

Comparative Study of the Defluorination of Hyperfluorinated Groundwater from the Diohine Senegal Borehole by Activated Carbon Based on Filao Litter and Zircon

Alioune Ly¹, Rose Ganga Tine¹, Mouhamadou Masseck Fall¹, Ndiouga Camara²,
Anabilaye Moussa Coly³, Diadioly Gassama³

¹Department of Civil Engineering, Laboratoire des Sciences et Technologies de l'Eau et de l'Environnement, Ecole Polytechnique, Thies, Senegal

²Department of Geotechnical, UFR Sciences de l'Ingénieur, Iba Der Thiam University, Thies, Senegal

³Department of Science and Technology, Iba Der Thiam University, Thies, Senegal
Email: lyaliou@hotmail.com

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Abstract

In Senegal, the existence of fluoride in groundwater is a major problem, particularly in the central zone. In these regions, Maastrichtian aquifers frequently have fluoride concentrations above recommended standards. These high fluoride levels cause health problems, particularly dental and bone health problems. This study focuses on defluoridation tests of hyperfluorinated Diohine Senegal groundwater using local adsorbents. The adsorbents used are activated carbon based on filao bedding and zircon. During the various tests, zircon and activated carbon columns based on filao bedding were used. Each of these columns was able to filter 500 mL of raw Diohine water with a fluoride concentration of 5.74 mg/L with a retention time of 10 mL/s. The results show that after treatment, zircon allowed a significant reduction in fluoride concentration, bringing it down to 1.84 mg/L for a reduction of approximately 67.9%. However, activated carbon reduced the concentration to 2.29 mg/L, with a reduction rate of 60.1%. The results obtained show that the effectiveness of zircon is slightly higher than that of carbon. These values, although significant, nevertheless remain higher than the WHO standard of 1.5 mg/L. Improvement could be possible with lowering the pH and increasing the retention time.

Keywords

Defluoridation, Groundwater, Fluoride, Activated Carbon, Filao Litter,

1. Introduction

In Senegal, the natural presence of fluorides in groundwater is a well-documented problem, particularly in the peanut basin where the Maastrichtian aquifers are often affected by fluoride levels exceeding recommended standards [1]-[4]. According to the World Health Organization (WHO), the maximum permissible concentration of fluoride in drinking water is set at 1.5 mg/L [5] [6]. At low doses, fluoride has beneficial effects on oral health and the acidification of tooth enamel, particularly for young children under eight years of age [7]-[9]. However, at high concentrations and prolonged consumption, it becomes toxic and causes diseases such as dental fluorosis (staining and discoloration of teeth) and bone fluorosis (deformation and joint pain), with serious consequences on the quality of life of the affected populations [10] [11].

However, in some localities in Senegal such as Diouroup, a neighboring commune of Diohine, values exceeding 6 to 10 mg/L are regularly observed, thus posing a real public health problem [3] [12]. Faced with this situation, several methods of treating hyperfluorinated water such as reverse osmosis, ion exchange, chemical precipitation, nanofiltration and adsorption have been tested [2] [13] [14]. Among these approaches, adsorption on natural or modified materials is attracting growing interest, particularly in rural areas, due to its simplicity, low cost, local adaptability and availability [15]-[17].

Among the most promising materials for water defluoridation, zirconium-based adsorbents have attracted increasing interest due to their high affinity for fluoride ions, their chemical and thermal stability, as well as their non-toxic and economical nature. A comprehensive study conducted by Savari *et al.* (2023) provides a comprehensive review of zirconium-modified adsorbents, highlighting their notable effectiveness in removing fluoride from aqueous media [18]. The use of zircon in this study is part of an international dynamic of valorization of high-performance and available materials for defluoridation. This choice also aims to explore its potential in comparison with another local natural material; activated carbon based on filao litter, in order to propose solutions adapted to the environmental and economic realities in the locality of Diohine.

The general objective of this study is to evaluate and compare the effectiveness of two adsorbent materials, namely activated carbon based on filao litter and local zircon, in the defluoridation process of hyperfluorinated Diohine groundwater. To achieve this objective, it is first necessary to characterize the groundwater in the study area in order to determine its physicochemical parameters, including fluoride content. Then, activated carbon was prepared from filao litter and characterized according to usage standards. Laboratory adsorption tests were used to evaluate the respective performances of the two materials in terms of adsorption

capacity and defluoridation efficiency. Finally, a comparative analysis is conducted to study the adsorption kinetics, maximum performance and optimal operating conditions of each adsorbent.

