

**Research article**

# Influence of Hydraulic Conditions on Reclaimed Water Polishing Using Soil and Sand Columns

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## Abstract.

The removal of residual pollutants from a synthetic effluent with a composition similar to that of urban effluent from secondary treatment was evaluated in vertical downflow columns. These were filled with soil, the fine fraction of the soil, and sand, and operated in discontinuous and continuous mode. The results showed high removal rates of organic matter, ammonium, nitrate and phosphate in the discontinuous and continuous experiments, especially for the fine fraction. Therefore, the soil is suitable for removing organic matter and nutrients (N-NH<sub>4</sub>, N-NO<sub>3</sub>, and P-PO<sub>4</sub>), and can be used for polishing wastewater before its infiltration.

**Keywords:** wastewater reuse, organics removal, nutrient removal, residual soil, river sand

## 1. Introduction

Adaptation and mitigation of climate change through water management are essential for sustainable development. Therefore, it is increasingly necessary to consider “unconventional” water resources in future planning [1]. Reclaimed water is a reliable alternative to conventional water resources for many uses [1-2]. Indeed, there are several successful cases of water reuse worldwide [3], including the artificial recharge of aquifers with treated wastewater. The complementary treatment of secondary effluents through the soil (Soil aquifer treatment - SAT), before its inclusion in the aquifers, has been gaining increased attention in several countries around the world [4-5], with studies pointing to the advantage of its application [4,6]. However, if the soil does


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not present favourable conditions for infiltration of treated wastewater, the residual loads of that water, characterized by an organic matter of difficult biodegradability (e.g. hydrocarbons, pesticides and several contaminants of emerging concern), forms of nitrogen (ammonium, nitrite, and nitrate), and phosphorus (orthophosphates and polyphosphates), heavy metals and pathogenic microorganisms, can be a disadvantage for groundwater quality.

Thus, the purpose of this research was to evaluate the influence of the hydraulic conditions on the removal of organic matter and nutrients (nitrogen and phosphorus forms), using laboratory columns filled with soil, the fine fraction of the soil and river sand, and verify the main mechanisms responsible for removing these pollutants.

## 2. Material and methods

### 2.1. Characterization tests of soil, fine soil, and sand

A granitic residual soil was collected at the Quinta de Gonçalo Martins (Guarda, Portugal) [7]. A component less than 0.075 mm (fine soil) was extracted from the soil, as this is where the most reactive soil fractions are found, namely silt and clay. In addition, river sand was also used [8].

To understand the importance of soil properties in the removal of pollutants, soil granulometric analysis was performed using the sieving method [9] and the fine soil analysis using the sedimentation method [10]. The specific surface was determined by laser diffraction, using the Coulter LS200 equipment. The density of solid particles was determined by the pycnometer method [11] and the porosity using the procedures described in [6]. The qualitative mineralogical composition of the soil was determined by X-Ray Diffraction (XRD) with a Rigaku diffractometer, DMAX III/C, USA. For the morphological and microstructural analysis, the Scanning Electron Microscope (SEM), Hitachi S-2700, USA, was used. The chemical composition (analysis in oxides) was determined with the Energy Dispersive Spectrometer (EDS), which is coupled to the SEM. Organic matter was determined by the Walkley-Black method [12], and soil pH was determined in H<sub>2</sub>O and KCl by the potentiometric method, in suspension (soil: water, 1:2.5), described by [13].

## 2.2. Columns experiments

Experiments were set up in three acrylic circular columns with vertical and downflow flux, with a height of 42 cm and 14 cm in diameter (Figure 1), and a synthetic effluent. Water samples were collected at column top ( $C_T$ , influent),  $P_1$ ,  $P_2$ , and  $C_B$  points (the  $C_B$  point was considered for the effluent at the column base). The three columns were filled with soil, fine soil, and river sand. The filling media had a height of 25 cm. The first column had 20 cm of soil (mass = 4467.4 g) and 5 cm of sand (mass = 891.4 g) at the bottom. The second column had 20 cm of fine soil (mass = 3588.1 g) and 5 cm of sand (mass = 891.4 g) at the bottom. The third column had 25 cm of sand (mass = 5912.1 g). All columns were filled with synthetic wastewater until the media was submerged with a water level of  $\pm 2$  mm above the  $C_T$  point. The lower sand layer was provided to protect the upper layers of soil and fine soil against the exit of fine particles and disturbance of the flow.

