

## Fabrication and Testing of Activated Alumina based Defluoridation filters with Yarn cartridges

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**Abstract:** We have fabricated and tested several defluoridation filters using activated alumina (DF-101) and yarn cartridges. The flow rate of the filter was controlled by fixing a pinhole plastic stopper at the outlet of the yarn cartridge. Both domestic and intermediate defluoridation filters of 11 liter capacity and 50 liter capacity were fabricated. The filters were tested using fluoride contaminated ground water. The various filter parameters like empty bed contact time, flow rate, fluoride uptake capacity, time before I<sup>st</sup> regeneration, and recurring cost of the filter were determined. Various water quality parameters like pH, TDS, Cl<sup>-</sup>, NO<sup>3-</sup>, Na<sup>+</sup>, K<sup>+</sup>, CO<sub>3</sub><sup>2-</sup>, HCO<sub>3</sub><sup>-</sup>, Total alkalinity, calcium hardness and magnesium hardness, total hardness were continuously monitored. These defluoridation filters with yarn cartridge being unbreakable can be handled easily. Moreover the yarn cartridge has been wrapped in several layer of nylon cloth which can be periodically washed and thus prevents the filter from choking. The performance of the defluoridation filters with yarn cartridges have been compared with similar filter fitted with ceramic cartridge.

**Keywords:** activated alumina, defluoridation, fluoride, filter.

### Introduction

In many parts of the world, high concentration of fluoride occurs naturally in ground water and causes wide spread fluorosis<sup>1-4</sup>. According to the World Health Organization (WHO), the desirable limits of fluoride in drinking water are 1 ppm and the maximum permissible limit is 1.5 ppm. The high fluoride level in drinking water has become a critical health hazard in many places of world. The countries which are most affected are India, Kenya, Turkey, Iraq, Iran, Afghanistan, Thailand, China and Japan<sup>1,1</sup>. Fluorosis is a disease which mainly affects the bones and teeth<sup>2,-3</sup>. About 95% of fluoride ingested the body is deposited in hard tissues like bones

and teeth, weakening their structure and making them brittle<sup>1,4</sup>. Dental fluorosis occurs on consumption of water containing greater than 2 ppm of fluoride for long periods. Dental fluorosis is characterized by yellow patches on the teeth which turns into brown streaks. The brown streaks may turn black and affect the whole tooth, which may get pitted<sup>4,6, 5</sup>. Skeletal fluorosis occurs when water containing more than 3-6 mg/L of fluoride is ingested. Some symptoms of skeletal fluorosis are stiffness and severe pain in the backbone, joints, hip and paralysis<sup>4, 5,6</sup>. The problem of fluorosis has reached alarming proportions, affecting 17 states in India including Rajasthan, Andhra Pradesh which are the most affected<sup>2,3</sup>. In Rajasthan state, all the districts have been declared as fluorosis prone areas<sup>7,-10</sup>. Fluoride containing rocks have been found in Sikar district of Rajasthan for example: Fluorospars ( $\text{CaF}_2$ ) and Fluoroapatite [ $\text{Ca}_5(\text{PO}_4)_3\text{F}$ ]. People of this region suffer from dental and skeletal fluorosis<sup>8,9,10</sup>.

Activated alumina (AA) is known to remove fluoride from drinking water and has been used extensively in defluoridation filter<sup>18-23</sup>. In the course of our study to design and fabricate defluoridation filters with activated alumina (AA), we had reported earlier the fabrication and testing of domestic defluoridation filters using ceramic cartridge and various quantity of AA<sup>24,25</sup>. These filters were sufficient to meet water requirement of one family (4-5 people). Further to meet the water demands of 3-4 households, the capacity of these filters was increased to 50 liters<sup>26</sup>.

We have now replaced the ceramic cartridges in the filter by yarn cartridges. These yarn cartridges are light in weight, unbreakable and can be handled easily. The ceramic cartridge gets choked with suspended particles in water thus need to be washed and cleaned with emery paper during the course of the operation of the filter. We circumvented this problem by using yarn cartridges wrapped with nylon cloth which can be washed easily, and in this paper we report the fabrication and testing of these defluoridation filters.

## Materials and Methods

### Fabrication of 11 litre capacity filters with single yarn cartridge without controlled flow rate {3kg (1)}:

Four layers of nylon cloth of 0.16 mm mesh size was wrapped around a yarn cartridge, fig 1. The nylon cloth was secured using a nylon thread. The nylon cloth covered yarn cartridge was fitted on the base of 11 liter food grade polypropylene containers (Bharati Houseware). A plastic tap was fitted at the base of second container. One hole of 1 cm diameter was made on the lid of a second food grade container. The container with the yarn cartridge was placed on top the second food grade container with tap, so that the outlet of the yarn cartridge protrudes through hole in the lid. The top container was filled with water washed 3 kg AA (DF-101, M/s Siddharth Industries, Surat, Gujrat, India). The photograph of the fabricated filter is shown in fig 2.



