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Estimation of some heavy metals and nutrients in dry sludge collected from Hammdan wastewater treatment plant in Basrah city-Iraq

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Abstract

The present study deals with the estimation of selected heavy metals (Cd, Cu, Ni, Pb and Zn) in dry sewage sludge collected seasonally during the period extended from summer 2015 until spring 2016 from Hammdan wastewater treatment plant(WWTP) in Basrah city . Flame atomic absorption (FAAS) was employed for measuring the mentioned metals . pH and available Nitrogen, Phosphorus and Potassium (N, P, K) in sludge were measured . Sludge texture as percentage and organic matter (OM) content as mg/kg have been determined to evaluate the presence of N, P, and K, OM and heavy metals in dry sludge samples .

The results showed that the range values of pH was from 6.6 to 7.7, while the range values of OM were from 12500 to 1800 mg/kg, also the rang values of N, P and K were (4180 – 5130), (55 - 68) and from (247 – 402.33) mg/kg respectively .

The concentration of heavy metals reveled the following order, Zn > Cu > Ni > Pb > Cd . Sludge texture description was silt clay . It concluded from this study that the sewage sludge from Hammdan WWTP, rich in the N, P and K also have different levels of heavy metals under the sludge, but these levels were in acceptable range for use this sludge in amended of agricultural soil according to (USEPA, 1983) .

Key words : Sewage sludge – Heavy metals – Nutrients – Hammdan wastewater treatment plant – Basrah .

Introduction

Sludge is an essential by product of wastewater processes, and the main sources of the sludge are sewage water treatment plants, (Wolejko, *et. al*., 2015) . It is mixture of water, inorganic and organic substances removed from wastewater coming from various sources, (domestic, sewage and industries), storm water ran-off from roads and other paved area through physical biological and/or chemical treatment (Janez, *et. al*., 2000) . Sludge contain toxic substances (heavy metals, pesticides) and pathogenic organisms (bacteria and parasites), these materials may make the sludge unsuitable for agricultural use, (Wilson and Jacobs, 1998 ; Watanabe, *et. al*., 2002) .

Heavy metals (HM) are elements which have atomic number more than 20, or have specific density more than 5 gm/cm^3 (Giadh and Al-khafaji, 2016) . Metals such as Cd, Pb and Hg they have no biological and they are toxic, even they exist in low concentration (Al-khafaji, 1996) . However the high level of HM in sludge frequently prevents reuse of its in agriculture application, so the removal of HM from the sludge before it is used in agriculture, it is safe step in keeping of public health (Indra and Sivaji, 2006), However it has been shown that wastewater sludge application at recommended rates increased microbial activity in soil and tied up the heavy metals making them unavailable to plant and soil (Sastre, *et. al*. 1996) .

Many studies have been done about the sludge outside Iraq, such as studding the characterization of sludge (Jia-Yin, *et. al*., 2006), using sludge in order to improve of mechanical properties of concretes (Barrera-Diaz, *et. al*., 2011), and use sludge as resource for sustainable agricuslture (Usman, *et. al*., 2012) . Because of the limited and scanty information about the sludge and all the previous aspects about it in Basrah city, the present study aimed to determine the concentration of selected HM, (Cd, Cu, Ni, Pb and Zn) in addition of N, P and K and OM in the sludge collected from Hammdan WWTP in Basrah city, to potentially evaluate using it in agricultural applications in order to sustain soil quality and agricultural productivity .

