

**PILOT SCALE STUDY OF A CHEMICAL TREATMENT PROCESS  
FOR DECONTAMINATION OF AQUEOUS RADIOACTIVE WASTE  
OF PAKISTAN RESEARCH REACTOR-1**

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## **Abstract**

Chemical treatment process for the low level liquid radioactive waste generated at PINSTECH was previously optimized on lab-scale making use of coprecipitation of hydrous oxides of iron in basic medium. Ferrous sulfate was used as coagulant. Batch wise application of this procedure on pilot scale has been tested on a 1200 L batch volume of typical PINSTECH liquid waste. Different parameters and unit operations have been evaluated. The required data for the construction of a small size treatment plant envisioned can be used for demonstration/teaching purpose as well as for the decontamination of the waste effluents of the Institute. The lab-scale process parameters were verified valid on pilot scale. It was observed that reagent doses can further be economized with out any deterioration of the Decontamination Factors (DF) achieved or of any other aspect of the process. This simple, cost-effective, DF-efficient and time-smart batch wise process could be coupled with an assortment of other treatment operations thus affording universal application. Observations recorded during this study are presented.

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## 1. INTRODUCTION

After upgrading the PARR-1 in Nov. 1990, the volume and radioactive content of the aqueous waste effluents has increased significantly. A major portion of the radioactivity in the waste effluents is due to  $^{110m}\text{Ag}$  and  $^{124}\text{Sb}$ . This is added into the waste, from reactor's control rod material through nuclear transformations.  $^{110m}\text{Ag}$  and  $^{124}\text{Sb}$  radionuclides with significant production abundance with from the standpoint of our storage capacity, and since internal norms no more support near-surface disposal of radioactive effluents without any treatment, warrant chemical treatment. Removal of  $^{110m}\text{Ag}$  and  $^{124}\text{Sb}$  radionuclides through different means including coprecipitation was studied [1,2] and a process based on scavenging precipitation of hydrous oxides of iron was optimized [3] on lab-scale for the decontamination of low level liquid waste (LLW) effluents.

Chemical treatment of LLW, usually used at nuclear installations, is always accompanied by physical methods like sedimentation, filtration, and centrifugation etc. Chemical treatment is relatively low cost and is based on well-proven conventional plant equipment. It has the ability to handle a large variety of radionuclides, non-radioactive salts and solid matter in solution. It is easy to change the chemical precipitants in order to accommodate changes in the composition of the liquid feed and at the same time, big liquid flows can be handled both continuously and batch wise very economically. Disadvantages being the rather low decontamination factors (DF) achieved, the relatively large volumes of sludge to be managed and difficulties with continuous automatic operation. High inactive salt content of the treated water can reduce the scope for a second treatment such as ion exchange and which may also be undesirable from environmental management standpoint. The upper activity level specified to categorize LLW and the levels for their release to the environment varies globally but usually are based on the guidelines and recommendations of the international commission on radiological protection (ICRP) and local regulations [4-7]. This in conjunction with other factors is taken into account while optimizing a treatment procedure. As a general guide, the upper activity level for LLW to be treated by a coagulation process will approximate a 100 times the *maximum permissible concentration* (MPC) in water and in accordance with the 'as low as reasonably achievable (ALARA)' concept [8]. However, for mixed radionuclides inventory, it is not customary to determine the concentration (MPC) of individual isotopes except for the radiologically most hazardous ones and gross activities are usually dealt with. At PINSTECH the release limits for liquid discharges have been set at ( $A \leq 3.7 \text{ MBq/m}^3$ ) by the Pakistan Nuclear Regulatory Authority (PNRA) [9, 10]. The selection of the treatment procedure [11] and the optimum doses of the reagents involved depended on chemical composition of the waste, nature and extent of radionuclides, extent of decontamination factors, volume reduction factor (VRF) and regulatory release limits for our site.

The present study is based on the already optimized process [3]. Objective of the current study was to evaluate lab-scale parameters of a scavenging decontamination process on pilot scale. Since a batch process that could be completed preferentially in a single working-day time (8 hours) was desired, it seemed to offer very little time for the sludge to age and adsorb radionuclides and to settle neatly thus affecting the optimum doses and filtration operation. It was further envisaged that testing will also provide baseline data for the assessment of personnel and environmental protection, permits and approval. Following points were under considerations:

- a. Evaluate in situ performance of the optimized reagent doses on above-lab-scale.
- b. Obtain design parameters for a working plant on small/demonstration scale.

- c. Evaluate the time dependence of the discrete and assembled unit processes.
- d. Obtain data for the development and validation of predictive techniques for options like additional radionuclides handling and separation of the solid phase etc.

The objective also was to have the runtime parameters (and cautions) both industrial and radiological, and to have parameters for possible automation and computerization.

