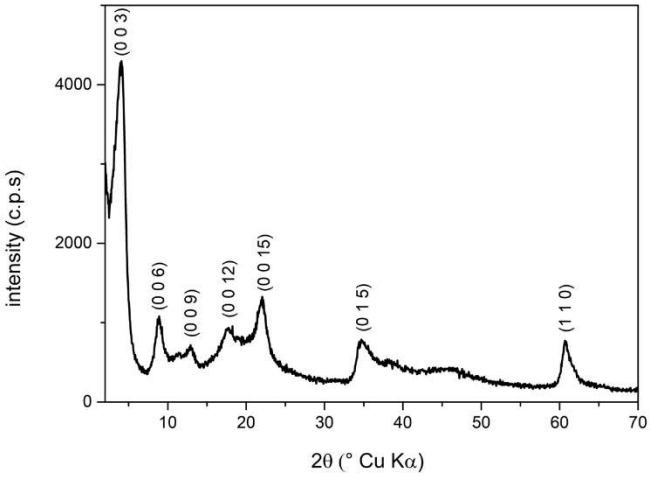

Figure S5. Powder X-Ray diffraction pattern of sample MgAl-8SO3.

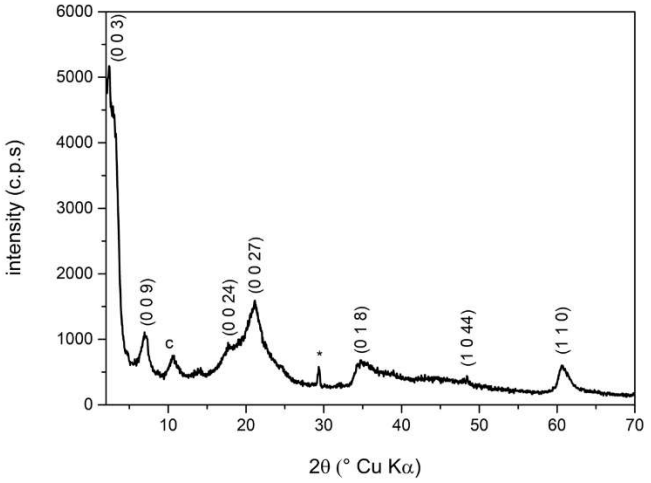

Figure S6. Powder X-Ray diffraction pattern of sample MgAl-12SO3. (c) carbonate impurity, (*) Sodium nitrate impurity.

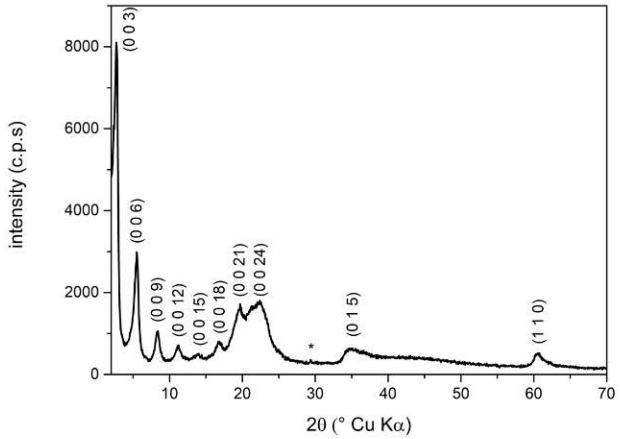

Figure S7. Powder X-Ray diffraction pattern of sample MgAl-18SO3. (*) Sodium nitrate impurity.

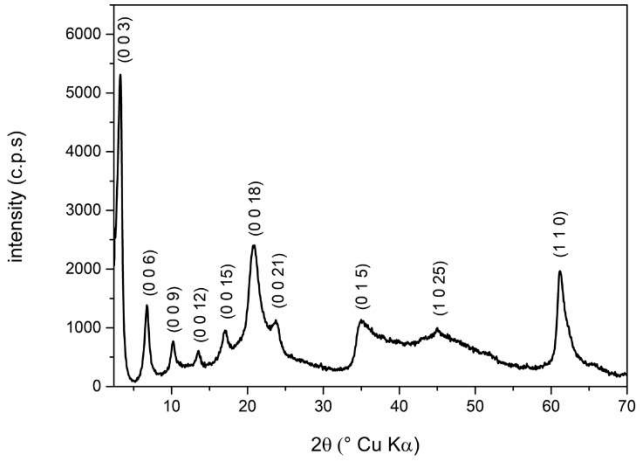

Figure S8. Powder X-Ray diffraction pattern of sample MgAl-12SO4.

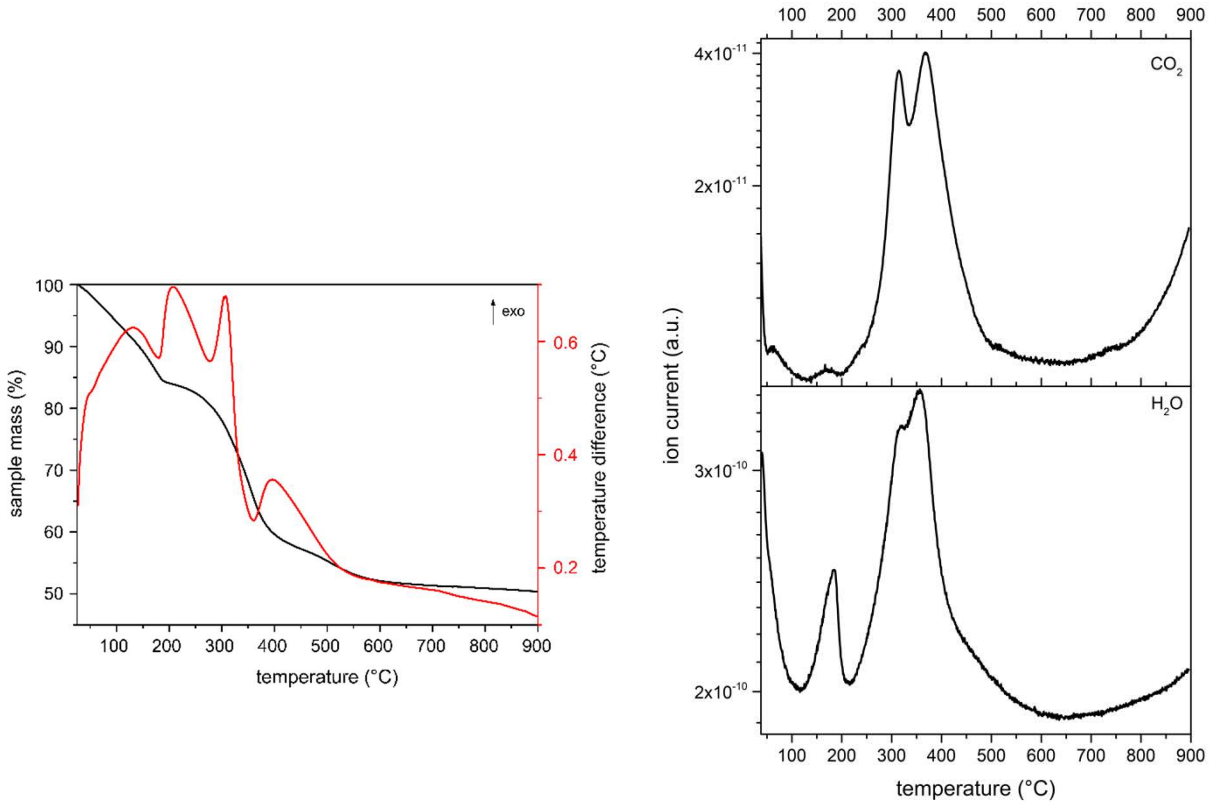

Figure S9. TG-DTA-MS analysis of the reference sample MgAl-CO3.

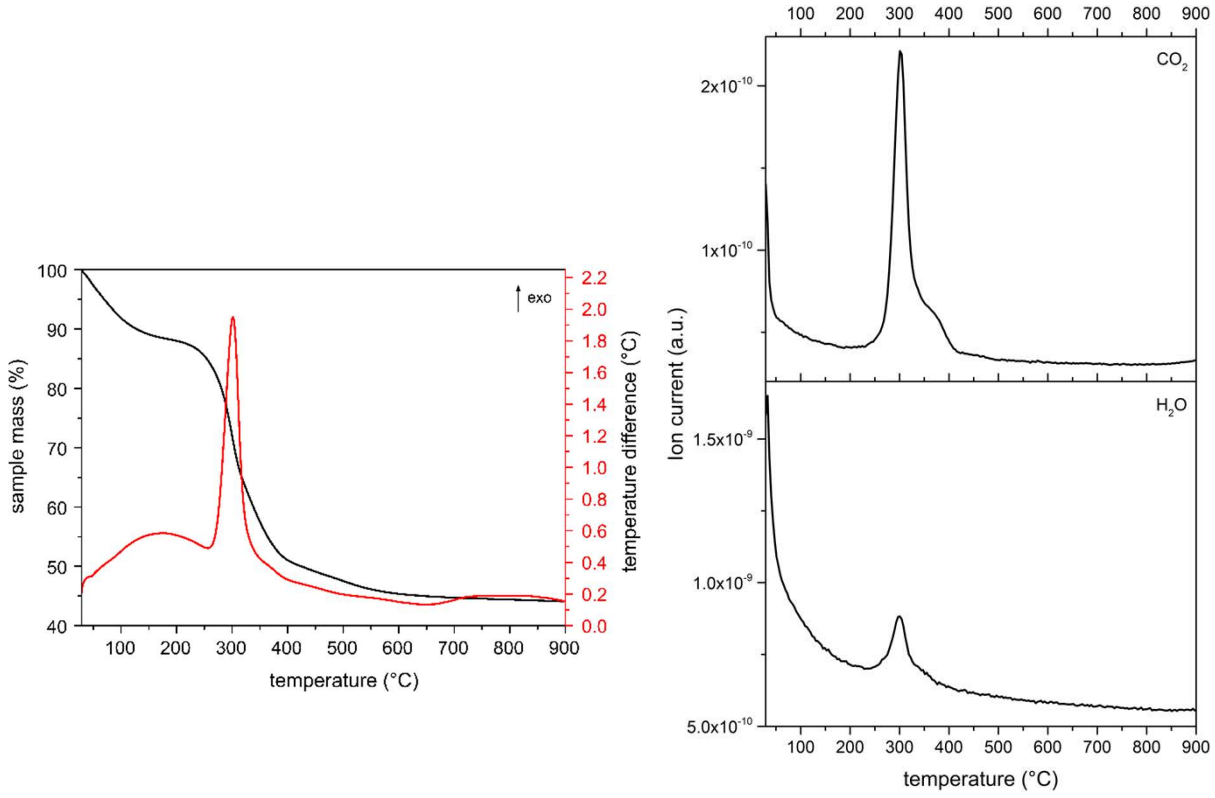

Figure S10. TG-DTA-MS analysis of sample MgAl-7COO.

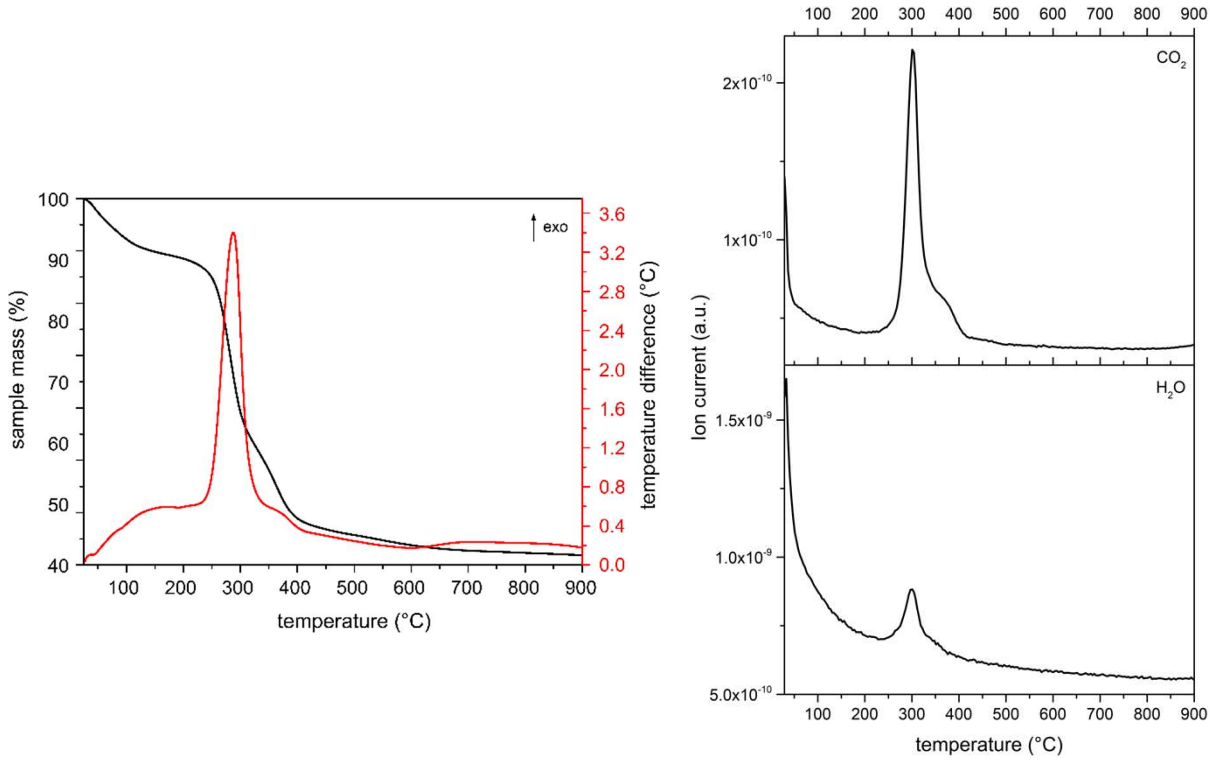

Figure S11. TG-DTA-MS analysis of sample MgAl-11COO.

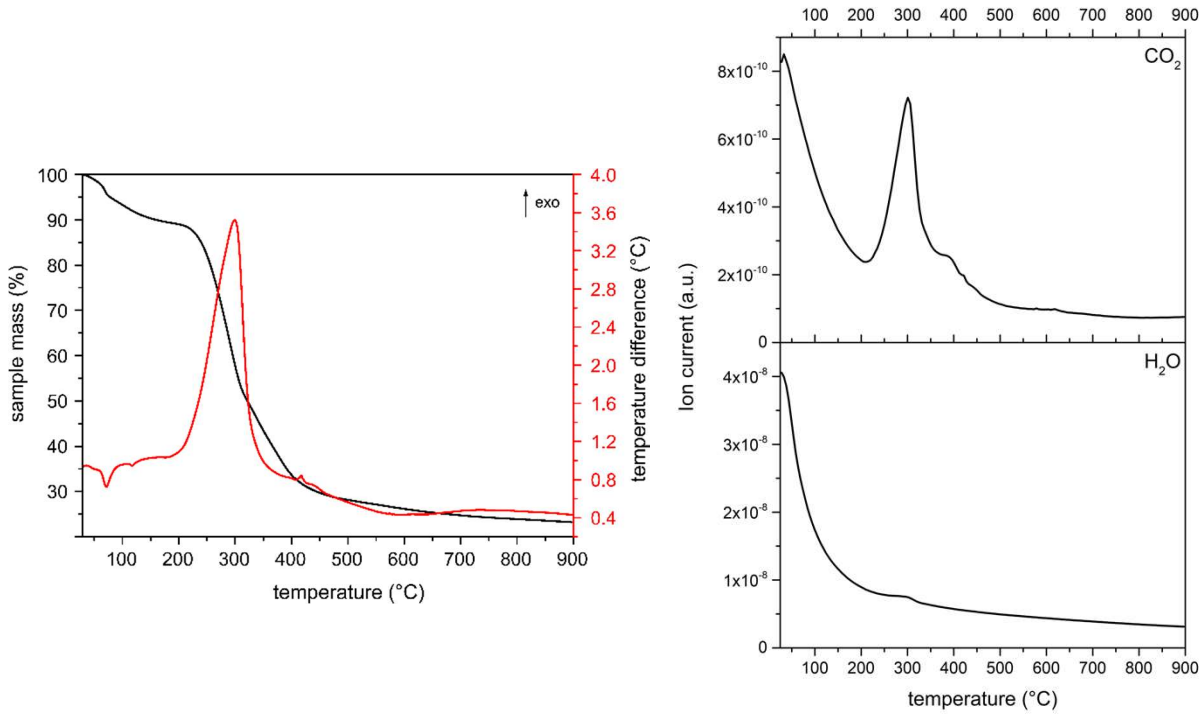

Figure S12. TG-DTA-MS analysis of sample MgAl-17COO.

