Desalination and Water Treatment
www.deswater.com

doi: 10.1080/19443994.2015.1119760

57 (2016) 15649–15659
July

Taylor & Francis
Taylor & Francis Group

Novel method for water purification using activated adsorbents developed from sewage sludge

Yehya Elsayed[a,*], Mustafa Khamis[a], Fatin Samara[a], Maitha Alqaydi[a], Ziad Sara[a], Isam Al Zubaidi[b], Md. Maruf Mortula[c]

[a]*Department of Biology, Chemistry and Environmental Sciences, College of Arts and Sciences, American University of Sharjah, P.O. Box 26666, Sharjah, UAE, Tel. +971 6 5152576; Fax: +971 6 515 2450; emails: yelsayed@aus.edu (Y. Elsayed), mkhamis@aus.edu (M. Khamis), fsamara@aus.edu (F. Samara), maitha.alqaydi@alumni.aus.edu (M. Alqaydi), zsara@aus.edu (Z. Sara)*
[b]*Faculty of Engineering and Applied Science, University of Regina, Regina, Canada, email: Isam.Al.Zubaidi@uregina.ca*
[c]*Department of Civil Engineering, College of Engineering, American University of Sharjah, Sharjah, UAE, email: mmortula@aus.edu*

Received 23 August 2015; Accepted 26 October 2015

ABSTRACT

Sewage sludge from Dubai was characterized using inductively coupled plasma and CHN elemental analysis. Selected sludge samples were washed with deionized water until constant conductivity was achieved. The sludge samples (washed and unwashed) were carbonized at 680˚C in a semi-static steam environment that was self-generated inside a homemade reaction chamber and with the absence of any external flow. The carbonized materials were then activated by impregnation with sodium hydroxide at 3:1 mass ratio followed by thermal treatment at 680˚C in the presence of water. The activated products were characterized using SEM/EDS, CHN elemental analysis, and sorption of nitrogen. The results showed that washing the sludge prior to the carbonization step significantly enhanced the surface area of the final activated product. Activation efficiencies were lower than the carbonization efficiencies. The surface area of the activated adsorbent developed from the washed sludge was 1,552 m^2/g compared to 1,182 m^2/g of the unwashed sample. Pore size distribution showed slight shift to higher average pore size in the washed sample. The activated adsorbent with the highest surface area was then evaluated for the adsorption of lead. The adsorption capacity of the activated product for lead removal was determined using Toth isotherm. The maximum adsorption capacity for the activated washed sludge was found to be 95 mg/g. The energy contents of the washed raw sludge and the tar by-products that were produced during the carbonization process were found to be 14.8 and 38.1 kJ/g, respectively.

Keywords: Sewage sludge; Adsorbent materials; Environmental remediation; Steam activation; Lead; Sustainable process

---

*Corresponding author.

Presented at the 3rd International Conference on Water, Energy and Environment (ICWEE) 24–26 March 2015, Sharjah, United Arab Emirates

1944-3994/1944-3986 © 2015 Balaban Desalination Publications. All rights reserved.

1. Introduction

Sewage sludge is the residual, semi-solid to solid material produced from wastewater treatment plants consisting of nutrients (nitrogen and phosphorus), fats, silica, proteins, and a wide variety of major and trace elements [1,2]. Disposal and management of sewage sludge is an environmental concern due to the massive amounts of sludge produced each year [3–5]. There are various sewage sludge disposal methods currently available including: landfilling [4–7], disposal into oceans [5–7], incineration [1,4], and farmland utilization [4,5]. Nowadays, many of these practices are restricted due to poor public image, high cost, and safety issues associated with the release of toxic materials and hazardous ash residues into the environment [1,4,6]. Furthermore, the disposal of sludge is not always done in a controlled manner which might introduce serious pollution issues [7]. The United Arab Emirates (UAE) and other Arabian Gulf countries manage their sewage sludge waste by conversion into organic soil conditioner and fertilizer [8]. However, the use of sewage sludge as an organic fertilizer in farmland applications is associated with several health issues due to the presence of toxic compounds, metals and trace elements such as lead, copper, antimony and selenium in the sludge [4]. Therefore, it is necessary to find a simple, efficient, inexpensive, and environment friendly disposal and management method for sewage sludge [8]. Conversion of sewage sludge to an adsorbent can be a suitable option. However, the production of an efficient adsorbent for environmental remediation from sewage sludge should provide a possible alternative away from the limitations of the above methods [9]. The produced adsorbents from sewage sludge have several properties in common with activated carbons, but with higher inorganic and lower carbon contents. Activated carbon adsorbents are usually produced from various natural materials including wood [10], coal, date stones [11], apricot stones [12], coconut husks [13], and rich hulls [14]. Activated carbon is a porous form of carbon with large surface area that makes it a highly efficient adsorbent for water and air purification applications in which organic pollutants and heavy metals are removed [15–23].

Activated sludge adsorbents are generally prepared using two processes. The first process involves carbonization of the organic material [18], while the second process involves activation of the carbonized products [18,22]. Several studies have been reported on the carbonization and the activation processes [1,3,5–7,18,21–32]. However, these methods have some drawbacks including the production of potentially hazardous chemical waste that might lead to air or water pollution. Although most of the produced adsorbent materials from sewage sludge in the literature were successful and effective for the removal of heavy metals such as copper II [29], lead [30], and strontium [33], all of them were done under dynamic flow of inert gases. In addition, the sludge samples were not modified prior to the carbonization step. The drawbacks of such processes are high cost, elevated ash content, and complexity of the process.