### **3. SCALE UP PARAMETERS**

#### **3.1. The optimized Process**

Detailed description of the optimized lab-scale process is given elsewhere [3]. The coagulant and other reagent doses, derived from lab-scale optimums, for a 1200 L batch are reproduced in Tables 1 and 2. The values in brackets (Table 1) were used. Freshly prepared solutions were used in plastic containers. On the basis of previous experience and recently studied bench-top behaviour of the process, some saving in the reagent doses was expected. Similarly sludge volume at the test dose and settling rate was expected to be around 10 % at 90 minutes. Feed activity and the required DF however were not favoring lower doses since lower than optimum activity range as observed on lab-scale experiments required somewhat higher doses. Decantation of the supernatant followed by dewatering of the expected 10% volume of the concentrates was to be tested. The reduced volume for filtration unit operation would then result in more efficient and quicker process.

#### **3.2. Description of the scale up process**

Behaviour of the decontamination process is shown in the Figures 2 through 9 (some reproduced from already published work). The process flow-sheet is very simple (Fig. 1). Basic steps and underlying chemical and physical principles are few for the current study.

Usual constituents of the wastewater entering the process approximated tap water with respect to total hardness, turbidity and pH. The latter two was being a little higher. The total hardness of the feed solution averaged to 56 ppm, turbidity ranged from 2.5 to 3.8 NTU and pH averaged to 8.8 pH units. The averaged chemical profile of a typical composite feed sample is presented in Table 3. The waste effluents are transferred to a centralized waste collection and storage system wherein these streams are held in a cascade of basins according to the activity concentration and storage capacity at different levels. These are finally routed to the final decay tanks (four, of 85m<sup>3</sup> each) in the controlled area. One of these tanks was temporarily designated as a settling/equalization basin for untreated discharge where the effluents also obtained system equilibrium. The strainer stage was purposely omitted to observe the effect of suspended particulate and bio-matter in the preliminary two runs; filtered (cloth strainer) inflow was incorporated in the third run. With no organic solvents and very low detergent levels (presumably, keeping in view the nature and frequency of addition of such substances into the waste line), these components were not considered at any stage of the operation.

### **4. EQUIPMENT AND OPERATION**

A used-up M.S. tank (rubber lined) with a capacity of >1200 liters was fitted with a top loaded motor-driven shaking mechanism. The vessel had sufficient capacity to hold all

the reactants till completion and was open from top to permit visual inspection. The 1400 RPM motor (3-Phase, 2HP from Mitsubishi, Japan) coupled with 2 pitched-blade turbine agitator was run at its natural speed (no gearing mechanism was provided since very little motor operation was needed). The blades (two, each 9" long and 2.5" wide) were adjustable in both their angle with the rotation plane and position in the tank to have the desired shaking control. Since no baffles were provided in the tank that would have caused the laminar circular flow of the bulk fluid to break, as observed during the non active runs, the position of the shaft was shifted a little off-centre in order to induce some degree of turbulence in the subsequent two runs. Usual centrifugal pump was used for transferring waste liquid from the equalization tank to the coagulation tank. A siphon, floating on the surface with lateral suction of the supernatant from the surface without disturbing the sludge sediments and with priming mechanism was provided in the tank. The tank also had a lateral bottom outlet that was used for cloth bag filtration. Two different grades of cloth were used to see the extent of retention and speed of filtration.

An underground 27 m<sup>3</sup> tank (one of two in the collection line cascade) was used as arising tank, off which a volume equal to about four batch volumes was drawn into equalizing tank. The batch volume, after equalization was shifted to reaction vessel without any strainer for the first two batches and with cloth straining for the third batch. A 60L stainless steel drum having a bottom outlet was used for holding coagulant solution for flash mixing. This arrangement however was not much appropriate and the contents were mixed through gravity at the centre of the whorl around the moving shaft of the mixer assembly. The open top of the reaction vessel was covered with polyethylene (polythene) sheet and manual bulk mixing of the coagulant was avoided to assess feasibility of continuous process operation. The reactor was run at its maximum handling capacity (1200 L) affording room for the marginal rise in the bulk volume due to the whorl dip in the rotating wastewater bulk. Batch volume in the next two runs was kept at 1000 L level.

## **5. DESCRIPTION OF THE UNIT PROCESS OPERATIONS**

The process consisted of two basic operations viz precipitation and filtration with associated phenomena like coagulation, flocculation clarification and settling of the sludge etc. Because of scantiness of the equipment, multiple unit operations were carried out in a single basin that was run like a coagulator. Different parameters included in the tests were: pH adjustment, separation of solids either settled or in suspension, flash mixing, coagulation, flocculation, settling of the solid phase, bulk and consistency of sludge, coagulant doses for maximum and clearance level activity and DF and VRF at these preset limits. Run-time optimization, space and cost effectiveness were to be evaluated. Other aspects like simplicity of the process and its extent of tailoring for automation were observed.

### **5.1 Coagulation unit operation**

A 1.3 hp centrifugal pump (1½ x 1¼ in) was used for transferring wastewater from equalization tank to the coagulation tank at a rate of ~50 lpm. The batch volume was prealkalized with the calculated volume of NaOH (~0.01M for pH 12) in the precipitation tank and was precipitated by the addition of coagulant solution while the batch had already attained the appropriate mixing speed. In the subsequent runs the pH was preadjusted to the optimum value (pH 10) and the rest of the alkali was added gradually lagging behind the coagulant keeping the pH nearly constant. Samples were drawn in-between and checked for flock characteristics and DFs. It was observed that

NaOH doses, in this way, can be reduced significantly that would help in the finishing stages and in meeting the EPA requirements for disposal to public sewer or environment. The precipitates were grown and flock formation commenced by shutting the speed-shaking. Any precipitation of hardness cations e.g Ca or Mg or frothing could not be observed in the low salt effluents.

