Dataset on infrared spectroscopy and X-ray diffraction patterns of Mg–Al layered double hydroxides by the electrocoagulation technique

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Article info
Article history:
Received 6 July 2019
Received in revised form 26 August 2019
Accepted 19 September 2019
Available online 25 September 2019

Keywords:
Layered double hydroxides
Al and AZ31 magnesium alloy electrodes
Electrochemical synthesis
Electrocoagulation

Abstract
The XRD profiles and FTIR analysis of sludge aggregates, Mg–Al layered double hydroxides, produced during electrocoagulation processes are presented. The data describes the composition of materials (LDH) produced at different operations conditions (atmospheric conditions and Mg²⁺/Al³⁺ ratio). The data show the diffraction peaks of (003), (006), (018) and (110) crystal planes for hydrotalcite structure.

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1. Data

The electrochemical method for the synthesis of Layered Double Hydroxides (LDHs) by electrocoagulation is used as an alternative procedure [1]. The LDHs are a class of anionic clays which have observed increasing attention due to their applications in many research areas [2]. Therefore, physicochemical properties of HDL materials, mainly explored from X-ray diffraction and FTIR analysis,
disclose their more specific applications. The dataset presents LDH characteristics prepared by electrocoagulation varying atmospheric conditions and Mg²⁺/Al³⁺ ratio. Figs. 1–6 show the diffraction peaks of (003), (006), (018) and (110) crystal planes for hydrotalcite structure. Tables 1–6 describe information on the phases and hkl-diffraction planes. Table 7 shows the band positions in the FTIR spectra. Figs. 7–12 displays the functional groups and bonding information. Table 8 exhibits the LDH-material specifications.

1.1. X-ray diffraction

X-ray diffraction (XRD) patterns of the materials were measured using a X’pert PRO—PANalytical diffractometer under the following conditions: 45 kV, 40 mA, monochromatic CuKα radiation (λ = 0.1542 nm) over a in the 2θ range from of 4° to -90°. The FTIR spectra was recorded with a JASCO FT/IR-4100 over a frequency in a range of 500–4000 cm⁻¹. The samples were prepared by mixing the powdered solids with KBr.

1.2. Infrared spectroscopy

The FTIR analysis was carried out in the spectral range (500–4000) cm⁻¹ by a Jasco FTIR-4100 spectrometer with a resolution of 4 cm⁻¹. The Figs. 7–12 represent the FTIR spectrum of composites and different vibrations attribution of the composites are represented in Table 7.
Fig. 1. XRD pattern of the AZ31-AZ31-1 material.

Fig. 2. XRD pattern of the AZ31-Al-N2-1 material.

Fig. 3. XRD pattern of the AZ31-Al-N2-3 material.

Fig. 4. XRD pattern of the HTX3-1 material.
Electrocoagulation experiments were conducted in a batch mode, using synthetic chloride solutions as supporting electrolyte. A 5.000 mg L⁻¹ of Sodium Chloride solution was prepared by the dissolution of Sodium Chloride (AR grade) in deionized water giving an overall final conductivity of 8.4 μS cm⁻¹. This solution was left to dissolve for 10 min. For nitrogen experiments, the beaker was covered and stirred with a speed of 100-rpm for 3.15 h. The sample was dried in a conventional oven for 2 h at 110 °C. The dried samples were then crushed into a fine powder using a ceramic mortar/bowl.

The electrocoagulation unit consisted on two plates that worked as anodes and cathodes, AZ31 magnesium alloy, Mg or aluminum, with an immersed area of 46.6 cm² each. The distance between electrodes was 5 mm, and the solution was mixing at 100 rpm using a hot magnetic plate mixer machine. Electrodes were connected to a DC power supply and the appropriate amount of the trivalent and divalent cations were carefully added to the beaker by a manual polarity inverter unit at an applied current of 0.36 and 0.15 mA. The Mg²⁺/Al³⁺ ratio and the operating time were calculated based on Faraday’s law, assuming that electro-dissolution only occurs at the anode. Before testing, electrodes were subjected to dry abrasion with emery paper No. 600 and then with abrasive paper No. 1000. Afterwards, the electrodes were rinsed with distilled water for approximately 5 min to remove traces (Table 8 describes the experimental conditions).