2. Methodology

In order to meet the objectives of this study and to rigorously compare the effectiveness of activated carbon based on filao litter and zircon in the defluorination of hyperfluorinated Diohine groundwater, a structured methodological approach was implemented. This section describes the different steps followed, from the location, collection and characterization of water samples to the preparation of adsorbent materials, including the implementation of adsorption tests.

2.1. Location of the Commune of Diohine

Diohine is a village in Senegal located in the west-central part of Senegal, in the region and department of Fatick, in the commune of Tattaguine. **Figure 1** shows its location in the Fatick region. The Diohine borehole, which is the subject of this study, is located in the commune and constitutes the source of water supply for the population.

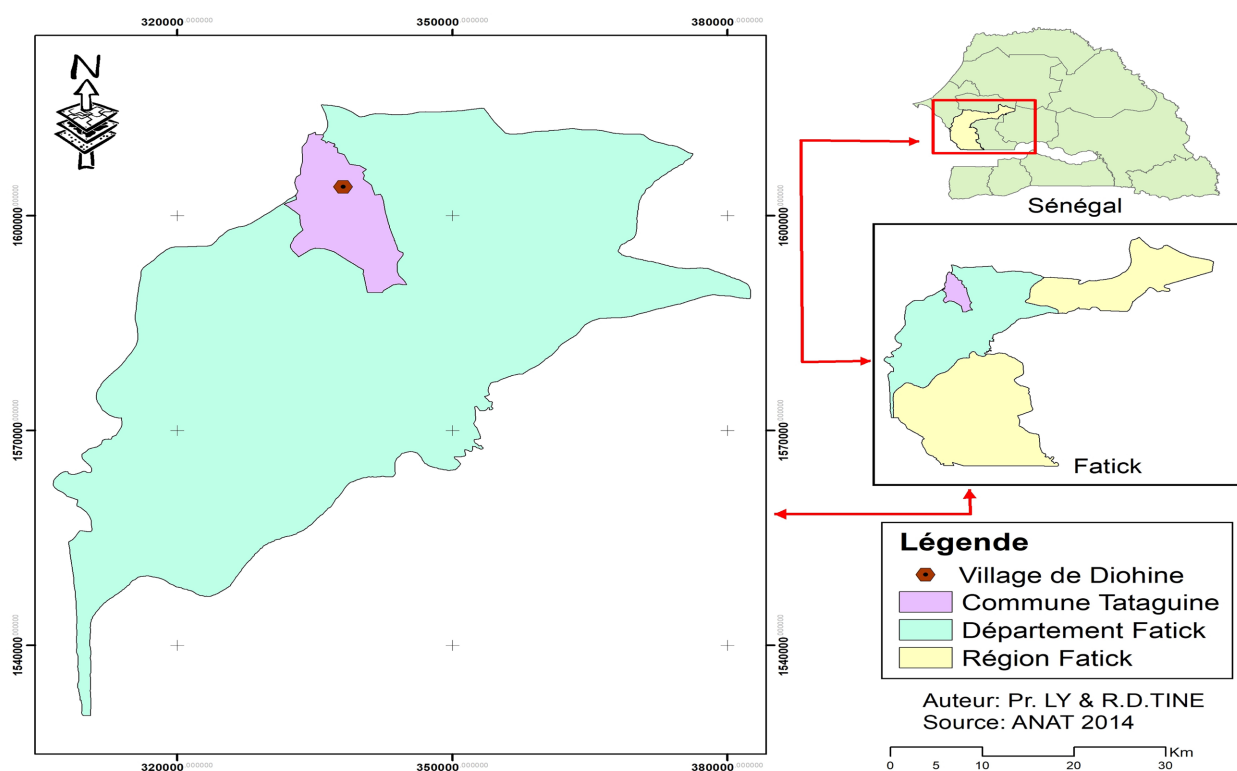


Figure 1. Location of the village of Diohine.

