Data Article

Data on ion composition and X-ray diffraction patterns of biosolids from wastewater treatment plants in Lufkin and Nacogdoches, Texas, USA

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ARTICLE INFO

Article history:
Received 27 June 2018
Accepted 23 August 2018
Available online 30 August 2018

Abstract

The data presented in this article is related to the research article entitled, “Structural Characterization and Evaluation of Municipal Wastewater Sludge (Biosolids) from two Rural Wastewater Treatment Plants in East Texas, USA” (Onchoke et al., [1]). The XRD profiles and composition of biosolids from two wastewater treatment plant is presented. This study describes the composition of XRD crystalline phase patterns of the wastewater sludge. After the removal of the Kα2 peaks the d-spacing and hkl values were determined. In addition, the ion chromatographic profile of the seven anions (NO3−, NO2−, Br−, Cl−, F−, SO42−, and PO43−) in biosolids is presented.

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Specifications Table

| Subject area                  | Environmental Chemistry |
|------------------------------|-------------------------|
| More specific subject area   | Wastewater sludge (biosolids) |
| Type of data                 | Table, graph, figure    |

DOI of original article: https://doi.org/10.1016/j.saa.2018.06.096
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https://doi.org/10.1016/j.dib.2018.08.087
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How data was acquired

- **Ion chromatography, XRD, SEM, EDX were used in the study.**
  - (a) Dionex Integrion HPIC ion chromatograph (Thermo Fisher Scientific Inc., USA) was used for anion analysis.
  - (b) A Bruker AXS D8 Advance diffractometer equipped with an X-ray tube (Cu Kα radiation: λ = 1.54060 Å, 40 kV, and 40 mA) using a Ni filter and one-dimensional LynxEye detector at scanning speed of 2°/min and 0.0125° step sizes and a 1 s/step.
  - (c) A JEOL-JSM 6100 scanning electron microscope equipped with a Horiba energy dispersive X-ray spectroscopy (SEM/EDX) was used.

Data format

- **Raw, filtered, analyzed**

Experimental factors

- (a) For XRD analysis: Biosolid samples were obtained from Nacogdoches and Lufkin wastewater treatment plant (NWWTP, LWWTP), air dried, and ground to powder.
- (b) For IC analysis: samples were filtered on a 0.45 μm filter.

Experimental features

- Wastewater sludge generated from the rural municipal wastewater treatment plants are applied for land. We provide the characterization of the crystalline phases in the biosolids. The powder diffraction file was acquired using Bruker AXS DIFFRAC.EVA program [2]. The fitted line profiles, peak search methods, and indexing of the lines were used to calculate the mineral identification via comparisons with the diffraction patterns with TOPAS program [3].

Data source location

- Nacogdoches, East Texas, in East Texas, USA latitude: 31° 33′ 31.2444″ N and longitude 94° 38′ 52.1808″ W,

Data accessibility

- All data are available within this article.

Related research article

- Associated Paper: “Structural Characterization and Evaluation of Municipal Wastewater Sludge (Biosolids) from two Rural Wastewater Treatment Plants in East Texas, USA”, Onchoke, K.K, Franclemont, C.M., Spectrochim Acta A, In press [1].

### Value of the data

- The data provided here is important for wastewater and wastewater treatment plants, water resources. The data provides important information for identification of elemental compositions in biosolids.
- The indexed hkl and d-spacing values can be used for referencing and identification of crystalline phases prevalent in biosolids/wastewater sludge.
- The XRD patterns are important for the identification of any newer crystalline phases in wastewater treatment plants, and in particular in East Texas. This data can also be used for comparisons to other wastewater treatment plants. The data serves as a benchmark for other researchers analyzing biosolids generated from wastewater treatment plants.

### 1. Data

Wastewater treatment plants generate large amounts of wastewater sludge (also known as biosolids) [4]. Wastewater biosolids can be disposed of in several ways, namely, for enrichment of soils, or for landfills [5–8]. The data in this paper presents information on the crystalline phases, their approximate compositions, their d-spacings and hkl patterns (Fig. 3A and B, and Tables 1 and 2). An ion chromatographic profile with parameters used for analysis of seven anions (Cl−, F−, NO3−, NO2−, Br−, SO42−, and PO43−) during the analyses is provided (Fig. 2).
2. Experimental design, materials, and methods

The Experimental methods and procedures that allowed the data here presented are described in Ref. [1] and in cited references. Here, only the protocol for XRD and SEM morphological analysis is provided, giving a large number of experimental details, usually omitted in research articles due to the words limit.