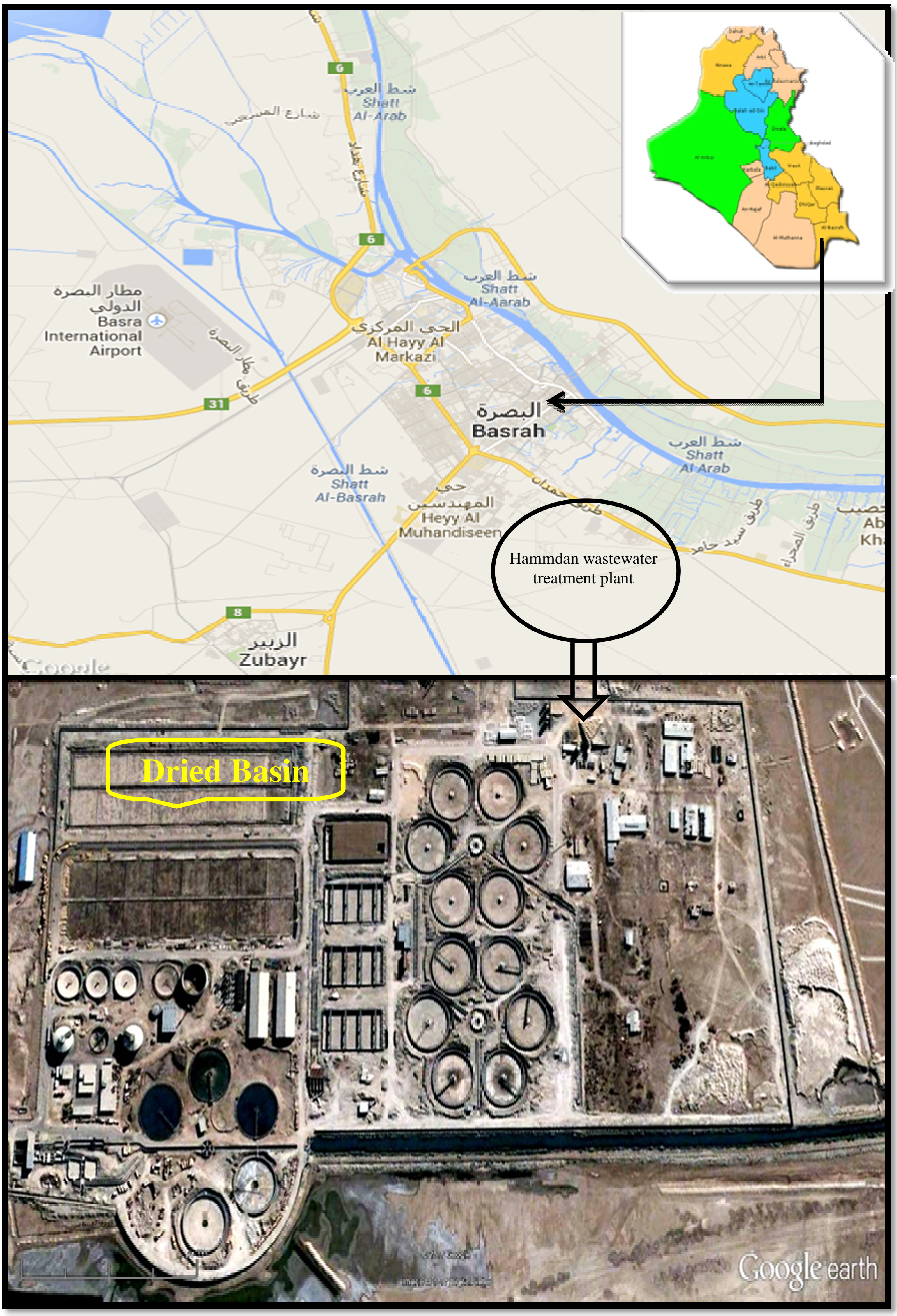

Figure 1. Sample station in Hammdan wastewater treatment plant – Basrah . Map from (Google Earth Pro.)

Material and Methods

Samples of dry sludge were collected seasonally from Summer 2015 up to Spring 2016 from the dried basin in Hammdan WWTP in Basrah city Fig.1 sterilized containers were used in sample collection with three replication by using plastic scoop. Samples containers were kept in refrigerator at 4°c to prevent any change in sludge condition (Rahmani, *et. al.*, 2015) . The extraction of HMs was done according to Yi, *et. al.*, (2007) by using 2gm of dry sludge, ground and sieved by <63 µm sieve, mixture of acids HNO_3 : $HCLO_4$ 1:1 was add to the sample for 60 minutes, then heated at 45°c for 60 minutes, beyond that cooling, then filtered by using 0.45µm filter paper, the filtered keep in 25 ml volume plastic vial and the volume was complete to 25 ml by deionized distilled water, and it become ready to measurement . Triplicates were used, and flam atomic absorption spectrophotometer type phoenix – 986 proved with hollow cathode lamps was employed for measuring HMs concentrations . Some parameters of sludge such as pH, N, P, K and OM content were determined according to standard methods (APHA, 2005 ; Mishra and Mahanty, 2012) . Sludge grain size was calculated according to a well tested method (Day, 1965) .