2.2. Characteristics of the Water from the Diohine Borehole

The assessment of the water characteristics of the Diohine borehole was carried

out in two stages: an *in situ* analysis followed by a laboratory analysis.

The *in situ* analysis focused on the measurement of physical parameters: temperature, hydrogen potential (pH), electrical conductivity and total dissolved solids (TDS).

A water sample was taken and sent to the laboratory for the analysis of physicochemical parameters and the treatment test process.

2.3. Description of Zircon and Activated Carbon Used

The zircon used in this research was supplied by Grande Côte Opérations (GCO), a subsidiary of the Eramet group, specializing in mineral sand mining. GCO operates a deposit located in the dune formations of the Thiès and Louga regions of Senegal, and has two industrial processing units located in Diogo. The extracted ores consist mainly of zircon, ilmenite, rutile, and leucoxene.

As for activated carbon, it was synthesized from the litter of *Casuarina equisetifolia* (filao) collected on the coastal strip of Mboro. The manufacturing process includes controlled pyrolysis followed by chemical activation using calcium chloride (CaCl_2), before grinding and sieving in the laboratory to obtain a particle size suitable for adsorption application. **Figure 2** illustrates in A the image of zircon and in B that of carbon.

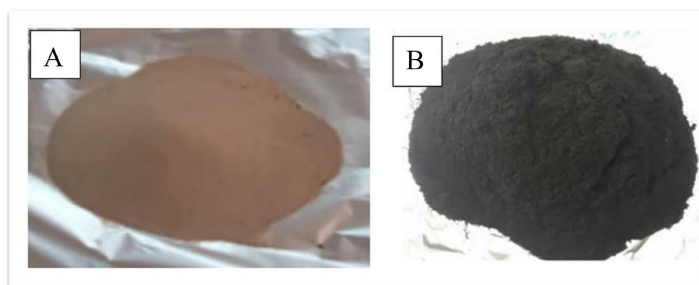


Figure 2. Materials used: (A) zircon; (B) active charcoal based on filao litter.

2.4. Process of Obtaining Activated Carbon

2.4.1. Carbonization of Filao Litter

Carbonization is a key step in the production of precursor charcoal for activation, aiming to enrich the biomass with fixed carbon and to initiate the formation of porosity. In this study, the previously collected *Casuarina equisetifolia* (filao) litter (**Figure 3**) was manually fragmented using metal scissors into small, homogeneous pieces to facilitate carbonization.

The process was carried out using a Nabertherm muffle furnace. The ground material was packaged in a metal pan with a perforated lid to allow controlled evacuation of volatile gases from pyrolysis. The whole was then introduced into the furnace to undergo dry pyrolysis in a controlled atmosphere at 600°C for 45 minutes.

The muffle furnace, having a limited capacity, required several carbonization cycles in series to accumulate a sufficient quantity of carbon for the subsequent

activation and characterization steps. **Figure 4** illustrates the visual appearance of the carbon obtained after carbonization of the filao litter called carbonizate at 600°C.



Figure 3. Filao litter.



Figure 4. The coal.

2.4.2. Drying, Grinding and Sieving of the Carbinisate

The carbonized material (**Figure 5(A)**) is washed with distilled water to remove impurities, then dried for 24 hours in an oven at 105°C. It was then ground in a mortar to obtain two (2) types of particle size: one with a diameter of less than 0.5 mm (**Figure 5(B)**) and the other with a diameter of between 0.5 and 1 mm (**Figure 5(C)**). **Figure 5** illustrates the stages of grinding and separation of the two aggregates of the carbonized material of the filao litter obtained in a muffle furnace at 600°C.

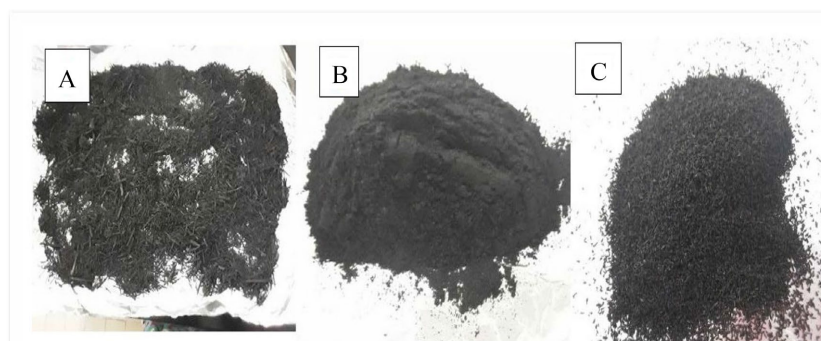


Figure 5. Crushing and separation of aggregates.

