# STUDIES ON TREATMENT OF EFFLUENT GENERATED FROM LATEX CENTRIFUGING FACTORIES

Project Report
Submitted in partial fulfillment of the requirements for
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POLYMER TECHNOLOGY
by
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(वाणिज्य एवं उद्योग मंत्रालय, भारत सरकार)
(Ministry of Commerce and Industry - Government of India)
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# CERTIFICATE

This is to certify that the project work entitled **"STUDIES ON TREATMENT OF EFFLUENT GENERATED FROM LATEX CENTRIFUGING FACTORIES"** is a bonafide record of the research and development activities undertaken by **Shri.P.Arumugam**, II year M.Tech student in Polymer Technology of Cochin University of Science and Technology. The work was undertaken under my guidance at Processing and Quality Control Division, Rubber Board, Kottayam -9. He has developed a more cost effective treatment for waste water generated at a latex centrifuging factory.

M Sunny Sebastian
Jt.Director (P&QC)
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# CERTIFICATE

Certified that this is the bonafide report of the project work done by shri P. ARUMUGAM, II year M.Tech ( polymer Technology) during the Year 2005-06 in partial fulfillment of the award of degree of Master of Technology in Polymer Technology of the Cochin University of Science and Technology, Kochi – 682022.

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Finally I thank all the teaching members of Dept of PS & RT who took interest on this work and suggested necessary modification.

**P. ARUMUGAM**

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**“ CONCENTRATE ON PREVENTION OF ENVIRONMENTAL EMERGENCIES. DIFFERENCE BETWEEN LIFE AND DEATH IS OF FEW SECONDS. THEREFORE PLEASE DONATE GENEROUSLY FOR PREVENTION OF NATURAL DISASTERS. IT IS BETTER TO THINK AHEAD AS HUMAN LIFE IS VERY PRECIOUS.”**

# CHAPTER 1

## 1. INTRODUCTION

### 1.1 General introduction of wastewater:

Every Community produces both liquid and solid wastes and air emissions. The liquid waste, wastewater is essentially the water supply of the community after it has been used in a variety of applications. From the standpoint of sources of generation, waste water may be defined as a combination of the liquid or water carried wastes removed from residences, institutions and commercial and industrial establishments, together with such groundwater, surface water and storm water as may be present.

When untreated wastewater accumulates and it is allowed to go septic, the decomposition of the organic matter it contains will lead to nuisance conditions including the production of malodorous gases. In addition, untreated wastewater contains numerous pathogenic microorganisms that dwell in the human intestinal tract. Wastewater also contains nutrients, which can stimulate the growth of aquatic plants, and may contain toxic compounds or compounds that potentially may be mutagenic or carcinogenic. For these reasons, the immediate and nuisance-free removal of wastewater from its sources of generation, followed by treatment, reuse or dispersal into the environment is necessary to protect public health and the environment.

To protect public health and the environment, it is necessary to have knowledge of

- Constituents of concern in waste water
- Impacts of these constituents when wastewater is dispersed into the environment.
- The transformation and long-term fate of these constituents in treatment processes.
- Treatment methods that can be used to remove or modify the constituents found in waste water.
- Methods for beneficial use or disposal of solids generated by the treatment systems.

## 1.1 Rubber plantation in India:

### 1.1.1 Brief history

Havea brasilinesis is the most important commercial source of natural rubber (NR). It is a native of the Amazon River basin of South America. It is one of the most recently domesticated crop species in the world and was introduced to tropical Asia through Kew gardens in the UK, with the seeds brought from Brazil by Sir Henry Wickham. The tree is now grown in the tropical regions of Asia, Africa, and America. Commercial Cultivation of NR was actually started during the 1870's when the British successfully transported Hevea seeds from Brazil for planting in the British India. The domestication history of Hevea was chronicled, analysed and reviewed by many. The growth of the Indian rubber plantation industry has been mainly through the expansion of rubber cultivation in Kerala. The geographical and agro-climatic suitability proved congenial for rubber cultivation in Kerala, Kanyakumari region (TN), Mangalore region (Karnataka), Northeast region, etc.

### 1.1.2 Natural Rubber:

Natural rubber (cis-1,4,polyisoprene), has been found in the latex of over 2000 species of plants belonging to 311 genera of 79 families. The minor sources of NR are manihot glaziovii ( Euphorbiaceae), Ficus elastica (Moraceae), Parthenium argentatum and Taraxacum koksaghyz ( compositae). In addition, many other species like Euphorbia intisy (Euphorbiaceae). Cryptotegia grandiflora, C.Madagascariensis (Ascleplodaceae),Funtumia elatica, Landolphias (Apocynaceae): etc. have been experimented as possible minor sources of natural rubber. However, Hevea braziliensis is the most important commercial source of natural rubber.