The following units were obtained beforehand and thoroughly cleaned:

- Digital scale
- Glass beaker (size: 1000 ml)
- Magnetic hotplate stirrer
### Table 1
X-ray diffraction planes related to the AZ31-AZ31(1)_MMH material.

| Magnesium Aluminium Hydroxide Carbonate Hydrate (0.5%) | Hydrotalcite (0.5%) | Carbon (97.6%) | Magnesite (1.2%) | Doyleite (0.2%) |
|------------------------------------------------------|---------------------|----------------|------------------|----------------|
| JCPDS: 98-004-0937                                    | JCPDS: 98-000-6183  | JCPDS: 98-003-1976 | JCPDS: 98-006-6643 | JCPDS: 98-004-9607 |
| Lattice parameters (Å):                              | Lattice parameters (Å): | Lattice parameters (Å): | Lattice parameters (Å): | Lattice parameters (Å): |
| a 3.0810                                             | a 3.054 a 14.26 a 4.314 | a 4.983 | b 3.0810                     | b 3.054 b 14.26 b 4.314 | b 5.000 |
| c 23.784                                             | c 22.81 c 14.26 c 12.775 | c 5.168 |
| 2 Theta degree | hkl | 2 Theta degree | Hkl | 2 Theta degree | Hkl | 2 Theta degree | Hkl | 2 Theta degree | Hkl |
| 11.154 0 0 3 | 11.630 0 0 3 | 10.737 1 1 1 | 37.00 1 0 4 | 18.560 0 0 1 | 0 1 |
| 22.409 0 0 6 | 23.382 0 0 6 | 17.578 0 2 2 | 47.192 1 1 3 | 20.731 1 0 0 | 1 0 |
| 34.419 0 1 2 | 34.098 1 0 1 | 20.643 1 1 3 | 61.105 1 1 6 | 21.263 1 0 0 | 1 0 |
| 36.892 1 0 4 | 34.792 0 1 2 | 21.570 2 2 2 | 63.276 0 1 8 | 21.723 0 1 0 | 2 3 |
| 38.657 0 1 5 | 35.390 0 0 9 | 35.583 0 4 4 | 22.926 0 1 1 | 23.779 0 1 0 | 0 0 |
| 45.651 0 1 8 | 37.455 1 0 4 | 37.273 1 3 5 | 35.526 1 1 1 | 36.002 0 1 2 | 1 2 1 |
| 45.738 0 0 12 | 39.343 0 1 5 | 37.825 0 0 6 | 37.114 1 2 1 | 37.637 0 0 2 | 0 2 |
| 61.243 1 1 3 | 46.811 0 1 8 | 39.956 0 2 6 | 38.766 2 1 -1 | 38.956 2 0 -3 | 2 0 -3 |
| 61.393 1 0 13 | 60.593 1 1 0 | 45.382 1 1 7 | 46.031 1 2 2 | 46.242 1 1 2 | 1 2 |
| 61.933 1 1 3 | 60.868 0 0 15 | 45.850 0 4 6 | 60.163 2 2 2 | 60.163 2 2 2 | 2 2 2 |
| 63.596 1 0 13 | 60.893 4 6 6 | 62.033 1 3 9 | 61.920 2 0 3 | 63.865 1 1 3 | 1 1 3 |
Table 2
X-ray diffraction planes related to the Al-AZ31_N2 material.

| Component                  | JCPDS: 98-000-4494 Lattice parameters (Å): | JCPDS: 98-004-0936 Lattice parameters (Å): | JCPDS: 98-000-7431 Lattice parameters (Å): | JCPDS: 98-007-4545 Lattice parameters (Å): | JCPDS: 98-005-6296 Lattice parameters (Å): | JCPDS: 98-006-6646 Lattice parameters (Å): |
|----------------------------|--------------------------------------------|--------------------------------------------|--------------------------------------------|--------------------------------------------|--------------------------------------------|--------------------------------------------|
| Carbon dioxide (0.2%)      | a 5.624 a 3.046 a 5.67                    | A 14.025                                  | A 6.756                                    | a 4.278                                    | b 6.756                                    | b 4.278                                    |
| Hydrotalcite (0.3%)        | b 5.624 b 3.046 b 5.67                    | B 14.083                                  | B 6.756                                    | b 4.278                                    | c 6.756                                    | c 12.546                                   |
| Nitrogen oxide (0.2%)      | c 5.624 c 22.77 c 5.67                    | C 14.486                                  | C 6.756                                    | c 12.546                                   |                                             |                                            |
| Magnesium zinc (98.3%)     |                                           |                                           |                                           |                                           |                                           |                                            |
| Magnesium carbonate (0.3%) |                                           |                                           |                                           |                                           |                                           |                                            |
| Sodium carbide (0.3%)      |                                           |                                           |                                           |                                           |                                           |                                            |
| Magnesite (0.7%)           |                                           |                                           |                                           |                                           |                                           |                                            |
Table 3
X-ray diffraction planes related to the AZ31-Al-N23 material.