2.4.3. Activation of the Carbonized Material

Activation was carried out with calcium chloride (CaCl_2) prepared beforehand in the laboratory.

The charcoal powder, passing through a 0.5 sieve, is poured into a saucepan, then the calcium chloride solution is poured into it while stirring so that all the powder is in contact with the solution. This was done until a pasty mixture was obtained. The saucepan was closed and the mixture was left to stand for 24 hours. Then, the carbonized material was removed from the solution while pressing it to drain the water. The previously activated charcoal was dried in an oven for 24 hours. The activated charcoal was washed with distilled water to remove the activating agent and then dried in an oven for 24 hours at 105°C . After activation of the carbonized material, the activated charcoal was obtained (**Figure 6**).



Figure 6. Activated carbon.

2.5. Determination of Iodine and Methyl Blue Values of Zircon and Activated Carbon

2.5.1. Iodine Index

The iodine value is an indicator of the microporosity of an adsorbent; it measures its ability to retain small molecules in aqueous solution. It is expressed in milligrams of iodine adsorbed per gram of material. In this study, a 0.1 N iodine solution was prepared from potassium iodide (KI) and diiodine (I_2), then standardized with a 0.1 N sodium thiosulfate solution. The zircon and activated carbon samples, dried at 105°C for 24 h, were brought into contact with the iodine solution. After stirring for 5 to 6 min and filtration, a portion of the filtrate was titrated with thiosulfate until discoloration. The amount of iodine adsorbed, calculated by the difference in concentration before and after contact, gives the iodine value of the material. This index allows to compare the adsorption efficiency of zircon and activated carbon and to evaluate their ability to trap small molecules in aqueous phase. The iodine index expressed in (mg/g) was calculated according to Morvan *et al.* by Equation 1 [19].

$$\text{Iodine index} = \frac{\left(C_0 - \frac{C_n V_n}{2V_{\text{I}_2}} \right) \times M_{\text{I}_2} \times V_{\text{abs}}}{m_{\text{CA}}} \quad (1)$$

with:

C_0 : the concentration of the initial iodine solution ($\text{mol}\cdot\text{L}^{-1}$), C_n : the concentration of the sodium thiosulfate solution ($\text{mol}\cdot\text{L}^{-1}$), V_n : the volume of the sodium thiosulfate solution at equivalence (mL), V_{I_2} : the volume of iodine solution dosed, M_{I_2} : the molecular molar mass of iodine, V_{abs} : the adsorption volume and m_{CA} : the mass of activated carbon.

2.5.2. Methylene Blue Index

The methylene blue index (MBI) is a parameter used to evaluate the macroporosity of an adsorbent material such as activated carbon or zircon. It is defined as the volume of standard methylene blue solution decolorized by 0.1 g of adsorbent. In this work, a 1200 mg/L stock solution was prepared by dissolving 1200 mg of dye in one liter of distilled water. Adsorption is monitored by UV-Visible spectrophotometry, a quantitative technique based on the Beer-Lambert law that relates absorbance to solution concentration, extinction coefficient, and optical path length. Absorbance measurements are performed for different wavelengths (330 - 690 nm) to determine the maximum absorbance wavelength (λ_{max}). The latter is used to establish a calibration line, an essential prerequisite for evaluating the adsorption capacity of the materials studied.

To measure the residual concentrations of methylene blue, 100 mg of adsorbent (charcoal or zircon) are brought into contact with 100 mL of dye solution for 20 minutes with stirring. The absorbance values obtained make it possible to determine the residual concentrations (C_{mr}), then to calculate the apparent adsorption capacity (Q_{BM} in mg/g) according to Equation 2 [20]:

$$Q_{\text{BM}} = \frac{(C_{\text{mi}} - C_{\text{mr}}) \times V}{m_{\text{CA}}} \quad (2)$$

C_{mi} : the initial mass concentration (mg/L) of the methylene blue (MB) solution. C_{mr} : the residual mass concentration (mg/L) of the MB solution. V : the volume of the MB solution (= 100 mL). m_{CA} : the mass of the adsorbent used (g).

