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Treatment by adsorption on a dual-layer filter of hyperfluoridated brackish water from the Ndamatou borehole in Touba, Senegal

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Abstract

Fluorine is a common component of natural water and plays an important role in many metabolic processes, when present in small quantities in the human body.

A concentration higher than 1.5 mg/L, as set by the World Health Organisation (WHO), can cause serious health problems, such as dental or bone disorders, Alzheimer's disease and infertility.

Various methods have often been used to remove excess fluoride from water, but their cost and difficulty of application are barriers to accessibility for middle-income populations.

The aim of this study is to apply adsorption technology by superimposing two adsorbents, namely zircon and carbon made from filao litter, activated with calcium chloride. The treated samples were taken from the Ndamatou borehole in Touba (Senegal), and raw water characterisation has led to the conclusion that this water is brackish and hyperfluorinated. A 500 mL sample was passed through a double-layer filter with a contact time of 10 minutes, resulting in a 66.45% reduction in fluoride ions, from a concentration of 4.65 to 1.56 mg/L. Analysis of the other constituents of natural water showed reductions of 25.22% for bicarbonates. On the other hand, increases

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were noted for chlorides (104.28%), sulphates (65.89%), nitrates (46.87%), calcium (1141.76%), magnesium (323.80%), sodium (13.93%) and potassium (41.43%). These results demonstrate the mixed filter's ability to retain certain pollutants and release others. In most cases, the concentrations obtained in the filtrate meet the standards or do not present a risk to human health. The increase in chlorides and calcium can be attributed to the activating agent.

Introduction

A multitude of chemical components are present in water, including fluorine [1][2]. A common constituent of natural water samples, it can originate from geological and anthropogenic sources [3]. The main contribution comes from geological resources such as biotite, sellaite, fluorite (or fluorspar), cryolite, topaz, tremolite, fluormica, epidote and fluorapatite) [1][3][4][5]. In phosphate-producing countries, the presence of hydroxyapatite is often associated with that of fluorinated minerals or fluorapatite [6]. Senegal, the sixth largest phosphate producer in Africa [7], may experience excessive fluorine levels in water from its confined aquifers [6].

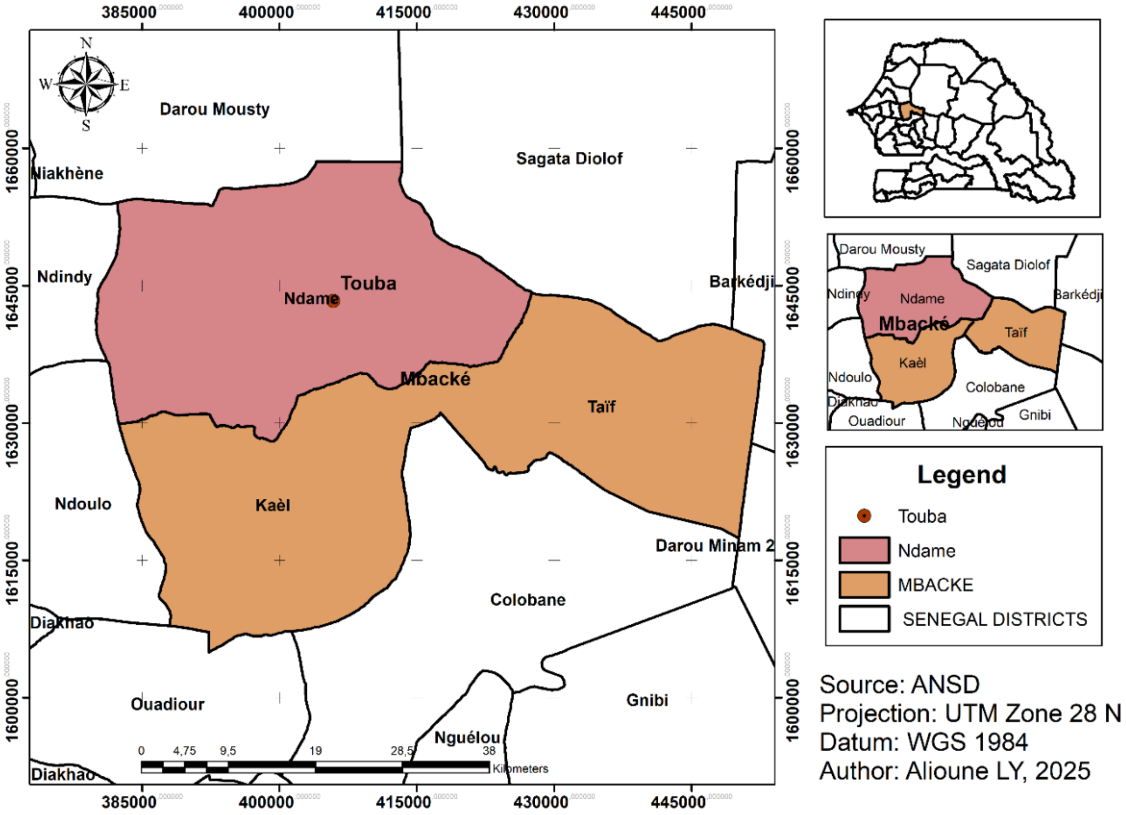
Adsorption could be a versatile, optimal and viable method of defluorination due to various factors, including cost, diversity of its end applications, socio-cultural acceptance, regulatory compliance, environmental safety and simplicity [8]. It describes the ability of certain materials to retain ions or molecules (gases, metals, organic molecules, etc.) on their surface in a more or less reversible manner [9]. It is a surface phenomenon in which a liquid or gas molecule, known as an "adsorbate", is attracted to another molecule present in a solid, known as an "adsorbent". These two molecules will strive to achieve the most advantageous energy configuration at the interface [10]. Adsorption is one of the water treatment methods used to remove dissolved non-degradable organic matter, which is extracted from the liquid or gaseous phase in which it is found [11]. Its application is limited by various challenges, including limited adsorption capacity, relatively slow adsorption rates, restricted pH range, and high cost of certain adsorbents [4]. Certain materials have shown significant potential for fluorine adsorption. Among them can be mentioned quartz, clay, fly ashes, calcite, sodium-exchanged montmorillonite (Na+), bauxite, bone carbon, activated alumina, zircon and activated carbon [6][12]. These elements are usually used alone or in combination with other approaches to ensure therapeutic efficiency [13].

