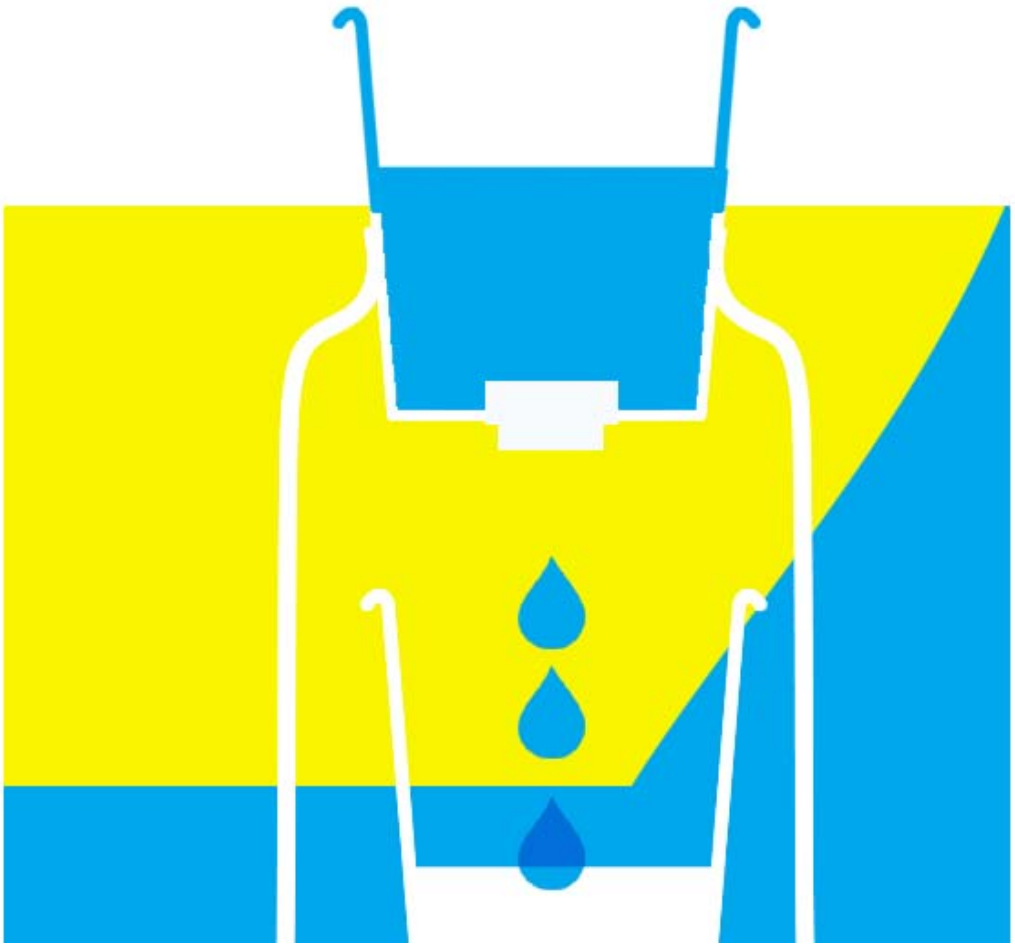
Regeneration Manual for Activated Alumina used in Domestic Defluridation Units



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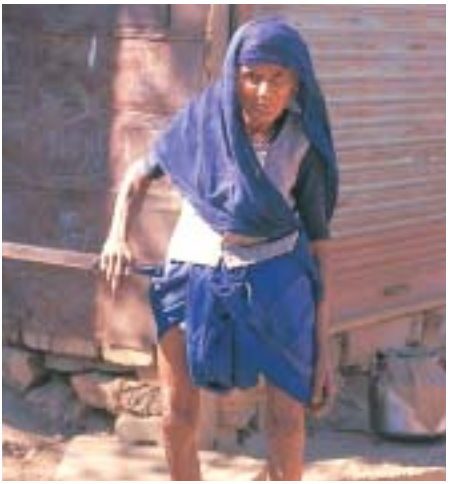
Introduction

Water and sanitation goals for India laid by the Government, highlights the need for access to safe drinking water. Around three decades back, India Mark II handpumps fitted on drilled wells were seen as sources of bacteriologically safe drinking water for rural populations. However, groundwaters in some areas have now shown the presence of certain inorganic chemical constituents that are hazardous to health and can lead to chronic health problems. Among these, fluoride and arsenic are of major concern in India. Nineteen states in India have reported high fluoride in groundwater.

Prolonged intake of excess fluoride leads to skeletal and dental fluorosis as well as non skeletal manifestations such as gastrointestinal problems.

