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Benefits and Risks of Liquid Sewage Sludge Recycling in Agricultural Spreading – A Case Study of WWTP of Skhirat, Morocco

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ABSTRACT

The sewage sludge recycling as an agricultural land resource has received a great deal of attention worldwide. This practice has highly increased because of ever-increasing municipal wastewater production and the awareness of its fertilizing potential as amendment resources. However, there is a concern about land spreading linked mainly to health associated risks due to the presence of diverse pollutants. Thus, sewage spreading management is a key factor the guarantees benefits and avoids risks. The present work aimed to investigate the benefits and risks of sewage sludge (SS) application on agricultural land. To this end, physicochemical main parameters and bacteriological indicators, fecal coliform (FC) and fecal streptococcus (FS), of the sewage sludge generated form WWTP of Skhirat, Morocco, were performed during the period 2018–2019. The obtained results of physicochemical parameters reveal high concentration of organic matter in SS, which reach 96.3 mg/l, and in nutrients. Indeed, total Kjeldahl nitrogen TKN reaches a maximum of 3791 mg/l, potassium K⁺ reaches 58.71 mg/l. In addition, the average content of FC and FS are around 5.40 CFU/ml and 5.85 CFU/ml, respectively, whereas total phosphorus reaches 508.25 mg/l. In addition, concentrations of micronutrients such as Cl⁻, SO₄²⁻, Ca²⁺, Mg²⁺, and Na⁺ were high, which is interesting and could benefit for both soils and plant. Furthermore, this sewage sludge contains high concentration of heavy metals, mainly zinc and copper which could limit reuse in land spreading. The obtained results were compared to the applied standards and directives established within the framework of the agricultural spreading.

Keywords: sewage sludge, WWTP Skhirat-Morocco, liquid sludge, physicochemical parameters, bacteriological parameters, heavy metals, agricultural.

INTRODUCTION

Rapid demographic growth is leading to an increase in water consumption in the world and, consequently, an increase in wastewater production, as well as digested sewage sludge, which accounts for about 0.3 to 0.5% of treated wastewater [1], the sewage sludge is an unavoidable by-product of wastewater treatment, posing environmental problems due to the presence of

undesirable components, such as heavy metals, synthetic organic compounds and pathogens [2], the recycling of sludge as an agricultural resource has received increased attention worldwide. This is not only due to the problems of sludge disposal, but also to the unprecedented increase in the demand for water for agricultural production worldwide, the scarcity of fresh water, especially in arid and semi-arid regions [3]. In Europe, the Urban Wastewater Treatment Directive 91/271/ EEC (Council of the European Union, 1991) [4], encouraged the implementation of wastewater treatment plants (WWTP) with secondary wastewater treatment in municipalities with more than 2000 population equivalent (p.e.), and the Water Framework Directive (Council of the European Union, 2000) [5] encouraged wastewater treatment even in municipalities with less than 500 p.e. As a result, sludge production has increased by 50% in the European Union since 2005 [6]; however, safe disposal of sewage sludge is a major environmental concern. Applying sludge to land after proper treatment would increase the sustainability of agricultural production, as it recycles nutrients in the soil and makes them available to plants [7, 8, 9]. The use of municipal sludge on agricultural land is largely applied on the basis of agronomic rate, which is the agronomic N needed to meet the demands of a particular crop [10, 11]. The agronomic rate is determined by a variety of factors, including sludge characteristics, edaphic properties, and climatic factors [12, 13]. Given the population growth, human, industrial and agricultural consumption and the repeated drought that Morocco has experienced, several WWTPs have been set up, including that of the city of Skhirat, which has equipped a treatment plant by lagooning and activated sludge to manage the discharge of wastewater without polluting the receiving environment [14], and this, as part of the National Strategy for Sustainable Development [15]. The present work aimed to investigate the benefits and

risks of liquid sewage sludge (SS) application on agricultural spreading form the WWTP Skhirat in Morocco. The physicochemical and bacteriological characteristics as well as micropollutants of sewage sludge were determined. In view of their recycling in agricultural spreading also to study their environmental impact in a comparative way.