| Hydrotalcite (20.4%) | Carbon dioxide (15.0%) | Brucite (1.1%) | Sodium Carbonate (15.4%) | Magnesite (48.1%) |
|---------------------|------------------------|----------------|--------------------------|------------------|
| JCPDS: 98-000-6183  | JCPDS: 98-001-3442     | JCPDS: 98-004-4736 | JCPDS: 98-003-6631        | JCPDS: 98-006-6646 |
| Lattice parameters (Å): | Lattice parameters (Å): | Lattice parameters (Å): | Lattice parameters (Å): | Lattice parameters (Å): |
| a | b | c | a | b | c | a | b | c | a | b | c |
| 3.054 | 3.054 | 22.810 | 5.63 | 5.63 | 5.63 | 3.148 | 3.148 | 4.779 | 5.208 | 5.208 | 6.454 | 4.278 | 4.278 | 12.546 |

2 Theta degree hkl 2 Theta degree Hkl 2 Theta degree hkl 2 Theta degree Hkl 2 Theta degree hkl 2 Theta degree Hkl

11.630 0 0 3 27.414 1 1 18.549 0 0 1 27.619 0 0 2 27.947 0 1 2
23.382 0 0 6 35.628 0 2 1 37.614 0 0 2 34.137 0 0 2 37.540 0 1 4
34.098 1 0 1 39.160 1 1 2 37.967 0 1 1 34.413 1 1 0 47.716 1 1 3
35.3900 0 0 9 61.588 1 2 3 62.027 1 1 1 39.945 0 2 0 62.015 1 1 6
46.811 0 1 8 46.746 0 1 3 64.469 0 1 8
60.593 1 1 0 49.252 0 2 2
60.868 0 0 15 60.936 0 1 4
61.933 1 1 3 61.468 1 2 2

Table 4
X-ray diffraction planes related to the HTX3_1 material.

| Hydrotalcite (12.7%) | Halite (12.5%) | Brucite (0.7%) | Gibbsite (74.1%) |
|---------------------|----------------|----------------|------------------|
| JCPDS: 98-000-6183  | JCPDS: 98-001-6223 | JCPDS: 98-003-4961 | JCPDS: 98-008-2783 |
| Lattice parameters (Å): | Lattice parameters (Å): | Lattice parameters (Å): | Lattice parameters (Å): |
| a | b | c | a | b | c | a | b | c | a | b | c |
| 3.054 | 3.054 | 22.810 | 5.653 | 5.653 | 5.653 | 3.148 | 3.148 | 4.779 | 3.052 | 3.052 | 4.779 |

2 Theta degree Hkl 2 Theta degree hkl 2 Theta degree Hkl 2 Theta degree hkl 2 Theta degree Hkl 2 Theta degree Hkl

11.630 0 0 3 27.303 1 1 1 18.577 0 0 1 18.675 0 2 0
23.382 0 0 6 31.632 0 0 2 37.671 0 0 2 22.393 1 1 1
34.792 0 1 2 45.341 0 2 2 37.979 0 1 1 27.736 1 2 1
37.455 0 1 4 62.040 1 1 1 27.736 1 2 1
39.343 0 1 5 28.669 1 1 2
46.811 0 1 8 34.984 1 2 1
47.810 0 0 12 35.509 2 0 0
60.593 1 1 0 36.989 1 1 3
60.868 0 0 15 37.871 0 4 0
61.933 1 1 3 38.269 1 1 6

- Spatula
- Al, Mg and AZ31 alloy electrode plates
- Sodium Chloride, AR grade
- Nitrogen (N₂) gas pipeline
- DI water
- Ceramic mortar/bowl
- Emery paper No. 600 and abrasive paper No. 1000
Table 5
X-ray diffraction planes related to the MgHP-1 material.

