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**35th WEDC International Conference, Loughborough, UK, 2011****THE FUTURE OF WATER, SANITATION AND HYGIENE:  
INNOVATION, ADAPTATION AND ENGAGEMENT IN A CHANGING WORLD****Experimentation on bone char-based treatment for fluoride  
removal from drinking water in Senegal***S. Sorlini & D. Palazzini, Italy***REFEREED PAPER 1260**

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*In several areas of Senegal fluoride concentration in drinking water exceeds World Health Organization (WHO) guide value. A safe, efficient, simple and low cost defluoridation technique needs to be developed in order to prevent the occurrence of fluorosis. This paper describes a laboratory and pilot experimentation carried out using animal bone char as adsorbent material for fluoride removal. Possible influencing parameters, such as specific ions in Senegalese drinking water, were investigated and the best process conditions were defined for the application in Senegal. The results attest the efficacy of bone char in removing fluoride from Senegalese water: the mean specific adsorption resulted 2.72 mgF/g of bone char at pilot scale, corresponding to a total treated volume of 4,000 L and a filter life of 3 months.*

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**Introduction**

Fluoride is a chemical compound naturally present in groundwater depending on the nature of the bedrocks (Fawell et al., 2006). WHO suggests that its concentration in drinking water should not exceed 1.5 mg/L, in order to prevent dental (yellowing of teeth) and skeletal fluorosis (permanent bone and joint deformations and osteosclerosis). Around 200 millions of people, living mostly in Developing Countries (DCs), are particularly affected by this disease. In these contexts, fluoride in groundwater becomes a problem when there aren't alternative sources and defluoridation treatment is the unique solution for delivering safe water.

In DCs, conventional treatments (chemical processes of precipitation, electro dialysis, adsorption with activated alumina and membrane process) aren't suitable, because of their high costs and technical complexity. Therefore, treatments capable to combine effectiveness, technical simplicity and low costs are necessary. One of the most promising defluoridation methods is the adsorption with bone char. It is a porous granular material, composed of calcium phosphate (57-80%), calcium carbonate (6-10%), and activated carbon (7-10%). Its ability to take up fluoride from water is due to hydroxyapatite,  $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ , where one or both the hydroxyl-groups can be replaced with fluoride (Fawell et al., 2006). Considerable researches at laboratory scale (Mwaniki, 1992; Larsen et al., 1994; Dahi and Bregnhøj, 1995; Phantumvanit and Legeros, 1997; Puangpinyo and Osiriphan, 1997; Mjengera and Mkongo, 2003; Watanesk and Watanesk, 2000; Abe et al., 2004) on fluoride removal from water with bone char-based treatment were conducted all over the world. In these studies the parameters influencing fluoride removal efficiency were investigated, like the calcination method (temperature and duration time of the process, oxygen content), the bone char quality (colour) and granulometry, the initial drinking water quality (content of F, Cl, I, Na<sup>+</sup>, K<sup>+</sup> ions). At domestic level, bone char defluoridation was experimented in Thailand (Phantumvanit et al., 1988), Tanzania (Mjengera and Mkongo, 2003; Jacobsen and Dahi, 1997), Ethiopia (Esayas et al., 2009) and Kenya (Korir et al., 2007).

In 2004, CeTamb (Research centre on appropriate technologies for environment management in Developing Countries) started a research on bone char-based treatment in Senegal, in collaboration with the University "Cheikh Anta Diop" of Dakar. During the experimentation, bone calcinations, batch tests, filtration tests and leaching tests were performed with particular attention to Senegalese local context in order to define the optimal process conditions, identify possible influencing parameters and calculate bone char efficiency.

## Materials and methods

### Animal bone calcinations

Five calcination tests were executed varying the type of kiln and other operational conditions.

