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Impact of Sewage Sludge Leaching on Soil Constituents and Quality

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Activated sludge treatment plants generate large quantities of sludge each year, thereby posing a serious environmental problem. This study aims to experimentally assess the effect of rainwater on the leaching of sludge components. In this context, a percolation test was set up, and composed of PVC cylinders into which the solid substrate was introduced. Five modalities of the solid substrate were used: a sludge modality, a soil modality and three modalities with increasing percentage of sludge (1%, 5% and 25%) in the soil. The percolation water is collected during the rainy months in bottles placed below each column. Solid substrate samples were taken before the test and after one year. The physicochemical analysis of the percolation water showed an increase in the electrical conductivity, BOD₅, COD, nitrogen compounds and phosphate compounds which were proportional to the percentage of sludge. The pH of the sewage sludge leachates varies from 7.61 to 7.98. Zinc and copper were the most mobilized metals. A year following the installation of the percolation test, electrical conductivity, total phosphorus (TP) and orthophosphate (PO₄) contents decreased for the solid substrates using the five modalities. Furthermore, ammonium (NH₄) and nitrates (NO₃) levels decreased in soil mixed with 1 to 25% of sludge due to their leaching by rainwater. Collectively, these data show that the leachates through the soil mixed with sludge are stable and loaded with NO₃, a plant nutrient that can contaminate the groundwater as well as the surface waters inducing their eutrophication. Furthermore, addition of sludge to the soil improves the levels of carbon, total nitrogen, TP and PO₄ in the soil and thereby soil fertility. The addition of sludge, however, is not without soil contamination with heavy metals. Such soil contamination would cause pollution of surface and ground water. Reaching certain severity, it should call for the adoption of prompt measures for the protection of environment and human health.

Keywords: sludge, leachate, soil, heavy metals, nitrates, percolation.

Introduction

The expansion of urban regions is incessantly generating alarming amounts of sewage that require appropriate treatment. The plants destined for sewage treatment generate a high amount of liquid and highly fermented waste, known as sewage sludge. In 2014, the sewage sludge quantity was around 392.000 tonnes of wet sludge in Morocco and is estimated to reach 2 million tonnes of sludge at 25% dryness per year by the year 2030 (Netherlands Enterprise Agency, 2018). Such continuous accumulation of sludge would dramatically impact the environment (Fuentes et al., 2004; Dong et al., 2015). Therefore, various processes are used to dispose of the sludge: incineration, land filling or agricultural spreading. The sludge contains compounds such as carbon, nitrogen, phosphorus and potassium (Lambkin et al., 2004). Many researchers have reported a positive impact of using sewage sludge in agriculture (Wu et al., 2015; Kominko et al., 2017), reconditioning of degraded natural and anthropogenic soil (Martínez et al., 2003), control of erosion as well as slope stabilization (Sort and Alcañiz, 1999). However, the recycling of sewage sludge into agricultural land has serious risks for public health and environment. The sewage sludge contains high amounts of toxic metals (Cesar et al., 2012). These sludge constituents are subjected to leaching by rainwater (Vettorazzo et al., 2001; Lehmann et al., 2003), and thereby liable to contaminate soil, water, and produce (Keller et al., 2002; Lavado et al., 2007), which limit the use of sludge in agriculture (Chaudri et al., 2000; Mosquera-Losada et al., 2010).

The City of Nador, located in the northeastern region of Morocco, is equipped with wastewater treatment plants (WTP) using activated sludge process. The Nador's sludge is disposed in landfill as seen in other WTPs in Morocco (Arisily and Hajji, 2020). Sludge, generated from the Nador's WTP, can be used in farming as fertilizer in the plains of Bou-Areg and Gareb, located in the Moulouya basin in the south of Nador city. However, under these two plains there is a shallow aquifer, which extends continuously and covers an area of 570 km². Unlike that of the Gareb aquifer, the thickness of Bouareg aquifer varies from 5 to 60m only (Lyazidi et al., 2019), thereby increasing the risk of its pollution by sludge leachate.

The objective of this research is to determine the physicochemical characteristics of sewage leachates in comparison with soil leachate, and assess its impact on the soil. We will use experimental models of leaching sludge by rainwater to assess the transfer of nutrients and toxic elements from sewage sludge, generated by water treatment plants of Nador city (Morocco).

