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Purification of Wastewaters by a Combined System Planted of Emerged Macrophytes

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Introduction

Since many years, industrial activities in Abidjan (Ivory Coast) have generated polluted wastewater, which is frequently discharged into lagoon bays. These liquid effluents bring organic matter, nutrients and micropollutants to Ebrié lagoon. Currently, waters of the bays record high levels of pollution, especially in organic biorecalcitrant and toxic pollutants. This usually leads to a multiplication of invasive microalgae and aquatic plants causing significant mortality of invertebrates and fishes. While the effects of global organic materials are relatively well known, there are still questions about those of micropollutants such as surfactants, whose conventional treatment processes fail to effectively remove them. Indeed, conventional wastewater treatment has often failed in developing countries due to complex operating procedures, costly maintenance actions, sludge production and high energy consumption (Dhote and Ingole, 2012). To meet the needs of communities and industries with technical and / or often financial constraints, new technologies have been developed. Filters planted with macrophytes are one of these alternative processes to conventional processes (Vymazal, 2014; Yalcuk and Ugurlu, 2009). Their operation and maintenance are easy and cheaper (Wu and Sansalone, 2013). In the wastewater treatment process, these systems involve physico-chemical and biological reactions. However, the basic principle of planted filters remains the infiltration of raw water through beds composed of a mixture of materials (sand and gravel) and assimilation by plants (Kadlec and Wallace, 2008).

ABSTRACT This work is focused on the purification of soap factory wastewaters by a combined system planted of emerged macrophytes. The exploitation of this combined system planted reveals substantial results after twenty weeks of the purification of wastewater: neutralization of the pH of filtered water and its cooling. Almost all suspended matter is removed (95%) from the filtered waters. Significant reductions in ammonium (75%) and nitrite (69%) contents are observed. Using this combined system planted, high removal rates of orthophosphates (56%) and anionic surfactants (81%) are obtained. The removal rate of organic matter is about 76% in BOD₅ and 42% in COD.

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The present study aims to improve the quality of industrial wastewater of the soap factory type. To achieve this purpose, a planted system consisting of the series association of two filters planted with macrophytes, one to vertical flow and the other to horizontal flow, is being tested. The association of these two models of planted filters will allow to strongly remove the soluble organic load in themes of BOD₅, COD, orthophosphates, nitrogenous compounds, anionic surfactants, and other substances such as suspended matter and fats contained in the wastewater.

Materials and Methods

Determination of Hydrodynamic Parameters

100g of sand sample is successively sieved in the standard AFNOR sieve. This device consists of 16 superimposed sieves; the dimensions are between 5 and 0.063 mm. The calculation of the rejection percentages is made using the mass of material rejected by each sieve. These percentages are used to plot the grain size curve of the sand samples using semi-logarithmic paper. Reading the values on the plot, the diameters d_{10} and d_{60} are determined. Thus, the ratio between these two values d_{60} and d_{10} gives the coefficient of uniformity (Cu): Cu = d_{60} / d_{10} .

The flow rate (Q in m^3/d) of the water is determined from the flow tests: Q = v / t. This corresponds to the recovery time (t) of a volume (v) poured into the filter.

Description of Wastewaters Purification Device

The wastewater treatment device is composed of 500 L wastewater storage drum (1), 3 m³ settling-digestion basin (2) and two filtration basins with a volume of 2 m³ and a useful storage capacity of 500 L each (Fig. 1). These filtration basins

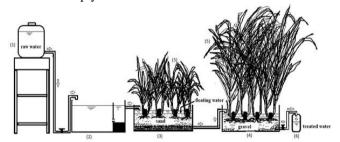
are arranged in series and constitute the combined system planted: the vertical flow filter (3) and the horizontal flow filter (4). The filter materials used consist of two layers of gravel with a grain size of 30-60 mm and 10-20 mm, a layer of siliceous sand and young stems of common reeds (5). The thickness of each layer of gravel and that of the sand are 10 cm and 30 cm respectively. A barrel of 60 L (6) is installed downstream of the device to collect the treated water

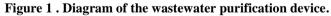
Conduct of the Experiment

The experiment of the combined system planted takes place in three phases. The first phase, which lasts two weeks, allowed the installation of filters planted with macrophytes. Setup first involves placing the gravel at the bottom of the bin and placing the sand just above the gravel. A geotextile filter separates these two materials. Then, young reed stems with a density of 15 stems/m² are planted on the sand bed. Then, you have to pass the tap water through the sand bed until a permanent layer of supernatant water is obtained of 5 centimeters.

