Guidelines

for

REGENERATION OF

EXHAUSTED ACTIVATED ALUMINA

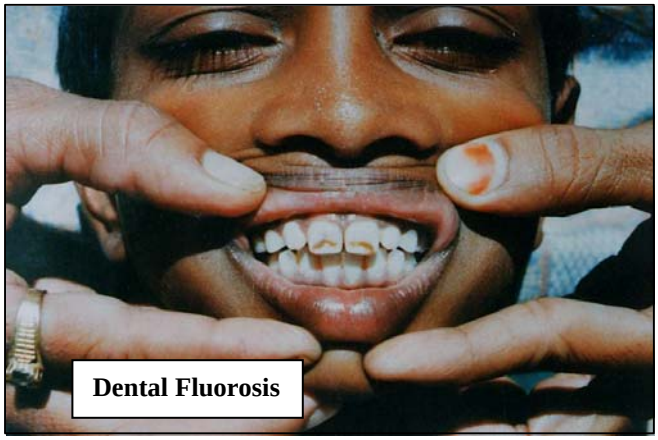
Table of Contents

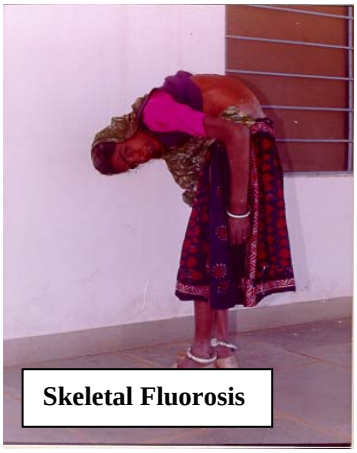
1.	Introduction	2
2.	Frequently asked questions	2
3.	Regeneration procedure	4
4	Safety precautions	7
5	How to dispose off fluoride rich effluent?	9
6	Requirement of equipment and consumables	10
7	The regeneration center	11
8	Details of settling tank	13

Introduction

Twenty states in India have reported high fluoride in groundwater - used extensively for drinking and cooking in rural areas. It is estimated that nearly 60 million people in twenty states are at health risk due to ingestion of high fluoride water

Long term ingestion of high fluoride water can cause several health problems. These include dental fluorosis, skeletal fluorosis and non-skeletal manifestations. It affects





young and old alike. It can also damage foetus if the mother, when she is pregnant, consumes water/food/other items with high fluoride concentration. Fluoride is known to induce aging. The malnourished children, pregnant women and lactating mothers are particularly at high risk.

The social and economic costs of this disease are enormous. It is therefore necessary to provide safe water to people as soon as possible. Considering the resource constraints, it is not possible to bring safe piped water to the doorstep of every household in the near future. In some cases implementation of such water supply schemes may take several years. In the meantime, the affected households can be provided domestic defluoridation units (DDU) as an emergency and interim measure.

As part of the DDU maintenance, the activated alumina needs to be regenerated periodically. This manual on regeneration of exhausted AA is prepared to provide information on the regeneration process, precautions to be observed, equipment and consumables required and lay out of the regeneration center. The target audience includes engineers, field workers and particularly the entrepreneur providing regeneration services at the village level.

Frequently asked questions

What is activated alumina?

AA, which is mainly Al_2O_3, is prepared by dehydration of Aluminium hydroxide in temperature range of 300 - 600^oC. It is white in colour and granular/spherical in shape. It

is inert in nature and safe to handle. It is used extensively for conditioning air i.e. removal of moisture, oil and other impurities from air in certain industries. It is also used for removal of fluoride and arsenic from drinking water.

What is its use in defluoridation of drinking water?

Since 1930's, many reports have been published on the use of Activated Alumina for defluoridation. Alcoa F-1 grade of AA (manufactured by Alcoa Company, USA) has been extensively used large-scale water treatment plants. Many investigators in India have carried out studies on the use of indigenously produced AA for defluoridation of water. The Indian Institute of Technology, Kanpur has done extensive R&D work on the use of AA for defluoridation at the household level under an UNICEF sponsored R&D project.