#### *Kilns*

The first four tests were executed in two kilns designed for cooking purposes in DCs. Both of them are made of: a combustion chamber obtained from an old oil drum, an external insulating layer (sand), an air inlet and a chimney for the gas exit. These kilns are different in dimension (kiln A:  $\Phi=50$  cm and height=1 m; kiln B:  $\Phi=30$  cm and height=80 cm) but they are substantially the same as regards their functioning. Subsequently, the kiln design was improved in order to satisfy the real needs of bone char production and to guarantee better conditions of insulation and air control during the process. As consequence, a third kind of kiln (kiln C) was tested, whose design was derived from a low-tech furnace experimented in Tanzania (Jacobsen and Dahi, 1997).

#### *Operational conditions of the tests*

Table 1 summarizes the operational conditions applied during the calcination tests. The calcinations 1 and 2 were performed to investigate the bones disposition influence on the process. Test 3 was performed to evaluate a different combustion method consisting in maintaining the bones in contact with the hot combustion smoke and not directly with the fuel. The calcination 4 was compared with the calcination 2 to verify the influence of different bones parts. The calcination method 5 was tested to verify its applicability at real scale: the kiln was enlarged, the fuel quantity was increased, the wood was used in addition to coal in order to facilitate the combustion start up and cow shoulder blades were used (no flesh residues and medulla that could remain unburned and that could negatively affect drinking water treatment).

Test	Kiln	Q.ty of raw bones (kg)	Type of bones	Fuel quantity (% of raw bones)	Fuel type	Raw bones disposition in the kiln
1	A	17.5	unselected cow bones	8	coal	coal at the bottom of the kiln and bones at the top
2	B	10	unselected cow bones	8	coal	bones and coal mixed together
3	A	8.8	unselected cow bones	122	coal	coal in the kiln and bones in an external pot crossed by the hot combustion smoke
4	B	2.5	cow horns (from Senegal)	8	coal	bones and coal mixed together
5	C	30-40	cow shoulder blades	20 10	coal wood	coal at the bottom of the kiln and bones at the top

#### *Bone char samples*

The products of calcinations were divided into black, white and grey portions, that were crushed, sieved and finally weighed to calculate the productivity of each calcination process. The bone char samples with grains smaller than 2 mm were employed in the batch tests, while the portion with a granulometry between 0.2 and 4 mm was employed in the filtration tests.

### Fluoride removal tests

#### *Batch tests*

In the first series of batch tests, all the samples of bone char (black, white and grey portions) obtained from calcinations 1, 2 and 3 were employed and mixed with distilled water containing 5 mg/L of fluoride (as in

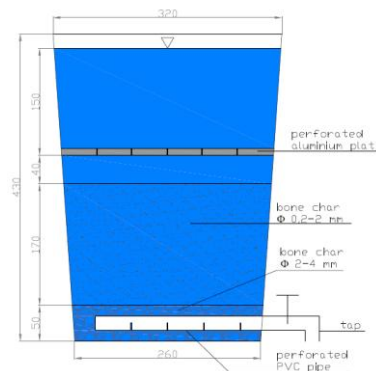
Senegalese groundwater). First, the contact time was fixed (30 min) and the adsorbent dosage was varied between 10 g/L and 80 g/L (10-20-40-60-80 g/L); this allowed to select the optimal dosage. Then, the optimal dosage was fixed and the contact time was varied between 2.5 min and 30 min (2.5-5-10-20-30 min) in order to select the optimal contact time used in the following tests.

The second series of tests were carried out in order to investigate the bone char granulometry influence on fluoride removal and to choose the optimal size range to be used in the filtration treatment. In this case, grey and black portions from calcination 5 were tested.

Finally, the third series of tests were performed to investigate the drinking water quality influence on fluoride removal, taking into account the specific characteristics of Senegalese drinking water that has a high salts content. Natural Senegalese water samples and distilled water samples, in which different chloride and sulphate concentrations were artificially added, were used. In these third series, the grey bone char from calcination 5, with 0.2-2 mm grain size, was tested. Initial fluoride concentration, bone char dosage and contact time were respectively 5 mg/L, 40 g/L and 5 min, during both the second and third series of batch tests.

