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Full Length Research Paper

Comparative study of improved treatment of oil produced-water using pure and chemically-impregnated activated carbon of banana peels and *Luffa cylindrica*

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Produced water (PW) during oil and gas production operations contains various hazardous substances including heavy metals (HM) with adverse impact on the environment. Disposal of PW interferes with environmental sustainability, making PW treatment obligatory prior to discharge into the environment. Among previously available treatments of PW, environmentally sustainable methods using low-pore space bio-adsorbents require further development. This study investigated the efficacy of chemically-modified activated carbon (cMAC) of *Luffa cylindrica* (LC) and Banana Peel (BP) for the treatment of PW obtained from Niger-Delta oilfield, treated (2, 4, and 6 h) with finely ground (425 µm) carbonized LC and BP impregnated separately with phosphoric-acid and sodium hydroxide. The derived cMAC was characterized by proximate analysis and FTIR spectroscopy. Treated PW was analysed for HM using AAS, Langmuir, and Freundlich isotherms. Obtained values of the surface area for the cMAC from LC were 880 (NaOH), 830 (H_3PO_4) m^2/g and BP was 810 (NaOH), and 920 (H_3PO_4) m^2/g. The existence of active functional groups is revealed on the FTIR spectra. Results revealed a substantial drop in HM concentrations (Zn: 30-86%, Cu: 78-88%, Ni: 33-55%, Fe: 17-52%) in PW after treatment with cMAC at varying contact times. All metals (Zn, Cu, Ni) in the treated PW except Fe were below WHO and USEPA guideline limits. Treatment of Niger Delta oilfield PW was effectively improved with acid-modified carbonized *L. cylindrica*.

Key words: Activated carbon, adsorption, bio-adsorbent, heavy metals, oil-produced water, treatment.

INTRODUCTION

Petroleum is produced with large volumes of water (Igunnu and Chen, 2014; Nonato et al., 2018; Gbadegesin et al., 2022) typically called Produced Water (PW). Thus, the petroleum industry is characterized by prolific

wastewater generation during extraction and development activities (Beech, 2006; Udeagbara et al.,2021). Produce water constitutes the largest volume wastewater stream from oil field operations and contains large amounts of salt, scale products, and hazardous substances including polycyclic aromatic hydrocarbons (PAH), benzene, toluene, ethylbenzene, and xylene (BTEX), radioactive materials, heavy metals (HM) and others resulting in the contamination of the environment (Neff et al., 1992; Igunnu and Chen, 2014; Olajire, 2020; Udeagbara et al., 2021). Among these contaminants, heavy metals are of particular interest because of their non-biodegradability, long-term persistence in the environment, and adverse impacts on human and environmental health (Kostecka et al., 2014; Nowicki et al., 2016; Ani et al., 2019; Briffa et al., 2020). Hence, PW during oil and gas extraction and development operations contain various hazardous substances including HM with adverse impact on the environment.

In Nigeria, PW is a major environmental pollutant in areas where oil exploration and production activities are ongoing, the disposal of the PW interferes with environmental sustainability, making PW treatment mandatory prior to discharge into the environment. This has led to increase in research works on plausible treatment management options (Joel et al., 2010; Isehunwa and Onovae, 2011; Onyema et al., 2015; Ofili et al., 2015; Udeagbara et al., 2021). Whilst, many treatment technologies for the management of PW exists, efficient treatment methods that are cost-effective and environmentally sustainable remain a challenge (Nwosi-Anele et al., 2016; Nonato, 2018) and as such requires further development. Among previously available treatments of PW, environmentally sustainable methods using low-pore space bio-adsorbents require further development. Current research efforts on cost-effective methods for the removal of toxic HM and other contaminants have received increased attention (Rasheed et al., 2017; Aclione et al., 2018; Oumam et al., 2020; Udeagbara et al., 2021; Gbadegesin et al., 2022).

Currently, appropriate management strategies are not only necessary for the treatment of PW to meet specified limits of water quality required by regulatory agencies for safe discharge and/or reuse (Nwosi-Anele et al., 2016; Olajire, 2020) but also to achieve relevant Sustainable Development Goals (Clean water and sanitation, Climate Action, Life below water, and Life on land) strategic to environmental sustainability and water use (UN, 2015). In particular, a major current and future priority of PW management should be directed to developing an eco-friendly and cost-effective technology with zero pollutant discharge (Olajire, 2020).

One way to achieve the aforementioned is by treating PW using bio-adsorbents made from natural organic materials, such as agricultural wastes or other raw materials which are readily available and low-cost. Some previous studies have evaluated the effectiveness of bio-adsorbents for the treatment of PW. Recently, Udeagbara et al. (2021) reported 18 to 100% removal of HMs from PW using non-carbonized bio-adsorbents produced from organic materials. Furthermore, other studies have demonstrated improved removal efficiency of various contaminants in PW using bio-adsorbents processed into activated carbon. Studies investigating possible treatments of PW from Niger Delta oilfields have recently reported improved removal efficiency for crude oil (50 = 74%) (Akinsete and Araoye, 2021) and HM (59 - 80%) (Popoola et al., 2022) using activated carbon of bio-adsorbents. The removal efficiency of contaminants, especially HM can be further enhanced through the modification of activated carbon using activating agents such as phosphoric acid, nitric acid, hydrochloric acid, zinc chloride, sulphuric acid, potassium hydroxide, and sodium hydroxide (Jankowska et al., 1991; Kumar and Namasivayam, 2009; Reffas et al., 2010; Li et al., 2015; Rahman et al., 2015; Ani et al., 2020; Zięzio et al., 2020). The increased removal efficiency of 61 to 83% of crude oil from PW using phosphoric acid-modified activated carbon has been demonstrated (Akinsete and Araoye, 2021).

Organic materials such as banana peel, a readily available agro-waste, and sponge gourd (*Luffa cylindrica*), a fast-growing vine that produces an inedible sponge-like structure when mature are promising candidates for oilfield PW treatment. Many organic materials do not only serve as raw materials but have the advantages of availability, low-cost, and can be readily processed into activated carbon (Zięzio et al., 2020).

Efficient treatment technology that is cost-effective for PW treatment to meet regulatory standards prior to discharge and/or reuse has remained a big challenge for the oil and gas industry (Olajire, 2020). Therefore, this comparative study was conducted to assist in the identification of an efficient oilfield PW treatment technology. The study adopted the adsorption technology using non-carbonized bio-adsorbent and activated carbons (AC) prepared from *L. cylindrica* and banana peel and modified with two activating agents (H_3PO_4 and NaOH) for the removal of HMs (Cu, Fe, Ni, and Zn) and other contaminants from oilfield PW at varying time intervals.

MATERIALS AND METHODS

The PW sample (Figure 1a) was collected from the Gbetiokun oilfield of Niger Delta, Nigeria. Banana peels (Figure 1b) and dried mature fruit of *L. cylindrica* (Figure 1c) were sourced from a local market in Ibadan, Oyo State, Nigeria. All reagents used were of high analytical grade. The pH was attuned (from red colour) to desired values (blue colour) using NaOH pellets.

Preparation of adsorbents

This study investigated the efficiency of chemically-modified activated carbon (cMAC) of Banana Peel (BP) and *L. cylindrical*

Figure 1. (a) Produced water sample; (b) Banana peels; (c) *Luffa cylindrica*.
Source: Authors

(LC) for the treatment of PW.

Pre-treatment of banana peels and *L. cylindrica*

Banana peels (BP) and *L. cylindrica* (LC) were thoroughly washed with distilled water to remove dirt, cut into pieces, and air-dried for 7 days. Excess water was removed by oven-drying (100°C) for 5 h (BP) and 3 h (LC) owing to the difference in their texture. Completely dried samples of BP (ground and sieved) and LC (cut into small pieces only due to very light texture) were stored in airtight containers.

Modified bio-adsorbent

The non-carbonized bio-adsorbent of the BP and LC was pre-treated with sodium hydroxide (1.25 M NaOH) by immersing 20 g of non-carbonized bio-adsorbent in 50 mL NaOH solution at room temperature for 24 h. The alkali solution was drained out and the adsorbents were thoroughly washed with distilled water (to remove NaOH) until pH 7 was attained. The obtained adsorbent samples (MBP and MLC) were sun-dried (6 h), then oven-dried (2 h), and stored in air-tight containers.

Production of activated carbon

The ground BP and LC samples were carbonized (Figure 2) in a muffle furnace (Heraeus RO/ROF Tube Furnace) in a steady flow of nitrogen gas (N_2), at a temperature of 450°C for 1 and 0.5 h, respectively. The carbonized samples were left to cool down to room temperature. Chemical activation of the carbonized samples (CBP and CLC) was achieved by impregnation using phosphoric acid (H_3PO_4; CBPP and CLCP) and sodium hydroxide (NaOH; CBPN and CLCN) at a ratio of 1:3 for 24 h. Briefly, 40 g of activated carbon was placed in a beaker and 120 mL of the activating agent was added. After the 24 h impregnation process, the CBP and CLC were filtered and washed repeatedly with water purified by distillation (for acid and alkali removal from the pore spaces) to obtain a neutral pH. The washed chemically activated carbons of BP (CBPP and CBPN) and LC (CLCP and CLCN) were dried at 200°C for 5 and 2 h, respectively.

Characterization of activated carbons and produced water

Activated carbons were characterized for moisture content, ash content, and volatile materials using methods described by Oladimeji et al. (2021). Total and fixed carbon was determined by the method described by Isehunwa and Onovae (2011), while the bulk density and surface area were determined according to the methods described by Sugumaran et al. (2012). Additionally, the functional groups of activated carbons were determined by Fourier transform infrared spectroscopy (FTIR; PerkinElmer Spectrum BXII) which is an essential technique for determining the chemical properties of activated carbon (Rahman et al., 2015).

Moreover, the PW (untreated and treated) was characterized for total dissolved solids (TDS) and total suspended solids (TSS) according to Oladimeji et al. (2021). The concentrations of heavy metals (HM) in PW (untreated and treated) were determined using atomic absorption spectrophotometer (AAS; PerkinElmer Analyst 200).

The effect of contact time was determined by adding 1 g of activated carbon sample to 50 mL of distilled water in a bottle and placed on a mechanical shaker for different contact times (2, 4, and 6 h). After shaking, the samples were filtered and HM, TDS, and TSS were determined in the filtrate.