### 1.1.3 Hevea braziliensis:

The rubber tree is sturdy, quick growing and tall. It grows on many types of soils, provided they are deep and well drained. A warm humid equable climate (21°c to 35°c) and a fairly distributed annual rainfall of not less than 200mm are necessary for the optimum growth. It however grows successfully under slightly varying conditions also. Rubber trees have a well-developed taproot and laterals. The bark on tapping yields latex. The cambium in between the wood and the bark is responsible for the increase in girth of tree including bark renewal.

The young plants show characteristic growth pattern of alternating period of rapid elongation and consolidated development. The leaves are trifoliate with long stalks. Normal annual leaf fall of mature trees, known as wintering, occurs during the period of December to February in South India. Refoliation and flowering follow wintering. Some trees may occasionally show off seasonal flowering during September – October also. Pollination is by insects. The fruits mature in about five months after pollination. They are three seeded and burst when mature, scattering the seeds 15 to 18 metres.

### 1.1.4 Present status:

Since the introduction of the rubber tree in 1878, the rubber plantation sector in India has grown manifold in area of planting, rubber production and productivity. Table1.1 gives the details of growth in these areas during the current decade. India is the third largest producer and the fourth largest consumer of NR in the world.

**Table 1.1. Total area, trappable area, production, average yield per hectare and consumption of natural rubber**

| Year | Total area (ha) | Tapped area (ha) | Production (M.T) | Average yield per hectare (kg) | Consumption (M.T) |
|---|---|---|---|---|---|
| 1990 - 91 | 475083 | 306413 | 329615 | 1076 | 364310 |
| 1991 - 92 | 488514 | 324540 | 366745 | 1130 | 380150 |
| 1992 - 93 | 499374 | 330500 | 393490 | 1191 | 414105 |
| 1993 - 94 | 508420 | 38550 | 435160 | 1285 | 450480 |
| 1994 - 95 | 515547 | 346270 | 471815 | 1362 | 485850 |
| 1995 - 96 | 524075 | 356444 | 506910 | 1422 | 525465 |
| 1996 - 97 | 533246 | 365580 | 549425 | 1503 | 561765 |
| 1997 - 98 | 544534 | 376970 | 583830 | 1549 | 571820 |
| 1998 - 99 | 553041 | 387100 | 605045 | 1563 | 591545 |
| 1999 - 00 | 558584 | 394800 | 622265 | 1576 | 628110 |
| 2000 - 01 | 562670 | 399901 | 630405 | 1576 | 631475 |
| 2001 - 02 | 566555 | 400713 | 631400 | 1576 | 638210 |
| 2004 - 05 | -- | -- | 749665 | -- | 755405 |

Source: Rubber Board (2005)

## 1.2 Natural rubber processing in India

### 1.2.1 Marketable forms of natural rubber:

The crop from rubber tree is highly susceptible to degradation on keeping. Latex or the different field coagulum rubbers are not generally used as such for the manufacture of rubber goods. So the processing of the crop into forms that allow easy storage, transportation and utilization by manufacturing industries is a necessity. Latex can be processed into any of the following forms.

- Ribbed smoked sheet (or) air-dried sheet.
- Pale latex crepe and sole crepe
- Technically specified block rubbers and different types of speciality rubbers.
- Preserved field latex or latex concentrate.

Field coagulum rubbers are processed into any of the following forms.

- Estate brown crepes.
- Technically specified block rubbers.

### 1.2.2 Ribbed smoked sheets or air dried sheets:

Raw rubber sheets are of various types like ribbed smoked sheets (RSS), air dried sheets (ADS) and sun dried sheets, depending on the method adopted for drying. The operations involved in making RSS and ADS include sieving, bulking and standardization of latex, addition of chemicals, coagulation, sheeting, dripping and drying.

### 1. Sieving and bulking:

Latex reaching the factory should be sieved through 40 and 60 mesh sieves to remove suspended impurities. Latex from different fields is blended in bulking tanks, which may be of aluminium or brickwork construction, lined inside with porcelain tiles.

### 2 .Standardization of latex:

The bulked latex is to be diluted to a standard DRC of 12.5 percent before it is coagulated. Dilution helps in settling denser and finer impurities at a faster rate and is obtaining a softer coagulum, which can be easily sheeted.

