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Research Article

Use of Zircon in the Physicochemical Process of Defluorination of Hyperfluorinated Brackish Water from the Diouroup Borehole (Senegal)

Alioune Ly[1,*], Anabilaye Moussa Coly[2], Ndiouga Camara[3], Baba Ngom[4], Diadioly Gassama[2], Séni Tamba[1]

[1]Department of Civil Engineering, Laboratoire des Sciences et Technologies de l'Eau et de l'Environnement, Ecole Polytechnique, Thiès, Sénégal

[2]Department of Science and Technology, Iba Der Thiam University, Thies, Senegal

[3]Department of Geotechnical, UFR Sciences de l'ingénieur, Iba Der Thiam University, Thies, Senegal

[4]Departement of Common Core, Laboratory of Water and Environmental Sciences and Technologies, Thiès, Senegal

Abstract

In phosphate-producing countries (United States, Commonwealth of Independent States, North and West Africa including Senegal), the presence of phosphate ores (hydroxyapatite) is often associated with that of fluorine ores (fluoro apatite). In these regions, water from confined aquifers may have excessive fluoride contents. While the potability standards in tropical climates are 0.7 mg/L at a temperature of 25 °C In the central zone of Senegal, hyperfluorinated brackish water constitutes a public health problem. It is in this context that it was decided to make our contribution to the purification of drinking water in this area. In our study, we used as an element in the physicochemical defluorination process. The results obtained, by this physicochemical treatment by filtration on the zircon column, showed overall a reduction in fluoride contents of 67.55% (i.e. from 6.41 mg/L to 2.08 mg/L) in chlorides of 10.44%. We can therefore affirm that zircon can be used for the defluorination of hyperfluorinated brackish waters. However, these results can be improved by lowering the pH of the water before treatment and increasing the retention time.

Keywords

Zircon, Defluorination, Adsorption, Brackish Water, Diouroup

1. Introduction

In phosphate-producing countries such as Senegal, the presence of hydroxyapatite is often associated with that of fluorine ores (fluoro apatite). In these countries, water from confined aquifers may have excessive fluoride contents. While the drinking standards in tropical climates are 0.7 mg/L at a temperature of 25 °C [1, 2].

Fluoride concentrations commonly encountered vary from 5 to 15 mg/L depending on the season. Exceptionally, maximum

*Corresponding author: lyaliou@hotmail.com (Alioune Ly)

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peaks of up to 20 mg/L can be recorded. The presence of excess fluoride ions in drinking water causes serious poisoning [3].

Like any trace element, the fluoride ion is necessary and beneficial for the body at low levels (prophylactic effect) but, as soon as its concentration is too high, it becomes toxic and leads to dental and bone fluorosis.

Fluoride poisoning is serious in certain regions of Senegal and represents a real public health problem. Indeed, an epidemiological survey carried out in 2008 in 45 villages in the departments of Mbour and Fatick (on a population estimated at 154,763 inhabitants) reveals that 70% of the 900 surveyed are in contact with fluoride levels greater than 1.5 mg/L. (WHO guide values) and that 83.6% are affected by dental fluorosis while 52.7% show clinical signs which could be associated with bone fluorosis [4].

Faced with such an alarming situation in the central zone of Senegal, we have carried out studies with a view to stemming this phenomenon.

The aim of this study is to propose a method for treating hyperfluorinated brackish water in Senegal, more precisely in the locality of Diouroup, Fatick region. The methodology consists of adsorption by zircon sand for domestic applications.

This study follows a first which was carried out on another membrane treatment method (low pressure reverse osmosis) for applications to larger cities [5].

We reviewed the state of knowledge acquired in Senegal on the problem of hyperfluorinated brackish groundwater. We were also interested in the solutions already studied, since its first discovery by Raoult, in Mont-Rolland (Thiès region) in 1957 [6, 7].

Subsequently, we presented a hydrogeological distribution of fluorides on the Senegalese territory. Afterwards, based on the results of epidemiological surveys carried out, we exposed the endemic health risks incurred (dental and bone fluorosis) by populations in exposed areas [6, 7].