Adsorbents have wide range of pore width that can be categorized as: micropores (<2 nm), mesopores (2–50 nm), and macropores (450 nm) [22]. Pore size distribution depends on the precursor material [28] and the activation method. Two methods are usually employed for activation: physical [1,22] and chemical activation [22]. In physical activation, partial combustion or thermal decomposition occurs, leading to the creation of the porous structure [1,22]. In chemical activation, the activating agents are responsible for development of the pores by dehydration and degradation [22]. The presence of pores increases the adsorption potential of the activated carbon. The interaction between the adsorbent and the adsorbate is a key criterion for an effective purification process in which an adsorbate adheres to the surface and the rest of the sample is eluted. The adsorption capacity of the activated carbon depends on the nature, distribution, size, and volume of its pores as well as its surface area [19–21]. The presence of high ash content in the activated adsorbents produced from sewage sludge might negatively affect its adsorption capacity by limiting the development of pores [31]. Sewage sludge-based activated carbon usually has 35–40% ash content compared to less than 10% in commercial activated carbon [32]. Nevertheless, the inorganic salts and metal oxides in ash might provide catalytic centers for some adsorption applications.

The aim of this paper is to develop a simple and effective process for the preparation of adsorbent materials with unique and developed surface properties using sewage sludge waste as a precursor in order to provide an effective solution for the sewage sludge disposal problem worldwide. Moreover, the application of the developed adsorbent materials for the removal of lead from water samples is evaluated.

2. Experimental section

2.1. Sludge samples

Sewage sludge samples were obtained from a Wastewater Treatment Plant in Dubai, UAE. Three sludge samples were selected based on the stage of treatment, including: (1) sludge sample before dewatering and prior to polymer addition (SD); (2) sludge sample before dewatering and after polymer addition (SP); and (3) sludge sample at the final stage of treatment where the sample is thermally dried and processed (S). Samples were oven dried at 105℃ overnight prior to analysis. As a result of the ICP and ash content characterization analysis discussed below, sample S was selected for carbonization and activation. The selected sample was then sieved to a particle size of 1–2 mm. Since sludge samples are known to be rich in inorganic content, 100 g of sample S was also washed with sequential aliquots of deionized water (mass ratio of 1:1 of sample/water) until constant conductivity of the effluent was achieved. The washed sample was labeled as SW.

2.2. Apparatus

Carbonization and activation processes were performed using a homemade stainless steel apparatus constructed in our facilities. The design of the apparatus was adopted from a previous study and is shown in Fig. 1 [30]. The stainless steel boat used for the carbonization and activation processes consisted of two sections, an inner section where the sample is placed and an outer section filled with distilled water which is converted to steam upon heating, creating a semi-static steam environment that is used for the carbonization and activation processes. In this apparatus, a tar trap was connected to the outlet line and designed to collect the by-products.

2.3. Carbonization and activation

The sewage sludge samples (S and SW) were carbonized in the self-generated steam environment described in Section 2.2. Thirty grams of a sieved sludge sample was placed in the inner section of the steel dual boat and 200 mL of deionized water were placed in the outer section of the dual boat. The boat was placed inside the steel reaction chamber which was adjusted inside a muffle furnace. The reaction chamber was purged with nitrogen for 5 min before carbonization to remove oxygen and other reactive gases from the inner chamber environment. The carbonization process was carried out at 680℃ and a heating rate of 5℃/min. Once the desired temperature was reached, the furnace was kept isothermal for 30 min. The carbonization was done on two replicates for each sample. After carbonization, the samples were labeled as S-C1, S-C2, SW-C1, and SW-C2. The tar produced was collected for further analysis.

The carbonized sludge samples were then activated by chemical treatment with potassium hydroxide (KOH). Ten grams of the carbonized samples were mixed with KOH powder at a mass ratio of 1:3 and then placed in the inner section of the steal dual boat. The activation was carried out in the steam environment in the same manner as described for carbonization. However, once the desired temperature of 680℃ is reached, the furnace temperature was kept isothermal for one hour. The product was washed with 60–100 mL 5 M HCl then rinsed with distilled water until pH of 6 was obtained. The activated samples were labeled S-A and SW-A. The “W” refers to the washed sludge as described before.

2.4. Characterization methods

2.4.1. Trace metals analysis

Bulk trace metal analysis of the sludge samples was done using sequential inductively coupled plasma (AX sequential ICP-OES-Varian-Liberty). Choice of metals was based on commonly found metals for similar samples reported in literature [4] and included Aluminum (Al), Cadmium (Cd), Cobalt (Co), Chromium (Cr), Copper (Cu), Iron (Fe), Manganese (Mn), Nickel (Ni), Lead (Pb), and Zinc (Zn). TraceCERT (Sigma Aldrich, Switzerland), a multi-element stock

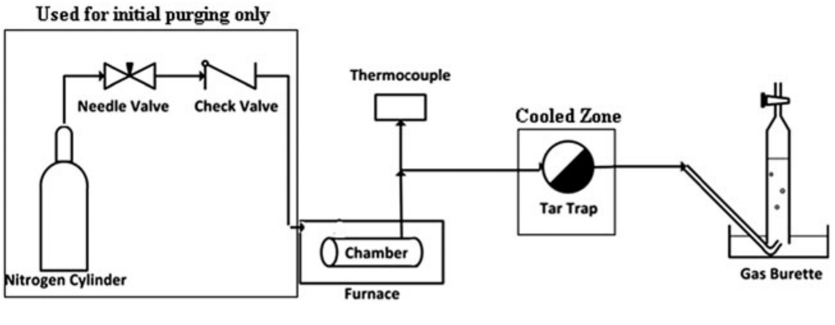

Fig. 1. A flow diagram of the experimental setup for the carbonization and activation process.

solution was used to prepare the standards in the range of 0.001–10 ppm. One gram of each sludge sample was ashed at 550˚C for 6 h in a muffle furnace. The ashed sludge samples were acid digested using microwave assisted digestion (Multiwave 3000, Anton Paar, Austria). In this process, 12 mL mixture of HNO_3 (16 M) and HCl (36%) in ratio of 3:1 by volume were added to each vessel. The digestion time was adjusted to 15 min at 800 W.