## **5.2 Mixing unit operation**

Since it provides relatively higher supersaturation within the vicinity of incipient precipitation, flash mixing is very important for achieving better decontamination factors. Increased relative supersaturation (RSS) acquired by rapidly mixing concentrated solutions results in larger number of seed nuclei thus forming highly viscous and gelatinous precipitates with very large active surface area available for adsorption to take place on. Rapid mixing also helps the precipitated particles to leave apart and afford enough time for the barren particles to adsorb contaminant cations before they coalesce. After completion of the coagulation, that took about 10 - 15 minutes, on the first active run, shaking was stopped and the bulk volume rotating under its momentum was allowed to stop naturally. The slow mixing during this course was required and was appropriate for flock formation. However it took a bit longer for the flock to settle down demanding early stopping of the shaking mechanism before precipitation was complete for time considerations. Shifting the shaft of the shaking propeller to off centre, somewhat reduced laminar bulk rotation, however, baffles along the walls of the tank, if were provided, would have greatly enhanced flash mixing and savings in time.

## **5.3 Flocculation and Sedimentation steps**

Slow and gentle agitation of the treated charge helped in bringing about more and more particles in contact thus coalesce to grow flock particles. The flock was so much well developed that no other treatment or aid was needed to settle it down. The viscosity of water (a function of both temperature and salt conc.) has a significant effect on the sedimentation velocity. Ambient temperature recorded was around 29 °C. The settling rate determined by a  $\phi$  1 cm graduated glass tube open at both ends was not in step with lab-scale experimentations in the tank having a height-to-diameter ratio of approx. 1.1:1. It took about two hours that the sludge was settled to a degree (ca 19%) the supernatant could now be siphoned out to leave behind the slurry. The speculated 10% slurry volume at about 90 minutes (1.1:1 h/ $\phi$ ) could not be achieved probably due to the total sludge quantity per unit length of the sludge column in the tank that caused the sludge density to grow faster retarding the compaction rate. However the time left was still enough to finish in time with some more time at hand to go for slurry volume reduction and compaction and to perform the mandatory house keeping. The batch which contained some biomass retarded sedimentation but to a less significant level. The extent of algal growth and other suspended matter in the feed however was not determined but it was conspicuous.

## **5.4. Separation of the solid phase**

Cloth bag filtration was adopted due to its simplicity. Two different grades of cotton filtering fabric were tested on non active batch. The compact grade available by that time suffered some leakage of finer particles when the lateral outlet of the precipitation tank ( $\phi$  6 cm) was used that caused a dragging of the settled sludge by the clear supernatant diluting and tearing apart the flock against cloth bag surface and taken out off the 50 x 30 x 2.5 cm bag by the pressurized effluents. This reduced the effective DF

drastically when used on active run. A bigger bag was not however tried, instead the siphon mechanism was applied that reduced the bulk volume of effluents and the concentrated slurry containing total sludge was bag filtered at reduced pressure with efficient filtration. The effective DF was much better. Properly calculated and appropriately designed cloth bag of the same compact grade fabric would have yielded better results for on stream filtration.

## **6. DISCUSSION**

Only very few, i.e. three runs in a time period of about 5 weeks were made with emphasis on the verification of the lab-scale doses. Two non active runs with tap water to see the behaviour of the assembly and of different individual steps were conducted before loading the waste volume in to it. The degree of decontamination required was not high and a compromise between the leftover short lived radioactivity in the treated effluents and the added chemical activity and condition for ground disposal was considered. Treatment for maximum removal was not worthwhile and the anticipated doses for a rational extent of decontamination appeared to be significantly low. Radiological conditions observed were quite safe for manual operation of a small scale plant based on these parameters, however, the process proved to be very flexible, easily tailor-able for individual radionuclides and subject to easy automation. Bioactivity was tolerated well with no biological pretreatment that deemed unnecessary. No elaborate filtration equipment was needed nor was any complex dosing and control arrangements required during these simple demonstrations that delivered quite reasonable results very cost effectively. Based on these observations a very economical demonstration/small scale plant can be constructed which could be utilized for training and teaching purpose as well as for treatment of low salt low activity waste effluents of a research reactor.

Though no close control of the process is required, in practice, few bench-top tests for ascertaining the coagulant dose and flocculation point (pH & electrolytes conc.) will greatly save time and post process adjustments. This will account for the slight changes in water temperature, pH and turbidity. These tests can easily be carried out in simple beakers or graduated cylinders.

