# Field testing of a magnesium oxide-lime-calcium chloride hydrochloric acid based filter

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**Abstract** Field trials of a MgO-Lime-CaCl<sub>2</sub>-HCl filter has been carried out to test the operation of the filter during actual user conditions. Water quality parameters like pH, TDS, Ca, Mg hardness, total alkalinity,  $SO_4^{-2}$ , Cl<sup>-</sup> were found to be within permissible limits in the water obtained from the filter except nitrate, whose concentration was above permissible limits in the raw ground water itself. The dosage of HCl had to be adjusted and fixed in the field trials. In comparison to the laboratory trials conducted earlier it was found that during the field trials there was on an average 6% reduction in the fluoride removal efficiency of the filter. Operational user problems of filter were found and were addressed in the village.

Keywords: magnesium oxide, water quality parameters, removal fluoride efficiency

#### **1. Introduction**

Fluoride is both beneficial and harmful to human health [1, 2, 3]. Its beneficial effects include its role in the formation of enamel etc. when present within desirable limits, however when present in excess it cause fluorosis a musculoskeletal disease characterized by weakening of bones, teeth inability to do physical labour etc [1, 4].

The World Health Organization sets the desirable limits of fluoride as 0.5 to 1.00 ppm and maximum permissible limit of 1.5ppm [5], while in India the Bureau of Indian standards (BIS) sets the desirable concentration at 1ppm and maximum permissible limits 1.5 ppm [6]. According to Indian Standard Organization (ISO) the desirable range is 0.6 to 1.2 ppm and maximum permissible limits is 1.5 ppm [7]. Ground water in many place in the world contain fluoride above permissible limits [8]. In India the groundwater in the state of Rajasthan

has higher fluoride concentration in 32 districts of its 33 districts [9, 10, 11].

Reverse Osmosis, Ion exchange, Activated alumina, Nalgonda Technique, Bone char, Clay bricks are known to remove fluoride from water and are used for defluoridation purposes [2, 12]. In view of the higher fluoride concentration in groundwater in Lakshmangarh Tehsil of Sikar district of Rajasthan, the Department of Science and Technology, Govt. of India has sponsored the laboratory and field trials of a magnesium oxide based defluoridation filter that has been recently developed at IISc Bangalore (India) [13, 14, 15]. The process involves adding calculated quantities of MgO, lime and aqueous solution of CaCl<sub>2</sub> to the fluoride contaminated ground water. The water is left overnight and the filtered to another container and NaHSO<sub>4</sub> is added to adjust the pH to within potable limits of 6.5 to 8.5 [13, 14, 15].

In view of the higher total alkalinity of local ground water, a suitable ratio of MgO, Lime and CaCl<sub>2</sub> solution has been arrived at in the laboratory. NaHSO<sub>4</sub> was replaced by dil HCl due to excess of  $SO_4^{-2}$  in the pH adjusted water. Laboratory trials of this technique has been conducted and has been recently reported [16]. To evaluate the performance of the filter under actual user conditions, we conducted field trials in the village Pilaniyon ki Dhani in Shola village of Lakshmangarh. In this paper we report the preliminary result of field trials and compare it with laboratory trials conducted earlier [16].

#### 2. Experimental

The fluoride filter was fabricated using the technique describes earlier [17]. The filter was installed in the covered verandah of a house in Pilaniyon ki Dhani (Village Shola).

The household was provided with seven bottles 7.5% CaCl<sub>2</sub> in dimineralised water, 0.38 N HCl in demineralised water and seven packets of 50,000.0 mg MgO + 37,500.0 mg lime mixture. The household was told to keep the chemicals out of reach of children in safe manner. The operation of the filters was first demonstrated and thereafter the head of the household was trained to operate the filter. It was observed that it took 2-3 days for consuming 50 liter water by a family of five adults and three children. The performance of the filter was monitored in the field for two weeks. The raw water and pH adjusted fluoride free water was brought from the village in a clean stoppered food grade PET container and analysed in the laboratory.