Finally, we reviewed the different physicochemical and membrane treatment methods for hyperfluorinated brackish water (chlorides and fluorides) already implemented in the world and in Senegal, while analyzing their suitability or not to local realities. [8-10].

Finally, we proposed a physicochemical type treatment, adsorption on zircon, for domestic application.

2. Materials and Methods

The study area is located in the municipality of Diouroup, district of Tattaguine, region of Fatick. The samples of the raw water studied were taken at the sampling point of the borehole in the said municipality, located almost one hundred (100) meters from the national road. Previously we present the study area.

2.1. Presentation of the Study Area

The commune of Diouroup belongs to the department of Tattaguine, Region of Fatick. This region is part of the central zone of Senegal.

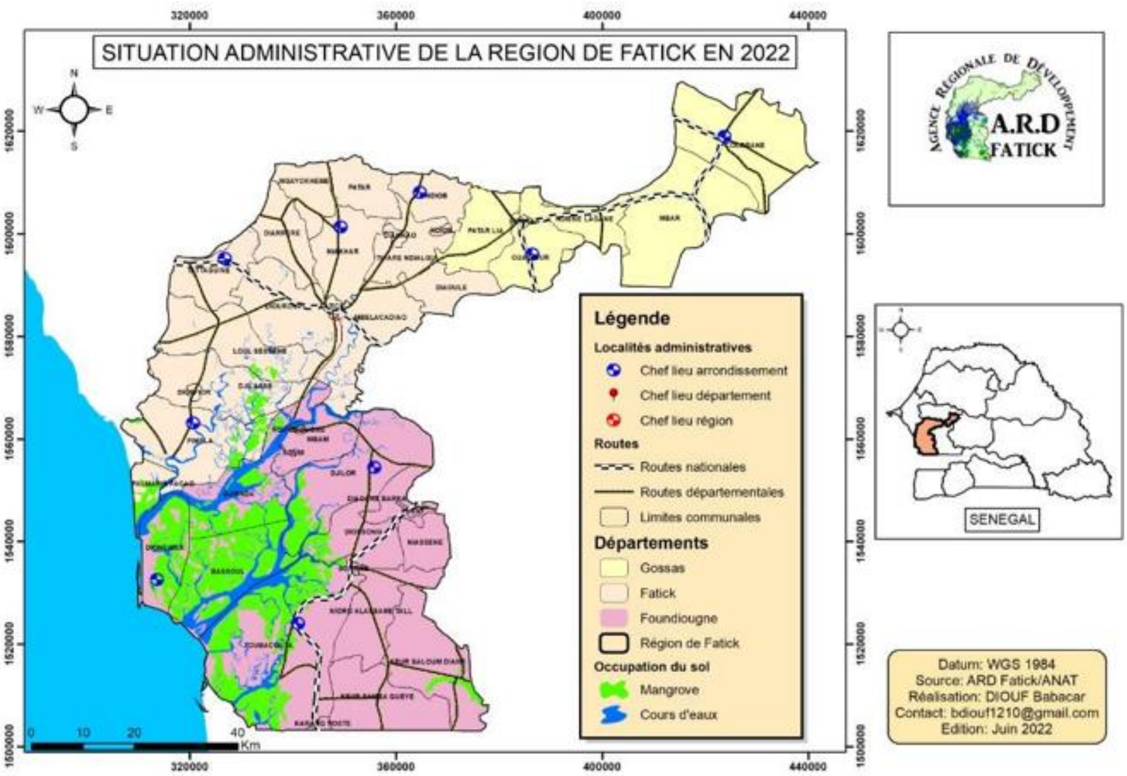

Figure 1. Administrative map of the Fatick region.

The archives and regional hydrogeological data obtained after the mechanical surveys carried out by the SDE present from top to bottom the succession of the following hydrogeological facies:

It is made up of lateritic sands and clayey sands with a thickness rarely exceeding 10 m. The aquifer in this sector can only be exploited by wells and the exploitation of these resources for the supply of drinking water is delicate.