The synthetic effluent was similar to those used by [14-15], which included a carbon source (sodium acetate trihydrate,  $\text{CH}_3\text{COONa}\cdot 3\text{H}_2\text{O}$ ), two nitrogen sources (ammonium chloride,  $\text{NH}_4\text{Cl}$ ; and potassium nitrate,  $\text{KNO}_3$ ), a phosphorus source (di-potassium hydrogen phosphate trihydrate,  $\text{K}_2\text{HPO}_4\cdot 3\text{H}_2\text{O}$ ), and a trace element solution (iron chloride, magnesium sulphate, and calcium chloride), according to the compositions and C/N/P ratios indicated in [6]. The solutions of sodium acetate, ammonium chloride, potassium nitrate, and di-potassium hydrogen phosphate were prepared in a concentrated form and diluted according to the desired loads. The loads used were similar to those observed by [15], approximately  $150 \text{ mg L}^{-1}$  of COD,  $30 \text{ mg L}^{-1}$  of N- $\text{NH}_4$ ,  $10 \text{ mg L}^{-1}$  of N- $\text{NO}_3$ , and  $10 \text{ mg L}^{-1}$  of P- $\text{PO}_4$ , which are typical of urban effluents from secondary treatment. In the analysis and discussion of results, the following terms will be used: organic matter (for COD values), ammonium or ammonia nitrogen (for N- $\text{NH}_4$  values), nitrate or nitric nitrogen (for N- $\text{NO}_3$  values), and phosphate (for P- $\text{PO}_4$  values).

Experiments were run in two phases. In the first phase, the columns were operated in a discontinuous mode, including filling with the feed solution, reaction for 3.5 days (totalling 35 days, 10 cycles), drainage, and sampling, as suggested by [16]. In the second phase, the columns were operated in a continuous mode, also for 35 days, with a feeding system composed of an ISMATEC MCP CA4 peristaltic pump (Switzerland) for pumping the influent to the inlet of the column, with a flow rate of  $0.36 \text{ L d}^{-1}$  and a hydraulic load of  $2.34 \text{ cm d}^{-1}$ , values previously tested in the laboratory and in agreement with those observed by [17-19], on similar filling materials. At the beginning and end of each cycle, in the first phase, water samples were collected at the influent ( $C_T$  point),  $P_1$ ,  $P_2$ , and  $C_B$



**Figure 1:** Column tests.

for measuring pH, temperature, COD, N-NH<sub>4</sub>, N-NO<sub>3</sub>, and P-PO<sub>4</sub>. For the second phase, water samples were collected at the same points every 3.5 days for determining the same parameters. A SenTix 41 probe connected to a Multi 340i meter (WTW, Germany) was used for determining pH and temperature. COD, N-NH<sub>4</sub>, N-NO<sub>3</sub>, and P-PO<sub>4</sub> were determined according to [20].

### 3. Results and discussion

#### 3.1. Characterization of soil, fine soil, and sand

The soil used in the study has a low clay fraction (particle size less than 2 μm) of approximately 5% (Figure 2), which means that most of the fines are essentially silty. According to [21], to prevent soil clogging and ensure wastewater treatment, the soil should not contain more than 10% clay. Figures 3-(a) to 3-(c) show the morphology of soil, fine soil, and sand particles.

Table 1 presents the physicochemical characteristics of the three materials, most relevant to the study. For fine soil (Table 1), the permeability ( $k$ ) is low ( $k = 3.72 \times 10^{-6} \text{ m s}^{-1}$ ). However, soils with permeability in the order of  $6.94 \times 10^{-6} \text{ m s}^{-1}$  are suitable for controlled infiltration of treated effluents [2]. It essentially contains silica (60.44%) and

alumina (31.76%), with lower levels of iron (3.99%) and potassium (3.81%) oxides. The organic matter content is very low (0.45%), and the soil is very acidic (pH = 4.44). The mineralogical composition consists of quartz, muscovite, illite, kaolinite, and smectite (identified in the fraction below 2 μm). Kaolinite constitutes about 60% of the clay mineral present in the soil.

This soil has a specific surface area suitable for the development of biofilm capable of removing pollutants and pathogens and the clay-colloidal complex of this soil also has reactive properties that allow the removal of pollutants by sorption mechanisms [6].