The aim of this study is to propose a method for decontaminating hyperfluorinated water by adsorption in Senegal, in Ndamatou area, in Touba, by superimposing two adsorbents. For this purpose, we used a double-layer or mixed filter, consisting of a column of activated carbon topped with a column of zircon. The zircon comes from the Diogo mine in Senegal and the carbon, made from filao litter, was activated with calcium chloride and carbonised at 600°C. The two adsorbents were combined in order to achieve a more significant reduction. After passing through the double-layer filter with a contact time of ten (10) minutes, the results showed a 66.45% reduction in the fluoride ions contained in the raw water. The concentrations of other elements present, both in the raw water and composition of the two materials constituting the filter, were also measured in the filtrate. This was done in order to quantify their reduction or release and measure the effectiveness of the filter in retaining those different substances. In order to meet health standards, the concentrations obtained were compared with WHO norms.

1. Methodology

1.1. Presentation of the study area

Located at 14°52' and 15°30' west and 14°30' and 15°00' north, in the west-central region on the edge of Ferlo, Touba city stands out for its demographic, economic and cultural importance in Senegal's urban architecture. It covers an area of 375 km² with a population density of 4409 inhabitants per square kilometre [hab. /km²]. Thus, it remains one of the localities with the highest population densities in the country. From only 400 hectares [ha] in 1928 (the year of the first lease), the built-up area did not change significantly until 1976, when it increased to 575 ha. It reached 3,975 ha in 1990 and 12,000 ha in 1997, before exceeding 30,000 ha in 2002. Touba changed from being a residence in 1888 to a village at the beginning of the 20th century until 1976, then to a Rural Community (RC) as a result of the 1996 territorial reform under the Act III of Decentralisation [14].



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Author: Alioune LY, 2025

Figure 1. Presentation of Touba city.

1.2. Sampling

The study was conducted on water samples taken from the Ndamatou borehole in Touba.

Sampling was carried out, at the test point, at the water tower. Before proceeding, glass and plastic bottles were washed, rinsed and labelled. Preservative agents were added, if required.

A hose was attached to the sampling tap during picking. The tap was opened, allowing the water to flow for a few minutes, to purge the pipe and obtain representative samples. Certain parameters (temperature, pH, conductivity and total dissolved solids) were measured in situ.

The pre-prepared bottles were filled from brim to bottom, in order to prevent gas dissolution or evaporation. They are then kept cool in a cooler and transported to the laboratory for further physicochemical analysis. As the water sampled is hyperfluorinated, laboratory analyses consist of determining the concentrations of major and minor cations and anions in natural waters.

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

For the analysis of potassium, sodium, calcium and magnesium, bottles containing a few drops of concentrated nitric acid (HNO_3) were used to lower the samples pH below 2.

1.3. Treatment process

1.3.1. Filtration equipment

Defluorination tests, using adsorption on an activated carbon column made from filao litter, topped with a zircon column, were carried out using the following setup:

- An inlet valve,
- A valve for admitting water to be filtered,
- A junction sleeve without filter,
- An adsorbent column (zircon),
- A junction sleeve with filter,
- An adsorbent column (activated carbon made from filao litter),
- A junction sleeve with filter,
- A shut-off valve and filtration flow regulator,
- A filtered water tank.