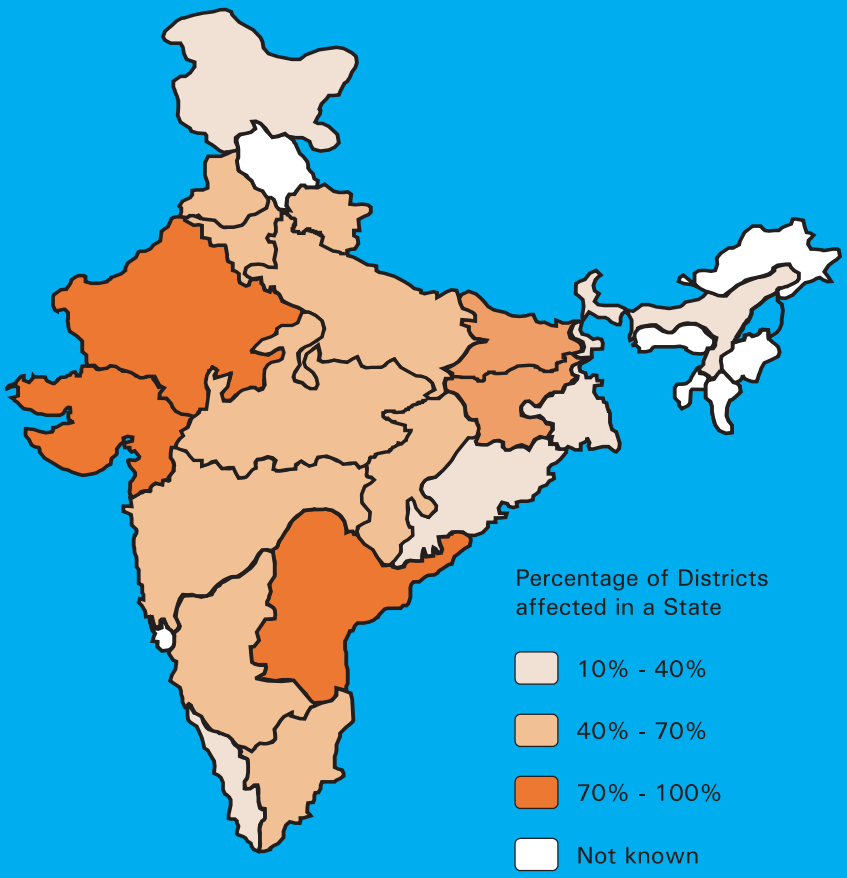
Dental Fluorosis



Skeletal Fluorosis

Fluoride affected States in India

19 States, 199 Districts affected,
66 million people at risk



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To overcome these health hazards, safe water with respect to fluoride, has to be provided to people as early as possible. Considering resource constraints, it may not be possible to bring safe piped water to every household in the near future and implementation of such water supply schemes may take several years. In the mean time, changes are necessary in nutritional habits of people and certain methods of removal of fluorides or "defluoridation" of water have to be adopted as an emergency and interim measure.

One of the effective methods of defluoridation is based on adsorption and uses Activated Alumina (AA) for the removal of fluoride from water. Households in affected areas can be provided with domestic defluoridation units (DDU) using activated alumina.

As raw water with high fluoride content passes through AA, its ability to remove fluoride from water gradually decreases. Hence, as a part of maintenance of DDUs, the AA in a DDU needs to be regenerated period to bring it back to its original fluoride removal capability.

This manual provides information on the regeneration procedure, disposal of spent regenerants, equipment and consumables required as well as the suggested layout for regeneration centre in a village.

The target audience for this manual includes engineers, field workers and other personnel providing regeneration services at the village level.

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Frequently Asked Questions

What is Activated Alumina?

Activated Alumina (AA), which is Al_2O_3. nH_2O, is prepared by the dehydration of aluminium hydroxide in the temperature range of 300ºC-600ºC. It is extensively used for the removal of fluoride and arsenic from drinking water. The fluoride uptake capacity of activated alumina depends upon the specific grade of AA and its particle size as well as the characteristics of the raw water being treated.