**Fig.1** Nylon cloth wrapped around a yarn cartridge



**Fig. 2** Domestic defluoridation unit (11 lit.) capacity with single yarn cartridge

**Fabrication of 11 litre capacity filters with single yarn cartridge with controlled flow rate {3kg (2)}:**

Using a sharp knife the sides of a mineral water bottle cap were cut, and the round plastic piece was fitted at the outlet of the yarn cartridge, fig 3. A fine hole of 1 mm diameter was made on the plastic cap to serve as an outlet for water. The fabrication of the rest of the filter was same as above.



**Fig. 3** Flow rate control plastic pin hole fitted at the outlet of the yarn cartridge

**Fabrication of 50 litre capacity filters {10 kg (Y)} with double yarn cartridge:**

Two nylon cloth covered yarn cartridges were fitted on the base of a 50 liter food grade polypropylene container (Time Technoplast, Bombay). A plastic tap was fitted at the base of second 50 lit. container. Two holes of 1 cm diameter was made on the lid of a second food grade container. The container with the two yarn cartridge was placed on top the second food grade container with tap so that one outlets of the yarn cartridge protrudes through hole on the lid. The top container was filled with water washed 10 kg AA. The photograph of the fabricated filter is shown in fig 4.



**Fig. 4** Intermediate level defluoridation unit (50 lit.) capacity with double yarn cartridge

**Analysis of water**

Concentration of fluoride was measured by Fluoride Ion Selective Electrode and meter (Orion Thermo Scientific, USA) after calibration using 1, 10 ppm standard fluoride solution. TISAB-III was used to control the ionic strength and de-complex fluoride. The pH and TDS was measured by pH and TDS meter. The pH meter was calibrated at buffer solution of pH = 7 and TDS meter was calibrated using by 1000 ppm KCl solution. The chloride ion concentration was determined by titrating with silver nitrate (0.0268 N) using potassium chromate as a indicator. The determination of calcium and magnesium hardness was done by 0.01 N EDTA titration using Erichrome Black-T and Patton and Reeder indicators (AR, CDH India Ltd). The carbonate and

bicarbonate concentration was measured by titration (0.02 N H<sub>2</sub>SO<sub>4</sub>) using phenolphthalein and methyl orange indicators. Nitrate was analyzed using Shimadzu 1800 UV-VIS Spectrophotometer by measuring the absorbance 220 nm and 270 nm wavelengths (1N HCl is added to prevent interference due to OH<sup>-1</sup> or CO<sub>3</sub><sup>-2</sup>). Sodium and potassium were measured by Flame Photometer (ESICO, India). The instrument was calibrating by using 20, 50 ppm Na<sup>+</sup> solution for sodium and 20 ppm K<sup>+</sup> ppm for potassium.

### Laboratory testing of the filter

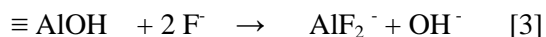
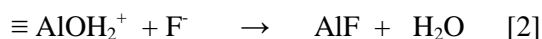
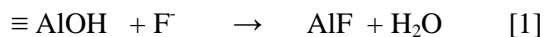
Fluoride contaminated ground water from nearby Bhooma Chota village was brought in the laboratory using 5000 liter tanker. The water was passed through the filter and various water quality parameters were monitored for both untreated and treated water.

### Characterization of Activated Alumina

The SEM and EDA X-ray analysis has been carried out at IIT Madras using Wave length Dispersive Spectroscopy (WDS).

### Result and Discussion

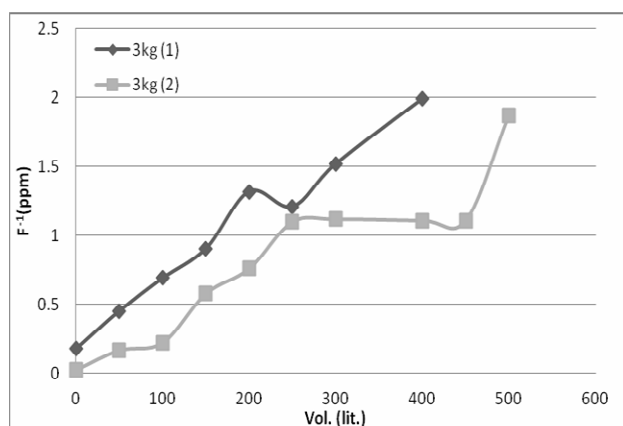
Fluoride is known to be absorbed on AA by the following mechanism <sup>27</sup>.



Where  $\equiv$  represents surface of AA, Eq. [3] is valid at very high concentration of fluoride <sup>28</sup>.