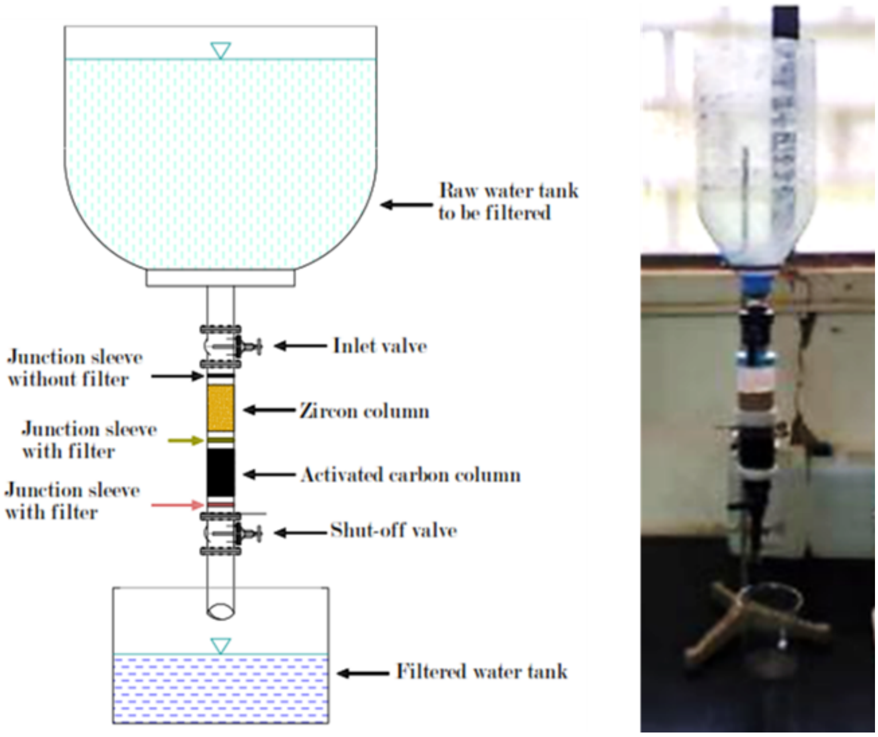

Figure 2. Double column diagram (activated carbon topped with zircon).

This experiment was carried out after carbon activation. The quantification of the constituent elements released of the two adsorbents into the filtrate was studied along with the efficiency of the process in water purification. The superposition adopted helps to retain the non-absorbed elements by the carbon, after the water has passed through the zircon column, as the carbon has a larger specific surface area.

1.3.2. Filtration procedure

After characterising the raw water, we introduced five hundred (500) mL of water to be treated into the tank. The inlet valve was then opened and the filtration flow rate was adjusted to 0.83 mL/s, using the stop valve. The filtrates are collected in beakers. The contact time between the water and the filter mass was ten (10) minutes. The treated water is then stored in bottles and refrigerated before being sent to the various laboratories for analysis.

2. Results and discussion

2.1. Composition and characterisation of zircon and activated carbon

The characterisation of the two materials was carried out entirely at the Sanitary Engineering Laboratory of the Ecole Polytechnique de Thiès (EPT). The activated carbon was characterised by determining its levels of humidity, volatile matter, ashes and fixed carbon, as well as its iodine and methylene blue indices.

For zircon, the iodine and methylene blue indices were determined.

The two adsorbents used in the study were zircon (A) and activated carbon (B).

The zircon comes from the Diogo mine, located in the dunes of the Thiès and Louga regions. The carbon was obtained after carbonisation at 600°C of filao litter collected along the Atlantic Ocean coast in the Mboro area and minced in the laboratory. It was then activated with calcium chloride ($CaCl_2$).

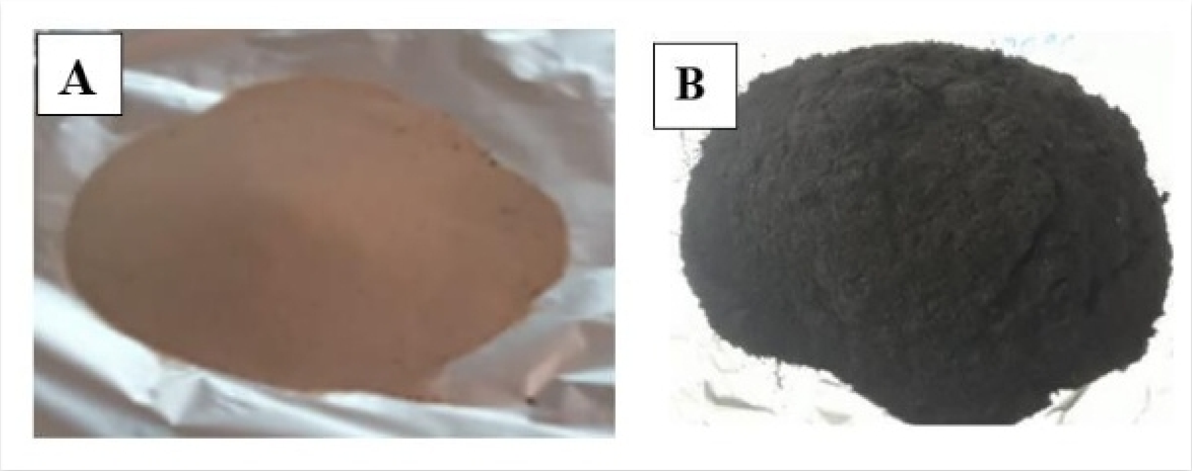

Figure 3. Zircon from the Diogo mine (A) and activated carbon made from filao litter (B).

The composition of the zircon and activated carbon was determined following analyses carried out with an X-ray fluorescence spectrometer, the Niton XLT900s, in the laboratory of the Institut Technologique de Nucléaire Appliquée (ITNA) at the Université Cheikh Anta Diop de Dakar (UCAD).