2.4.2. CHN analysis

Carbon, hydrogen and nitrogen elemental analysis for all raw sludge samples (SD, SP, S, and SW) was performed using EA3000 elemental analyzer (EuroVector).

2.4.3. Ash content

Ash content of the raw sludge materials was determined using standard method [34]. Briefly, one gram of each sample (SD, SP, S, and SW) was weighed in a crucible then heated in a muffle furnace at 550˚C for 6 h. The mass of the ash was then measured.

2.4.4. SEM/EDS elemental analysis

Scanning electron microscope (Oxford Instruments, UK) was used to study the structural heterogeneity of the carbonized and activated samples by collecting magnified images of the surface. Elemental analyses of the surface of the washed, carbonized, and activated samples were measured using energy dispersive X-ray spectroscopy INCA X-act. The SEM was operated at an accelerating voltage of 15 and 20 keV. The scanning electron microscopy coupled with energy-dispersive spectroscopy analyses were done on the same batch of samples.

2.4.5. Sorption of nitrogen

Nitrogen adsorption and desorption isotherms were constructed at 77 K using a gas sorption analyzer (Autosorb iQ, Quantachrome). Before the experiment, the activated samples were heated at 423 K and then outgassed under a vacuum of 10–5 torr to constant pressure. Characterization of pore sizes and pore structure were accomplished using Density Functional Theory (DFT). The Brunauer–Emmett–Teller (BET) isotherm was used to determine the surface area for the activated sample.

2.4.6. Energy content

Energy content of the raw material and the tar produced during the carbonization process were determined using a bomb calorimeter (Parr Instrument Co., USA). 0.5 g of sample was used in each case.

2.5. Adsorption studies

The activated adsorbent with the highest surface area produced in the proposed process was evaluated using the adsorption of lead. The adsorption capacity was estimated using Toth models [31]. A set of different concentrations of lead nitrate solution ranging from 10 to 500 ppm were added to 0.05 g of the activated adsorbent. The solutions were mixed in the thermal shaker at 200–250 rpm and 30˚C for 48 h. The suspensions were then filtered and the concentration of lead was determined using Varian 220 FS flame atomic absorption spectrometer (FAAS).

2.6. Adsorption models

Langmuir–Freundlich and Toth isotherms are used to describe adsorption from aqueous solutions on microporous heterogeneous surfaces [35]. Based on our previous results [30], Toth adsorption isotherm model was selected.

Toth isotherm is described by the following equation:

$$q_e = \frac{K_t C_e}{\left(a_t + C_e^t\right)^{1/t}}$$

where q_e is the amount adsorbed at equilibrium per gram of adsorbent (mg/g), K_t is the maximum adsorption capacity (mg/g), C_e is the equilibrium concentration (mg/L), a_t is the adsorption equilibrium constant, and t is a dissociation parameter.

3. Results and discussions

3.1. Sludge samples evaluation

3.1.1. Major and trace metals analysis

ICP analysis was performed on the ashed sludge sample before dewatering and prior to polymer addition (SD), sludge sample before dewatering and after polymer addition (SP) and sludge sample at the final stage of treatment where the sample is thermally dried and processed (S) samples to determine the concentrations of major and trace metals in each sample. The results from the ICP analysis showed various amounts of heavy metals and trace elements that are potentially hazardous to the environment (Table 1). Table 1 reveals that the final stage sludge sample (sample S),

Table 1
Heavy metal composition of the studied sludge samples (mg/kg). S: final stage sludge (thermally dried and processed); SD: before dewatering and prior to polymer addition; and SP: before dewatering and after polymer addition

Concentration (mg/kg)				
Element	S	SD	SP	Standard limits [36]
Al	6,874 ± 547	5,471 ± 116	5,370 ± 101	–
Cd	6.14 ± 0.11	1.21 ± 0.01	1.16 ± 0.02	30
Co	1.89 ± 0.04	1.00 ± 0.07	1.01 ± 0.28	100
Cr	163 ± 3.1	54.3 ± 2.2	55.9 ± 2.0	1,000
Cu	845 ± 10	311 ± 2.6	295 ± 0.88	1,000
Fe	12,569 ± 954	10,049 ± 165	9,868 ± 359	–
Mn	129 ± 1.2	134 ± 1.9	135 ± 1.8	–
Ni	3.01 ± 0.04	1.40 ± 0.06	1.42 ± 0.05	200
Pb	46.6 ± 2.1	25.9 ± 0.18	28.1 ± 0.86	1,000
Zn	2,428 ± 10	1,599 ± 13	1,513 ± 8.5	1,000

used as a fertilizer, had the highest concentrations of metals. The metals found at the highest concentrations were iron (12,569 mg/kg) followed by aluminum (6,874 mg/kg), zinc (2,428 mg/kg), copper (845 mg/kg), and chromium (163 mg/kg). On the other hand, cobalt showed the lower concentration (1.89 mg/kg) followed by nickel (3.01 mg/kg), cadmium (6.14 mg/kg), and lead (46.6 mg/kg). It is noteworthy to mention that the concentrations of all analyzed metals were within the standards set by the environment department in Dubai municipality, which has set maximum concentration limits to some trace elements in sewage sludge for its use as a compost or fertilizer in agricultural activities, except for zinc [36]. The presence of excess concentrations of zinc in sludge and subsequently in soil might lead to serious pollution and health issues including diarrhea, vomiting, nausea, and abdominal cramps [37]. Metals might be taken up by the plants and animals and thus enter the food chain. Furthermore, water-soluble metals might leach into the groundwater leading to contamination [36].