| Index | Angle (2θ) | d-Value | Net Intensity | Gross Intensity | Rel. Intensity | h, k, l | Mineral        |
|-------|------------|---------|---------------|----------------|---------------|--------|---------------|
| 0     | 6.217      | 14.20528 | 2756          | 10,258         | 31.40%        | 0 0 2  | Vermiculite   |
| 1     | 20.756     | 4.27606  | 1703          | 6741           | 19.40%        | 1 2 1  | Alunogen      |
| 2     | 22.663     | 3.92039  | 258           | 4887           | 2.90%         | 0 3 1  | Laumontite    |
| 3     | 25.201     | 3.53096  | 255           | 4588           | 2.90%         | 2 2 1  | Laumontite    |
| 4     | 26.422     | 3.37054  | 3254          | 7472           | 37.10%        | 3 1 2  | Laumontite    |
| 5     | 26.53      | 3.35703  | 8779          | 12,982         | 100.00%       | 0 1 1  | Quartz        |
| 6     | 27.266     | 3.26809  | 341           | 4425           | 3.90%         | 1 0 1  | Quartz        |
| 7     | 27.359     | 3.25717  | 296           | 4365           | 3.40%         | 1 1 6  | Vermiculite   |
| 8     | 27.886     | 3.19679  | 225           | 4182           | 2.60%         | 1 1 2  | Annite Mica   |
| 9     | 31.569     | 2.83182  | 196           | 3637           | 2.20%         | 0 2 8  | Vermiculite   |
| 10    | 32.373     | 2.7633   | 212           | 3602           | 2.40%         | 2 3 1  | Gypsum        |
| 11    | 35.837     | 2.50374  | 274           | 3498           | 3.10%         | 1 3 3  | Vermiculite   |
|       |            |          |               |                |               | 1 3 1  | Talc          |
| 12    | 35.963     | 2.49521  | 171           | 3401           | 1.90%         | 1 3 11 | Antigorite T  |
| 13    | 36.148     | 2.48288  | 157           | 3389           | 1.80%         | 1 3 2  | Annite Mica   |
| 14    | 36.39      | 2.46693  | 501           | 3726           | 5.70%         | 1 1 0  | Quartz        |
| 15    | 38.122     | 2.3587   | 157           | 3189           | 1.80%         | 0 0 3  | Kaolinite     |
| 16    | 38.325     | 2.34672  | 160           | 3168           | 1.80%         | 1 3 1  | Kaolinite     |
| 17    | 39.336     | 2.28866  | 502           | 3449           | 5.70%         | 1 0 2  | Quartz        |
| 18    | 40.173     | 2.24291  | 243           | 3181           | 2.80%         | 1 1 1  | Quartz        |
| 19    | 42.263     | 2.13671  | 265           | 3079           | 3.00%         | 0 2 0  | Quartz        |
| 20    | 45.719     | 1.98288  | 275           | 2919           | 3.10%         | 2 0 1  | Quartz        |
|       |            |          |               |                |               | 0 2 1  |               |
| 21    | 48.994     | 1.85774  | 131           | 2513           | 1.50%         | 0 6 2  | Laumontite    |
| 22    | 49.996     | 1.82283  | 1566          | 3964           | 17.80%        | 1 1 2  | Quartz        |
| 23    | 55.225     | 1.66197  | 145           | 2426           | 1.70%         | 0 1 3  | Quartz        |
| 24    | 59.809     | 1.54506  | 598           | 2789           | 6.80%         | 2 1 1  | Quartz        |
| 25    | 67.57      | 1.38524  | 305           | 2359           | 3.50%         | 1 2 2  | Quartz        |
| 26    | 68.019     | 1.37718  | 781           | 2833           | 8.90%         | 2 1 2  | Quartz        |
| 27    | 68.16      | 1.37468  | 553           | 2596           | 6.30%         | 2 0 3  | Quartz        |
| 28    | 73.358     | 1.28957  | 239           | 2203           | 2.70%         | 0 1 4  | Quartz        |
| 29    | 75.597     | 1.25684  | 393           | 2319           | 4.50%         | 0 3 2  | Quartz        |
| 30    | 79.662     | 1.20261  | 271           | 1528           | 3.10%         | 2 1 3  | Quartz        |
Table 2
Analysis of the crystalline phases, d-spacing, and h, k, l values of biosolids in LWWTP [1].