What is regeneration of AA?

Regeneration is a chemical procedure used for cleaning up the used activated alumina, so that it can be reused. Caustic soda and sulphuric acid are used for regeneration.

Why to regenerate AA?

Fluoride uptake capacity of AA depends on the physical and chemical properties of alumina as well as water to be treated. After certain length of use, AA gets saturated with fluoride ions and loses its capacity to remove fluoride ions from water. This stage of AA is known as "Exhausted AA". Users have two options. Replace the exhausted AA with the new AA Or regenerate the exhausted AA. The regeneration option is cost effective as it can be done at $1/15^{th}$ the cost of replacing the exhausted AA with the new AA.

When to regenerate AA?

AA should be regenerated when it looses its capacity to remove fluoride ions from drinking water. AA is considered "exhausted" when fluoride in treated water from a DDU exceeds 1.5mg/L - recommended guideline value for fluoride by the World Health Organization.

How to check if fluoride in treated water exceed 1.5 mg/L?

Fluoride test kits of YES/NO type can be used to know if treated water contains fluoride below or above the permissible limit of 1.5 mg/L.

How to regenerate exhausted AA?

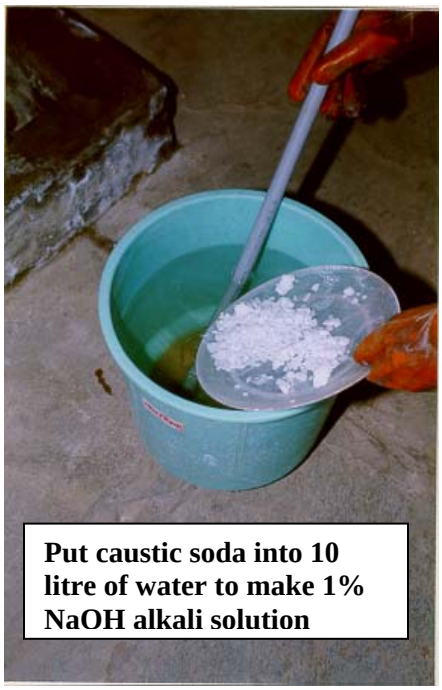
Exhausted AA is treated with caustic soda solution followed by rinsing in clean water, treating it with dilute acid and lastly, rinsing it in clean water till the rinse water pH is between 6.and 7. This procedure results in regeneration of the exhausted AA.

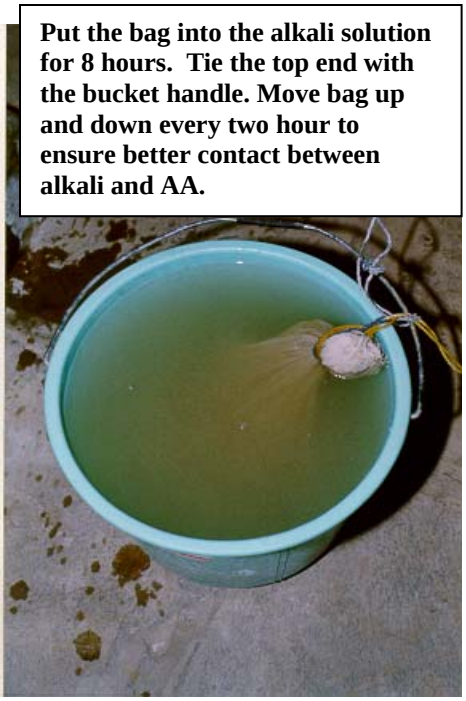
Regeneration Procedure:

1. Transfer the full quantity of exhausted AA from the domestic filter into the nylon bag (mesh size 0.106 mm). Make sure that there is no spilling during the transfer of AA.