3.1.2. Washing

Sewage sludge is known to contain a wide range of pathogens since it is mainly composed of fecal materials [36]. In order to decrease the inorganic content of the sewage sludge and provide a better precursor for the carbonization and activation processes, sample S was washed with de-ionized water in a ratio 1:2. The conductivity of the leachate was monitored as a function of the washing cycle (Fig. 2). The results of the washing experiment showed that the leachate's conductivity decreased after multiple washings until a constant conductivity was achieved. Hence, suggesting that the majority of ions in the sludge were leached out.

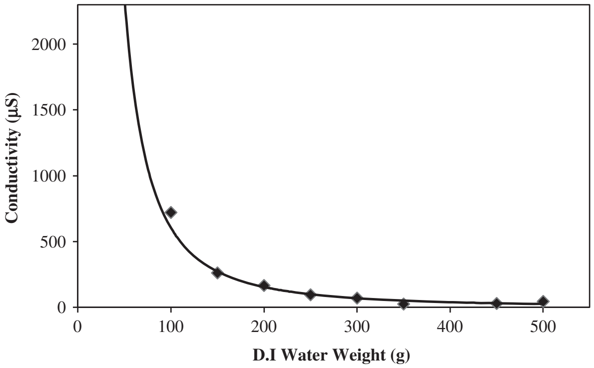

Fig. 2. The change in the conductivity of sample "S" leachate with equal water aliquots.

3.1.3. Ash content and CHN analysis

The ash content of SW was 26.2% by weight as reported in Table 2. The difference in the ash content compared to sample S is about 1% indicating that some of the inorganic content is present within the sewage sludge and not on the surface [35]. The carbon, hydrogen, and nitrogen elemental composition and the ash content of the different types of sewage sludge samples were determined and presented in Table 2. The hydrogen and nitrogen content of the four types is approximately identical and it ranges between 4.8–5.5 wt% and 4.7–5.3 wt%, respectively. However, there is a slight variation of the carbon and the ash content. S and SW sample results were close to those reported in literature [38]. SW has the highest percentage of carbon and the lowest percentage of ash

Table 2
CHN and ash content of studies sludge samples. S: final stage sludge (thermally dried and processed); SD: before dewatering and prior to polymer addition; SP: before dewatering and after polymer addition and SW: washed sludge

Sample	C (%)	H (%)	N (%)	Other (%)	Ash content (%)
S	37.8	5.5	4.9	51.8	27.1
SW	38.1	5.2	4.7	52.0	26.2
SD	34.3	4.8	5.3	55.6	32.4
SP	33.9	5.2	4.8	56.1	34.1

content compared to the other three types of sewage sludge. The presence of high ash content hinders the development of pores and limits the adsorption capacity of the adsorbent material produced. Therefore, sample SW is the best for the development of adsorbent materials.

3.1.4. SEM/EDS elemental analysis

To further evaluate the composition, SEM/EDS elemental analysis was performed on the ash of samples S and SW and the results are summarized in Table 3. A broad range of elements including alkali and alkaline earth metals as well as other heavy metals were detected. Since the ash was developed by burning the samples in air, oxygen was the predominant element on the surface of sludge ash film. Calcium, silicon, potassium, phosphorous sulfur, magnesium, aluminum, and iron are present in major proportions. Other elements such as manganese, sodium, zinc, titanium, copper, chloride, strontium, lead, nickel, and chromium are present in lower amounts. Since the EDS results showed the presence of soluble inorganic salts on sample S, it was expected that washing would leach out some of these salts especially with the observed decrease in the conductivity upon washing. As shown in Table 3, the percentages of calcium, sodium, sulfur, chloride, potassium, manganese, nickel, and copper decreased upon washing sample S. The final product, after carbonization and activation processes, is expected to be highly dependent on the ash content and the characteristics of the raw material.

Table 3
EDS elemental analysis for final stage sludge (S) and washed sludge (SW) ash (wt%)

Element	S	SW
C	5.46	4.71
O	50.28	51.62
Na	0.91	0.42
Mg	3.12	2.84
Al	3.10	3.24
Si	7.18	8.62
P	4.98	5.36
S	3.86	2.22
Cl	0.34	0.26
K	5.12	12.62
Ca	9.26	0.55
Ti	0.43	0.04
Cr	0.03	0.04
Mn	1.57	5.55
Fe	2.59	0.01
Ni	0.16	0.27
Cu	0.41	0.85
Zn	0.73	0.32
Sr	0.29	0.47
Pb	0.17	4.71

3.2. Preparation of activated adsorbents

3.2.1. Carbonization

Table 4 summarizes the efficiency of the carbonization process for each sample. The efficiency is calculated as the weight percent of the carbonized material produced from the starting sludge sample. The efficiency of the carbonization process decreased slightly when SW is used as a starting material compared to sample S, with calculated efficiencies of 45.58 ± 0.50% and for S and 41.22 ± 0.50% for SW when used as starting materials. The slight decrease in efficiency was attributed to the presence of smaller amounts of inorganic salts in sample SW during the preliminary washing step.

Table 4
The efficiency of the carbonization process for different carbonized products. S-C: carbonization of final stage sludge and SW-C: carbonization of washed sludge

Sample	Starting material	Efficiency (%)
S-C1	S	45.58
S-C2	S	45.02
SW-C1	SW	41.22
SW-C2	SW	40.70

3.2.2. Characterization of the carbonized samples

The pH and conductivity of the carbonized samples are summarized in Table 5. The electrical conductivity of the leachate of samples SW-C1 and SW-C2 were lower than those of samples S-C1 and S-C2. This is expected due to the pre-washing treatment of the staring material. Samples SW-C1 and SW-C2 were prepared from sample SW and therefore are expected to have lower conductivity since some ions leached out during the washing process. It is important to note that all four samples displayed basic pH. This could be due to the presence of basic metal oxides.