| Index | Angle (2θ) | d Value  | Net Intensity | Gross Intensity | Rel. Intensity | h k l     | Mineral                |
|-------|------------|----------|---------------|-----------------|---------------|-----------|------------------------|
| 0     | 6.196      | 14.2529  | 2843          | 10,629          | 36.2%         | 0 0 2     | Vermiculite            |
| 1     | 11.645     | 7.59308  | 554           | 4410            | 7.0%          | 0 2 0     | Gypsum                 |
| 2     | 19.881     | 4.46235  | 524           | 4195            | 6.70%         | 1 0 1     | Andalusite             |
| 3     | 20.825     | 4.26215  | 1469          | 5156            | 18.70%        | 0 1 0     | Quartz Gypsum          |
| 4     | 25.287     | 3.51915  | 198           | 3358            | 2.50%         | 2 2 1     | Laumontite             |
| 5     | 26.622     | 3.34568  | 7847          | 10,898          | 100.00%       | 0 1 1     | Quartz                 |
| 6     | 27.008     | 3.29879  | 313           | 3309            | 4.00%         | 2 4 1     | Palygorskite           |
| 7     | 27.737     | 3.21372  | 792           | 3667            | 10.10%        | 2 2 1     | Palygorskite           |
| 8     | 29.104     | 3.06578  | 478           | 3225            | 6.10%         | 1 4 1     | Gypsum                 |
| 9     | 31.083     | 2.87493  | 297           | 2862            | 3.80%         | 2 2 2     | Vermiculite, Hexahydrite |
| 10    | 31.761     | 2.81512  | 131           | 2644            | 1.70%         | 0 1 1     | Laumontite             |
| 11    | 33.327     | 2.68633  | 225           | 2515            | 2.90%         | 1 3 1     | Annite Mica            |
| 12    | 33.411     | 2.67978  | 139           | 2421            | 1.80%         | 0 6 1     | Vermiculite            |
| 13    | 35.938     | 2.49691  | 150           | 2436            | 1.90%         | 2 0 2     | Acolite                |
| 14    | 36.5       | 2.45972  | 537           | 2768            | 6.80%         | 1 3 4     | Vermiculite            |
| 15    | 37.734     | 2.3821   | 150           | 2246            | 1.90%         | 1 1 0     | Corundum               |
| 16    | 39.426     | 2.28366  | 479           | 2517            | 6.10%         | 0 1 2     | Quartz Vermiculite     |
| 17    | 40.251     | 2.23874  | 216           | 2246            | 2.80%         | 1 1 1     | Quartz + Palygorskite  |
| 18    | 42.44      | 2.12817  | 254           | 2218            | 3.20%         | 3 5 1     | Quartz + Palygorskite  |
| 19    | 43.236     | 2.09083  | 121           | 2029            | 1.50%         | 0 2 0     | Quartz                 |
| 20    | 43.647     | 2.07209  | 141           | 2014            | 1.80%         | 1 1 3     | Corundum               |
| 21    | 45.571     | 1.989    | 126           | 1929            | 1.60%         | 2 0 1     | Hexahydrite            |
| 22    | 45.769     | 1.98085  | 274           | 2070            | 3.50%         | 0 2 1     | Quartz                 |
| 23    | 46.944     | 1.93396  | 125           | 1844            | 1.60%         | 3 1 1     | Turquoise              |
| 24    | 47.998     | 1.89391  | 137           | 1818            | 1.70%         | 0 0 2     | Boehemite              |
| 25    | 48.374     | 1.88009  | 106           | 1778            | 1.30%         | 4 0 4     | Laumontite             |
| 26    | 50.115     | 1.81878  | 758           | 2421            | 9.70%         | 1 1 2     | Quartz                 |
| 27    | 54.851     | 1.6724   | 318           | 1872            | 4.10%         | 2 0 2     | Quartz                 |
| 28    | 59.932     | 1.54218  | 513           | 1930            | 6.50%         | 1 2 1     | Quartz                 |
| 29    | 67.736     | 1.38224  | 357           | 1704            | 4.50%         | 1 2 2     | Quartz                 |
| 30    | 68.112     | 1.37553  | 603           | 1954            | 7.70%         | 4 4 0     | Quartz Vermiculite     |
| 31    | 68.106     | 1.37564  | 625           | 1976            | 8.00%         | 0 3 1     | Quartz Vermiculite     |
| 32    | 73.466     | 1.28794  | 234           | 1546            | 3.00%         | 0 1 4     | Quartz                 |
| 33    | 75.625     | 1.25645  | 133           | 1320            | 1.70%         | 0 3 2     | Quartz                 |
| 34    | 75.702     | 1.25536  | 88.4          | 1266            | 1.10%         | 0 3 2     | Quartz                 |
| 35    | 85.016     | 1.14001  | 20.7          | 66.3            | 0.30%         | 0 2 4     | Quartz                 |
2.1. Study area description