2.6. Absorption Device

The adsorption or filtration device is illustrated in **Figure 7**. In the context of the defluorination tests of hyperfluorinated brackish water, two experimental devices or single-column systems were designed; one filled with activated carbon based on filao bedding (**Figure 7(A)**), the other with zircon (**Figure 7(B)**). Each single-column device consists of a 5 - 10 L raw water reservoir, an inlet valve, a perforated connecting sleeve without a filter, an adsorbent column (zircon or activated carbon), a perforated connecting sleeve with a filter, and a filtration flow control valve as shown in **Figure 7**.

After characterizing the raw water, a volume of 500 mL was introduced into the reservoir of each device. The inlet valve was opened, and the filtration flow rate was adjusted to 0.83 mL/s using the shut-off valve. The contact time between the water and the adsorbent was set at 10 minutes. The filtrates obtained were col-

lected in beakers and then stored in refrigerated bottles before being sent to the laboratories for physicochemical analyses.

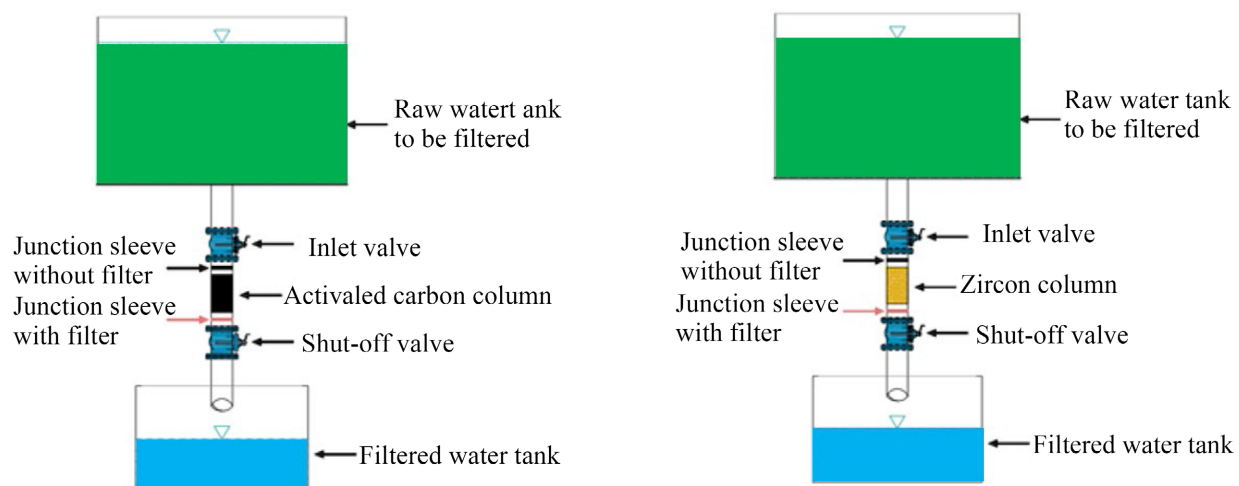


Figure 7. Diagram of the measuring device.

3. Discussion Results

3.1. Characterization of Raw Water from Dihine Drilling

The analysis of the physicochemical parameters of raw water (Table 1) compared to the standards and limits recommended for drinking water, reveals that the majority of the measured values exceed the accepted thresholds, thus confirming the need for prior treatment before consumption. However, the present study does not aim to directly make the analyzed water potable, but rather to evaluate the relative effectiveness of adsorption on zircon and adsorption on activated carbon based on filao litter for the removal of fluorides.

Table 1. Characteristics of raw water.