2. Using rubber gloves, weigh 100gms of caustic soda flakes in a plastic plate or vessel.

3. Add cautic soda slowly into 10 litre of water. Stir with a plastic rod to dissolve the flakes. The alkali solution thus made is 1% NaOH.
4. Tie the neck of the nylon bag containing exhausted AA. Place the nylon bag in the





bucket containing diluted caustic soda (NaOH) solution. **Move the bag up and down inside the bucket every two hours to ensure good contact between the caustic soda solution and the exhausted AA.** After eight hours, lift the bag from the bucket and drain out excess alkali from the bag into the bucket. Note that caustic soda solution is used only once to treat nearly 5 Kg of AA.

5. Drain out the used caustic soda solution from the bucket into the settling tank.

6. Place the nylon bag in a plastic bucket containing 10 litre of clean water. This is the washing step. Move the nylon bag up and down a few times as described in step 4 above. Repeat the washing procedure at least 2 times, each time with fresh water. Raw water with fluoride can be used for this washing step.

7. Using gloves, transfer 100ml of concentrated sulphuric acid from the acid bottle to a measuring cylinder. Pour acid slowly along the side of the 15 litre capacity plastic bucket containing 10 litre of water. Stir water with plastic rod while adding acid. The acidic solution thus made is 0.4 N sulphuric acid (H_2SO_4). **Never add water to acid. It can cause a serious accident.**

8. After draining excess water, transfer the nylon bag containing activated alumina, to the bucket containing acid solution. Move the bag up and down as described in step 4 above to ensure good contact between AA in the nylon bag and the acid solution. After four hours, preferably overnight, lift the bag and drain out excess acid.

9. Drain out the used acid solution from the bucket into the settling tank.

Transfer 100 ML of acid from bottle to a measuring cylinder carefully. Use rubber hand gloves.

Pour acid slowly along the glass or PVC rod. Never put acid directly into water. It can result in accident. This acid solution is of 0.4 N strength.

10. Place the nylon bag with AA in a plastic bucket containing 10 L clean water. This is a washing step. Repeat the procedure as outlined in step 6 two or three times till the pH of water draining out of AA bag is between 6-7. Use the broad range pH strip to check pH. *This is a very important step. If pH is less than 6, the pH of treated water will be in unacceptable range in the first few cycles of defluoridation.*

11. When pH of drain water is between 6-7, lift the nylon bag and drain water into the bucket. Allow the regenerated AA to dry inside the bag in the regeneration center.

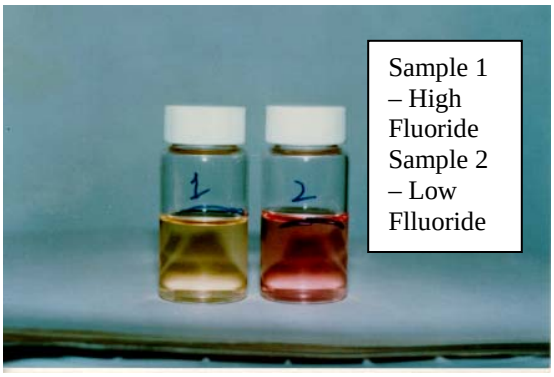
Check wash wat er pH. It must be between 6 and 7. Use a broad range pH strip.

12. AA is now ready for use.

Interventions at the Household Level

13. Transfer the clean activated alumina from the nylon bag back to the domestic defluoridation unit. Take care not to lose activated alumina while transferring.

14. Domestic filter is now ready for use. Test the treated water for fluoride to make sure that fluoride level is well below 1.5 mg/L.



For ease of handling, an arrangement of rod and pulleys as shown below may be considered wherever feasible.

Safety precautions to be taken during regeneration

While handling caustic soda and acid, certain precautions need to be strictly observed. If you do not take the following precautions, it can cause serious injury to you and others. **Precautions listed below must be strictly observed to avoid injury.**

General Precautions

1. Always wear rubber hand gloves and goggles while handling acid or alkali.
2. Keep chemicals away from children in a secure place preferably in a locked almirah.

Precautions while handling acid

Extreme care has to be taken while handling acid. Concentrated sulphuric acid causes burns on the skin and has a destructive action on all tissues. Sulphuric acid burns are deep and produce scars. If eyes are exposed to acid, it can lead to blindness. Fumes and mists can produce severe irritation of the respiratory tract, which includes nose and throat.