Domestic defluoridation units were fabricated at first using a single yarn cartridge with 3 kg of AA [3kg (1)]. This filter had a flow rate of 30 lit./h. which is approximately 15 times higher than that of similar filter with ceramic cartridge (1.7 lit./hr) <sup>24</sup>. The bed volume of this filter was found to be 4.05 liter and it had an empty bed contact time of 8.1 min. The filter produced 300 liter of safe water with an average fluoride inlet concentration is 3.57 ppm. The variation of fluoride concentration in treated water is shown in fig 5. The fluoride uptake capacity was found to be 282 mg/kg of AA. The fluoride uptake capacity of AA in this filter, is approximately four time less, (due to high flow rate) as compared to similar filter with same quantity of AA and ceramic cartridge (FUC was 1106.6 mg/kg AA) <sup>24</sup>. The lower FUC is due to two factors, a) low volume of safe water, b) high average fluoride concentration in raw water. In the raw water used for testing this filter the average fluoride concentration was 3.57 ppm, earlier when filter with ceramic cartridge was tested, the raw water had an average fluoride concentration of 2.41 ppm <sup>24</sup>.

The volume of safe water that is obtained from this filter was 300 liter, which is approximately 6 times lower than ceramic cartridge filter (volume of safe water was 2000 liter) <sup>24</sup>. Less volume of safe water produced from the filter requires that the filter need to be regenerated at a shorter time interval. The time before I<sup>st</sup> regeneration is 15 days for yarn cartridge and 3 month 10 days for ceramic cartridge filter <sup>24</sup> (at a water consumption of 20 lit./day). The recurring cost of 1 lit. water is 1.16 Rs./lit. (0.017 USD) as compared to 0.17 Rs./ lit (0.0025 USD) for ceramic cartridge filter in the I<sup>st</sup> defluoridation cycle <sup>24</sup>. The cost of the defluoridated water by yarn cartridge filter is 10 times more expensive than ceramic cartridge filter. The initial capital cost of the fabricated filter is however same for both filters. The filter needs to be refilled 37.5 times before I<sup>st</sup> regeneration as compared to 500 times for the same filter with ceramic cartridge <sup>24</sup>.



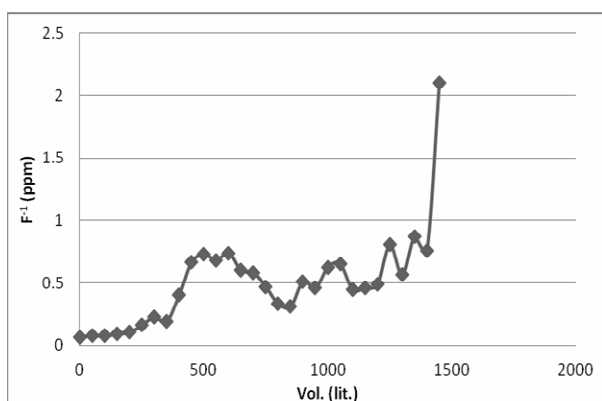
**Fig. 5** Variation of fluoride in the treated water from the filter (3kg AA, with Single Yarn Cartridge Filter (av. F in inlet 3.57 ppm for 3kg (1) and 4.21 ppm for 3 kg (2))

To increase for 3 kg (1) filter, the fluoride uptake capacity and volume of safe water produced we reduced the flow rate of this fabricated filter while using same amount of AA, 3 kg (2). The flow rate was reduced by approximately 10 times to 3.67 lit./hr. The bed volume of this filter remains same and the empty bed contact time was found to be 66.3 min. which is approximately 8 times higher from 3 kg(1) filter as mentioned above. Now the volume of safe water obtained from this filter was 400 liter (average fluoride inlet concentration is 4.21 ppm) an increase of only 100 lit. from 300 lit. in the 3 kg(1) filter. The volume of safe water is low due to higher concentration of fluoride in raw water. The variation of fluoride concentration in treated water is shown in fig 5. The fluoride uptake capacity was found to be 461.3 mg/kg AA, (approx. 1.5 times higher as compared to 3kg(1) filter). This is because of the volume of safe water in 3 kg (2) filter is more than that of 3 kg (1) filter.

The time before 1<sup>st</sup> regeneration is 20 days, an increase of only 5 days from 3 kg (1) filter (at a water consumption of 20lit./day). The recurring cost of the filtered water obtained from this filter with yarn cartridge was found to be the 0.87 Rs./lit. (0.0131USD). The 3kg (2) filter needs to be refilled 80 times before 1<sup>st</sup> regeneration, as compared to 37.5 times for 3kg (1) filter.

To increase flow rate and filter capacity, defluoridation filter using 50 lit. containers and double yarn cartridges with controlled flow rate of 7.8 lit./h were fabricated. This flow rate was approximately half in 10 kg (y) than that of 10 kg (c) (flow rate was 20.6 lit./hr) with same quantity of AA (10kg) <sup>26</sup>. Different water quality parameters were continuously monitored. The bed volume of this filter was found to be 13.5 lit. and it had an empty bed contact time of 103.84 min., it was approximately three times higher than 10 kg (c) with same quantity of AA (EBCT of 39.35min) <sup>26</sup>. The filter produces 1400 lit. of safe water with average fluoride inlet concentration 3.3 ppm.