They are in the sector of marls, marl-limestones and fissured limestones productive with salt water unsuitable for drinking water supply and irrigation. Note also that the salty Paleocene limestones rest directly on the Maestrichtian sands and sandstones.

It is made up in the sand sector, clayey sands of good production. The depth is approximately 300 to 350 m, but its piezometric level is generally located less than 10 m from the ground. The fluoride content is greater than 4 mg/L.

With nearly 30 billion m^3 of water reserves assessed, it constitutes the most important drinking water reserve in Senegal in the future [11].

2.2. Choice of Treatment Process

There are several processes for the removal of fluorides and chlorides. Among these, we can cite membrane processes (electrodialysis, reverse osmosis and nano filtration) and physicochemical processes (precipitation, adsorption and ion exchange).

Based on the advantages, disadvantages and the nature of the pollutants to be removed (fluorides and chlorides), our choice fell on adsorption on zircon for domestic applications, due to its abundance in Senegal, its ease of use and its adsorption capacity.

On the other hand, adsorption makes it possible to reduce MES and organic matter; in addition, these adsorbents are easily regenerable.

To carry out a good adsorption treatment, it is important to use a product with a high adsorption capacity, easily regenerable and which withstands several passes before being regenerated. Thus, studies on adsorption processes, Metal Organic Frameworks based on zirconium (Zr-MOF) have demonstrated a higher adsorption capacity and are thermally and chemically stable in hyperfluorinated water [12, 13].

2.3. Sampling

Regardless of the quality of an analysis, its result is only valid under the following conditions:

1) The sample analyzed is well representative of the body of water considered;
2) This sample did not deteriorate between the time of collection and that of analysis.

To do a good job, the same attention must be given to the collection and storage of samples as to the analysis itself. Therefore, the sampling method must be adapted to the measurement to be carried out and the characteristics of the water. In addition, the analysis must be done as soon as possible, generally within 24 hours.

For sampling, as part of this study, before going into the field, we used plastic and glass bottles that we previously washed well, rinsed and perfectly identified. In these bottles, we introduced, when necessary, preservative agents in the laboratory.

When sampling, we attached a hose to the sampling tap. We opened the tap and let the water flow for a few minutes to purge the pipe, to have truly representative samples.

Then we filled the bottles, to the brim from the bottom, to prevent gas dissolution or evaporation.

Sampling was carried out at the test point at the water tower.

Immediately, certain analyzes (temperature, pH, conductivity and total dissolved solids) are carried out in situ.

Subsequently, the sample was stored in vials containing preservation reagents, cooled in a cooler and sent to the laboratory for further testing and physicochemical analyses.

The study was carried out on the water sample taken from the borehole in the commune of Diouroup in accordance with the protocol described. This water is brackish and hyperfluorinated.

Laboratory analyzes consist of determining the concentrations of major and minor cations and anions in natural waters.

For the analysis of chlorides, fluorides, sulfates, carbonates and hydrogen carbonates we use plastic and glass bottles containing no preservatives.

As for the analyzes of potassium, sodium, calcium and magnesium, we used bottles in which we put a few drops of concentrated nitric acid (HNO_3) in order to lower the pH of the samples to a lower pH at 2.

The water studied was collected at the borehole in the commune of Diouroup and, during sampling, we took care to collect information on the hourly and daily flow rates from the borehole. The analyzes carried out on the raw water focused on the major ions (Ca^{2+}, Mg^{2+}, Na^{+}, K^{+}, Cl^{-}, F^{-}, SO_4^{2-}, NO_3^{-}).

The zircon used was kindly offered to us by the Senegalese company Grande Côte Opération (GCO), a subsidiary of the Eramet group. This company, whose two processing plants are established in Diogo, and the mining deposit is located in the dunes of the Thiès and Louga regions, specializes in the valorization of zircon, ilmenite, rutile and leucoxene.

The zircon image is shown in Figure 2.

Figure 2. *Zircon.*

Once the zircon was obtained, we proceeded to determine its composition and its characterization. The determination of the composition of zircon, as well as the chemical analyzes of the filtrate are carried out in the Chemistry laboratory of the Applied Nuclear Technology Institute (ITNA) of the Cheikh Anta Diop University of Dakar (UCAD).