The principle involves presenting zircon and activated carbon samples directly to the X-ray fluorescence analyser (figure 4-a), with a silver (Ag) anode as the excitation source and an optimised wide-geometry detector fitted with several filters as secondary sources. The sample is subjected to intense X-rays, which ionise the atoms by ejecting electrons in the layers close to the nucleus. The atom then returns to a stable state by reorganising its electron cloud. In particular, the space left by the ejected electron is filled by an electron from a more external layer.

The Niton XLT900s X-ray tube with the filter arrangement is shown in figure 4-b.

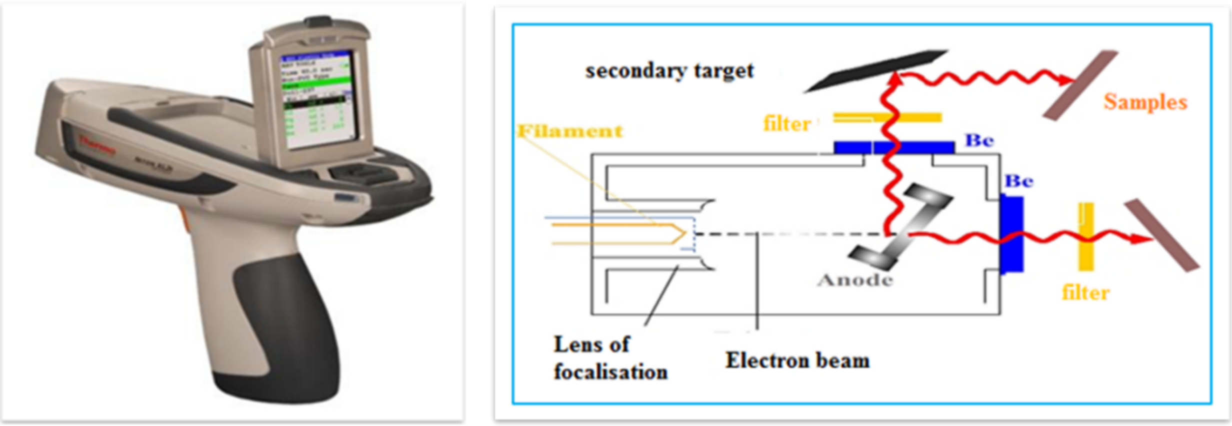

fig 4-a **fig 4-b**

Figure 4. X-ray fluorescence spectrometer, the Niton XLT900s (fig. 4-a) and X-ray tube with typical excitation geometry (fig. 4-b).

Figure 5 illustrates the composition of the zircon and activated carbon used, as well as the level of each of the constituent elements.

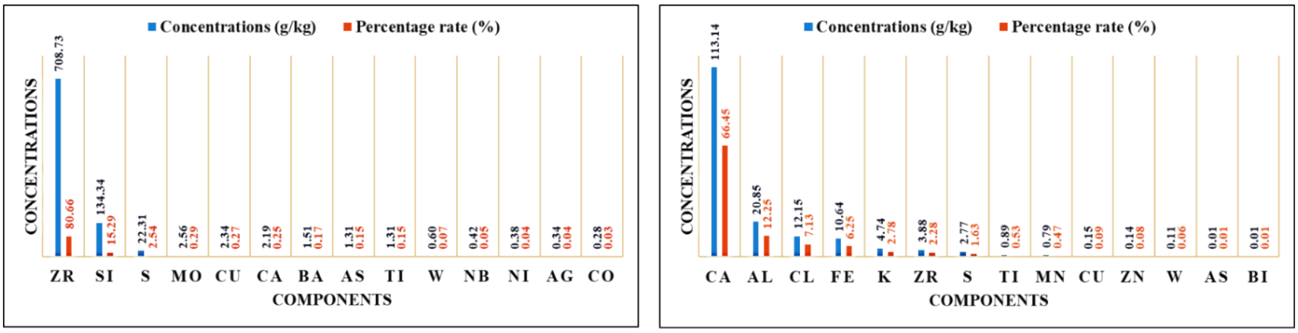

fig 5-a **fig 5-b**

Figure 5. Composition of zircon (fig. 5-a) and activated carbon (fig. 5-b).

Both materials are essentially made up of transition metals, alkaline earth metals, metalloids, metals and non-metals. The presence of halogen (chlorine) and alkali metal (potassium) is noted in activated carbon.

The transition metals present in both zirconium and activated carbon are zirconium, titanium, copper and tungsten. The zirconium content of zircon is estimated at 80.66%, compared with 2.28% for activated carbon. The levels of copper, tungsten and titanium are low in the composition of both materials. Tungsten and copper are higher in zircon, while titanium in activated carbon (0.53%) is higher than in zircon (0.15%). Niobium, nickel and cobalt are present at very low levels in zircon, 0.05%, 0.04% and 0.03% respectively. Activated carbon contains 6.25% of iron and 0.47% of manganese.