1. **Always use protective rubber gloves and goggles, and if possible wear a rubber apron while handling the acid.**
2. Take off the wristwatch, ring, bangles etc. before handling acids. Folding of shirtsleeves for men and tying of chunni or sari for women is recommended. Use of rubber apron is always preferable.
3. If acid spills on any part of the body, flush it immediately with water. Apply some antiseptic cream. If acid is spilled on the eye, wash it profusely with water for at least 10-15 min. **Remember vision may be lost if not washed immediately**.
4. Concentrated sulphuric acid is a liquid and is normally supplied in glass bottles. **Acid bottles should be stored so that it is kept securely away from the reach of unauthorised persons, particularly children.**
5. Extreme care has to be taken while transporting the bottles. Do not hold the bottle from the neck, as the bottle can break leading to a serious accident. Hold the bottom of the bottle with both hands. Always use a bottle carrier such as a bucket for taking the bottle from the storing cabinet to the work place. Use small size bottles, which are easy to handle.
6. **Store sulphuric acid away from caustic soda.**
7. If acid is spilled on the floor, use lime stone or sodium bicarbonate to neutralise acid. This neutralisation reaction using carbonates release CO_2 gas. Lack of gas fizzing indicates that acid has been neutralised. Since heat is generated which can cause splattering of the spilled material, add the chemical slowly. After the reaction is completed, remove the solid mixture and put it in the sludge drying bed. **Use of caustic soda pellets is not recommended.**

Precautions while handling caustic soda

Caustic soda can cause burns and exposure to eyes is dangerous. Therefore it should be handled carefully.

1. Caustic soda absorbs water and loses its strength. Therefore, the caustic soda container must be kept tightly closed.
2. If caustic soda pellets fall on the body, wash the affected portion with water, after which 2% acetic acid (2 ml glacial acetic acid in 100ml water) can be used. Apply cream on the affected area. The caustic soda solution is extremely damaging to eyes. If exposure occurs, the eyes should be washed immediately with cold running water for 15 min.
3. **Use rubber gloves and goggles (as in the case of acids) while handling caustic soda or its solution.**

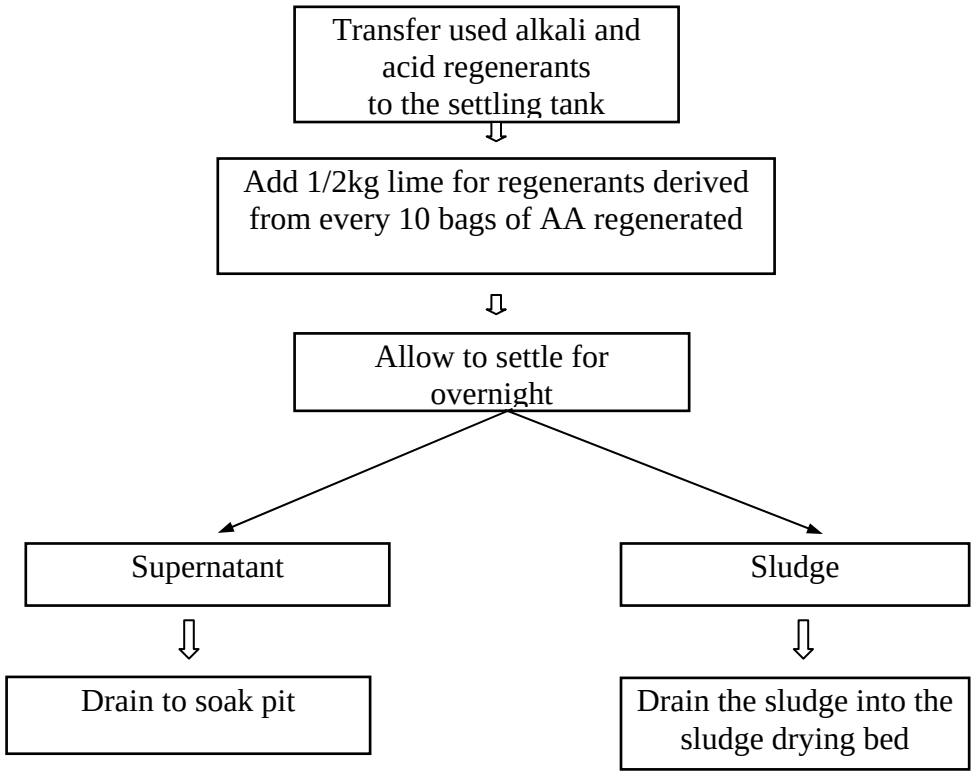
4. If caustic soda pellets are spilled on the floor, collect the pellets either using a broom or hands (after wearing gloves). Remove the collected pellets into a plastic bag. Wash the floor with water.