The Fluoride uptake by Activated Alumina

The main component of activated alumina is aluminium oxide. In an aqueous phase (upon hydration) hydroxyl groups develop at the surface of alumina. These groups behave amphoterically.

$$AlOH_2^+ \Leftrightarrow \quad AlOH + H^+ \quad \Leftrightarrow \quad AlO^- + H^+$$

Binding of fluoride to activated alumina is mainly ascribed to ligand exchange and can be represented as follows:

$$\overline{\text{Al - OH}} + F^- \Leftrightarrow \overline{\text{AlF}} + OH^- \text{ (pH >7)}$$

$$\overline{AlOH_2} + F^- \Leftrightarrow \overline{AlF} + H_2O \text{ (pH <6)}$$

At higher fluoride levels, loading (exceeding 15mg F/lit for 1 gm AA/ lit) polynuclear surface complex formation has been suggested

$$\overline{AlOH} + 2F^- \rightarrow \overline{AlF_2^-} + OH^-$$

Fluoride uptake can take place only when activated alumina surface is in the form of

$$\overline{Al\ (OH)^+_2} \text{ or } \overline{Al(OH)}$$

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Why Regenerate?

After a certain period of use, AA gets exhausted. This results in an increase in fluoride concentration of treated water till it goes beyond the permissible levels. At this stage users have two options, replace the exhausted AA with the fresh AA or regenerate the exhausted AA. Activated Alumina used for fluoride removal is a costly material. The economics of the application of AA for defluoridation of drinking water dictates that this material has to be reused for several defluoridation cycles. The regeneration option is cost effective as it can be done at 1/15th the cost of replacing the exhausted AA with the fresh AA.

What is Regeneration of AA?

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

Caustic soda strips fluoride from the surface of AA granules. After this treatment, activated alumina surface will be in the basic AlO^- form. To restore fluoride removal capacity, basic alumina will have to be treated with acid. Either sulphuric acid (H_2SO_4) or hydrochloric acid (HCl) can be used for the purpose. H_2SO_4 is generally used, as it works is cheaper than HCl.

Reactions during regeneration with caustic soda and sulphuric acid can be represented as given below:

$$\overline{AlF} + NaOH \rightarrow \overline{AlO^-} + Na^+ + F^-$$

$$\overline{AlO^-} + H_2SO_4 \rightarrow \overline{AlOH} \text{ or } \overline{AlSO_4^-}$$

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When to Regenerate?

Activated Alumina is considered saturated when fluoride levels in treated water begin to exceed the limit set for potable water, which is 1.5 mg/lit (this is the maximum permissible level of fluoride in potable water as per Bureau of Indian Standards and the permissible level as per WHO Guidelines). Commercial fluoride test kits of YES/NO type can be used to detect when fluoride in treated water exceeds permissible levels, at which point the AA can be considered exhausted after which regeneration is necessary.

**Pink.....Fluoride within acceptable limit*

**Yellow.....Fluoride beyond acceptable limit*

How to Regenerate?

To regenerate exhausted Activated Alumina, a dilute solution of Caustic Soda is used. To restore the fluoride removal capacity, basic alumina has to be acidified by treating with dilute acid. AA treated with acid is then washed with fresh water to bring pH in the range 6-7. The regenerated Activated Alumina is then ready for reuse.

Regeneration of Exhausted Activated Alumina

Household level activity

Transfer activated alumina from the domestic filter to a nylon (or woven plastic) bag. Make sure that all the material is transferred. Take care that there is no spilling while transferring.

Activities at Regeneration Centre

Precautions while handling Acid and Caustic Soda: Refer Annex 1

Preparation of Regenerants




Preparation of Alkali Regenerant

Weigh 100 gm of Caustic Soda in a plastic container and add it slowly into a plastic bucket containing 10 litres of water. Stir with a plastic rod to dissolve the flakes or pellets.

Keep the caustic soda container tightly closed. Otherwise caustic soda absorbs water and loses its strength.

Preparation of Acid Regenerant

Transfer 100 ml of concentrated Sulphuric acid from an acid bottle to a plastic measuring cylinder. Pour the acid slowly along a plastic rod or tube or along the side of the 15 lit. capacity plastic bucket containing 10 lit. of water.

Stir gently with the plastic rod or pipe while adding acid.

Never add water to acid. This results in extreme heat generation and can cause accidents.

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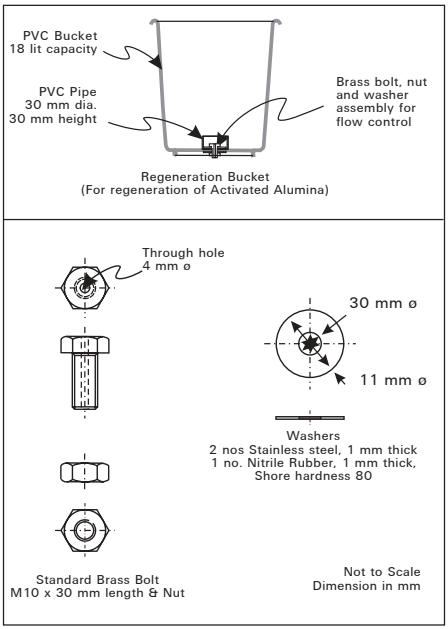
Regeneration Bucket