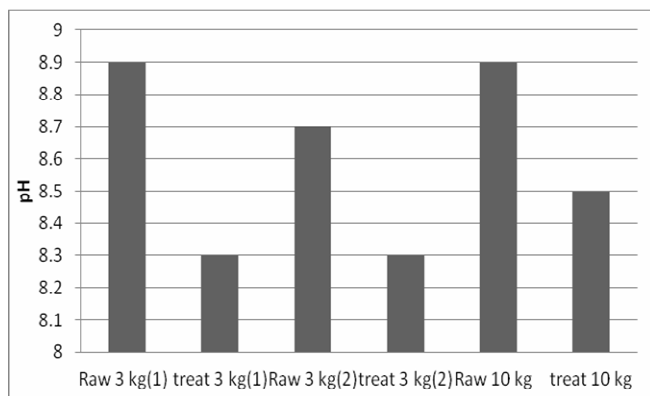
The volume of safe water is approx. 2.5 times lower than similar filter with 10 kg (c) (volume of safe water was 3300 lit.) <sup>26</sup>. The variation of fluoride concentration in treated water shown in fig 6. The fluoride uptake capacity was found to be 367.43 mg/kg which is approximately half compared to that of a similar filter with 10 kg (c) (FUC of 722.7 mg/kg) due to higher concentration of fluoride and alkalinity of raw water. The less volume of safe water produced from this filter requires that the filter need to be regenerated at a shorter time interval. The time before 1<sup>st</sup> regeneration is 2 month 10 days for 10 kg (y) and 5 month 15 days for 10 kg (c) (at a water consumption of 20 lit./day). The recurring cost of 1 lit. filtered water is 0.80 Rs./lit. (0.0121 USD) for 10 kg (y) and 0.35 Rs./lit. (0.0053USD) for 10 kg (c). The capital cost of the filter is Rs. 2690 (40.75 USD) for 10 kg (y) and Rs. 2775 (42.04 USD) for 10 kg (c) and cost of filter is nearly same for both 10 kg filters. The 10 kg (y) filter needs to be refilled 35 times before 1<sup>st</sup> regeneration as compared to 82.5 times for 10 kg (c).



**Fig. 6** Variation of fluoride in the treated water from the filter 10 kg AA with Double Yarn Cartridge (av. F in inlet 3.28 ppm)

### Monitoring of pH

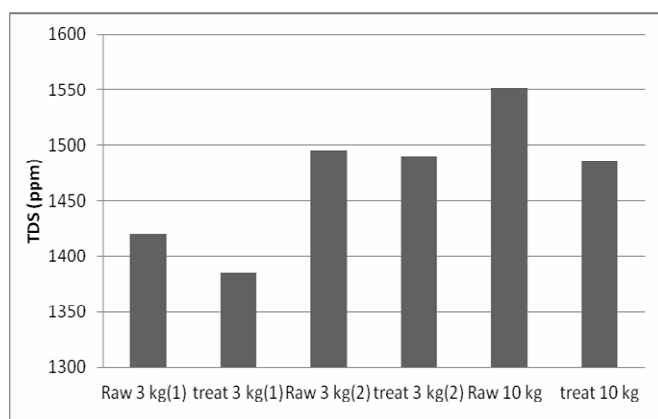
pH is used to express the intensity of acid or alkaline condition of water. Acidic water (low pH) can leach metals from plumbing systems, which can cause health problems. High alkalinity can cause aesthetic problems, such as an alkaline taste to the water that makes beverages taste bitter<sup>29</sup>. The maximum permissible range of pH according to BIS standard is 6.5 to 8.5<sup>30</sup>. Notice that pH of treated water is lower than raw water in all the filters and is within the permissible limit, fig 7.



**Fig. 7** Comparative average pH values of raw and treated water from filters with 3 kg and 10 kg AA

### Monitoring of TDS

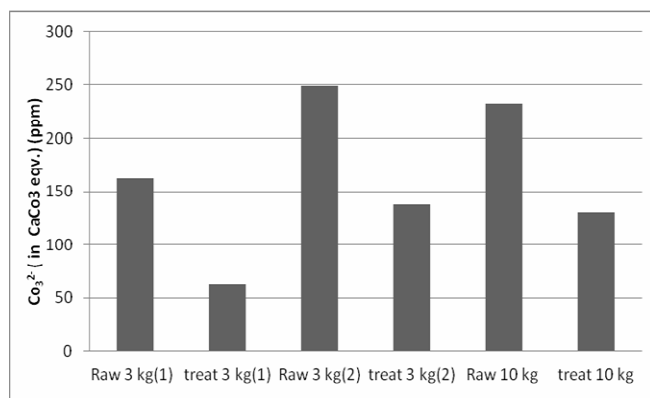
The TDS represents the various type of minerals present in water in the dissolved form. The maximum permissible limit of TDS according to BIS standard is 2000 ppm in absence of any alternative source of water<sup>30</sup>. Notice that TDS of treated water is lower than raw water by all the filters, fig 8.