### ***Filtration tests***

For these tests, a pilot filter (Figure 1 and Photograph 1) was constructed taking into account its applicability at domestic level in Senegal. It consists of a plastic bucket with a 30 L capacity and equipped with a tap, a perforated PVC pipe rapped with nylon cloth and connected to the tap, a perforated aluminium plate that protects bone char filter and avoids turbulence and a cap. The bucket was filled with 8 kg of grey bone char from the calcination 5: 2 kilos, with 2-4 mm grain size, were put at the bottom of the bucket and they constituted the draining and supporting layer; 6 kilos, with 0.2-2 mm grain size, were put on the coarse bone char and they constituted the main adsorption layer. Two tests were conducted varying the operational conditions of the filter. In the first case the filter was used discontinuously and the total treated volume was 100-200 L/d. The second test was conducted in order to simulate the local families practices; the filter was daily used and the total treated volume was 50 L/d, assuming 10 components per family and a per-capita drinking water use of 5 L/d. In each case, the filter was fed with Senegalese drinking water, with an initial natural fluoride concentration between 5 and 7 mg/L, in downflow condition and with an EBCT of 8 min.



**Figure 1. Scheme of the pilot filter**



**Photograph 1. Picture of the pilot filter**

### **Leaching tests**

These tests were conducted in order to verify the possible leaching of undesired substances from bones to the water. The method, defined in the UNI 14456 for the characterization of bone char used for drinking water treatment, was applied. An amount of 330 g of grey bone char from calcination 5, with a granular size of 0.2-2 mm, was put in a column, having 50 cm of height and 3 cm of inner diameter, and drained with 21 bed volumes of water (80 mL necessary to fill the gap between the bone char particles). The 20<sup>th</sup> and the 21<sup>st</sup> bed volumes were left in contact with bone char for 24 h and retained for the analyses (1<sup>st</sup> and 2<sup>nd</sup> leachates respectively). Three tests were executed varying water and bone char samples. During the tests 1 and 2 distilled water was left in contact with grey and black bone char respectively, while in the test 3 Senegalese water was maintained in contact with grey bone char.

## Analytical methods

During the calcination tests, the temperature was measured in the firebox (at the top and the bottom). At the end of the processes the medium values were calculated and compared.

During the fluoride removal and leaching tests, drinking water quality was monitored before and after treatment. Fluoride and pH concentration were measured by using a fluoride selective electrode (WTW pH/Ion 340i Pocket Meter). Total Ionic Strength Adjustment Buffer (TISAB) was added to water samples before the analysis in order to stop the effect of the interfering ions during fluoride measurements. Chloride, sulphate and other ions concentrations were measured by using a photometer (WTW Photoflex Turb Set).

## Results

### Animal bone calcinations

The influence of the bone char disposition in the kiln during the combustion process isn't so evident, as the total produced bone char and the temperature curves (Figure 2) reached in bones (mean value between the top and the bottom of the kiln) during the tests 1 and 2 are very similar. In any case, the calcination 1 results better than the method 2, because it produces a higher amount of black and grey portions, able to give the best performances in terms of fluoride removal (Mwaniki, 1992; Larsen et al., 1994; Phantumvanit and Legeros, 1997; Puangpinyo and Osiriphan, 1997). This was one of the reason why the method 5, defined for the application in Senegal, reproduces the same conditions of the calcination 1, but at higher scale. Also the calcination 3 offers good results, in terms of quantity and quality of bone char, but it has the following disadvantages: the smoke exits freely disturbing the calcination executor (the chimney isn't used); a lower amount of raw bones can be loaded in the kiln; an higher amount of fuel is used with respect to bone char and it isn't so easy to perform if compared with the calcination 1.

