



Characterization of Industrial Wastewater Sludge in Oman from Three Different Regions and Recommendations for Alternate Reuse Applications

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(Received 15 Feb 2015; accepted 18 Jul 2015)

Abstract

Background: Domestic and industrial wastewaters are mostly treated by biological process such as activated sludge, aerobic pond, and anaerobic treatment. This study focuses on characterizing the quality of sewage sludge in the Sultanate of Oman chosen from three industrial sewage treatment plants (STPs): Rusayl Industrial Estate (RSLIE); Sohar Industrial Estate (SIE); and Raysut Industrial Estate (RIE).

Methods: Samples of recycled activated sludge (RAS) and wasted activated sludge (WAS) were collected over a period of 12 months across above mentioned STPs. Parameters analyzed are electrical conductivity (EC), potential of hydrogen (pH), cations, anions and volatile content (VC).

Results: The obtained values for pH and EC were low for both RAS and WAS samples, except EC values of RIE that was more than 1000 $\mu\text{S}/\text{cm}$. The range of VC percentages in RAS and WAS samples were 44 to 86% and 41 to 77%, respectively. The measured values for chloride, sulfate, nitrate and phosphate were higher than the other anions.

Conclusion: The average values of the cations in RAS and WAS samples were within the Omani Standards, suitable for the re-use of sludge in agriculture except for Cd in RSLIE. The study recommends that a regular maintenance should be performed at the studied STPs to prevent any accumulation of some harmful substances, which may affect the sludge quality, and the sludge drying beds should be large enough to handle the produced sludge for better management.

Keywords: Industrial Wastewater sludge, Characterization, Analysis, physicochemical, Heavy metals, Sultanate of Oman

Introduction

The industrial wastewater is mostly treated by biological process such as activated sludge, aerobic pond, and anaerobic treatment. In Oman, wastewater treatment plants use activated sludge process (1). Activated sludge process is an effective technology to meet stringent standard if properly functioned. This process results in the generation of a large amount of waste activated sludge (2). The cost for excess sludge treatment has been estimated to be up to 60% of the total operating cost of a wastewater treatment plant (3).

Moreover, the customary disposal method of landfilling causes lesser pollution problems. Therefore, an interest in methods to reuse the sludge instead of dumping it has been grown rapidly. Before studying the best method to reuse wastewater sludge, characteristics and types of sludge produced in biological treatment plants should be identified.

There are more than 350 wastewater treatment plants distributed across different parts of Oman (4). During site visits and reviewing of database

for thirty seven sewage treatment plants (STP) consisting of 15 main STP and 22 small STP, it has been observed that roughly 96% of STPs were functioning as extended aeration processes, with the remainder operating as stabilization ponds which do not produce sludge at monthly and yearly intervals (5). Approximately 60% of STPs visited treat the sludge in drying beds. Numerous of these STPs were operating without proper planning and were poorly maintained, with either too much sludge being supplemented to the beds without providing the opportunity for the sludge to dry or allowed the sludge to remain too long in beds with resultant development of weeds (1). Around 8% of covered STPs used filter press. The remaining percentage of STPs visited had no sludge treatment processes, and in general, these sites periodically collect the sludge in trucks and get rid of it at landfills. Within Muscat Municipality, the dried sludge is transported from the Municipality STPs to Al-Ansab STP then to collect by Oman Fertilizer Company, which transports this sludge to Al-Khabourah for composting. Roughly, 69% of sludge is sent to dumping sites (5).