**Fig. 8** Comparative average TDS values of raw and treated water from filters with 3 kg and 10 kg AA

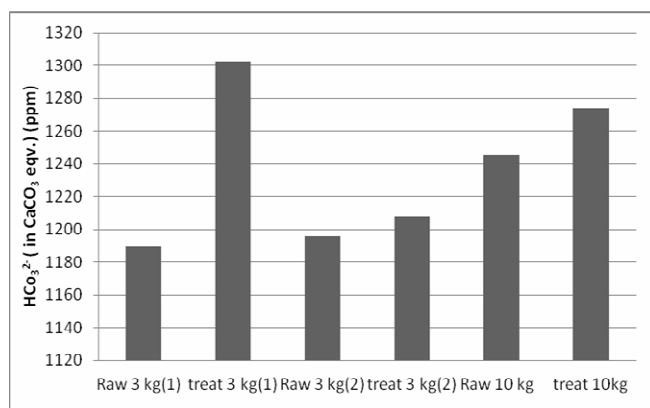
### Monitoring of $\text{CO}_3^{2-}$ , $\text{HCO}_3^-$ and Total alkalinity

Alkalis, when dissolved in water, create a bitter taste and a slippery feel. Highly alkaline waters, above pH 7.0, can cause drying of the skin<sup>31</sup>. The alkalinity of water is due to the presence of certain hydroxides, carbonates and bicarbonates in water. Notice that average value of carbonate decreases and bicarbonate increases, this is probably because of the low pH of treated water, fig 9 & 10. The maximum permissible limit for Alkalinity is 600ppm<sup>30</sup>.



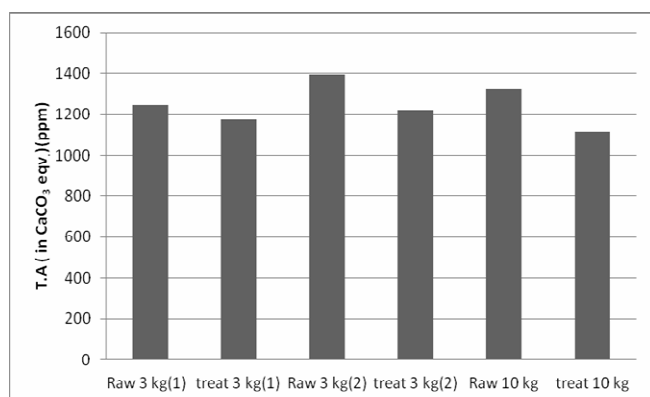
**Fig. 9** Comparative average carbonate values of raw and treated water from filters with 3 kg and 10 kg AA

It has been reported that if bicarbonate is present in fluoride contaminated water then fluoride adsorption by AA is lowered<sup>32</sup>.



**Fig. 10** Comparative average bicarbonate values of raw and treated water from filters with 3 kg and 10 kg AA

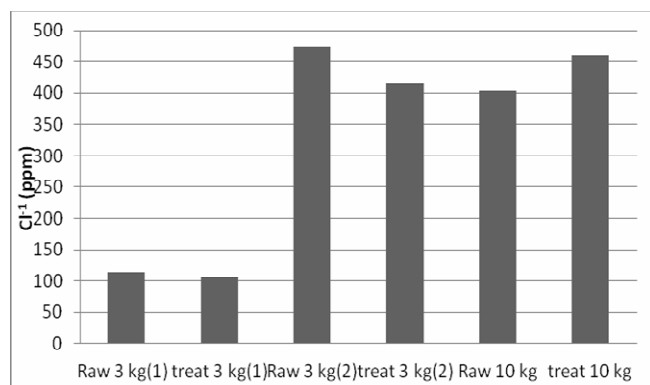
Notice that T.A is lower in all the filters, this may be due to part of the adsorption of  $\text{CO}_3^{2-}$  and  $\text{HCO}_3^-$  ion on the surface of AA, fig 11.



**Fig. 11** Comparative average Total Alkalinity values of raw and treated water from filters with 3 kg and 10 kg AA

### Monitoring of Chloride

Chloride is present in water in the form of compounds like  $\text{CaCl}_2$ ,  $\text{MgCl}_2$ ,  $\text{NaCl}$ . High chloride may cause bladder cancer and rectal cancer and deficiency of chloride may cause muscle weakness, loss of appetite, irritability, dehydration<sup>33</sup>. For chloride, the maximum permissible limit of chloride is 1000 ppm<sup>24</sup>. Chloride concentration of treated and raw water is not much different, fig 12. The fluoride adsorption is very little affected by chloride concentration<sup>34</sup>.