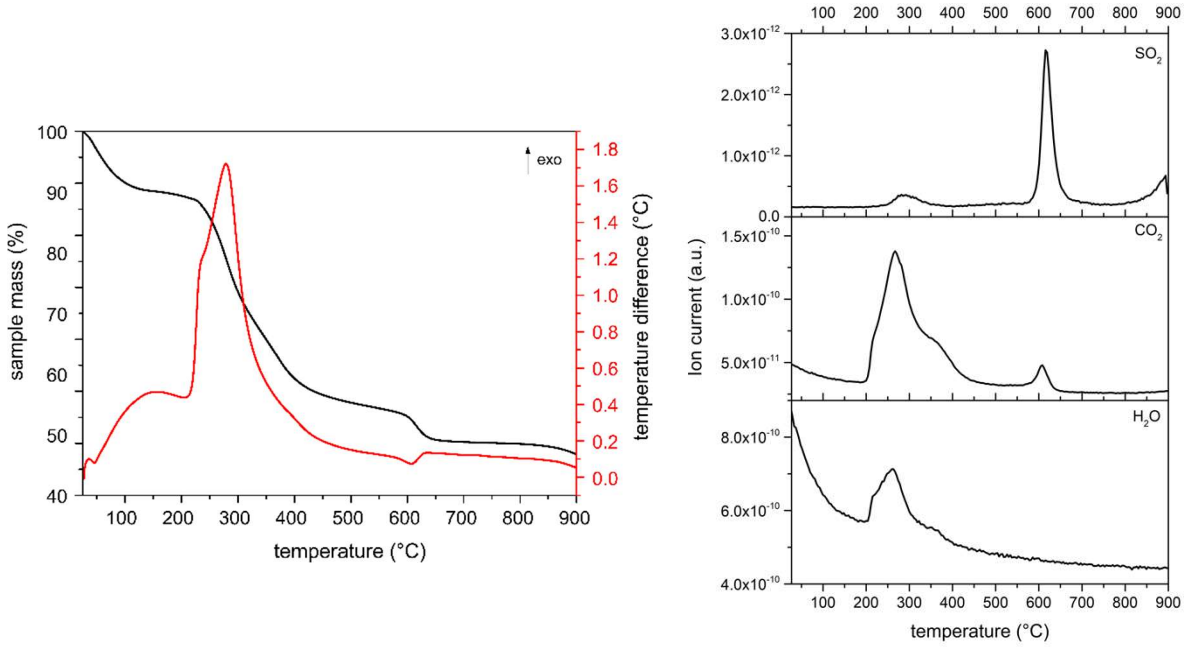

Figure S13. TG-DTA-MS analysis of sample MgAl-12SO3.

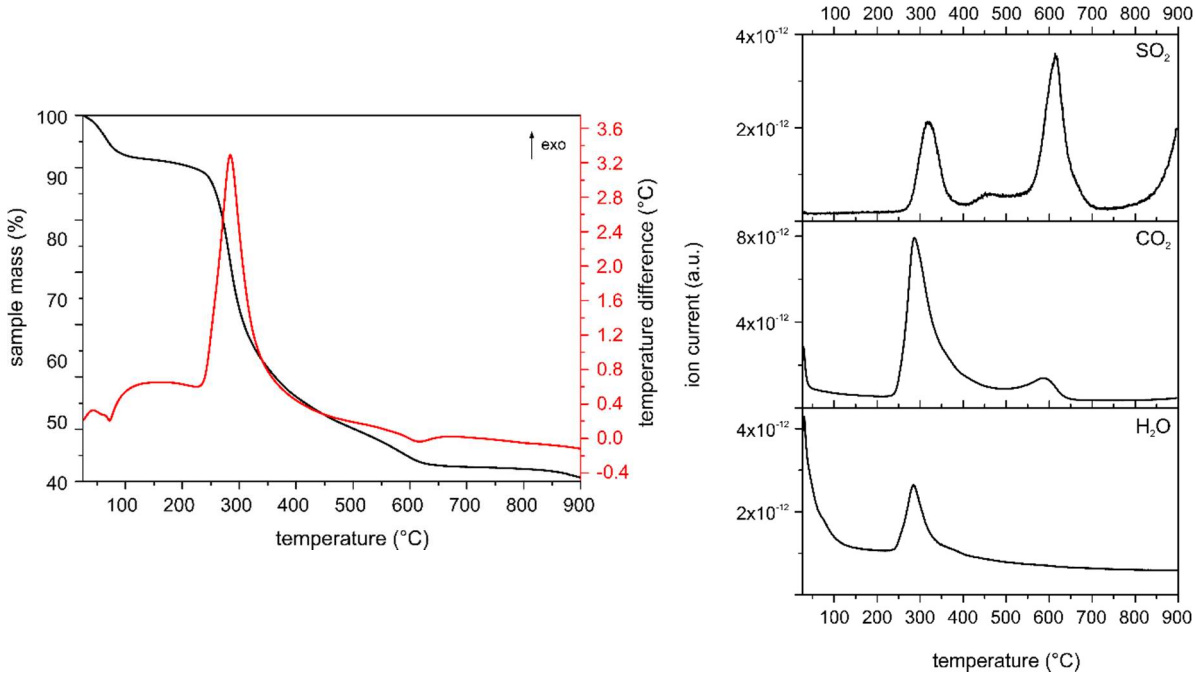

Figure S14. TG-DTA-MS analysis of sample MgAl-18SO3.

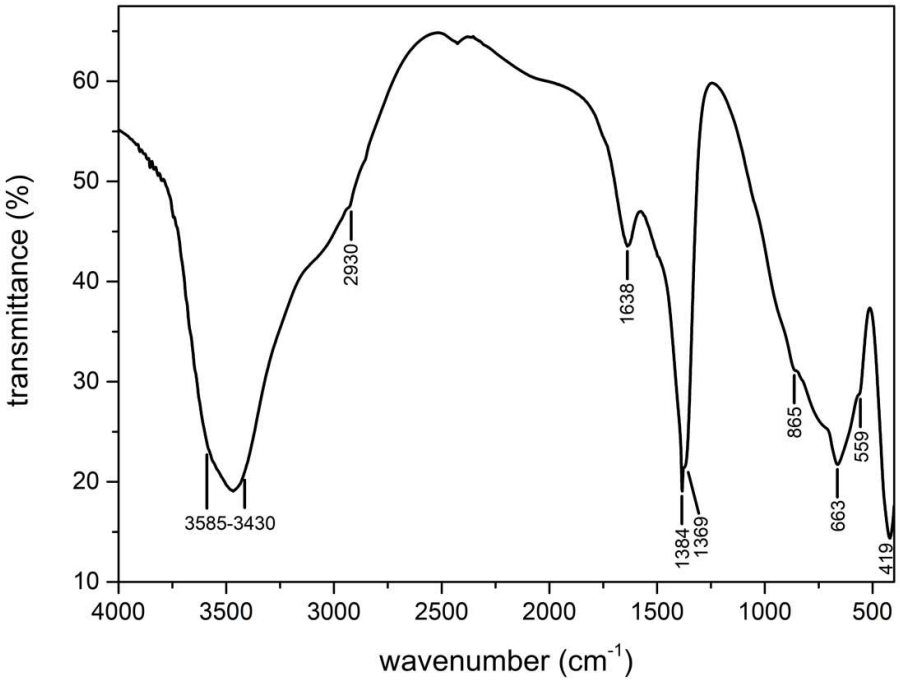

Figure S15. FTIR spectrum of sample MgAl-CO3.

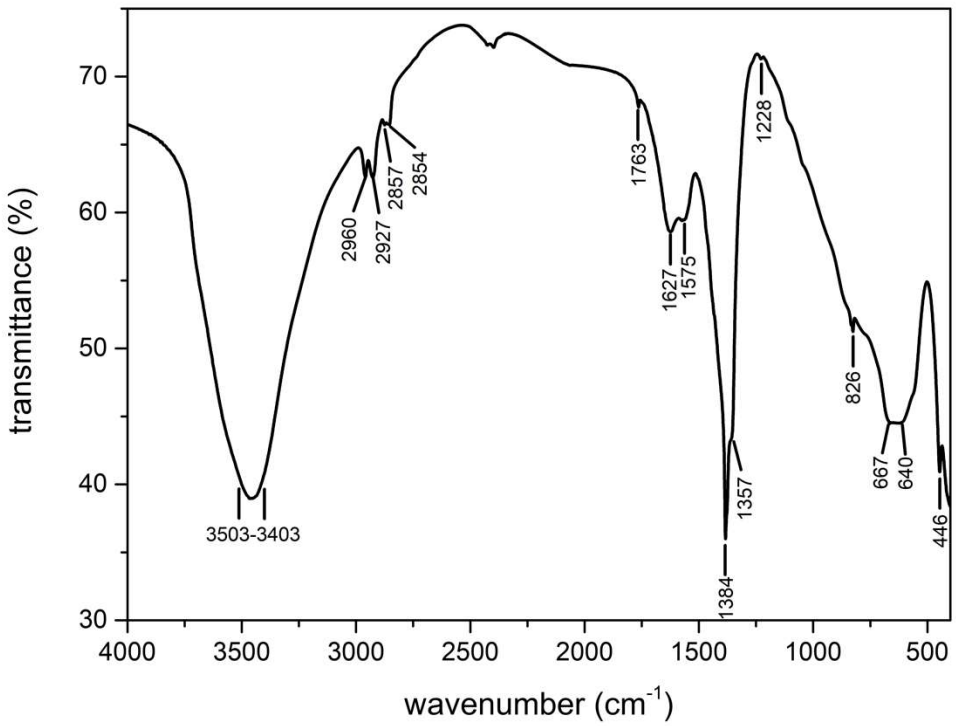

Figure S16. FTIR spectrum of sample MgAl-5COO.