### 3. Addition of chemicals:

#### i. Sodium bisulphite:

Enzymes of the polyphenol oxidise type catalyse the oxidation of phenols and amino-phenols, naturally present in latex, by oxygen from the air to give ortho-quinones. Ortho-quinones react with naturally occurring amino acids and proteins present in latex to give coloured products resembling melanin. Reduced melanin has brown colour and oxidized melanin black colour. Addition of sodium bisulphite to latex prevents this discoloration by preferential reaction with oxygen, getting it converted into sodium bisulphate in the process.

#### ii. Para Nitro Phenol:

Another chemical, which is added to latex at this stage, is Para Nitro Phenol (PNP) and it prevents mould growth on the surface of the dried sheets.

### 4. Coagulation:

Coagulation is the process of destabilization of latex by some means with a view to recovering rubber from it. There are several methods of coagulation like coagulation by addition of chemicals, natural coagulation and assisted biological coagulation.

### 5. Sheeting and dripping:

After draining out the serum, the coagulum is washed with water before sheeting. The sheeting rollers consists of a pair of plane rollers and another set of grooved ones. The rollers usually made of cast iron, are of length 60cm and diameter 15cm. On the grooved rollers, grooves are cut at 3mm intervals with 3mm width and depth and at $45^0$ spirals. For washing, provision is made for continuous flow of water through a line of holes drilled at the lower side of a pipe fitted over the rollers. It is essential that, while sheeting sufficient quantity of waterfalls over the coagulum to remove the serum residues and excess chemicals retained. The wet sheets are allowed to drip in shade for 1-2 hrs before they are transferred to smoke houses for producing RSS or to hot chambers for making air-dried sheets.

### 6. Drying:

Freshly machined and dripped sheets contain about 20 percent moisture. For the preparation of RSS, these sheets are dried in smoke houses, while for the production of ADS, smokeless drying chambers are used.

### 1.3.3 Crepe Rubbers

When coagulation from latex or any form of field coagulum or RSS cuttings after necessary preliminary treatments is passed through a set of creping machines, crinkly, lace like rubber is obtained. This when dried is called crepe rubber. Pale latex crepe and sole crepe are processed from field latex. The other forms of crepe rubbers are processed from field coagulum rubbers or RSS cuttings. Pale latex crepes are mainly used for the production of surgical and pharmaceutical articles, light coloured and transparent goods, adhesives and tapes, derivatives like chlorinated rubber, etc. Sole crepe is used for the production of translucent shoe soling material. The other forms of crepes are used for the manufacture of tyres, foot- wears, re-treading materials, etc.

Processes involved in the production of latex crepes include sieving, bulking and standardization of latex, addition of chemicals, coagulation, creping of coagulum and drying.

### 1.3.4 Technically specified Block Rubbers:

#### i. Processing of latex:

#### 1. Preservation:

For centralized processing, latex may have to be brought from long distances. Ammoniation of latex is necessary in such cases and usually the level of ammoniation is kept at 0.07-0.10 percent on latex to ensure adequate preservation.

#### 2. Reception and bulking:

Latex on arrival at the factory is strained into the bulking tank using a 40-mesh sieve. Sodium bisulphite at the rate of 1 g/kg DRC rubber is added as 1% solution to the bulking tank to prevent surface darkening of the coagulum.

#### 3. Coagulation:

Latex is then coagulated either in conventional aluminium tanks or coagulation troughs at field DRC using either formic or acetic acid.

#### 4. Pre- machining:

In the case of latex coagulated in conventional aluminium tanks, the individual slabs of coagula are cut into smaller pieces before feeding to an extruder or one or two passes are given through a creeper before feeding to a hammer mill for final size reduction.

#### ii. Processing of field coagulum:

#### 1. Reception and storage:

Field coagulum such as cup lumps, tree lace, shell scarp and pre-coagulated lumps require reception, storage and pre-cutting for easy handling prior to pre-cleaning, blending and final size reduction. The best method of storing is to keep the material dry under shade.

### 2. Pre- cleaning:

Field coagulum materials usually arriving at the factory from estate sources are fresh, clean and fairly discrete pieces or lumps. Dry and dirty scrap requires soaking in water for about 24 hrs, before pre cleaning can be effectively carried out by repeated passes through a macerator and a pre- cleaning hammer mill.

### 3. Blending:

The blending of rubber from different sources can be achieved by adopting the following procedure.

i. By evenly distributing the different lots of rubber on arrival at the factory into several receptacles.

ii By recycling a portion of the crumbs.

iii By multiple and overlapping passes of crepes through macerators.

### 4. Initial Processing:

Initial processing of the blended materials is required mainly to impart a good shearing to the rubber particles, so that it can be dried easily by hot air convection drier. Shearing is done by creping the material by using creepers/ macerators.