Adsorption batch experiments

The batch experiments for adsorption equilibrium were carried out to estimate the adsorption isotherms of metal ions onto the surface of the adsorbents used in this study. Equation 1 gives the quantity of adsorbed metal ion at time t:

$$Q_t = \frac{(C_o - C_t) \times V}{w} \qquad (1)$$

While Equation 2 gives amount of metal ion adsorbed at equilibrium conditions $q_t = q_e$ and $c_t = c_e$:

$$Q_e = \frac{(C_o - C_e) \times V}{w} \qquad (2)$$

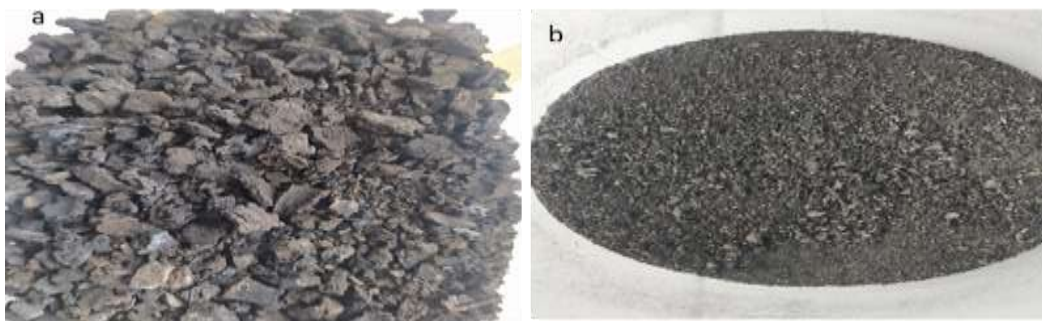The percentage adsorption is given by Equation 3:

Figure 2. Carbonized: (a) banana peels; (b) *Luffa cylindrical.*
Source: Authors

$$\% \; Adsorption = \frac{(C_o - C_e)}{C_o} \times 100 \tag{3}$$

Adsorption isotherms

In this study, equilibrium isotherms were used to describe the experimental bio-sorption data. The equations of Langmuir and Freundlich are the most frequently used to describe experimental data of adsorption isotherms. The parameters and assumptions of these equilibrium isotherms continually provide certain acumen into the sorption mechanism, the surface properties, and the affinity of the sorbent (Langmuir, 1917).

The Langmuir isotherm

This Langmuir isotherm correlates the amount of extracted material to its equilibrium concentration in the majority of solutions. Langmuir (1971) showed that the model is only acceptable for monolayer adsorption. Langmuir isotherm model (Equation 4) assumes uniform adsorption on the surface and transmigration in the plane of the surface.

$$\frac{C_e}{q_e} = \frac{1}{bk_L} + \frac{1}{b}C_e \tag{4}$$

where q_e signifies adsorption capacity at equilibrium in mg/g, Ce is the equilibrium concentration (mg/L), k_L is the Langmuir constant in mL/mg and b is the adsorption capacity of the substrate (gram solute/gram adsorbent). Plotting Ce/qe against Ce gives a straight line of slope 1/b and an intercept $1/bk_L$ that is equal to homogenous and the adsorption energies to be equivalent for each adsorption site. The key features of the Langmuir isotherm given in a dimensionless equilibrium parameter R_L are defined by Equation 5:

$$R_L = \frac{1}{1 + bC_o} \tag{5}$$

where Co is the original solute concentration, b is the Langmuir's adsorption constant (L/mg) and R_L value gives the grouping of the isotherm to be either unfavorable (RL>1), linear (RL =1), favorable (0< R_L <1) or irreversible (R_L = 0) (El-Nafaty et al., 2013).

The Freundlich Isotherm

This is an empirical model describing the adsorption process

occurring on multilayer (that is, heterogeneous) surfaces (Freundlich, 1906); the model (Equation 6) suggests that adsorption capacity is correlated to the adsorbent concentration (Udegbara et al., 2021).

$$\ln q_e = \ln k_f + \frac{1}{n} \ln C_e \tag{6}$$

where qe represents the equilibrium concentration (mg/g), Ce denotes equilibrium concentration in solution (mg/L), k_f signifies the Freundlich adsorption isotherm constant and 1/n indicates the heterogeneity of the data distribution which is correlated to the degree of the adsorption driving force (Konggidinata et al., 2017).

RESULTS AND DISCUSSION

Adsorbent characterization

Characteristics of the adsorbent after the activation with H_3PO_4 and NaOH are shown in Table 1. The total ash content ranging from 2.8 to 3.9% indicates that the produced adsorbents contained high amounts of organic materials. Additionally, the high carbon content indicated that the biomass used in this study could serve as a good bio-adsorbent in adsorption studies. The bulk density of the chemically activated carbons (0.22 – 0.32 g/cm^3) are similar to the values obtained for phosphoric acid impregnated carbonized plantain stem (0.32 g/cm^3) (Ekpete et al., 2017) but slightly lower than (0.39-0.56 g/cm^3) phosphoric acid impregnated carbonized spent coffee grounds (Zięzio et al., 2020). The obtained values of the surface area for the chemically activated carbons from BP were 810.0 (NaOH) and 920.0 (H_3PO_4) m^2/g and from LC were 880.0 (NaOH) and 830.0 (H_3PO_4) m^2/g.

The large surface area exhibited by the chemically activated carbons in this study is higher than (Ramutshatsha-Makhwedzha et al., 2022) but comparable (Zięzo et al., 2020) to those reported in the literature for activated carbons using similar activation agents. Generally, a larger surface area indicates better-developed porosity for the increased adsorption process (Zięzio et al., 2020; Udeagbara et al., 2021).

Table 1. Chemical characteristics of produced adsorbent.

| Parameter | Activated Carbon | | | |
| | Banana peel | | Luffa cylindrical | |
	NaOH	H_3PO_4	NaOH	H_3PO_4
Moisture Content (%)	8.03000±0.15	8.40000±0.10	8.23000±0.12	7.97000±0.15
Volatile Matter (%)	0.23000±0.06	0.80000±0.10	0.33000±0.06	0.60000±0.10
Total Carbon (%)	93.83000±0.32	86.53000±0.29	88.20000±0.17	90.97000±0.31
Total Fixed Carbon (%)	81.70000±0.20	74.57000±0.21	76.17000±0.15	79.17000±0.15
Total Ash (%)	3.87000±0.25	2.77000±0.15	3.80000±0.79	3.23000±0.25
Bulk Density (g/cm^3)	0.22000	0.32000	0.31000	0.28000
Surface area (m^2/g)	810	920	880	830

Mean ± Standard Deviation.
Source: Authors

FTIR spectroscopy analysis of adsorbents

The Fourier Transform Infrared (FTIR) spectra of the chemically activated carbons, recorded between 4000 and 400 cm^{-1} are as shown in Figure 3. Varying absorption peaks were displayed on the spectra of the activated carbons, suggesting the presence of active functional groups that will increase the removal of HM from PW (Popoola et al., 2022). In this study, all activated carbons revealed common board peaks from 1628.21 to 1637.94 cm^{-1} corresponding to the C=O stretch of amine (Ekpete et al., 2017). The defined peaks ranging from 3833.98 to 3885.29 cm^{-3} were assigned to the O–H stretch of alcohol. This range is similar to values previously reported for activated carbons from banana waste (Sugumaran et al., 2012). The presence of the hydroxyl (OH) group observed in all the activated carbons has been reported to be important for the adsorption of metals (Ramutshatsha-Makhwedzha et al., 2022). The bands observed around 1053 and 1080 cm^{-1} can be associated with C–O stretching vibrations of carboxylic groups. Similar FTIR spectra were observed for the different activating agents regardless of the carbonized material. Sodium hydroxide impregnated activated carbons had similar bands between the regions of 550 and 3500 cm^{-1}, while the bands between 500 and 2800 cm^{-1} were similar for phosphoric acid impregnated activated carbons. Additionally, the phosphoric acid activated carbons displayed pronounced bands within the region of 902.32 to 995.24 cm^{-1} corresponding to the P–H band of phosphine, which is absent when the activating agent was sodium hydroxide.

Produced water treatment

Table 2 shows the adsorption of heavy metals; Zinc (Zn), Nickel (Ni), Iron (Fe), and Copper (Cu) from produced water using the prepared activated carbons. The PW used in this study contained higher concentrations of HMs than reported in recent studies from the Niger Delta area of Nigeria (Udeagbara et al., 2021; Popoola et al., 2022). The results obtained demonstrated the efficiency of the bio-adsorbent in removing heavy metals. The results revealed a substantial decrease of heavy metal concentrations present in produced water after treatment with the prepared activated carbons at varying contact times. The pattern of removal efficiency of HM was 2 < 4 < 6 h, indicating the highest removal efficiency was achieved at 6 h (Figure 4). This confirms that increasing contact time improves the effectiveness of HM removal by bio-adsorbents (Udeagbara et al., 2021). At 6 h, higher Zn (86%) and Cu (88%) were removed via the phosphoric acid impregnated LC activated carbon compared to corresponding 9 and 85% for NaOH impregnated BP activated carbon. Overall, CLCP was most effective in the removal of all HMs compared with the other activated carbons. Only Ni was completely removed (100%) at 4 and 6 h using banana peel modified activated carbon (MBP), thus demonstrating the high adsorption capacity of Ni. Again, at 6 h, the most adsorbed HMs by the bio-adsorbents were Cu, corresponding to 61 to 88% removal, while the least was Fe corresponding to 2 to 52% removal. While all the prepared bio-adsorbents demonstrated effective removal of Cu, only those prepared from LC adsorbed Fe better. This confirms that locally prepared activated carbon has a strong affinity to removing heavy metals from PW at varying contact times (Popoola et al., 2022). At 6 h, the modified and chemically activated carbons are better adsorbents for Zn, Cu, and Ni when compared with non-carbonized adsorbents prepared from similar materials used in a related study (Udeagbara et al. 2021). All metals in the treated PW except Fe were below the guideline limits by the regulatory bodies (Table 2).