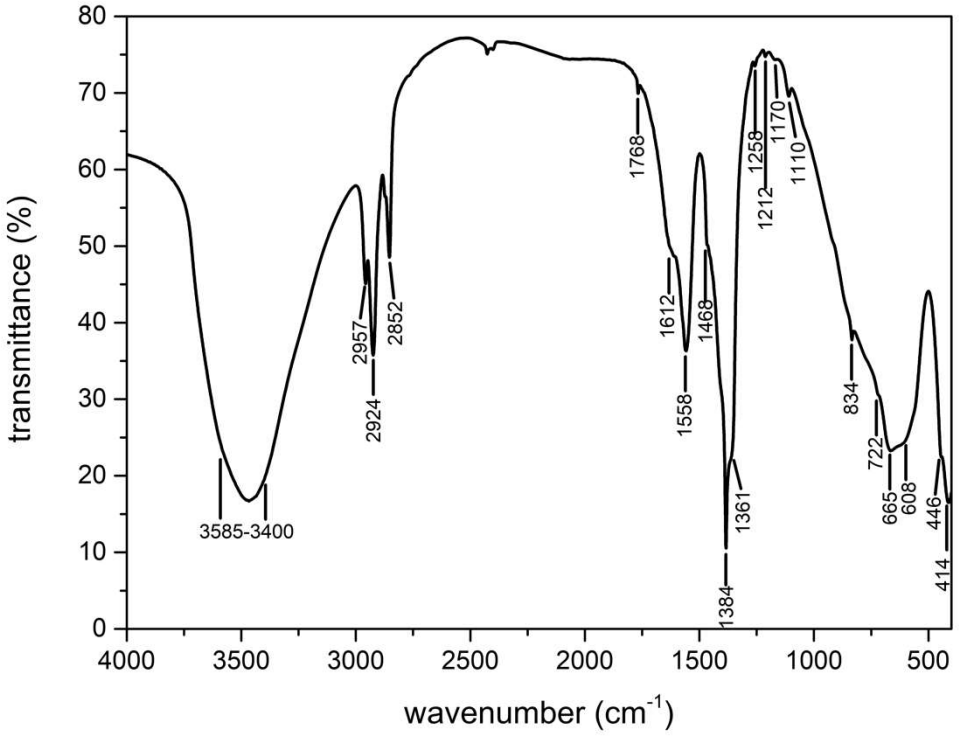

Figure S17. FTIR spectrum of sample MgAl-7COO

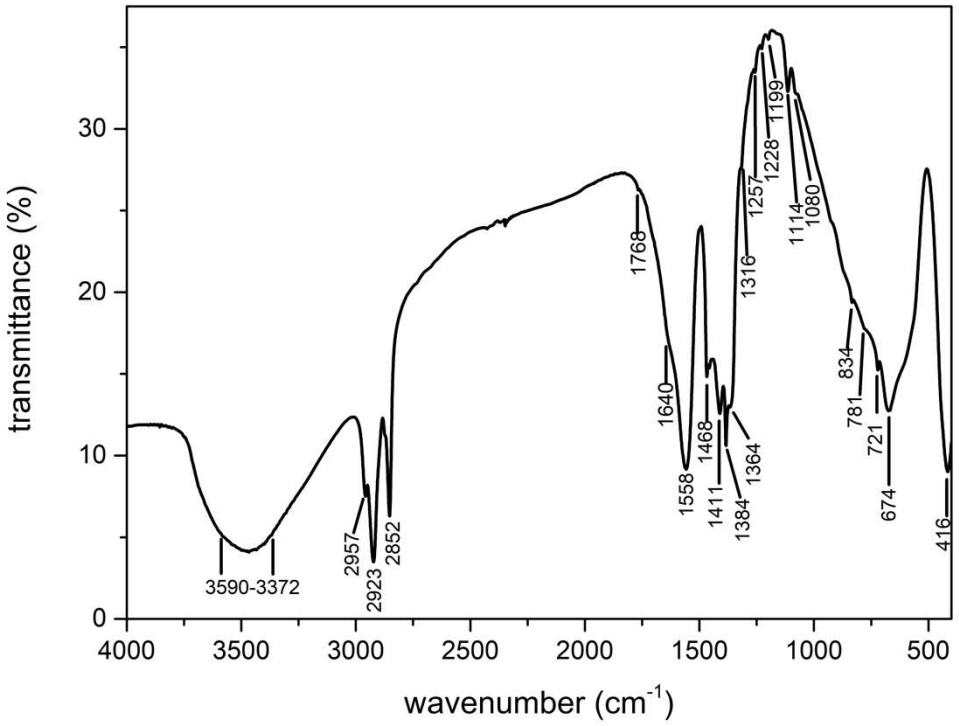

Figure S18. FTIR spectrum of sample MgAl-11COO

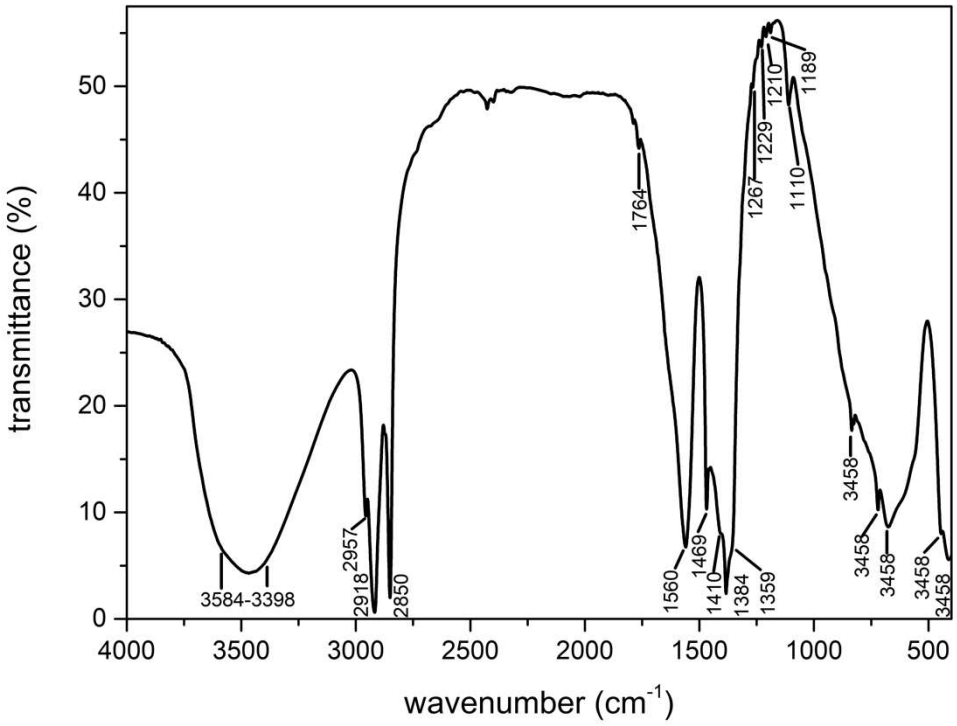

Figure S19. FTIR spectrum of sample MgAl-17COO

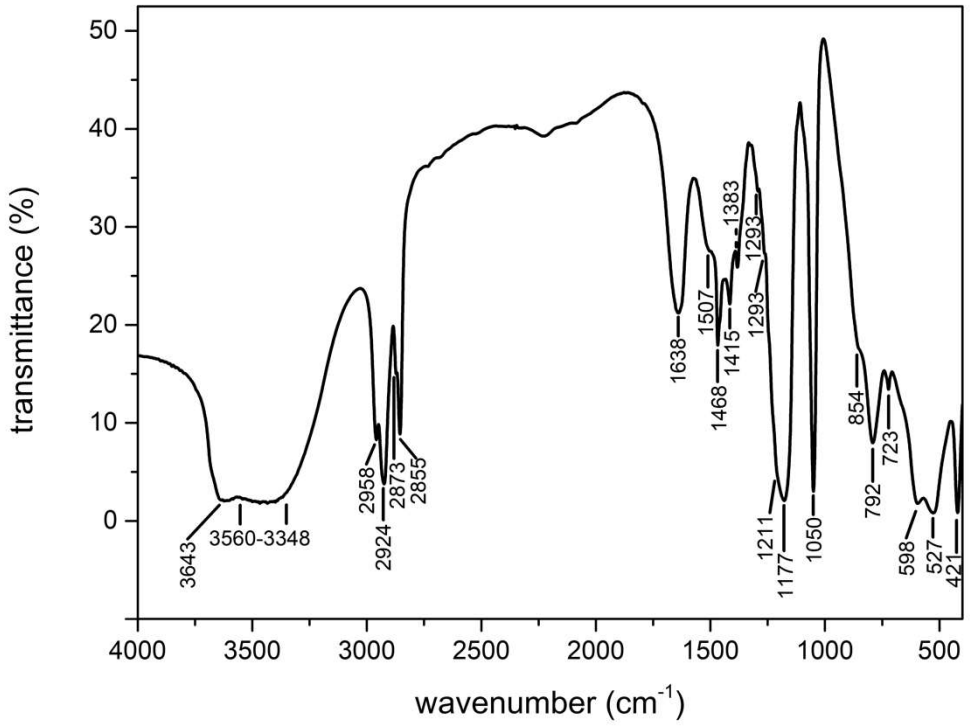

Figure S20. FTIR spectrum of sample MgAl-8SO3.

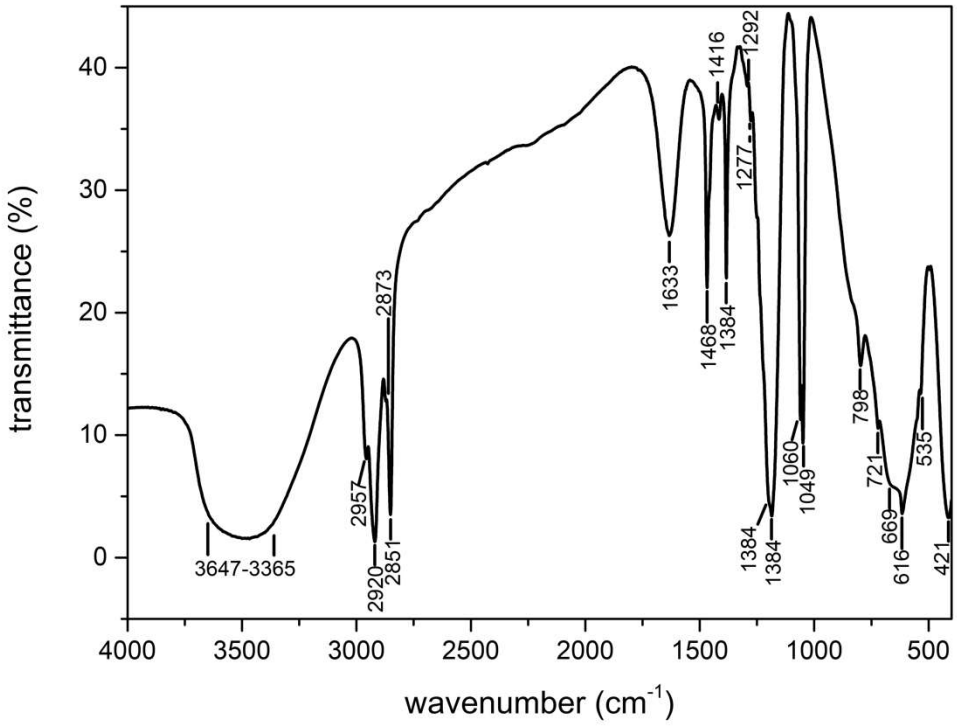

Figure S21. FTIR spectrum of the sample MgAl-12SO3.

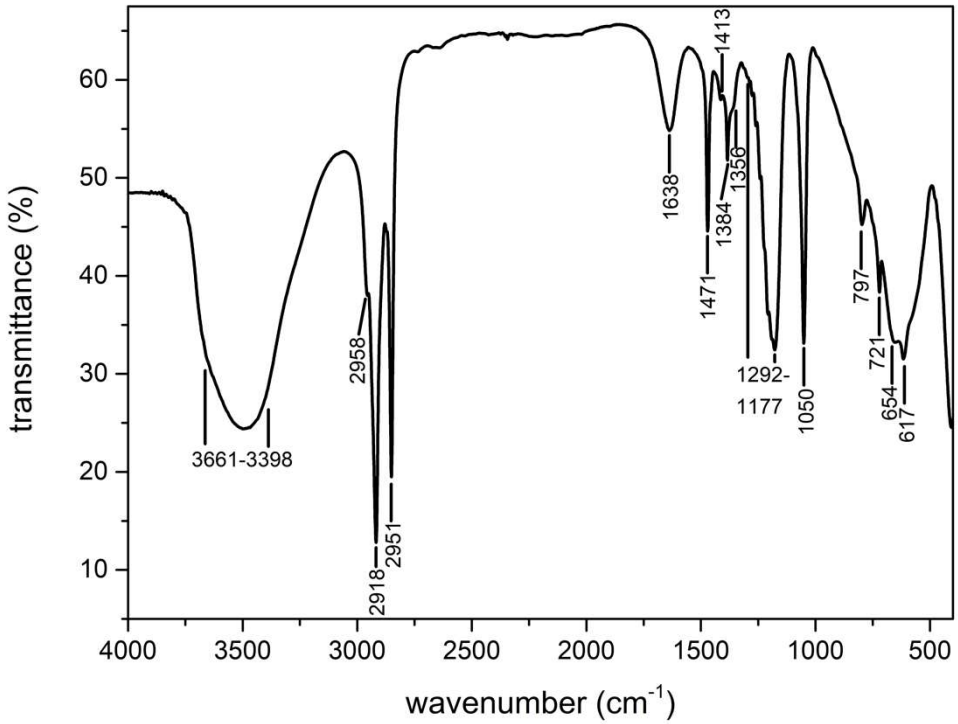

Figure S22. FTIR spectrum of sample MgAl-18SO3

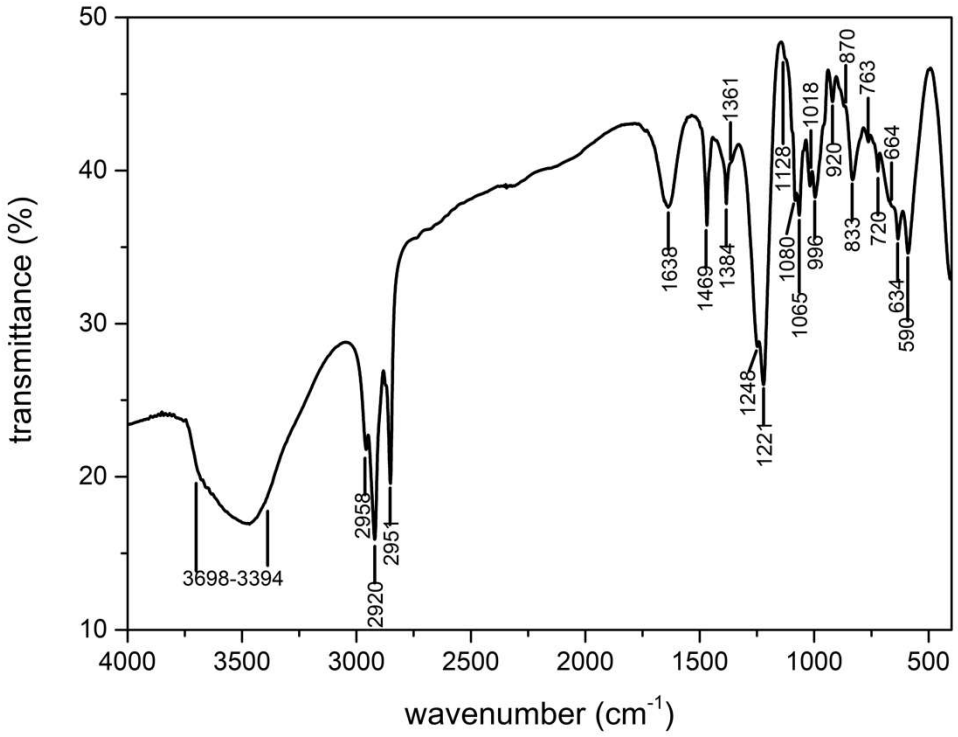

Figure S23. FTIR spectrum of sample MgAl-12SO4.