The main paths to reuse sludge are composting and recovering energy and the major parameters to check for the quality of sludge are heavy metals, anions, and volatile content. The effect of metal ions, anions, ortho-phosphate, polyphosphate and organic phosphorus on the activity and kinetics of alkaline phosphates in aerobic activated sludge are Investigated (6). Results showed that there were three impact models of heavy metals on alkaline phosphatase activity (APA) and most inorganic anions were somewhat innocuous at concentrations from 0 to 5.0 mM. However, phosphorus had an inhibitive effect on APA and pyrophosphate was the most effective inhibitor. A study assessed the distributions of total and chemical fractions of Fe, Mn, Ni, Cu, Zn, Cr, Pb, and Mo in different sludges (7). The highest concentrations were found for Fe, Mn, Pb, and Mo in digested sludge (DS), Ni and Cr in thickened sludge (TS), Zn in dewatering sludge (DWS), and Cu in active sludge (AS). Furthermore, organic matter contents, total nitrogen, total phosphate, electrical conductivity (EC) and, pH of sludges were deter-

mined. A significant positive correlation with concentrations of exchangeable and reducible fraction of Pb, Mo, Cr, Cu, and Fe were found, while sludge pH demonstrated significant negative correlations with concentrations of these metals.

Two sewage sludge samples collected from wastewater treatment plants in Brazil and one sludge sample produced from South Germany were studied in terms of heavy metals, polychlorinated dibenzo-p-dioxin and polychlorinated dibenzofuran (PCDD/F), and polychlorinated biphenyl (8). The concentrations of PCDD/F found in the samples were below specified legislation for final disposal or agricultural use in soils. On the other hand, PCB and heavy metal values exceeded this limit. A study conducted on the extraction for heavy metals Cu and Zn and for competing metals Ca and Fe (9). The main findings showed that Cu can be extracted for 60-70% and Zn for 90-100% by citric acid at pH 3-4. A study carried out using physicochemical methods for reducing the heavy metal content of waste activated sludge (10). Acid thermal hydrolysis effectively reduced the concentrations of most heavy metals, excepted Cu and Pb while alkaline thermal hydrolysis is most effective in solubilizing these two metals. Fenton peroxidation released Cd, Cu, Ni and Zn largely, but it cannot be used for a reduction of Pb and Hg.

In the year 2001, analysis of the sludge from STP in Oman, indicated that none of them could be used for application to land under current International Regulations (4). However, only some of them exceeded the maximum concentration according to Omani Regulations. It should be noted that metals content would be reduced in admixture with non-hazardous domestic wastes through composting processes and may therefore be suitable for land spreading (5). This sludge can be reused instead of current dumping practice, which may pose a great risk to the environment. However, the reuse of contaminated sludge as fertilizer may harm life by affecting the crops, which we consume. Furthermore, dumping of contaminated sludge to landfills might cause problems to the groundwater and hinder its suitability for different uses (11). Therefore, it is the responsibility of the

decision makers to set strategies and policies to control the pollution source to protect the environment and human health (12, 13).

Therefore, the objective of this study was to 1) to characterize industrial wastewater sludge collected from different wastewater treatment plants in Muscat, Sohar, and, Salalah, 2) to recommend alternative ways to reuse this sludge.

Materials and Methods

Study Sites

STPs covered under this study are classified as industrial according to the source of raw sewage and samples were collected from the following STPs in the Sultanate of Oman:

Industrial STPs

1. Rusayl Industrial Estate (RSL.IE) located in Muscat Governorate

2. Sohar Industrial Estate (SIE) located in Sohar Governorate

3. Raysut Industrial Estate (RIE) located in Salalah Governorate

Table 1 shows information for industrial STPs. Following three types of samples was collected for characterization:

1. Retained/recycled Activated Sludge (RAS): all STPs

2. Waste Activated Sludge (WAS): all STPs

3. Primary Sludge: only from SLL.STP

Table 1: Basic information on studied industrial sewage treatment plants (STPs), Oman

STP	Capacity	Treated effluent (m ³ /day)	Sludge (ton/month)	Sludge conditioning
RSL.IE	1200	800	3.6	Drying Beds
RIE	470	125	0.58	Drying Beds
SIE	700	300	0.7	Drying Beds

Sampling and Analysis

The samples were collected over a period of one year approximately (monthly manner in the year 2010), through grab sampling method, and the collected samples were kept inside polythene bags until reaching the lab. Samples were first dried by utilizing sun heat and dried each sample was grinded to increase the surface area to have better extraction. The collected sludge samples were analyzed in the Environmental Engineering Laboratory at Sultan Qaboos University. All tests were conducted according to the Standard Method for the Examination of Water and Wastewater (14).

