



ELSEVIER

Contents lists available at ScienceDirect

## Data in Brief

journal homepage: [www.elsevier.com/locate/dib](http://www.elsevier.com/locate/dib)

CrossMark

## Data Article

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

Kefa K. Onchoke<sup>a,\*</sup>, Christopher M. Franclemont<sup>a</sup>,  
Paul W. Weatherford<sup>b</sup>

<sup>a</sup> Department of Chemistry & Biochemistry, Stephen F. Austin State University, Box 13006 – SFA Station, Nacogdoches, TX, 75962-13006, USA

<sup>b</sup> Department of Agriculture, Stephen F. Austin State University, Soil, Plant and Water Analysis Lab, P.O. Box 13025 SFA Station, Nacogdoches, TX 75962-3025, USA

## 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  $K\alpha_2$  peaks the d-spacing and hkl values were determined. In addition, the ion chromatographic profile of the seven anions ( $\text{NO}_3^-$ ,  $\text{NO}_2^-$ ,  $\text{Br}^-$ ,  $\text{Cl}^-$ ,  $\text{F}^-$ ,  $\text{SO}_4^{2-}$ , and  $\text{PO}_4^{3-}$ ) in biosolids is presented.

© 2018 The Authors. Published by Elsevier Inc. This is an open access article under the CC BY license

(<http://creativecommons.org/licenses/by/4.0/>).

## Specifications Table

Subject area	<i>Environmental Chemistry</i>
More specific subject area	<i>Wastewater sludge (biosolids)</i>
Type of data	<i>Table, graph, figure</i>

DOI of original article: <https://doi.org/10.1016/j.saa.2018.06.096>

\* Corresponding author.

E-mail address: [Onchokek@sfasu.edu](mailto:Onchokek@sfasu.edu) (K.K. Onchoke).

<https://doi.org/10.1016/j.dib.2018.08.087>

2352-3409/© 2018 The Authors. Published by Elsevier Inc. This is an open access article under the CC BY license (<http://creativecommons.org/licenses/by/4.0/>).

How data was acquired	<p><i>Ion chromatography, XRD, SEM, EDX were used in the study.</i></p> <p>(a) Dionex Integrion HPLC ion chromatograph (Thermo Fisher Scientific Inc., USA) was used for anion analysis.</p> <p>(b) A Bruker AXS D8 Advance diffractometer equipped with an X-ray tube (Cu <math>K_{\alpha}</math> radiation: <math>\lambda = 1.54060 \text{ \AA}</math>, 40 kV, and 40 mA) using a Ni filter and one-dimensional LynxEye detector at scanning speed of <math>2^{\circ}/\text{min}</math> and <math>0.0125^{\circ}</math> step sizes and a 1 s/step.</p> <p>(c) A JEOL-JSM 6100 scanning electron microscope equipped with a Horiba energy dispersive X-ray spectroscopy (SEM/EDX) was used.</p>
Data format	Raw, filtered, analyzed
Experimental factors	<p>(a) For XRD analysis: Biosolid samples were obtained from Nacogdoches and Lufkin wastewater treatment plant (NWWTP, LWWTP), air dried, and ground to powder.</p> <p>(b) For IC analysis: samples were filtered on a <math>0.45 \mu\text{m}</math> filter.</p>
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: $31^{\circ} 33' 31.2444'' \text{ N}$ and longitude $94^{\circ} 38' 52.1808'' \text{ W}$ ,
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 ( $\text{Cl}^{-}$ ,  $\text{F}^{-}$ ,  $\text{NO}_3^{-}$ ,  $\text{NO}_2^{-}$ ,  $\text{Br}^{-}$ ,  $\text{SO}_4^{2-}$ , and  $\text{PO}_4^{3-}$ ) during the analyses is provided (Fig. 2).