This study was used to analyze the variance (ANOVA), F-test, mean, standard deviation and correlation coefficient to find the significant among the parameters by the statistical system (SPSS – 10) .

Results and Discussion

Estimation of HMs concentration in dry sludge is a good indication of the situation of the sludge as amendment of the agricultural soil . The concentration of HMs expressed on dry weight basis in the sludge of the present study are given in Table 1 .

Table 1. Mean Concentration mg/kg dry weight ±SD and range of heavy metals in sewage sludge during the study period.

Metal / Seasons	Cd	Cu	Ni	Pb	Zn
Summer 2015	1.83± 0.05 (1.5 – 2.2)	220.66 ±9.5 (156 – 280)	96 ± 5.51 (54.3–0.2)	73.33 ±4.51 (69 – 78)	1272±80.11 (1050–321)
Autumn 2015	2.4 ± 0.4 (2.7 – 2.9)	211.66±5.9 (180 – 265)	83.5 ± 6.1 (44.1–8.6)	63 ± 4.36 (58 – 66)	1455 ± 81.3 (1378–540)
Winter 2016	2.5 ± 0.26 (1.9 – 2.8)	470.40±11.5 (495 – 562)	70.5 ± 4.3 (38.5–5.4)	72 ± 4.36 (69 - 77)	1265 ± 6.81 (1090-1320)
Spring 2016	2.2 ± 0.03 (1.8 – 2.9)	350.33±22.72 (295 – 411)	98.22 ± 9.2 (126 –8.2)	65.55±6.81 (58 – 71)	1196.33±19.7 (1031 –1255)
Annual mean	2.23	312.75	87.06	68.48	1297

SD: Standard deviation

Cadmium

Concentration of the Cd in the dry sludge was detected in the range of 1.83 to 2.5 mg/kg . Its annual mean was 2.23 mg/kg . The higher concentration was recorded in winter season, Cd clearly indicate is absorbed by the soil particles, and this in agreement with the result that found by (Kudakwashe and Janlaulani, 2014) of Cd in winter season related with higher content of OM 18200 mg/kg in the mentioned season Fig.2 . Significant correlation r=0.832 p≤ 0.05 was observed between Cd concentration and OM during the study period . Also, a significant correlation r=0.643 and r=0.735 p≤ 0.05 between Cd concentration and clay and silt respectively. This maybe due to the line grain size of silt and clay pocess large surface area lead to accumulate high concentration of Cd this result is agreement with that found by (Al-Khafaji, 1996) .

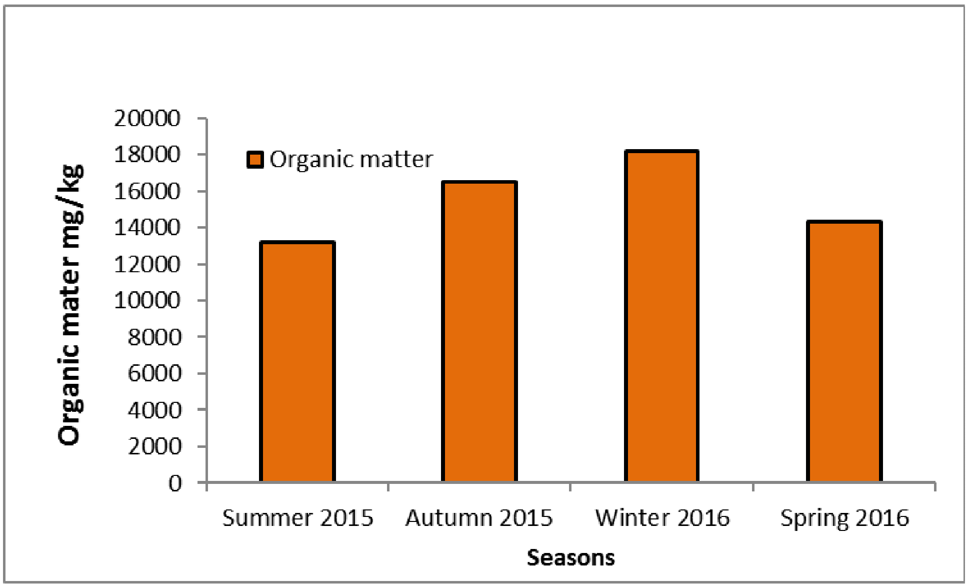

Fig 2. Organic matter content mg/kg in dry sludge during the study period.