Methods

In order to provide more information about the importance of sludge leaching by rainwater, a percolation test was carried out. The experimental test setup consists of a polyvinyl chloride (PVC) column (microcosms), one meter high and an internal diameter of 10.5 cm, into which the solid substrate was introduced. Five modalities of the solid substrate were used: a sludge modality (100% S) used to understand the effects of sludge stocks on water resources, a control modality consisting solely of soil (0% sludge) and three modalities with increasing percentages of sludge (1%, 5% and 25%). The soil used for the experiment, comes from an agricultural plot that has never received sewage sludge or irrigated by sewage. This soil was collected at a depth of 0–40 cm, dried in the open air and sieved to 2 mm. The sludge used in the percolation test comes from a sludge stock of the Nador's WTP. The column of the solid substrate weighs 2.75 kg each and measures approximately 25 cm in height. The microcosms were placed in the open air for one year.

Below, a mesh of fabric holds the ground column while allowing a good drainage of the water. The percolation water was collected in bottles placed below each column in the same day. The percolates collected during five rainy months (January to May) were subsequently filtered and stored at 4°C to be analyzed the following day. The samples intended for the determination of heavy metals were acidified to pH 2 with H₂SO₄.

The leachate samples subjected to the percolation test underwent several analysis: pH, electrical conductivity (EC), salinity, ammonium NH₄ (NF T90-015), nitrates NO₃ (Rodier, 1984), orthophosphate (PO₄) and total phosphorus (TP) (NF T90-023), total Kjeldahl nitrogen

(TKN) (NF T90-110), chlorides (NF T90-0114), sulfates (SO_4) (Tardat-Henry and Beaudry, 1992) and chemical oxygen demand (COD) (NF T90-101). Biochemical oxygen demand (BOD_5) was determined after incubation at 20°C for five days. Water soluble heavy metals were analyzed after filtration using inductively coupled plasma emission spectrometry (ICP-AES). The results were generated from the analysis of leachate samples ($n = 8$), carried out during the five months of monitoring are expressed as average value. The sampling was carried out after rain. Table 1 shows the number of percolate samples taken per month. Eight samples in total were analyzed.

Table 1. Number of leachate samples

	January	February	March	April	May
Sample numbers	1	2	1	1	3

Substrate samples were taken from the 0–10 cm layer, tested at the time of collection and after one year using the five modalities in order to check the impact of rain leaching on nitrogen and phosphate levels. Afterwards, the samples were subjected to several physicochemical analysis. The pH measurement is carried out on a substrate-water mixture with a ratio of 1/5 (volume / volume). The mixture is stirred for 5 min and left to rest for 2 hours (ISO 10390). Electrical conductivity and salinity are measured on a substrate-water extract with a ratio of 1/5 (mass/volume) after stirring for 30 min (ISO 11265). The extraction of the mineral nitrogen is carried out with a 2M KCl solution (Keeney and Nelson, 1982). The forms of mineral nitrogen (NH_4 , NO_3 and NO_2) in extracts are analyzed by colorimetry assay according to the methods described for leachate. Total nitrogen is measured according to the Kjeldahl method (NF ISO 11261: 1995). Orthophosphate is extracted from solid substrates in distilled water with a ratio 1/10 substrate/water and stirred for 1 hour (Self-Davis et al., 2000). The assay is performed using the standard (NF T90-023). The total phosphorus is always determined using the NF T90-023 standard after mineralization of organic phosphorus into orthophosphate ions (Olsen and Somner, 1982). Total organic carbon (C) is measured by the Walkley and Black method (Gigliotti et al., 2001).

Heavy metals are analyzed by Inductively Coupled Plasma Atomic emission spectrometry (ICP- AES) ICP (after mineralization with aqua regia (HNO_3 and HCl with a ratio of 1:3) (EPA-ROC, 1994).

Results and Discussion

Characteristics of leachate sludge

pH, electrical conductivity and salinity

Generally, leachate is considered a high strength wastewater with high conductivity, rich in ammonium and organic matter. It can be described as a multi-component mixture of insoluble, soluble, inorganic, organic, non-ionic, ionic, bacteria and bacteriological substances in an aqueous solution. Since these components are water soluble, there is a high risk that they will leak in surface and groundwater (Dounavis et al., 2019).