The alkaline-earth metal common to both materials is calcium, with a very high level in activated carbon (66.45%) compared with 0.25% in zircon. Activated carbon contains 12.25% of aluminium. A silica content of 15.29% was recorded in the zircon composition, along with a low barium content (0.17%).

The metalloid and non-metal present in the two materials are arsenic and sulphur respectively. The level of arsenic in both zircon and activated carbon is relatively low, although the level in zircon (0.15%) is higher than that in activated carbon (0.01%). The sulphur content of zircon (2.54%) is higher than that of activated carbon (1.63%).

Silver is present in zircon with a rate of 0.04% The two metals present in activated carbon are bismuth (0.01%) and zinc (0.08%).

The composition of activated carbon contains 7.13% of chlorine and 2.78% of potassium.

Figure 6 provides information on the characterisation of activated carbon dried and then carbonised in oven at 600°C.

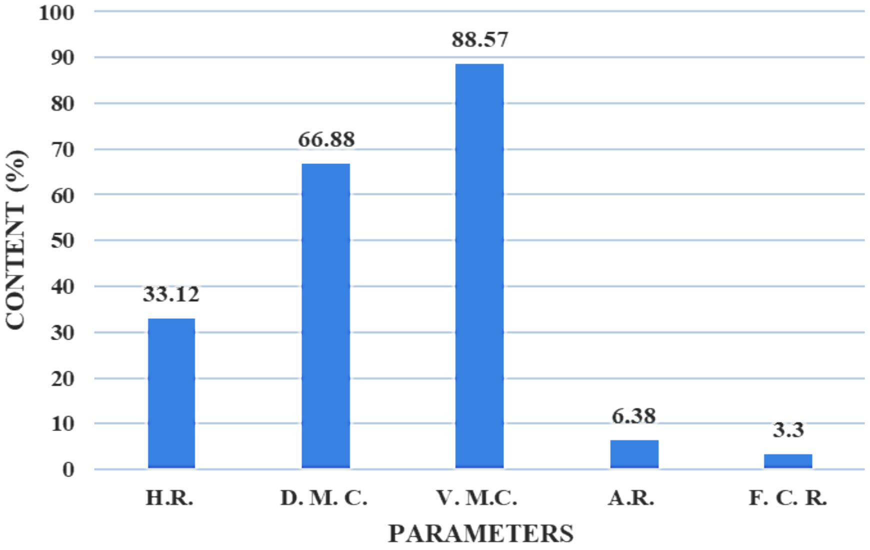

Figure 6. Characterisation of activated carbon.

Humidity and dry matter obtained immediately after drying the activated carbon were, respectively, 33.12% and 66.88%.

After carbonisation in oven at 600°C, volatile matter dominated with a rate of 88.57%, followed by ashes (6.38%). Fixed carbon had a lower rate compared with the other characteristics (3.3%).

Activated carbon has higher indices for both iodine and methylene blue. The iodine values for activated carbon and zircon are 1063.56 and 526.61, respectively. The methylene blue indices are 842.80 for activated carbon and 705.98 for zircon.

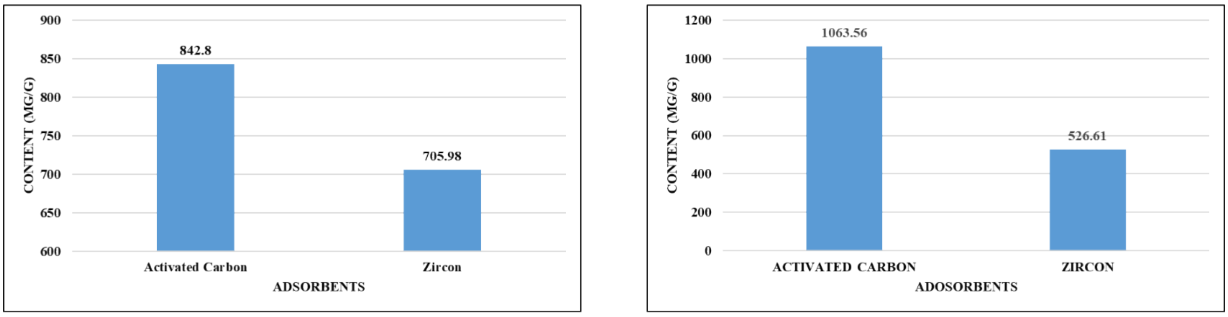

fig 7-a **fig 7-b**

Figure 7. Methylene blue indice (fig. 7-a) and iodine indice (fig. 7-b).

After identifying the elements present both in the raw water and the two adsorbents composition, the differences in concentrations between the raw water and the treated water are listed in Figure 8.