| Zinc Aluminium Hydroxide Chloride Hydrate (7.6%) | Magnesite (12.3%) | Diamond (2.3%) | Sodium carbide (40.0%) | Hydrotalcite (5.2%) | Gibbsite (32.4%) |
|-----------------------------------------------|------------------|---------------|----------------------|-------------------|-----------------|
| JCPDS: 98-005-8141                            | JCPDS: 98-006-6646 | JCPDS: 98-005-4252 | JCPDS: 98-005-6291 | JCPDS: 98-000-6183 | JCPDS: 98-011-2963 |
| Lattice parameters (Å):                        | Lattice parameters (Å): | Lattice parameters (Å): | Lattice parameters (Å): | Lattice parameters (Å): | Lattice parameters (Å): |
| a 3.083                                        | a 4.278           | a 4.591        | a 6.778              | a 3.054           | a 8.675          |
| b 3.083                                        | b 4.278           | b 4.591        | b 6.778              | b 3.054           | b 5.069          |
| c 23.47                                        | c 12.546          | c 4.591        | c 12.74              | c 22.81           | c 12.508         |
| 2 Theta degree                                | hkl               | 2 Theta degree | Hkl                  | 2 Theta degree    | Hkl              |
| 11.3 0 0 3                                    | 27.947            | 0 1 2          | 39.212               | 0 02              | 23.206           |
| 22.711 0 0 6                                  | 37.540            | 1 0 4          | 48.536               | 112              | 27.991           |
| 34.363 0 0 9                                  | 47.716            | 1 1 3          | 36.387               | 1 2 3            | 34.792           |
| 38.772 0 1 5                                  | 62.015            | 1 1 6          | 37.501               | 2 2 0            | 35.390           |
| 45.920 0 1 8                                  | 64.469            | 0 1 8          | 38.766               | 0 2 4            | 37.455           |
| 58.983 0 0 15                                 | 46.758            | 1 1 6          | 39.343               | 0 1 5            | 37.486           |
| 62.002 1 0 13                                 | 47.439            | 2 2 4          | 46.811               | 0 1 8            | 28.714           |
| 48.939 2 3 1                                  | 47.810            | 0 0 12         | 31.649               | 3 0 2            | 35.159           |
| 50.697 0 2 6                                  | 60.593            | 1 1 0          | 35.159               | 1 1 4            | 35.385           |
| 61.093 2 4 0                                  | 60.868            | 0 0 15         | 35.385               | 0 2 0            | 35.809           |
| 61.224 2 3 5                                  | 61.933            | 1 1 3          | 35.809               | 3 1 3            | 38.327           |
| 61.412 1 3 6                                  | 63.586            | 1 0 13         | 38.327               | 1 2 2            | 40.117           |
| 61.983 0 4 4                                 | 40.117            | 0 2 2          | 40.249               | 2 1 5            | 45.440           |
| 64.610 0 2 8                                 | 47.175            | 1 0 4          | 47.287               | 4 1 5            | 50.512           |
| 47.175 1 0 4                                 | 47.287            | 4 1 5          | 50.512               | 3 1 1            | 58.612           |
| 50.512 3 1 1                                 | 50.512            | 3 1 1          | 58.612               | 2 3 2            | 60.468           |
| 58.612 2 3 2                                 | 60.468            | 4 2 6          | 64.616               | 6 0 6            | 72.237           |
| 64.616 6 0 6                                 | 72.237            | 1 1 8          |                      |                   |                  |
Table 6

X-ray diffraction planes related to the MgHP-Al_2 material.