### 5. Final size reduction:

Final size reduction largely depends on the type of rubber produced. The hammer mill is used for final size reduction.

### 6. Drying:

Drying is done in oil fired or electrically heated or gasifier driers. For light coloured latex rubbers, the temperature of drying is normally kept around 100°C– 105°C whereas for scrap rubber it is around 105°C – 110°C. The normal drying time is 4 – 5 hrs.

### 1.3.5. Preserved Latex Concentrate:

The process of latex concentration involves the removal of a substantial quantity of serum from field latex, thus making latex richer in rubber content. Concentration of latex is necessary because of the following four reasons.

i. Economy in transportation.

ii. Preference for high DRC by the consuming industry.

iii. Better uniformity in quality.

iv. Higher degree of purity.

Various processes have been proposed for concentrating latex. Out of these, four have emerged as of special importance. These are evaporation, creaming, centrifuging and electro-decantation. Evaporation method involves the removal of water only. Hence the ratio of non-rubber constituents to rubber remains unaffected by this concentration process. The particle size distribution also is substantially unaffected. On the other hand, the other three processes involve the partial removal of non-rubber constituents and small rubber particles. Because of this, the range of particle size is reduced and a higher degree of purity is obtained.

### 1.3.6. Creaming:

#### 1. Principle of creaming:

Rubber latex is a dispersion of very fine particles of rubber in serum. Like any other dispersion or suspension, the particles have an inherent tendency to sediment under the influence of gravity. Rubber particles being lighter than serum tend to cream up. The velocity of creaming, can be deduced approximately from Stokes law, which can be stated mathematically as follows.

$$V = \frac{2g\,(Ds-Dr)\,r^2}{9\eta}$$

Whereas, V – velocity with which the particles rise (cm/sec)

g - Gravitational force (cm/sec$^2$)

Ds – Density of serum ( g/ml)

Dr - Density of rubber particle (g/ml)

r - Effective radius of the rubber particle (cm)

$\eta$ - Viscosity of the serum (poise)

## 2. The creaming process:

Latex is collected and ammoniated to 1 % and preferably kept for a few days for aging. The creaming agent solution at 3% concentration is prepared by boiling the required quantity of the material with water. The preserved and de-sludged latex is taken in the creaming tank. Calculated quantity of the creaming agent solution, which has been sieved to remove uncooked materials, is then added. A 10 % solution of soap is added so as to make its concentration 0.3% on latex. The latex is then thoroughly stirred for about 1 hr. as the creaming agent solution is viscous, it does not readily mix with latex unless broken up by agitation. The complete dissolution of creaming agent and the through mixing of the solution in latex are very important. After stirring, latex is allowed to remain undisturbed till the desired level of creaming is obtained.

Although a minimum period of 48 hrs is usually required for satisfactory creaming fixed time can be set for all conditions. After the creaming agent is added there is an induction period of several hours before any creaming is visible. For the first 24 – 40 hrs, creaming is rather rapid and then it becomes slower. When the desired level of separation is obtained, the skim layer is drained off. The DRC and ammonia content of the concentrate are checked and adjusted to standard levels. Several batches of cream are bulked together packed and dispatched.

### 1.3.7 Centrifuging:

Although Biffin observed in 1898 that latex could be concentrated by centrifuging no commercial development of the process took place until about 1923 when Utermark apparently rediscovered the process. The process was further developed by Dunlop Rubber Company on a large scale in the Far East. Now this is the most widely used method for the concentration of latex. Over 90% of the concentrated latex is produced by centrifuging

#### 1. Principle of centrifuging:

The theory of centrifugal concentration of latex is basically the same as that of creaming. In centrifuging, centrifugal force replaces gravitational force, which brings about separation of rubber particles in creaming. Latex is subjected to strong centrifugal force, several thousand times the gravitational force, in a bowl rotating at high speeds where by the individual rubber particles tend to separate into a layer surrounding the axis of rotation leaving an outer serum layer having a comparatively low rubber content. Each layer is removed through annular spacing around the axis of rotation. By controlling the time to which latex is subjected to such a forces and by controlling the conditions of operation, latex having an original DRC of 30- 38 % can be concentrated to DRC of 60 % or more. Usually the speed of the machine is fixed, so that the variables for the purpose of control are.

i The rate of passage of the latex through the machine

ii Certain mechanical adjustments on the machine.

It should also be noted that with increasing temperatures up to 50°C an increasing proportion of the total rubber is obtained in the concentrate.