Analysis based on adsorption isotherms

To study the connection between adsorption capacity
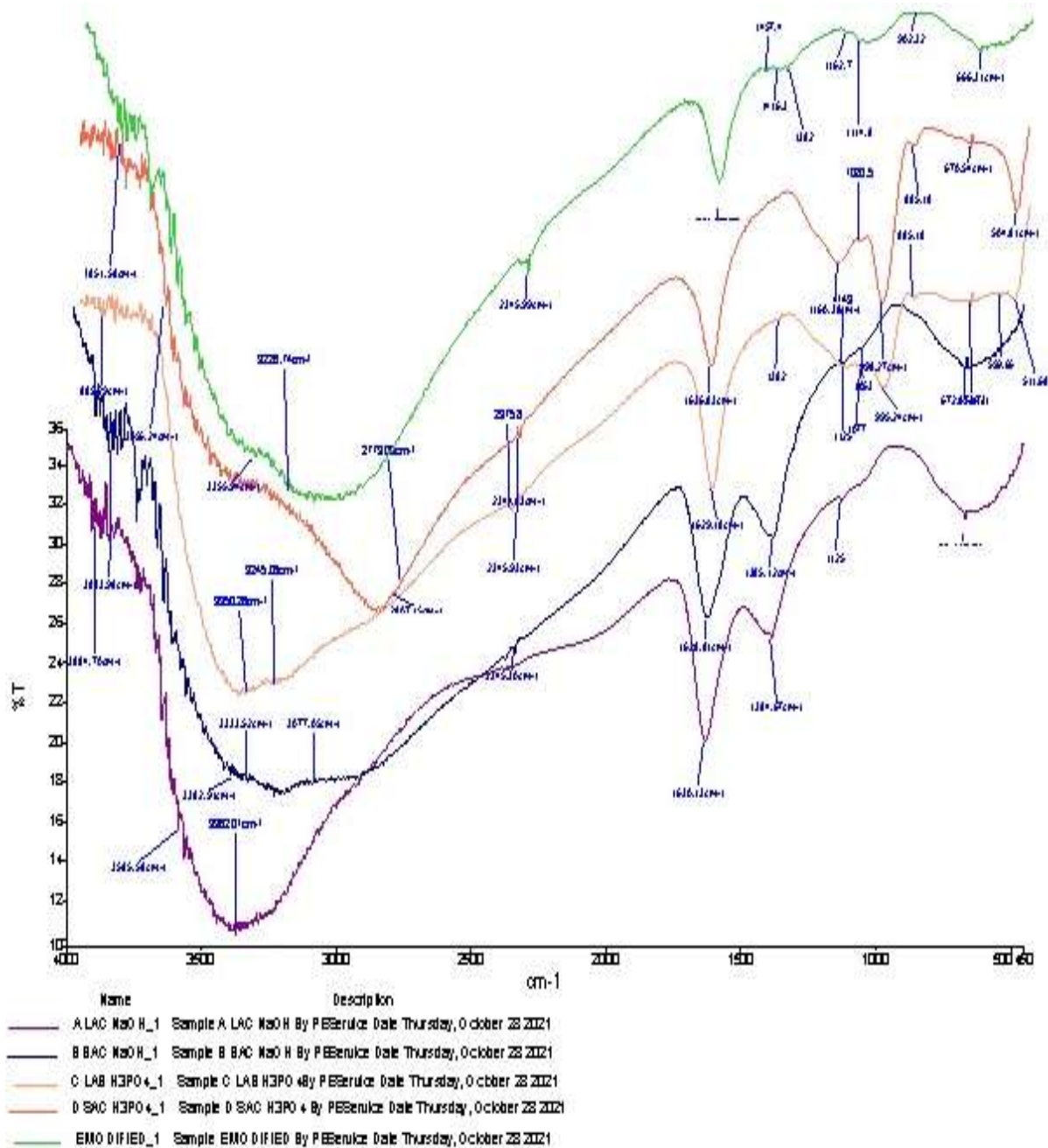
Figure 3. FTIR spectrum of adsorbents.
Source: Authors

(*qe*) and aqueous concentration (*Ce*) at equilibrium, Langmuir's adsorption isotherm reported by Naseem et al. (2019), Ramutshatsha-Makhwedzha et al. (2022) and Freundlich's adsorption isotherm stated by Rasmey et al. (2018), Ramutshatsha-Makhwedzha et al. (2022) models were deployed to fit the equilibrium data in this study. Table 3 presents the correlation coefficient for the adsorbents using the Langmuir and Freundlich isotherm models. Results of the dimensionless equilibrium parameter (R_L) revealed that the sorption of Zn, Cu, Ni,

and Fe on the adsorbents is satisfactorily good as values fall between the favorable range of $0<R_L<1$. The adsorption of Zinc by *L. cylindrica* (LC) favored multilayer as agreed by Freundlich isotherm, while adsorption of Zinc by Banana Peel (BP) favored monolayer as agreed by Langmuir. Results also showed that the Langmuir isotherm is good for the equilibrium study of the adsorption of Copper by *L. cylindrica* and banana peel, which suggests homogeneous treatment of the adsorbate on the adsorbent surface for the Copper ion. Nickel and

Table 2. Concentrations of metal (mg/L) in produced water before and after treatment.

Treatment type	Time (h)	Metal concentration (mg/L)				Parameters	
		Zn	Cu	Ni	Fe	TDS	TSS
Raw PW	-	0.25800	0.14400	0.30000	3.60000		
MLC	2	0.18800	0.06100	0.10800	3.28900	44.00000	50.00000
	4	0.16500	0.05800	0.08300	2.76000		
	6	0.04800	0.04500	0.08200	1.72000		
CLCP	2	0.18100	0.03100	0.20000	3.00200	14.33000	36.40000
	4	0.05500	0.01900	0.17200	2.43800		
	6	0.03700	0.01700	0.13600	1.72500		
CLCN	2	0.20100	0.09300	0.28900	3.44200	14.00000	36.40000
	4	0.19900	0.09000	0.23800	3.36900		
	6	0.15400	0.04900	0.21100	3.05200		
MBP	2	0.22100	0.03100	0.12500	3.43100	16.00000	38.80000
	4	0.16200	0.02100	0.00000	3.44300		
	6	0.07500	0.02000	0.00000	3.43400		
CBPP	2	0.25800	0.07200	0.16400	3.48500	10.00000	37.60000
	4	0.24500	0.05800	0.11100	3.29600		
	6	0.23500	0.02100	0.08800	3.52200		
CBPN	2	0.25700	0.14000	0.26600	3.34000	13.33000	35.60000
	4	0.17100	0.11300	0.25200	3.33600		
	6	0.16800	0.05600	0.15200	3.33000	17.33000	39.20000
EGASPIN[†]	-	5.00000	1.00000	-	1.00000	<2000	30
USEPA[††]	-	0.02000	1.30000	0.30000	0.30000	<4.00000	1200
SDL[†††]	-	1.00000	0.02000	0.03000	1.00000	-	-

[†]EGASPIN (2018); [††]USEPA (2018) – United States Environmental Protection Agency;
[†††]SDL – Standard disposal level.
Source: Udeagbara et al. (2021)

Iron give the Freundlich-type model a better result than the Langmuir model, which gives an indication of multilayer coverage of the adsorbate on the adsorbent surface for the ions.

Conclusion

Modified and chemically activated carbons were prepared from banana peel and *L. cylindrica* for the treatment of produced water at different contact times. All prepared activated carbons were characterized by large surface area and the presence of active functional groups was revealed on the FTIR spectra. The bio-adsorbents showed improved surface area.

The efficiency of prepared activated carbons at varying contact times, indicating the highest HMs removal was achieved at 6 h (2 < 4 < 6 h). Overall, phosphoric acid impregnated activated carbon of *L. cylindrica* was most effective in the removal of all heavy metals compared with the other activated carbons. Copper was the most adsorbed heavy metal by the bio-adsorbents while the least adsorbed was iron. While all the prepared bio-adsorbents demonstrated effective removal of copper, only those prepared from *L. cylindrica* adsorbed iron better.

Results further showed that Langmuir isotherm gave the best fit for Copper and Zinc, while Freundlich isotherm gave the best fit for Iron and Nickel for *L. cylindrica* while Freundlich isotherm gave the best fit for Iron and Copper and Langmuir isotherm gave the best fit for Nickel and Zinc for Banana Peel. Treatment of Niger Delta oilfields PW was effectively improved with acid-modified *L. cylindrica* activated carbon.
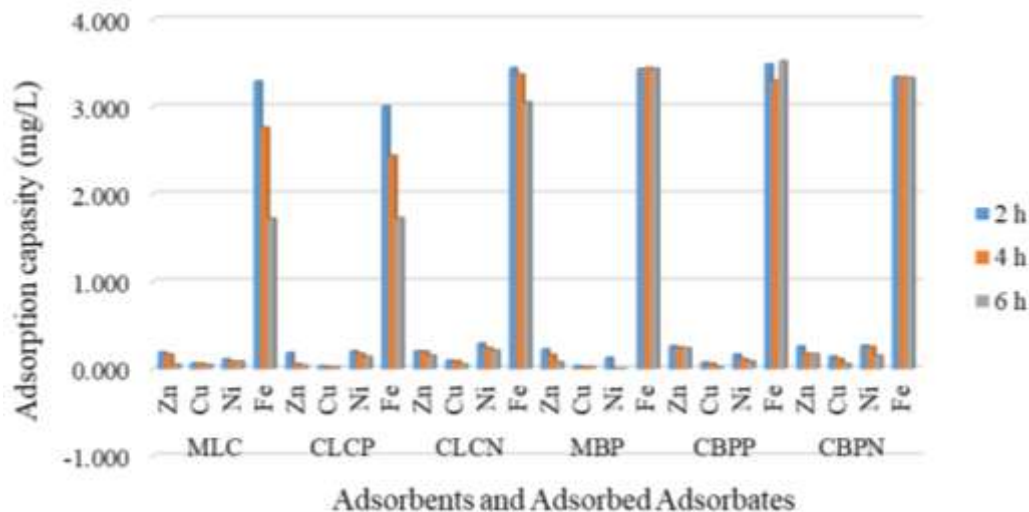
Figure 4. Contact time effect on the adsorption of heavy metals from produced water by modified and chemically impregnated carbonized adsorbents.
Source: Authors

Table 3. Isotherms analysis on the rate of metal adsorption by the different adsorbent.

Metal	Adsorbent	Langmuir			Freundlich		
		b	R_L	R^2	k_f	N	R^2
Zinc	MLC	0.00310	0.99920	0.95160	0.00110	-1.33941	0.97300
	CLCP	0.00319	0.99918	0.99470	0.00120	-1.43123	0.97150
	CLCN	0.00117	0.99970	0.99910	0.00008	-0.44719	0.99970
	MBP	0.00138	0.99965	0.83900	0.00030	-0.73692	0.88140
	CBPP	0.00006	0.99999	1.00000	6.28924E+43	0.01329	0.74300
	CBPN	0.00002	1.00000	0.99910	2.25339E-11	-0.09296	0.99910
Copper	MLC	0.00288	0.99958	0.99910	0.00084	-1.74980	0.99870
	CLCP	0.00496	0.99929	0.99980	0.00284	-5.05050	0.99760
	CLCN	0.00172	0.99975	0.99760	0.00027	-1.05030	0.99860
	MBP	0.00485	0.99930	1.00000	0.00269	-4.66850	0.99500
	CBPP	0.00318	0.99954	0.98280	0.00128	-2.44620	0.97250
	CBPN	0.00014	0.99998	0.63590	1.22815E-06	-0.34430	0.78380
Nickel	MLC	0.00696	0.99792	1.00000	0.00343	-2.16076	1.00000
	CLCP	0.00276	0.99917	0.98000	0.00067	-0.79183	0.98470
	CLCN	0.00015	0.99995	0.91660	1.22887E-07	-0.14576	0.94950
	MBP	0.00875	0.99738	1.00000	0.00002	0.00875	1.00000
	CBPP	0.00471	0.99859	0.99170	0.00185	-1.37627	0.98420
	CBPN	0.00094	0.99972	0.92810	0.00007	-0.40515	0.98100
Iron	MLC	0.00004	0.99987	0.99980	1764264982	-0.04730	0.99990
	CLCP	0.00037	0.99868	0.91660	193347568.4	-0.05130	0.98270
	CLCN	0.00098	0.99650	0.99990	50312.05553	-0.07950	1.00000
	MBP	0.00890	0.96895	0.79350	0.40694	-0.39186	0.98200
	CBPP	0.01591	0.94584	0.90810	0.29449	-0.50161	0.94520
	CBPN	0.00133	0.99524	0.92250	1645.50071	-0.10156	0.90150

Source: Authors

The prepared activated carbons have the ability to effectively treat produced water contaminated with Cu, Zn, and Ni. The efficiency of heavy metal removal demonstrated in this study confirms that locally prepared activated carbon enhanced with activating agents is an environmentally sustainable method for the treatment of produced water from the oil industry.