Fluoride was measured using an Ion Selective Electrode (Orion-Thermo Scientific, USA) using TISAB III as buffer. Flouride standards of 1 and 10 ppm were prepared from 100 ppm F<sup>-</sup> standard (Orion-Thermo Scientific, USA) for caliberation. pH, was measured using Hanna pH meter (USA) and calibrated using a buffer capsules of pH 7 (CDH India Ltd.) in demineralised water. TDS was measured using Hanna electronic TDS meter and calibrated using 1000 ppm KCl solution in dimenineralised water. Calcium hardness was determined by EDTA (0.02 N) using Patton and Reeders reagent (AR, CDH India Ltd.). Magnesium was determined using EDTA (0.02 N) using EBT Indicator (AR, CDH India Ltd.). Alkalinity was determined by titrating with dil  $H_2SO_4$  (0.02N) using phenolphalien indicator. Chloride was determined by titrating with AgNO<sub>3</sub> (0.0268 N) solution prepared using AR grade AgNO<sub>3</sub> (CDH India Ltd.). Sulphate was determined by turbidity method (BaCl<sub>2</sub> .2H<sub>2</sub>O Qualigens India Ltd.) using a digital photocolorimeter (Instrument India Ltd.) by measuring absorbance at 220 nm and 270 nm. K<sup>+</sup> and Na<sup>+</sup> were determined by a flame photometer (ESICO, India), caliberated using 50 ppm Na<sup>+</sup> and 20 ppm K<sup>+</sup> solutions. Nitrate was analyzed using a Shimadzu 1800 UV-VIS Spectrophotometer by measuring the absorbance at wavelengths at 220nm and 270nm (1 N HCl is added to prevent interference due to OH<sup>-1</sup> or  $CO_3^{-2}$ ).

#### 3. Results and Discussions

Before field work was performed six trials laboratory experiments were carried out using water brought from the village. The raw water and MgO-CaO-CaCl<sub>2</sub> treated water was analysed for various water quality parameters. If was found that in the final pH adjusted water the measured quality parameters (the pH, TDS, Ca, Mg, hardness, CO<sub>3</sub><sup>2-</sup>,  $HCO_3^{-}$ , Total alkalinity,  $SO_4^{2-}$ ,  $Cl^{-}$ ) were within the permissible except nitrate. limits whose concentration in the raw water itself was high. After the laboratory trials of the filter were successful, the filter was put to field testing.

During laboratory trials it was found that 600 to 620 ml of 0.38 N HCl was sufficient to lower the pH of 45 liters MgO-CaO–CaCl<sub>2</sub> treated water to 8.1 in six trials. Under field conditions it was found in 7 trials that 660ml of 0.38 N HCl was required for to bring down the pH to an average value of 7.9 (**Fig. 1.**)

Notice that in field trials and lab trials the reduction of  $F^-$  concentration occurred by 65% and 59% respectively (**Fig. 2**.).



**Fig. 1**. Comparative pH values of raw and pH adjusted water during Laboratory and Field Trials in Vill. Pilaniyon ki Dhani.



**Fig. 2.** Comparative F<sup>-</sup> values of raw and pH adjusted water during Laboratory and Field Trials in Vill. Pilaniyon ki Dhani.

Fluoride removal in this process is a combined precipitation of  $Mg(OH)_{2-x}F_x$  [18] [19] [20] and  $CaF_2$  [21]. There is considerable reduction of  $HCO_3^-$  ion concentration (**Fig. 3**).



**Fig. 3.** Comparative HCO<sub>3</sub><sup>-</sup> values of raw and pH adjusted water during Laboratory and Field Trials in Vill. Pilaniyon ki Dhani.