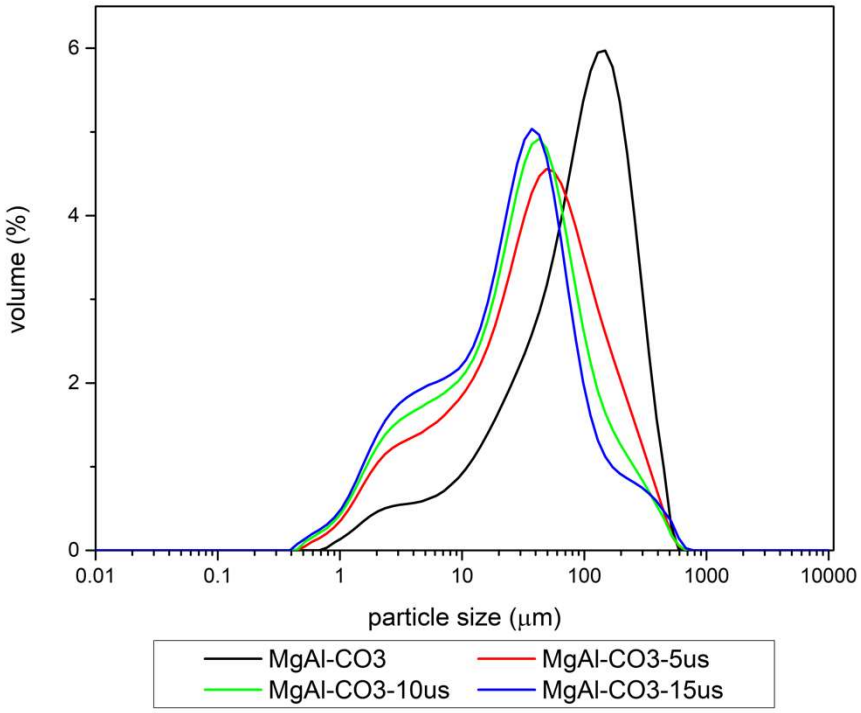

Figure S24. Particle size distribution of the dried reference sample MgAl-CO3

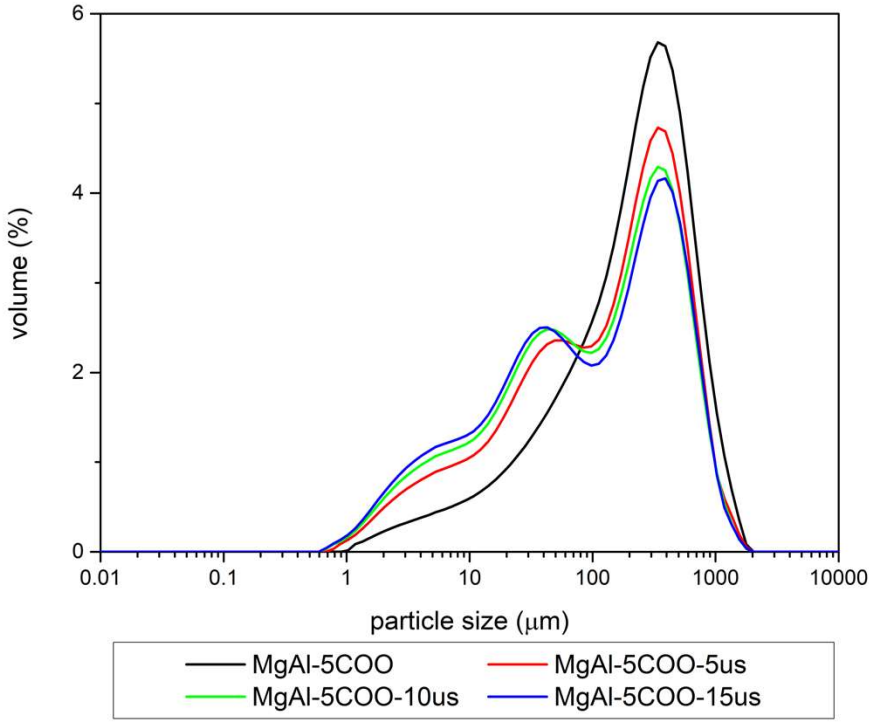

Figure S25. Particle size distribution of the dried sample MgAl-5COO

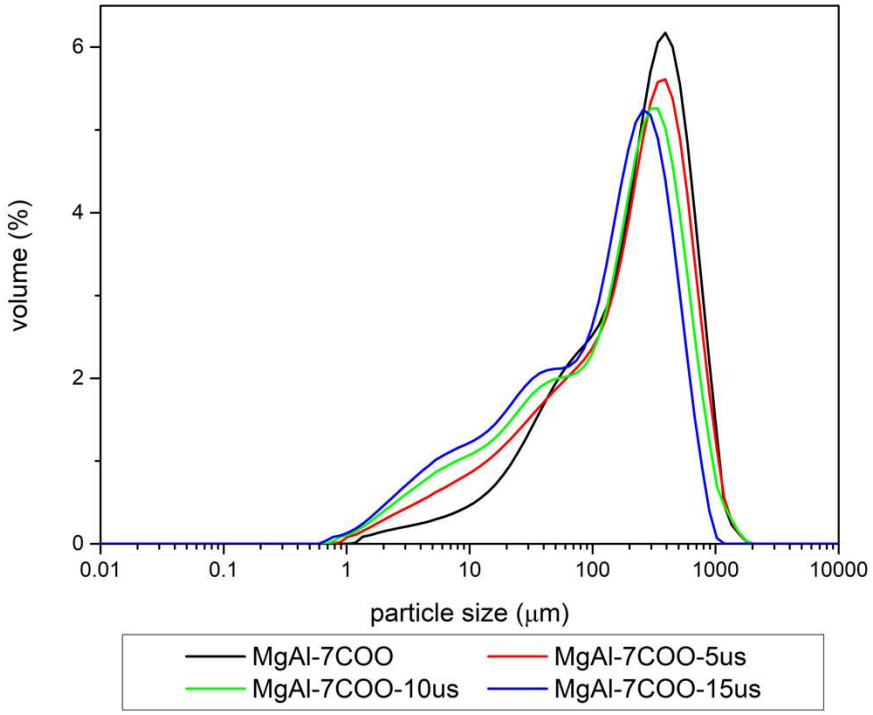

Figure S26. Particle size distribution of the dried sample MgAl-7COO

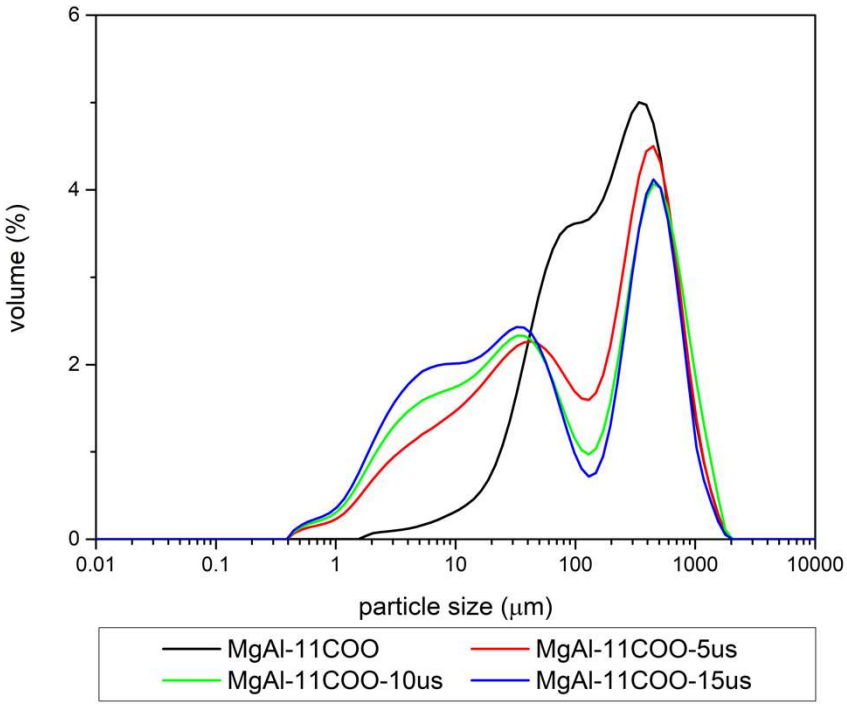

Figure S27. Particle size distribution of the dried sample MgAl-11COO

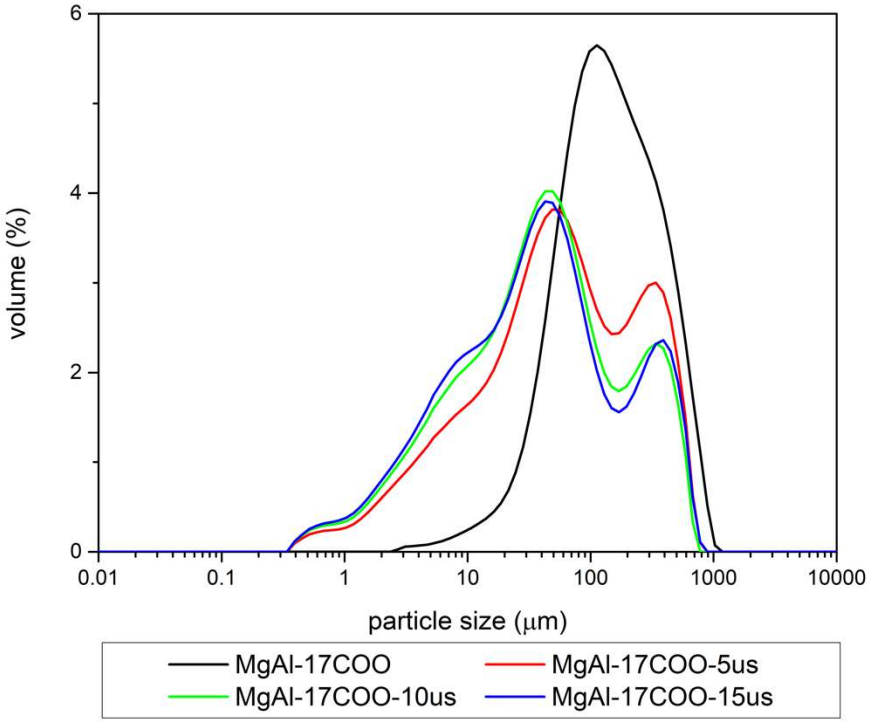

Figure S28. Particle size distribution of the dried sample MgAl-17COO

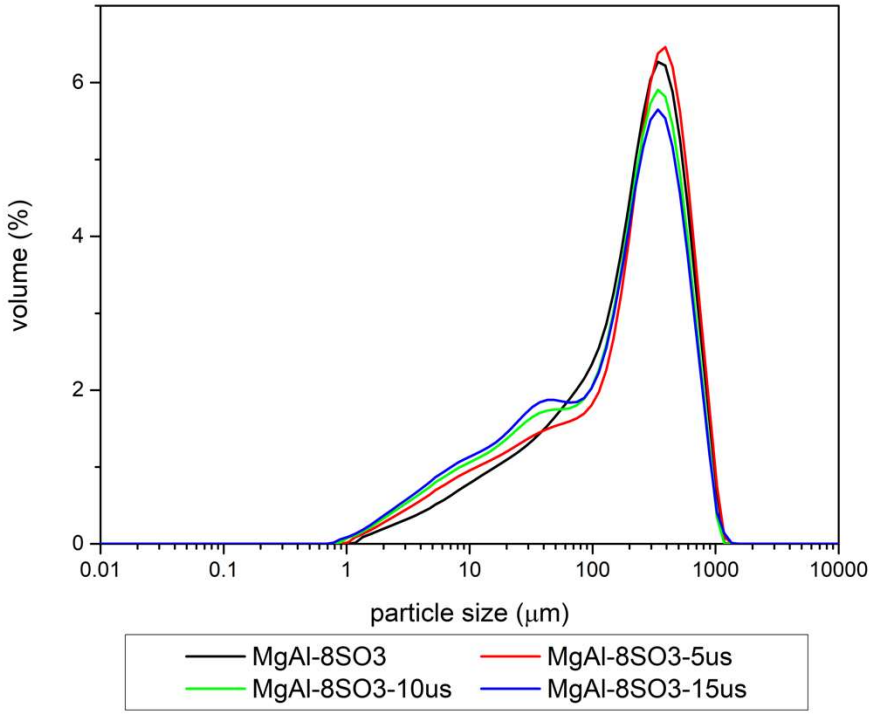

Figure S29. Particle size distribution of the dried sample MgAl-8SO3

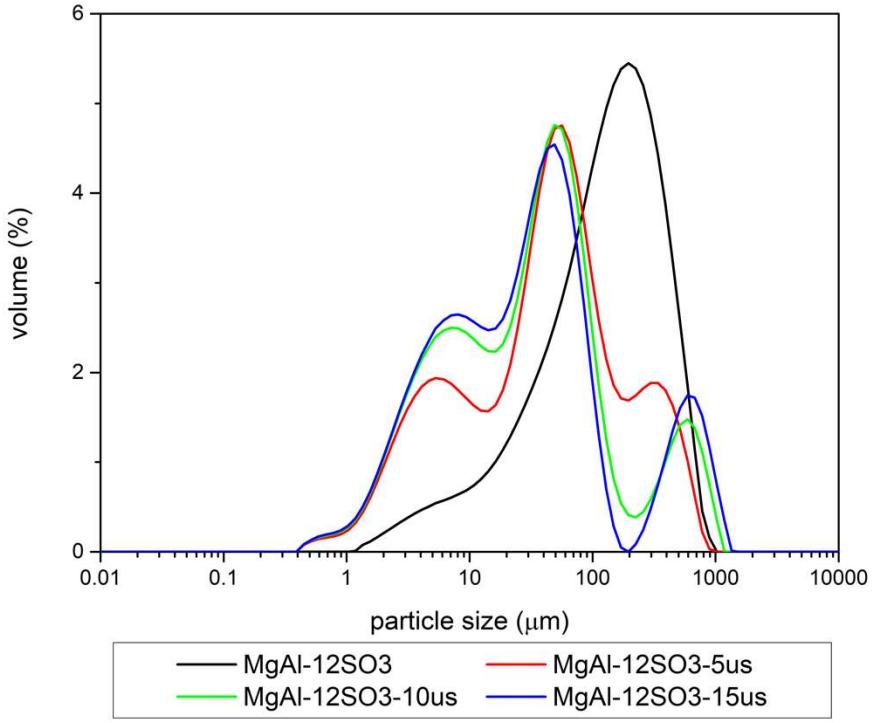

Figure S30. Particle size distribution of the dried sample MgAl-12SO3

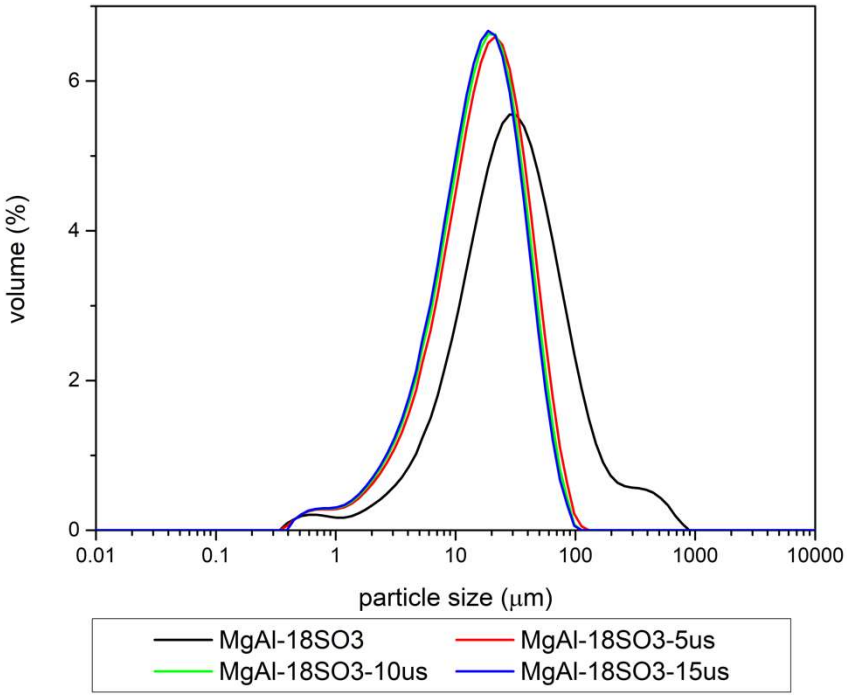

Figure S31. Particle size distribution of the dried sample MgAl-18SO3

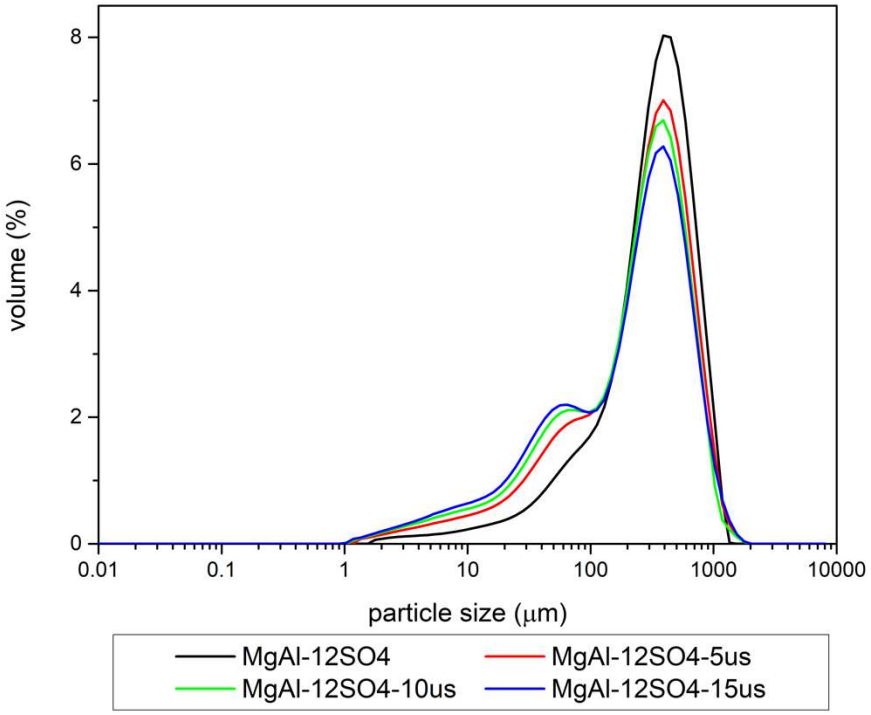

Figure S32. Particle size distribution of the dried sample MgAl-12SO4.

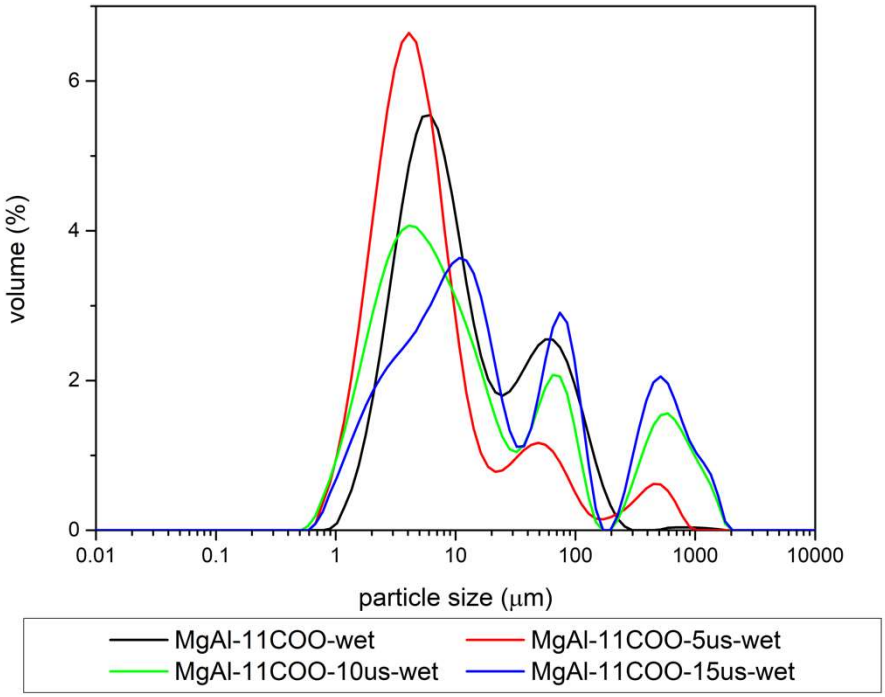

Figure S33. Particle size distribution of the wet sample MgAl-11COO.

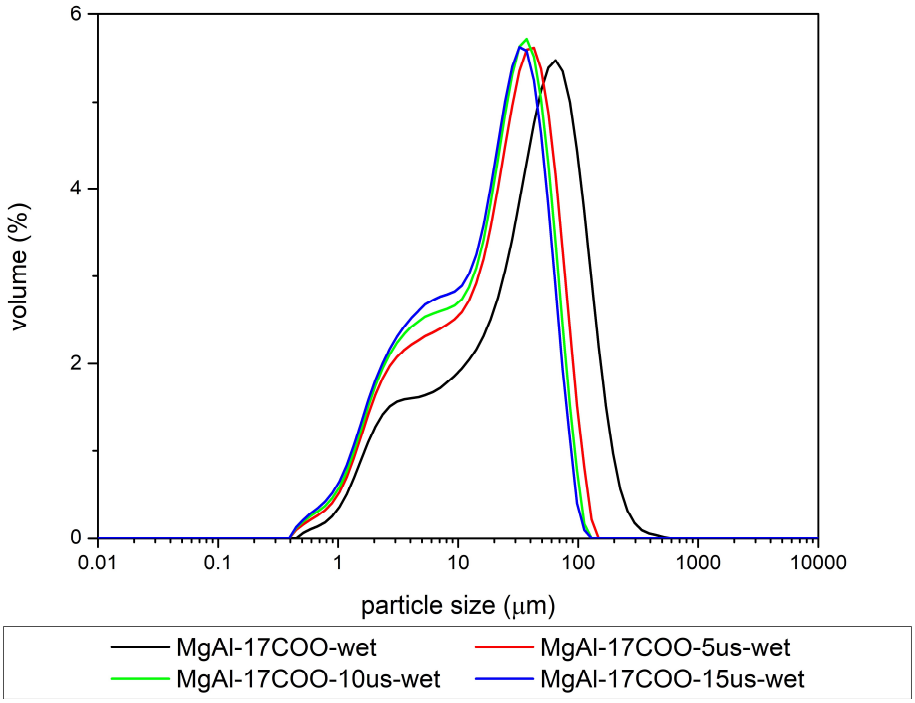

Figure S34. Particle size distribution of the wet sample MgAl-17COO.