MATERIALS AND METHODS

Geographic location and Synoptic diagram of the WWTP – Skhirat

The Skhirat wastewater treatment plant, of the natural lagoon type with anaerobic digestion of sludge, is located about 20 km south-west of the city of Rabat, and covers an area of about 13 ha ($33^{\circ}49'39^{\circ}$ N, $7^{\circ}05'22^{\circ}$ E) (Fig. 1). The plant treats a nominal flow of 6000 m³/day, and a gross load in average of about 950 kg BOD₅/d (Fig. 2) which corresponds to the discharges of about 63,500 PE, and generates up to 1000 Kg of sludge a day [16].

Collection of the samples

In the present study, In the present study, In the present study, the samples of sewage sludge of "Liquid" nature (generated by the WWTP of Skhirat Morocco) were selected significantly at the level of the valves of each basin (V1, V2, V3, V4)



Figure 1. Geographic location map of the Wastewater Treatment Plant of the city Skhirat-Rabat Morocco)



Figure 2. Synoptic diagram of the WWTP - Skhirat-Rabat Morocco

(Fig. 3) during the following period: March 2018/ June 2018 and February 2019/May 2019. It is important to note that the samples were transported in a cooler to the National Laboratory of Studies and Surveillance of Pollution Rabat he Ministry of Energy Transition and Sustainable Development, Morocco (33°59'18° N 6°51'41° W) to be kept in the refrigerator at a temperature below 4 °C for a time not exceeding 2 hours in polypropylene containers according to the NF EN ISO 5667-13 standard. This enabled to follow the evolution of their physico-chemical and bacteriological characteristics according to time and note the changes in their state as well as make a decision on the possibility of their valorization and to choose the delicate die for this regard. The samples of sludge "Liquid" taken were the subject of analyses and characterizations and various parameters were raised.

Sewage sludge analysis

The measurements of the pH of the liquid sludge samples were performed according to the standards (NM ISO 10523 (2012)), the temperature (ISO 10523:2008), the electrical conductivity (NM ISO 7888 (2001)), are measured in-situ and carried out using a multi-parameter Type HACH LANGE/Model: Sension+ MM374. The content of organic and ammoniacal nitrogen (NKT) was determined after distillation and determination of ammonium by mineralization with an Auto-Kjeldahl Unit K-370 [17]. The inorganic nitrogen (NH₄-N and NO₃-N) was



Figure 3. Sampling point of sludge samples (V1, V2, V3, and V4)

determined by the Bremner method [17]. Total organic carbon (TOC) was determined by the dichromate oxidation method [18]. Phosphorus (P) was measured by NaHCO₃ - ascorbic acid method [19]. The organic matter (OM) content was measured by drying the samples at 105 °C until the sample masses were constant, followed by calcination at 550 °C for 4 hours in a muffle furnace [20]. The percentage of dry matter mass was calculated from the percentage weight after drying at 105 °C for 24 hours. The difference between the weight before and after drying expresses the water content of the initial sample according to (NF ISO 11465), The Chemical Oxygen Demand (COD) was determined by the classical standard method, using the oxidation by potassium dichromate $(K_2Cr_2O_7)$ in acid medium in the presence of silver sulfate and sulfate of Mercury and ammonium at the temperature of 150 °C (NF T90-101). The Biochemical Oxygen Demand (BOD₅) was measured by incubating at 20 °C for 5 days in a WTW thermostat spirometer for liquid sludge, according to ISO 5815-1986.

Heavy metals (Cu, Cd, Pb, Zn, Cr, Ba, Sn, Ni) and Major elements (Na⁺, K⁺, Ca²⁺, Mg²⁺)

The mineralization of the sludge liquid was realized according to the AFNOR standard N°31-151, 2 ml of sludge liquid was put in a Teflon tube, the samples contain organic matter which can cause interferences during the passage to the ICP, The following protocol enabled to eliminate them: 5 ml of 65% Nitric acid and 2 ml of 40% Hydrofluoric acid and 2 ml of Hydrogen peroxide were added with a micropipette, then the tubes were placed in a NOVA-8 type microwave at a temperature 180 °C for 40 min. The heavy metals and major elements were then determined by microwave plasma Atomic Emission Spectrometry: MP-AES 4210 Agilent at the Laboratoire National des Études et de Surveillance de la Pollution Rabat-Maroc.