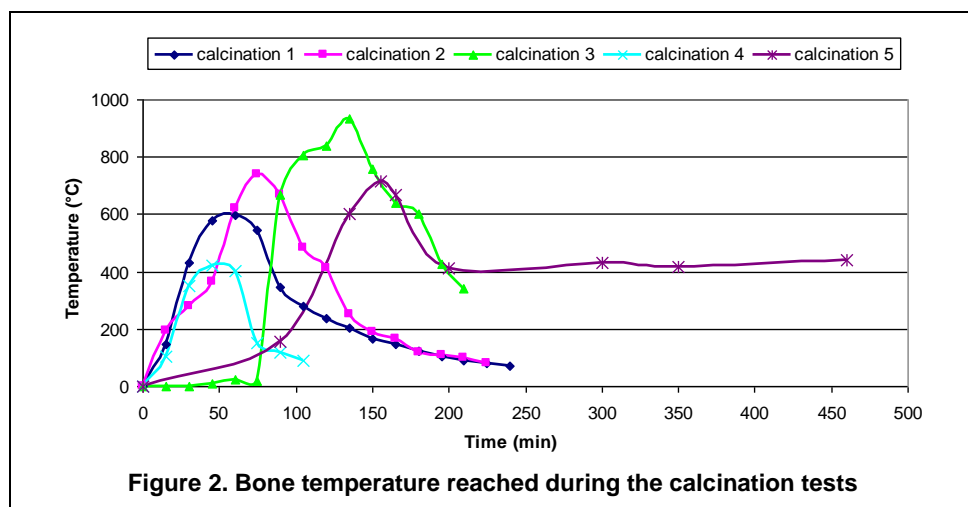


Figure 2. Bone temperature reached during the calcination tests

Test 4 demonstrates that the cow horns aren't suitable for the calcination process. This type of bones burns too fast and strongly, even in presence of lower temperatures, and it is very difficult to control the fire. As consequence, the process produces an high quantity of white bone char, that offers a low fluoride removal (Mwaniki, 1992; Larsen et al., 1994; Phantumvanit and Legeros, 1997; Puangpinyo and Osiriphan, 1997). Otherwise, the use of cow shoulder blades during the test 5 is successful, as the desired result of obtaining an important percentage of grey bone char is reached.

The results also indicate that during the transformation process from animal bones to bone char (including crushing and sieving) a material loss of 70%-80% occurs. This phenomenon is due to the bones weight loss, that in the previous experiences was 35-40% (Dahi and Bregnhøj, 1995), the presence of unburned pieces at the end of the calcinations processes and the natural dispersion of the volatile bone char. The reduction in weight during the calcination 4 is lower (50%) than in the other cases because the horns are much dry and, as consequence, the water content loss is less important. Finally, it was calculated that the calcination method 5 can produce a bone char quantity sufficient to fill one pilot filter.

Finally, the tests demonstrate that this kind of kilns, designed for the use in DCs, doesn't allow to fix accurately the temperature around the optimal value of 550°C for about 4 hours (Fawell et al., 2006), as the

only way to control it consists of opening or closing the air inlet. After the fire ignition, the calcination process continues naturally without adding any other fuel. As consequence, the temperature rises slowly, it reaches a peak value (controlled by the air inlet) and then it begins to go down. In Figure 2 it is possible to note that during the calcination 5 the temperature in the kiln rises slower than in the other cases, probably due to the kiln dimension, but after the peak value it remains constant, around 400°C, for about 4 hours.