How to dispose off the effluent from a regeneration center?

Used alkali and acid will contain high fluoride and have extreme pH. It should be disposed off in an environmentally friendly manner. The following procedure is recommended for safe disposal of high fluoride effluent.

1. Drain out used alkali and acid from buckets into the settling tank.
2. Add lime (1/2 kg. per ten regenerations) in the settling tank to improve precipitation of the sludge. The supernatant water left after settling of the sludge has near neutral pH and low fluoride concentration.
3. Drain out the supernatant into a soak pit.
4. The settled sludge with high fluoride concentration is allowed to dry in the sludge drying beds.
5. Dispose off the dried sludge in a concrete lined pit Or Use the sludge in the manufacture of bricks Or Sludge can also be filled in plastic bags and buried in a place 100 meters away from water sources.

Flow Chart for Disposal of Effluent:



⇩

Transfer the dried sludge into a concrete- lined pit and finally get it mixed in bricks or dispose it off in a chemically safe environment

Requirement of Equipment and Consumables

Chemicals

1. Tezaab (Concentrated Sulphuric acid-Commercial grade)
2. Caustic soda (Sodium hydroxide-Commercial grade)
5. Lime
6. Weak base (Sodium bicarbonate or Baking soda)
7. Weak acid (Acetic acid)
8. Fluoride YES/NO test kit with reagent

Equipment

Weighing balance	5kg	1 number
	500gm	1 number

Other Items

1.	15L plastic buckets (with graduations)	12 Nos.
2.	200L plastic container (for storing raw water)	1 No.
3.	Nylon mesh bag (mesh size – 0.106 mm)	50 Nos.
4.	pH strip (wide range 2-10.5)	5 Nos.
5.	Plastic measuring cylinder	
	(i) 1000 ML	2 Nos.
	(ii) 100 ML	2 Nos.
6.	Rubber gloves	3 pairs
7.	Rubber apron	3 Nos.
8	Plastic rod (1m long, half inch diameter PVC pipe closed at both ends)	1 No.
9.	Goggles (for protection of eyes from acid and alkali)	2 pairs

Indicative Cost of Caustic Soda and Concentrated Sulphuric acid

Commercial Grade

(a) Caustic soda (50kg pack): Rs. 24-36 per kg; and (b) Sulphuric acid (loose): Rs. 3-6 kg

Laboratory Grade

(a) Caustic soda (5kg pack): Rs. 80-95 per kg; and (b) Sulphuric acid (2.5L): Rs 60-65 per kg

The Regeneration Center

Regeneration centre comprises of a room, a settling tank, a soak pit, two sludge drying beds and a concrete-lined sludge drying bed constructed specifically for regeneration of the exhausted AA. It should be near a water source so that water is easily available. The layout plan of a regeneration unit is given on the next page.