Parameters studied are electrical conductivity (EC), potential of hydrogen (pH), cations, anions and volatile content. The obtained values of RAS (retained / recycled activated sludge) and WAS (waste activated sludge) samples are presented using box plots which show the maximum, minimum, median, first quartile, and third quartile of the results.

Method followed for determining pH and EC

Electrometric determination of pH involves measuring the electromotive force (EMF) of a cell

comprising of an electrode, responsive to hydrogen ions such as a glass electrode, and a reference electrode. The EMF of this cell is measured using a high impedance electrometer calibrated in terms of pH. Approximately a mass of 1 g of each powdered samples were weighed and 100 ml-distilled water was added. Then, samples were put in Ultrasonic instrument for 15 minutes to atomization particles by vibration because the sound is faster in water and it can separate the particles fast. After that, the samples were taken out of the Ultrasonic instrument. The samples were then filtered, and pH tests were conducted using the pH meters and EC tests were conducted by using EC meter.

Methods followed for analysis of Anions

Anions were analyzed using a Metrohm Professional Compact Ion Chromatography (IC) system 881 with conductivity detector and packed bed suppressor unit and Metrohm 858 Professional Sample Processor. After same procedure of sludge digestion mentioned in measuring pH the anions were measured.

Methods followed for analysis of Cations

Approximately a mass of 0.5 g of each sample was taken. Then, nitric acid of 5 ml and hydrochloric acid of 15 ml were added to each sample, digested using classical wet digestion methods. During the processes of digestion, the containers were cooled by distilled water for half an hour until the contents transferred to 100 ml capacity bottles. Finally, they were put in the laboratory at the room temperature for a whole day before the filtration. After the filtration, the samples were put in closed plastic containers and analyzed by an Inductively Coupled Plasma (ICP) spectrometer at the Soil and Water Laboratory, College of Agricultural and Marine Sciences at Sultan Qaboos University.

Methods followed for analysis of Volatile Content

First, the solid sludge samples were dried in the sun in small containers for three days to a week. Then, the samples were grinded into powder or fine materials. After that, the weight of empty containers and samples weight was considered to have in total approximately 4 g (the weight of samples with containers should not exceed 4 g). Next, the containers were kept in the oven at a temperature of 550 °C for fifteen to thirty minutes to remove some of organic contents and to simplify digesting process. After the ashing process, the samples were weighed again to find out the

weight lost to calculate volatile content. The volatile content of the sludge is an indication of the organic content of the sludge.

Results

pH and EC

The values of pH for RAS samples were found in the range of 5.8 to 8.0. This wide range appeared in RSL.IE samples (Fig. 1).

The range of pH values of WAS being found similar to the RAS samples with the overall average around 6.7. Since methanogenic bacteria are extremely sensitive to pH with an optimum range of 6.5 to 7.2 (15, 16) while the fermentative microorganisms are somewhat less sensitive and can function in a wider range of pH of 4.0 to 8.5 (17) in aerobic digestion, it would be advisable to use fermentative microorganisms for treating this sludge in aerobic digestion. By checking the relevant standard deviation (STD) values in both RAS and WAS samples (0.6 and 0.5, respectively), it appears that the seasonal change does not have significant effects on this parameter due to the narrow ranges observed.

Figure 2 shows EC values of RAS samples from different industrial STPs. The highest value detected was in RIE.

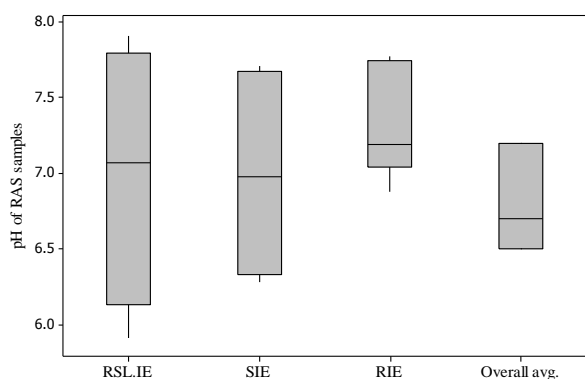


Fig. 1: Results of pH for samples of RAS

The differences between maximum and minimum values for WAS samples were relatively not very high compared to RAS samples. Most of the results were below 1000 $\mu\text{S}/\text{cm}$ except that for RIE.