Methods of microbiological analysis

For bacteriological analysis, the sampling of sludge was carried out in sterile bottles with a capacity of 0.5 l and brought back to the laboratory in a cooler and analyzed in the first 24 hours. The enumeration of the test bacteria of the fecal contamination, in particular the fecal coliforms (FC), fecal streptococci (FS), was carried out according to the techniques described in the standard ISO 4831.2006. The enumeration of FC was done by calculating the most probable number (MPN) after incubation at 37 °C in liquid medium for 24 hours [21]. For the research and counting of FS, the most probable number (MPN) technique was performed by inoculation of Rothe medium for 48 h. Thus, only the tubes positive to this last one were transferred in the medium of Litsky during 48 h according to the standard NF in ISO 7899 / T90-432 [22].

RESULTS AND DISCUSSION

The results of the physico-chemical analyses and the heavy metals, the major elements and bacteriology of the liquid sludge at the level of the valves of the digester of the wastewater treatment plants (WWTPs) located in Skhirat city of morocco are gathered in the following tables (Table 1).

Physical and chemical properties of liquid sludge

pH, Temperature, EC, and dry matter

The results obtained during this study revealed the pH values that varied between 7.5 and 7.9 between the spring and summer periods, respectively, with an average of about 7.73. The values higher than 7 were observed; however, no sample reached 8 (Fig. 4). These values are compatible with the studies carried out by Peterson [23], favorable to the development of bacteria that ensure the purification of sludge and promote the availability of nutrients to plants. This shows that the pH of the sludge is a critical factor to be taken into account because it influences the quality of the soil in the long term.

The Minimum and Maximum temperatures recorded during the study period (March 2018 – May 2019) are 19.4 °C and 24.1 °C, respectively. The temperatures recorded in the month of June are higher while that of the other month is corresponding to the summer period. It should be noted that the temperature is favorable to the microbial activity and mineralization of sludge. Indeed, to evaluate the characteristics of a treatment plant, the temperature is a very important parameter. Its variation as well as the pH has considerable

Month	March 2018	June 2018	February 2019	May 2019
Sewage Sludge	LS1	LS2	LS3	LS4
рН	7.72	7.91	7.5	7.61
Temperature °C	19.2	24.15	20.34	21.2
Electrical conductivity mS/cm	4.61	5.24	3.19	4.5
Dry matter %	4.29	7.44	3.62	5.75
Organic matter %	82.14	96.3	91.25	93.52
Mineral matter %	12.86	3.13	8.75	6.48
TOC mg/l	49.22	56.11	44.37	53.24
Total nitrogen Kjeldhal(TKN) (mgN/l)	987	3791	1201	1964
C/N	0.05	0.02	0.03	0.01
Phosphorus total (mg/l)	427.35	461.41	346.83	508.25
NO ₃ -N (mg/l)	1151.02	1607.85	921.4	1248.34
NO ₂ -N (mg/l)	<0.05	< 0.05	<0.05	<0.05
NH ₄ -N (mg/l)	10.81	19.44	12.2	14.38
Cl ⁻ (mg/l)	737.20	1107.97	2015.41	2486.6
SO ₄ ⁻² (mg/l)	13950	22530	18915	19860
COD (mg O ₂ /l)	31243	33212	29726	36127
$BOD_5 (mg O_2/I)$	479	510	487	564
Ca²+ (mg/l)	1217.07	2583.21	1787.42	2259.09
K⁺ (mg/l)	55.92	49.25	52.38	58.71
Na⁺ (mg/l)	37.5	41.9	47.74	52.03
Mg ²⁺ (mg/l)	103.37	99.68	100.54	105.2

Table 1. Physicochemical properties of liquid sewage sludge from of wastewater treatment plants skhirat morocco

effects on the behavior of the purifying bacteria of the environment. Thus, it was noted that in the warm period, the biological activity is more important than in winter, because it plays a major role on the kinetics of reactions.