ABBREVIATIONS

AAS, Atomic absorption spectrophotometer; **BP,** banana peel; **CBP,** carbonized banana peel; **CBPN,** sodium hydroxide impregnated carbonized banana peel; **CBPP,** phosphoric acid impregnated carbonized banana peel; **CLC,** carbonized *Luffa cylindrical*; **CLCN,** sodium hydroxide impregnated carbonized *Luffa cylindrical*; **CLCP,** phosphoric acid impregnated carbonized *Luffa cylindrical*; **Cu,** copper; **cMAC,** chemically modified activated carbon; **Fe,** iron; **FTIR,** Fourier transform infrared; **HM,** heavy metals; **LC,** *Luffa cylindrical*; **MBP,** modified banana peel; **MLC,** modified *Luffa cylindrical*; **Ni,** nickel; **PW,** produced water; **TDS,** total dissolved solids; **TSS,** total suspended solids; **UN,** United Nations; **Zn,** zinc.

CONFLICT OF INTERESTS

The authors have not declared any conflict of interests.

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Full Length Research Paper

Biological removal of iron from groundwater in a pilot column on a filter support material with coated jujube seeds

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A co-flow pilot filter with a packing support material (coated jujube seeds) was constructed and tested for the biological removal of iron from Senegalese groundwater. Indeed, it is a PVC tube with an internal diameter of 40 mm and a height of 90 cm. The performance of the filter was tested at a constant hydraulic head of 261.67 m^3/m^2 per day while the iron feed concentrations varied between 0.5-1, 0.5-1.5 and 0.5-2 mg/L, respectively. Coated jujube seeds, covered with a plastic layer and a biofilm containing microorganisms, were used as the filter material. The iron removal was carried out in a single stage, for which a period of two months was required for the maturation of the bed. In optimal working conditions (Q_L = 8.11 L/h; pH = 9.5; $[Fe^{2+}]_0$ = 2 mg/L; P = 1.013 bar and T = 25±1° C; Redox potential = 300 mV), an operating cycle time of 1 day has proven to be sufficient for the filter bed backwash operation. The results show that the biological treatment gives reduction percentages of 39.3, 48.76 and 67.23%, respectively for the boreholes of Pout Kirène (PK2), Pout Sud (PS5) and Koungheul (KK5).

Key words: Biofilm, continuous test, biological oxidation, iron removal, column, doped water.

INTRODUCTION

Iron is present in some sites in drinking water intended for the consumption of the Senegalese population, at concentrations that exceed WHO water quality standards (0.3 mg/L) (Ahmed and Mir, 2022). Dissolved oxygen precipitates Fe(II) to Fe(III) and imparts a reddish color, metallic taste and undesirable odor to the water, domestic softeners become clogged with Fe (III) precipitates, the formation of precipitates in the pipes reduces the inside diameter and increases energy losses. When the ferrobacteria formed in the pipes die and disappear, bad odors and unpleasant tastes can be caused. Iron oxidation rates increase with the amount of oxygen

present in the medium, pH and redox potential can reach conversion rates of around 90% at pH=7 and Redox potential = 400 mV (Elliott et al., 2014). Oxidation of iron is markedly slower at pH<6 and precipitates may persist for some time in aerated waters. Oxidation yields can be increased by the presence and action of certain microorganisms (Bray et al., 2017; Nayyeri et al., 2021). There are several kinds of bacteria that oxidize dissolved Fe (II) by different mechanisms. Indeed, microorganisms in particular *Leptothrix ochracea*, *Crenothrix polyspora* and *Gallionella* species can cause biological oxidation if the operating conditions are established.

The aeration operation followed by solid-liquid separation on a sand bed is the most widely used chemical treatment method in groundwater treatment plants in Senegal. Michalakos et al. (1997) have shown in their scientific work that aeration is the adequate method for the oxidation of iron in groundwater where concentrations are greater than 5 mg/L to avoid chemical costs and the complexity of technology. Separation from liquid to solid phase is possible using sand filters. If necessary, other treatments can be added, such as chemical oxidation with chlorine (Vayenas and Lyberatos, 2005), potassium permanganate or ozone, ion exchange processes and biological oxidation (Cai et al., 2015).

In this work, the treatment method that relied on a continuously operated fixed bed column to remove dissolved Fe (II) was used. The column was filled with plastic coated jujube seeds with an average diameter of 8.08 mm, used as the filter material. The limits between the physical, chemical and biological removal of iron are not identified in this work. Nevertheless, the conditions in the field of biological oxidation are fixed. Experimental tests are carried out on the determination of the bed backwashing operating cycles, the oxidation rates of Fe (II), the effect of the Fe (II) concentration, the influence of the height of the filtration bed and the characterization of the withdrawn sludge, resulting from the biological treatment of groundwater.

MATERIALS AND METHODS

The biological filter used at the pilot scale is a non-transparent PVC tube with an internal diameter of 40 mm and 90 cm in height with an ascending type supply. The height of the pilot column is half that of full-scale industrial filters measuring on average 1.80 m (Katsoyiannis and Zouboulis, 2004). Along the depth of the filter there are 3 orifice ports for the determination of the iron content which passes through the bacterial bed. The aeration takes place at the start of the process by a *MECAFER* type air compressor at a constant flow rate of 760 L/h (Figure 1). Once the pseudo-continuous speed is reached, the air supply to the compressor is cut off. The natural air draft mode is used and no external source of mechanical ventilation is used in the biofilter. The advantage of using this type of biofilter is that it does not always require an external air supply, because air enters naturally through the filter due to the temperature gradient between the inside and the outside of the biofilter.

A hydraulic head of 261.67 m^3/m^2 per day was used in the experimental work to avoid flooding the biofilter. Water doped with iron to filter through biological filter body, could flow in the same direction as compressed air. Backwashing of the filters was necessary due to the clogging of the pores due to the growth of biomass and the deposition of iron precipitates on the surfaces of the coated jujube seeds. The backwashing was performed, according to operating conditions, using high flow velocities of air flow. Before to sampling, the biofilter was backwashed with a solution of 1 mg/L concentration and then operated in continuous mode while maintaining the feed conditions for at least 24 h, to ensure pseudo-stable conditions, to stabilize the distribution of the biofilm population as much as possible (Tekerlekopoulou et al., 2008). A syringe was immersed up to the center of the filter for the collection of water samples. The biological filter is filled with coated jujube seeds with an average diameter of 8.08 mm, a specific surface area (A_s) of 548.13 cm^2/cm^3 and a porosity of 0.423. On this support material, microorganisms responsible for removing iron from the water to be treated are cultivated (fixed culture), and were used for several days (2 months). In all the experiments, the water temperature is practically constant (25± 1°C). The oxygen dissolved in the liquid over the entire depth of the filter was between 6 and 8 mg/L. We have also tried to vary the redox potential from 200 to 400 mV, to be sure of being in the domain of biological iron removal. Once the shake flask culture reached the stationary phase (measured by a wavelength of λ_{max} = 530 nm), the suspension was transferred to the bioreactor. The volume of cell suspension added to the bioreactor was 1 L (Zhang et al., 2002).

To start the experiments, a start-up time of 10 days was necessary for the maturation of the biofilter. As earlier stated in batch mode after 10 days, microscopic observations were made. During continuous operation, solutions were added at iron concentrations between 0.5 and 2 mg/L for the column feed. The feed solution was the result of mixing a solution of ferrous iron prepared from iron sulfate (containing small amounts of ammonia and phosphorus) (Tekerlekopoulou et al., 2006).

For the laboratory experiments of this study based on monitoring the kinetics of biological degradation of iron, the measurement technique by UV-Visible spectrophotometry was used, in particular the ortho-phenanthroline method. The total microflora count in the sludge sample was made on the nutrient agar medium (GN), the number of germs in our sample was determined according to the following rule:

$$N = \frac{n}{d.V} \tag{1}$$

where N: number of microorganisms in CFU/mL; n: number of colonies counted; V: volume sampled (0.1 mL); d: dilution.

RESULTS AND DISCUSSION

For the tests, we chose to work under operating conditions with a volumetric flow rate of the sample 8.11 L/h and an air supply flow rate Q_G = 760 L/h to avoid entraining the ferrobacteria cultivated and to have a good residence time. However, different flow rates will be tested in order to gauge the efficiency according to the feed flow rates (Sample-Air). This is because the pilot scale ascending filter was kept in continuous operation for two and a half months to ensure its stable state and the development of the biofilm on the outer walls of the coated jujube seeds.
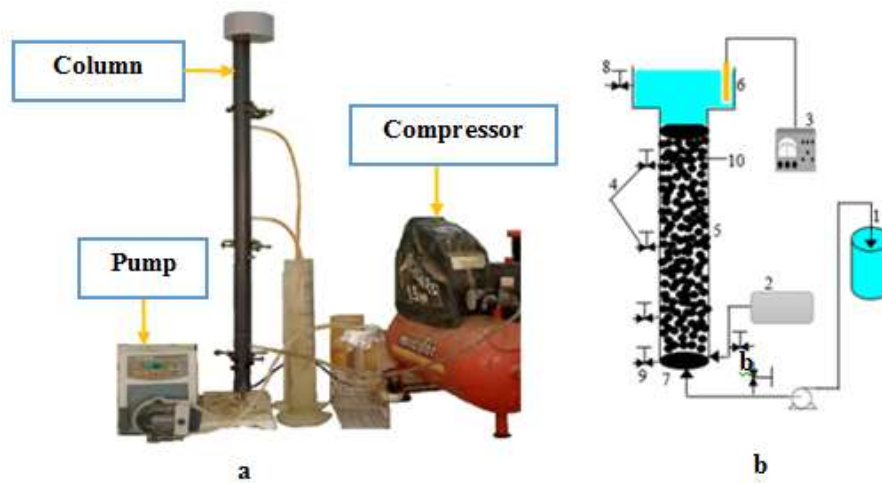
Figure 1. (a) Experimental setup and (b) general diagram.
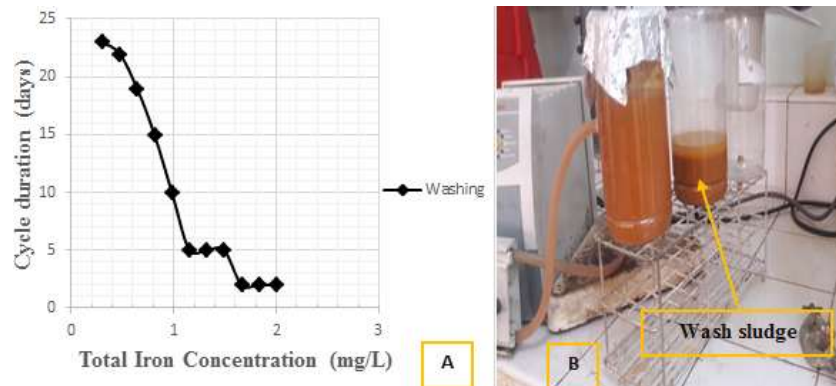Source: Authors

Figure 2. **A**-Required backwashing frequency (cycle time) as a function of iron concentration; **B**-photo: washing sludge from the filtration bed; Sample feed rate Q_L = 8.11 L/h; Air supply flow Q_G = 760 L/h; $[Fe^{2+}]_{0(max)}$ = 2 mg/L; pH = 9.5; P =1.013 bar; T = 25 ± 1°C; Redox potential = 300 mV.
Source: Authors

In all of the experiments, the water temperature was almost constant (25±1°C), as was the ambient temperature in the laboratory. The pH in the liquid phase over the entire depth of the filter is between 9.5 and 10. The redox potential considered is 300 mV in this study with a dissolved oxygen concentration between 6 and 7.5 mg/L. Under these operating conditions, and since the measured redox potential varies from 0.2 to 0.4 V, the oxidation of biological iron is predominant.