The determination of the zircon composition is carried out with an X-ray fluorescence spectrometer, the Niton XLT900s.

The zircon samples are directly presented to the X-ray fluorescence analyzer (Figure 3) with a silver Ag anode as an excitation source and an optimized large geometry detector equipped with several filters as secondary sources. Figure 4 clearly illustrates the Niton XLT900s X-ray tube with the filter layout.

Figure 3. *X-ray fluorescence spectrometer, the Niton XLT900s.*

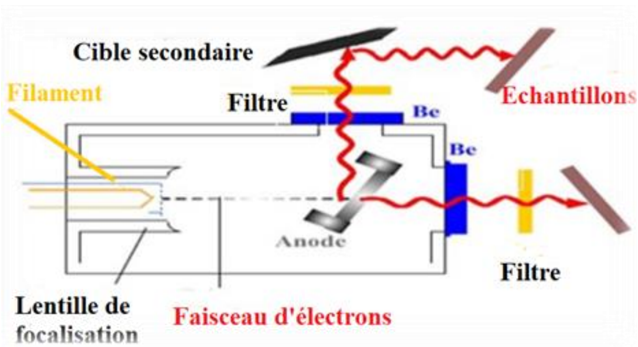

Figure 4. *X-ray tube with typical excitation geometry.*

Regarding the analysis of the filtrates, we used the EDXRF spectrometer (Figure 3) to determine the metals and other elements they contain.

For water analysis in non-destructive testing (NDT: Non-Destructive Testing) mode, a volume of 2 µL of sample was taken and then deposited on a nucleopore film. And with the dispersive energy X-ray fluorescence technique we determine all the metals present in the effluent if the contents are not lower than the detection limit. However, matrix effects must be corrected using standard solutions.

2.4. Adsorption Devices (Column Filtration)

2.4.1. Filtration Equipment

To carry out our defluration tests on hyperfluorinated brackish water by adsorption or filtration on a zircon column, we created a single column device with zircon.

This single column device consist of:

1) a 10 liter tank of raw water to filter,
2) a water inlet valve to be filtered,
3) a perforated connection sleeve without filter,
4) an adsorbent column (zircon)
5) a perforated connection sleeve with filter,
6) a shut-off valve and regulation of the filtration flow and
7) from a treated (filtered) water tank.

The zircon column is a plexiglass tube 50 mm in diameter and 100 mm in height containing 155 g of zircon with a density of 3156 g/L.

The diagram and photo of this device are illustrated in Figure 5.

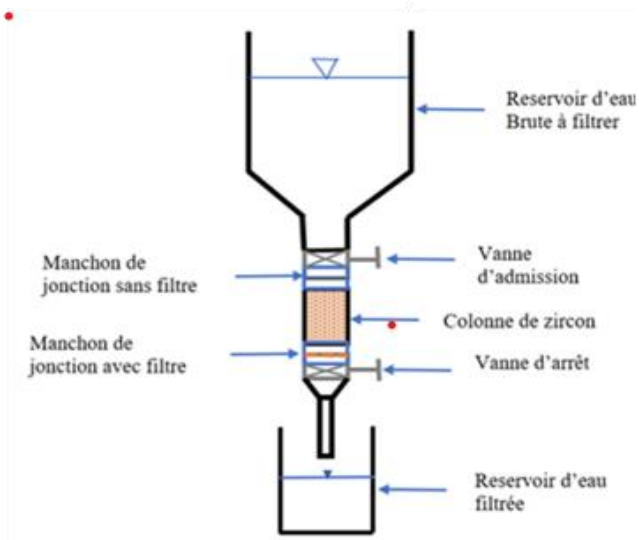

Figure 5. *Schematic of zircon filter (B).*

2.4.2. Filtration Procedure

The methodology adopted for these tests is batch filtration on an absorbent column.