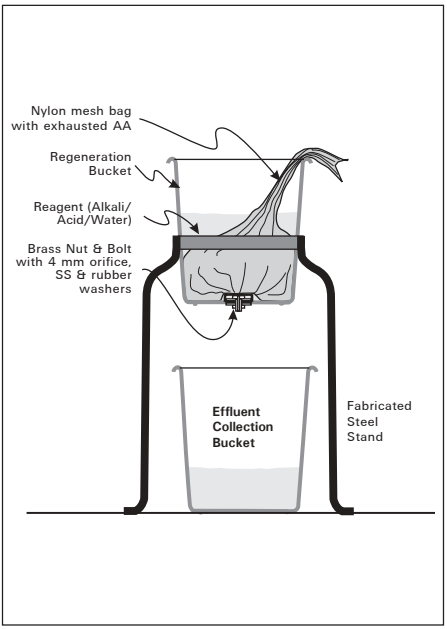
A Regeneration Bucket is made by fitting a brass or plastic nut and bolt along with stainless steel and rubber washers in the centre of the bottom of a plastic bucket of 18 lit. capacity. The bolt should have a hole of 4 mm for the regenerants flow through. A short piece of PVC pipe (3 cm dia. X 3 cm height) is placed around the bolt head in the regeneration bucket, so as to provide a clear space over the orifice. This facilitates flow of regenerant through the orifice and prevents it from choking.

A fabricated steel stand, as shown in the figure below, holds the Regeneration Bucket when it is filled with regenerating chemicals so as to allow it to drain into an Effluent Collection Bucket, placed below the regeneration bucket.

Regeneration Bucket for Activated Alumina



Set-up for Regeneration of Activated Alumina



Regeneration Procedure
Stepwise Description

Alkali Treatment
Step-1

Fig. 1.1

Fig. 1.2

Fig. 1.3

- 10 lit. of 1% NaOH regenerant is prepared in an 18 lit. plastic bucket, as mentioned earlier (Fig. 1.1).
- The exhausted AA is transferred to a bag made from nylon mesh (Fig. 1.2).
- The nylon mesh bag containing exhausted AA is dipped in the alkali in this bucket. Opposite sides of the bag are raised and lowered alternately, thoroughly agitating and churning the AA so that the entire volume of AA is well mixed and comes into full contact with the alkali (Fig. 1.3).
  This step is essential for effective regeneration of AA.

Step-2

Fig. 2.1

Fig. 2.2

- The Regeneration Bucket is placed in its stand. An empty bucket is placed below the Regeneration Bucket (Fig. 2.1).
- The nylon bag with AA is transferred to the Regeneration Bucket. 10 lit of alkali from the earlier step are poured into the Regeneration Bucket (Fig. 2.2).

Step-3

Fig. 3.1

Fig. 3.2

- Alkali flows through the orifice in the Regeneration Bucket and collects in the lower effluent collection bucket. (Fig. 3.1)
- The lower bucket is removed and a fresh empty bucket is placed below the Regeneration Bucket.
- 10 lit. of fresh water is poured in to the Regeneration Bucket and allowed to pass through the AA bed and collected into the lower bucket. (Fig. 3.2)

Acid Treatment

Step-4

Fig. 4.1

Fig. 4.2

- 10 lit. of 1% H_2SO_4 is prepared in an 18 lit. plastic bucket, as mentioned earlier (Fig. 4.1).
- As described in Step 1, opposite sides of the nylon bag with AA are alternately raised and lowered in the acid bucket to thoroughly churn the AA in the acid solution (Fig. 4.2). This step is essential to bring about proper contact between AA and acid to facilitate proper contact between the AA and the acid.
  This step is essential for effective regeneration of AA.

Step-5

Fig. 5.1

Fig. 5.2

- The empty Regeneration Bucket is placed in its stand and an empty collection bucket is placed below it to collect the effluent.

Fig. 5.3

- The nylon bag containing AA is then placed back in the Regeneration Bucket and the acid is poured into this (Fig. 5.1 and Fig. 5.2).
- After acid is drained out from the bucket, nylon bag is lifted to check the pH of the drained water, which should be in the range of 4-5 (Fig. 5.3).

Final Washing

Step-6

Fig. 6

- After acid treatment, AA in the nylon bag is washed repeatedly (2 to 3 times) (Fig. 6) with fresh water in the Regeneration Bucket, until the pH of water dripping from the orifice is in the range of 6.0 - 6.5 (Fig. 7).

Step-7

Fig. 7

- The effluents (alkali, acid and wash water) collected in the lower buckets are disposed in the Settling Tank.