Determination of bed backwashing operating cycles

Once the seeding is done and the continuous mode

established, the inlet flow rate of the water to be treated is set at 8.11 L/h. For iron concentrations of 0.3 to 0.6 mg/L, no backwashing was necessary (Figure 2). It appears that a state of equilibrium has been established in the biofilter. For higher iron concentrations, the duration of the operating cycles becomes short. For 2 mg/L, an operating cycle time of one day was found to be sufficient, while the iron concentration at the top of the column was approximately 0.983 mg/L (limited operation). These experiments verify the work of Michalakos et al. (1997) which showed that generally, an increase iron concentration decreases the required duration of the operating cycles. At the exit of the biofilter, suspended solids or iron precipitates of small sizes could
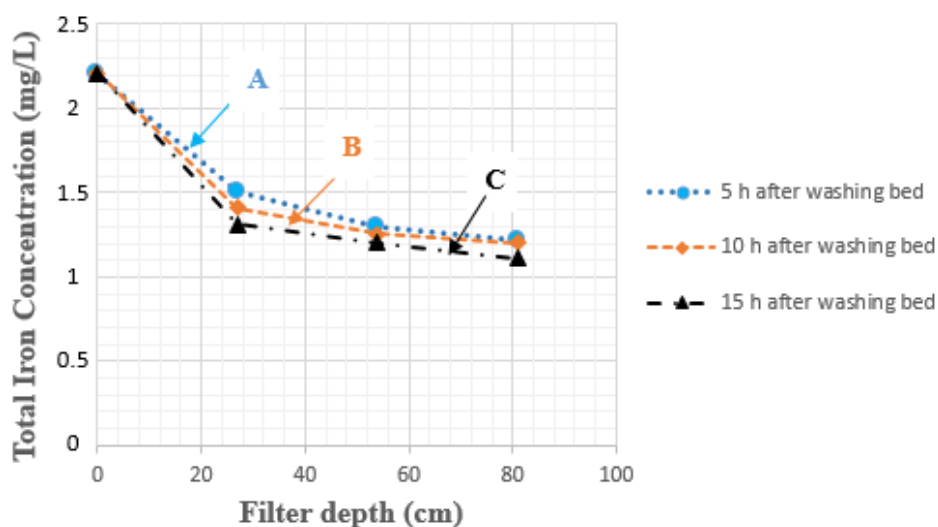
Figure 3. Iron concentration profiles in the biofilter: (**A**) 5 h after backwashing; (**B**) 10 h after backwashing; (**C**) 15 h after backwashing; Air supply flow Q_G = 760 L/h; Sample feed rate Q_L = 8.11 L/h; $[Fe^{2+}]_0$ = 2 mg/L; pH = 9.5; P = 1.013 bar and T = 25±1°C; Redox potential = 300 mV.
Source: Authors

be observed, while the end of each cycle is related to a decrease in outlet volume flow rate at the top of the column. This was used as a criterion to start the backwash of the biological filter.

Iron concentration profiles in the biofilter

The biologically active filter for iron removal, previously used in experiments for determining bed backwash duty cycles, was used to assess iron concentration profiles in the biofilter at different dates after backwashing of the bed filtration. Monitoring the pilot column will allow us to study the iron removal efficiency over the 24 h period of continuous operation. The effectiveness of the biofilter is illustrated in Figure 3 for a theoretical initial concentration of 2 mg/L. At the start of a filtration cycle (5 h after backwashing), the iron concentration decreases but well below the limit set by WHO for drinkability recommendations. This profile is practically constant after 15 and 20 h of filter operation, thus showing a tendency towards saturation of the biofilter.

Iron concentration profiles in the filter at different air flow rates

Monitoring of the filtration cycle (total concentration of iron at the outlet of the 3 intake points of the column) at different air supply rates (760, 900 and 1200 L/h) for a

supply rate of l the constant sample and equal to Q_L=8.11 L/h a shown, even at high air flow rate, the iron concentration in the collected sample decreases with the height of the bed (Figure 4). This has decreased and is slightly approaching the standard value recommended by the WHO at a filter height of 80 cm for a residence time of 24 h and a flow rate Q_G = 760 L/h. A slight shift in concentration profiles is observed with increasing air supply flow rates (900 and 1200 L/h). However, for an air flow greater than 1200 L/h the pilot column is unstable with the abundant presence of precipitates formed in the sample collected at the top of the column. These results are similar to those of Benjwal and Kar (2015) and Benjwal et al. (2016) which were obtained following the study of the iron concentration profiles in the biofilter they designed. Thus, since the feed rate supplied by the air compressor Q_G =760 L/h seems to be more adequate for the proper functioning of our pilot column, the biological treatment of iron was carried out at this rate.

According to the work of Štembal et al. (2005), continuous air aeration is important for the correct operation of a co-current biofilter. Tekerlekopoulou and Vayenas (2007) showed that the main factor responsible for airflow in an open-top biological filter is natural draft. The driving force behind the air flow is caused by the temperature difference between the ambient air and the air inside the pores of the filter bed. Thus, the use of an ascending filter has the advantage of not requiring an external air supply or a continuous ventilation system (air compressor).
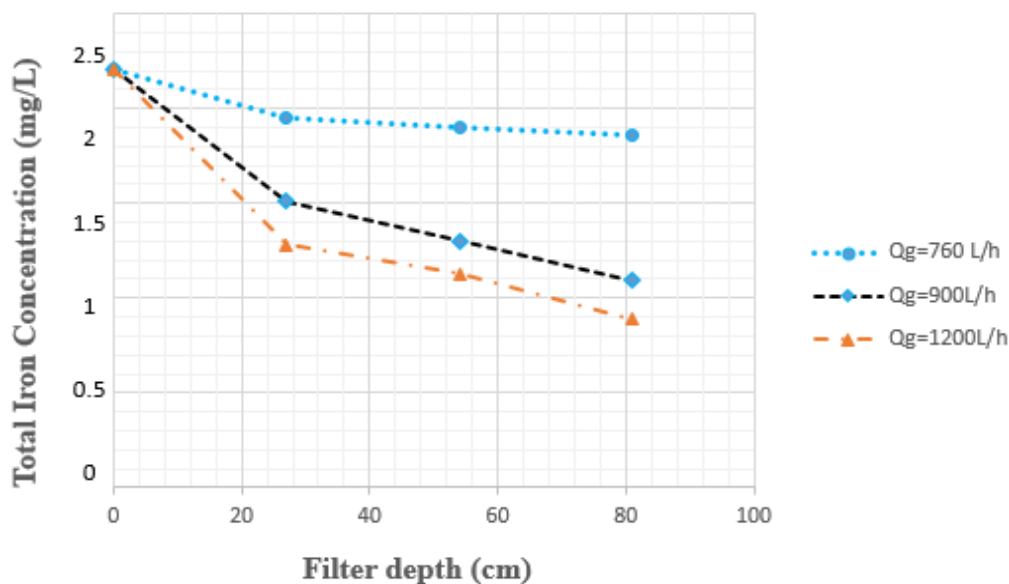
Figure 4. Iron concentration profiles in the biofilter at different air flow rates; Sample feed rate Q_L = 8.11 L/h; $[Fe^{2+}]_0$ = 2 mg/L; pH = 9.5; P=1.013 bar; T=25±1°C; Redox potential = 300 mV; t = 24 h.
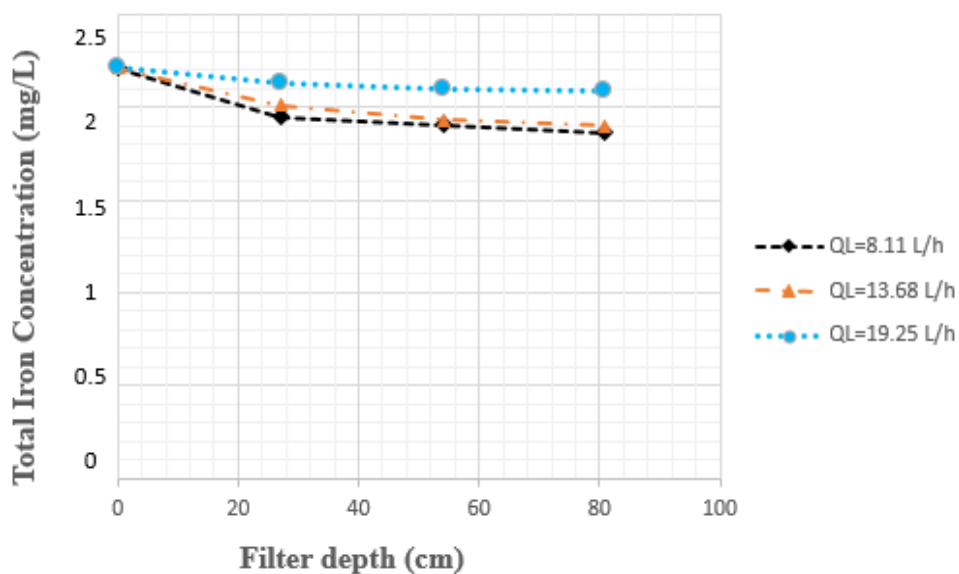Source: Authors

Figure 5. Iron concentration profiles in the biofilter at different sample flow rates; Air supply flow Q_G=760 L/h; $[Fe_{2+}]_0$ = 2 mg/L; $[O_2]$ = 6.5 mg/L; pH = 9.5; P =1.013 bar; T=25 ± 1°C; Redox potential = 300 mV; t =24 h.
Source: Authors