The second phase corresponds to the commissioning of the combined system planted, and it lasts six weeks. During this period, the purification basins are first irrigated with tap water for four weeks, then with wastewater to be treated in increasing quantities: 10 L in the 5th week then 20 L in the 6th week. This allows the young reed stems (macrophytes) on the one hand to adapt and grow, and on the other hand the biological membrane to develop just before the functioning phase of the system.

The third phase of the experiment concerns the actual utilization and operation of the purification device. It lasts twenty-four weeks. During this period, the combined system planted is powered by "tarpaulins"; it consists of regularly supplying a quantity of wastewater of 25 L per day. The vertical and horizontal flow filters successively receive the wastewater so as to submerge their entire surface. The filtered water leaving this combined system is collected in an empty barrel using a drain placed at the base of the horizontal filter. From this moment, the combined system planted operates without interruption until the quantity of filtered water decreases sharply.





The total duration of the experiment is 30 weeks during which the operation of the combined system planted is monitored by measuring chemical and physical parameters. A total of twenty-four soap factory wastewater samples, one hundred and twenty feeds to the combined system planted and twenty analyzes of the filtered water samples were carried out.

Choice of Sampling Site and Analysis of Samples

The study initially concerned three soap factories located near the bay of Bietry, in the city of Abidjan, due to the intensification of their liquid discharges into the bay. But only one soap factory was selected. The choice of the target soap factory is based on the easy access to the site and the physicochemical quality of the moderately fluctuating wastewater. Once a week, the wastewater from the target soap factory is collected in 60 L barrels and transported to the test site. Upon arrival, the collected water is immediately transferred to the storage barrel where it will be used to supply the pilot device. The pilot device is checked daily by taking samples at the entry and exit of the system. These samples are then stored in the freezer between -10 and 0°C.

Before carrying out analyzes, the water samples are thawed and brought to room temperature. The physicochemical analyzes are carried out taking into account the retention period of each parameter. The chemical and physical parameters measured, according to pre-established methods, are as follows: temperature, pH, redox potential, oils and fats, SST, anionic surfactants, ammonium, total phosphorus, BOD₅ and COD.

Results

Hydraulic System Operation

The sand used as filter material is composed of grains with an effective diameter of 220 μ m and a diversity equal to 600 μ m; which gives a uniformity coefficient of 2.72.

At the beginning of the commissioning of the combined system planted, the flow tests carried out give a flow rate of approximately 632 mL / min. Gradually, the flow of filtered water decreases until the end of the experiment.

Physicochemical Characteristics of Wastewater

At the sampling site, it was noticed the presence of sludge, whitish coloration and floating matter, as well as the formation of foams in the wastewater. The analysis of this water gives an idea of the physicochemical quality of the raw water before its treatment (Tab. 1).

It appears in this table that the wastewater from the target soap factory has a temperature of $(36\pm3)^{\circ}$ C, a very low redox potential of (-116±43) mV and a pH of (9.4±1.7). This wastewater has an alkaline and reducing character.

Table 1. Parameters of	wastewater	at the	outlet of soap
	fastan		

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Parameters	Range of	Mean ± standard		
	values	deviation		
pH	7.4 to 11.5	9.4 ± 1.7		
Eh (mV)	– 176 to - 44	- 116 ± 43		
Temperature (°C)	33 to 42	36 ± 3		
NH_4^+ (mg.L ⁻¹)	1.64 to 3.83	2.47 ± 0.41		
NO_{2}^{-} (mg.L ⁻¹)	2.42 to 4.56	3.45 ± 1.12		
NO_3^{-1} (mg.L ⁻¹)	0.01 to 0.21	0.01 ± 0.00		
PO_4^{3-} (mg.L ⁻¹)	23.43 to 42.26	33.87 ± 9.63		
MBAS (mg. L^{-1})	7.21 to 15.26	8.45 ± 2.27		
$BOD_5 (mg.L^{-1})$	679 to 1195	820 ± 228		
$COD (mg.L^{-1})$	1298 to 1789	1563 ± 392		
TSS (mg. L^{-1})	1340 to 1810	1533 ± 246		
Oils and fats (mg.L ⁻¹)	737 to 1325	958 ± 320		

The nitrite content of the wastewater is (3.45 ± 1.12) mg.L⁻¹. Those of nitrate and ammonium are equal to (0.01 ± 0.00) mg.L⁻¹ and (2.47 ± 0.41) mg.L⁻¹ respectively.