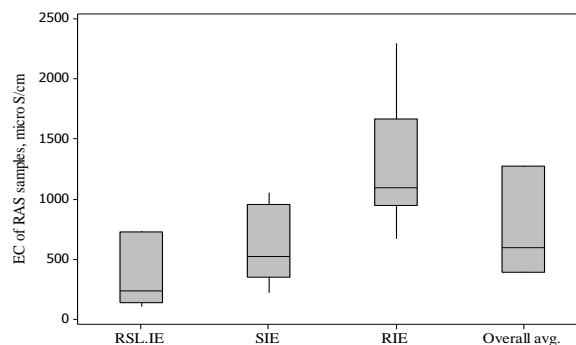


Fig. 2: Results of EC for samples of RAS

Therefore, the values of anions and cations will be relatively small. The values of STD for both RAS (388 $\mu\text{S}/\text{cm}$) and WAS (254 $\mu\text{S}/\text{cm}$) being high showing that the seasonal change has significant

effects. The general average for RAS and WAS samples were 755 $\mu\text{S}/\text{cm}$ and 433 $\mu\text{S}/\text{cm}$, respectively.

Anions

Tested anions are Fluoride, Chloride, Nitrite, Bromide, Nitrate, Phosphate, and Sulfate and most of the STDs for these samples (9, 3397, 1160, 73, 5848, 6822, and 6012 mg/kg, respectively) were particularly high which might be due to the effect of seasonal change. Fluoride concentrations were the lowest compared with the other anions. The average values of fluoride concentration of RAS and WAS samples was found about 14 mg/kg. The chloride concentration in RIE STP showed wider range than the other STPs with a maximum concentration of 51891 mg/kg for RAS samples. The overall average chloride for all STPs was around 12610 mg/kg for RAS samples (Fig. 3) while the overall average of WAS sample was 2923 mg/kg.

There is an outlier point in RAS samples of SIE, and there are outlier points in WAS samples for SIE and RIE which might be associated to some of the experimental errors or operational problems in the treatment plant.

The range of nitrite concentration in SIE was wider than the other STPs for RAS samples (Fig. 4) which may be due to accumulation of nitrite in settling tank. The overall average for all STPs was 51 mg/kg for RAS samples while the overall average of WAS sample was 586 mg/kg.

Routinely, the differences in WAS values were small in most STPs except for RSLIE in which the maximum value was too far from the median. There was an outlier point in WAS samples in RIE. Overall average of Nitrate of RAS and WAS samples were 1525 mg/kg and 2986 mg/kg, respectively. The ranges of RAS and WAS samples were narrow except for RAS samples in RIE (Fig. 5 and 6, respectively).

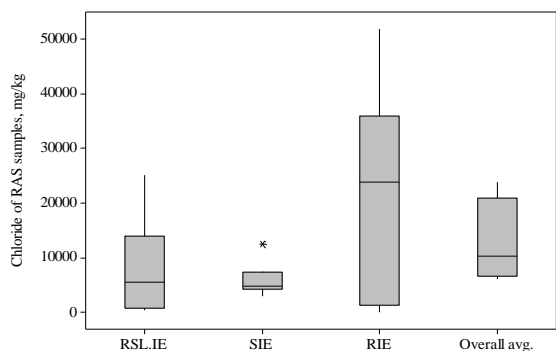


Fig. 3: Results of chloride values in samples of RAS (* denote extreme value/outlier)