Iron concentration profiles in the filter at different sample flow rates

Figure 5 shows the evolution of the iron content in the column at different volumetric flow rates. Unlike studies of iron concentration profiles in the biofilter at different air flow rates, we noticed, for a constant air supply rate Q_G=760 L/h, that the increase in the supply rate of the
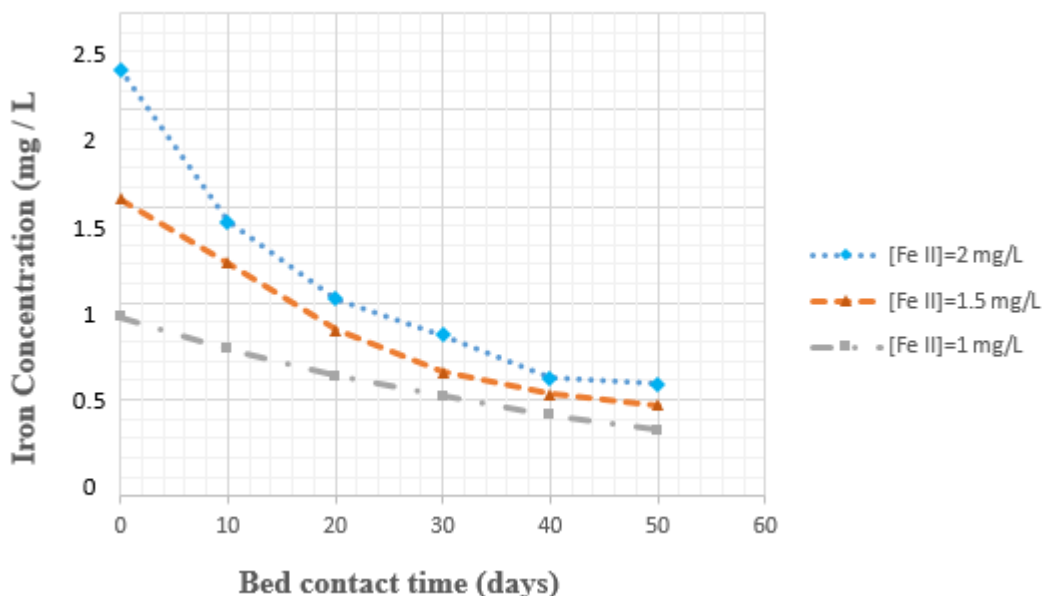
Figure 6. Biological removal of Fe (II) as a function of contact time at different iron concentrations; Q_L = 8.11 L/h; Q_G = 760 L/h; $[O_2]$ = 6.5 mg/L; pH = 9.5; P=1.013 bar and T = 25 ± 1°C and Redox potential=300 mV.
Source: Authors

sample decreases iron removal rates. This is because increasing the sample flow rate decreases the residence time in the biological bed. This study confirms that Q_L = 8.11 L/h is more adequate for proper operation of the pilot column.

The experimental data of the current study are in very satisfied agreement with the prediction of the model that was developed in the studies of Štembal et al. (2005). Their research showed that an increase in volumetric flow reduces the efficiency of the filter. In particular, for a feed iron concentration of 2 mg/L, an increase in flow from 1000 to 2000 mL. min^{-1} reduces filter efficiency from 100 to 81% (Tekerlekopoulou et al., 2006).

Biological oxidation kinetics of iron

It is established that iron can be removed from groundwater through the application of biological processes (Katsoyiannis and Zouboulis, 2004). It is also documented that iron oxidation rates are much higher in the process of chemical removal. Iron removal was compared to values reported in the literature in this study. These experiments were performed by adding Fe (II) to distilled water, at different initial iron concentrations (Figures 6 and 7).

According to the experimental data (Figure 6), the effect of varying the initial iron concentration improves the efficiency of the biofilter by approximately 5.84 to 4.47%

under the particular experimental conditions and concentration range of 1 to 1.5 mg/L and 1.5 to 2 mg/L. The efficiency of the biofilter increased by 5% on average. However, increasing the depth of our biofilter would be necessary to reach the authorized limit in drinking water. Tests carried out by Cai et al. (2015) and Tekerlekopoulou and Vayenas (2008) show that a decrease in the entry concentration of 1.5 to 1 mg/L, increases the effect of biological oxidation or the efficiency of the biofilter by about 5% while reducing the required depth by about 40%. The rate of oxidation is a function of the content of ferrous ions but independent of the amount of ferro-oxidant bacteria in the environment (Winklehaus et al., 1966).

Correlation of the curves revealed that the biological oxidation of iron in the pilot column also followed a first order kinetic rate, given by the following equation:

$$-\frac{d[Fe^{2+}]}{dt} = K[Fe^{2+}] \tag{2}$$

For a constant pH and iron concentration in the sample, the kinetic constants are equal to 7.52, 10.86 and 13.054 per day, respectively for the initial theoretical iron concentrations $[Fe^{2+}]_0$ =1, 1.5 and 2 mg/L. This clearly shows that the biological oxidation of iron is a slow reaction, under the set experimental conditions, occurring in the reconstituted water to be treated. This can also be indicated by the calculated half-life constant for iron
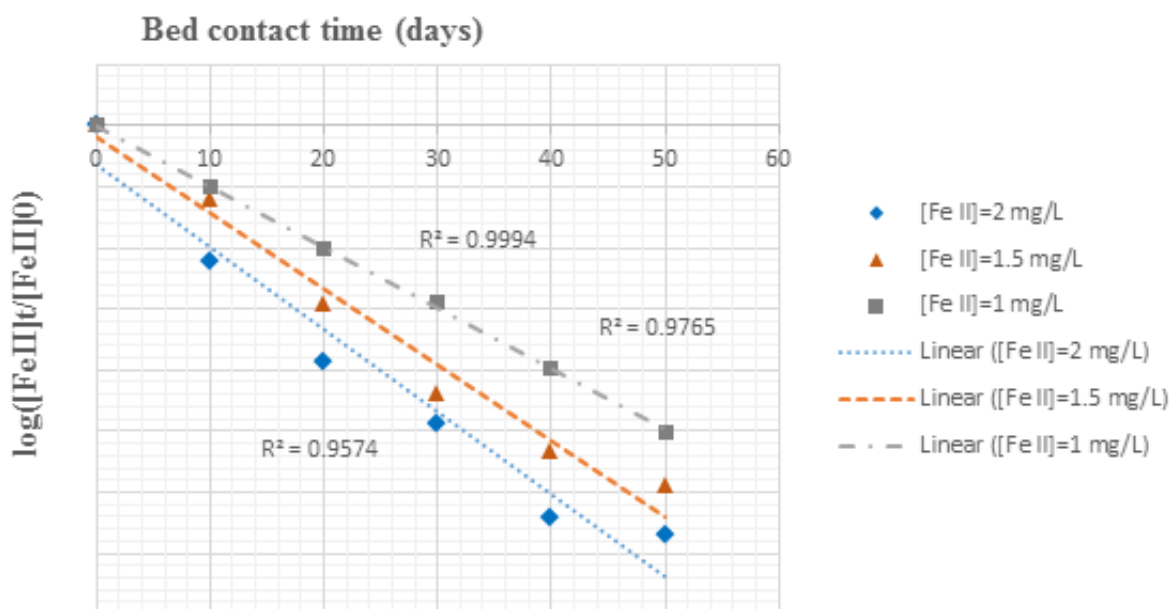
Figure 7. Linear regression analysis of Fe (II) depletion as a function of contact time; Q_L= 8.11 L/h; Q_G = 760 L/h; $[O_2]$ = 6.5 mg/L; pH = 9.5; P = 1.013 bar; T=25±1°C and Redox potential = 300 mV.
Source: Authors

oxidation, which was found to be 21, 28 and 34 days.

Compared to the work of Katsoyiannis and Zouboulis (2004) and Vaclavikova et al. (2008) the oxidation of iron with *L. ochracea* and *Gallionella ferruginea*, was far faster than ours. Biological oxidation of iron at pH=7.2 showed a half-life of 12 min (Gleeson et al., 2012; Van Beek et al., 2012). This could be explained by the catalysis of FeOOH precipitates, which can increase the removal rate by a factor of 10. This has also been observed in studies by Gleeson and Wada (2013), Taylor et al. (2013) and Wada et al. (2010).

Biological iron oxidation kinetics and biomass monitoring

Experiments based on varying the theoretical initial iron concentration of 2 mg/L, was carried out in the pilot column under prolonged aeration and the presence of isolated ferrobacteria (Figure 8). We studied the biological oxidation kinetics of iron and the development of biomass as a function of bed contact time. The liquid samples withdrawn from the top of the column, after filtration on filter paper, were analyzed by the UV-Visible spectrophotometric technique. To determine the concentration of biomass in our experiments, 20 mL samples were taken and then filtered through filter paper to remove the precipitated iron. Liquid samples were filtered again through filter paper to estimate the bacterial

dry weight on the volume collected.

Figure 8 shows the iron concentration profiles as a function of the contact time with the bed, in particular the depletion of iron in the medium, the concentration of biomass and the application of the kinetic model of Tekerlekopoulou and Vayenas (2008). These results indicate that a decrease in concentration leads to an increase in biomass. The increased biomass significantly induces precipitates of iron and biomass content in the samples collected. This could be due to the detachment of bacteria colonies at the bottom of the column caused by the air supply. We also notice a decrease in filtration speed as a function of time. Indeed, the trapped biomass further reduces the empty space between the coated jujube seed particles and the filtration rate thus becomes the dominant parameter to describe the deposition of iron precipitates. The biological oxidation of iron can be expressed by the expression of the kinetics of Tekerlekopoulou and Vayenas (2008):

$$r_{Fe\ total} = -\frac{1}{Y_{Fe}} \cdot \frac{\mu_{max} C_{Fe}}{K_s + C_{Fe}} X_{Fe} \qquad (3)$$

The kinetic constants obtained are: μ_{max} = 3.2 L/day, K_S = $0.826.10^{-3}$ mg/cm^3, Y_{Fe} = 16.72 mg Cells/mg of Fe (II) oxidized.