3.2.3. Activation

S-C1 and SW-C2 were activated and labeled as S-A and SW-A. The efficiency of the activation processes was 27.42 ± 0.50% and 26.54 ± 0.50% for S-A and SW-A, respectively (Table 6). When compared to carbonization process, the activation efficiencies are much lower, potentially due to the degradation of the sample as a result of the chemical reaction that occurs between potassium hydroxide and the surface.

3.2.4. SEM/EDS Analysis of the activated samples

EDS analysis on the activated samples was used to determine the elemental composition of their surfaces. As shown in Table 7, heavy metals as well as alkali and alkaline earth metals were found. The carbonization and activation processes eliminate a significant amount of the inorganic content resulting in a noticeable increase in the organic (carbon) content. Hence, carbon was found at high percentages followed by oxygen and silicon. Other elements such as phosphorus, sulfur, titanium, and iron are present in moderate amounts ranging from 2.65 to 7.80 mg/g in S-A and 1.00 to 8.10 mg/g to 1.00 mg/g in SW-A.

Table 5
pH and EC of different carbonized samples. S-C: carbonization of final stage sludge and SW-C: carbonization of washed sludge

Sample	pH ± 0.01	EC (μS) ± 0.1
S-C1	8.65	290.0
S-C2	9.43	326.0
SW-C1	9.72	183.4
SW-C2	9.63	178.6

Table 7
Surface elemental composition of the activated samples (mg/g sample). S-A and SW-A are the activated samples produced from final stage sludge and washed sludge, respectively

Element	S-A	SW-A
C	551	679
O	337	252
Si	85.6	48.1
Fe	7.80	1.30
Cl	7.05	0.80
S	2.65	8.10
P	2.40	3.05
Ti	2.00	4.85
Al	1.15	0.05
Cu	0.90	B.D.
Zn	0.90	B.D.
Cr	0.70	1.00
Na	0.30	0.10
K	0.25	0.55
Ca	0.25	0.60
Ni	0.20	0.10
Pb	0.15	0.35
Mg	0.10	0.10
Mn	0.10	0.20
Sr	B.D.	B.D.

Note: B.D.: Below detection limit.

3.2.5. Nitrogen sorption isotherms

Fig. 3 shows the nitrogen adsorption/desorption isotherms which were determined at 77 K for both S-A and SW-A. The minimal hysterisis between the two curves at high P/P_o in SW-A indicates that the activated carbon is mainly microporous. On the other

Table 6
The efficiency of the activation process. S-A and SW-A are the activated samples produced from final stage sludge and washed sludge, respectively

Sample	Starting material	Carbonized sample	Efficiency (%)
S-A	S	S-C1	27.42
SW-A	SW	SW-C2	26.54

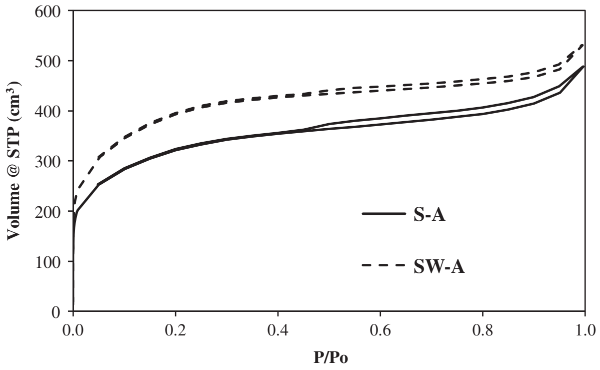

Fig. 3. Adsorption/desorption isotherms of N_2 at 77 K for KOH activated samples, S-A and SW-A.

hand, the slightly larger hysterisis is observed at high P/P_o in S-A indicating that the pores on the surface of the sample are large. The results observed in Fig. 3 are supported by the pore-size distribution (PSD) curves (Fig. 4) which prove that most of the pores present in the two activated samples are micropores. The presence of such pores indicate the effectiveness of this activated carbon in applications that involve volatile organic matters since the size of these compounds require the presence of microporous adsorbent. For S-A, several peaks are seen at 8.89 and 11.44 Å indicating that mostly micropores are formed during the activation process. For SW-A, the peaks are seen at 15.56 and at 20.07 Å indicating that micropores and some mesopores were formed during the activation process with relatively higher micropore volume. This accounts for the high surface area obtained for both samples. However, S-A has smaller pores than SW-A, this is expected since washing the starting material is expected to result in more opened pores.

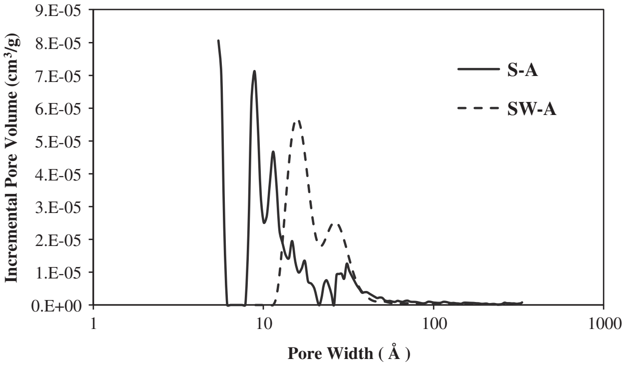

Fig. 4. Pore size distribution of the activated samples (S-A and SW-A) using DFT.