Actinides including Np and Am, isotopes of Cr, Co, Cs and certain activation products [8, 9 (ref. 61, 62 therein)] are likely to be removed to greater extent than isotopes of Cs and Sr. Similarly, Ru [10], due to the excellent reducing characteristics of Fe(II) ion, is likely to show efficient removal. For more specific needs, the process could, with little modifications, be operated in series or in combination with others reagents specific for those nuclides [4, 8 (ref. 62 therein)] for liquid waste of medium/high activity followed by a polishing step like ion-exchange etc. Total salt content of the feed however must be considered. One should expect better kinetics at increased batch temperatures likely to be prevailed at summer in our country.

### **6.1. Run time and throughput**

Flock build-up and settling alone were wanting for about 30 % of one complete cycle of the matured process. Siphoning and cloth bag filtrations were next in time consumption. These unit operations required about one and a half hours while half an hour was required for transferring wastewater into the reaction vessel. Forty-five minutes were taken by preliminary preparations and preparation of the reagent solutions. The remaining working hours were enough for concluding the batch, isolation and containing the active concentrates and the mandatory housekeeping.

The throughput was limited only by the available equipment and was kept proportionate to the production rate and the storage capacity. A further larger batch could have been handled without undue radiological implications and time



considerations with the current activity level; however, this if needed, an hour or more would be required for the finer flock particles to settle and save the achieved DF from losing it through poor solid phase separation at higher activity level.

## **6.2. Composition of activity in the waste stream after treatment**

In all the three runs the average activity of the processed effluents was not much high. Since the batches were arisen from the already decayed out waste streams, the level of activity was little above the allowable levels for near-surface disposal. The low activity level however, was favouring the dose optimization, since; low activity being difficult to remove was yielding a low DF value that was on the safer side. With the test doses, 96% of the silver activity and almost all of the antimony activity was removed from the waste in the first run while for removal of the available activity to the extent of allowable limits for near-surface disposal, as high as 80 % of the lab-scale doses worked well in the subsequent runs. This seemed a little higher, but the reason probably was inappropriate flash mixing stage and insufficient time allowed before sampling for analysis at times when there were finer flock particles still suspended in the batch.

The pH was basic enough, around 10, which needed neutralization of the effluents to meet EPA recommendations for their discharge into either public sewer or ground. Similarly the  $\text{Fe}^{2+}/\text{Fe}^{3+}$  content neither was studied nor adjusted for this purpose. Optimum reduction of effluent hardness is very important because the on-stream life of the polishing filters is greatly increased by decreasing the hardness of the effluents at headend; for this purpose anions like phosphate may impede calcium removal which in turn detracts Sr and Cs decontamination. This adjustment was not made as no polishing stage was included in the test.

## **6.3. Removal Efficiency and Cost Estimates**

The degree of decontamination achieved is a direct measure of the amounts of coagulants used up to a certain limit where the process achieves a plateau response [3]. However, the treatment costs are very reasonable and can further be economized by keeping the extent of treatment given to the waste effluents up to the allowable regulatory levels for the radioactivity. Economic use of reagents can be effected by holding the pH at 9-10 during precipitation step with controlled addition of NaOH (in case the coagulant is used last [3]). In this case the batch to batch variations will be eliminated. The efforts involved doing this however, could barely justify the saving in the costs.

Factors like 'Volume Reduction' (VRF), the ratio of the volume of waste before treatment to the volume of the process residues containing the bulk of radioactivity), 'Decontamination Factor (DF)',  $(\text{unit activity} \times \text{Feed volume})/(\text{unit activity} \times \text{Effluent volume})$  and the 'Concentration Factor',  $(\text{volume of waste processed}/\text{volume of sludge to be stored})$  for process in which volume changes occur during treatment should be accounted for. However, if the sludge is dewatered then and there, volume changes may be insignificant.

The results of the pilot scale and bench-top work indicate that the process can have expanded application and will yield an effluent that will not require dilution before final disposal. A very cost effective, in terms of both initial capital and running costs, can be designed with major components and large capacity vessels made from glass fiber reinforced resin polymer with polyethylene/polypropylene piping where necessary.

## 6.4 Small scale plant design considerations

The basic treatment is a  $\text{FeSO}_4/\text{NaOH}$  precipitation at pH 9-10 of the wastewater. This can be carried out in a single flocculator appropriately designed with an operational capacity of  $5 \text{ m}^3$  ( $6 \text{ m}^3$  max.) per batch per day. Two runs in a month will do with the current production rate. If a continuous process is preferred, additional pretreatment tanks will be required and the flocculator may be altered (reduced in size) and in operating principle. Very little retention time of the order of 10 – 20 seconds will be sufficient then as, reaction between the reagents is very fast. In this case, two parallel flocculators, each with  $0.1 \text{ m}^3$  capacity will give the required processing rate. The DFs will also improve further because the pretreatment stage would allow greater time for the initial flock to mature and age. Centrifuge filtration and filtration using filter cloth of appropriate mesh can be potentially and economically adopted. Sand filtration can help in improved preconditioning before final disposal. A continuous vacuum filtration, for example, can be applied when the process is adopted for large operation or used as a combined process utilizing titanium hydroxide, barium sulfate, cobalt sulfide and nickel ferrocyanide etc. for other specific radionuclides. Local clay which has very attractive ion exchange capacity and reasonable filtration characteristics can be integrated with sand bed at finishing stage if salt content is high. It however needs to be optimized.