The Electrical Conductivity (EC) of the different samples in June varied between 3.19 mS/ cm and 5.24 mS/cm (Fig. 4). It appears that this liquid sludge is mineralized with respect to fermentable organic pollutants in the June of 2018, thus, a too high salinity causes the clogging of the soil [24]. The DM content is low at the level of the four valves, it is between 3.62% to 7.44% which explains a very high moisture content 67.7% that depends on the time of sampling and the mode of stabilization [25].



Sulfates and chlorides (SO⁻, Cl⁻)

The chloride ions (Cl⁻) are highly mobile, negatively charged elements that migrate easily and are often considered as inert and conservative species, and their contents do not generally depend on the different phases of degradation of the waste [26]. The concentrations recorded at the levels of these sludges are high and ranging from 737.20 mg/l to 2486.6 mg/l during the summer period, so in these samples the concentration of sulfates (SO₄²⁻) is very high reaching an average of 18813.75 mg/l (Fig. 5), which is relatively very high compared to the values found by Zerrouqi et al [27]; this can be explained by the fact that sulfates (SO₄²⁻) are highly present in leachates [28].

Organic matter, ash and total organic carbon content

This study shows high contents of organic matter in the samples which vary from 82.14% to 96.33% (Fig. 6) during all the sampling periods, which could be explained by the composition of the wastewater that is rich in these elements. Thus, the high contents of organic matter make this sludge a better soil amendment, improving the fertility of degraded soils. In addition, its application leads to a substantial increase in soil organic carbon, and other necessary nutrients [29]. The inorganic fraction of this liquid sludge, such as ash content expressed as mineral matter (MM), the values of



Figure 5. The concentration of chloride and sulfates in liquid sludge during the period 2018-2019



Figure 6. Concentration of organic matter, ash and total organic carbon content

which are between 3.13% and 12.86%, is probably due to the high water content. Moreover, this liquid sludge has not yet undergone physical treatment at the drying beds. The total organic carbon (TOC) was also determined, and it was found that the values recorded were in the range of 44.37 mg/l and 56.11 mg/l (Fig. 6). These high levels of TOC in the month of June 2018 are related to the high content of organic matter. TOC is often considered a positive indicator of soil health [30], and the relative increase of TOC in the soil after organic amendments with varying stability could offer an additional benefit [31].

Charge organic – BOD, and COD

Regarding the organic pollution, the Biochemical Oxygen Demand (BOD₅), and the Chemical Oxygen Demand (COD), gave high levels during the summer period, which varied between 479 mg O_2/l and 564 mg O_2/l on the one hand and 29726 mg O_2/l and 36127 mg O_2/l on the other hand (Fig. 7). This could be explained by the increase in microbial activity and quantities of organic matter [32]. The elevation of this microbial activity will lead to the release of water-soluble organic molecules and the organic matter formed essentially of proteins, lipids and carbohydrates will undergo a degradation and transformation in aerobic or anaerobic environment by the action of microorganisms that develop there, mainly autotrophic or heterotrophic bacteria. These bacteria transform the biodegradable organic carbon pollution. The COD/BOD₅ ratio is high, which means that the sludge is mineralized and is no longer fermentable.

Kjeldahl nitrogen, inorganic nitrogen and C/N

Nitrogen is an essential soil nutrient to support agricultural productivity. When sludge is used, it is applied extensively according to the nitrogen needs of the plants [33]. The concentration of total Kjeldahl nitrogen and inorganic nitrogen (NH₄-N, NO₃-N and NO₂-N). In this study, the dominant fraction of nitrogen observed was NO3-N followed by Kjeldahl nitrogen and NH₄-N while NO₂-N was very low, this liquid sludge shows high values of nitrates, with an average of about 1248.34 g/l. However, a high content reaching 1607.85 mg/l was observed in the June of 2018 (Fig. 8); this denotes a good process of nitrification during dehydration of sludge in the summer period. The recorded contents of Kjeldahl nitrogen are between 987 mg/l and 3791 mg/l in the June of 2018. This can be attributed mainly to the mineralization of organic nitrogen contained in the sludge. Indeed, the ammonium NH₄-N released by this mineralization, and of the order of an average of 14.20 mg/l and oxidizes biologically into NO3⁻ with transient production of NO₂-N [34]. A high C/N ratio related to low nitrogen content (nutritional source) leads to a slowing down of the activity or growth of microbial populations. Conversely, a very low C/N ratio linked to an excess of nitrogen leads to a production of ammoniacal nitrogen characterized by a foul odor. In this study, the C/N ratio <20 is low, which varies between 0.01 and 0.05 (Fig 8: The content of total Kjeldahl nitrogen, inorganic nitrogen and ratio C/N), which indicates that these sludge liquids are rapidly biodegradable [35].