Settings	Concentration in mg/L	Standards and limits
Potassium K ⁺	21.08	12
Sodium Na ⁺	651.61	200
Magnesium Mg ²⁺	4.63	30
Calcium Ca ²⁺	9.44	200
Bicarbonate HCO ₃ ⁻	579.5	250
Nitrate NO ₃ ⁻	6.59	50
Chloride Cl ⁻	614.5	250
Fluorine F ⁻	5.74	1.5
Sulfate SO ₄ ²⁻	107.15	250
Total Dissolved Solids TDS	2005.85	1000
pH	7.92	6.5 – 8.5

3.2. Characteristics of Zircon and Activated Carbon

These results were obtained following analyses carried out with an X-ray fluorescence spectrometer, the Niton XLT900s, at the laboratory of the Applied Nuclear Technology Institute (ITNA) of the Cheikh Anta Diop University of Dakar (UCAD). **Table 2** gives the composition of activated carbon and **Table 3** that of zircon.

Table 2. Composition of coal.

Elements	Nature of matter	Symbols	Concentration (g/kg)	Percentage (%)
Calcium	Alkaline earth metal	That	113.14	66.50
Aluminum	Alkaline earth metal	Al	20.85	12.25
Chlorine	Halogen	Cl	12.15	7.14
Iron	Transition metal	Fe	10.64	6.25
Potassium	Alkali metal	K	4.74	2.79
zirconium	Transition metal	Zr	3.88	2.28
Sulfur	Non-metal	S	2.77	1.63
Titanium	Transition metal	You	0.89	0.52
Manganese	Transition metal	Mn	0.79	0.46
Copper	Transition metal	Cu	0.15	0.09
Tungsten	Transition metal	W	0.11	0.06
Arsenic	Metalloid	Ace	0.01	0.01
Bismuth	Metal	Bi	0.01	0.01
Total mass (g/kg)			170.13	100.00
Density (g/L)			0.25	-

The results obtained highlight notable differences between activated carbon from filao litter and zircon in terms of fluoride removal. Chemical analysis showed that filao litter is particularly rich in calcium (66.5%), aluminum (12.25%) and iron (6.25%), elements known for their ability to interact with fluoride ions by precipitation or adsorption on metal hydroxides. In addition, its low density (0.25 g/L) and its porous structure after activation constitutes an advantage for the development of a large specific surface area, which favors adsorption. These characteristics explain the good performances observed during adsorption tests, consistent with the iodine indices close to those of commercial carbons (838 - 876 mg/g) [19].

In contrast, zircon has a composition dominated by zirconium (80.83%) and silica (15.32%), with a much higher density (3.16 g/L). Zirconium is known for its high affinity towards fluoride ion through the formation of stable complexes (Zr-F), giving the material remarkable selectivity and efficiency for defluorination [21]. Although its structure is denser and less porous than that of activated carbon, the high zirconium concentration compensates for this limitation by providing many specific active sites.

These results are consistent with those reported in the literature, particularly on activated carbons obtained from bitter almond shell and olive seeds, which also exhibited interesting fluoride adsorption capacities. Similarly, the performance observed with zircon confirms the conclusions of previous studies highlighting the effectiveness of zirconium-based materials in the defluoridation of groundwater [22] [21].

Table 3. Composition of zircon.

Elements	Nature of matter	Symbols	Concentration (g/kg)	Percentage (%)
Zirconium	Transition metal	Zr	708.73	80.83
Silica	Alkaline earth metal	If	134.34	15.32
Sulfur	Non-metal	S	22.31	2.54
Copper	Transition metal	Cu	2.34	0.27
Molybdenum	Transition metal	Mo	2.26	0.26
Calcium	Alkaline earth metal	That	2.19	0.25
Barium	Alkaline earth metal	Ba	1.51	0.17
Titanium	Transition metal	You	1.13	0.13
Tungsten	Transition metal	w	0.60	0.07
Niobium	Transition metal	Nb	0.42	0.05
Nickel	Transition metal	Neither	0.38	0.04
Money	Metalloid	Ag	0.34	0.04
Arsenic	Metalloid	Ace	0.29	0.03
Total mass (g/kg)			876.85	100.00
Density (g/L)			3.16	-

It therefore appears that activated carbon from filao litter constitutes a versatile and accessible adsorbent, capable of treating several pollutants thanks to its porosity and the diversity of its constituent elements, while zircon, more specific, proves to be particularly effective in the targeted elimination of fluoride. The choice between these two materials therefore depends on the context of use on the one hand, the overall treatment of water quality for activated carbon and on the other hand, the selective and optimized treatment of fluoride for zircon.