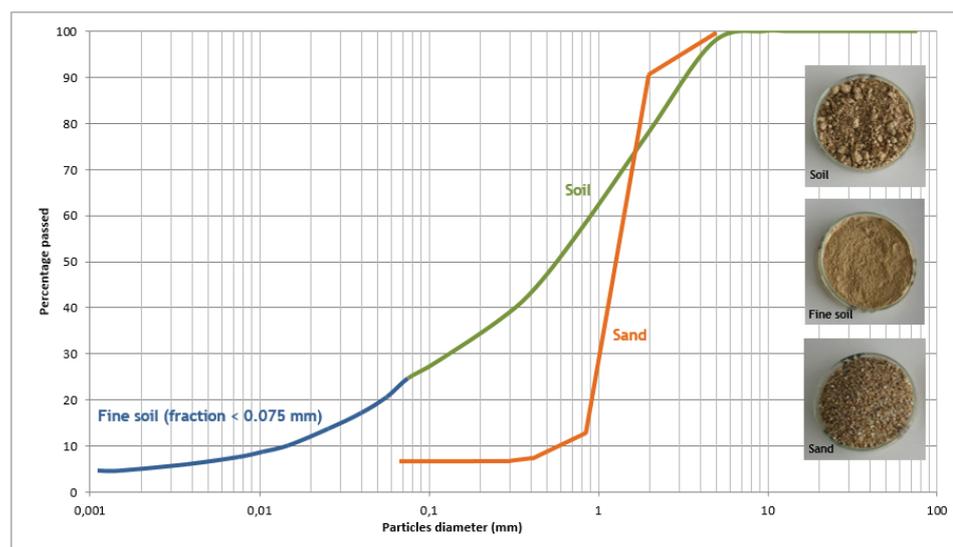


Figure 2: Particle size of soil, fine soil and sand.

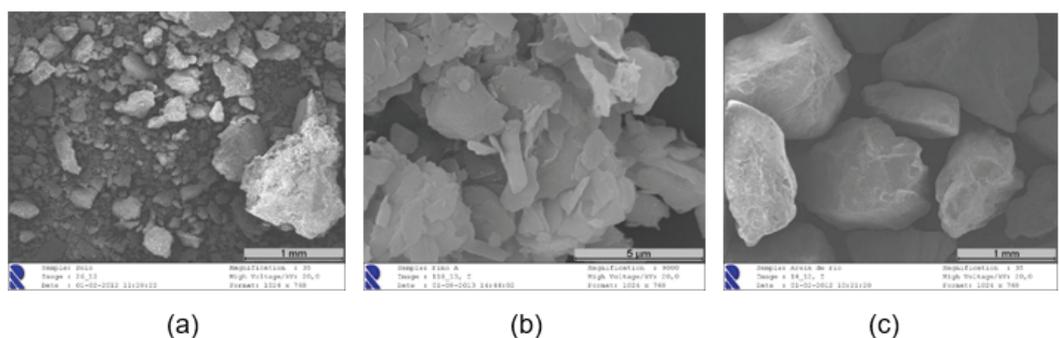


Figure 3: SEM images: (a) Soil, (b) Fine soil, and (c) Sand, with a magnification of 35x, 9000x, and 35x, respectively.

TABLE 1: Physicochemical characteristics of the materials.

Parameters	Soil	Fine soil (< 0.075 mm)	Sand
Effective diameter D <sub>50</sub> (mm)	0.59	0.027	1.28
Porosity (%)	38.5	48.0	41.0
Solid particle density (G <sub>s</sub> )	2.65	2.65	2.65
Permeability (k) (m s <sup>-1</sup> )	3.23 x 10 <sup>-5</sup>	3.72 x 10 <sup>-6</sup>	8.98 x 10 <sup>-4</sup>
Specific surface area (m <sup>2</sup> g <sup>-1</sup> )	-	0.29	-
Oxides (%): SiO <sub>2</sub> ; Al <sub>2</sub> O <sub>3</sub> ; Fe <sub>2</sub> O <sub>3</sub> ; K <sub>2</sub> O	58.64; 33.17; 3.96; 4.23	60.44; 31.76; 3.99; 3.81	86.44; 7.99; 1.54; 4.03
Organic matter content (%)	0.25	0.45	0.03
pH (1:2.5) H <sub>2</sub> O; KCl	4.91; 3.84	4.44; 3.48	6.32; 5.26

### 3.2. Evaluation of the removal of organic matter and nutrients

Table 2 and Table 3 show the variation of pH, temperature, COD, N-NH<sub>4</sub>, N-NO<sub>3</sub>, and P-PO<sub>4</sub>, for the sampling points C<sub>T</sub> and C<sub>B</sub> in the first and second phases.