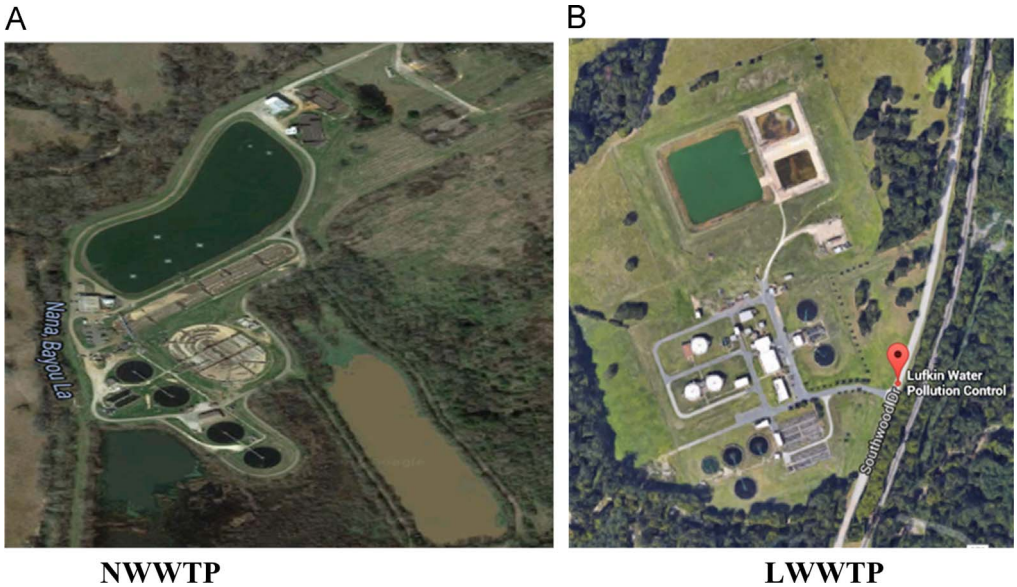
**Table 1**  
Analysis of the Crystalline Phases, d-spacing, and h, k, l values of biosolids in NWWTP [1].

Index	Angle ( $2\theta$ )	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
						1 -2 -1	Hexahydrate
						1 -2 -1	Quartz
2	22.663	3.92039	258	4887	2.90%	0 3 1	Gypsum
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
						1 -3 1	Mirabilite
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%	3 2 -3	Vermiculite
						1 3 3	Antigorite T
						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
						0 1 2	
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
						1 2 1	
25	67.57	1.38524	305	2359	3.50%	1 2 2	Quartz
						2 1 2	
26	68.019	1.37718	781	2833	8.90%	0 2 3	Quartz
						2 0 3	
27	68.16	1.37468	553	2596	6.30%	3 0 1	Quartz
						0 3 1	
28	73.358	1.28957	239	2203	2.70%	0 1 4	Quartz
						1 0 4	
29	75.597	1.25684	393	2319	4.50%	0 3 2	Quartz
						3 0 2	
30	79.662	1.20261	271	1528	3.10%	2 1 3	Quartz
						1 2 3	

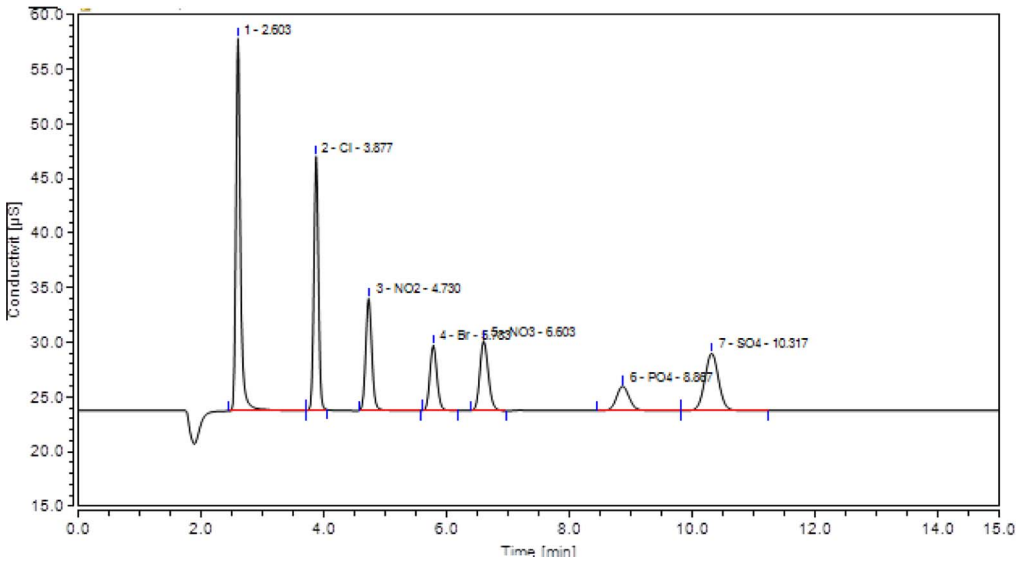
## 2. Experimental design, materials, and methods

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.