### Batch tests

The first series of the batch tests allows to identify the optimal bone char dosage and contact time values, 40 g/L and 5-10 min respectively. In these conditions (Figure 3) and with an initial fluoride concentration of 5 mg/L the highest fluoride removal (more than 90%) is obtained with the black portions of bone char. Also the grey portions show good removals, between 56% and 80%, while the white bone char samples have very low removals (lower than 20%) and they aren't suitable for drinking water treatment. It is worth to notice that the water samples, maintained in contact with black bone char, get a yellow colour, but this phenomenon rarely happens in case of grey bone char use. All these statements agree with the previous experiences (Mwaniki, 1992; Larsen et al., 1994; Phantumvanit and Legeros, 1997; Puangpinyo and Osiriphan, 1997). As consequence, it was decided to use grey bone char, as it has good fluoride removal and, at the same time, it doesn't alter treated water quality. Finally, it is possible to notice that all the samples obtained from the calcination 1 have the highest fluoride removal in comparison with the same samples from the other calcinations. This is the second reason why the conditions applied in the calcination 1 were reproduced in the calcination 5 at real scale.

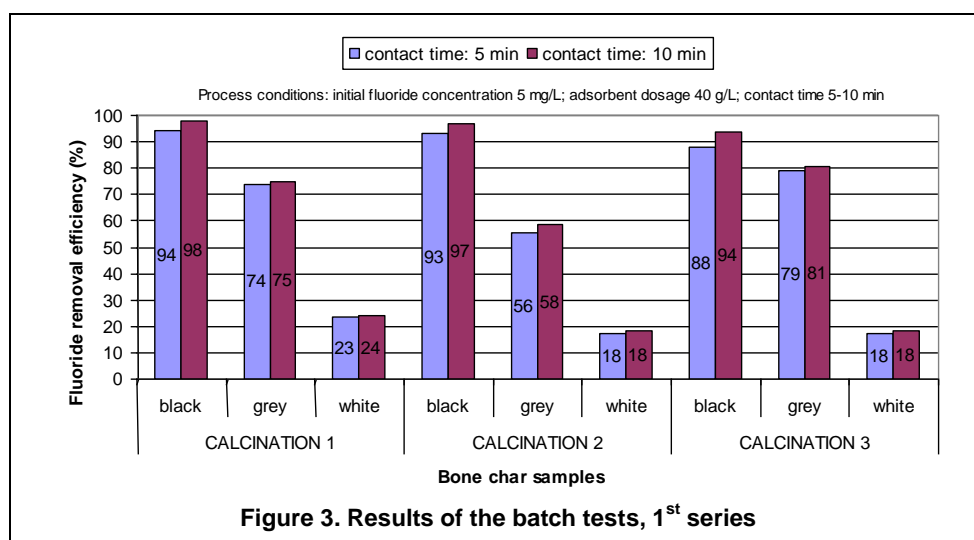


Table 2 indicates the more significant results of the second series of batch tests, during which the bone char quality was kept similar while the bone char granulometry was varied.

The 0.18-2.36 mm size was chosen as optimal value. Thanks to the finer particles, the grey portion with this granulometry has an high fluoride removal efficiency (90%), similar to that of the black bone char (99%). In fact, in a previous study, it was found that the particles smaller than 0.42 mm have a fluoride adsorption higher than 90% (Mwaniki, 1992). Furthermore, this granulometry can guarantee the correct hydraulic filter operation. On this regard, another study (Mjengeru and Mkongo, 2003) indicates a recommended range of 0.5 - 4 mm for bone char filtration.