Figure 8 also shows the efficiency of iron removal in the biofilter. This model makes it possible to evaluate a complete oxidation of iron. The data from this model can
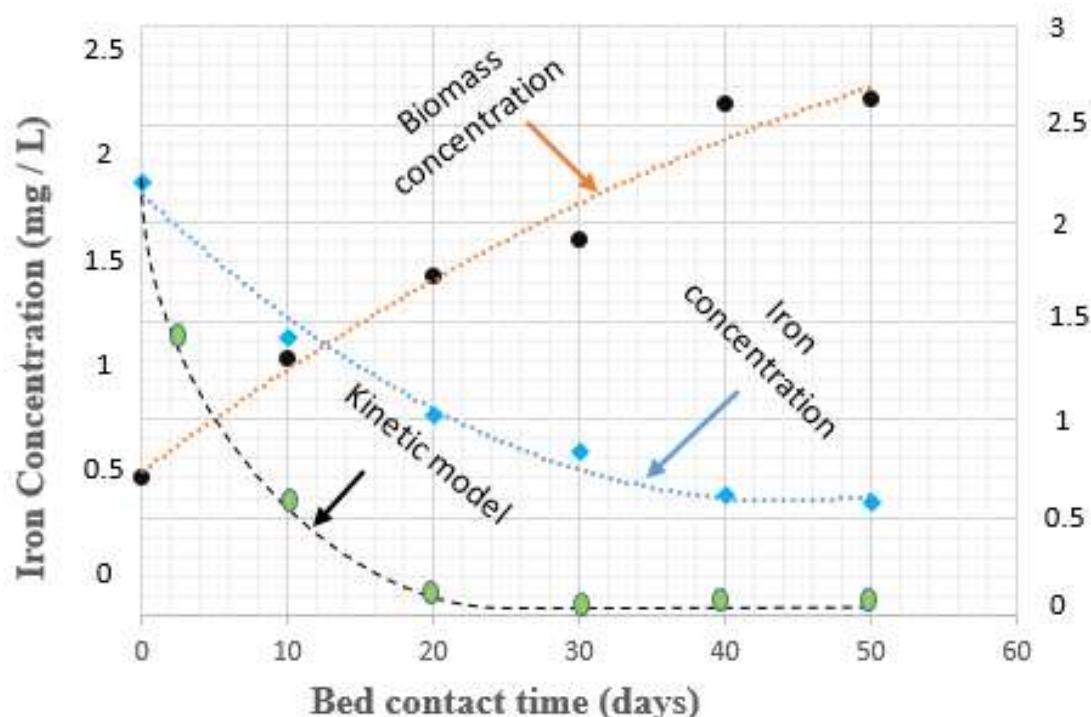
Figure 8. Experiments of the biological oxidation kinetics of iron and biomass as a function of bed contact time: Q_L = 8.11 L/h; Q_G =760 L/h; $[Fe^{2+}]_0$ = 2 mg/L; $[O_2]$ = 6.5 mg/L; pH = 9.5; P =1.013 bar; T=25 ± 1°C; Redox potential = 300 mV.
Source: Authors

facilitate the sizing of the pilot columns and the predictions of the effectiveness of biological iron treatments according to Tekerlekopoulou and Vayenas (2008) who used it in their research. In the context of our study, it is evident that the monolayer biofilter is not effective according to the operating conditions tested, because for all the iron contents chosen, we do not manage to reach the standards of drinkability recommended by WHO.

Iron concentration profiles along the depth of the single-layer filter

The effect of the initial concentration of iron along the depth of the monolayer filter was carried out in order to study the performance of the biofilter (Figure 9).

Figure 9 refers to the oxidation of iron for a constant sample volumetric flow rate Q_L =8.11 L/h and theoretical iron concentrations of 1, 1.5 and 2 mg/L. It is evident that an increase in the iron concentration at the entrance of 1 to 2 mg/L increases the depth of the biofilter needed to achieve safe iron removal from 26 to 60 cm. The rate of iron oxidation (mg Fe^{2+}/m^2.day) increases accordingly with increasing concentration. In particular, an increase in

iron concentration from 1 to 1.5 mg/L and finally to 2 mg/L increases the rate of iron oxidation from 87.535 to 572.957 and 891.267 mg Fe(II)/m^2.day, respectively.

Overall, the general observation that has been made shows that at the top of the filter, the efficiency of the filter is not sufficient, which implies that the height of the biofilter bed is a crucial parameter for the biological oxidation of iron. On the wafer from the top half of the filter, the iron oxidation efficiency increases to about 7.54% on average between the mid-column set point and the outlet. A general observation of the redox potential at the exit of the top of the column increased significantly. This is consistent with the remarks of Mouchet (1992) that the redox potential increases due to the oxidation of iron. Indeed, the biological oxidation of iron does not require more stringent conditions than the biological oxidation of manganese and ammonia with a redox potential greater than 300 to 500 mV.

Determining the depth of the filtration bed

For the study of the optimal bed height of our pilot column, we used the concentration profile equation for the biological reaction in a packed-bed bioreactor
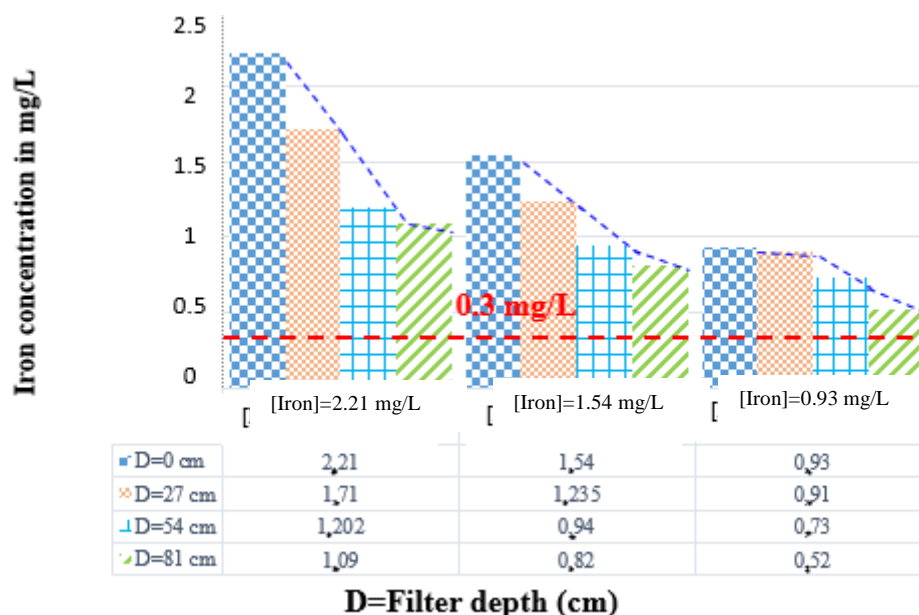
Figure 9. Experimental profiles of iron concentrations along the depth of the single-layer biofilter as a function of the initial iron concentrations in the feed: Q_L=8.11 L/h; Q_G=760 L/ h; $[O_2]$ =6.5 mg/L; pH = 9.5; P = 1.013 bar; T=25 ± 1°C and redox potential = 300 mV. Source: Authors

Table 1. Review of experimental data (k/Un) as a function of flow velocity.

Flow velocity (m/h)	Experimental values of k/U^n (m^{-1})		
	Iron	Manganese	Ammonia
5	1.20	1.09	0.99
6	1.06	1.01	0.95
9	0.94	0.94	0.88
11	0.83	0.77	0.71
15	0.79	0.72	0.6
19	0.64	0.58	0.56

Source: Štembal et al. (2005)

developed by Štembal et al. (2005):

$$\frac{c_A}{c_{A0}} = exp(-\frac{k_c a}{U} L) \qquad (4)$$

$$\frac{c_A}{c_{A0}} = exp(-\frac{k}{U^n} L) \qquad (5)$$

The experimental values of the term (k/U^n) found by Štembal et al. (2005) are grouped together in Table 1.

The dimensions and optimal operating conditions of the biofilter according to the composition of the groundwater to be treated made it possible to show the importance that binds the kinetic parameters. The kinetics of removal

of contaminants during groundwater treatment is considered to be an important issue, as it could provide information on the time required to effectively remove the specific contaminant, which is required to size treatment units (Table 2).

Application to Senegalese groundwater

The main objective of this paper was to achieve the installation of a pilot for the biological treatment of excess iron in groundwater in Senegal. In this wake, after the installation and start-up of the biological reactor in continuous mode, we carried out tests on groundwater

Table 2. Expected filter depths at 0.3 mg/L to assess the iron removal efficiency, calculated using the k/U^n values.

Flow	k/U^n	Limit value	$[Fe^{2+}]_0$ in mg/L	Bed height in m
			5	2.90
			4	2.59
			3	2.37
Q_L = 8.11 L/h selected	0.97 m^{-1}	0.3 mg/L	2	1.95
			1.5	1.65
			1	1.24
			0.5	0.52

Source: Authors

sampled at Dakar-Pout Kirene (Drilling PK2), South Dakar-Pout (Drilling PS5) and in Kaolack-Koungheul (Drilling KK5). In addition, in previous tests, iron concentrations were determined from a limited number of analyses of artificial water spiked with iron sulphate ($FeSO_4$). It therefore seemed advisable to us to undertake a more systematic study, based on water intended for the production of drinking water. In order to study at best a typical water coming from soils rich in iron, while taking into account the different ions present, we tried to make a biological removal of iron from some groundwater which comes to us from different areas mentioned earlier. We started from an original compilation of groundwater compositions intended for the production of drinking water, whose catchments contain an iron concentration between 0.5 and 2 mg/L. This will allow us to better justify the advantages and disadvantages of the realistic application of this method, in the field, for the treatment of groundwater.

Physical and chemical characterization of groundwater

The analysis of groundwater from boreholes PK2, PS5 and KK5 was carried out at the Senegalese water control laboratory. The results of the specific physical and chemical parameters of these waters are summarized in Table 3.

Biological removal of iron from groundwater

The experiences previously developed in continuous operating mode are repeated with groundwater. The treatment was carried out according to the optimized operating parameters and the results obtained are shown in Tables 4 and 5.

The results of continuous treatment show that it is possible to treat reconstituted water. However, the application to this groundwater gives values after treatment which are satisfactory but do not comply with the limit of the WHO standard recommended in drinking water. Table 5 shows that the biological treatment gives results of reduction percentages of 39.3, 48.76 and 67.23%, respectively for the PK2, PS5 and KK5 boreholes. The difficulties of treatment for biological removal appear mainly at the level of groundwater with an increasingly important iron concentration. Figure 10 shows the bar graph of groundwater treatment from boreholes PK2, PS5 and KK5 at the inlet and outlet of the biofilter. Indeed, after 14 days of contact time (continuous recirculation in a closed circuit), the greatest removal takes place for the initial concentration of 0.94 mg/L (Table 5). This means that for a high initial oxidation rate, the capacity will not necessarily be the greatest after 14 days of contact time. This confirms the biological treatment tests which were carried out in tubes.

Nevertheless, we record low biological removal percentages. This could be linked to the electrical conductivity, the very high turbidity in these collected groundwater, which exceeds the WHO standard standards (COND: ≤ 300 µs/cm Turb: ≤5 NTU), as well as to the influence metals dissolved in water. This drawback means that we recommend regulating these parameters before proceeding with the biological treatment of iron.

Characterization of the withdrawn sludge: Case of drilling PK2

The characterization of the products of biological oxidation has made it possible to show that with the X-ray fluorescence technique, the oxidized products contain a significant amount of ferric iron Fe (III). These results are in agreement with those of Katsoyiannis and Zouboulis (2004), where it was reported that the oxidation of Fe (II) by oxygen in reconstituted solutions mainly gives $Fe(OH)_3$ (Figure 11).