The orthophosphate content of the waste water is very high with (33.87 ± 9.63) mg.L⁻¹. The organic compound contents in terms of BOD₅ and COD are (820 ± 228) mg.L⁻¹ of O₂ and (1563 ± 392) mg.L⁻¹ of O₂ respectively. Part of the organic matter consists of anionic surfactant substances, the measured value of which varies between 7.2 and 15.3 mg.L⁻¹ MBAS. As for solids and fats, the recorded contents of (1533 ± 246) mg.L⁻¹ TSS and, (958 ± 320) mg.L⁻¹ fats and oils are large.

Purifying Performance of the Treatment Device Physical Characterization of the Filtered Water

The quality of the filtered waters fluctuates greatly throughout the operation of combined system planted. The

temperature of the filtered water is 28°C and its pH is around 6.7; which corresponds to a drop-in temperature of about 8 units and a drop in pH of 3 units.

The total suspended solids content after treatment with the planted filters is approximately 84 mg.L⁻¹; which gives a very significant reduction of 95%.

Elimination of Nitrogen Compounds

Mainly three forms of nitrogen (ammoniacal nitrogen, nitrite and nitric nitrogen) were analyzed during the experimental period of the combined system planted. It was observed that the concentration of nitrogen compounds in wastewater has an influence on that of the filtered water by the combined system planted of emerged macrophytes. At the outlet of the combined system planted, the filtered water has an acidic character.

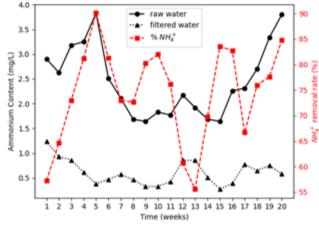


Figure 2. Evolution of ammonium content and rate of removal by combined system planted.

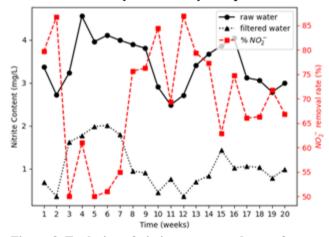


Figure 3. Evolution of nitrite content and rate of removal by combined system planted.

The Figure 2 shows an oscillation of the ammonium content and its removal rate during the experimentation period. However, the combined vertical and horizontal subsurface flow system was found to reduce the NH_4^+ content of the wastewater. This decreased from 2.46 mg.L⁻¹ to 0.61 mg.L⁻¹, which corresponds to NH_4^+ elimination rate of 75%.

The nitrite concentration decreased from 3.44 mg.L^{-1} to 1.08 mg.L^{-1} providing an approximate elimination rate of 69% as shown in Figure 3. An enrichment is observed in nitrites from filtered water during the first six weeks, followed by depletion to the twelfth week, then further enrichment until the end of the experiment. The rate of nitrite removal varies between 50 and 87%.

Elimination of Orthophosphates

The orthophosphates (PO_4^{3-}) content in the filtered water varies according time and depending on the quality of the

wastewater (Fig. 4). The average content of the wastewater was 32.58 mg.L^{-1} and using the purification device, it dropped to 14.30 mg.L⁻¹ in the filtered water. This equivalent to an average removal of 56% of PO₄³⁻ presents in the wastewater from the soap factory.

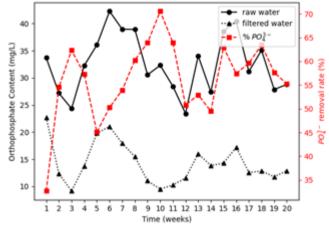


Figure 4. Evolution of orthophosphates content and rate of removal by combined system planted. Elimination of Anionic Surfactants

During the period of operation of the combined system planted, the anionic surfactants (MBAS) content in the filtered water varies between 0.9 mg.L⁻¹ and 3.3 mg.L⁻¹, with an average value of 1.7 mg.L^{-1} (Fig. 5). This corresponds to an elimination rate of approximately 81%. This variation in concentration of 2 units is negligible, despite the strong fluctuation in the quality of the wastewater.