References

D	Door	-	120x210cm
W1	Window	-	100x150cm
W2	Window	-	150x150cm

1. Sludge settling tank - Masonry built 100x100x100 cm with a bed slope of 1 in 10. Interior of the tank should be lined with acid/alkali resistant bricks or tiles. Exterior surfaces plastered with cement mortar 1:3. Heavy duty PVC pipes and acid/alkali resistant valves should be used.
2. Sludge drying bed – Masonry built of size 120 x 120 x 30 cm for alternate use. It is constructed partially below and partially above the ground level. Plastering is done on the inner side and the exposed outer side with cement mortar 1:3.
3. Soak pit - Pit of size 60 x 60 x 75 cm is dug. It is filled with 4.75 cm gravel

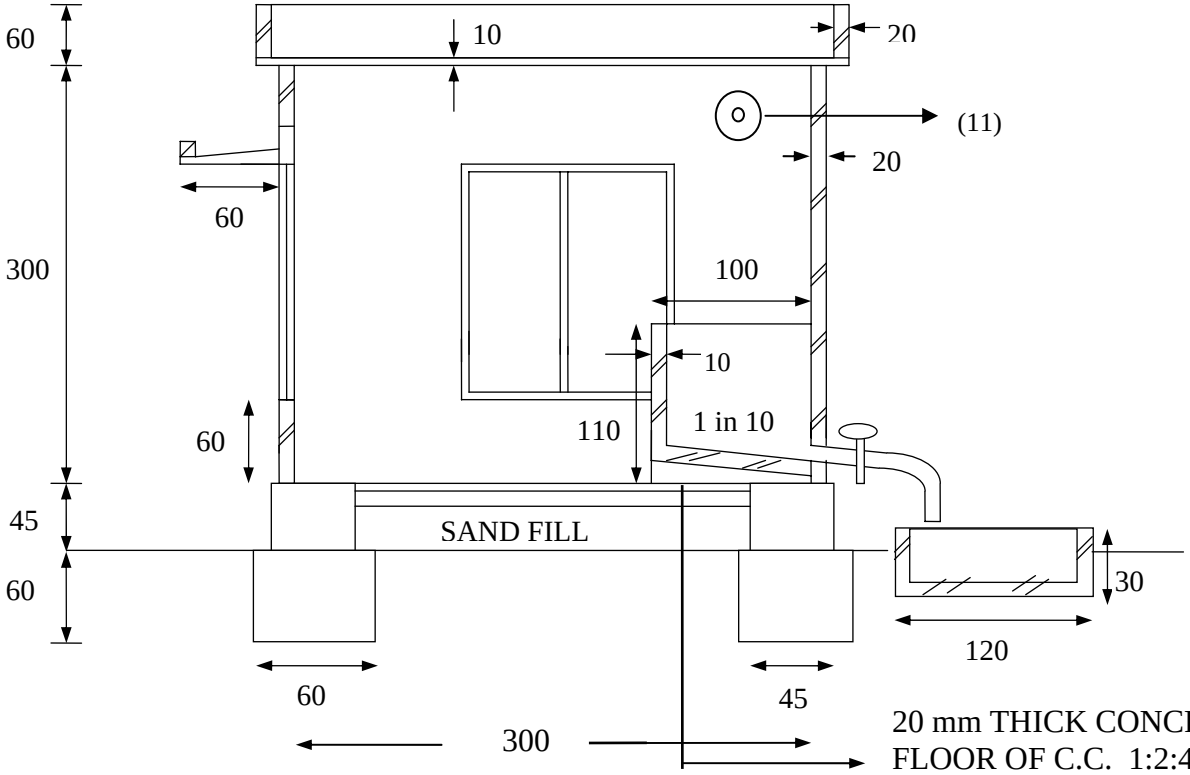
4&5 Corner with lockable shelf to store lime and caustic soda

6. Lockable shelf with wire mesh door for storing acid.
7. Shelf for keeping other equipment and consumables.
8. Masonry platform with sanitary tiles for placing fluoride meter and test reagents.
9. Masonry platform for placing balance
10. Water containers on elevated masonry platform
11. Exhaust fan

Masonry pit 75 x 75 x 75 cm (not shown in figure), plastered inside with cement mortar 1:3 is made to store dried sludge for later disposal.