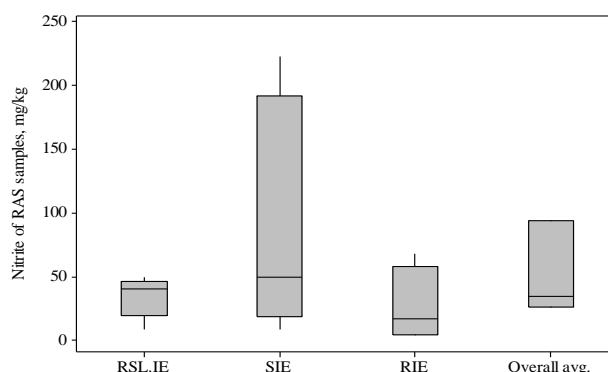


Fig. 4: Results of Nitrite values in samples of RAS

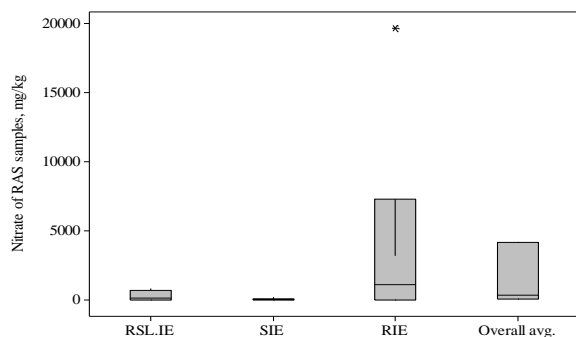


Fig. 5: Results of Nitrate values in samples of RAS (* denote extreme value/outlier)

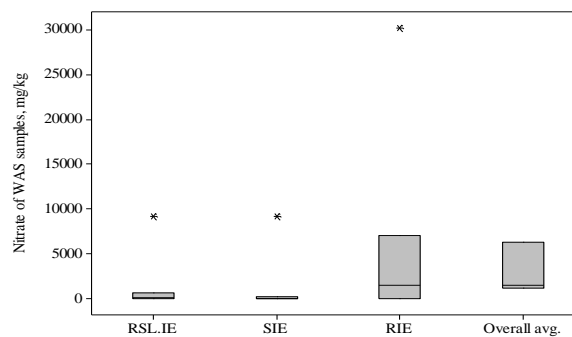


Fig. 6: Results of Nitrate values in samples of WAS (* denote extreme value/outlier)

The ranges of bromide in RAS samples were narrow with outlier points in each STP (Fig.7).

The STD of RAS in RIE (42 mg/kg) was relatively high. The differences in WAS value in RIE was small compared to the other two STPs in which the maximum value was too far from the median. All STDs were large in WAS samples (23 to 126 mg/kg) which reflect the wide change of bromide concentrations in each season. The overall average for all STPs was 88 mg/kg for RAS samples while the overall average of WAS sample was 61 mg/kg.

The range of phosphate concentration 19 to 18250 mg/kg, in SIE was wider than the other STPs for RAS samples (Fig. 8).

On the other hand, the range in WAS samples was wider in RSL,IE (60 to 44399 mg/kg). The overall mean for all STPs was 2218 mg/kg for RAS samples while the overall average of WAS samples were 3921 mg/kg. All STDs were very high in both RAS and WAS (3620 and 6822 mg/kg, re-

spectively) which means that the change in each season can appear clearly.

The range of overall average of sulfate concentration was from 1684 to 16681 mg/kg in RAS samples (Fig. 9).

The range of sulfate concentration in WAS samples was from 2656 to 5233 mg/kg which was narrower than RAS. The overall average for all STPs was 7412 mg/kg for RAS samples while the overall average of WAS sample was 5158 mg/kg, which is relatively high compared to the other anions. This might be due to the values of sulfate in raw sewage, which were high. Furthermore, all STDs were very high in both RAS and WAS (7214 and 6012 mg/kg, respectively) with outlier points in RAS and WAS samples in RIE.

Cations

The average obtained values for cations of different industrial STPs for RAS and WAS has been given in Tables 2 and 3.