Now AA is ready for reuse

Regeneration steps for Exhausted Activated Alumina (Flow Chart)

Steps in 18 lit. Plastic Bucket

Prepare 1% NaOH (10 lit.) in the Plastic Bucket (A)

Place the Nylon bag containing exhausted AA in (A) Agitate thoroughly for 1 min.

Prepare 1% H_2SO_4 (10 lit.) in Plastic Bucket (C)

Transfer Nylon bag (with AA) to bucket (C) Agitate thoroughly for 1 min.

Steps in Regeneration Buckets

Transfer Nylon bag (with AA) to Regeneration Bucket (B)
Pour alkali from (A) to (B). Allow the alkali to percolate through AA (≈ 12 min.)

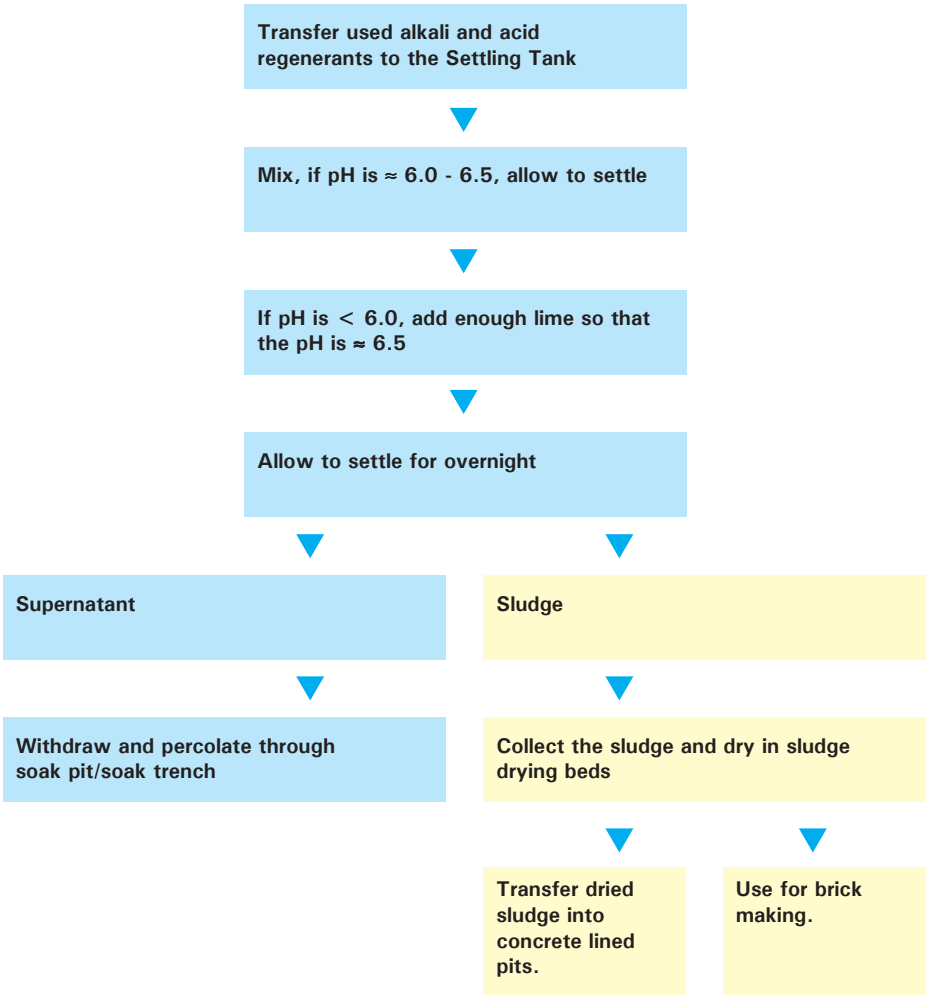
Pour 10 lit. fresh water, agitate the bag few times. Allow the water to flow through AA (≈ 12 min)

Transfer the nylon bag back to (B).
Pour the acid into (B). Allow the acid to percolate through AA (≈ 12 min)

Wash with fresh water till pH of wash water dripping from the bag is 6.0- 6.5

Regenerated Activated Alumina is ready for reuse

Disposal of Spent Regenerants



Requirements of Regeneration Centre

Chemicals for 10 regenerations

1.	Concentrated Sulphuric acid (commercial grade, 97% purity)	1 lit.*
2.	Sodium hydroxide (commercial grade, 97% purity)	1 Kg*
3.	Lime (90% purity)	0.5 Kg
4.	Fluoride Yes/No test kit	
5.	pH strip broad range	(2-14 range) (Litmus paper is not acceptable)

**Regenerant chemical requirements are for 4 Kg AA.*

Raw water requirement (for 10 regenerations per day) For 4 Kg bag of AA

10 lit. (Alkali) + 10 lit. water (rinsing) + 10 lit. (Acid) + 20 - 30 lit. final washing	= 50 lit per bag
Total fresh water requirement per 10 of AA bags	= 50 lit. x 10 = 500 lit.
Spent acid/alkali regenerants (20 lit.) to be treated by adding lime.	
Wash water can be mixed in the Settling Tank and disposed.	