3.2.6. Surface area analysis

BET surface area (S_{BET}) was measured for both S-A and SW-A and found to be 1,183 and 1,552 m^2/g, respectively. The high surface area indicates the existence of microporous structure for the activated products. The surface area obtained in this study was higher than that of commercially available activated carbon and other adsorbents prepared from sewage sludge [3,5,6,24–32]. In addition, the modification introduced in the current work resulted in a more porous adsorbent compared to the material presented in a past study in spite of the fact that similar carbonization and activation methods were employed [30]. Such comparison is presented in Table 8. In addition to the type and composition of the starting material, the BET surface area depends on the chemical reagent used for activation and the environment inside the reaction chamber. Since S-A and SW-A were carbonized and activated under the same experimental conditions, the lower surface area of S-A compared to SW-A is linked to the difference in ash content of the starting material as it was previously discussed in this paper. As a result, SW and its carbonization and activation products were chosen for further characterization.

3.2.7. SEM images

SEM images of SW-C2 and SW-A are shown in Fig. 5. SEM images show some morphological changes in the developed carbon after activation indicating the formation of pores. Fig. 5(B) reveals the presence of pores on the external surface of SW-A after chemical activation. The pores were formed due to the release of volatile matters and the reaction between potassium hydroxide and the carboneous structure of the precursor (SW-C2).

3.3. Adsorption studies

In order to test the efficiency of the newly developed adsorbent for environmental remediation, the adsorption of lead, which represents a toxic heavy metal, was studied. Different concentrations of lead were used to generate an adsorption isotherm while keeping the mass of the activated sludge adsorbent constant. Toth isotherm was constructed to determine the maximum adsorption capacity of SW-A sample (Fig. 6). It was found that the data fits well on Toth isotherm with correlation coefficient of 0.93. The fitting parameters of the Toth isotherm were used to estimate the maximum adsorption capacity of SW-A which was calculated to be 95 mg/g. This value is much larger than the value reported in a previous study that

Table 8
BET surface area (m^2/g) for different activated samples. AC stands for commercial activated carbon. S-A and SW-A are the activated samples produced from final stage sludge and washed sludge, respectively

Study	S-A	SW-A	Rehman, S [30]	Commercial AC [17]
Sample surface area (m^2/g)	1,183	1,552	838	500–1,500

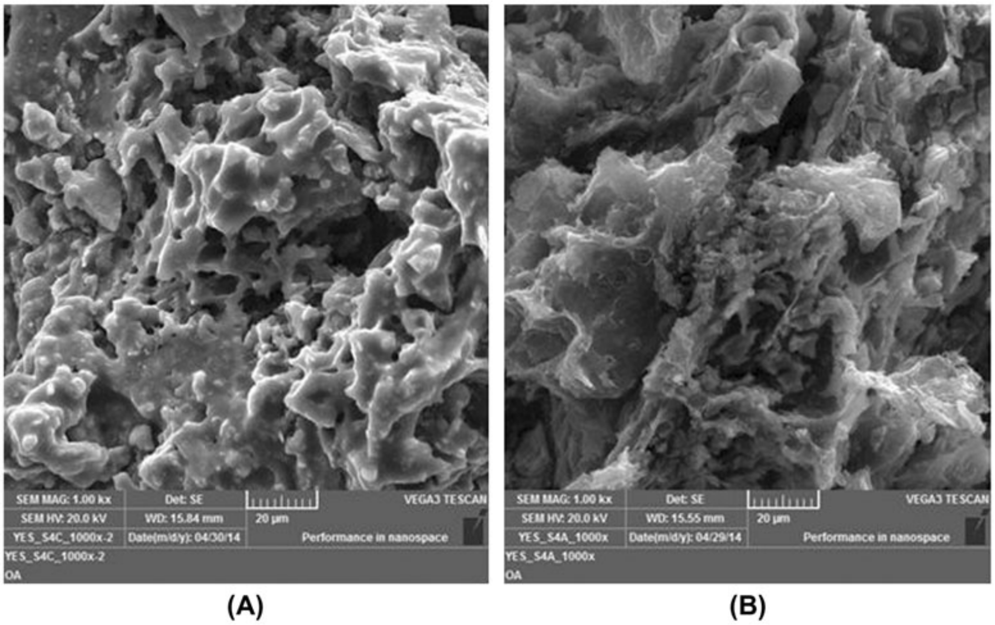

Fig. 5. SEM images taken at 1,000× for (A) the carbonized sample, SW-C and (B) the activated sample SW-A.

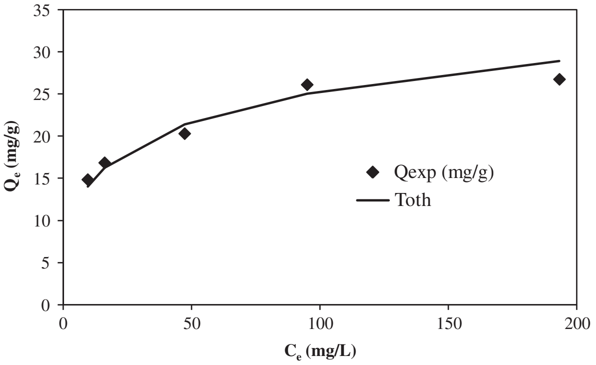

Fig. 6. Fitting for Toth adsorption isotherm for SW-A.

employs a similar carbonization and activation method where the maximum adsorption was estimated to be 49.2 mg/g [30]. The parameters that correspond to the fitting of the adsorption to the chosen isotherms are summarized in Table 9. Since the BET surface area of the developed material in a previous study was 838 m^2/g [30], there seem to be a good correlation between the surface area and the adsorption capacity using Toth fitting isotherm.