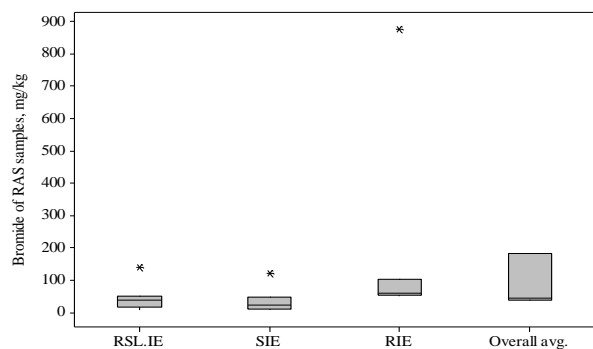


Fig. 7: Results of Bromide values in samples of RAS (* denote extreme value/outlier)

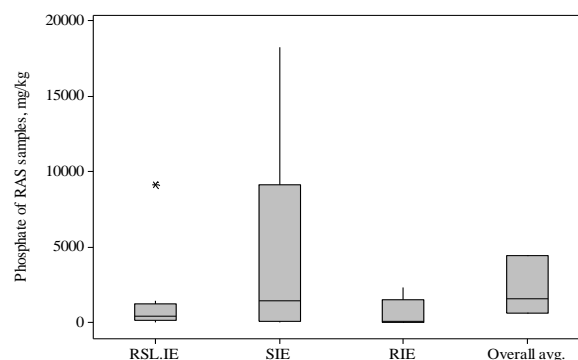


Fig. 8: Results of Phosphate values in samples of RAS (* denote extreme value/outlier)

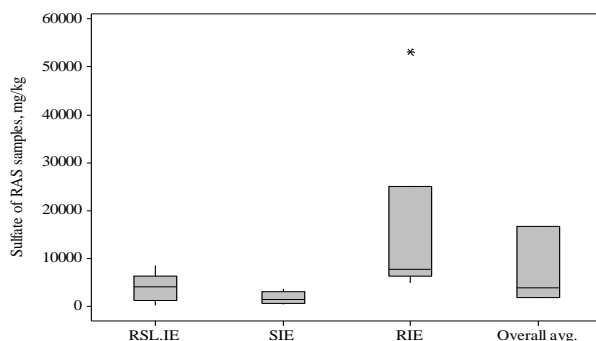


Fig. 9: Results of Sulfate values in samples of RAS (* denote extreme value/outlier)

Table 2: Average obtained value of cations (mg/kg) for RAS samples

Sample	Cd	Cr	Cu	Pb	Mo	Ni	Zn
RSL.IE	73	124	205	73	11	54	169
SIE	ND	158	144	11	19	101	277
RIE	4	136	99	39	8	34	2800
Overall average	38	139	149	41	13	63	1082
STD	23.1	23.6	28.1	17.5	5.1	14.4	182.4

Table 3: Average obtained value of cations (mg/kg) for WAS samples

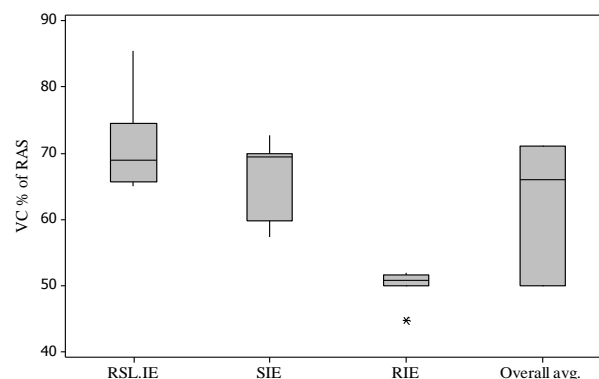
Sample	Cd	Cr	Cu	Pb	Mo	Ni	Zn
RSL.IE	64.5	77.9	226.9	64.5	6.1	44.7	73.8
SIE	ND	126.0	123.0	15.6	17.2	153.6	251.2
RIE	2.1	51.1	50.5	10.4	10.4	24.8	1791.0
Overall average	33.3	85.0	133.5	30.2	11.2	74.4	705.3
STD	5.1	54.9	129.4	7.0	3.4	14.5	275.2

These cations are: Cadmium (Cd), Chromium (Cr), Copper (Cu), Lead (Pb), Molybdenum (Mo), Nickel (Ni) and Zinc (Zn). The average obtained values of the cations in both RAS and WAS samples were found within the Omani standard for the reuse of sludge in agriculture except for Cd in RSL.IE (Tables 2 and 3). The value of Cd in SIE was very small (it was recorded as Not Detected (ND)). The effect of seasonal change appeared in Cr, Cu, and Zn in both RAS and WAS and Cd in RAS samples because STD was relatively high (Tables 2 and 3).