Copper

The concentration of Cu was in the range of 311.66 to 270 mg/kg with annual mean 242.67 mg/kg, higher concentration of Cu was observed in winter, the high concentration of cu in the sludge is due to the adsorption of Cu containing matrices in the sludge particles, also may be due to the texture of the sludge particular percentage of silt and clay in winter season (Fig. 3) . Positive correlation r=0.527 ($p \leq 0.05$) was observed between Cu content and OM, also significant correlation r=0.821 and r=0.759 ($p \leq 0.05$) between Cu concentration and clay and silt respectively during the study period . This mean that fine grain size and OM in sludge attract the metal to concentrated in the sludge, this result is in agreement with the result that is found by (Al-Awady, 2012 ; Al-Enazi, 2014) respectively . Also, the high concentration of Cu could have been caused probably by the corrosion of the water supplies pipes, whose composition includes copper . The present study was compared to the findings of Marrison, *et. al*., (2004) in which they found Cu values in the range of 245 to 441 mg/kg . Another explanation for the high level of Cu in sludge, was due to the use of brass contains Cu and Zn, and copper scrubbers as abrasives in general cleaning in household lichens and the washing of pots .

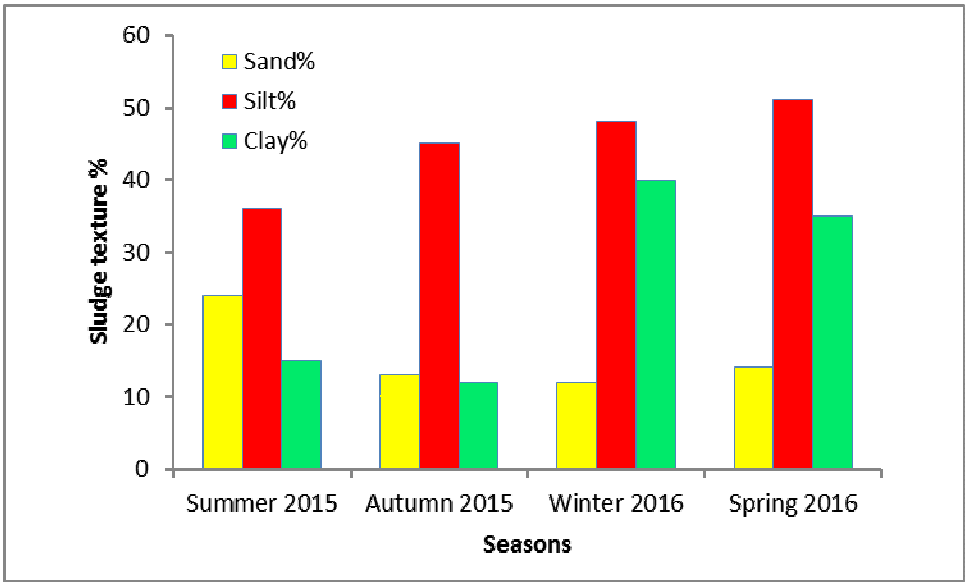

Fig 3. Percentage of Sludge texture during the study period.

Nickel

Ni showed fluctuation in its concentration during the study period, its range was from 70.5 to 98.22 mg/kg, with annual mean 87.06 mg/kg (Table 1), higher concentration 98.22 mg/kg of Ni was recorded in spring season, this may be due to the quantities and qualitative of sewage water which entered Hammdan WWTP in this season, also the high concentration of Ni in this study was in agreement with the result found by (Jia – Yin, *et. al*., 2006)

. Ni is one among many substances that enter in the composition of crud oil and its derivative, which highly using in many purpose in house hold, and finally discharged with wastewater (Giadh and Al-khafaji, 2016) .