The lay out in the following pages is indicative and can be modified depending on site conditions. The area for regeneration should be acid/alkali proof tiled floor with a 25 mm high parapet and with satisfactory arrangements to drain spilled water, alkali or acid into a soak pit.

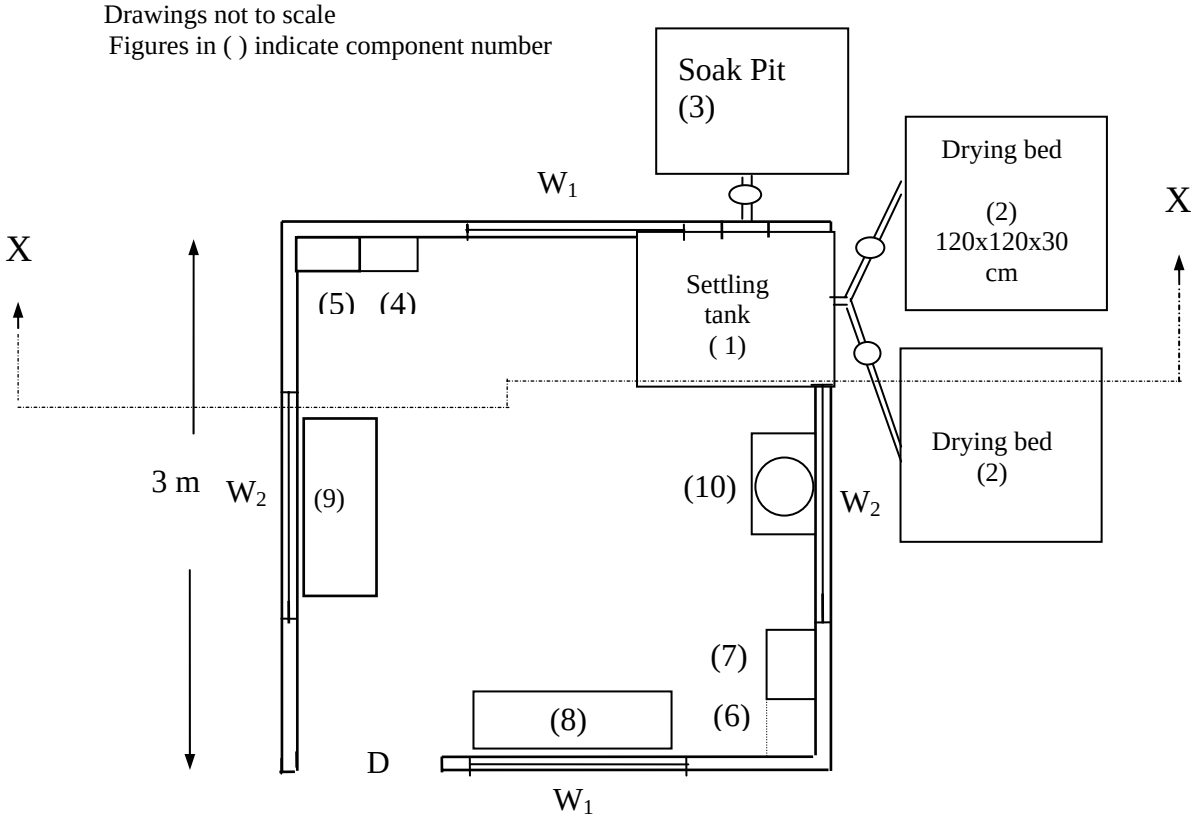
Suggested Layout Plan of Regeneration Centre



20 mm THICK CONCRETE FLOOR OF C.C. 1:2:4 OVER 10 cm C.C. 1:5:10

SECTION AT XX

Dimensions unless specified are in centimeter
Drawings not to scale
Figures in ( ) indicate component number