TABLE 2: Results of the first phase in discontinuous feeding.

Parameters	Column top <sup>1</sup>	Soil <sup>2</sup>	Column base	
			Fine soil <sup>3</sup> (< 0.075 mm)	Sand <sup>4</sup>
pH	6.67 - 7.32	4.90 - 6.78	6.32 - 7.12	6.67 - 7.28
Temp. (°C)	21.96 ± 0.37	21.8 ± 0.23	22.35 ± 0.51	21.90 ± 0.15
COD (mg L <sup>-1</sup> )	154.70 ± 11.57	36.01 ± 10.84	29.11 ± 6.66	51.12 ± 32.11
N-NH <sub>4</sub> (mg L <sup>-1</sup> )	33.73 ± 2.31	2.20 ± 1.13	1.15 ± 0.78	16.95 ± 3.09
N-NO <sub>3</sub> (mg L <sup>-1</sup> )	9.95 ± 0.59	1.61 ± 0.82	0.31 ± 0.08	6.50 ± 1.59
P-PO <sub>4</sub> (mg L <sup>-1</sup> )	10.28 ± 0.22	0.33 ± 0.29	0.13 ± 0.10	9.83 ± 0.37

Assuming a 95% confidence level, for the following sample sizes:

<sup>1</sup>10 (CQO); 10 (N-NH<sub>4</sub>); 9 (N-NO<sub>3</sub>); 8 (P-PO<sub>4</sub>); <sup>2</sup>9 (CQO); 8 (N-NH<sub>4</sub>); 6 (N-NO<sub>3</sub>); 8 (P-PO<sub>4</sub>);

<sup>3</sup>10 (CQO); 8 (N-NH<sub>4</sub>); 6 (N-NO<sub>3</sub>); 4 (P-PO<sub>4</sub>); <sup>4</sup>10 (CQO); 9 (N-NH<sub>4</sub>); 9 (N-NO<sub>3</sub>); 8 (P-PO<sub>4</sub>).

TABLE 3: Results of the second phase in continuous feeding.

Parameters	Column top <sup>1</sup>	Soil <sup>2</sup>	Column base	
			Fine soil <sup>3</sup> (< 0.075 mm)	Sand <sup>4</sup>
pH	7.31 - 8.17	5.38 - 6.97	5.12 - 6.40	7.37 - 7.85
Temp. (°C)	22.38 ± 0.40	22.07 ± 0.41	22.12 ± 0.45	22.08 ± 0.38
COD (mg L <sup>-1</sup> )	93.78 ± 13.89	24.69 ± 4.54	18.18 ± 4.07	47.15 ± 6.45
N-NH <sub>4</sub> (mg L <sup>-1</sup> )	26.84 ± 1.12	5.64 ± 2.48	0.41 ± 0.06	21.81 ± 3.55
N-NO <sub>3</sub> (mg L <sup>-1</sup> )	10.94 ± 0.49	2.24 ± 0.63	0.14 ± 0.04	9.66 ± 0.43
P-PO <sub>4</sub> (mg L <sup>-1</sup> )	10.51 ± 0.40	3.05 ± 0.88	1.16 ± 0.59	10.25 ± 0.30

Assuming a 95% confidence level, for the following sample sizes:

<sup>1</sup>10 (CQO); 10 (N-NH<sub>4</sub>); 10 (N-NO<sub>3</sub>); 10 (P-PO<sub>4</sub>); <sup>2</sup>10 (CQO); 10 (N-NH<sub>4</sub>); 10 (N-NO<sub>3</sub>); 10 (P-PO<sub>4</sub>);

<sup>3</sup>10 (CQO); 10 (N-NH<sub>4</sub>); 10 (N-NO<sub>3</sub>); 10 (P-PO<sub>4</sub>); <sup>4</sup>10 (CQO); 10 (N-NH<sub>4</sub>); 10 (N-NO<sub>3</sub>); 10 (P-PO<sub>4</sub>).