To tailor the process to a particular waste stream and if particularly nuclides like Cs has to be removed, the chemistry of the process may be reviewed. A number of IAEA [8-13] and other publications can provide excellent guidelines for this purpose. The precipitate of barium sulfate at pH ~8.5 for example, or a phosphate treatment can be used for the removal of strontium. The pH value is compatible with ferrocyanide precipitation (of Cu, Ni or Zn) used for the removal of cesium; the only requirement after the ferrocyanide precipitation might be to raise the pH value before the next stage. The alpha emitters in general are hydrolyzed in alkaline medium and, are readily entrained by hydroxides of iron and of aluminum and titanium as well. A further possibility is the precipitation of insoluble oxalates. Uranium co-precipitation has also been indicated for such treatments. The precipitation of plutonium and other alpha emitters at low pH values [8] can be used to separate them from majority of fission products which remain in solution. More exacting conditions can be expected for the disposal of alpha-bearing wastes. After separation of the actinide sludge, cesium can be precipitated by nickel ferrocyanide. The pH value then may be increased to 9, for example, and Fe-hydroxide can then be employed for the co-precipitation of ruthenium [8 (refs. 66, 67 therein)] and antimony.  $\text{HNO}_3$  and  $\text{HCl}$  flowsheets be accommodated for any future annexes to the process. Unusual constituents in the process wastewater entering the plant should also be accounted for which are not included in this study.

At present, no recycling of water was considered, however this aspect might be of interest and if dilution factor is to be incorporated in the process; a saving could be achieved in both the consumption of water and the reagents. Thus the effluents from the treated low activity waste, discharged to public sewage system, can instead be used to further dilute treated category-3 or -4 [15-17] effluents.

Enough downtime for maintenance is available since no continuous operation and complex equipment are required. Because of low radiation levels that would normally prevail, no remote operation/handling of any equipment would in principle be required. The applicability of the automation to the process has high degree of feasibility for demonstration purpose although it may not be required for the treatment of the waste on routine basis. Continuous on-line monitoring of parameters like pH, turbidity, hardness etc. will reduce man-hour/manpower requirement.

Cement immobilization of concentrates should be evaluated, for example, if slurries are dewatered and interim stored for an appropriate length of time, it might render them cleared resulting in reduced disposed of volume and burial space. First-batch sludge if

aged in contact with the mother liquor [3] till next operation of the second batch of the month (14 days, one run on fortnight basis) and then recycled in the next batch will greatly reduce doses as well as the hardness/saturation of soluble hardness-ions e.g. of Ca to deposit scum and scales and reduce on-stream life of polishing filters. Pre-alkalization (if coagulant is used in the last) of a batch with high hardness and/or Sr content before coagulation will also reduce Ca, Mg and Sr by precipitation; and suspended solids and dirt will be removed before adsorption on flock.

Capital cost reduction by incorporation of optimum usage of resources like only one SS pipe line for occasional use acid back wash, PVC lined carbon steel piping and valves; unplasticized schedule-80 PVC with screw joints etc. even for continuous operation and alike can be considered. However, considering any anticipated radiological hazard, the plant equipment and connecting lines should be designed in a simple, trouble free and efficient manner.

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Table-1: Optimum reagent doses based on lab-scale parameters

Reagent	Optimum Dose	Activity treated on Lab-scale	DF Obtained
Wastewater	1200 L	Ag, ~2700 Bq/L Sb, ~5950 Bq/L	Ag, > 450 Sb, > 290
FeSO <sub>4</sub> , commercial	1.68 – 1.92 kg		
NaOH (commercial lye)	3.84 – 4.8 L		
H <sub>2</sub> SO <sub>4</sub> A.R grade	0.36–[0.48] L		
Distilled water	to make 24–[36] L		

Table-2: Parameters for testing on extra-lab-scale

Reagent	Optimum dose	Activity treated on large-scale	DF expected for 37 Bq /L (clearance level) <sup>a</sup>
Wastewater	1200 L	Ag, ~1600 Bq/L Sb, ~50 Bq/L	Ag, ~ 43 <sup>b</sup> (~ 432) <sup>c</sup>
FeSO <sub>4</sub> , commercial	1.5 kg		
NaOH	1.94 kg		
H <sub>2</sub> SO <sub>4</sub> , commercial	0.5 L		
Distilled water	to make 36 L		

<sup>a</sup> On the basis of <sup>110m</sup>Ag only, all activity due to Sb radionuclides rather will be carried along according to removal efficiency for Sb.

<sup>b</sup> With cloth bag filtration that had low efficiency for the available grades.

<sup>c</sup> Decantation till 10% slurry volume left for dewatering later on expecting a 10-fold higher DF.