In the treated water as compared to the raw water in field trials and lab trials (by 627 ppm and 668 ppm respectively) due to the formation of insoluble  $CaCO_3$ : Eq. (1).

$$HCO_3^- + Ca^{+2} + OH^- \rightarrow CaCO_3 \downarrow + H_2O \qquad (1)$$

Notice that the carbonate ion concentration in treated water reduces as compared to the raw water, (both by 41 ppm and 25 ppm) in field and lab trials **Fig. 4** as some of the carbonate possibly gets precipitated as CaCO<sub>3</sub> due to the presence of added calcium in water: Eq. (2).

$$Ca^{+2} + CO_3^{-2} \to CaCO_3 \downarrow$$
 (2)

Also, there is a carbonate bicarbonate equilibrium occurring in the water due to addition of basic lime in the first step and hydrochloric acid in the second step. For the above mentioned reasons the Total Alkalinity decreases by 57 ppm and 62 ppm in treated water as compared to raw water both for field and lab trials (**Fig. 5**.).



**Fig. 4**. Comparative  $CO_3^{2-}$  values of raw and pH adjusted water during Laboratory and Field Trials in Vill. Pilaniyon ki Dhani.



**Fig. 5.** Comparative  $TA(CaCO_3 eqv)$  values of raw and pH adjusted water during Laboratory and Field Trials in Vill. Pilaniyon ki Dhani.

The added calcium in form of calcium chloride and lime gets precipitated in the form of  $CaCO_3$ (discussed above) and hence there is reduction  $Ca^{+2}$ ion concentration by 66 ppm and 64 ppm in field and lab trials respectively (**Fig. 6**.) The Mg<sup>+2</sup> concentration in raw water and treated remains nearly constant (increases very slightly by 1 ppm, (**Fig. 7**) due to very low solubility of Mg(OH)<sub>2</sub> in water ( $K_{sp}$ =5.0 × 10<sup>-12</sup>) [22].

On addition of MgO, CaO and 7.5% CaCl<sub>2</sub> solution the pH of water rises to 10.0-11.3. During this stage the precipitation of  $Mg(OH)_2$  and  $CaCO_3$ occurs as the solubility of Mg(OH)<sub>2</sub> [23] and CaCO<sub>3</sub> is very low in pH greater than 10 [24]. In this step the fluoride removal also occurs as fluoride ions gets trapped in the interstitial spaces of Mg(OH)<sub>2</sub> and also HCO<sub>3</sub><sup>-1</sup> gets converted to CaCO<sub>3</sub> and gets removed from the solution[15]. The precipated salts are removed by filtration, however, at this pH the water is not potable (drinkable). The drinkable range of water is between 6.5 to 8.5 as per WHO [5] and ISO standards [6]. Therefore dil. HCl is being added to adjust the pH to within potable limits. Also, at this pH range, the solubility of Mg(OH)<sub>2</sub> or CaCO<sub>3</sub> is sufficient to prevent their precipitation [23,24].

In line with above the Total Hardness decreases by 18 ppm in treated water as compared to raw water both for field and lab trials (**Fig. 8**).



**Fig. 6**. Comparative Ca<sup>+2</sup> values of raw and pH adjusted water during Laboratory and Field Trials in Vill. Pilaniyon ki Dhani.



**Fig. 7.** Comparative Mg<sup>+2</sup> values of raw and pH adjusted water during Laboratory and Field Trials in Vill. Pilaniyon ki Dhani.



**Fig. 8**. Comparative Total Hardness values of raw and pH adjusted water during Laboratory and Field in Vill. Pilaniyon ki Dhani.

Due to pH adjustment by HCl the chloride concentration increases considerably by 99 ppm in field trials as compared 123 ppm in lab trial (**Fig. 9.**)



**Fig. 9.** Comparative Cl<sup>-</sup> values of raw and pH adjusted water during Laboratory and Field Trials in Vill. Pilaniyon ki Dhani.

The sulphate concentration slightly increases by 10 ppm in treated water as compared to raw water (**Fig. 10**).