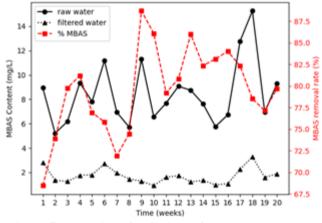


Figure 5. Evolution of anionic surfactants content and rate of removal by combined system planted. Elimination of Organic Matter

The BOD₅ and COD contents of the wastewater and the filtered water are shown in Figure 6 and 7. During the operation period of the combined system planted, BOD₅ and COD contents of the wastewater fluctuate widely. This is also the case with filtered water, which undergoes an alternation of decreases and increases in organic matter content. The concentrations of these compounds at the outlet of the combined system vary between 143 and 384 mg.L⁻¹ for the BOD₅ and, between 679 and 1048 mg.L⁻¹ for the COD. It was observed that the rate of BOD₅ removal increases during the first 4 weeks of operation of the combined system planted, and then decreases sharply in the fifth before rising again (Fig. 6). There is an alternating rise and fall in the performance of the purification system, which is linked, to the variation in the BOD₅ content in the effluent – affluent. The average BOD₅ removal rate is approximately 76%.

The COD content of the wastewater decreases after passing through the combined system planted with

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macrophytes (Fig. 7). The difference between raw COD and COD after filtration consists essentially of suspended solids. At the outlet of the combined system, COD varies between 679 and 1048 mg.L⁻¹, i.e. an average value of 893 mg.L⁻¹. This corresponds to an elimination rate, which oscillates between 25 and 60%, or on average 42%, despite the high concentration of raw COD.

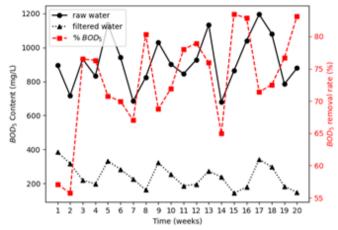


Figure 6. Evolution of BOD₅ content and rate of removal by combined system planted.

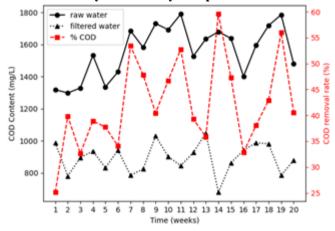


Figure 7. Evolution of COD content and rate of removal by combined system planted.

The experimental analysis of the filtered water by the combined system planted of emerged macrophytes was performed. This combined system was found to have an average removal efficiency of 76% BOD₅ and 42% COD in an operating time of 20 weeks (Fig. 6 & 7).

Discussion

The particle size characteristics show that this sand used consists of grains of various sizes, and it seems better suited for a slow filtration system (Kadlec et Wallace 2008).

After six weeks, the root system and buds of the young reed stems began growing up. This shows that the combined system planted device is in full swing. At this time, the flow of water leaving the combined system has dropped to approximately 479 mL.min⁻¹. This flow rate value is characteristic of a rapid filtration system (Morvannou et al., 2013). Such a system can allow to treat a large volume of wastewater. The development of macrophytes stems helps on the one hand to reduce the clogging of the sand bed and, on the other hand to maintain sufficient porosity to ensure the percolation of the filtered water (Prochaska et al., 2007).

Temperature and pH values don't respect the recommended standards for waste water in nature. Therefore, the temperature above 30°C and sometimes reaching 42°C is likely to affect the growth metabolism of microorganisms and

the life of fish in the aquatic environment (Yao et al., 2009). A receiving environment with a pH greater than 10 is harmful to living organisms.