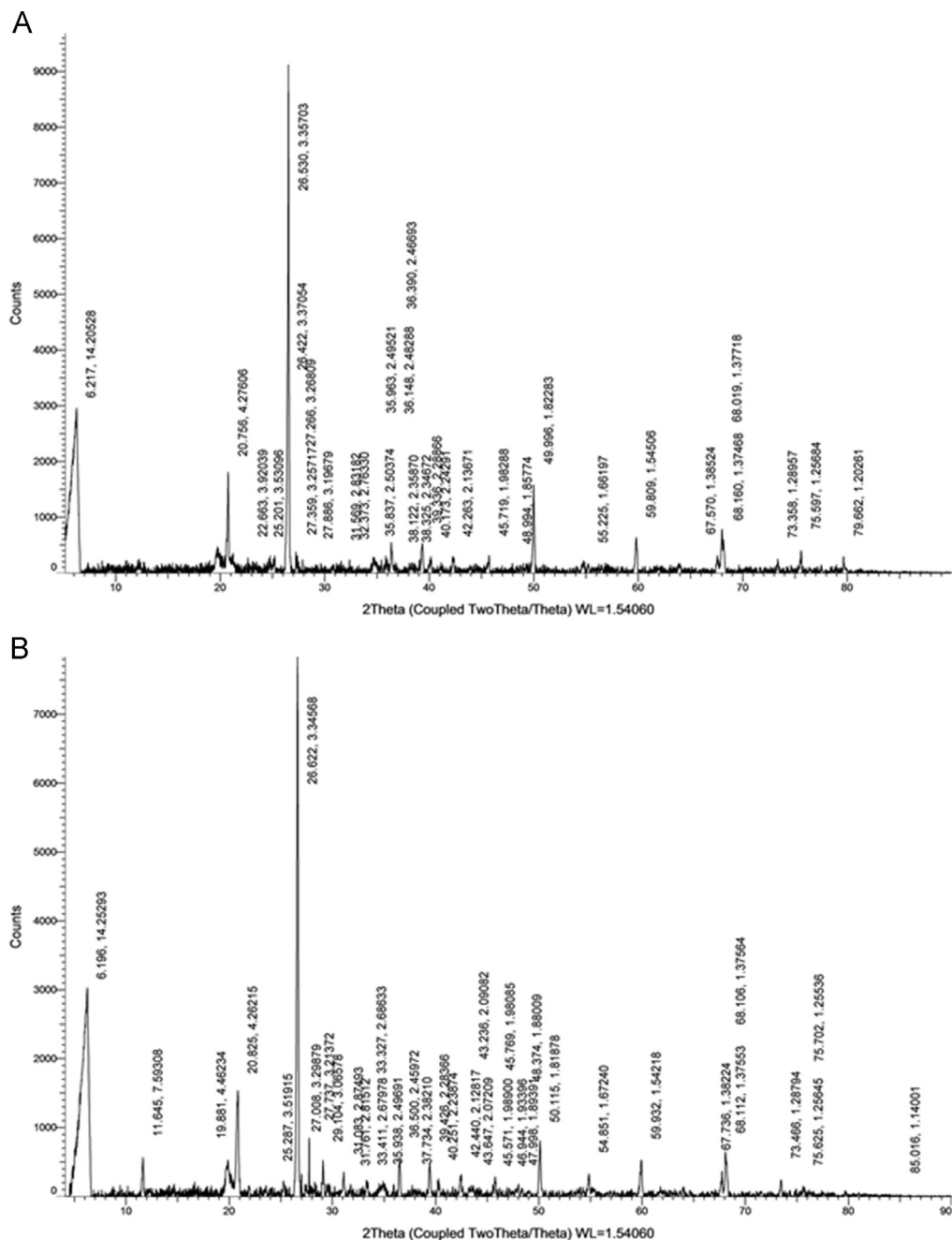
**Fig. 1.** Aerial photographs of (a) Nacogdoches wastewater treatment plant (NWWTP), and (b) Lufkin wastewater treatment plant (LWWTP). In each of the pictures, 4 clarifiers are observed.