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Equipment & Materials required (for 10 regenerations per day)

	Items		No.
1.	Weighing balances	5 Kg. 500 gm.	1 1
2.	500 lit. Syntex (HDPE) container fixed with outlet tap (for storing raw water)		2
3.	Plastic buckets (18 lit. capacity)		25
4.	Plastic measuring cylinders:	1000 ml 100 ml	2 6
5.	Plastic mugs (1 lit.)		20
6.	Plastic small plate for Weighing		5
7.	Cast iron stands for holding Regeneration bucket		10
8.	Nylon mesh bags, 30 cm width x 45 cm in depth, width of aperture -0.106 mm, IS 460: 1962 specification 106 (Width of aperture 0.106 mm)		50
9.	Fabricated steel stands or Concrete counter, for placing 6 nos. Regeneration buckets 45 cm above ground level, so that they can be drained into collecting buckets below		10
10	Heavy duty rubber gloves, protective Goggles		3 sets
11.	PVC pipe, 20 mm ND, 60 cm long, for mixing chemicals		2
12.	First Aid box		1
13.	Tools & materials for spill management		

Regeneration Centre

The Regeneration Centre for upto 24 bags per day in 3 batches, comprises of a room, two settling tanks, soak pits or trenches, two sludge drying beds and a concrete-lined sludge drying bed. **The Regeneration Centre should be near a water source so that water is easily available.** The schematic layout of The Regeneration Centre is given on the next page. As mentioned earlier, its main components are:

1. Two Neutralisation-cum-settling tanks – Masonry structures, approximately 100x100x100 cm with a bed slope of 1 in 10, partitioned to allow sedimentation in the first compartment. 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. Two Sludge drying beds – Masonry built of size 80 x 80 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. Lockable shelf to store lime.
4. Lockable shelf to store caustic soda.
5. Shelf for keeping other equipment and consumables.
6. Shelf for storing technical and administrative records.
7. Office table with chair
8. Lockable shelf with wire mesh door for storing acid.
9. Masonry platform with sanitary tiles for placing fluoride meter, test reagents, balance, etc.
10. Masonry platforms for placing Regeneration Buckets, with plumbing for reagents, drain for effluents, etc.
    Water (5000 liters), Acid and Alkali storage tanks of 200-250 litres each, on the roof of the structure, with appropriate plumbing lines to the Regeneration room and with drainage for easy washing and cleaning, connected to the Settling Tanks.

Not shown:

1. Exhaust fan
2. Masonry pit 75 x 75 x 75 cm, plastered inside with cement mortar 1:3, to store dried sludge for later disposal.
3. Two Soak pits - size 1 m x 1 m x 1.25 m deep, filled with coarse gravel or two Soak trenches made from 6 m long 150 mm dia. perforated PVC pipes, set in trenches, 1 m to 1.5 m deep, 0.5 m wide, covered with a layer of 0.3 m depth of coarse gravel, and then back filled with excavated soil.