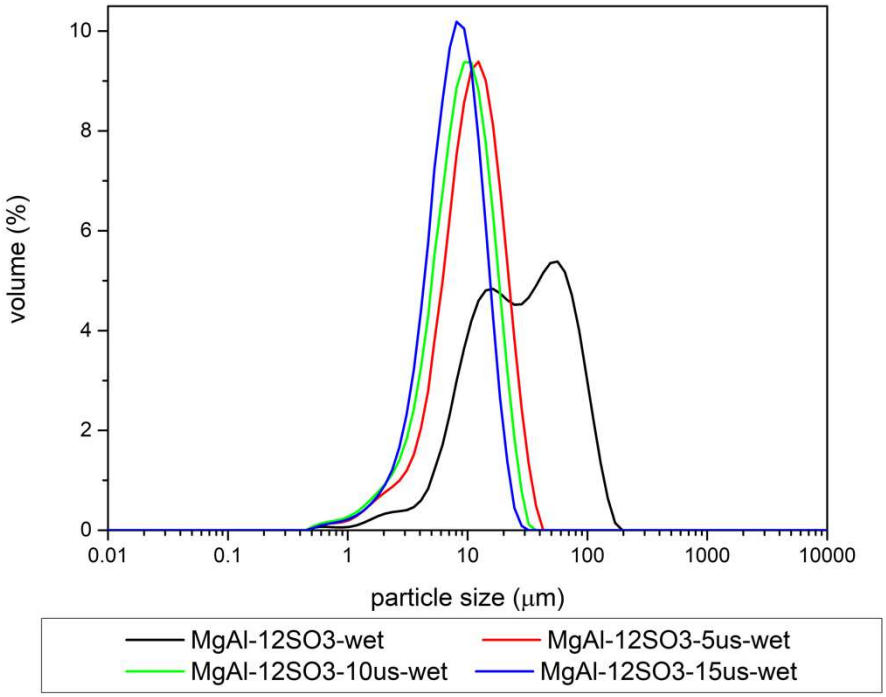

Figure S35. Particle size distribution of the wet sample MgAl-12SO3.

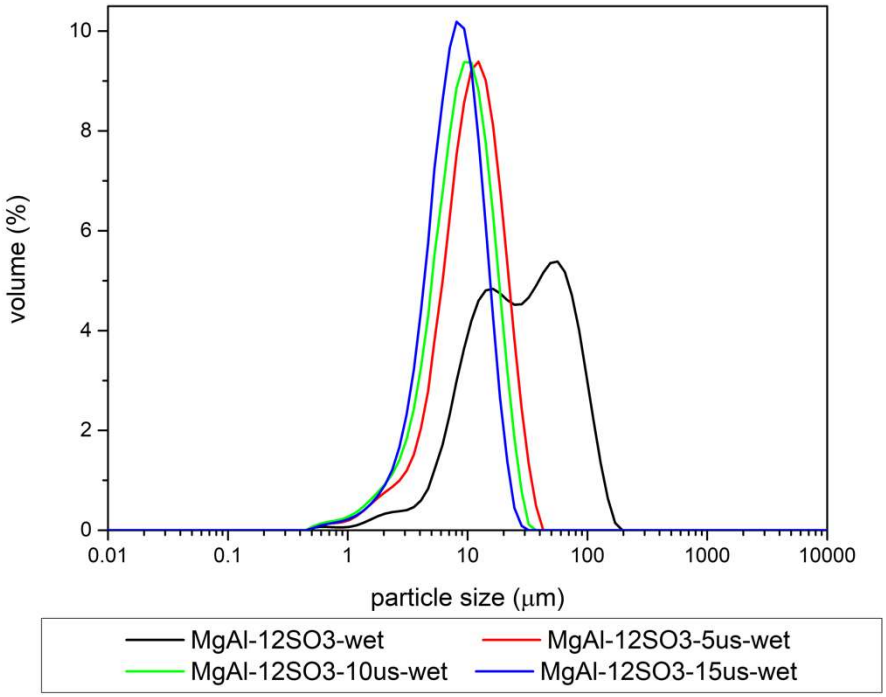

Figure S36. Particle size distribution of the wet sample MgAl-18SO3.

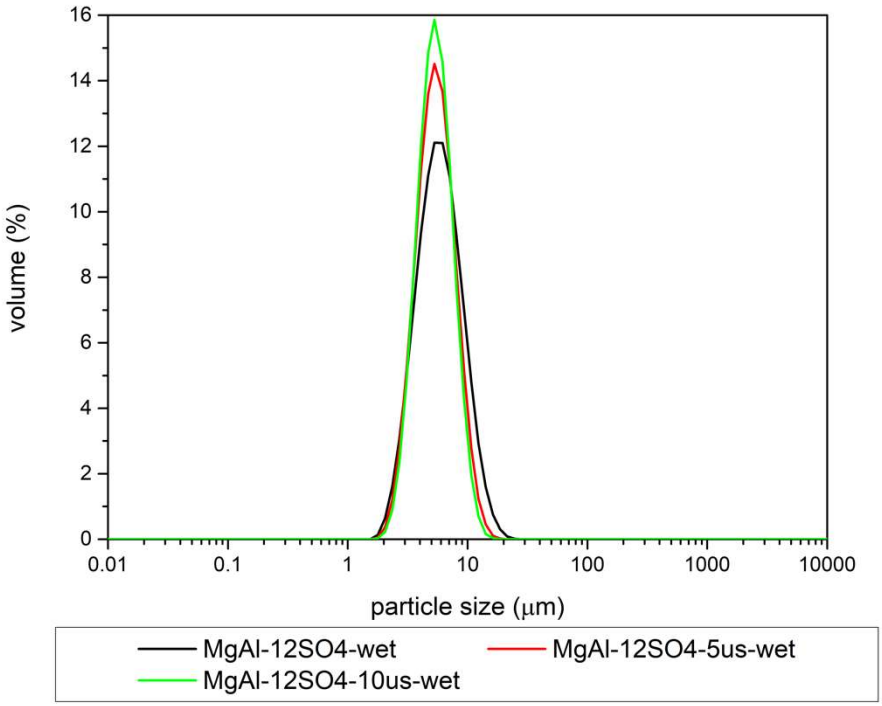

Figure S37. Particle size distribution of the wet sample MgAl-12SO4.

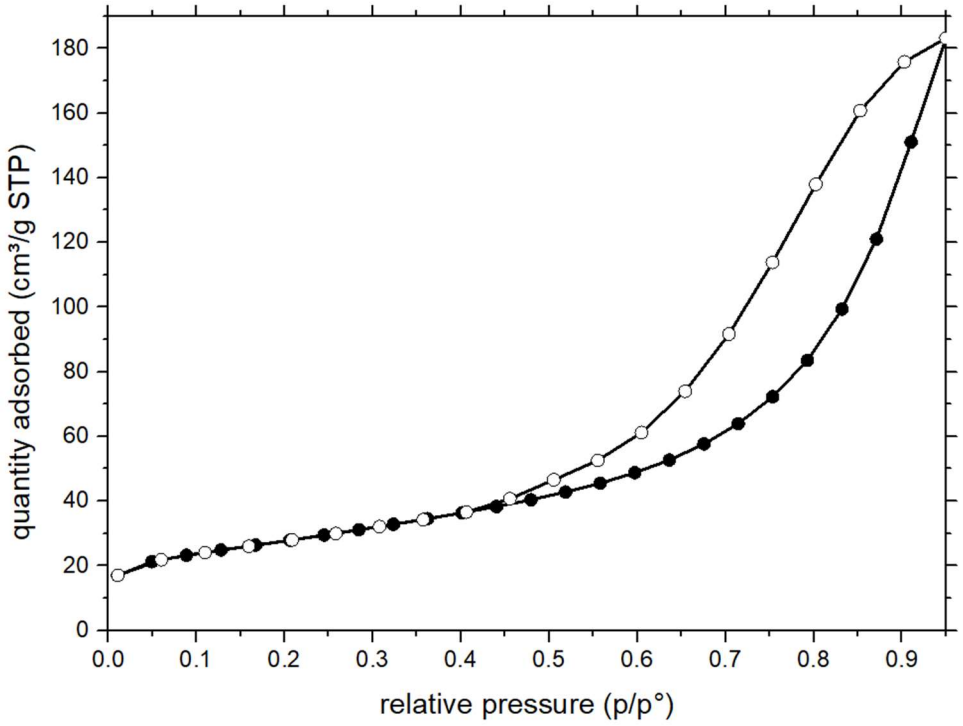

Figure S38. N_2 adsorption (filled circles)-desorption (empty circles) isotherm of the refence sample MgAl-CO3.

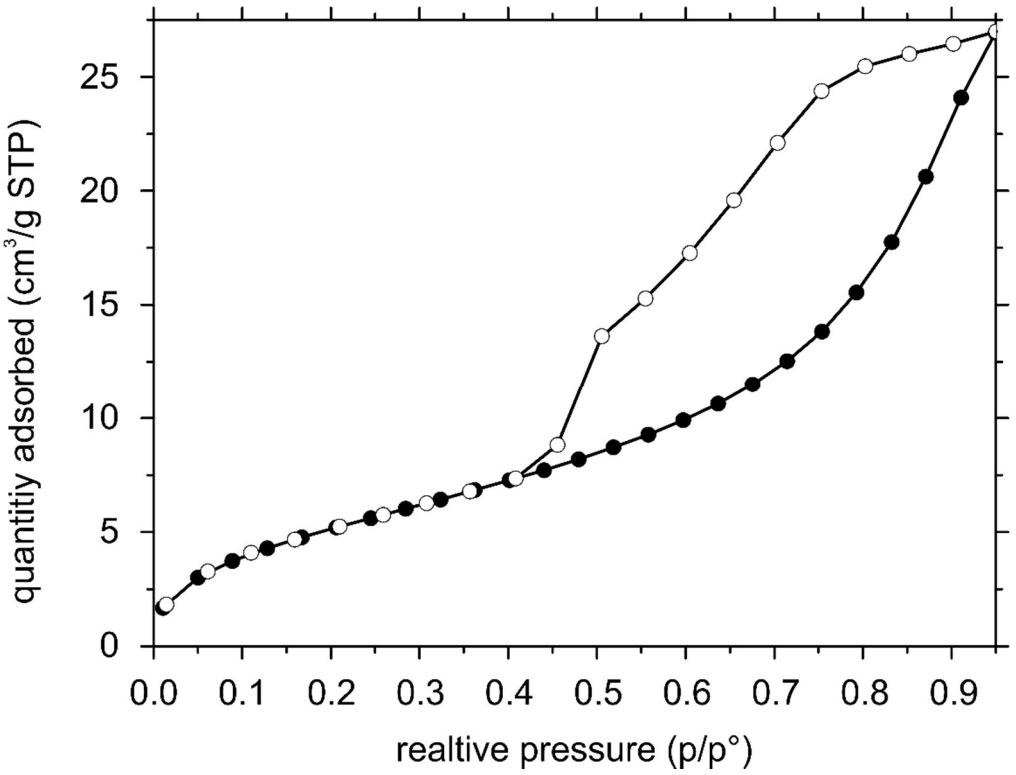

Figure S39. N_2 adsorption (filled circles)-desorption (empty circles) isotherm of sample MgAl-5COO.

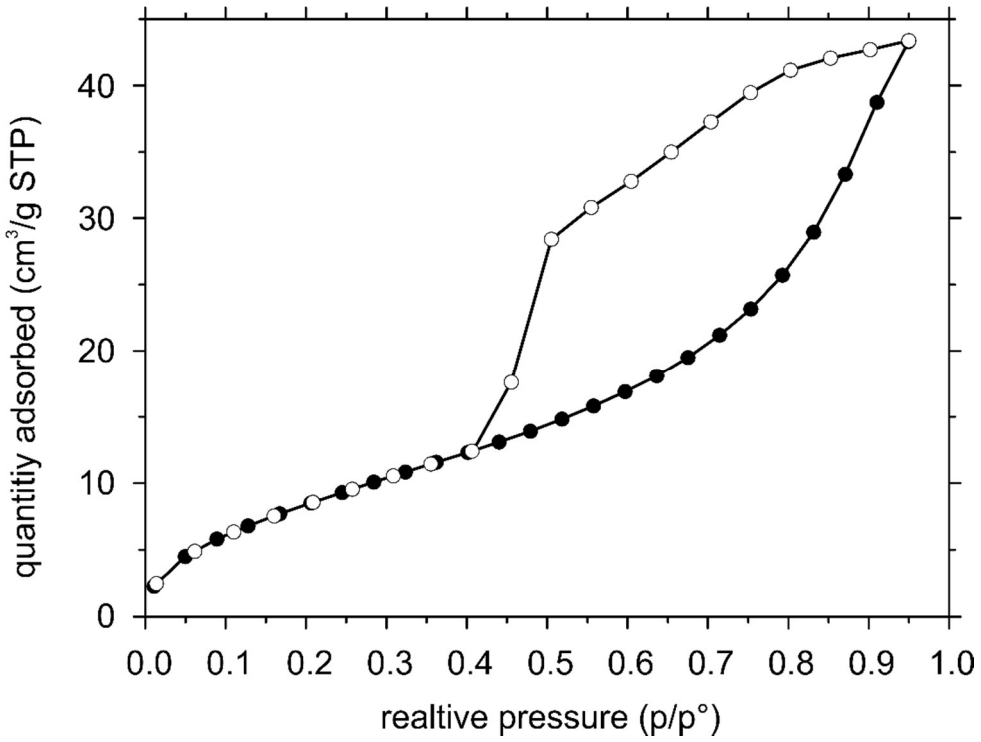

Figure S40. N_2 adsorption (filled circles)-desorption (empty circles) isotherm of sample MgAl-7COO.

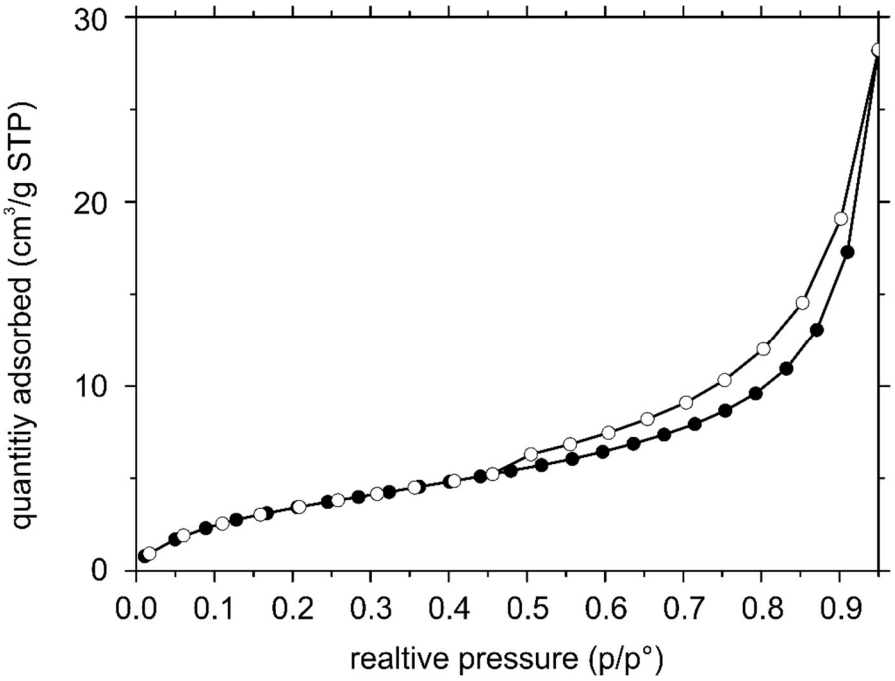

Figure S41. N_2 adsorption (filled circles)-desorption (empty circles) isotherm of sample MgAl-11COO.