After characterizing the raw water, we introduced five hundred (500) mL of water to be treated, into the device tank, we opened the inlet valve, we adjusted the filtration flow to 0.83 mL /s with the stop valve and we collected the filtrates in beakers. For each filtration test, the contact time between the water and the filter mass is ten (10) minutes.

Once the filtrations were completed, we stored the treated water in bottles that we refrigerated before sending them to the various laboratories for analysis.

The results of analyzes of these filtrates will be presented in the results and discussion section.

3. Results and Discussions

After having exposed the problem of hyperfluorinated brackish water in the world in general and in Senegal in particular and especially in its central region, we opted for the defluoridation process by the adsorption treatment process on zircon at the domestic level.

The need to verify the effectiveness of this technique requires the inventory and acquisition of equipment and methods allowing practical tests to be carried out in order to achieve convincing results.

Thus, after sampling and laboratory tests following defined protocols, we present in this part the results obtained.

The chronology that we adopted for the presentation of these results is as follows:

1) the results of the characterization of the raw water;
2) the results of the determination of the composition of zircon;
3) the results of the characterization of the zircon used;
4) the results of the analysis of the filtrates from the adsorption column.

3.1. Studies of Physicochemical Parameters of Raw Water

We present in Table 1 the results of the data collected in the field, analyzes carried out in situ and those carried out in the chemistry laboratory of the Department of Geology of the Cheikh Anta Diop University of Dakar (UCAD) on the raw water from the borehole. from Diouroup.

***Table 1**. Physico-chemical characteristics of raw water.*

Nature of the sample	Underground water
Sampling location	Diouroup
Drilling Information	
Hourly Flow (m^3/h)	20
Period Dry season, Daily flow (m^3/d)	50
Winter period, daily flow (m^3/d)	40
Physico-chemical parameters	
pH	8.16
Temperature (℃)	33.1
Conductivity (µS/cm)	3500
Total dissolved solids: TDS (mg/L)	2302.46

Cations	Concentrations (mg/L)	Anions	Concentrations (mg/L)
It $^{2+}$	8.16	Cl^-	750.33
Mg^{2+}	3.75	SO_4^{2-}	123.80
Na^+	759.19	HCO_3^-	610.00
K^+	24.79	NO_3^-	5.16
		F^-	6.41

3.2. Zircon Compositions

The result of determining the compositions of zircon (the adsorbent used in this study of defluoridation of brackish water in the commune of Diouroup) is presented in Table 2.

These results are obtained following analyzes 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. *Composition of the Zircon used.*

Elements	Nature of matter	Symbols	Concentrations (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	MB	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	Ti	1.13	0.13
Tungsten	Transition metal	W	0.60	0.07
Niobium	Transition metal	No.	0.42	0.05
Nickel	Transition metal	Neither	0.38	0.04
Money	Metal	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	-

Table 2 indicates that the predominant constituents of zircon are zirconium (80.83%) and silica (15.32%). We can say that it is mainly made up of zirconium and metal-based elements. Zircon acts as both an adsorbent and an ion exchanger.

This zircon is made up of 97.39% metals, the most important of which form numerous compounds with halogens or neutral or ionic molecules. These properties of the majority constituents of zircon make it an adsorbent with high defluorination capacity.

3.3. Characterization of Zircon

This characterization was entirely carried out in the sanitary engineering laboratory of the Ecole Polytechnique de Thiès (EPT). After acquiring the zircon, we proceeded with its characterization, the results obtained below which we present.

This characterization is essentially the determination of the iodine and methylene blue indices of zircon. We present these results in Table 3.

Table 3. *Iodine and methylene blue indices of zircon.*

Materials	Iodine index	Methylene Blue Index
Zircon	526.61	705.98

The iodine index value of zircon (526.61 mg/g) is slightly lower than those of commercial activated carbons (between 838 and 876 mg/g) [14]. These results show that zircon develops a good adsorption capacity of MB.

3.4. Results of Filtrate Ion Analyzes

In the following figure 6, we present the concentrations of ions in the zircon filter filtrate. This analysis was carried out in the chemistry laboratory of the geology department of the Cheikh Anta Diop University of Dakar (UCAD).