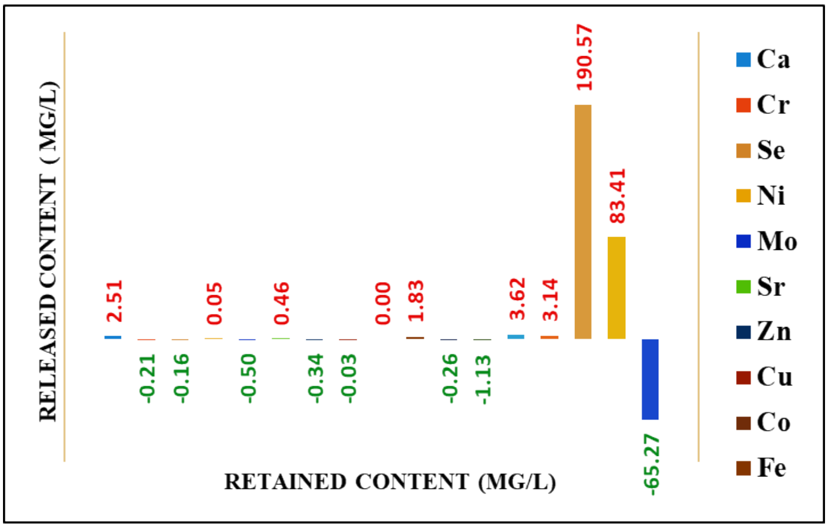

Figure 8. Material release and retention.

An increase is noted in the concentration of certain elements in the filtrate. These are calcium (Ca), nickel (Ni), iron (Fe), barium (Ba), zirconium (Zr), sulphur (S), with respective additional percentages of 19% (2.51 mg/L), 2% (0.05 mg/L), 48% (1.83 mg/L), 8% (3.62 mg/L), 22% (3.14 mg/L), and 25% (83.41 mg/L), compared with the concentration in the raw water. The release of calcium (Ca) and zirconium (Zr) can be attributed to both materials (zircon and activated carbon), as they are present in the composition of each. Nickel (Ni), barium (Ba) and sulphur (S) are only found in the composition of zircon, so their release depends on it. Iron is only present in carbon composition, so its release can be affected to it.

Molybdenum (Mo), copper (Cu), manganese (Mn), potassium (K), chlorine (Cl) and zinc (Zn) were found to be 3% (0.50 mg/L), 2% (0.03 mg/L), 13% (0.26 mg/L), 16% (1.13 mg/L), 25% (65.27 mg/L) and 14% (0.34 mg/L) lower, respectively than the initial concentration. Despite their presence in one or other of the two materials, these elements were retained by the two-layer filter.

Cobalt has the same concentration both in raw water and the filtrate.

Humidity level of activated carbon should be less than 5%, while the ashes rate should not exceed 10%. Higher values can raise the pH and cause carbonates to precipitate from the water onto the carbon, thereby reducing its adsorption capacity [15]. Low humidity can be an advantage for activated carbon with a high gross calorific value [16]. The humidity content of the activated carbon (33.4%) used in the study greatly exceeded the prescribed value (5%), and bicarbonates were reduced by 25% while carbonate concentration remained the same. The formation of precipitates on the activated carbon during the process cannot be ruled out, leading to a reduction in adsorption capacity. The ashes content (6.38%) does not exceed the recommended value (10%). A high ashes content reduces the specific surface area, as the inorganic nature of the ashes makes the activation process difficult. Ashes obstruct pores of the carbon structure [17].

The volatile matter or loss on ignition values is 88.57%. This indicates a high degree of graphitisation, a high gross calorific value and a large number of functional groups [18].

The level of fixed carbon found (3.3%) is very low. A high level of fixed carbon reduces the adsorption capacity of activated carbon. Its inorganic nature makes the activation process difficult, as it contributes to clogging the pores of the carbon, thereby reducing the adsorption sites [19].

The iodine number is the quantity of iodine adsorbed by 1 g of adsorbent in an aqueous solution of I_2 of normality 0.02 N [16][18]. It is used to measure the content of micropores (< 1 nm) in adsorbent, indicating the possibility of significant adsorption of fine molecules [20]. Activated carbons with an iodine value greater than 950 are able to adsorb small molecules such as those responsible for tastes and odors [16]. The values obtained for zircon and activated carbon are, respectively, 526.61 and 1063.56.

The methylene blue index is used to evaluate mesopores (between 1 and 25 nm) and macropores (> 25 nm), which is synonymous with a high adsorption capacity for medium and large molecules [16][20][21][22][23]. The methylene blue indices obtained for activated carbon and zircon are, respectively, 842.80 and 705.98.

According to the iodine and methylene blue indices, activated carbon has a higher porosity than zircon. This justifies the position of the carbon column below the zircon one, in order to retain as many particles as possible that have escaped when passing through the first column.

Activated carbon made from filao litter and zircon are both quality adsorbents with a greater adsorption capacity for carbon than for zircon. However, the humidity level of the carbon, which is very high, needs to be adjusted.

2.2. Treatment effectiveness

The effectiveness of fluorination is assessed by comparing the values measured before and after filtration.

Figure 9 shows the evolution of physicochemical parameters in water before and after treatment.