**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 Integriion HPIC ion chromatograph (Thermo Fisher Scientific Inc., USA) was used. A Dionex IonPac AS22 analytical column ( $2 \times 250$  mm) thermostated at  $30^\circ C$ , guard column (IonPac AG22), a Dionex AS 22 Eluent Concentrate (4.5 mM sodium carbonate/1.4 mM sodium bicarbonate) was used.

### 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:  $\sim 35,000$ ). These



**Fig. 3.** Powder XRD patterns of samples from the Nacogdoches Wastewater Treatment Plant (A), and Lufkin Wastewater Treatment Plant (B). The  $2\theta$  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

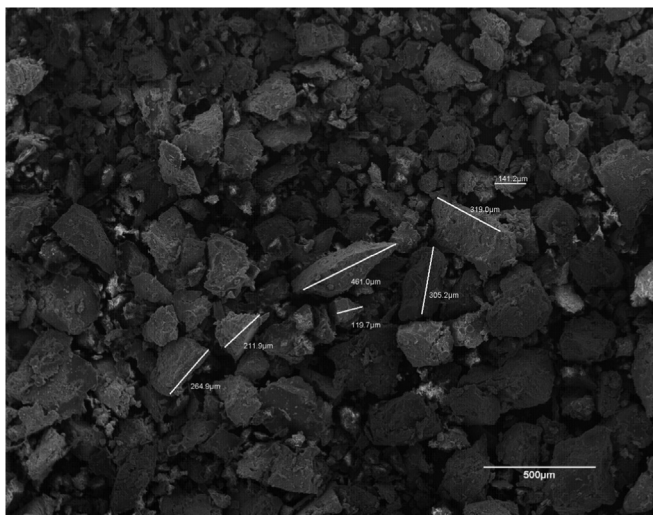
Biosolids were collected from the Nacogdoches Wastewater Treatment Plant (NWWTP) and Lufkin Wastewater treatment Plant (LWWTP) during the Summer 2016 and 2017. Multiple grab samples from the Nacogdoches and Lufkin WWTPs were dried in the lab. Biosolid samples were stored in plastic containers or 5-gallon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

## 2.3. Ionic analysis elution profile

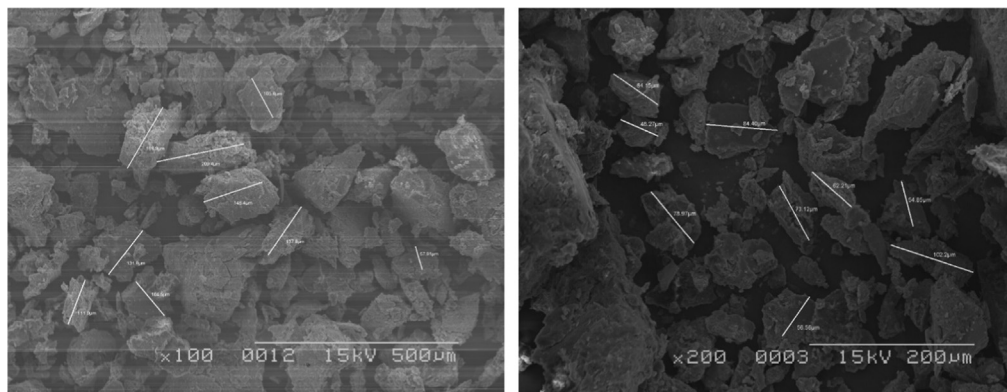
Approximately 28 mg of finely crushed biosolids was first added to a 25 mL volumetric flask and the volume filled to the mark using 18.2 M $\Omega$  water. The contents of the flasks were then sonicated for 20 min and the sample split into two separate 15 mL Falcon tubes. Following this, the samples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