3.3. Characterization of Activated Carbon

The characterization of activated carbon based on filao litter consisted of determining the moisture, volatile matter, ash, fixed carbon, iodine index and methylene blue index. The results obtained during this characterization are presented below. The characterization results are shown in **Table 4**.

The results obtained on filao litter (**Table 4**) are generally in agreement with those reported in the literature [23]. Indeed, the reference study on biomasses also showed low ash contents, low humidity levels and high proportions of volatile matter. This convergence confirms that filao litter, like other biomasses studied, constitutes a precursor suitable for the production of quality activated carbons,

presenting both a good adsorption capacity and an interest for water treatment.

Table 4. Characteristics of activated carbon based on filao litter.

Biomass	Humidity level (%)	Dry matter content (%)	Volatile matter content (%)	Ash content (%)	Fixed carbon rate (%)
Filao litter	33.12	66.88	88.57	6.38	3.30

3.4. Iodine and Methylene Blue Index of Zircon and Coal

The iodine and methylene blue indices of zircon and coal are grouped in **Table 5**.

Table 5. Iodine and methylene blue values of zircon and activated carbon.

Materials	Iodine index	Methylene blue index
Coal	1063.56	842.80
Zircon	526.61	705.98

The results show that both zircon and activated carbon prepared from filao litter have iodine values comparable to those of commercial activated carbons [19] [23], indicating good efficiency in terms of adsorption capacity. However, activated carbon from filao litter develops a slightly higher performance than zircon, due to its porous structure more suited to the adsorption of methylene blue molecules. Zircon, although efficient, could release some mineral constituents into the treated water, which justifies the complementary use of activated carbon downstream.

3.5. Adsorption Spectrum of Methylene Blue

In **Figure 8**, we presented the results on the adsorption spectrum of methylene blue which allowed us to choose the best wavelength for the determinations of the methylene blue indices of coal and zircon.

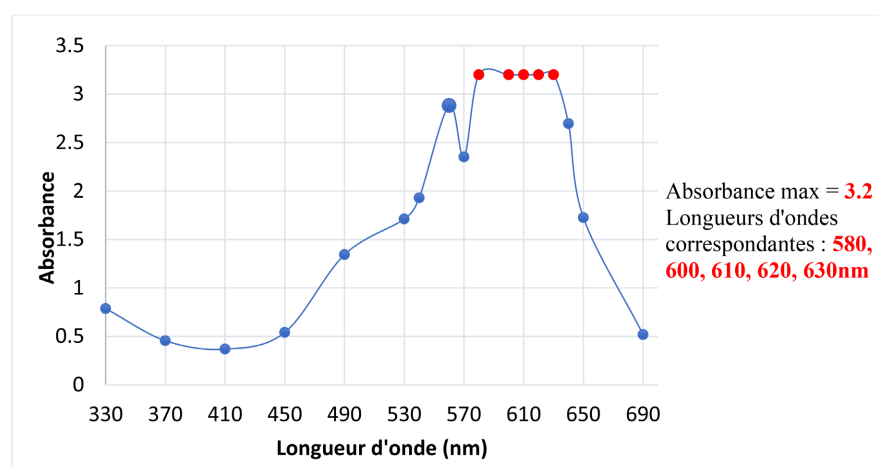


Figure 8. Adsorption spectrum of methylene blue.

For different wavelengths, we find that in the adsorption spectrum of methylene blue, the absorbance reaches its maximum at the values of 580, 600, 610, 620, 630 nm. Thus, we chose the wavelength 620 nm for the rest of the work.

3.6. Methylene Blue Calibration Line

In the following **Figure 9**, we have illustrated the calibration line obtained during the determination of the methylene blue indices. The equation of the calibration line, representing the absorbance as a function of the concentration of Methylene Blue, with a regression coefficient $R^2 = 0.998$, can be considered as a good linear fit. This equation is used, subsequently, for the calculation of the concentration of a given solution.