Table-3: Chemical profile of a treated liquid waste at PINSTECH

Entity	Concentration <sup>a</sup>	Entity	Concentration <sup>a</sup>
Calcium	~ 60	Zinc	0.02
Cobalt	bd	Bromide	bd
Iron	0.09	Carbonate, bicarbonate	570
Lead	bd	Chloride	623
Magnesium	5.5	Nitrate	90
Mercury	bd	Nitrite	bd
Potassium	1.1	Phosphate	10
Silicon	0.5	Sulfate	101
Sodium	459	Detergents	Very low levels <sup>b</sup>
Strontium	bd	EDTA/Decontaminants	Traces <sup>c</sup>
pH	8.8 pH unit	Conductivity	2390 (μ mohs)
TDS	625	Turbidity	3.1 NTU
Total Hardness	56	BOD/COD	Not measured
Organics	Traces <sup>b</sup>	Oil/Greases	- <sup>b</sup>

<sup>a</sup> Values are in ppm or otherwise stated determined by ICP and Ion Chromatography.

bd: Below detection.

<sup>b</sup> See text, sec. 3.2.

Table-4: Sludge volume at different coagulant concentrations in tapwater

Coagulant concentration (g/200 ml)	Slurry volume after 4 hr	Slurry volume after 48 hr	Slurry volume after 72 hr	Slurry Vol., % of total after 72 hrs
0.12	12	3.5	1.4	0.70
0.20	21.0	6.5	3.2	1.45
0.28	27.4	10.7	4.0	1.82
0.36	48.3	18.5	10.5	4.77
0.44	44.5	17.5	10.0	4.54
0.52	49.0	21.2	13.2	6.0
0.60	- <sup>a</sup>	42.5 <sup>a</sup>	36.5	16.59
0.80	68.6	21.1	15.1	6.86
1.20	72.2	18.2	12.2	5.55

<sup>a</sup> values are doubtful, could not have been properly recorded.

Table-5: Sludge characteristics observed in bench-top tests before large scale runs were conducted.

<b>Sludge characteristics after flocculation is over at pH 9-10.5 in waste-water</b>	
Coagulant concentration	5g/1L.
Settling + compaction time	24 hr.
Vol. Of slurry	50 ml.
Weight of slurry	50 g.
Specific gravity of slurry	1.
Water contents till complete drying	96 W%.
Weight of dry residues	1.96 g. 3.9 W% of slurry. 0.3 W% of freshly filtered slurry.
<b>Sludge characteristics after flocculation is over at pH 9-10.5 in tape-water</b>	
Coagulant concentration	5.6 g/3L.
Settling + compaction time	18 hr.
Vol. of slurry	125.6 ml (16.72 % of starting volume, 9.72% after 24 hrs). 28 ml after shaking the compacted sludge and one week later.
Volume of maximum compacted slurry	28 ml (13.63% of fresh 290 minuet slurry).
Weight of slurry	28.8558 g.
Specific gravity of slurry	1.0306.
Water contents till complete drying	89.643 W%.
Weight of dry residues	2.9886
Coagulant concentration	6.4 g/3L.
Settling + compaction time	106 ml when shaken vigorously and kept for 4 days, and 28 ml tar like after 10 days.
Vol. of slurry	
Texture of dried mass	Black-brown flacks to black chocolate like mass
Solubility of ppt.	
a) freshly filtered	Soluble in conc. Or dil. Mineral acids.
b) 24 hrs later	Soluble in conc. acids and >95% soluble in dil mineral acids.
c) after complete drying	Insoluble in 50% HCl, HNO <sub>3</sub> or mix., even on heating and decomposed slowly in conc. HCl giving bubbles of pungent smelling gas.