3.4. Energy content

Based on the unique properties and potential applications of SW, the energy content of the starting raw material (SW) and the tar produced throughout the carbonization process were measured using a bomb calorimeter. The goal was to evaluate the potential of generating energy within the process. The energy content of the tar was found to be 38.1 kJ/g, whereas the energy content of SW was found to be 14.8 kJ/g. The energy content of the tar is comparable to that of different forms of fuel including ethanol (30.0 kJ/g), crude oil (46.3 kJ/g), and anthracite coal (32.5 kJ/g) [39]. Therefore, the tar can be collected and used as an alternative source of energy. There was no ash residue after the combustion of tar indicating a complete and efficient burning.

Table 9
The fitting parameters for Toth isotherms

Toth		
K_t (mg/g)	a_t	t
95	0.80	0.21

4. Conclusions

This paper reports the successful production of a new adsorbent material using sewage sludge through a novel process. The different sludge samples contained high concentration of zinc, compared to other metals. Washing the sludge with deionized water produced an activated sludge of higher micropores size than that produced from unwashed sludge. Based on the ash content and CHN analysis, it can be concluded that SW is the best sludge sample for production of adsorbent materials. S had the higher carbonization efficiency than SW. The carbonized samples were basic in nature. Activation efficiencies were lower than the carbonization efficiencies. Nitrogen sorption isotherms revealed that most of the pores are micropores. The surface area of the activated sample that was produced using the washed sludge sample is 1,552 m^2/g compared to 1,182 m^2/g for the activated sample that was produced using un-washed sludge sample. Moreover, the prepared carbon material proved capable of removing lead from water. Toth isotherm was capable of fitting lead adsorption results. The tar produced in the carbonization process had an energy content of 38.1 kJ/g and thus it can be used as a source of energy. Finally, the results of this study provided an efficient and environmentally friendly management method for sewage sludge.

Acknowledgments

The authors of this paper would like to thank the American University of Sharjah for funding this research (Grant number: FRG13-2-06). Special thanks to the Dubai Municipality for providing the raw materials used in this research. Finally, thanks to the Department of Biology, Chemistry and Environmental Sciences at AUS, specially, Mr Nedal Abu Farha and Mr Thomas Job for their support during the analysis. Thanks are extended to Ricardo De Jesus from the Engineering workshop for construction the reaction chamber.

References

[1] K.M. Smith, G.D. Fowler, S. Pullket, N.J.D. Graham, Sewage sludge-based adsorbents: A review of their production, properties and use in water treatment applications, Water Res. 43(10) (2009) 2569–2594.
[2] B. Zhang, S. Xiong, B. Xiao, D. Yu, X. Jia, Mechanism of wet sewage sludge pyrolysis in a tubular furnace, Int. J. Hydrogen Energy 36(1) (2011) 355–363.
[3] V.M. Monsalvo, A.F. Mohedano, J.J. Rodriguez, Activated carbons from sewage sludge, Desalination 277 (1–3) (2011) 377–382.
[4] M.K. Hossain, V. Strezov, P.F. Nelson, Thermal characterisation of the products of wastewater sludge pyrolysis, J. Anal. Appl. Pyrolysis 85(1–2) (2009) 442–446.
[5] W.-H. Li, Q.-Y. Yue, B.-Y. Gao, X.-J. Wang, Y.-F. Qi, Y. Zhao, Y. Li, Preparation of sludge-based activated carbon made from paper mill sewage sludge by steam activation for dye wastewater treatment, Desalination 278(1–3) (2011) 179–185.
[6] D. Jung, Y. Chun, Characteristics on carbonization and steam activation of sewage sludge, Open Process Chem. J. 2 (2009) 12–19.
[7] W.-H. Li, Q.-Y. Yue, B.-Y. Gao, Z.-H. Ma, Y.-J. Li, H. Zhao, Preparation and utilization of sludge-based activated carbon for the adsorption of dyes from aqueous solutions, Chem. Eng. J. 171(1) (2011) 320–327.
[8] A. Geethakarthi, B.R. Phanikumar, Characterization of tannery sludge activated carbon and its utilization in the removal of azo reactive dye, Environ. Sci. Pollut. Res. 19(3) (2012) 656–665.
[9] Kawasaki Heavy Industries, Ltd., Production of Activated Carbon from Sewage Sludge and Cost Reduction of Sludge Treatment by Effective Utilization, (2011). Available from: <http://nett21.gec.jp/rerss/data/rerss-03.html>.
[10] K. Okada, N. Yamamoto, Y. Kameshima, A. Yasumori, Porous properties of activated carbons from waste newspaper prepared by chemical and physical activation, J. Colloid Interface Sci. 262(1) (2003) 179–193.
[11] C. Bouchelta, M.S. Medjram, O. Bertrand, J. Bellat, Preparation and characterization of activated carbon from date stones by physical activation with steam, J. Anal. Appl. Pyrolysis 82(1) (2008) 70–77.
[12] Ç. Şentorun-Shalaby, M.G. Uçak-Astarlıogˇlu, L. Artok, Ç. Sarıcı, Preparation and characterization of activated carbons by one-step steam pyrolysis/activation from apricot stones, Microporous Mesoporous Mater. 88(1–3) (2006) 126–134.
[13] J.H. Tay, X.G. Chen, S. Jeyaseelan, N. Graham, Optimising the preparation of activated carbon from digested sewage sludge and coconut husk, Chemosphere 44(1) (2001) 45–51.
[14] Y. Guo, D.A. Rockstraw, Activated carbons prepared from rice hull by one-step phosphoric acid activation, Microporous Mesoporous Mater. 100(1–3) (2007) 12–19.
[15] Y. El-Sayed, T.J. Bandosz, Adsorption of valeric acid from aqueous solution onto activated carbons: Role of surface basic sites, J. Colloid Interface Sci. 273 (2004) 64–72.