Figure 7. Variation of organic pollution (BOD, COD) of Liquid sludge in WWTP Skhirat)



Figure 8. The content of total Kjeldahl nitrogen, inorganic nitrogen and ratio C/N)

Macronutrient content

The application of sewage sludge on degraded agricultural soils could be considered as an alternative to fertilization [36] due to their high concentrations of essential nutrients [26]. However, application rates must be carefully calculated. The concentrations of macronutrients (Mg, Ca, Na, K and P) of these sludges are shown in Table 1, the calcium (Ca) content in the samples is high, which varies from 1217.07 mg/l and 2583.21 mg/l, respectively in the June of 2018 (Fig 9: concentrations of macronutrients (Mg, Ca, Na, K and P). Thus, the concentration of Magnesium (Mg) is independent of the type of sludge [37]. It amounts to an average of 102.19 mg/l in the March of 2018; Phosphorus (P) and potassium (K) vary according to the raw material treated, the content of K in this study varies between 49.25 mg/l and 58.71 mg/l. Therefore, the P content is between 346.83 mg/l and 508.25 mg/l in the summer period. Sodium (Na) is a very important parameter in soil amendment. The recorded contents varied between 37.5 mg/l and 52.03 mg/l (Fig. 9). In general, sludge is characterized by considerable variability in nutrient content, depending on the source of the wastewater and the treatment process [38]. The concentration of sewage sludge provides essential nutrients for plant growth.



Figure 9. Concentrations of macronutrients (Mg, Ca, Na, K and P)

Parameter	Present study of Liquid Sludge -WTPP Skhirat (Morocco)	Seleiman et al. (2013a)	Enrica et al (2012a)		
		Pure sewage sludge	Bassin 1	Bassin 2	Limit values
Zn (mg/kg)	309.01 ± 8%	470	874	1189	2500–4000
Pb (mg/kg)	50.68 ± 7%	20.0	57	72	750–1200
Ni (mg/kg)	7.00 ± 5 %	20.0	76	_	300–400
Cd (mg/kg)	<0.003 ± 5%	0.4	1	1	20–40
Sn (mg/kg)	2.76 ± 7%	-	-	-	NS
Cr (mg/kg)	9.51 ± 7%	30.0	-	-	NS
Cu (mg/kg)	93.32 ± 6%	270	305	440	1000–1750
Ba (mg/kg)	57.25 ± 8%	-	-	-	NS

Table 2. Heavy metal concentrations (mean \pm standard deviation) in the sludge of the WTPP-Skhirat Morocco and example of studies by Seleiman et al. (2013a) and Enrica et al. (2012a)

Note: Limit values according to the current European legislation (Council of the European Union, 1986) [39]; NS: The maximum limit is not specified by legislation.

Heavy metals and fecal bacteria

The heavy metal content in sludge is one of the main factors limiting the use of sludge as a soil amendment.. Thus, the risk of heavy metal pollution in soils has been well documented in the literature, highlighting sludge as a possible source [40]. However, the limits for Hg, Ni, Pb, Zn Cd, Cr, and Cu, have been established by European legislation (European Directive 86/278/EEC) [41]. Table 2 shows the concentrations of heavy metals in liquid sludge. The content of Zinc and Copper is higher, it is about (Zn – 309.01 mg/kg, Cu – 93.32 mg/kg), these values are lower than the concentration of sludge from Greve wastewater treatment plant (Denmark) [42], and the concentration published by Seleiman et al. (2013a) [43].