Compared to the fixed maximal values, set, it is noted that the nitrate and ammonium ions are very weak quantities compared to the nitrite ions. Regarding ammoniacal nitrogen, the fact is in an alkaline environment the non-ionized form (NH_3) predominates over the ionized form (NH_4^+) .

$$NH_3(g) + H_2O \Leftrightarrow NH_4OH \Leftrightarrow NH_4^+ + OH^- \text{ pKa} = 9,25$$

In general, the contamination of wastewater with nitrogen compounds is moderate. This is characteristic of industrial wastewater. It is well known that in wastewater, nitrogen mainly exists in ammoniacal form but our results highlight that it is on nitrous form (NO₂⁻). Nitrite is the intermediate product of several oxidation and reduction reactions involved in different nitrogen transformation processes, including nitrification and denitrification (Verma and Suthar, 2018). These two processes are linked by the following balances:

$$NH_4^+ + 3/2 O_2 + H_2 O \Leftrightarrow NO_2^- + 2H_3 O^+$$

$NO_3^- \Leftrightarrow NO_2^- + 1/20_2$

During the nitrification process, there is an accumulation of nitrite when the oxidation reaction of ammonium (NH_4^+) is greater than that of nitrite. Baeand et al., (2001) show that in an alkaline environment, the conversion of nitrite to nitrate in wastewater is inhibited by the presence of free NH_3 or the low level of dissolved oxygen. Also, in the denitrification process there is an accumulation of nitrite when the reduction reaction of nitrate is greater than that of nitrite. Through these two processes, nitrite is likely to accumulate in the wastewater. However, due to the reducing nature of the wastewater, denitrification would best justify the high level of nitrite observed in this environment.

An effluent, very loaded with orthophosphate ions, is one of the characteristics of those generated by the cosmetics and cleaning products industries (Ritter, 1989). This wastewater is also characterized by the presence of soluble, solid and fatty organic compounds, solids and fatty whose contents are also important (Achak et al., 2011).

The COD/BOD₅ ratio is around 2; this shows that we have wastewater from the food industry, which contains elements that bacteria love. Consequently, the wastewater from the target soap factory must not be discharged into the lagoon bays without prior treatment.

Temperature and pH corrections of this water are mainly linked to the nature and thickness of the inert materials used (i.e. sand and gravel) (Kadlec and Wallace, 2008). It should be remembered that the layers of sand and gravel contained in the filtration basins have thicknesses of 30 cm and 20 cm respectively, i.e. a total filter layer of 50 cm. In this bed of materials, the raw water stays there for at least 24 hours and the ambient temperature is on average 27 ° C. The sand used is of the siliceous type. In addition, siliceous or limestonepoor soils have a pH less than 7. These different factors therefore help to refresh the water and neutralize alkaline water (Achak et al., 2011).

The reduction rate of total suspended solids is initially due to physical settling processes in the settling-digester basin. At the beginning of operation of the combined system planted, there is a low hydraulic conductivity allowing good surface distribution of the water. This leads to efficient deposition of suspended solids on the surface of the filter bed (Wallace and Knight, 2006). So, the removal of solid particles is the result of absorption and ion exchange phenomena at the solid-water interface. As the system works, the surface deposit is pierced by the rhizomes and roots newly emitted from the reed stems. This reduces the risk of filter material clogging and increases hydraulic conductivity.

In the acidic environment, most of the ammoniacal nitrogen is in its ionized form NH_4^+ (Vazquez-Rodriguez and Rols, 1997).

An earlier study revealed that the treatment of domestic wastewater with the combined vertical and horizontal subsurface flow system provides an average removal of 72.2% for N–NH₄⁺ (Zurita et al., 2009).

The evolution of the ammonium content in the combined system reveals a retention of ammoniacal nitrogen by physical processes of filtration and sedimentation (Al-Saedi and *al.*, 2018). This would limit the risk of in-depth training, but also the instant availability for the plants. Within the planted filters, there is therefore an increasing biological activity of the soil which also consumes ammonium and transforms it into nitrites (Al-Saedi et al., 2018; Fan et al., 2013): this is the nitrification process. This microbial activity is supported by the presence of plants. When the operating time of the planted system increases, ammoniacal nitrogen is better eliminated both by biological degradation and by plant assimilation (Liu et al., 2018).