#### 2. Centrifuging Process:

Several types of centrifuging machines are used for the concentration of latex. Of these, the most widely used type is the Alfa Laval. The rotating mass of latex is broken up into a number of thin conical shells within the bowl, which rotates at speeds around 6000 rpm. The maximum distance, which a particle has to traverse in order to pass from the skim to the cream, is very small.

The degree of ammoniation of the latex prior to centrifuging depends upon the period, which has elapsed since collection and ranges from 0.25%; if centrifuging is immediate, to 0.8%, if the period is two days or more. Only the minimum required quantity of ammonia should be added. Most of the ammonia that is added to the field latex goes in the skim, which makes the coagulation of skim more difficult. Moreover, if the concentration of ammonia in latex is more, larger quantity escapes into the atmosphere causing discomfort to workers. Usually latex is to be ammoniated and kept overnight before being centrifuged, thus giving time for the sludge to settle down. Latex is fed to the centrifuge from a constant head device so as to ensure a constant flow through the machine. It enters the bowl through a central feed tube and then passes through a distributor to the bottom of the bowl. Where it flows through a series of holes into the interior of the bowl. The cream centrifuges inwards towards the axis of rotation and the empties from the bowl through holes into a stationary gully. The skim flows outwards away from the axis of rotation and leaves the bowl through orifices, which are formed by regulating screws. It passes out of the centrifuge through a second stationary gully. The parts of the machine, which come into contact with latex, should be constructed preferably out of stainless steel for avoiding contamination and preventing corrosion. The cream is separately collected in a bulking tank, its ammonia content estimated and made up to 0.7% on latex and packed in drums

### 1.3.8 Skim Latex:

When field latex is centrifuged/ creamed in addition to the concentrate containing most of the rubber, an equal fraction in volume, containing a very small proportion of rubber is also obtained. This is known as **skim latex**. As the normal efficiency of centrifuging process is only 85-93% about 3 to 6 % of the incoming rubber goes in the skim. Hence on economic grounds, it is essential to recover the rubber in the skim latex.

### 1.3.9 Composition of skim latex:

The important factors governing the composition of skim latex produced are the type of centrifuge, DRC of field latex through output and efficiency of operation. Normally the rubber content of skim latex lies between 3.5 to 6 %. It contains about two thirds of the total serum from the field latex. The average size of its rubber particles is smaller than that of the field latex. Since the larger particles separate more readily into the concentrate fraction. In addition to the water-soluble substances in serum, latex contains proteins, which are mainly distributed as absorbed film on the surface of the rubber particles and as the particles are small the protein content per unit weight of rubber is more in the case of skim latex. This is not only renders coagulation more difficult, but also has a marked effect on the properties of the rubber. Skim also contains ammonia, which adds to the cost and difficulty of acid coagulation. With increasing efficiency in concentration, the DRC of the skim fraction decreases and the difficulty and cost of any recovery process increase.

## 1.4. Treatment of Effluent from Latex Concentrate factories:

In 2005, India produced about 749665MT of natural rubber of which 15% was in the form of latex concentrate produced by centrifugation and creaming. A survey on the chemical and physical properties of various types of effluent discharged from rubber/latex factories showed that effluent from latex concentrate factories was the most polluting. High ammoniation of field latex and subsequent auto and or acid coagulation of skim latex (a by product of latex concentrate manufacture) occurring during the processing of Latex concentrate contribute towards the high pollutant effluent. The coagulation of skim latex produces a serum that contains a much higher concentration of proteins, sugars, lipids and inorganic and organic salts compared to serum obtained by the acid coagulation of field latex in ISNR.