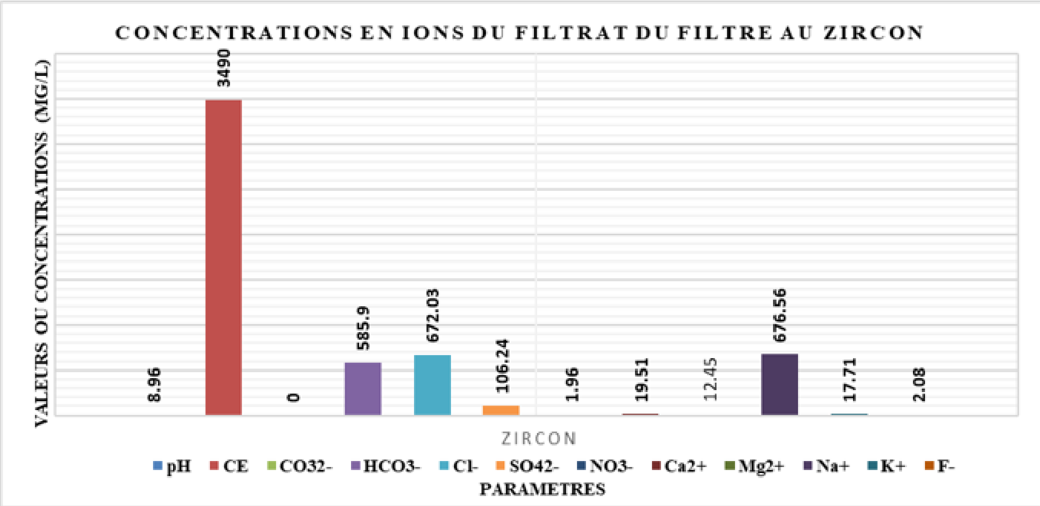

Figure 6. *Graph of zircon filter filtrate analysis results.*

The recovery rates obtained with the zircon filter are presented in Figure 7.

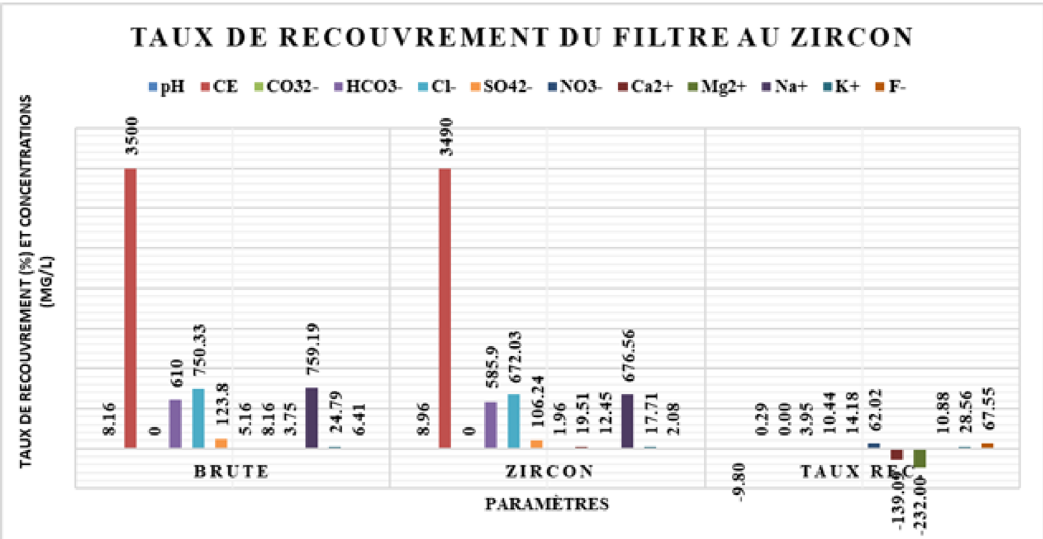

Figure 7. *Recovery rate with the zircon filter.*

The results obtained show that filtration on the zircon column considerably reduced fluorides, however, the reduction of chlorides and sodium is less significant.