The Nacogdoches and Lufkin Wastewater Treatment Plants (NWWTP, LWWTP, shown in Fig. 1) are located in Nacogdoches City (Population: 33,000) and Lufkin City (Population: ~35,000). These

Fig. 1. Aerial photographs of (a) Nacogdoches wastewater treatment plant (NWWTP), and (b) Lufkin wastewater treatment plant (LWWTP). In each of the pictures, 4 clarifiers are observed.

Fig. 2. Representative elution profile of the seven anions and retention times. $1 = F^-$, $2 = Cl^-$, $3 = NO_2^-$, $4 = Br^-$, $5 = NO_3^-$, $6 = PO_4^{3-}$, $7 = SO_4^{2-}$. The standard was diluted X20 times. The Dionex Integrion HPIC ion chromatograph (Thermo Fisher Scientific Inc., USA) was used. A Dionex IonPac AS22 analytical column (2 × 250 mm) thermostated at 30 °C, guard column (IonPac AG22), a Dionex AS 22 Eluent Concentrate (4.5 mM sodium carbonate/1.4 mM sodium bicarbonate) was used.
Fig. 3. Powder XRD patterns of samples from the Nacogdoches Wastewater Treatment Plant (A), and Lufkin Wastewater Treatment Plant (B). The 2θ values and d-spacing values corresponding to each crystalline phases are also shown. The crystalline phases corresponding to each peak(s) are presented in Tables 1 and 2, respectively.
wastewater treatment plants are activated wastewater treatment plants. The NWWTP and LWWTP have wastewater treatment capacity of 12.88 million gallons per day (MGD) 11.3 MGD, respectively.

2.2. Sampling and collection of biosolids

Biosolids were collected from the Nacogdoches Wastewater Treatment Plant (NWWTP) and Lufkin Wastewater treatment Plant (LWWTP) during the Summer 2016 and 2017. Multiple grab samples from the Nacogdoches and Lufkin WWTPs were dried in the lab. Biosolid samples were stored in plastic containers or 5-gallon plastic buckets. Proper care was taken to avoid any contamination during each sampling period.

2.3. Ionic analysis elution profile

Approximately 28 mg of finely crushed biosolids was first added to a 25 mL volumetric flask and the volume filled to the mark using 18.2 MΩ water. The contents of the flasks were then sonicated for 20 min and the sample split into two separate 15 mL Falcon tubes. Following this, the samples were centrifuged at 7650 rpm for 20 minutes and filtered through 0.45 μm filters. Samples were then analyzed with anion chromatography.

2.4. Morphological characterization of biosolids

The biosolids were air dried, crushed with mortar and pestle, and analyzed with JEOL-JSM 6100 scanning electron microscope equipped with a Horiba energy dispersive X-ray spectroscopy (SEM/EDX) with an accelerating voltage of 15 kV. The surface morphology, particle diameters (Figs. 4-6) of biosolids were measured at X40, 100 - 200 × magnifications. Powder XRD analysis was performed in the 2θ range of 2° –90° on a Bruker AXS D8 Advance diffractometer equipped with an X-ray tube (Cu Kα radiation: λ = 1.54060 Å, 40 kV, and 40 mA) using a Ni filter and one-dimensional LynxEye detector at scanning speed of 2°/min and 0.0125° step sizes and a 1 s/step. The diameters of select pores (Fig. 6) were measured at 1–5 k magnification. Powder XRD patterns (Figs. 3–5) and their hkl values was used to identify the crystalline structural phases present in biosolids (Tables 1 and 2).

![Fig. 4. Representative SEM micrograph of LWWTP biosolid showing particle size diameters (magnification 40 ×, Voltage applied =15 kV).](image-url)
Acknowledgments

The authors gratefully acknowledge financial support from Stephen F. Austin State University Faculty Research Grants (ORSP # 107552, and Research minigrants) and Robert A. Welch Foundation Grant Number AN-0008.

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