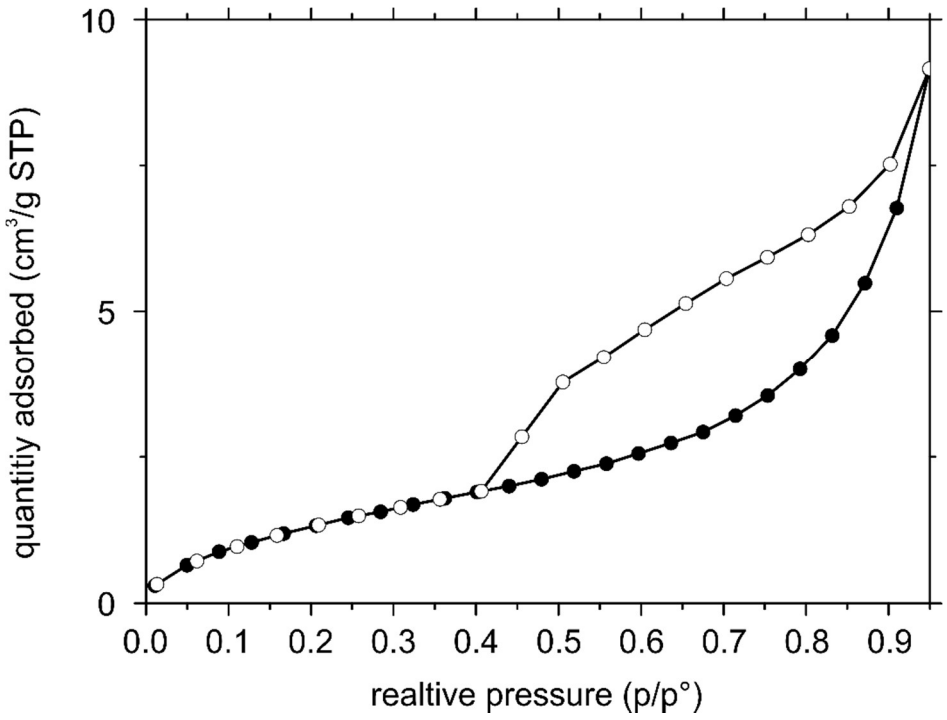

Figure S42. N_2 adsorption (filled circles)-desorption (empty circles) isotherm of sample MgAl-17COO.

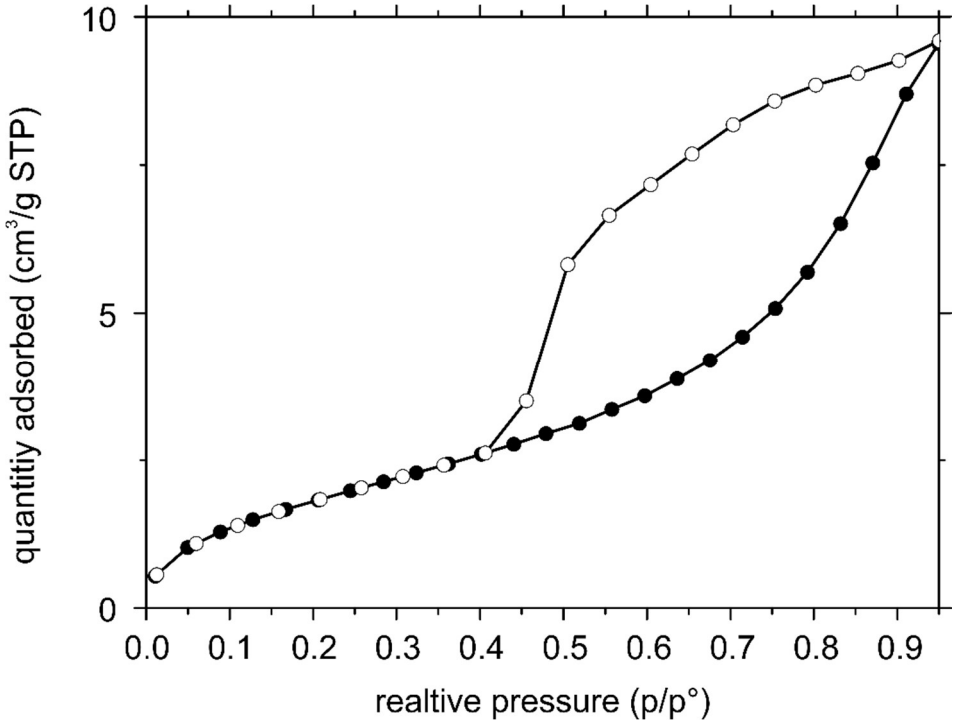

Figure S43. N_2 adsorption (filled circles)-desorption (empty circles) isotherm of sample MgAl-8SO3.

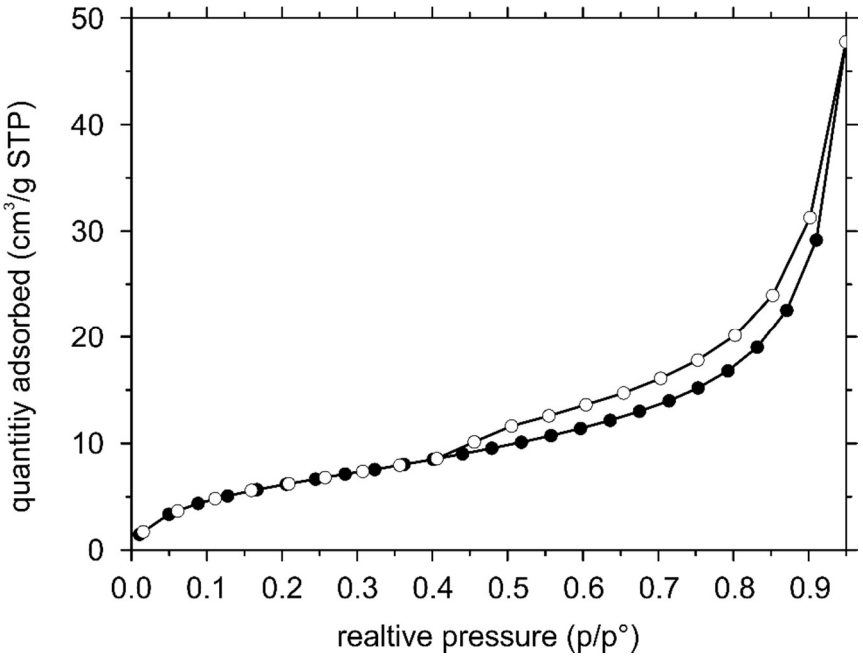

Figure S44. N_2 adsorption (filled circles)-desorption (empty circles) isotherm of sample MgAl-12SO3.

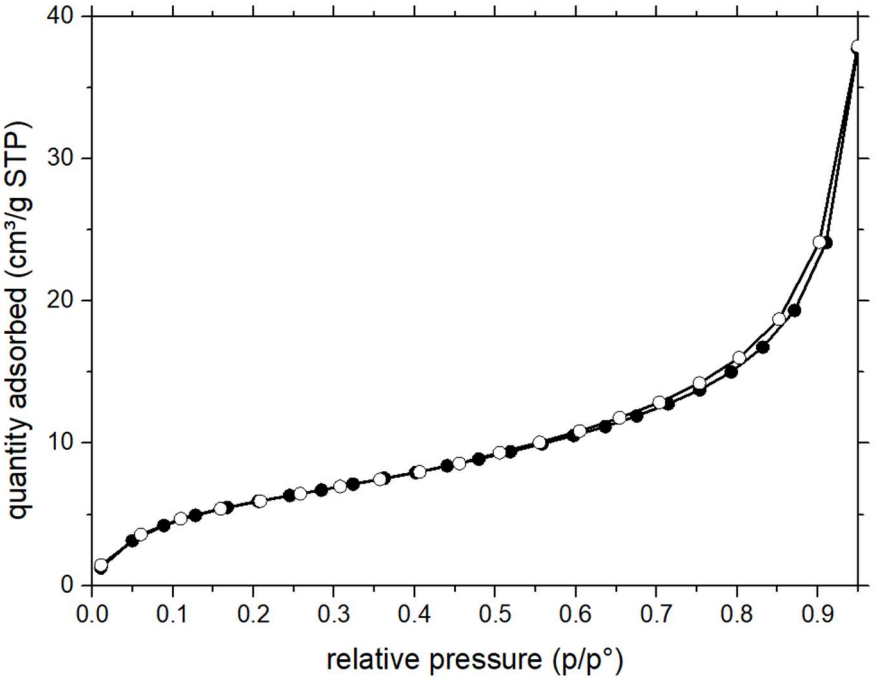

Figure S45. N_2 adsorption (filled circles)-desorption (empty circles) isotherm of sample MgAl-18SO3.

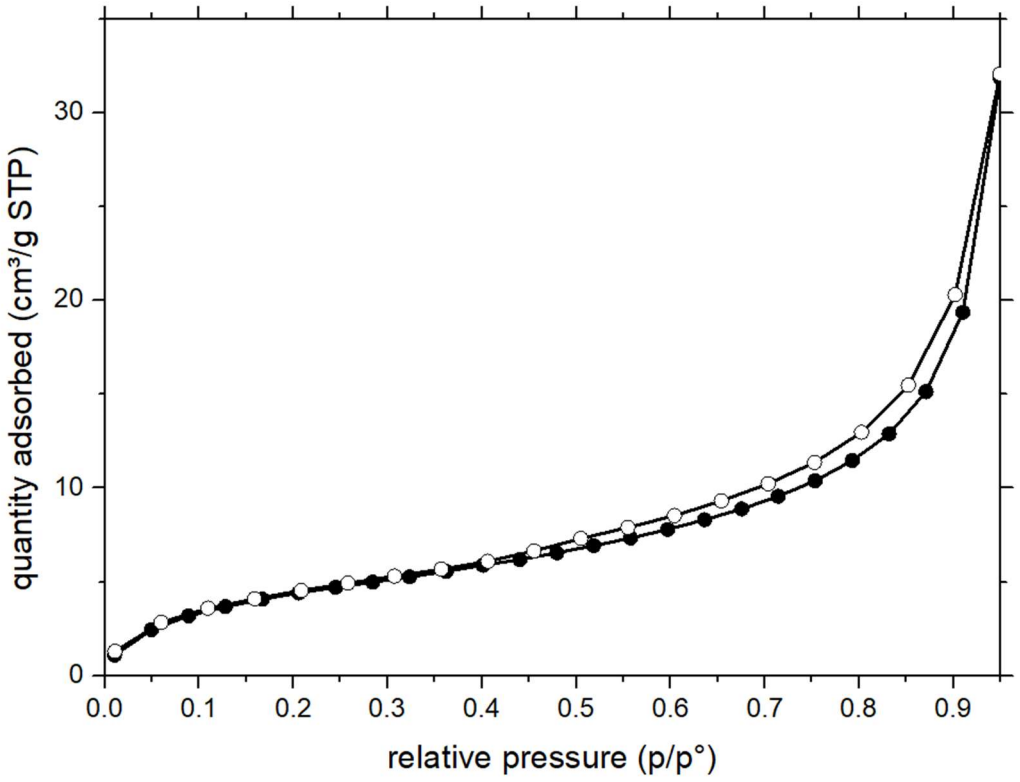

Figure S46. N_2 adsorption (filled circles)-desorption (empty circles) isotherm of sample MgAl-12SO4.

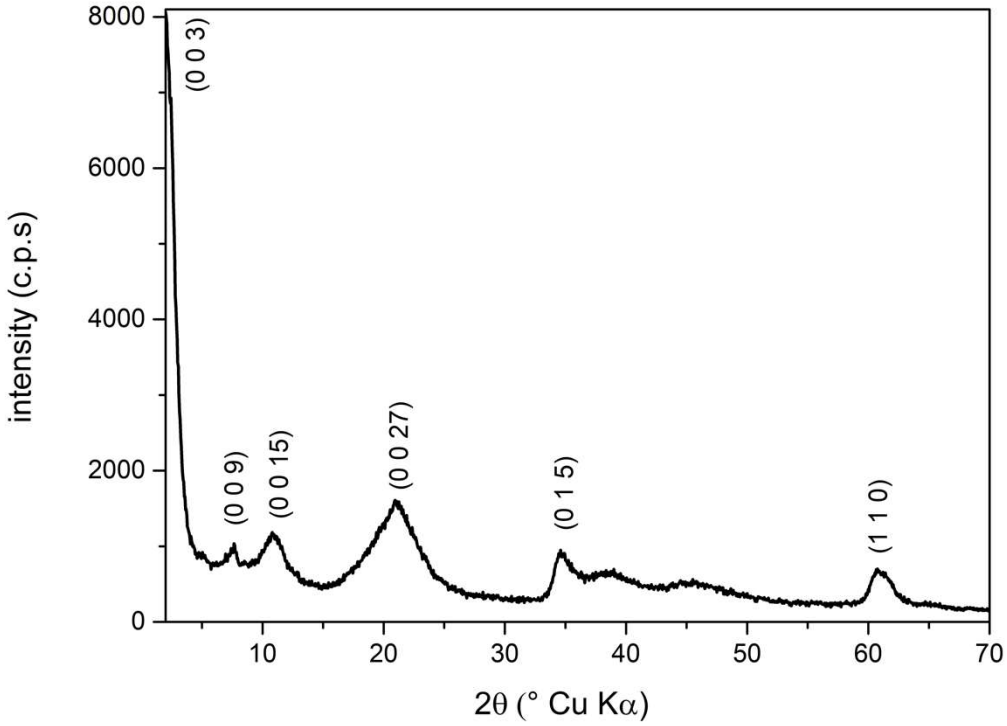

Figure S47. Powder X-Ray diffraction pattern of sample E-MgAl-11COO after a batch experiment with 1,1,2-TCA.

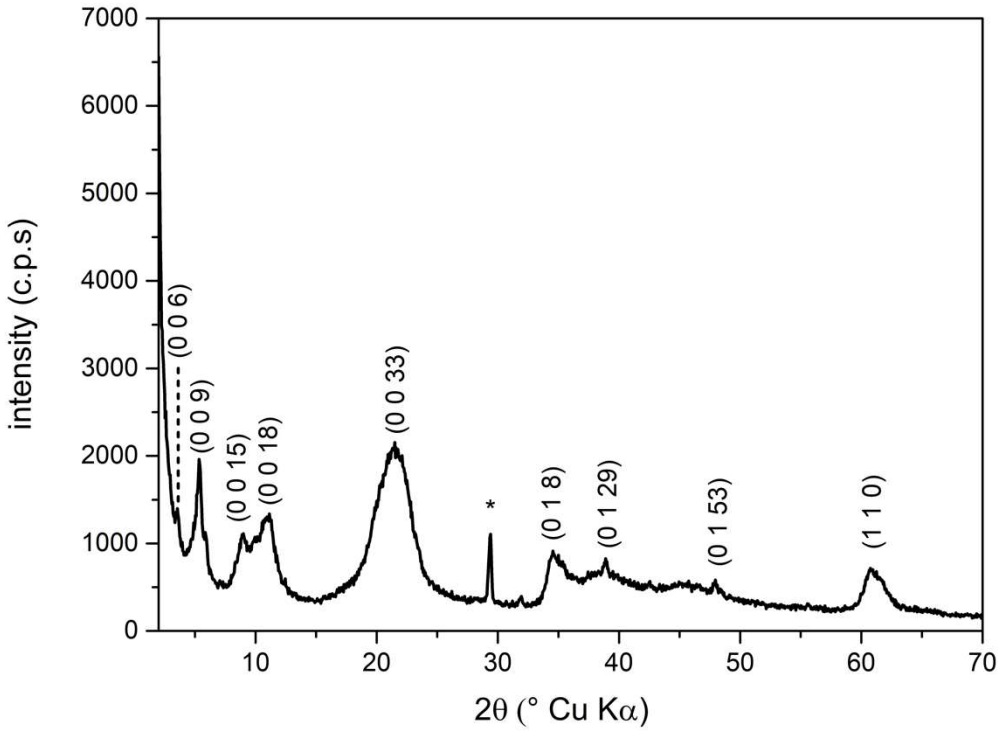

Figure S48. Powder X-Ray diffraction pattern of sample E-MgAl-17COO after a batch experiment with 1,1,2-TCA. (*) Sodium nitrate impurity.