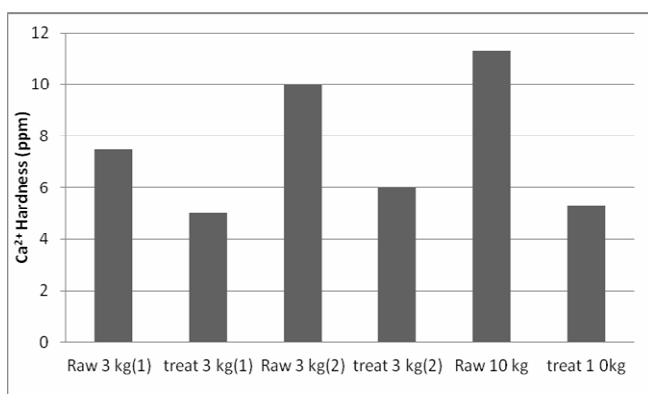


**Fig. 12** Comparative average chloride values of raw and treated water from filters with 3 kg and 10 kg AA

### Monitoring of Calcium Hardness, Magnesium Hardness and Total Hardness

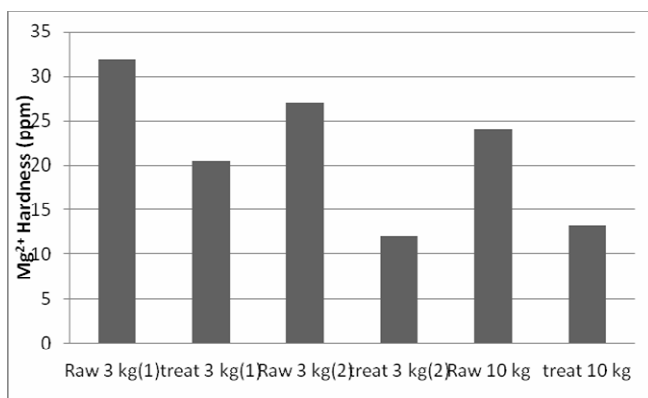
The deficiency of calcium causes osteoporosis, dry skin and brittle nails. High intake of calcium may also decrease the risk of heart disease. The maximum permissible limit of calcium is 200 ppm<sup>30</sup>. Notice that average value of calcium hardness, magnesium hardness and Total Hardness is decreases in treated water as compare to raw water, fig 13, 14 &15.





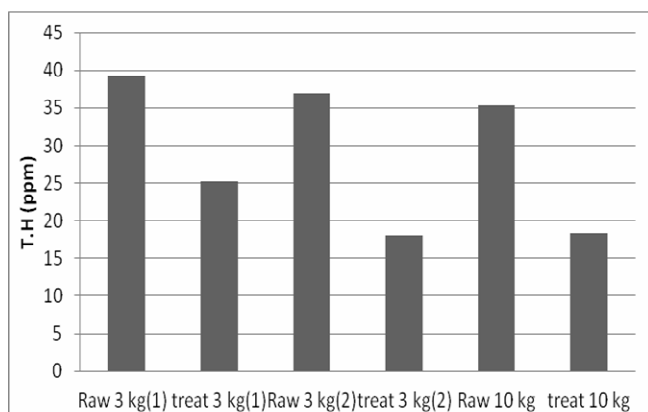
**Fig. 13** Comparative average calcium values of raw and treated water from filters with 3 kg and 10 kg AA

High doses of magnesium in medicine and food supplements may cause muscle slackening, nerve problems, depression and personality changes. Magnesium deficiency may cause vomiting, loss of appetite, nausea, personality changes. For magnesium, the maximum permissible limit is 100 ppm<sup>30</sup>.



**Fig. 14** Comparative average magnesium hardness values of raw and treated water from filters with 3 kg and 10 kg AA

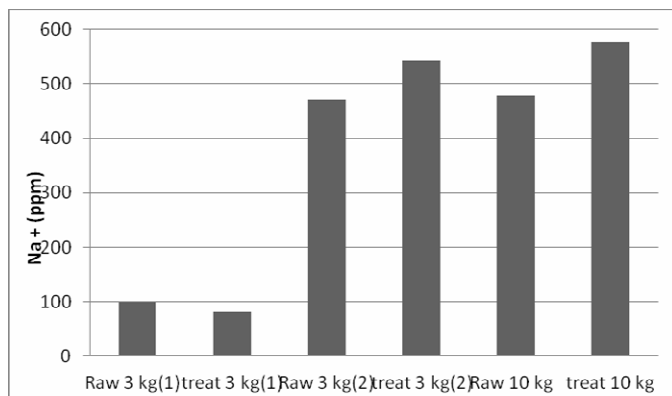
The maximum permissible limit of total hardness is 600 ppm<sup>30</sup>. Notice that in all the filters, the Total Hardness is decreasing in treated water, this is due to the lowering of both calcium hardness and magnesium hardness.