This can be explained by the fact that Cu and Zn are the most mobilized metals and are considered to be the most bioavailable and mobile of the cationic heavy metals. Thus, the concentration found in Ni was lower than the concentrations reported in Germany by Fijalkowski et al. (2017) [44]. The concentration of lead is in the order of (Pb - 50.68 mg/kg)although higher than that found by Seleiman et al. (2013a) in Egypt, and lower than the concentrations found by Enrica et al. (2012a) [42] at pond level. In turn, the content of Barium (Ba - 57.25 mg/kg) and Tin (Sn - 2.76 mg/kg), recorded is lower than the value reported by Sérgio Siqueira et al. (2021a) (Ba-178.95 mg/kg) [45]. The contents of chromium and cadmium in this present study are in the order of (Cr -9.51 mg/kg, Cd <0.003 mg/kg), these levels are higher than those found by Seleiman et al. (2013a) (Cr - 30.0 mg/kg, Cd - 0.4 mg/kg) [46], which can be explained by the fact that Cd and Cr are less mobilized metals. The values of heavy metals found in these sludge were ranked in the following order: Zn > Cu > Ba > Pb > Cr > Ni > Sn > Cd. In summary, according to this study, the concentrations of heavy metals are lower than the limits allowed by the European Union (EU). These sludges could be safely applied to the soil in accordance with current legislative recommendations. However, caution is advised and it is recommended to evaluate the longterm effects of soil amendment with these sludges.

Fecal coliforms and Streptococcus are bacteria present in the human digestive tract. The presence of fecal coliforms and streptococci in the environment usually indicates contamination of fecal origin (sludge, wastewater, ...). Their survival in the environment is generally equivalent to that of pathogenic bacteria such as E. coli, Enterobacter aerogenes, Citrobacter freundii, In this study, the average concentration of fecal coliforms and fecal streptococci in liquid sludge is higher than that found in dried sludge (Table 3), at the level of drying beds (average sludge aged more than one month of storage), which can be explained by the fact that this high content is due to the decrease in the amount of water (desiccation), the increase in temperatures and the presence of large amounts of organic matter, in addition, the storage of sludge in drying beds gradually decreases the bacterial load under the effect of light, leaching

Table 3. Bacteriological characteristics of raw sludge

Type of sludge	Faecal coliforms (FC) Faecal Streptococci (FS)	Concentration in ulog 10 or CFU/ml
Liquid sludge	FC/ml	5.40
	FS/ml	5.85
Dry sludge	FC/ml	2.30
	FS/ml	1.27

and antagonism between the germs (FINDLAY, 1972) [47]. It is necessary to investigate the research of other bacteriological analysis in the way of valorization of the sludge as soil amendment.

CONCLUSIONS

This study was interested in the characterization of liquid sludge in organic and mineral matter and bacteriological, of the wastewater treatment plant of the city Skhirat-Rabat Morocco which presents a variation of their physicochemical and bacteriological quality. During the period of study, the physicochemical and microbiological results of the sludge have provided vital data, fitting perfectly into the strengthening of knowledge on the complex nature of sludge for use as organic soil amendments as a very important strategy to conform to the Directive on the landfill of waste (Directive 1999/31/EC of the Council), the results obtained indicate a richness in nitrates and phosphorus and that it contains nutrients as part of the overall program of crop fertilization. The nutrients found in the sludge, which are important in the agriculture, are the nitrogen fertilizing value, the phosphorus and sometimes potassium and zinc. Other macro and micronutrients may also be present; the sludge is considered as nitrogen fertilizer. The concentrations of heavy metals: Cu, Pb, Cd, Cr, Ni, and Zn, are below the required standards. It is therefore recommended to develop a management plan based on regulatory standards and laws (top-down approach) and on partnership with non-traditional stakeholders (private sector and civil society) and collaborators (research and educational institutions). The "sludge-energy" approach using anaerobic digestion-based technology appears to be favorable with several advantages, including the high degree of sludge stabilization and the reduction of sludge quantity, as well as the conversion of organic matter into biogas and several value-added products, such as soil conditioners and fertilizers. This approach could be established in collaboration with the private sector to maximize the economic and environmental benefits.

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