A good reduction in concentration of nitrogen compounds was found in the combined system planted on emerged macrophytes, with average yields of 75% for NH_4^+ and 69% for NO_2 . The rate of nitrogen compounds removal is mainly the result of physical filtration - sedimentation mechanisms and biological degradation at the level of the filter bed (Bae et al., 2001). The combined system has therefore been shown to be effective against nitrogen compounds. Observing the evolution of the removal rates of ammonium and nitrites, it notices three phases during the process of removal of mineral nitrogen by the combined system planted. First there is the nitrification of nitrogen, then the denitrification in the second phase and finally nitrification in the third phase. In this process, the phenomenon of biological nitrification observed from the beginning of the purification device is subsequently reinforced by the phenomenon of plant assimilation (Verma and Suthar, 2018). Thus, the conditions for removing nitrogen compounds are more favorable when the reed stems start growing up.

The orthophosphates are eliminated directly, at the beginning of operation of the combined system planted, by adsorption or sedimentation on the surface of inert materials (Comeau et al., 2001; Kadlec and Wallace, 2008). The removal of phosphorus is strongly influenced by the physicochemical characteristics of the substrate used (Brix and Arias, 2005; Prochaska and Zouboulis, 2006). As plants grow, the performance of the combined system will increase due to the uptake of phosphorus in the form of phosphate ions in solution. This phenomenon is reinforced by biological transformation under the effect of microorganisms until the of the experiment. Microorganisms mineralize end phosphorus compounds, and thus make them assimilable by plants (Verma and Suthar, 2018). This assumes that more the plant biomass grows up, the phosphorus compounds are better eliminated.

After nine weeks of operation, the MBAS elimination rate peaked at 89%. During this period, the anionic surfactants are mainly eliminated by adsorption on the matrix and by biological degradation (Šíma et al., 2009). The growth of the plants was observed for a long time. After that, the rate remained virtually static until the end of the experiment. Our results confirm that the presence of vegetation improves LAS removal, with higher biomass systems associated with higher LAS removal rates (Thomas et al., 2017). The increase in the root network and rhizomes of young plants creates the best conditions for biological degradation and plant assimilation of the anionic active material (Ramprasad and Philip, 2016).

The elimination of BOD₅ by the combined system planted is due to the processes of aerobic microbial degradation and sedimentation (Choudhary et al., 2011; Zhu et al., 2014). Soluble organic compounds are removed by microbial growth on the surfaces of filter materials and, attached to the roots and rhizomes of plants (Verma and Suthar, 2018). The organic matter in wastewater contains sufficient carbon, which is consumed by a wide range of microorganisms as a source of energy.

The COD/BOD₅ ratio of the effluent wastewater to the combined planted system varied from 1.5 to 2; which indicates that wastewater is easily biodegradable.

Oxygenated environment allow aerobic bacteria to proliferate and consequently ensure better mineralization or oxidation of organic matter (Liu et al., 2018; Zhu et al., 2014). Horizontal and vertical flow planted systems have been shown to remove over 90% of the organic charge, in addition to total nitrogen and phosphorus with multiple processing steps (Luederitz et al., 2001).

An earlier study showed that the macrophytes planted system is quite effective against organic matter (BOD₅ and COD) with average removal yields of 78% for a pilot system (0.57 m^2) (Stefanakis and Tsihrintzis, 2012). In the process of organic matter removal by biological phenomena of degradation and decomposition associated with the bacterial flora, there is also an intervention of the physical phenomena of sedimentation and filtration of organic matter (Thalla et al., 2019; Verma and Suthar, 2018).

Conclusion

The present study is achieved in the aim to test the combined system planted of emerged macrophytes to intermittent flow. This system is exploited in the purification of wastewater from soap factory. The chemical and physicochemical analysis of the wastewater samples shows that it is about an alkali and very loaded sewage in organic matters of low biodegradability. The sewages of the soap factory contain as various contaminating as the strong particles, orthophosphates (PO₄³⁻), nitrogenous compounds (NH₄⁺ and NO₂⁻), fat matters and anionic surfactants.

The performance of the combined system planted has been demonstrated. And it reveals that this combined system provides filtered waters lucid having a pH very close to the neutrality. This combined system assures an important removal of phosphorus compounds, nitrogenous compounds and anionic surfactants contained in wastewaters. The application of this combined system also permits to eliminate the organic matter efficiently under shape of BOD₅ and COD.

The combined system planted of emerged macrophytes can be considered like a lasting alternative to the conventional industrial wastewater purification, permitting a reuse thus. For it, it is suggested a long enough working length to eliminate the quasi-totality of the pollutants.

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