In fact, this filtration of raw water from the Diouroup borehole made it possible to obtain reduction rates of:

1) fluorides of 67.55% (i.e. from 6.41 mg/L to 2.08 mg/L),
2) sodium of 10.88% (i.e. from 759.19 mg/L to 676 mg/L),
3) chlorides of 10.44% (i.e. from 750.33 mg/L to 676 mg/L) and
4) calcium by -139.09% (i.e. from 8.16 mg/L to 19.51 mg/L).

While the limits (standards) set are respectively 1.5 mg/L for fluorides, 200 mg/L for sodium, 250 mg/L for chlorides and 200 mg/L for calcium [15-17].

Thus, the recovery rate of 67.55% shows that this process is well suited to the specific elimination of fluorides but requires the control of certain parameters, namely pH and contact time. In fact, the adsorption capacity depends on the specific surface area of the material, the nature of the adsorbent-adsorbate bond, the contact time, the pH and the initial fluoride concentration.

On the one hand, let us mention that in this study, we worked with raw alkaline water (pH = 8.16) while the ionic radii of the hydroxyl and fluoride ions are comparable; in addition the hydroxyl and fluoride ions are isoelectronic. This means that at high pH (OH^- numerous), hydroxyl ions compete with fluoride ions [18, 19].

Furthermore, during our tests, our retention times were 10 minutes, while many studies on defluorination by zirconium-based composites recommend a minimum retention time of 30 minutes, to obtain a better rate. yield [20, 21, 18].

Furthermore, the very high concentrations of bicarbonates (610 mg/L) significantly reduce the defluoridation capacity [20, 18].

Thus, the purifying efficiency of the process implemented can be improved, by intervening on the influencing parameters, such as the retention time or the pH.

3.5. Results of Analyzes of Elements Released by the Filter

Below, in Table 4 we present the results of the analyzes of the elements released by the zircon filter.

Table 4*. Concentrations of elements released by the zircon filter.*

Elements	Raw water (mg/L)	Zircon Filtrate (mg/L)	Releases (mg/L)
That	12.9	12,953	0.053
Cr	0.864	0.89	0.026
Se	0.89	0.628	-0.262
Neither	2.46	2.46	0
MB	18.02	16.57	-1.45
Zn	2,277	1,779	-0.498
Cu	1,465	1,492	0.027
Co	1,413	1,361	-0.052
Fe	5,914	5,783	-0.131
Mn	2.015	2.015	0
K	6,542	5,338	-1.204
Ba	50.06	50.21	0.15
Zr	17.71	17.57	-0.14
S	425.9	353.66	-72.24
Cl	281.16	76.47	-204.69

These results reveal that the release of zircon filter elements is insignificant. Only calcium and chromium were released into the filtrate at very low levels of respectively 0.053 mg/L for calcium and 0.026 mg/L for chromium.

This release into the filtrate really has no alarming effects since:

1) As an indication, the old directives of the Council of the European Communities indicated a guide level of 100 mg/L as the calcium content in water intended for human consumption. While the limits (standards) set by the WHO for calcium are 200mg/L [15-17] and;
2) the WHO as well as the directives of the Council of the European Communities and French regulations relating to the quality of water intended for human consumption have adopted the figure of 0.05 mg/L as the limit value for chromium [15-17].

In short, we note that neither of these two elements released in the zircon filtrate exceeds the permitted limits.

4. Conclusion

In Senegal, a phosphate producing country, the presence of phosphate ores (hydroxyapatite) is often associated with that of fluorine ores (fluorapatite).

In addition, geotechnical and Hydrochemical studies on the Senegal sedimentary basin have shown that water from the Maastrichtian aquifer is an important source of water supply for the populations of the peanut basin. However, they contain significant quantities of fluorides and chlorides.

Commonly encountered fluoride concentrations can reach maximum values of 20 mg/L. These excess fluorides in drinking water are the cause of serious poisoning, dental and bone fluorosis. This fluoride poisoning is confirmed by an epidemiological survey carried out in 2008 in 45 villages in the departments of Mbour and Fatick. This survey revealed that 70% of the 900 people surveyed are in contact with fluoride levels above 1.5 mg/L (value accepted by the WHO guide). 83.6% are also affected by dental fluorosis while 52.7% show clinical signs that could be associated with bone fluorosis.