Table 2 and Table 3 show a drop in pH from  $C_T$  to  $C_B$ . The average temperature remains practically constant throughout all cycles. The best removal rates were obtained for fine soil, and, therefore, the analysis will only be focused on the fine soil results.

The results for the first phase (Table 2) show average concentration removals of 125.59 mg COD L<sup>-1</sup>, 32.57 mg N-NH<sub>4</sub> L<sup>-1</sup>, 9.52 N-NO<sub>3</sub> L<sup>-1</sup>, and 10.24 mg P-PO<sub>4</sub> L<sup>-1</sup>, which correspond to mass removals rates of 232.0 mg of COD, 60.17 mg of N-NH<sub>4</sub>, 17.59 mg of N-NO<sub>3</sub> and 18.91 mg of P-PO<sub>4</sub>, and removal efficiencies of 80.78% COD, 96.55% N-NH<sub>4</sub>, 96.81% N-NO<sub>3</sub> and 98.73% P-PO<sub>4</sub>. For the second phase (Table 3), the average concentration removals were 75.60 mg COD L<sup>-1</sup>, 26.43 mg N-NH<sub>4</sub> L<sup>-1</sup>, 10.80 N-NO<sub>3</sub> L<sup>-1</sup>, and 9.35 mg P-PO<sub>4</sub> L<sup>-1</sup>, corresponding to mass removals rates of 139.65 mg of COD, 48.82 mg of N-NH<sub>4</sub>, 19.95 mg of N-NO<sub>3</sub> and 17.28 mg of P-PO<sub>4</sub> and removal efficiencies of 80.43% COD, 98.45% N-NH<sub>4</sub>, 98.70% N-NO<sub>3</sub> and 88.95% P-PO<sub>4</sub>.

In the first phase (discontinuous feeding), the removal of organic matter starts to be significant in the 4th cycle, because the biofilm was already stable and well developed to be able to continuously remove acetate, as was also observed by [22]. In the second phase (continuous feeding), the higher removal efficiency (86.27%) was observed on the 8th day. In both experiments, carbonaceous removal has occurred, either in the presence of oxygen (closer to the surface of the column), by aerobic removal mechanisms such as aerobic respiration and nitrification, or in the absence of oxygen, by anaerobic removal mechanisms, such as anaerobic respiration, denitrification and fermentation processes.

Ammonium reached a higher removal efficiency of 98.66% (7th cycle), for the discontinuous feeding, and higher removal efficiency (99.46%) for the 3rd day, for the continuous feeding. This means that nitrification was present. Therefore, ammonium oxidation may have occurred closer to the surface, where oxygen is able to dissolve in the water mass by diffusion, as admitted by [23].

In both phases (discontinuous and continuous experiments), the removal of nitrate was very significant, which have occurred through denitrification at the lower layers where oxygen was low. Nitrate was present through the feeding solution and also produced due to the ammonia oxidation (nitrification). The decrease in pH indicates that there was more alkalinity consumption, which is characteristic of the nitrification mechanism, than production, which is characteristic of the denitrification mechanism,

as mentioned by [24]. The pH variation observed (from around 7 to around 5) is also favourable to the presence of unconventional removal mechanisms, as mentioned by [25].

Phosphate removal was also significant and reached a higher removal efficiency of 99.54% in the 5th cycle, for discontinuous experiments, and higher removal efficiency of 94.62% on the 15th day, for continuous experiments. However, contrary to what would have happened with organic matter, ammonium, and nitrate, the biological removal of phosphate is low [26] and usually occurs by biosorption (i.e. it is adsorbed on the polymers that make up the biofilm) and by complexation and precipitation at low pH (usually complexes with aluminium and iron, precipitating in the form of phosphates), as reported by [27-28].

## 4. Conclusions

The soil and its fine fraction have reactive properties that allow a good capability for removing organic matter, ammonium, and nitrate by biodegradation mechanisms and phosphate by sorption mechanisms, either in discontinuous or continuous mode experiments, allowing it to act as a barrier to the contamination of groundwater during infiltration with reclaimed water. However, fine soil has showed to be a better media for residual pollutant removal.

## 5. Acknowledgments

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