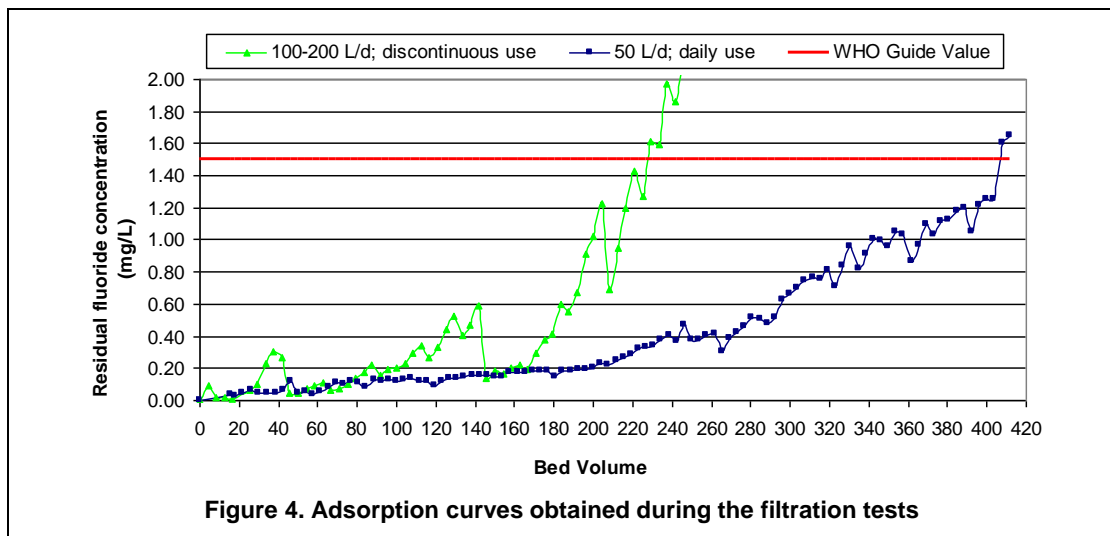
Bone char colour	Bone char dosage (g/L)	Contact time (min)	Initial fluoride (mg/L)	Bone char granulometry (mm)	Fluoride removal efficiency (%)	Treated water organoleptic quality
Black	40	5	5	0.18-0.6	99	colour and odour
Grey	40	5	5	0.18-0.6	90	no colour and odour
Grey	40	5	5	0.6-1.18	72	slight colour and odour
Grey	40	5	5	0.6-2.36	60	no colour and odour
Grey	40	10	5	0.6-2.36	80	no colour and odour
Grey	40	5	5	0.18-2.36	90	slight colour and odour

Table 3 indicates the more significant results of the third series of the batch tests, during which the initial drinking water quality was varied. The initial chloride and sulphate concentration, responsible for the high salts content in Senegalese water, doesn't significantly influence bone char fluoride removal efficiency, that is always higher than 90%. These results don't respect the statements of the previous experiences (Mwaniki, 1992; Abe et al., 2004), that observed an increasing fluoride uptake by bone char in presence of chloride ion. The difference could be due to the fact that, in this case, the initial fluoride, chloride and sulphate concentrations weren't sufficiently high to note important differences in fluoride removal by bone char. Even during the treatment of Senegalese water, a good fluoride removal was obtained.

Water sample	Bone char dosage (g/L)	Contact time (min)	Initial fluoride (mg/L)	Initial chloride (mg/L)	Initial sulphate (mg/L)	Fluoride removal efficiency (%)
Distilled water	40	5	5.0	0	0	97
Distilled water	40	5	5.0	900	0	97
Distilled water	40	5	5.0	450	0	97
Distilled water	40	5	5.0	0	70	93
Distilled water	40	5	5.0	00	35	98
Distilled water	40	5	5.0	900	70	100
Senegalese water	40	5	4.8	872	84	94
Senegalese water	40	5	5.4	830	74	84

### Filtration tests

In Figure 4 the fluoride adsorption curves of the two pilot filters are represented, where the bone char is considered exhausted when the WHO guide value is reached in the treated water.



The residual concentration of fluoride detected in water after filtration on filter 1 (irregularly used with a 100-200 L/d down flow) increases fast after a Bed Volume (BV) of 160 and the exhaustion of the filter is reached at a BV of 230. For the filter 2 (daily used with a 50 L/d down flow), fluoride residual concentration slowly increases until the bone char exhaustion is reached at a BV of 410. As consequence, filter 2 has a fluoride specific adsorption (3.72 mgF/g of bone char) higher than the filter 1 (1.72 mgF/g). Finally, assuming that a Senegalese family of 10 components needs about 50 L/d of drinking water, the corresponding life of the filter 1 and 2 is 1.8 and 3.5 months respectively. The different behaviour of the filters could be due to their operational conditions (the use of the filter 1 is more intensive than that of the filter 2) and to the bone char quality (as the selection of the bone char portions with different colours can't be precise, it is possible that the filter 2 contained traces of black particles, that remove fluoride more efficiently). Finally, in Figure 4 it is also possible to note that fluoride concentration in the water treated by filter 1 sometimes falls down significantly and then it begins again to rise. This happens when filter isn't continuously used and water remains in contact with bone char for a long time. As consequence, the first treated water, after the re-start of the process, has a lower residual fluoride concentration.