Among the factors that affect the composition of the leachate is the pH. pH is known to influence the mobility of heavy metals in the environment (Bourg and Loch, 1995). However, its effect on the concentration of heavy metals in soil solution differs among metals. The analysis of the sludge leachate collected during the monitoring period is shown in Table 2. We found a slight variation in pH between the five modalities studied. The pH difference between soil versus soil mixed with sludges (at 1%, 5% and 25%) varies from 0.01 to 0.16 pH unit (Table 2).

The electrical conductivity (EC) was also measured. The results show increased EC as salinity tied to the sludge percentage increases from 2.38 mS/cm for the soil modality to 15.17 mS/cm for the sludge modality. Conductivity values are strongly related to the total salts content in leachate reflecting its total concentration of ionic substances. In fact, the elevation of EC and the salinity of sludge percolates can be explained by the importance of the leaching through the sludge of mineral salts such as sulphates, chlorides and nitrates.

Ca, Mg, Na, K, SO_4 , Cl

The biodegradation and mineralization of organic matter involves the production of nutrients through microbial communities (bacteria, fungi) and the release of assimilable elements (K, Ca, Mg, K, etc.). The most

leached anions are sulphates (SO_4). Sulphates are generally strongly present in leachates (Robinson and Lucas, 1985). We found that their concentration in sludge leachates was found to be 22 times higher than in soil leachate. Sewage sludge contains a significant concentration of sulphur. Various authors (Sommers et al., 1977; Gutenmann et al., 1994) report a total sulphur concentration in waste sludge between 0.3 and 2.3% on a dry solid basis. The most dominant inorganic sulphur containing molecules are sulphates and (metal-) sulphides. Moreover, some sulphur is incorporated in the organic material (Dewil et al., 2008). Due to their high mobility (Lehmann and Chroth, 2003), the sulphates would ultimately enrich the leachates: the fall of rain on sludges would lead to significant leaching of sulphates. Chlorides (Cl) are often considered as conservative and inert species (Christensen et al., 2001) and their contents do not generally depend on the different phases of waste degradation. Chloride ions are highly mobile elements. Negatively charged, they are not fixed by the clay-humic complex and migrate easily. These facts may explain our data as the concentration of chloride (Cl) was 1.5 times in sludge percolate higher than in soil percolate (Table 2).

We found that the most leached cation is calcium. Its concentration increased from 212.2 mg/L for leachate soil to 369.9 mg/L for sewage sludge percolate (Table 2).

In summary, the leached concentrations of the studied anions and cations were almost proportional to the

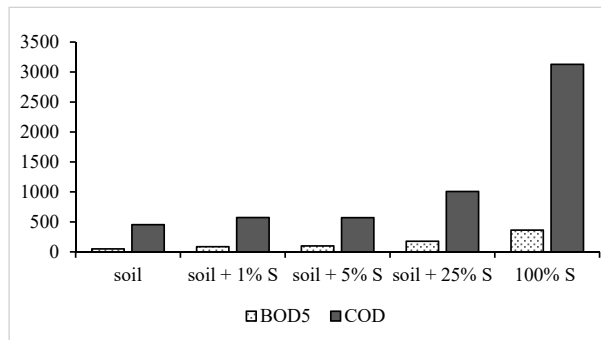
percentage of sludge in the solid substrates.

Organic matters are usually quantified as BOD_5 and COD (Lee and Nikraz, 2014). We found that the rate of sewage sludge positively correlates with BOD_5 and the COD (Fig. 1). Indeed, BOD_5 and COD increased from 50 mg/L to 364 mg/L and from 454.9 mg/L to 3129.6 mg/L for soil leachate and sewage sludge leachate, respectively. The increase in BOD_5 and COD of sludge leachate can be attributed to the increase in microbial activity and the amounts of leached organic matter after the rainfall. In fact, the activity of microorganisms is found to be stimulated by the addition of sludge (White et al., 1997). The increase of this microbial activity is likely responsible for the high concentration of water-soluble organic molecules, which could have been carried along by the percolation water.