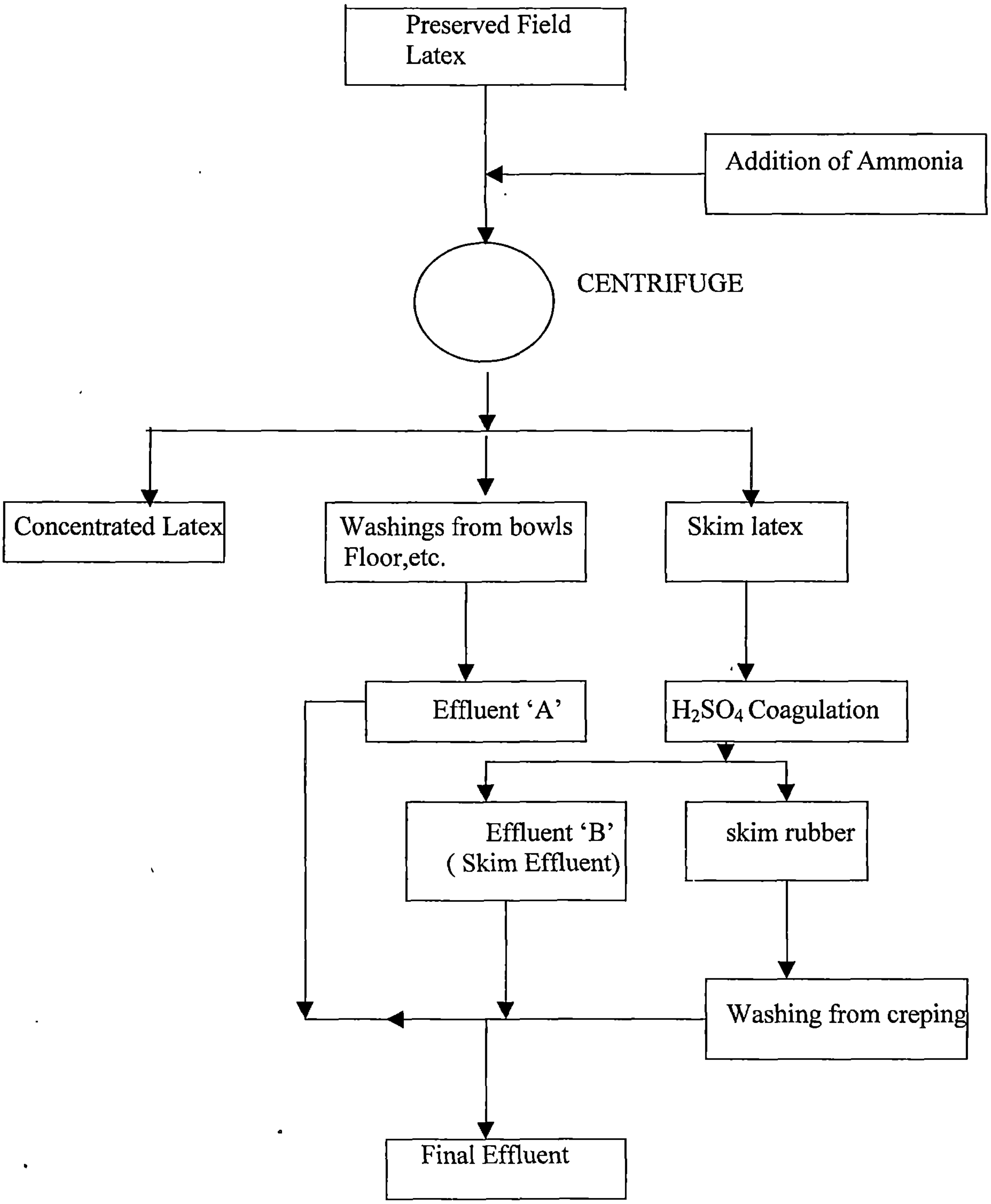
The added presence of a significant number of viable and indicative bacteria in the skim serum makes it a high oxygen absorbing liquor, necessitating its treatment prior to discharge into public water ways.