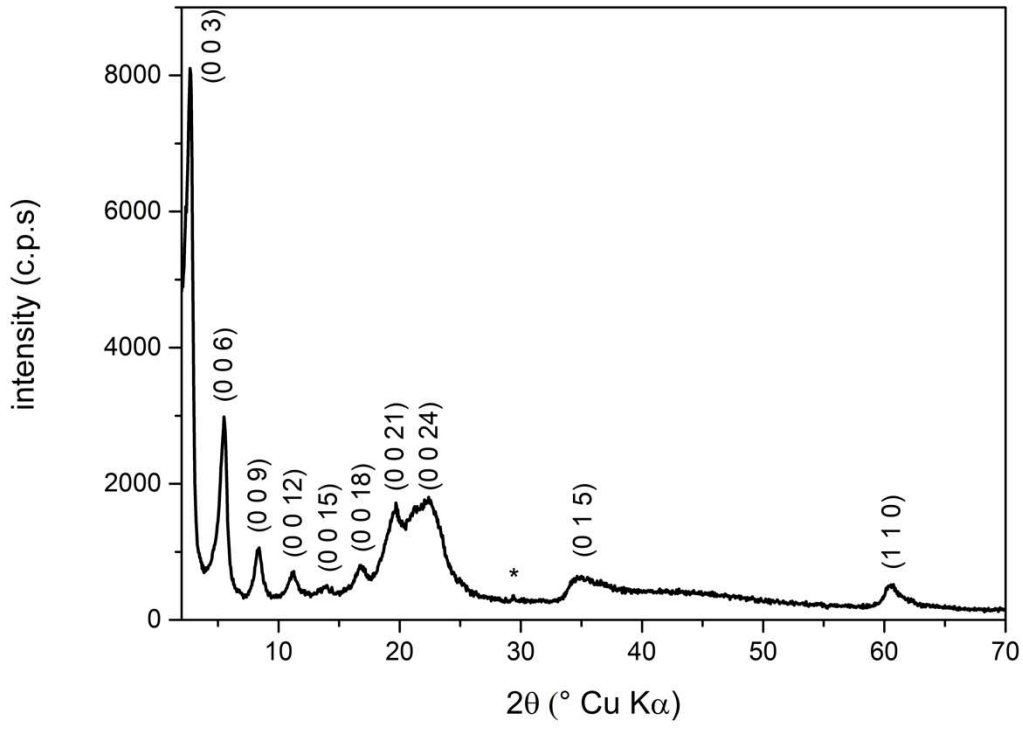

Figure S49. Powder X-Ray diffraction pattern of sample E-MgAl-18SO3 after a batch experiment with 1,1,2-TCA. (*) Sodium nitrate impurity

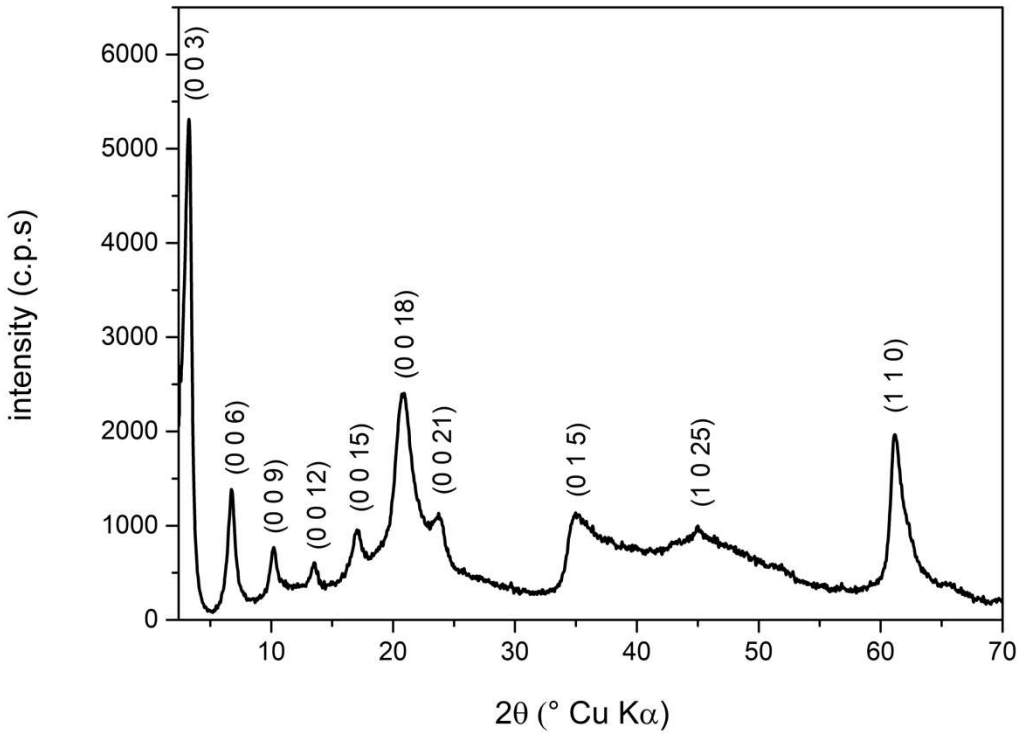

Figure S50. Powder X-Ray diffraction pattern of the reference sample E-MgAl-12SO4 after a batch experiment with 1,1,2-TCA.

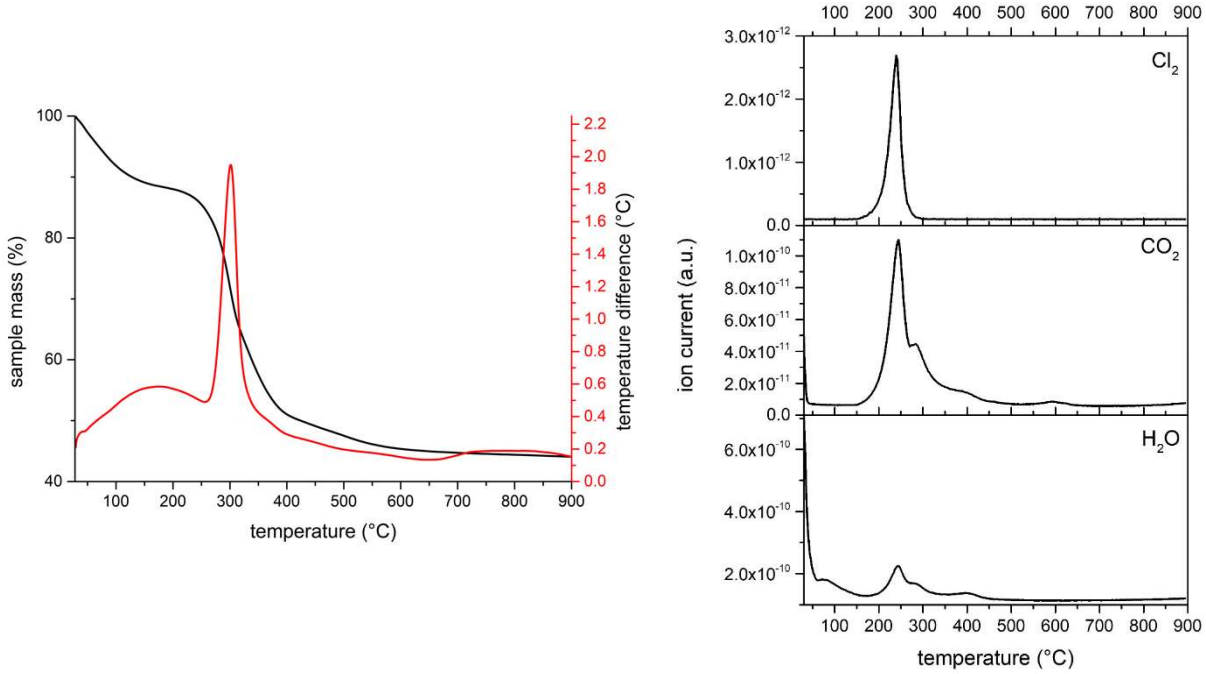

Figure S51. TG-DTA-MS analysis of sample E-MgAl-11COO after a batch experiment with 1,1,2-TCA.

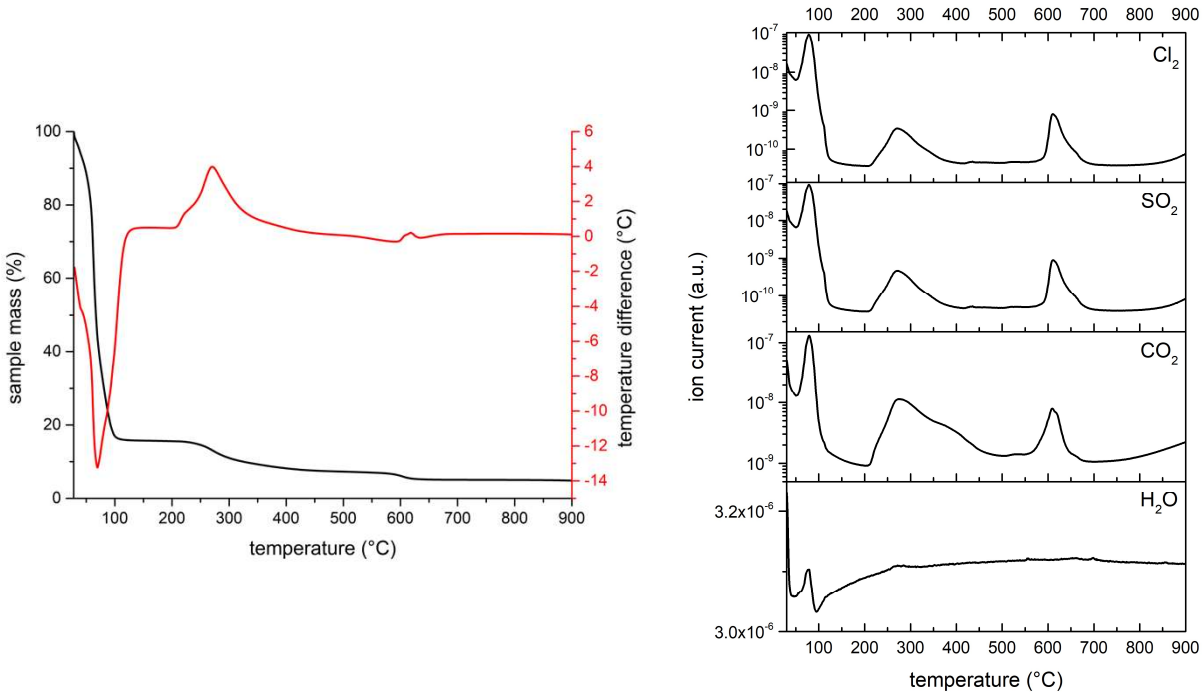

Figure S52. TG-DTA-MS analysis of sample E-MgAl-18SO3 after a batch experiment with 1,1,2-TCA.

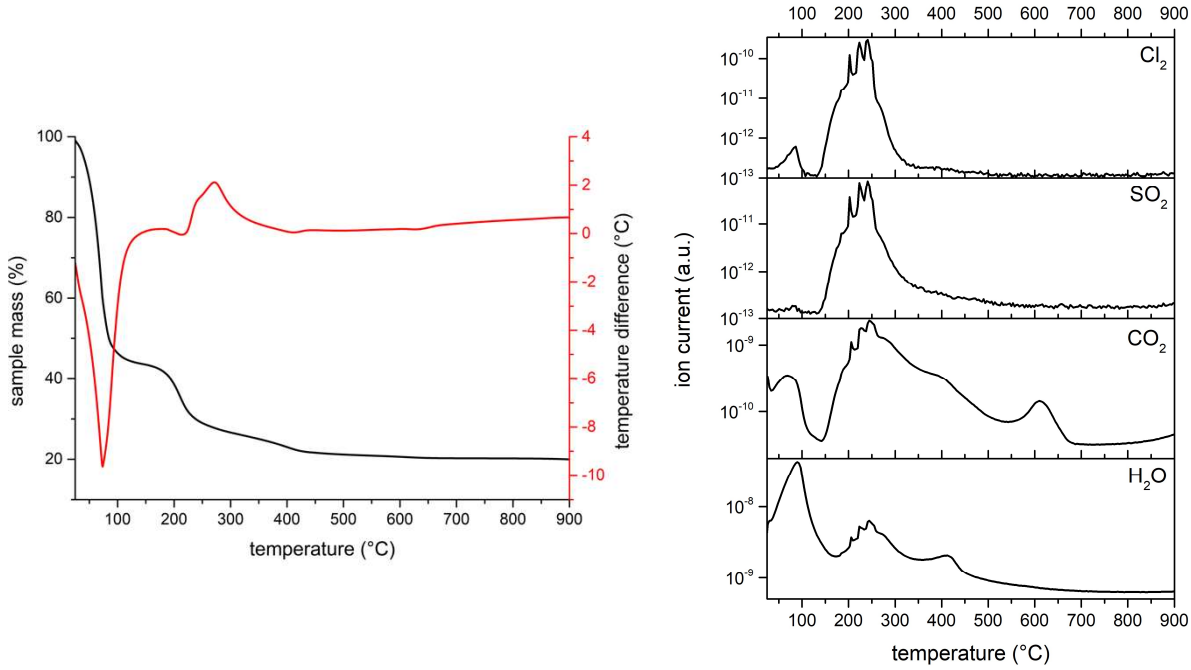

Figure S53. TG-DTA-MS analysis of sample E-MgAl-12SO4 after a batch experiment with 1,1,2-TCA.

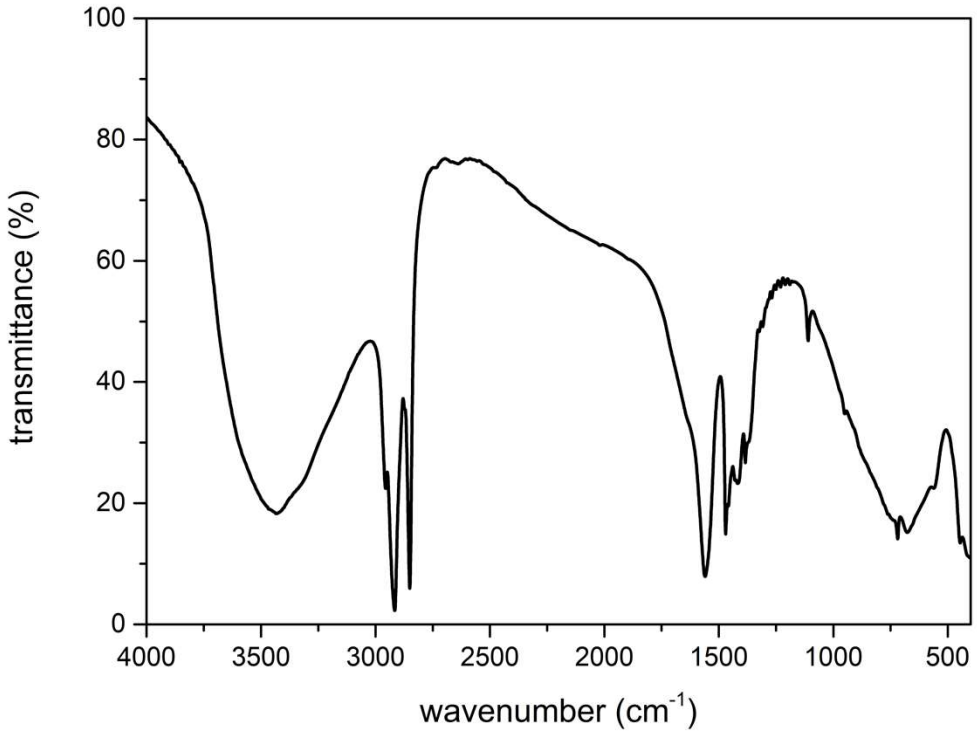

Figure S54. FTIR spectrum of sample E-MgAl-11COO after a batch experiment with 1,1,2-TCA.

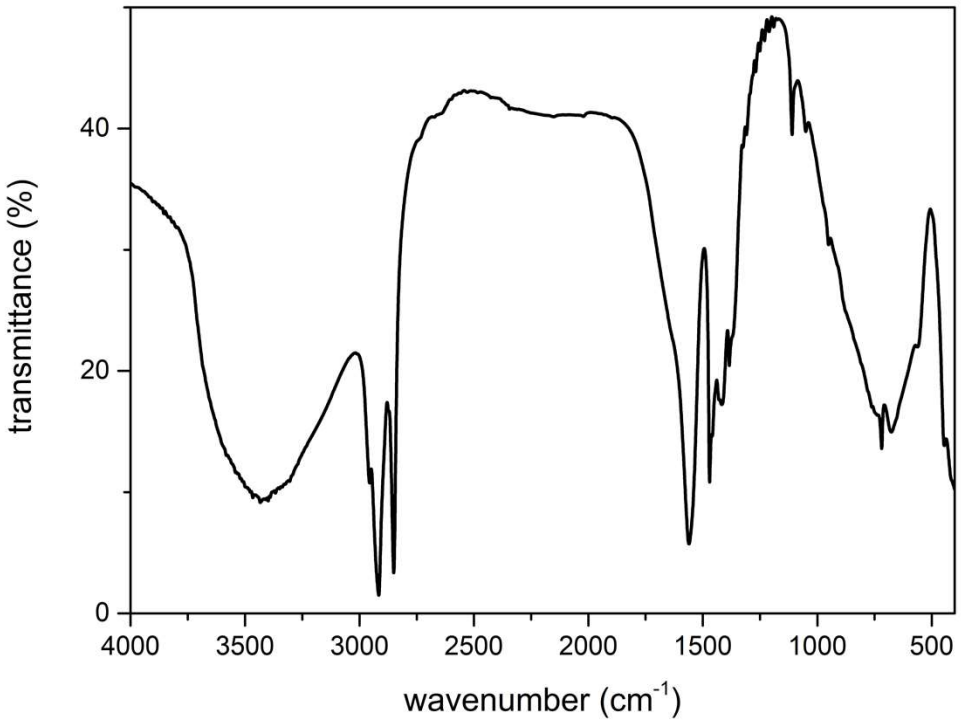

Figure S55. FTIR spectrum of sample E-MgAl-17COO after a batch experiment with 1,1,2-TCA.