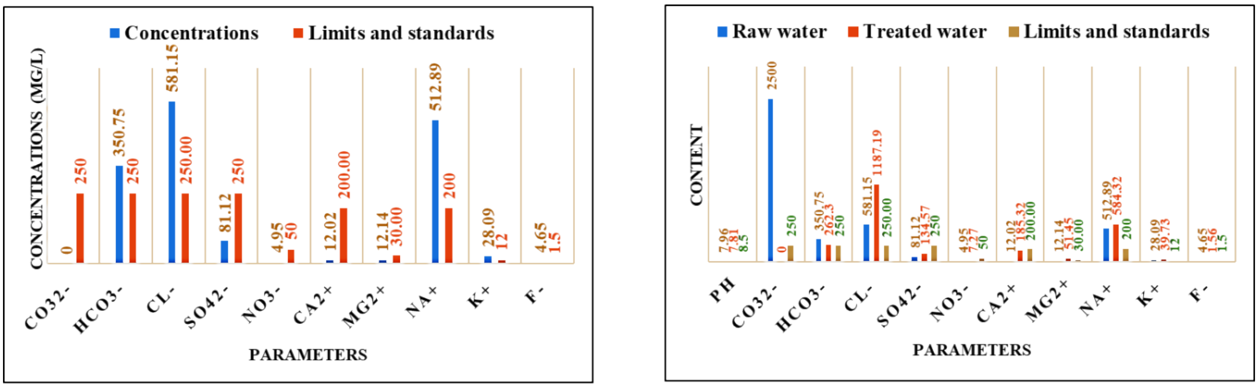

fig 9-a

fig 9-b

Figure 9. Raw water characterisation (fig. 9-a) and treatment efficiency (fig. 9-b).

A pH of 7.96 was measured in the raw water, compared with 7.81 in the filtrate obtained, corresponding to 1.88% reduction from the initial value.

Conductivity increased from 2500 µS/cm in the raw water to 4080 µS/cm in the treated water, corresponding to a 63.2% increase of the initial value.

An increase was noted in the concentration of elements such as chlorides, sulphates, nitrates, calcium, magnesium, sodium and potassium. Bicarbonates and fluorides decreased while carbonates' concentration remained constant both in raw and treated water.

Chlorides rose by 104.28%, sulphates by 65.89%, nitrates by 46.87%, calcium by 1441.76%, magnesium by 323.81%, sodium by 13.93% and potassium by 41.44%.

Reductions of 25.22% and 66.45% were respectively recorded for bicarbonates and fluorides.

The defluorination process with activated carbon can give good efficiency, but the sorption process is very pH-dependent and is effective when the pH is below 3 [20]. The pH values obtained before and after treatment are respectively 7.96 and 7.81. These values are within the optimum range of 6.5 to 9.5 recommended by the WHO for drinking water [24]. However, acidification of the sample prior to treatment would enable the sorption capacity of the activated carbon to be fully exploited.

Conductivity is used to assess the overall mineralisation of water. It depends on the ionic strength of water and increases as a function of the dissolved ion content, the nature of the various dissolved substances, their concentration and the temperature at which it is measured. According to the World Health Organisation (WHO), the quality limit for water intended for human consumption should be between 200 and 1100 µS/cm [19][25][26]. The conductivity of both raw water (2500 µS/cm) and treated water (4080 µS/cm) exceeds the maximum value for the range. This high conductivity value in raw water confirms its brackish nature [27]. The increase after treatment could be due to the high presence of sodium chloride, generated by the treatment process [19].

Results obtained, after activating the carbon and passing the sample through the mixed filter, showed that copper had a reduction of 0.03 mg/L (2%) and sulphur, an increase of 83.41 mg/L (25%), compared with the initial concentrations. These elements are present both in raw water, zircon and carbon compositions. Iron, potassium and chlorides are present only in raw water and carbon. An increase of 1.83 mg/L (48%) of iron concentration is noted in the filtrate, showing its release during the treatment process. Potassium and chlorides have respective reductions of 1.13 mg/L (16%), and 65.27 mg/L (25%) compared with the initial concentration. These elements are therefore not released during the treatment of fluoride by the filter and this one is able to retain a certain proportion of these constituents, even if it is not very significant.

Iron, potassium and chlorine do not have guide values for drinking water. However, a taste can be detected above a chlorine concentration of 250mg/L. Residual copper concentration in the filtrate (1.47 mg/L) does not exceed the standard set by the WHO (2mg/L) [24].

Surpluses of 2.51 mg/L (19%) and 3.14 mg/L (22%), compared with the initial concentration, were found in the filtrate, respectively for calcium and zirconium, present both in the composition of the two adsorbents and in the raw water. Nickel and barium present in raw water and zircon composition showed respective increases in concentration of 0.05 mg/L (2%) and 3.62 mg/L (8%). Molybdenum, also present both in the two absorbents and raw water shows a decrease of 3% (0.5mg/L) in the filtrate, compared to the initial value. Manganese, present only in raw water and carbon composition, saw its concentration decrease by 0.26 mg/L (13%) compared with the initial concentration. This shows that the filter is able to retain certain elements with an acceptable rate but at the same time unable to do so with some of them.

WHO standards do not specify guide values for zirconium and calcium. For barium, molybdenum, manganese and nickel, WHO standards are 0.7 mg/L, 0.07 mg/L, 0.4 mg/L and 0.07 mg/L respectively [24]. In the filtrate, standards are not met for these elements, with much higher values: barium (49.42 mg/L), molybdenum (16.14 mg/L), manganese (1.83 mg/L), nickel (2.51 mg/L). It should be noted that concentrations of raw water were higher than the standards and the filter used to treat it, even able to retain some of them, was not able to meet WHO standards.