The BOD_5/COD ratio can be considered as a measure of the biodegradability of the organic matter, and hence of the maturity of the leachate, which typically decreases with time (Quasim and Chiang, 1994). High BOD_5/COD ratios indicate elevated concentrations of biodegradable organic compounds, while low BOD_5/COD ratios indicate a resistance to biological degradation due to the predominance of non-biodegradable compounds (Bhalla et al., 2013). A BOD_5/COD ratio greater than 0.5 indicates an unstable leachate, whereas when the ratio is less than 0.1, the leachate can be considered mature (SWANA, 1997). For the five modalities studied, the value of BOD_5/COD ratio does not exceed 0.2 from which all the leachate collected is stable.

Table 2. Physicochemical characteristics of sewage leachate

	Soil	Soil + 1% S	Soil + 5% S	Soil + 25% S	100% S
pH	7.60	7.65	7.76	7.61	7.98
EC (mS/cm)	2.38	2.97	3.08	11.60	15.17
Sal. (g/L)	0.86	1.12	1.19	5.59	7.72
Ca (mg/L)	212.26	274.23	349.10	686.57	369.94
Mg (mg/L)	43.93	84.08	96.81	447.27	184.15
Na (mg/L)	58.75	67.75	46.50	240.90	587.50
K (mg/L)	27.37	15.69	26.60	238.30	486.66
SO_4 (mg/L)	169.22	243.37	527.75	1939.43	3837.65
Cl (mg/L)	232.19	200.40	174.01	315.70	390.38

Fig. 1. BOD_5 and COD of leachates (mg/L)

Nitrogen and phosphorus compounds

The nitrogen compounds (NO_3 , NH_4 , TKN)

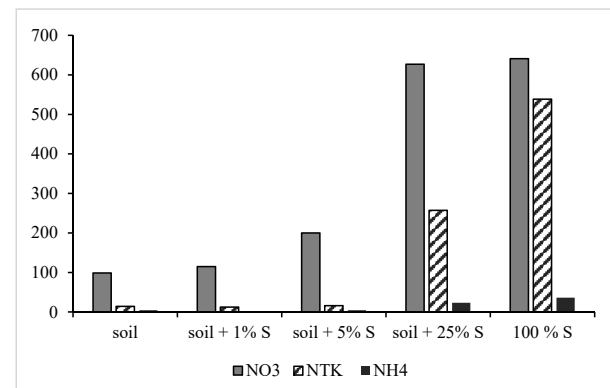
The concentrations of NO_3 , TKN and NH_4 in the percolates of sewage sludge were determined (Fig. 2). The nitrate concentration (max $NO_3 = 640.9$ mg/L) was higher than ammonium concentration (max $NH_4 = 36.2$ mg/L). Similarly, for nitrogenous compounds, their content in the percolation water increased in parallel with the increase in the sludge content in the solid substrates. The nitrate content in the percolate increased from 114.95 mg/L for soil +1% S to 627 mg/L for soil +25% S. Leaching sludges induced by rainwater enrich the percolate with nitrates, which could contaminate the groundwater, as well as the surface waters inducing their eutrophication.

The high concentrations of NO_3 found in the percolates of the soil + sludge mixtures and in the sludge modality can be attributed to i) the leaching of the NO_3 , which already exists in the solid substrates of the sludge, and ii) the mineralization of the organic nitrogen contained in the sludge. This later generates NO_3 via a biologic mediated oxidation of the released ammonium (NH_4) with a transient production of NO_2 , as reported by Laudelout (1990). Due to their negative charge, nitrates are not retained by soil clay colloids and can be easily washed away by rainwater and thus contaminate the natural environment, and, eventually, the ground waters (Atteia, 2015).

The organic content in the leachate (NTK) is also determined in this study. The sludge percolates have high content of NTK. This content was 539 mg/L in sewage sludge leachate, which is 38 times higher than the concentration found in soil leachate (Fig. 2). This strong increase can be attributed to the increase in the amount

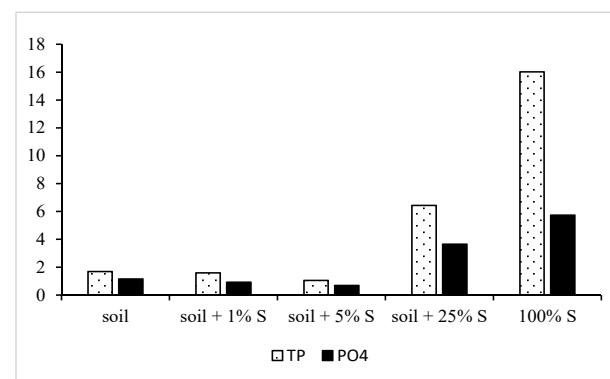
of leached nitrogenous organic matter. These data are supported by the recent study showing elevated concentrations of organic compounds (either susceptible to biological degradation or not) and ammoniacal nitrogen substances in the leachate (Mor et al., 2006).