Lead

Lead concentration was in the range 65.55 to 73.33 mg/kg, with annual mean 68.48 mg/kg, but the annual mean was less than the total maximum threshold and maximum permissible level of Pb in sludge . Table 2. significant correlation r=0.954 ($p \leq 0.05$) was observed between Pb concentration and OM content in sludge, also significant correlation r=0.55 and r=0.72, ($p \leq 0.05$) between Pb concentration with silt and clay percentage respectively . Sludge texture description was silt clay (Fig.3), also high OM content was observed in sludge of the present sludge during the study period Fig. 2 . Higher concentration of Pb was observed in summer season (Table 1), also the annual mean concentration of this metal was less than it concentration according to Guidelines (2008) .

Table 2. Comparison between the level of metal concentrations mg/kg dry weight in sludge from the present study with the other studies.

Country	Cd	Cu	Ni	Pb	Zn	Ref.
The European union number states	0.4 – 3.8 2.6	319 - 361 333	9 - 90 89	13 - 221 150	142 - 2000 1305	European commission 2001
India	2 – 9 3.6	- -	212 - 596 250	26 -154 88	- -	Dubey, *et al*. 2006
China (Shanghi)	17 – 81 65	1056-3873 2200	22 - 523 130	56.9 – 63.5 58	1685-15890 3705	Jia – yin, *et. al* 2006
Iraq Hammdan-Basrah	1.83 – 2.5 (*2.3)	2.11 - 470 (*312.72)	70.5–98.22 (*87.06)	63 –73.33 (*68.48)	1196 - 1455 (*1297)	Present study
Standard values	8.5	4300	420	840	7500	USEPA, 1983
Maximum permissible level	5	375	200	150	700	DWARF guidelines,2008

Mean values of heavy metals concentration in the dry sludge of the present study . *
Maximum values of heavy metals in dry sludge using in a agriculture lands*

A major source of Pb was probably due to the use of leaded petrol . The high concentration of Pb in the sludge of the present study was due to the located of Hammdan WWTP close of high traffic roads, and numbers of private electric generators from which Pb may have entered the storm water drains which one connected to the sewage plant, this result is in correspondence with the result that recorded by (Jia – Yin, *et. al*., 2006) .

Zinc

The concentration of Zn in the present study ranges from 1265 to 1455 mg/kg, with annual concentration 1297 mg/kg (Table 1) . The high concentration was in summer season and this may be due to the differences in amount and quality of sewage water that enter this plant during the study period . Also, this higher concentration maybe not only result from industrial and household source, but also from galvanized steel including that water distribution pipes, and the possibility is that it presence might be due to corrosion and Zn find its way to WWTP . Positive correlation r=0.453 ($p \leq 0.05$) observed between Zn concentration and OM during the study period, also significant correlation r=0.783 and r=0.821 $p \leq 0.05$ were recorded between Zn concentration with silt and clay in the sludge respectively .

Concentration of Zn in this study was higher than its concentration which was recoded by DWARF guidelines, (2008) Table 2., whereas it was lower than that reported by USEPA (1983), this depended on the nature of wastewater amount and quality which entered to WWTP from country to another .

The high concentration of Cd, Cu, Ni, Pb and Zn in the present study came mostly from domestic sewage effluent, which entered the Hmmdan WWTP . The presence of the mentiond metals in faces has been reported (Davis and Coker, 1980), also the quantity of HM in the study depend upon the type of domestic sewage and mixing of varieties of waste into its (Indra and Sivaji, 2006) . In Basrah city, many small scale private industries, and the effluents of these industries get mixed with the city sewage and flows through city drains to the Hammdan WWTP . This agreement with that found by (Shrivastave, 1995) . The concentration of all studied HMs in the sludge of the present study were less than the standard maximum levels of HMs which stipulated by USEPA (1983) in the sludge, using in amendment the agricultural soils (Table 2) .

pH, OM, Nutrients

pH values of the dry sludge in the present study were ranged from 7.1 to 7.7, higher value was recorded in Autumn season (Table 3) . The fluctuation of pH value during the seasons reflect the chemical, physical and biological reactions in the study components . The pH is a very important factor that it can affect crop production at land application sites by changing the pH of the soil and influencing the uptake of metals by soil and plants . Low pH (less than approximately pH 6.5) promotes leaching of heavy metals, which high pH sludge (greater than pH 11) kill many species of bacteria and conjunction with soil of neutral or high pH, and can inhibit the movement of HM in soil .