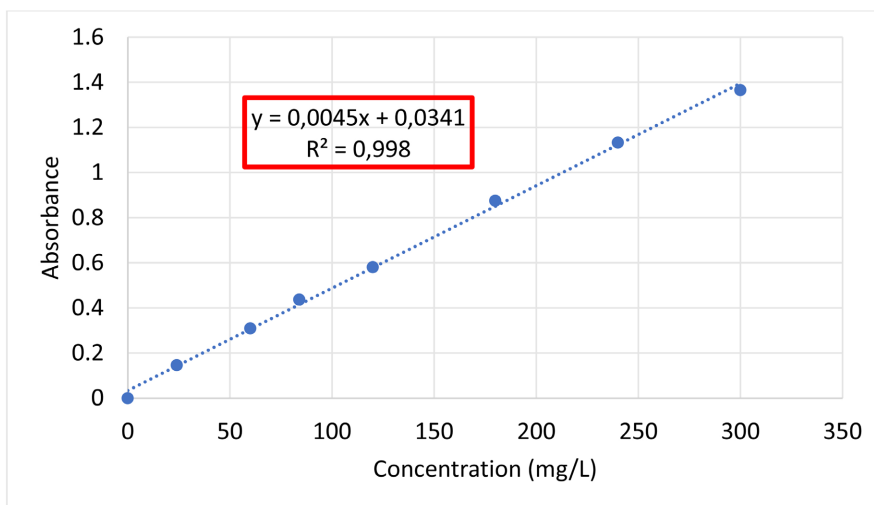


Figure 9. Methylene blue calibration line.

3.7. Treatment Results

The following device **Figure 10** was adopted for the treatment as written in the methodology.



Figure 10. Device used for treatment.

The results of the analysis of raw water, treated water and standards are presented in **Table 6**.

Table 6. Comparison of fluoride contents of different waters and limits.

	Concentration in (mg/L) of Fluorides
Raw water	5.74
Zircon treated water	1.84
Water treated with activated carbon	2.29
Limits and standards	1.5

Analysis of the results obtained highlights an initial fluoride concentration in the raw water from the Diohine borehole of 5.74 mg/L, a value well above the standard recommended by the WHO set at 1.5 mg/L. This high content confirms the need for defluoridation treatment before any consumption [24]. After treatment, two distinct behaviors are observed depending on the adsorbent used. Zircon allows a significant reduction in the fluoride concentration, bringing it down to 1.84 mg/L, which corresponds to a reduction of approximately 67.9%. Although this value remains slightly above the limit set by the WHO, it demonstrates the high efficiency of this material and its strong affinity for fluoride ions [22] [25] [26]. In comparison, activated carbon made from filao litter reduced the concentration to 2.29 mg/L, a reduction of 60.1%. This significant result, however, remains lower than that obtained with zircon and thus reflects a more limited effectiveness with respect to fluoride.

These observations indicate that zircon performs better than filao activated carbon for fluoride removal. However, neither adsorbent can achieve the recommended potability standards. On a practical level, activated carbon has the advantage of being sourced locally and is very accessible, making it an attractive alternative for decentralized and sustainable treatment. Zircon, on the other hand, appears to perform better but could pose economic or supply constraints. In this sense, process optimization, particularly through combined or sequential treatments (zircon-carbon, or coupling with other adsorbents such as activated alumina), could make it possible to achieve fluoride concentrations that comply with international standards.

4. Conclusions

This present work has highlighted the persistence of the fluoride problem in Diohine groundwater, with concentrations well above WHO standards. In order to reduce the fluoride contents of these waters, defluoridation tests by adsorption on zircon columns and activated carbon based on filao litter have made it possible to obtain significant reductions.

The results obtained confirm that zircon has a higher efficiency (67.9% reduction) compared to activated carbon from filao litter (60.1%). However, neither

adsorbent was able, under the experimental conditions applied, to reach the recommended potability threshold (1.5 mg/L). Activated carbon nevertheless retains the major advantage of being a local resource, inexpensive and easily mobilizable in a rural context, which makes it relevant from a decentralized treatment perspective.

In addition to the results obtained, this study offers new opportunities for process improvement. In particular, extending the retention time from 10 to 30 minutes, or even 120 minutes, could significantly improve the efficiency of activated carbon by promoting longer contact between the adsorbent and fluoride ions.

The study, therefore, highlights the importance of continuing studies on improving operational parameters and potential synergies between various adsorbents, in order to design viable, sustainable solutions adapted to the socio-economic and environmental realities of Senegal.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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