Results obtained for the treatment effectiveness of the mixed filter showed a consistency in carbonate and reduction in bicarbonate and fluoride concentrations.

Carbonates and bicarbonates do not have any limit values recommended by WHO [24]. The pH of most natural waters is between 4.5 and 8.3. Their alkalinity is mainly due to bicarbonates (HCO_3^-), which are weak bases, converting into free CO_2, when neutralised by an acid [19]. At pH values above 8.3, carbonates may be present, and at pH values above 9.5 hydroxides. Alkalinity, in combination with hardness, pH and acidity, contributes to the calcium-carbonate balance. The effects of this balance can sometimes be scaling (protective layer) or incrustation (attacks on pipes and tanks) [28]. Alkaline pH causes precipitation of these ions and a drop in adsorption capacity [15]. Reductions have been observed for bicarbonates, showing a certain retention capacity for this pollutant by the adsorbents while carbonates concentration remains the same. In addition, pH of the water to be treated, near to the alkaline one, which favours bicarbonates precipitation, alters this result's positivity.

From a value of 12.02 mg/L in raw water, calcium concentration rose to 185.32 mg/L in filtrate. This increase may be due to the activating agent, calcium chloride.

Calcium, dominant element in drinking water, is the major component of water hardness. Its influence on human health has often been discussed. It exists mainly in form of hydrogen carbonates and in smaller quantities in form of sulphates, chlorides, etc. There is no danger in drinking water with a high calcium concentration if you are in good health. A limit of 200 mg/L is recommended by WHO [19].

WHO standards for drinking water recommend a concentration of 50 mg/L for nitrates [24]. From 4.95 mg/L in raw water, the concentration increases to 7.27 mg/L in treated water. The dual-layer filter is therefore not suitable for removing nitrates from water where the concentration could exceed the standards, even if in this case, the concentration recorded is far lower than the standard set.

WHO standards for drinking water set a limit of 1.5 mg/L for fluoride [24]. From an initial value of 4.65 mg/L, the concentration fell to 1.56 mg/L. This value is a little higher than the limit recommended by WHO (1.5 mg/L). However, the treatment quality can be improved by increasing contact time between adsorbents and raw water. Contact time between water and adsorbent is a key parameter in any adsorption process, as it influences the size of the installation and adsorbent consumption [29]. Some studies have used contact times up to 60 minutes [30], 180 minutes [20], 10 to 360 minutes [31], to ensure better treatment quality. A more substantial abatement, which can be far lower than the WHO standards, can therefore be obtained by adjusting this parameter.

Chlorides do not have a limit value, but a taste can be noted above a concentration of 250 mg/L [24]. The taste may be attenuated by the presence of calcium and magnesium, for an equivalent quantity of chlorides [19].

A concentration of 1187.19 mg/L was found in the filtrate, compared with 581.15 mg/L in the raw water. This increase could be due to the activating agent in the carbon, calcium chloride.

Sodium levels were 512.89 mg/L in the raw water, compared with 584.32 mg/L in the filtrate. For taste reasons, a concentration of 200 mg/L is recommended by the WHO [28].

The concentration of potassium in raw water rose from 28.09 mg/L to 39.73 mg/L in the filtrate. Its presence in natural water does not usually exceed 10 to 15 mg/L and the perception threshold for potassium chloride is around more than 20 times this value [19].

These high concentrations of chloride, calcium, sodium and potassium are indicative of brackish water.

Sulphates can cause gastrointestinal problems, particularly in children. Their laxative action is accentuated by the presence of magnesium. Magnesium salts, and sulphates in particular, have a laxative effect above 400 to 500 mg/L. Above a concentration of 100 mg/L, magnesium gives water an unpleasant taste [19].

Despite the increase observed, sulphates do not exceed the standard limit value (500 mg/L) [24]. The concentrations found in the raw and treated water were respectively 81.12 and 134.57 mg/L. The magnesium concentrations found in the raw water and treated water were respectively 12.14 mg/L and 51.45 mg/L, indicating an increase in the filtrate. These values are well below the risk values. These elements therefore present no danger in this scenario.

Conclusion

This study explored a new approach, using a superimposed filter made up of two adsorbents, zircon and activated carbon, to ensure greater removal of fluoride ions. The activated carbon made from filao litter, which has a larger specific surface area, was placed below the zircon column to retain anything that might have escaped. A 66.45% reduction in fluoride ions was obtained. The final concentration found in the filtrate (1.56 mg/L) is a little higher than WHO standards (1.5 mg/L). Analysis of other components of natural water, such as carbonates, bicarbonates, calcium, magnesium, sulphates, sodium, potassium, etc., showed increases or decreases in the filtrate, reflecting the filter's ability to retain or release them. The concentrations found do not present any danger to human health, as they are below standards or do not present any risk at certain concentrations.

Conductivity, chloride, calcium, potassium and sodium values show that the water to be treated is brackish, as well as being hyperfluorinated. Results obtained in the filtrate, after carbon activation, showed a significant release of calcium chloride, contributing to an increase in the water salinity.

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