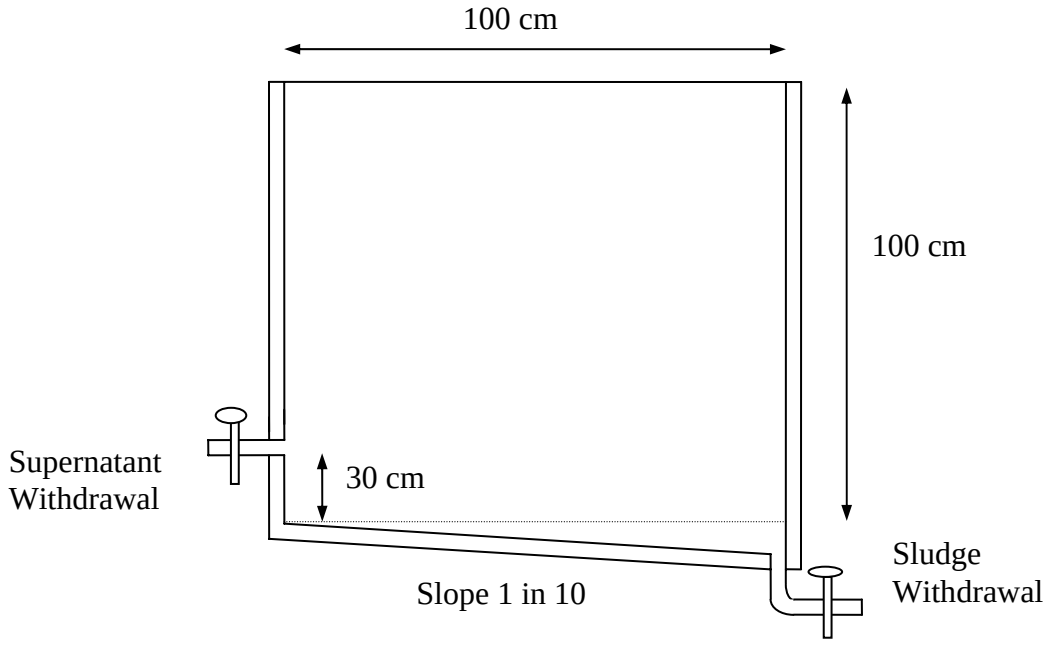
Details of the Settling Tank

Specifications for a settling tank to treat alkali and acid effluent (capacity 40 regeneration) are given below.

Dimension of the tank	1x1x1 meter
Bottom slope provided	1 in 10

The capacity of the tank can is sufficient for regeneration of 20 AA bags. Sludge as well as supernatant can be withdrawn once in two days.

Two withdrawal ports are provided on the adjacent sides as shown in the figure. Generally, tank is constructed at one corner of the regeneration center and the adjacent ports facilitate sludge and supernatant withdrawal directly outside the room.



Address of the chemical manufacturing Companies

Commercial grade alkali manufacturers

1. Thakkar Chemicals Pvt.Ltd
   307, Mandvi Navjivan,
   121/127, Kazi Sayed Street,
   Mumbai – 400003
2. Modi Alkalies & Chemicals Ltd
   18,Community centre,
   New Friends Colony,
   New Delhi – 110065
3. Gujarat Chemicals
   International Trade Tower,
   F -block,Second Floor, Nehru place,
   New Delhi – 110019
4. Kanodiya Fertilisers and Chemicals
   405/premier Chambers,
   R.C.Dutt Road, Vadodara –7

Addresses of laboratory grade acid and alkali

1. S.D.FINE - CHEM LTD
   Regd.Office:315– 317 T.V. Ind. Estate,
   248,Worli road Mumbai – 400025
2. Qualigens Fine Chemicals
   (A Division of Glaxo India Limited)
   Regd.Office: Dr. Annie Besant Road,
   Mumbai- 400025
3. Thomas Baker (Chemicals) Ltd
   Regd.Office: 4/86 Bharat Mahal
   Marine Drive Mumbai – 400002
4. E. Merck (India) Limited
   801,Swastik chambers,Swastik Mill compound
   Jn.of CST Road & Sion – trombay Road
   Chembur,Mumbai – 400071

Manufacturers of commercial grade H_2SO_4

Local manufacturers of acid may be contacted