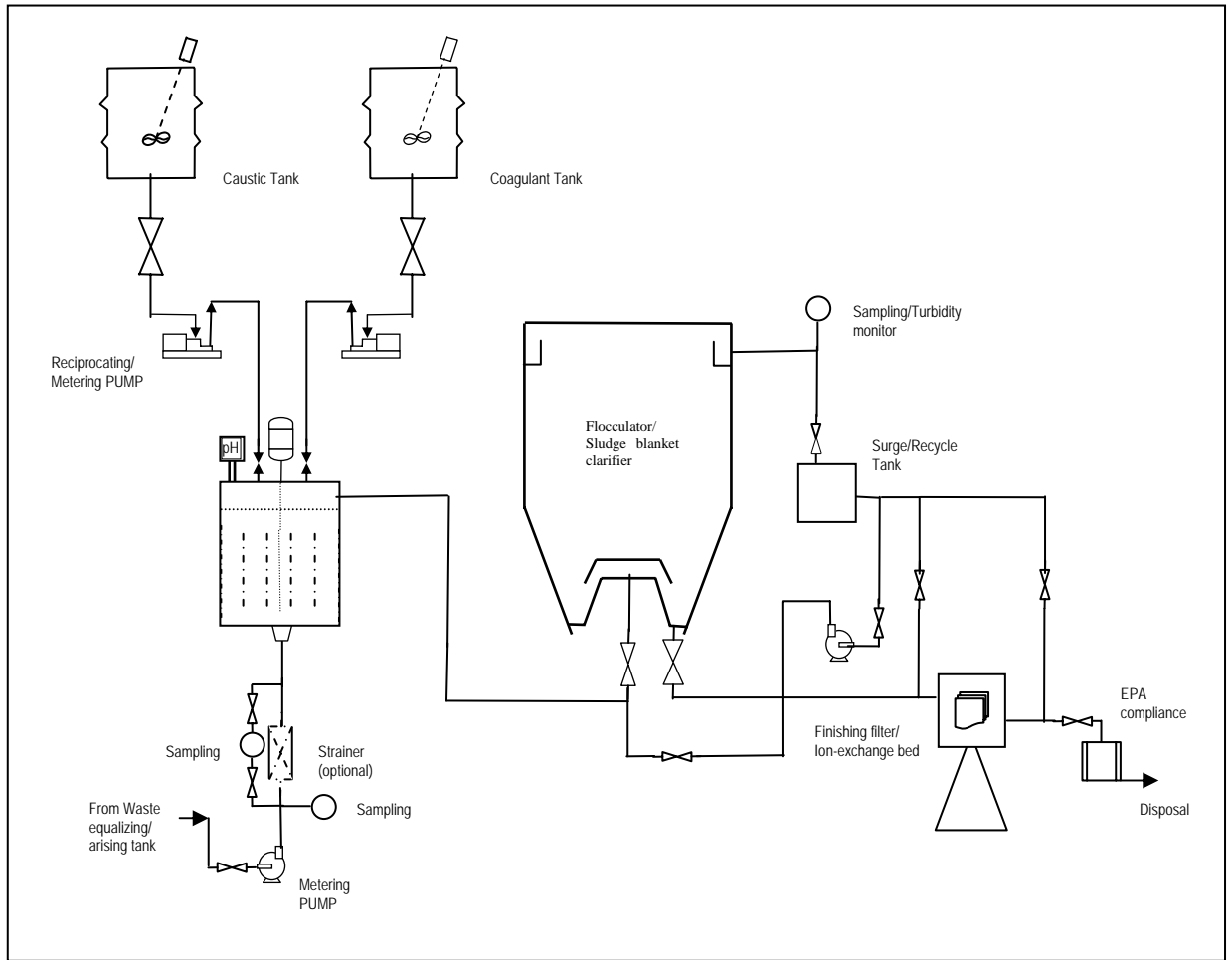


Fig. 1: Simplified flow-diagram of the process.



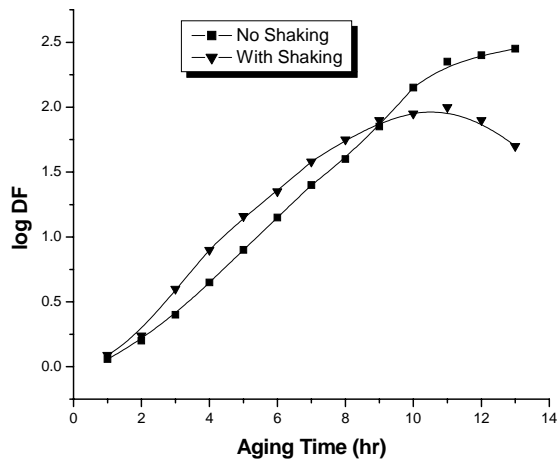


Fig. 2: Aging behavior with intermittent shaking.

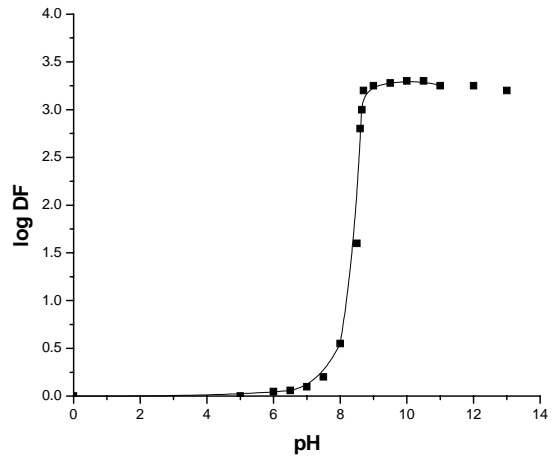


Fig. 3: Variation of DF with pH.

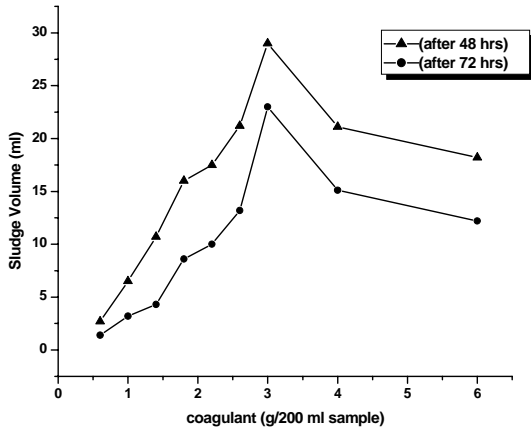


Fig. 4: Sludge volume as a function of coagulant concentration.