| JCPDS: 98-007-4545 | Lattice parameters (Å): | 2 Theta degree | Hkl | 2 Theta degree | hkl | 2 Theta degree | Hkl | 2 Theta degree | Hkl |
|---------------------|--------------------------|----------------|-----|----------------|-----|----------------|-----|----------------|-----|
| Magnesium Zinc (98.5%) | Magnesium Aluminium Hydroxide Carbonate Hydrate (0.3%) | a 14.025 | 12.210 | 0 0 2 | 11.684 | 0 0 3 | 11.630 | 0 0 3 | 23.415 | 2 0 -1 |
| Sodium Carbonate (0.9%) | JCPDS: 98-003-6621 | b 14.083 | 12.562 | 0 2 0 | 23.492 | 0 0 6 | 23.382 | 0 0 6 | 23.762 | 1 1 -1 |
| JCPDS: 98-004-0937 | Lattice parameters (Å): | c 14.48 | 17.835 | 2 2 0 | 34.205 | 1 0 1 | 34.098 | 1 0 1 | 27.897 | 0 0 2 |
| JCPDS: 98-00-61-83 | Lattice parameters (Å): | JCPDS: 98-00-61-83 | JCPDS: 98-00-61-83 | JCPDS: 98-00-61-83 | JCPDS: 98-00-61-83 |
| a 3.045 | 23.201 | 1 2 3 | 39.486 | 0 1 5 | 35.390 | 0 0 9 | 35.464 | 2 0 2 |
| a 3.054 | 23.223 | 2 1 3 | 39.486 | 0 1 5 | 35.390 | 0 0 9 | 35.464 | 2 0 2 |
| a 9.015 | 23.492 | 3 1 2 | 48.058 | 0 0 12 | 37.455 | 1 0 4 | 36.557 | 3 1 -1 |
| b 5.209 | 27.675 | 0 2 4 | 60.786 | 1 1 0 | 39.343 | 0 1 5 | 38.070 | 3 1 1 |
| b 5.209 | 28.413 | 4 2 0 | 61.193 | 0 0 15 | 47.810 | 0 0 12 | 47.893 | 4 0 -2 |
| b 5.209 | 34.741 | 1 5 2 | 62.140 | 1 1 3 | 60.593 | 1 1 0 | 50.244 | 2 2 2 |
| b 5.209 | 40.633 | 6 2 0 | 72.053 | 2 0 2 | 60.868 | 0 0 15 | 55.692 | 0 2 -3 |
| b 5.209 | 45.335 | 4 5 3 | 61.933 | 1 1 3 | 58.6 | 2 2 -3 |
| b 5.209 | 46.523 | 4 6 0 | 72.160 | 1 1 9 | 60.730 | 2 2 3 |
| b 5.209 | 47.310 | 1 7 2 | 71.203 | 1 3 3 |
| b 5.209 | 48.359 | 6 4 2 |
| b 5.209 | 50.238 | 5 1 6 |

Table 7

Positions of the bands (in cm⁻¹) in the IR spectra (Figs. 7–12) [4,5].

| Vibration/Assignmet | Material | AZ31-AZ31-1 | AZ31-Al-N2 | AZ31AIN2-3 | HTX3-1 | MgHP-1 | MgHP-2 |
|---------------------|----------|-------------|------------|------------|--------|--------|--------|
| Water and hydroxyl groups | OH stretching | 3459.67 | 3443.28 | 3443.28 | 3450.99 | 3216.68 | 3465.46 |
| | Bending | Ad sorbed water | 1641.13 | 1639.2 | 1639.2 | 1641.13 | 1646.91 | 1642.09 |
| Nitrogen | N–H stretching | 2095.28 | 2095.28 | 2095.28 | 2098.17 | 2100.1 | 2101.06 |
| Carbonates | C = O | 1475.28 | 1501.31 | 1501.31 | 1508.06 | |
| | v3 asymmetric stretching | 1364.39 | 1363.43 | 1363.2 | 1364.39 | 1360.53 | 1365.35 |
| | v1 symmetrical stretching | 1267 | 675.93 | |
| Others | Al–O and Mg–O deformation | 1032.69 | 1069.33 | 1069.33 | 1073.19 | 1087.66 | 1075.12 |
| | Mg–O | 1188.9 | 639.2 | |
| | Mg–O | 557.33 | 598.80 | 589.15 | 544.79 |
| | Mg–O | 447.40 | 452.22 | 452.22 | 412.692 | |
| | Mg–O | 378.94 | 367.37 | |

Fig. 7. IR Spectrum of the AZ31-AZ31-1 material.
Fig. 8. IR Spectrum of the AZ31-AL-N2-1 material.

Fig. 9. IR Spectrum of the AZ31-AL-N2-3 material.

Fig. 10. IR Spectrum of the HTX3-1 material.

Fig. 11. IR Spectrum of the MgHP-1 material.
Acknowledgements

The authors would like to acknowledge the Universidad del Valle (Colombia) for supporting the study under Grant No. 2863: Electrocoagulation of textile industrial effluents using magnesium ‘Case study of indigo carmine solutions’. The Royal Academy of Engineering –Newton Caldas Fund (United Kingdom) and Loughborough University (United Kingdom) for the financial support for the development of this research, framed in the project: Treatment of petroleum production wastewater by combined adsorption and oxidation process using double layer hydrotalcites under Grant No. IAPP1617-70.

Conflict of Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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