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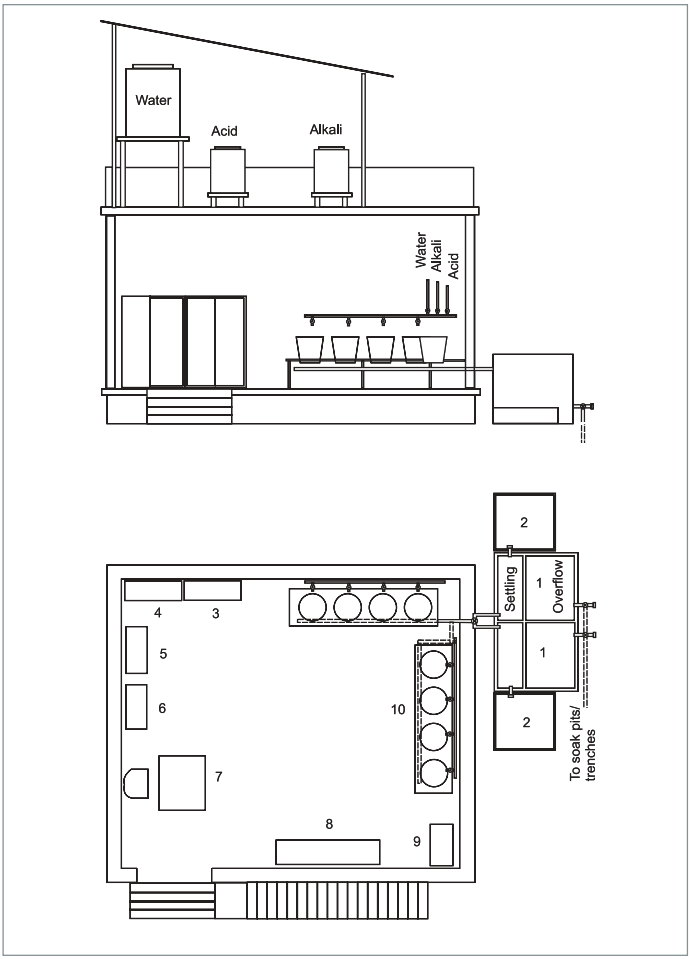
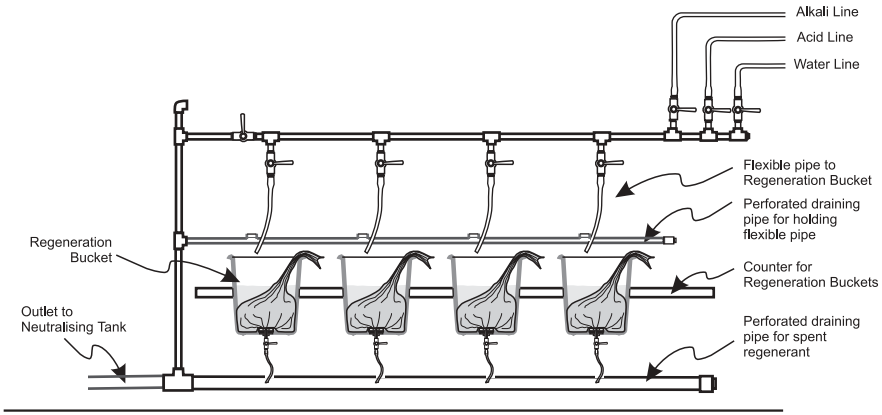
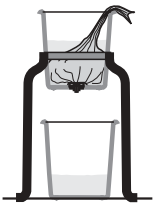
Schematic Layout of Regeneration Centre



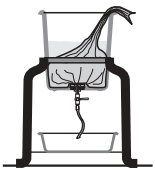
Layout of Multiple Bucket Regeneration Set-up

All piping should be in thick walled PVC pipe with threaded joints. All faucets in PVC or SS, very good quality, corrosion and leak proof. Flexible lines should be in braided, heat resistant PVC hose.





Conventional Regeneration Set-up



Lower Steel Stands for handling Buckets

Annex 1

Handling of Sulphuric acid and Caustic soda during Regeneration of Activated Alumina

Precautions and Safety Measures

Sulphuric acid and Caustic soda are chemicals used for regeneration of exhausted Activated Alumina from defluoridation units at the domestic level. Owing to their hazardous nature, the transportation, storage and use of these chemicals can result in accidents. Hence, it is important that the properties of these chemicals, the risks involved in their handling and the precautions and safety measures to be taken, are properly understood and well known.

The chemicals can enter the body orally or by inhalation. Contact with skin causes burning sensation and can even result in burns and wounds. These chemicals can affect the eyes, nose and skin directly.

Sulphuric acid is liquid in nature and Caustic soda is solid (in the form of pellets or flakes). Both these chemicals, when used for regeneration, are of commercial grade. Sulphuric acid is available in glass bottles whereas Caustic soda is available in sealed boxes/ containers.

Chemicals should be stored in a dry, airy place, under lock and key and away from the reach of animals and people, especially children. The chemicals should always be kept covered and should not be kept very close to each other. Care should be taken that the bottles or the boxes are not held by the lid or the neck while carrying from one place to another. Instead, both hands should be used for the purpose. In case of more than two chemicals, trays or buckets should be used.

For cleaning of the table or the floor in case of spillage of chemicals, the following things should be readily available:

Brush (1 no.), Mopping cloth (1 no.), Goggles (1 pair), Gloves (I pair), Plastic bags for waste disposal, Polythene bag (1 no.), Lime/sand/mud (1 bucket), First-aid box (1 no.).

Precautions while using Sulphuric acid

1. Sulphuric acid is extremely dangerous and can cause deep burns and scars on the skin and has a destructive action on all tissues. If eyes are exposed to acid, it can lead to blindness. Fumes and mist can produce severe irritation of the respiratory tract, which includes nose and throat. Hence care should be taken that it does not come in contact with the skin.