Ammonium levels, however, have remained low, presumably because of its low stability and rapid conversion to other nitrogenous forms (NO_2 , NO_3).

Fig. 2. Concentrations of NO_3 , TKN, NH_4 in sewage sludge leachate (mg/L)

The phosphorous compounds (PO_4 , TP)

The leached amounts of the phosphate ion (PO_4) and total phosphorus (TP) vary from 0.1 mg/L to 5.8 mg/L for PO_4 and from 1.2 mg/L to 16.2 mg/L for the TP (Fig. 3). The leached concentrations of PO_4 and TP from the sludge were respectively 5 and 9 times higher than those leached from the soil (Fig. 3). Indeed, phosphorus from sludge is likely to migrate in the soil solution by leaching or diffusion (Vanden Bossche, 1999).

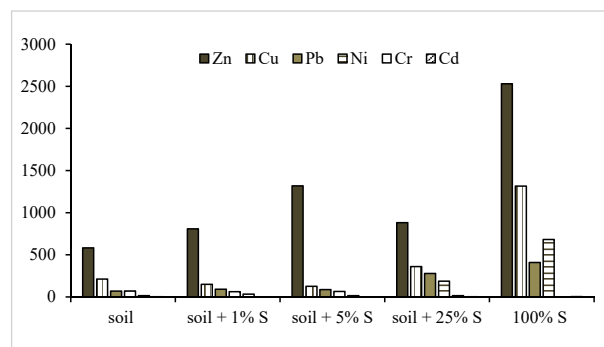
Fig. 3. Concentration of leached TP and PO_4 (mg/L)

Phosphates were the main compounds of TP present in the column effluents, as reported by Łuczkiwicz (2006). Rain water drains soluble and particulate phosphorus from soil or sludge. Despite the high rate of PO_4 and TP leached, the amounts are less significant in comparison with the amount of nitrates. These data are consistent with the previous findings indicating that phosphorus is less easily leached than nitrates and ammonium (Lavelle, 2007).

Heavy metals

The results show that the concentrations of heavy metals found in the leachates of the five studied modalities were markedly high by 2–5 folds when the soil was mixed with sludge at 25% (Fig. 4). Zinc and copper were the most mobilized metals. These data indicate that the sludge may pollute the ground and surface water with heavy metals by several folds depending on the amount of the sludge mixed with the soil.

Fig. 4. Concentration of heavy metals in leachate ($\mu\text{g/L}$) depending on the percentage of the sludge (S) in the soil



Because heavy metals mobility in soils is negatively correlated with pH (Robertson and Blowes, 1995; Geohring et al., 2001), the pH of the sludge-soil mix seems enforcing the retention of heavy metals. Therefore, the leachates pH varies from 7.6 to 7.98. The high pH ($7 \leq \text{pH} \leq 10$) at the surface and slightly neutral ($6 \leq \text{pH} \leq 7$) at depth seems to favour the retention of the heavy metals (Kouame et al., 2006) by the adsorption mechanism (Swift and McLaren, 1991; Kouame et al., 2006).

Several studies have shown that heavy metals from sludge are not completely immobilized within solid soil fractions, but some of these metals can travel into the solution (Camobreco et al., 1996; Richards et al., 1998).

Thus, the biodegradation of sludge contributes to the supply of metals, resulting in greater leaching of heavy metals in the sludge modality. More heavy metals then pass into solution for this modality. Zinc is considered the most mobile and bioavailable among cationic heavy metals (Kiekens, 1995; Fjällborg et al., 2005). Cadmium and chromium were the least mobilized metals. In fact, the sludge would play a retention role for Cd and Cr. These metals could then be immobilized within stable organometallic complexes.