**Fig. 15** Comparative average Total Hardness values of raw and treated water from filters with 3 kg and 10 kg AA

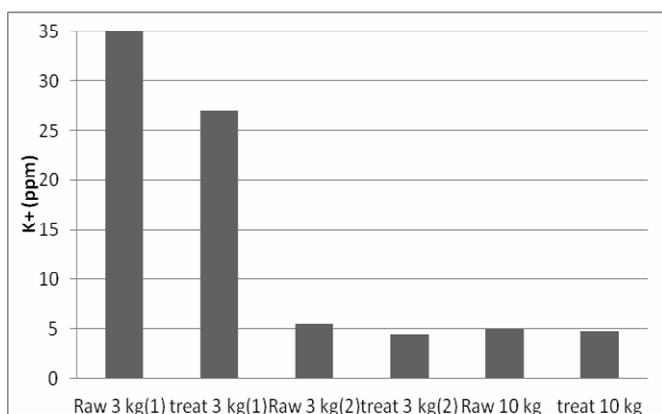
### Monitoring of Sodium and Potassium

Excessive sodium intake causes increased blood pressure, confusion and increased risk of infection. A low salt diet appears to increase the risk of death in those with heart failure<sup>35</sup>. There is no consistent trend in variation of sodium and potassium in the treated water for the various quantity of AA, fig 16 & 17.



**Fig. 16** Comparative average Sodium values of raw and treated water from filters with 3 kg and 10 kg AA

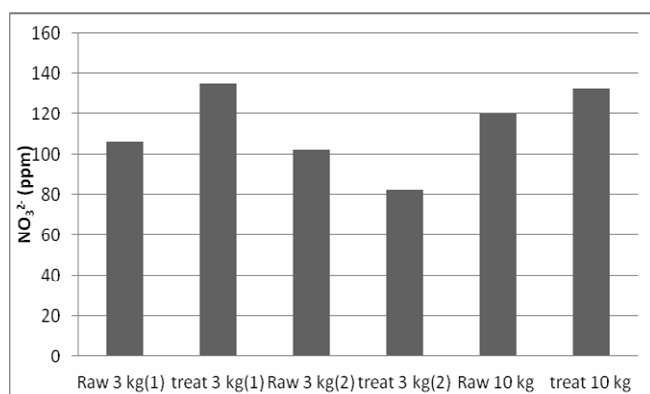
High potassium can reduce the risk of Hypertension (high blood pressure) and due to lack of potassium causes muscle weakness, cramps and paralysis.



**Fig. 17** Comparative average potassium values of raw and treated water from filters with 3 kg and 10 kg AA

### Monitoring of Nitrates

High concentration of nitrate in drinking water may causes shortness of breath and methemoglobinemia (blue baby syndrome) in infants<sup>36</sup>. The maximum permissible limit of nitrate in drinking water is 45 ppm<sup>30</sup>. There is no consistent trend in variation of nitrates in the treated water for the various quantity of AA, fig 18.

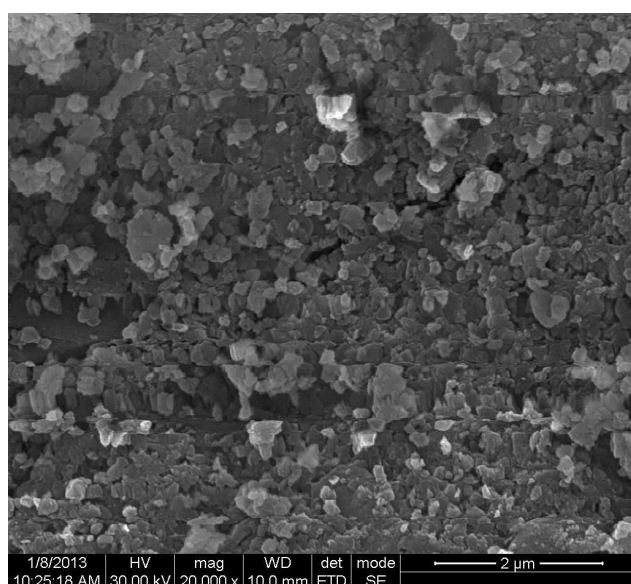


**Fig. 18** Comparative average nitrates values of raw and treated water from filters with 3 kg and 10 kg AA

### Characterization of Activated Alumina (DF-101)

The SEM images of fresh AA and exhausted AA at different resolutions have been carried out. At low resolutions of 200 x there is no perceptible difference in surface morphology. Difference start to emerge at 5000 x and become clear at 10,000 and 20,000 x. Notice that the pores on the fresh AA surface are filled up and the surface appears smooth in the case exhausted AA. The SEM analysis of a fresh and exhausted sample of AA is shown in fig. 19 and 20.