Table 3. Mean ±SD of pH value and major variable nutrients mg/kg in sewage sludge during the study period.

Parameter / Seasons	pH	N	p	K
Summer 2015	7.1± 0.06 (7.02 - 7.19)	4180 ± 26.5 (3060 – 4290)	65 ± 2.2 (52.1 – 70.3)	318.33 ± 9.7 (250 – 360)
Autumn 2015	7.7 ± 0.1 (7.6 – 7.81)	5120 ± 33.4 (4090- 5280)	68.2 ± 5.1 (59.2 ± 70.2)	289 ± 14.73 (280 – 306)
Winter 2016	6.6 ± 0.3 (6.3 – 9.6)	4580±30.54 (3900 – 4650)	55.8 ± 2.5 (47.1 – 60.5)	402.33 ± 17.79 (383 – 418)
Spring 2016	7.3 ± 0.12 (7.2 – 7.5)	5130 ± 60.1 (4880 – 5630)	65.44 ± 6.1 (59.11 – 70.4)	247 ± 29.55 (214 – 271)
Annual mean	7.17	4752	63.79	314.17

SD: Standard deviation

OM values were ranged from 12500 to 1800 mg/kg with annual mean 15175 mg/kg . Higher OM content n the sludge was recorded in winter Fig. 2, this may be due to the decline in degradation of OM by microorganisms, because the reducing of water temperature in the mentioned season (Giadh and Al-khafaji, 2016) referd that the high water temperature in Summer season will increase the microorganisms activity in degradation of OM in sewage water .

The high content of OM in the dry sludge is very important in agricultural applications . Usman, *et. al*., (2012) showed that the OM content of the sludge in urban sewage is generally high, usually more than 50% of dry matter . As mentioned, significant correlation was observed between OM and studied HMs, this relation lead to restriction of these metals by OM and inhibit their toxicity .

Usman, *et. al*., (2012) referd that the OM in sewage sludge reduces the negative environmental effects of HM and other pollutants by binding contaminants also OM in the sludge has important roles in soil properties, such as preserving water and nutrients and preventing soil degradation . Nutrient (N, P and K) content in the sludge in the present study showed in Table 3 . The range of the mentiond nutrients was from (4180 to 5130) (55.8 – 68.2) and (247 – 402.33) mg/kg respectively, which their annual means were 4752, 63.79 and 314.17 mg/kg for the mentiond nutrients respectively (Table 3) . Significant correlation r= 0.865 and r= 0.742 ($p \leq 0.05$) were observed between OM, N and P respectively, which negative correlation r= – 0.326 ($p \leq 0.05$) was observed between OM and K . The nutrients content in the sludge of the present sludge were (less than these were reported in Pakistan (Usman, *et. al*., 2012), they refers that the content of available N, P and K were 5200, 70, and 288 mg/kg respectively . USEPA (1983) showed that the sludge typically contain between (1 – 6 %) N, (0.8 – 6.1 %) P and from (1 – 5%) K respectively .

Conclusions

From the present study, it we conclude the following :

1- Dry sewage sludge consider main by – products in Hammdan WWTP in Basrah city .
2- The sludge is a rich source with nutrients (available N, P and K), it contain substantial amount .

3- High content of organic matter exist in this sludge .
4- The concentrations of HM in the sludge was in an acceptable range according to the standard values which stipulated by USEPA (1983).
5- Fluctuation in a level of HM, OM and nutrients during the study season reflect the quantity and quality of wastewater which enter to Hammdan WWTP in the mentioned season .
6- According to the comparison of the present results for HM, OM and nutrients with standerd levels which stipulated by USEPA (1983), the sludge of Hammdan WWTP can be used in amendment the agricultural soils in Basrah city .

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