### Leaching tests

The results of the leaching tests show that (Table 4):

- in general, the ions concentration increases after the contact between bone char and distilled water (test 1 and 2) and the phenomenon is more evident with the black bone char (test 2). On the contrary, after the contact between bone char and Senegalese water (test 3), only the phosphate concentration increases, while the other ions keep quite constant (sulphate). These observations are confirmed by the increase/decrease of the conductivity;
- the Dissolved Organic Carbon (DOC) always increases, but this phenomenon is very clear in the case of the black bone char (test 2). This could be due to the fact that black bone char is produced at low temperature, in accordance with the literature data (Mwaniki, 1992; Larsen et al., 1994; Phantumvanit and Legeros, 1997; Puangpinyo and Osiriphan, 1997), and it could be partially unburned;
- the hardness increases after the contact between black bone char and distilled water. On the contrary, the hardness remains unaltered/decreases if grey bone char is put into contact with distilled/Senegalese water.

These results confirm the decision to use grey bone char to treat water in Senegal.

TEST		pH	Cond. ( $\mu\text{S/cm}$ )	DOC (mg/L)	Hard. ( $^{\circ}\text{f}$ )	Ca <sup>2+</sup> (mg/L)	Mg <sup>2+</sup> (mg/L)	Na <sup>+</sup> (mg/L)	PO <sub>4</sub> <sup>3-</sup> (mg/L)	SO <sub>4</sub> <sup>2-</sup> (mg/L)	Cl <sup>-</sup> (mg/L)
1	blank	7.00	33	7	0.4	2.94	0.98	2.6	< DL*	< DL*	0.24
	1 <sup>st</sup> leachate	8.30	834	178	0.4	4.30	1.13	173	23	49	19.6
	2 <sup>nd</sup> leachate	8.06	904	192	0.8	4.98	1.19	182	22	55	28.9
2	blank	6,70	23	6	0.2	1.76	0.59	1.35	< DL*	< DL*	0.1
	1 <sup>st</sup> leachate	7.13	2120	5958	66	134	78	302	22.3	91	144
	2 <sup>nd</sup> leachate	6.90	2320	6302	75	150	100	337	20.6	98	176
3	blank	8.85	2330	8	3.4	6.8	4.69	492	< DL*	78	700
	1 <sup>st</sup> leachate	8.21	1080	231	0.4	1.5	1.06	223	15.3	76	43
	2 <sup>nd</sup> leachate	7.76	1393	184	0.4	1.6	1.50	264	12.9	80	45.6

\* DL: Detection Limit

## Conclusions

The results of this experimentation suggest the following concluding remarks:

- grey bone char with 0.2-2 mm grain size is suitable to treat drinking water in Senegal in a filtration process, as it is able to remove fluoride up to 90% (batch tests), it doesn't alter the organoleptic water quality and it maintains high efficiencies in presence of higher sulphate and chloride concentrations;
- a bucket filter filled with 8 kg of grey bone char, fed with Senegalese water at 5-7 mgF-/L and in downflow condition (EBCT 8 min), can last 2-3.5 months, considering a water treatment of 50 L/d. The specific adsorption ranges between 1.7 and 3.7 mgF-/g of bone char;
- the calcination conducted in the kiln C can produce an amount of grey bone char sufficient to fill one domestic filter.

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