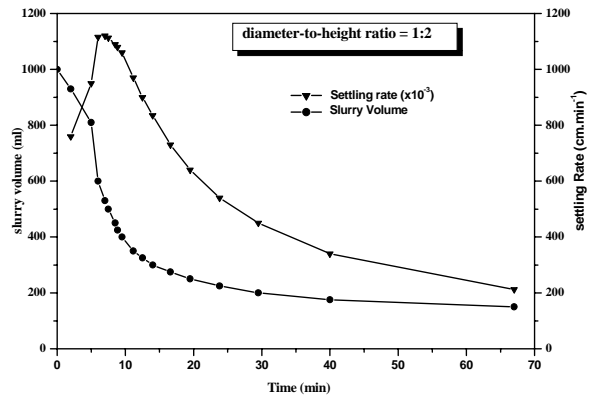


Fig. 5: Settling behavior of the sludge from 1.6g/L coagulant conc. at 1:2 diameter-to-height ratio.

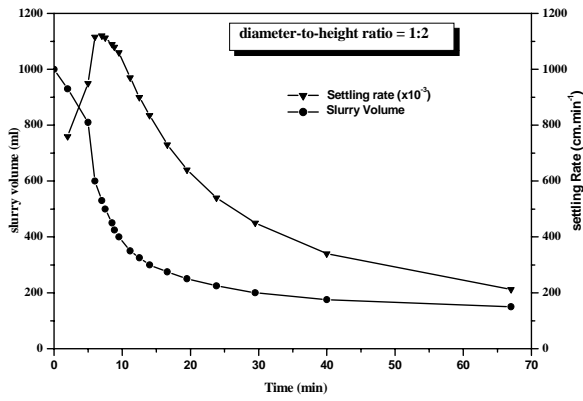


Fig. 6: Settling behavior of the sludge from 1.6g/L coagulant conc. at 1:2 diameter-to-height ratio.

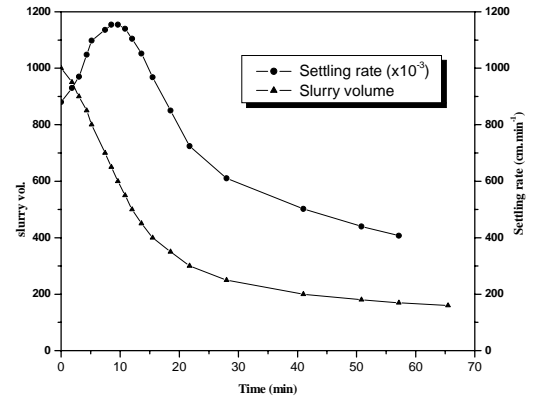


Fig. 7: Settling behaviour of the sludge from 1.6g/L coagulant conc. at 1:4 diameter-to-height ratio.

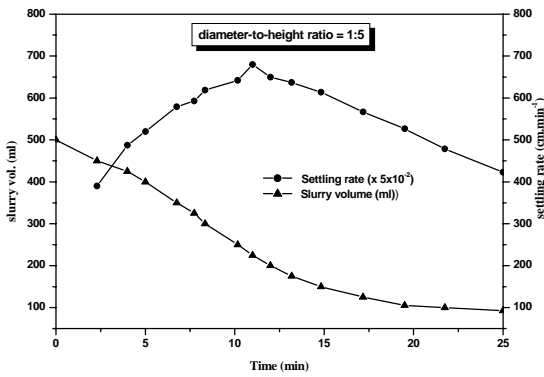


Fig. 8: Settling behavior of the sludge from 1.6g/L coagulant conc. at 1:5 diameter-to-height ratio.

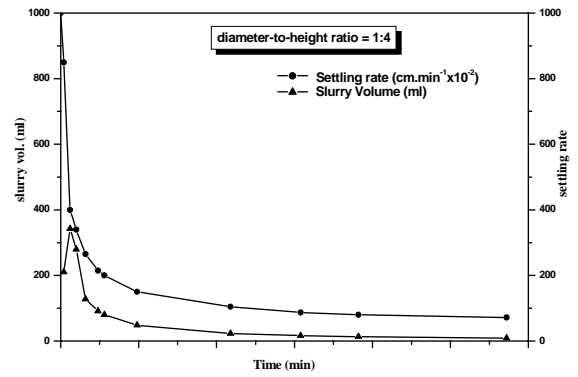


Fig. 9: Settling behavior of the sludge from 1.4g/L coagulant conc. at 1:4 diameter-to-height ratio.

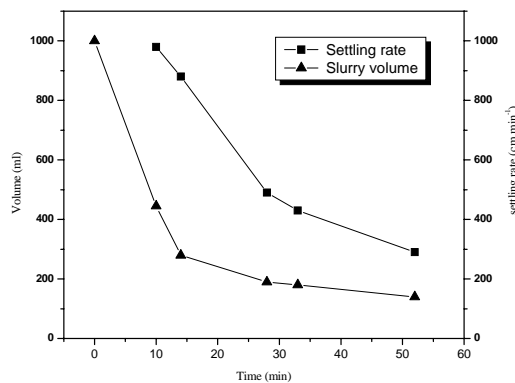


Fig. 10: Behavior of the sludge (of Fig.6) when vigorously shaken with supernatant after heavy compaction.