**Fig. 10.** Comparative  $SO_4^{2-}$  values of raw and pH adjusted water during Laboratory and Field Trials in Vill. Pilaniyon ki Dhani.

This may be due to the introduction of sulphate in form of impurities in lime (commercial grade) and technical grade of MgO used. The nitrate concentration was more than the permissible limit of



**Fig. 11**. Comparative  $NO_3^-$  values of raw and pH adjusted water during Laboratory and Field Trials in Vill. Pilaniyon ki Dhani.

The TDS values of the treated water increased marginally by 127 and 131 ppm in field and lab trials respectively **Fig. 12**.



**Fig. 12**. Comparative TDS values of raw and pH adjusted water during Laboratory and Field Trials in Vill. Pilaniyon ki Dhani.

 $Na^+$  and  $K^+$  ion concentration did not change significantly and were in the range of 400-500 ppm and 13-14 ppm respectively.

When filter with single tap was put in field trial the cloth got choked repeatedly. Therefore two taps were fitted with six layers of cloth. No choking of the cloth was subsequently observed. For convenience, a line was drawn outside the drum using a waterproof permanent marker to indicate 50 liter volume of water for easy filling of water. Every time after the upper container was emptied, the settled sludge and water at the bottom of the container (approximately 5L) was removed, and stored in a 60 liter plastic container and covered. When the plastic sludge container became full the supernatant water was decanted and disposed off in a common drain outside the house. The fluoride concentration in the supernatant water was found to be 1.49 and 1.51 ppm in two sludge buckets that were emptied during the trials. The sludge was taken back to the laboratory for safe disposal in form of cement stabilized sludge mud blocks to be made later.

There was a considerable effort in filling the water in the filter as the house had only one common tap at a very low level in the courtyard and there was no overhead tank in the house. Filling the filter with water using a pipe was not possible as the water supply was at very low pressure. A bucket with a 10 liter mark outside was provided to fill the water into the drum from the common tap. To reduce the effort in filling the water in the filter, the filter was relocated from the open verandah of the house to a thatched shed near the water tap.

It was observed that when the villagers operated the filter, the six layer cloth tied at the mouth of the tap fell off several times due to the pressure of water in the upper container. Therefore it was important to tie the cloth securely using a strong rope or twine to the tap. Since the location of the village is remote, it was necessary to keep spare plastic pipes, taps and Teflon tape for securing the threads of the plastic pipes in case of leakage and breakage. Only a slight change of taste of water from the filter as compared to raw water was observed.

An important advantage of this filter process as compared to activated alumina filter is that the

fluoride removal efficiency remains almost constant when used repeatedly as fresh dosage of adsorbents and chemicals are added, whereas in activated alumina based filters the fluoride removal capability of the filter gradually decreases. Though no bacteriological studies were carried out, chances of bacterial contamination is low as each day the filter has to be washed and refilled afresh with water and chemicals. The filter media also does not need any periodic regeneration moreover the process does not need any skilled personnel for operation.

## 4. Conclusions

To conclude we have carried out the user field trials of the MgO-Lime-CaCl<sub>2</sub>-HCl base filter and found that HCl dosage had to readjusted in field trails. Also there is a slight reduction (6%) in the efficiency of the filter. Otherwise the filter performance almost similarly to the trails conducted in the laboratory earlier. Practical issues of filling the water in the filter, crucial aspect of improper tying of six layers cloth, disposal of sludge and water mixture from the filter, acceptability of the filter in term of tastes of water and user friendliness were understood and out resolved within the limitations of a single user trials described above.

### 5. Acknowledgments

Financial help from (Water Technology Initiative) DST, Govt. of India is acknowledged in form of project No. (DST/DT/WTI/2k8/95). The authors thank the MITS for providing facilities and support. Prof. S. Sudhakar Rao, IISc, Bangalore is thanked for advice and support.

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Submitted: February 25<sup>th</sup> 2013 Accepted in revised form:May 27<sup>th</sup> 2013