Characteristics of the solid substrates of the percolation test

At t_0 , prior to the run of the percolation test, it was noted that the addition of the sludge to the soil with percentages of 1 to 25% did not affect all the pH, which remained almost constant (Table 3). Indeed, the pH values of the modalities were close to neutrality and showed that these modalities favoured the assimilation of the nutrients. However, the electrical conductivity increased in parallel with the increase in the sewage rate, from 291 $\mu\text{S/cm}$ for the soil modality to 4150 mS/cm for the soil + 25% of sludge; similar results were according to the findings of other authors (Dridi and Toumi, 1999; Bipfubusa et al., 2006; Amadou, 2007; Bahri and Annabi, 2011). Total phosphorus, PO_4 and NH_4 levels were also improved following sludge addition. Spreading of sludge will have a fertilizing effect of the soil and, therefore, would increase the crop yield, promote the growth of herbaceous species in natural environment and ultimately reduce the rate of erosion.

After one year (t_1) since the percolation test installation, the five modalities showed, on the one hand, a decrease in electrical conductivity, which can be explained by the leaching of ions by rain water and decreasing in TP and PO_4 contents due to the mineralization of the organic matter and the leaching of the latter by rainwater, respectively (Table 3). Furthermore, NH_4 , NO_3 and PO_4 levels decreased in soil and in mixtures of 1 to 25% of sludge with soil, due to their dominant leaching. Whereas they increased for the sludge modality due to the mineralization of organic nitrogen into ammonium followed by the nitrification process. However, the NO_3 level decrease from 1360.95 to 682.06 mg/kg , and from 1482.8 to 863.25 mg/kg , and from 1354.62 to 1069.77 mg/kg , for 0%, 1%, 5%, and 25% of the sludge-soil mixture, respectively.

Table 3. Physical and chemical characteristic of solid substrates of percolation test at t0 and t1

		pH	EC ^a	NO ₃ ^b	NH ₄ ^c	TP ^d	PO ₄ ^e
Soil	t0	7.95	291	1360.95	8.61	848.02	84.56
	t1	7.82	169	682.06	5.95	376.29	82.79
Soil + 1% S	t0	7.95	334	1482.8	20.18 **	872.18	119.29
	t1	7.92	170.4	863.25	5.1	351.83	85.77
Soil + 5% S	t0	7.83	601**	938.42*	33.74*	990.56	190.56
	t1	7.75	196.6	864.05	6.63	762.55*	89.69
Soil + 25% S	t0	7.25*	4150***	1354.62	38.44**	1224.91	362.4*
	t1	7.52	558*	1069.77*	17.09*	728.42*	104.49
100% S	t0	6.92**	4550**	892*	11.25	1275.65	688.56**
	t1	6.97	3090***	3266.28**	64.39**	1069.08*	332.2*

a: $\mu\text{S}/\text{cm}$; b, c, d, e: mg/kg ; *: the difference is considered significant when $p < 0.05$ (*, $p < 0.05$; **, $p < 0.01$, ***, $p < 0.001$) using unpaired Student t test to compare sample values vs control group – soil alone

Conclusion

The leaching test carried out in this study provides an explanation of how the rain allows percolation of the most mobile chemical compounds from sewage sludge applied to land. The percentage of the added sludge to soil determines the leaching amount of nitrogen, phosphate, salt compounds and heavy metals, mainly zinc and copper. Undoubtedly, this percolation influences the chemical composition of shallow aquifers, especially when soils are characterized by high water permeability. Hence, together with heavy metals, nutrient compounds should be regarded as limiting factors for sewage sludge land application. Also, the effects of short-term addition of sludge studied in this experiment indicate that this addition has a beneficial fertilizing effect on the soil for

the short term in the absence of significant drain by rain water. Therefore, it is of great interest to expand the use of sludge in non-agricultural environments such as forest soils or degraded soils with the aim of replanting or increasing wood production.

However, in order to protect the quality of ground and surface water, this study provides evidence of deleterious effects of WTP-sludge on soil and water quality, as well as a strong rationale for the necessity of a careful monitoring, management and storage of sludge produced by sewage treatment plants. Regulatory measures for setting the conditions and technical requirements have to be implemented to ensure the protection of environment and population's health.

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