Table 3. Physical and chemical characterization of groundwater PK2, PS5 and KK5.

Parameter	COND (µs/cm)	pH	Turb (NTU)	Cl⁻ (°F)	SO₄²⁻ (mg/L)	Ca²⁺ (°F)	Mg²⁺ (°F)	NO₃⁻ (mg/L)	NO₂⁻ (mg/L)	Fer total (mg/L)	Mn²⁺ (mg/L)
					Site: Pout Kirene (PK2)						
Tablecloth: Maastrichtien	599	7.33	12.1	2	5	21.1	6.1	1.4	0	**2.01**	0.034
					Site: Pout Sud (PS5)						
Nappe Captée: Paléocène	718	7.01	17.2	5	23	28.4	6.4	4.4	0.007	**1.21**	0.036
					Site: Koungheul (KK5)						
Nappe Captée: Miocène	291	6.73	14.4	2	12	4.4	4.4	2.1	0.054	**0.94**	0.1

Source: Authors

Table 4. Treatment Parameters.

Operating parameter	Values
[Iron]$_{total}$- PK2	2.01 mg/L
[Iron]$_{total}$- PS5	1.21 mg/L
[Iron]$_{total}$- KK2	0.94 mg/L
Temperature	25 ±1°C
pH	9.5
Amount of dissolved oxygen [O_2]	6.5 mg/L
Filter media pressure	1.013 bar
Redox potential	300 mV

Source: Authors

Table 5. Treatment of groundwater withdrawn.

Parameter	Groundwater			
	Before treatment		After treatment	
	Result (mg/L)	WHO recommendation (mg/L)	Result (mg/L)	WHO recommendation (mg/L)
Pout Kirene (PK2)	2.01	0.3	1.22	0.3
Pout Sud (PS5)	1.21	0.3	0.62	0.3
Koungheul (KK5)	0.94	0.3	0.308	0.3

Source: Authors

The iron peak is observed at 6.305 keV (Figure 12). According to previous work published by Katsoyiannis and Zouboulis (2004), this is the characteristic energy of iron oxides, probably goethite or ferrihydrite. Goethite should peak at 6.411 keV, while ferrihydrite should peak at 6.116 keV. Therefore, the peak at 6.405 keV could correspond to goethite, which is in agreement with previous studies on the biological removal of iron carried out by Madeleine et al. (2001). Since iron and manganese are intimately linked, we can see the manganese peak at a binding energy of 6.503 keV. Manganese oxides are generally expressed with the chemical formula of MnO_x, due to the multiple valence states exhibited by Mn. Therefore, it is reasonable to measure the average of the oxidation states of a manganese mineral (Eusterhues et al., 2008). In summary, *Niton XL3T XRF* Analyzer allowed us to detect the iron peak in the washing sludge. However, it would be necessary to use the Scanning Electron Microscope-Energy Dispersion Spectroscopy (SEM-SDE) to observe the exopolymers of bacteria,

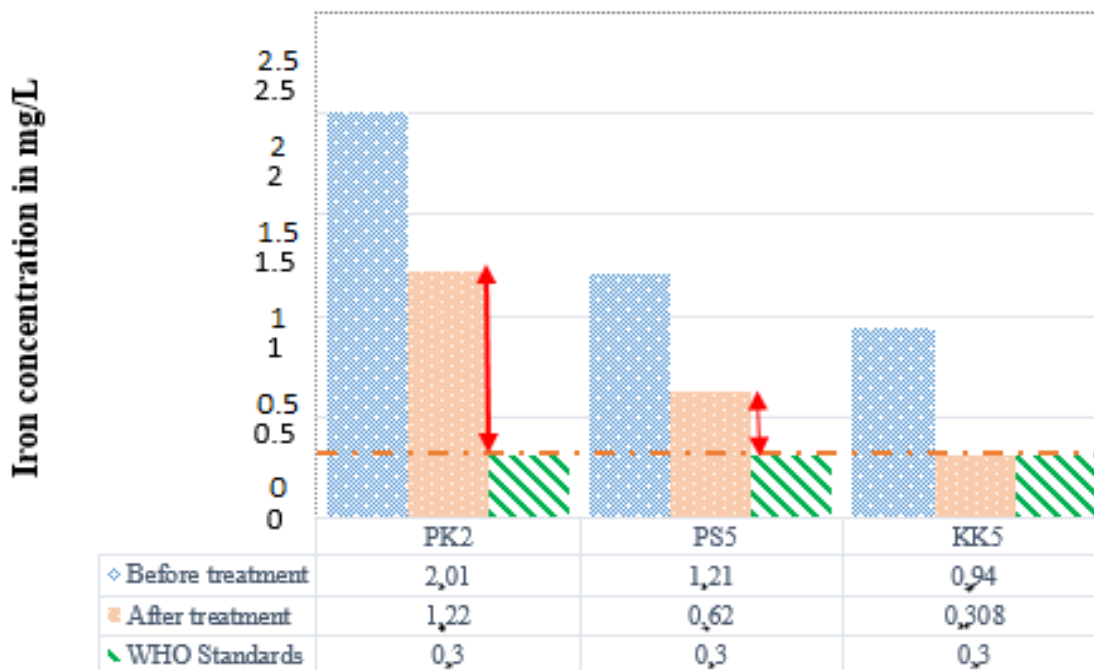## Pout Kirene (PK2); Pout Sud (PS5); Kaolack- Koungheul (KK5)

	PK2	PS5	KK5
◇ Before treatment	2,01	1,21	0,94
▪ After treatment	1,22	0,62	0,308
↖ WHO Standards	0,3	0,3	0,3

Figure 10. Bar graph of the concentrations of the PK2, PS5 and KK5 boreholes at the entry and outflow of the biofilter.
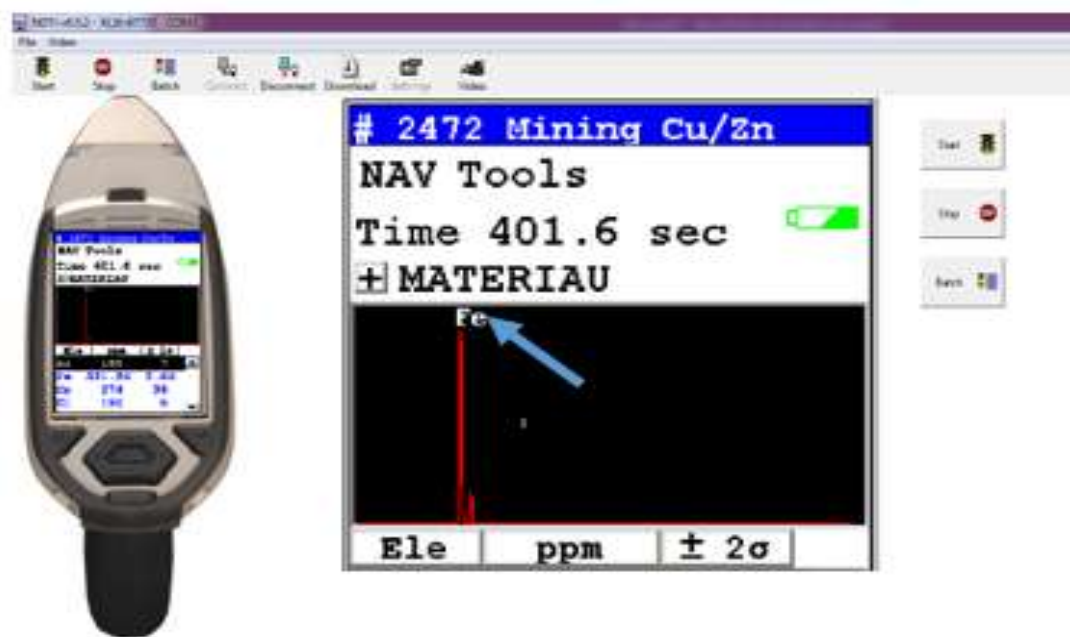Source: Authors

Figure 11. Visualization of the spectrum on the Niton XL3T XRF Analyzer.
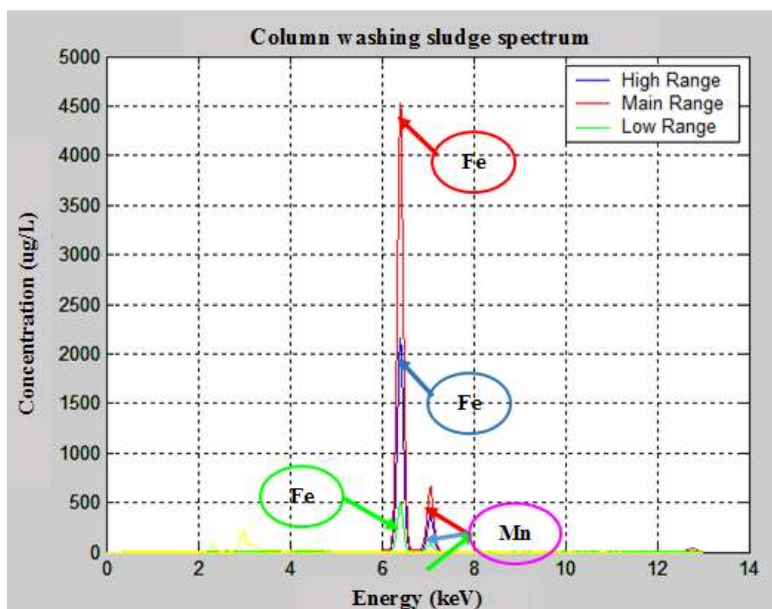Source: Authors

Figure 12. Spectrum plot on Matlab (version 6.1).
Source: Authors

which have been immobilized in the filtration column.

Conclusion

This study contributes to the improvement of Senegalese groundwater treatment techniques in order to provide a technical solution to industrialists in the treatment of drinking water. Biological treatment has been studied for this purpose to remove the excessive quantities of iron in Senegalese groundwater.

As part of this work, we have carried out pilot tests in a continuous bioreactor. These tests made it possible to control the behavior of the bioreactor. In addition, the determination of the bed backwashing operating cycles showed that an operating cycle time of 1 day was sufficient for an initial concentration of 2 mg/L, while iron concentrations included 0.3 to 0.6 mg/L, no backwashing is necessary. Thus, the effect of varying the initial iron concentration improves the filter efficiency by approximately 5.84 to 4.47% under the specific experimental conditions and initial iron concentrations of 1.5 and 2 mg/L, respectively. During these tests on the pilot column, the biological oxidation of iron is a slow reaction, under the set experimental conditions, occurring in the reconstituted water to be treated.

The process has been tested on groundwater. The results show that the biological treatment gives results of reduction percentages of 39.3, 48.76 and 67.23%, respectively for the PK2, PS5 and KK5 boreholes. The difficulties of treatment for biological removal appear mainly at the level of groundwater with an increasingly important iron concentration.

CONFLICT OF INTERESTS

The authors have not declared any conflict of interests.

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