2. In case the acid comes in contact with the skin, the affected part should be washed thoroughly with water and an anti-burn cream should be applied. In case, of eyes, they should be washed repeatedly with water for 10-15 minutes, since absence of immediate action can lead to blindness.

3. Concentrated Sulphuric acid is a liquid and is normally supplied in glass bottles. These bottles should be preferably stored on a low shelf which is kept securely away from the reach of unauthorised persons and children. Always store Sulphuric acid away from Caustic soda.

4. Extreme care has to be taken while transporting the bottles. Do not hold the bottle from the neck, as the bottle is liable to be broken 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.

5. All the materials required should be kept ready before using the chemicals. Watches, rings and metal bangles should be taken off and gloves should be worn. In case the regeneration operator is wearing a full sleeved shirt, he should fold up the sleeves before working. Women should tie up their hair and *dupattas, chunnis, sari,* etc. so as to prevent any contact with the chemicals. Gloves and goggles should necessarily be worn while the acid is being poured and the solution is being prepared. After opening the bottle, the cork should be removed gently to prevent any spillage onto the face or hands.

6. The acid should be poured slowly into the measuring cylinder to the required level. The acid should then be carefully poured into the water from near the edge of the bucket or along the side of a plastic pipe/ rod.

7. Water should never be poured into the acid as this could lead to accidents. It should always be the other way round; the acid should be poured into the water instead.

8. In case of spillage of acid on the floor, lime should be spread over it immediately so as to prevent spreading of acid and spoiling of the floor. Ash/ sand/ mud balls can also be used to cover the acid if lime/ soda-ash is not available. It should be seen that no one ventures near the affected area.

9. Putting ash/lime over spilt acid leads to effervescence and rise in temperature. The sludge should therefore be collected in a plastic bag with the help of a plastic spoon/ scoop or dust collecting pan only after two minutes.

10. It may be difficult to remove acid remnants from leather articles like belts, shoes and caps even after repeated washing. Such articles should be discarded. Even clothes spoiled by acid spillage should be removed and should not be worn without washing.

To contain spilled concentrated H_2SO_4 even sand can be used first and thereafter a weak base (lime) can be used.

Use of Sodium hydroxide pellets to neutralise an acid spill is not recommended.

Things not to do

1. **Water should never be poured directly on spilt acid. Cotton or jute cloth should not be used for wiping up the spilt acid.**
2. **The spilt acid should also not be covered with any jute bag or paper or cloth as this can lead to fire accidents.**
3. **No iron or aluminum article should come in contact with the acid as it can lead to explosion.**
4. **Empty acid bottles should not be used for any other purpose.**

Precautions while using NaOH/Caustic soda (Solid)

1. Caustic soda can cause burns, hence should not be allowed to come in contact with skin.
2. Caustic soda (pellets or flakes) spilt on the floor should be collected using a broom.
3. Rubber gloves should also be used while collecting Caustic soda (pellets or flakes) from the floor.
4. After the Caustic soda is removed, the floor should be washed thoroughly.
5. Rubber gloves and goggles should be used while Caustic soda is being lifted, weighed or mixed in solution.
6. In case of the Caustic soda (pellets, flakes or solution) coming in contact with the skin, the affected part should be washed thoroughly with water and 2% Acetic acid solution (2 ml glacial acetic acid in 100 ml water). Apply an anti-burn cream on the affected area. Hydroxides are extremely damaging to eyes. If exposure occurs, eyes should be washed immediately with cold running water for 15 min.
7. Medical advice should be taken if necessary.
8. Caustic soda bottle/ container should not be left uncovered as contact with air/ moisture may spoil the chemical.
9. Caustic soda should always be weighed in a plastic or glass container.
10. After the weighing is completed, the container used for the purpose should be washed and cleaned thoroughly.
11. 100 gm Caustic soda should be put in a bucket of water and stirred slowly using a plastic/ glass rod till complete dissolution takes place.
12. The bag containing Activated Alumina should be very gently immersed in the Caustic soda solution so as to avoid spillage.

Things not to do

1. **The Caustic soda bottle/ container should not be kept uncovered or in a wet place and should be kept away from contact of water.**
2. **No oil or cream should be used if the chemical enters the eyes.**
3. **Caustic soda should not be put directly on the plates of the weighing balance for weighing. Instead containers should be used.**
4. **Caustic soda should be removed from the bottle/ container using a spoon and never by the hand.**
5. **Empty Caustic soda bottles should not be used for any other purpose.**