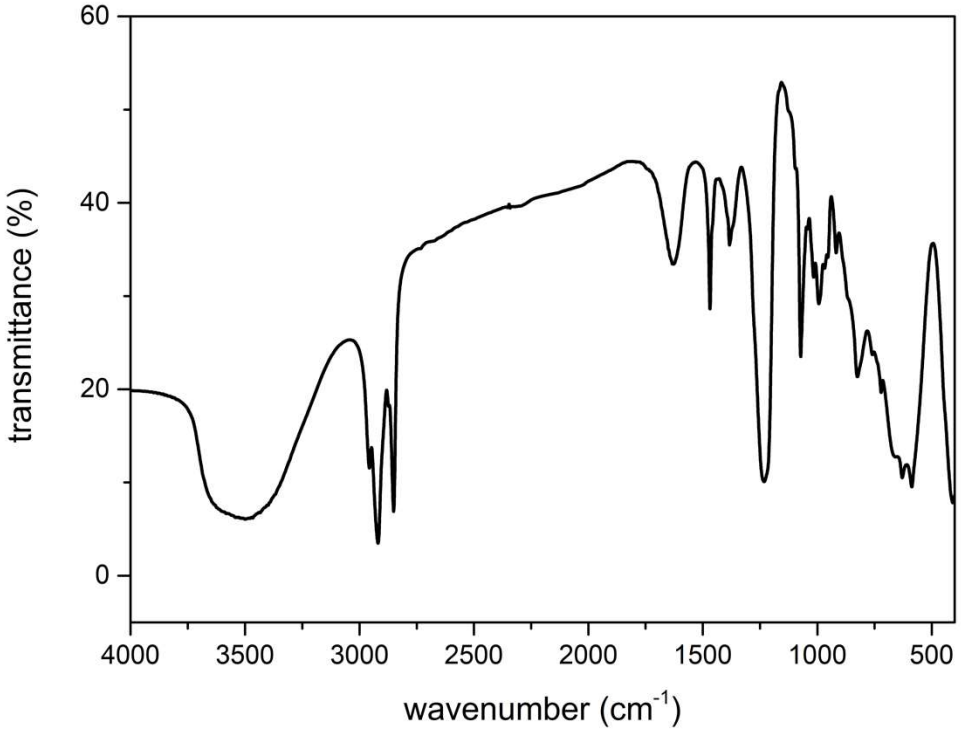

Figure S56. FTIR spectrum of sample E-MgAl-18SO3 after a batch experiment with 1,1,2-TCA.

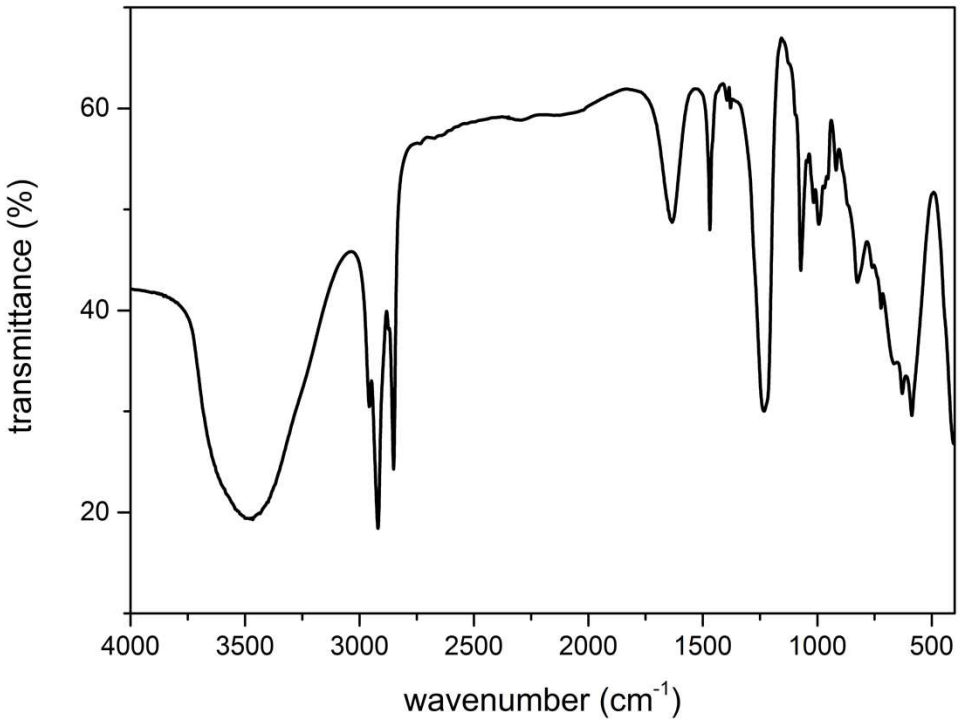

Figure S57. FTIR spectrum of sample E-MgAl-12SO4 after a batch experiment with 1,1,2-TCA.

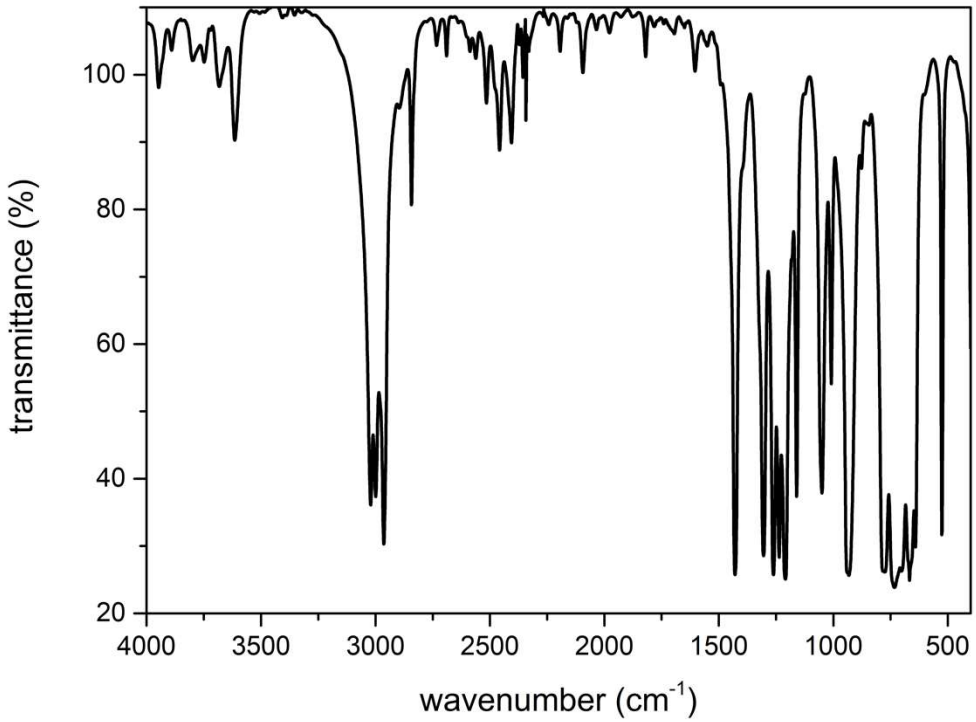

Figure S58. FTIR spectrum of 1,1,2-TCA.

Supplementary Table S1. List of the chemicals used within this study

chemicals	chemical formula	producer	purity	denotation in paper
sodium hydroxide, pellets	NaOH	Panreac	98 %	
Aluminium oxide	Al2O3	Fluka	99.99 %	
Aluminium Nitrate 9-hydrate pure	$Al(NO_3)_3 \bullet 9H_2O$	PanReac AppliChem ITW Reactants	min. 98 %	
Magnesium Nitrate 6-hydrate, for analysis	$Mg(NO_3)_2 \bullet 6H_2O$	PanReac AppliChem ITW Reactants	min. 98 %	
sodium hydrogen carbonate	$NaHCO_3$	Panreac	min. 99%	
sodium hexanoate	$C_6H_{11}NaO_2$	TCI Chemicals	min. 99.0 %	5COO
sodium caprylate	$C_8H_{15}NaO_2$	Glentham Life Sciences	99.3 %	7COO
Sodium Laurate	$C_{12}H_{23}NaO_2$	TCI Chemicals	min. 97 %	11COO
Sodium stearate	$C_{18}H_{35}NaO_2$	Alfa Aesar	-	17COO
1-Octanesulfonic acid sodium salt	$C_8H_{17}NaO_2S$	Sigma Aldrich	~ 98 %	8SO3
1-dodecane-sulfonic acid sodium salt	$C_{12}H_{25}NaO_3S$	Molekula	99.7 %	12SO3
Sodium 1-Octadecanesulfonate	$C_{18}H_{37}NaO_3S$	TCI Chemicals	min. 99 %	18SO3
sodium dodecyl sulfate pure, pharma grade	$C_{12}H_{25}NaO_4S$	PanReac AppliChem ITW Reactants	96.1 %	12SO4
Chloroform stabilized with ethanol (0,5%), for analysis	$CHCl_3$	PanReac AppliChem ITW Reactants	99.5 %	TCM
Trichloroethylene, reagent grade, stabilized with ethanol (0,49%)	C_2HCl_3	Scharlab	99.5 %	TCE
1,1,2-Trichloroethane	$C_2H_3Cl_3$	Acros Organics	98 %	1,1,2-TCA

Supplementary Table S2. List of other lab material used within this study.

product	producer	product ID	description
Amber storage bottles	Sigma Aldrich	23230-U	volume 120 mL, O.D. × H 49 mm × 114 mm, thread 22-400, PTFE/silicone septum, black phenolic hole cap, pre-assembled
Septa	Sigma Aldrich	27237-U	transparent PTFE/silicone, 20 mm diam. × 0.1 inch thickness × 10 mil PTFE, temperature limit 250 °C
Clear Boston Round Bottles	Thermo Fisher	S329-0250	250ml clear bottles, recommended for EPA Volatile Organic Analysis (VOA) Methods, cap with molded-in PTFE-faced silicon septa; certified
Membrane filtering Paper	Advantec® Toyo Roshi Kaisha Ltd.	25CS020AN	0.2 μm pore size, membrane filters, cellulose acetate
Hamilton syringe	Sigma Aldrich	20740-U	1000 Series, Gastight, 1001LTN, volume 1 mL, needle size 22 ga (bevel tip point style 2), needle L 51 mm (2 in.), mfr. no. 81317 (Hamilton)
Headspace vials	Agilent Technologies	5188-5392	Vial, screw top, headspace, clear, round bottom, 10 mL, 23 × 46 mm, vial size: 22.75 × 46 mm
screw cap	Agilent Technologies	5188-2759	Screw cap, headspace, steel, magnetic cap, PTFE/silicone septa (top white, bottom blue), 18 mm, cap size: 18 mm

Supplementary Table S3. d-spacing of the (003) reflexes of the synthesised organo-LDHs, the calculated contour chain length of each organic anion and the calculated tilt angle of the alkyl chain of each organic anion.

sample name	d-spacing (003) (Å)	contour chain length (Å)	tilt angle (°)
MgAl-5COO	8.3	8.5	2.5
MgAl-7COO	16.4	11.1	19.4
MgAl-11COO	36.0	16.1	54.9
MgAl-17COO	47.7	23.6	37.1
MgAl-8SO3	21.3	12.6	28.4
MgAl-12SO3	36.2	17.6	43.1
MgAl-18SO3	31.7	25.2	7.2
MgAl-12SO4	37.9	18.5	41.9

Supplementary Table S4. Band positions in the FTIR spectra of the reference sample MgAl-CO3 and MgAl-12SO4 with corresponding assignments

absorption bands	**MgAl-CO3**	**MgAl-12SO4**
ν(O-H)	3585-3430	3698-3394
ν_1,ν_3(H-O-H)	3585-3430	3698-3394
$\nu_{as}(CH_3)$		2958
$\nu_{as}(CH_2)$		2920
$\nu_s(CH_3,CH_2)$		2851
$\delta(H_2O)$	1638	1638
$\delta(CH_2)$		1469
$\delta_{as}(CH_3)$		1469
$\delta_s(CH_3)$		1384
$\nu_3(CO_3)$	1384, 1369	1384, 1361
$\nu_{as}(SO_3)$		1248, 1221, 1128
$\nu_s(SO_2)$		1080, 1050, 1018
ν_{as}(C-O-S)		996
$\nu_2(CO_3)$	865	883
ν(C-S)		763
ϱ(CH2)		722
δ(Me-OH)		722
$\nu_4(CO_3)$	663	664
(Me-OH)	559	634, 590
$\delta_{as}(SO_2)$		535
(O-Me-O)	419	421

Supplementary Table S5. Band positions in the FTIR spectra of the MgAl-xCOO samples with corresponding assignments.

absorption band	**MgAl-5COO**	**MgAl-7COO**	**MgAl-11COO**	**MgAl-17COO**
ν(O-H)	3503-3403	3585-3400	3590-3372	3584-3398
ν_1,ν_3(H-O-H)	3503-3403	3585-3400	3590-3372	3584-3398
ν_{as}(CH_3)	2960	2957	2957	2957
ν_{as}(CH_2)	2927	2927	2923	2918
ν_s(CH_2)		2873	2874, 1411	1410
ν_s(CH_3,CH_2)	2857, 2854	2852	2852	2850
ν(C=O)	1763	1768	1768	1764
δ(H_2O)	1627	1612	1640	1640
$\nu_{as/s}$(COO^-)	1575	1558, 1468	1558, 1468	1560, 1469
δ_{as}(CH_3)	1575	1558, 1170	1558, 1199	1560, 1189
δ_{as}(CH_2)	1575, 1228	1558, 1212, 1110	1558, 1228	1560, 1229, 1210, 1110
ν_3(CO_3)	1384, 1357	1384, 1361	1384	1384, 1359
δ_s(CH_3)		1384, 1361	1384	1384, 1359
δ_{as}(COO^-)		1258	1257	1267
ν_2(CO_3)	826	834	834	835
δ(Me-OH)			781, 721	720
ϱ(CH_2)		722	721	720
ν_4(CO_3)	667	665	674	676
(Me-OH)	640	608, 446		
(O-Me-O)	446	414	416	445, 411

Supplementary Table S6. Band positions in the FTIR spectra of the MgAl-xSO3 samples with corresponding assignments.

absorption band	MgAl-8SO3	MgAl-12SO3	MgAl-18SO3
ν(O-H)	3643	3647-3365	3661-3398
ν_1,ν_3(H-O-H)	3560-3348	3647-3365	3661-3398
ν_{as}(CH_3)	2958	2957	2958
ν_{as}(CH_2)	2924	2920	2918
ν_s(CH_3,CH_2)	2873, 2855	2873, 2851	2851
δ(H_2O)	1638	1633	1638
δ(CH_2)	1468	1468	1471
δ_{as}(CH_3)	1468	1468	1471
δ_s(CH_3)	1383, 1359	1384, 1359	1384, 1356
ν_3(CO_3)	1359	1384, 1359	1384, 1356
ν_{as}(SO_2)	1293, 1211, 1177	1292, 1277, 1199, 1185	1292-1177
ν_s(SO_2)	1050	1060, 1049	1050
ν_2(CO_3)	854		
ν(C-S)	792	798	797
δ(Me-OH)	792, 723	721	721
ϱ(CH_2)	723	721	721
ν_4(CO_3)		669	669
(Me-OH)	598	616	616
δ_{as}(SO_2)	527	535	535
(O-Me-O)	421	421	421