### 1.4. 1 Treatment and disposal of Effluents:

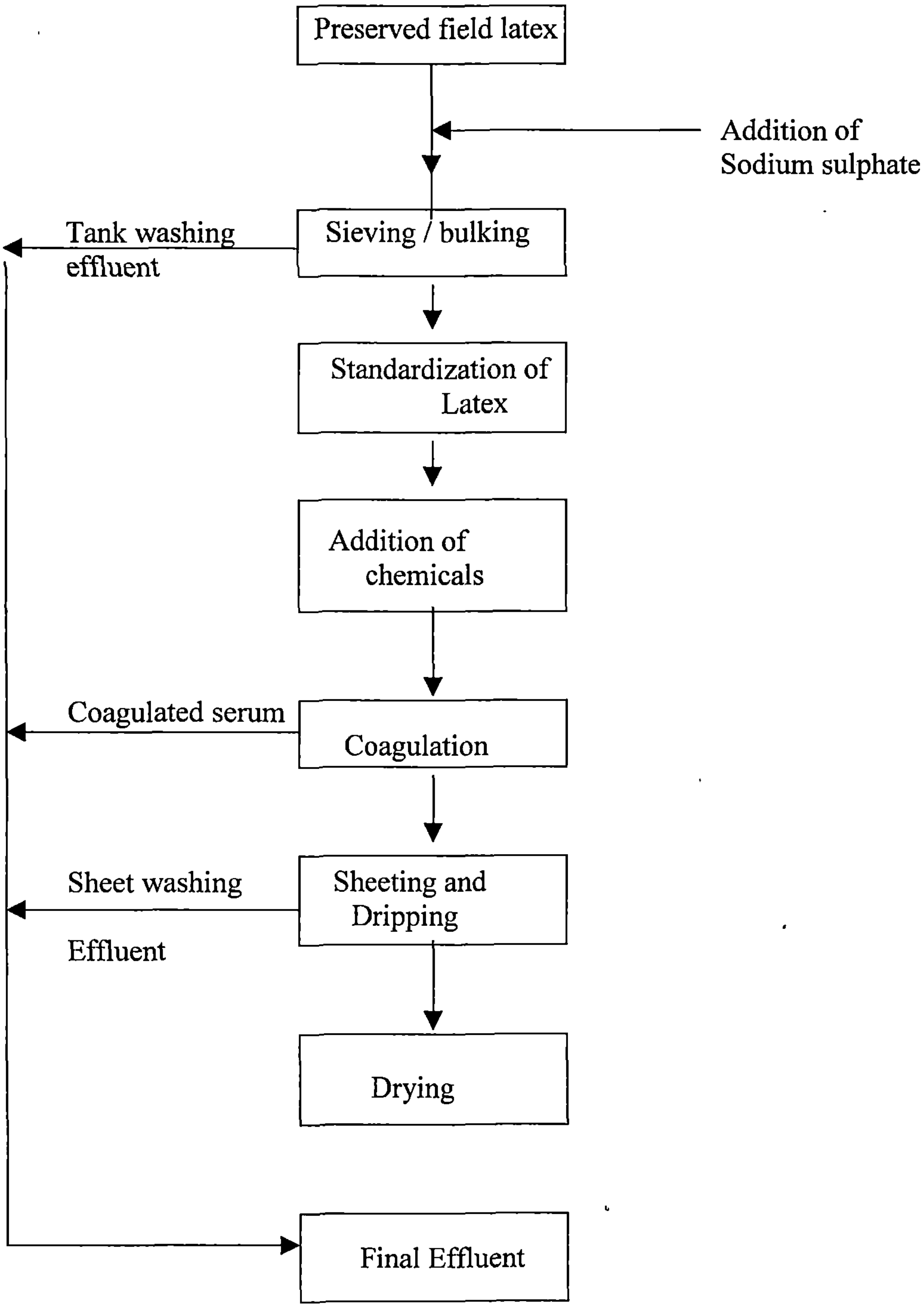
During the processing of the field latex, large quantity of water (approximately 20 l/kg DRC) is used for washing, cleaning and for dilution of latex. The serum obtained after coagulation and the water used for washing and cleaning during processing constitute the effluent, which is generally discharged into Public waterways. This effluent contains small quantity of rubber particles, varying quantities of proteins, sugars, lipids, carotenoids and inorganic and organic salts. The presence of these materials increases the bio chemical oxygen demand (BOD), chemical oxygen demand (COD), dissolved and suspended solids and nitrogen content of the receiving waterways. BOD measures the quantity of oxygen utilized by a mixed population of microorganisms in the aerobic oxidation. COD test on the other hand, measures the quantity of oxygen required for chemical oxidation of organic matter in the sample to carbon dioxide and water. Thus, high BOD and COD of effluents tend to deplete dissolved oxygen in water and endanger aquatic life. A large population of bacteria is also present in the effluents discharged from the factories.