In the world in general and more particularly in countries facing this problem, several types of defluoridation processes have been studied and implemented for the purification of hyperfluorinated brackish water.

In this context, we have thus made a contribution to the defluoridation of hyperfluorinated brackish water intended for the consumption of the populations of the commune of Diouroup region of Fatick.

We first presented the problem of hyperfluorinated brackish water in the world in general and in Senegal in particular and especially in its central region. We then opted for physicochemical treatment by adsorption on zircon.

It appears from the characterization results that zircon has an iodine number of 526.61 mg/g. Regarding the methylene blue index, the results showed a value of 705.98 mg/g. These two indices prove that zircon is a good adsorbent.

The results were presented in the following sequential order: raw water characterization, zircon composition determination, zircon characterization, and adsorption column filtrate analysis.

In the process of defluoridation of hyperfluorinated brackish water from Diouroup, we adopted a physicochemical type treatment process based on the principle of adsorption on an adsorbent column (zircon).

The samples taken from the site were analyzed and then tested using a physicochemical process.

We summarize below the results from this process, we obtained overall a reduction in fluoride contents of 67.55% (i.e. from 6.41 mg/L to 2.08 mg/L) in chlorides of 10.44% (i.e. from 750.33 mg/L to 676 mg/L) and sodium of 10.88% (i.e. from 759.19 mg/L to 676 mg/L).

Filtration with this zircon column released small amounts of calcium and chromium. The contents of these releases are respectively 0.053 mg/L for calcium (limits 200 mg/L) and 0.026 mg/L for chromium (limits 0.05 mg/L).

We have thus observed that none of these elements released in the filtrate of the zircon column exceeds the permitted limits. We can therefore affirm that zircon can be used for the defluoridation of hyperfluorinated brackish water without harmful consequences on the health of consumers.

Therefore, from our results, it appears that the adsorption defluorination process is effective.for the treatment of hyperfluorinated brackish water. Indeed, the physicochemical process implemented made it possible to considerably reduce the fluoride and chloride contents.

Furthermore, it should be noted that WHO and European standards are not rigorously respected. In fact, the standards set by the WHO and the European Union are respectively 1.5 mg/L for fluorides, 200 mg/L for sodium, and 250 mg/L for chlorides. However, chloride levels can be exceeded and reach thresholds of 900 mg/L, particularly in arid areas. The only drawback of these high chloride values is an unpleasant taste, except for certain patients.

Our study opens up perspectives that we can discuss below.

The purification yields of the physicochemical process (filtration on a zircon column) implemented can be improved, by intervening on the influencing parameters, such as the retention time or the pH. In fact, we carried out our tests with a retention time of 10 minutes, whereas in most studies the minimum time is 30 minutes to 120 minutes. This retention time plays an important factor in the adsorption treatment process increases the removal capacity.

In addition, the pH of the samples used is alkaline, whereas in previous adsorption studies carried out around the world, it turned out that the adsorption phenomenon is optimal with an acidic pH.

Therefore, further studies should be considered to adjust operating parameters such as retention time and pH to improve the purification efficiencies of the zircon-based adsorption column.

After which, we will be able to produce prototypes of filters for home uses.

Finally, we propose to extend this study to other groundwater in the area to test the effectiveness of this technique for eliminating fluorides and chlorides in order to confirm the results from this study.

Author Contributions

Alioune Ly: Funding acquisition, Investigation, Methodology, Resources, Validation, Writing – original draft

Anabilaye Moussa Coly: Formal Analysis, Validation, Writing – original draft

Ndiouga Camara: Validation

Baba Ngom: Writing – review & editing

Diadioly Gassama: Conceptualization, Supervision. Validation, Visualization, Writing – original draft

S éni Tamba: Project administration, Supervision

Conflicts of Interest

The authors declare no conflicts of interest.

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