[16] Y. El-Sayed, T.J. Bandosz, A study of acetaldehyde adsorption on activated carbons, J. Colloid Interface Sci. 242 (2001) 44–51.

[17] Calgon Carbon, Activated Carbon & Specialty Products, (2013). Available from: <http://www.calgoncarbon.com/products/activated_carbon_specialty_products>.

[18] J. Hayashi, A. Kazehaya, K. Muroyama, A.P. Watkinson, Preparation of activated carbon from lignin by chemical activation, Carbon 38(13) (2000) 1873–1878.

[19] O.A. Ioannidou, A.A. Zabaniotou, G.G. Stavropoulos, M.A. Islam, T.A. Albanis, Preparation of activated carbons from agricultural residues for pesticide adsorption, Chemosphere 80(11) (2010) 1328–1336.

[20] D. Xin-hui, C. Srinivasakannan, W. Qu, W. Xin, P. Jinhui, Z. Li-bo, Regeneration of microwave assisted spent activated carbon: Process optimization, adsorption isotherms and kinetics, Chem. Eng. Process. Process Intensif. 53 (2012) 53–62.

[21] R. Hoseinzadeh Hesas, A. Arami-Niya, W. Wan Daud, J.N. Sahu, Preparation of granular activated carbon from oil palm shell by microwave-induced chemical activation: Optimisation using surface response methodology, Chem. Eng. Res. Des. 91(12) (2013) 2447–2456.

[22] J.M. Dias, M.C.M. Alvim-Ferraz, M.F. Almeida, J. Rivera-Utrillaand M. Sánchez-Polo, Waste materials for activated carbon preparation and its use in aqueous-phase treatment: A review, J. Environ. Manage. 85 (4) (2007) 833–846.

[23] Y. Tang, Q. Liu, F. Chen, Preparation and characterization of activated carbon from waste ramulus mori, Chem. Eng. J. 203 (2012) 19–24.

[24] M. Seredych, T.J. Bandosz, Removal of copper on composite sewage sludge/industrial sludge-based adsorbents: The role of surface chemistry, J. Colloid Interface Sci. 302(2) (2006) 379–388.

[25] A. Méndez, G. Gascó, M.M.A. Freitas, G. Siebielec, T. Stuczynski, J.L. Figueiredo, Preparation of carbon-based adsorbents from pyrolysis and air activation of sewage sludges, Chem. Eng. J. 108 (2005) 169–177.

[26] X. Chen, S. Jeyaseelan, N. Graham, Physical and chemical properties study of the activated carbon made from sewage sludge, Waste Manage. 22(7) (2002) 755–760.

[27] M.J. Martin, A. Artola, M.D. Balaguer, M. Rigola, Activated carbons developed from surplus sewage sludge for the removal of dyes from dilute aqueous solutions, Chem. Eng. J. 94(3) (2003) 231–239.

[28] F. Rozada, L.F. Calvo, A.I. García, J. Martín-Villacorta, M. Otero, Dye adsorption by sewage sludge-based activated carbons in batch and fixed-bed systems, Bioresour. Technol. 87(3) (2003) 221–230.

[29] X. Wang, X. Liang, Y. Wang, X. Wang, M. Liu, D. Yin, S. Xia, Adsorption of Copper (II) onto activated carbons from sewage sludge by microwave-induced phosphoric acid and zinc chloride activation, Desalination 278 (2011) 231–237.

[30] S.U. Rehman, Production of activated carbon from sewage sludge for the removal of lead from water, Master's Thesis, Sharjah, American University of Sharjah, UAE, 2012.

[31] A. Linares-Solano, I. Martín-Gullon, C. Salinas-Martínez de Lecea, B. Serrano-Talavera, Activated carbons from bituminous coal: Effect of mineral matter content, Fuel 79(6) (2000) 635–643.

[32] S. Rio, C. Faur-Brasquet, L.L. Coq, P. Courcoux, P.L. Cloirec, Experimental design methodology for the preparation of carbonaceous sorbents from sewage sludge by chemical activation—Application to air and water treatments, Chemosphere 58(4) (2005) 423–437.

[33] E. Kaçan, C. Kütahyalı, Adsorption of strontium from aqueous solution using activated carbon produced from textile sewage sludges, J. Anal. Appl. Pyrolysis 97 (2012) 149.

[34] M.J.F. Llorente, J.E.C. Garcia, Concentration of elements in woody and herbaceous biomass as a function of the dry ashing temperature, Fuel 85 (2006) 1273–1279.

[35] L.J. Uranowski, C.H. Tessmer, R.D. Vidic, The effect of surface metal oxides on activated carbon adsorption of phenolics, Water Res. 32 (1998) 1841–1851.

[36] Environmental standards and allowable limits of pollutants on land, water, and air environment, Environment Department, Dubai Municipality, Dubai, 2003. Available from: <http://www.esisys.com/Documents/allowablepollutants.pdf>.

[37] Institute of Medicine, Food and Nutrition Board, Dietary Reference Intakes for Vitamin A, Vitamin K, Arsenic, Boron, Chromium, Copper, Iodine, Iron, Manganese, Molybdenum, Nickel, Silicon, Vanadium, and Zinc, National Academy Press, Washington, DC, 2001.

[38] M. Kalyani, D.S. Ramteke, L.J. Paliwal, Production of activated carbon from sludge of food processing industry under controlled pyrolysis and its application for methylene blue removal, J. Anal. Appl. Pyrolysis 95 (2012) 79.

[39] Y. Demirel, Energy: Production, conversion, storage, conservation, and coupling, Springer Publications, London, 2012, pp. 27–70, doi: 10.1007/978-1-4471-2372-9.