## 2.4. Morphological characterization of biosolids

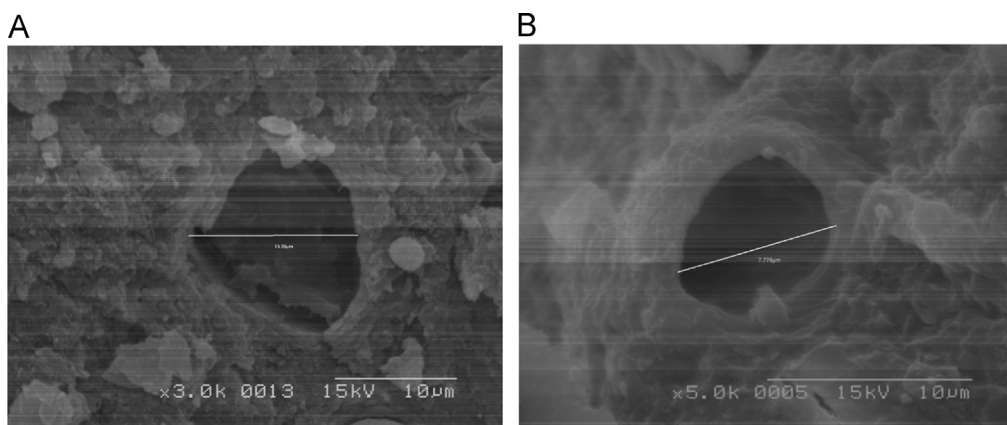
The biosolids were air dried, crushed with mortar and pestle, and analyzed with JEOL-JSM 6100 scanning electron microscope equipped with a Horiba energy dispersive X-ray spectroscopy (SEM/EDX) with an accelerating voltage of 15 kV. The surface morphology, particle diameters (Figs. 4–6) of biosolids were measured at X40, 100–200 $\times$  magnifications. Powder XRD analysis was performed in the  $2\theta$  range of  $2^\circ$ – $90^\circ$  on a Bruker AXS D8 Advance diffractometer equipped with an X-ray tube (Cu  $K_\alpha$  radiation:  $\lambda = 1.54060 \text{ \AA}$ , 40 kV, and 40 mA) using a Ni filter and one-dimensional LynxEye detector at scanning speed of  $2^\circ/\text{min}$  and  $0.0125^\circ$  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 $\times$ , Voltage applied = 15 kV).



**Fig. 5.** Representative SEM micrograph of the LWWTP biosolid with measurement of smaller particles; magnification 100 ×, and 200 ×.



**Fig. 6.** SEM micrograph of a pore from the NWWTP biosolid with measurement of its diameter; magnification 3 kV (a) and 5 kV (b).

## 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.

## References

- [1] K.K. Onchoke, C.M. Franclemont, W.P. Weatherford, Structural characterization and evaluation of municipal wastewater sludge (biosolids) from two rural wastewater treatment plants in East Texas, USA, *Spectrochim. Acta A* 204 (2018) 514–524.
- [2] DIFFRAC EVA, Madison, WI, USA, (2008.). DIFFRAC EVA, BrukerAXS, Madison, WI, USA, 2008.
- [3] BrukerAXS. TOPAS, V5.0. (Computer Software), Bruker AXS: Karlsruhe, Germany, 2011.
- [4] S. Bai, S. Srikantaswamy, V. Krishnamandan, O.P. Naik, Speciation of heavy metals in biosolids of wastewater treatment plants at Mysore, Karnataka, India, *Environ. Monit. Assess.* 184 (2012) 239–249. <https://doi.org/10.1007/s10661-011-1964-3> (Epub 2011 Mar 23).
- [5] P. Westerhoff, S. Lee, Y. Yang, G.W. Gordon, K. Hristovski, R.U. Halden, P. Herckes, Characterization, recovery opportunities, and valuation of metals in municipal sludges from U.S. wastewater treatment plants nationwide, *Environ. Sci. Technol.* 49 (2015) 9479–9488. <https://doi.org/10.1021/es505329q>.



- [6] M.A. Kiser, P. Westerhoff, T. Benn, Y. Wang, J. Pérez-Rivera, K. Hristoviski, Titanium nanomaterial removal and release from wastewater treatment plants, *Environ. Sci. Technol.* 43 (2009) 6757–6763. <https://doi.org/10.1021/es901102n>.
- [7] E. Donner, G. Brunetti, B. Zarcina, P. Harris, E. Tavakkoli, R. Naidu, E. Lombi, Effects of chemical amendments on the lability and speciation of metals in anaerobically digested biosolids, *Environ. Sci. Technol.* 47 (2013) 11157–11165 ([pubs.acs.org/doi/abs/10.1021/es400805j](https://pubs.acs.org/doi/abs/10.1021/es400805j)).
- [8] D. Zhang, Y. Chen, Y. Zhao, X. Zhu, New sludge pretreatment method to improve methane production in waste activated sludge digestion, *Environ. Sci. Technol.* 44 (2010) 4802–4808. <https://doi.org/10.1021/es1000209>.