Microanalysis data of fresh AA show that, the percentage of Al and O are close to the calculated formula of Al<sub>2</sub>O<sub>3</sub>, for fresh (found 51.28 % for Al and 48.72 % for O) and (calculated 52.9 % for Al and 47.05 % for O). The exhausted AA (found 48.26 % for Al, 50.54 % for O and 1.20 % for Ca) show the presence of calcium. The exhausted AA show the presence of calcium probably from adsorbed calcium salts in water. The EDS x-ray analysis spectra of a fresh and exhausted sample of AA is shown in fig. 21 and 22. Aluminium tests in treated water indicates that the concentration of Al in water using a fresh AA is 0 ppm and after the AA was exhausted is 0.3 ppm. The permissible limit of Al in drinking water is 0.2 ppm.



**Fig. 19** SEM analysis of fresh activated alumina (DF-101)

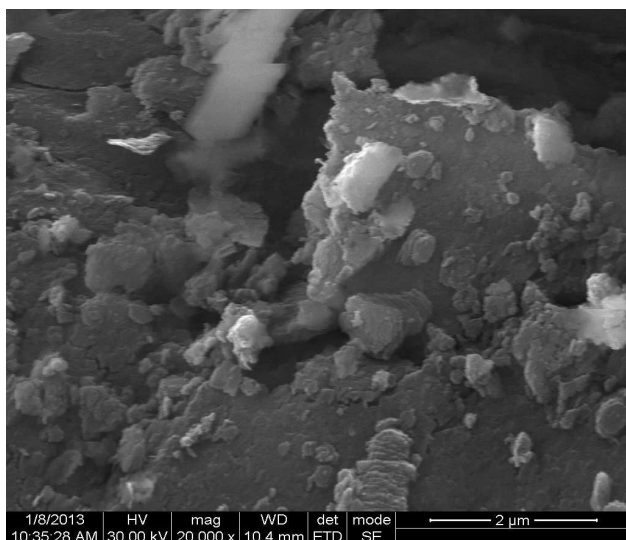


Fig. 20 SEM analysis of exhausted activated alumina (DF-101)

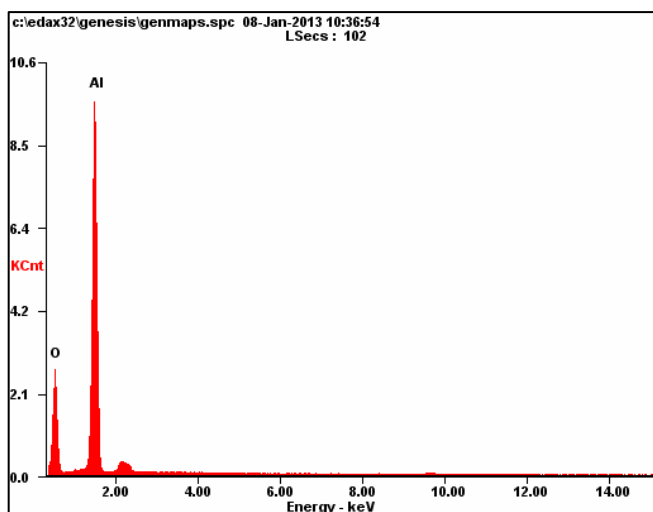


Fig. 21 EDS X-ray analysis of fresh activated alumina (DF-101)

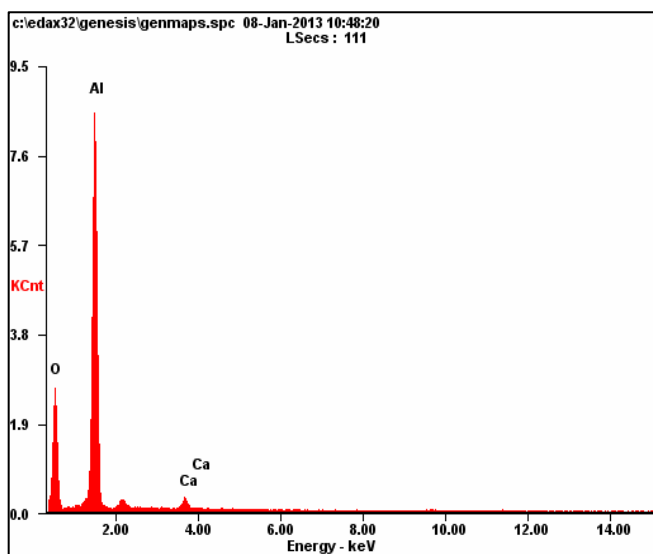


Fig. 22 EDS X-ray analysis of exhausted activated alumina (DF-101)

## Conclusion

From the above study of fabrication and testing of defluoridation filter using yarn cartridge and comparing them with similar filters with ceramic cartridge conducted earlier, we conclude, that in term of volume of safe water, fluoride uptake capacity and time before 1<sup>st</sup> regeneration, the defluoridation filter with ceramic cartridge is better. However, when flow rate and ease of use and breakability of the cartridges are considered, the defluoridation filters with yarn cartridges are at an advantage.

For intermediate level use between 3-4 families, the defluoridation filter with yarn cartridge is a good choice for both urban and rural household.

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