Volatile Content (VC)

The VC in RAS samples ranged from 44 to 86% (Fig. 10) compared to a range of 41 to 77% for WAS samples.

These ranges are wider than the range of secondary sludge which is 50 to 70% taken from a previous study (18). According to the statistical analysis using MiniTab software, outlier points were found for RIE RAS, and SIE WAS results indicated possible errors during the sampling or measurement phases. The overall averages of RAS and WAS being 62 % and 61%, respectively. The STDs were relatively low in both RAS and WAS (5 and 6%, respectively).

**Fig. 10:** Results of VC values in samples of RAS (* denote extreme value/outlier)

Discussion

A wide range of pH values observed in RSL.IE samples and it would be advisable to use fermentative microorganisms for treating this sludge in aerobic digestion. The highest value of EC detected was in RIE sample and indicated that the values of anions and cations will be relatively small. The values of chloride were very high especially for the RAS in RIE and this may be due to high concentration of chloride in raw sewage or because of supplementing chloride in the pre-aeration tank, which might have caused accumulation

in the settling tank. For nitrite, there is one outlier point in RAS samples in RIE, and also one outlier point in each STP for WAS samples. This might be related to the seasonal change effect. The relative high nitrate content could be due to aeration process in which most of ammonia (NH_3) was converted to nitrite and nitrate. Phosphate concentration dissimilarities observed in RAS values were very big in SIE and in RSL.IE for WAS samples in which the maximum value is too far from the median. There were outlier points in RAS samples in RSL.IE and in WAS samples in SIE. The comparison of cation results to the typical values for metals in biosolids of United States, all concentration were within the typical range except Zn in RIE in both RAS and WERE samples.

Conclusion

This study aim was to characterize industrial wastewater sludge in Oman from three different regions (Muscat, Sohar and Salalah) and to suggest alternate ways to reuse this sludge. The results showed higher concentrations of nitrate in all samples. Moreover, the values of chloride, sulfate and phosphate were higher than the other anions. The average values of the cations in recycled activated sludge (RAS) and wasted activated sludge (WAS) samples were within the Omani Standards, suitable for the reuse of sludge in agriculture except for Cd in RSL.IE. However, it is suggested that a regular maintenance should be performed at the studied sewage treatment plants to prevent any accumulation of some harmful substances, which may affect the sludge quality, and the sludge drying beds should be large enough to handle the produced sludge for better management.

Recommendations

Based on the results of this study, the following recommendations can be considered;

- Chloride sources and content might need further investigation as the values of chloride were found particularly high at some sewage treatment plants.

- Fermentative microorganisms are recommended for treating the sludge in aerobic digestion due to the suitable pH range observed for such process.
- Composting the produced sludge rather than dumping is highly recommended for the industrial STPs because the values of heavy metals were small. Furthermore, a pilot study has to be conducted to make sure that there will be no harmful effects to the environment if this sludge is used as a fertilizer.
- If the only way to get rid of this sludge is through dumping sites, the weight and volume of the produced sludge has to be reduced through proper processes to reduce the land use and associated aesthetic effects.
- This study also recommends further research investigations to provide the needed analysis to recommend other ways to reuse this sludge for energy in the terms of biogas such as methane etc.

Ethical considerations

Ethical issues (Including plagiarism, informed consent, misconduct, data fabrication and/or falsification, double publication and/or submission, redundancy, etc.) have been completely observed by the authors.

Acknowledgement

The authors would like to acknowledge the financial support received from Sultan Qaboos University through His Majesty Strategic Research Grant number SR/ENG/CAED/08/01. The authors declare that there is no conflict of interests.

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