## CHAPTER - 2

### 2.1. SOURCES OF EFFLUENTS IN LATEX CONCENTRATE FACTORY:

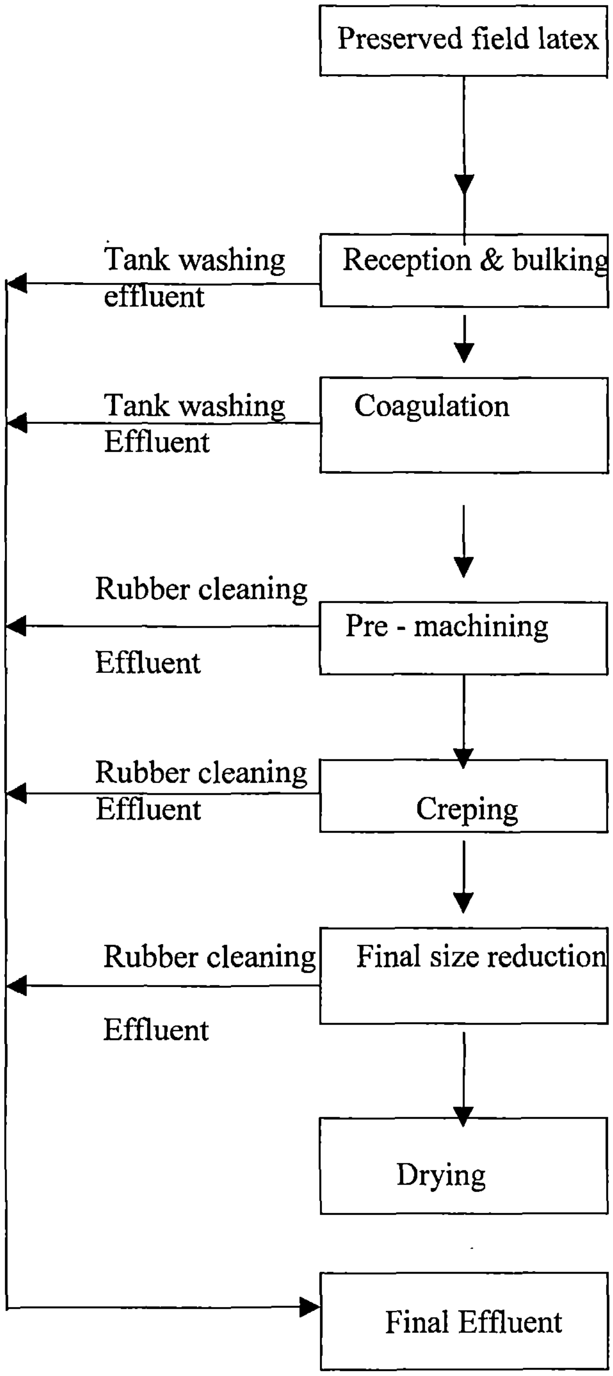


## 2.1 SOURCES OF EFFLUENT IN SHEET FACTORY:



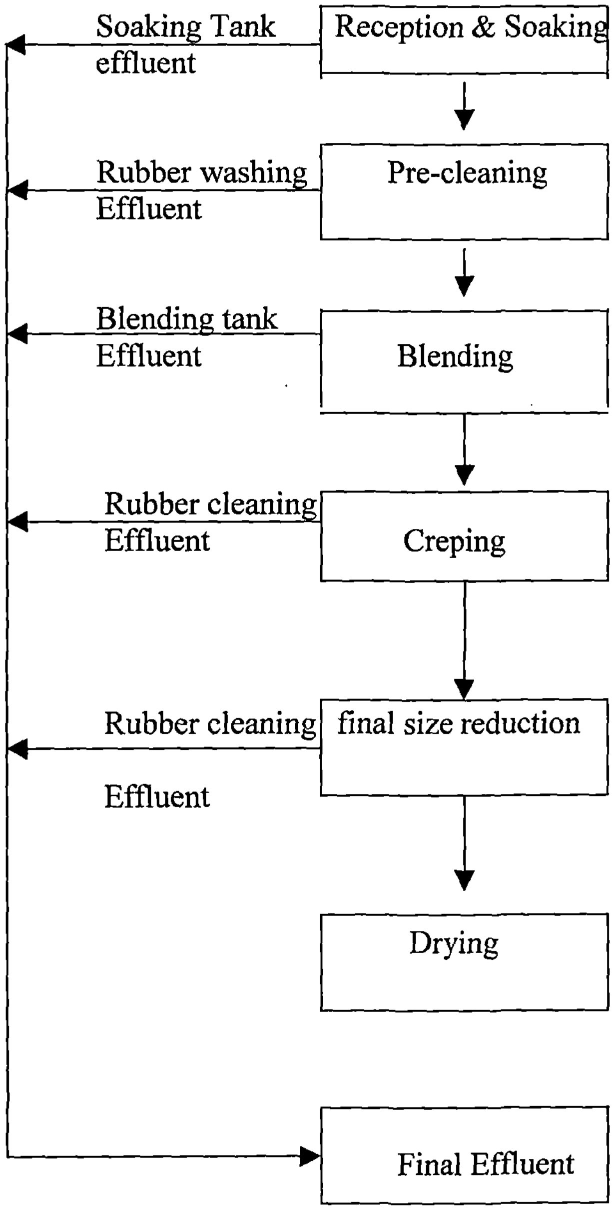
## 2.2. SOURCES OF EFFLUENT IN TECHNICALLY SPECIFIED BLOCK RUBBER FACTORY

### i) PROCESSING OF LATEX



## 2.2. SOURCES OF EFFLUENT IN TECHNICALLY SPECIFIED BLOCK RUBBER FACTORY:

### ii) PROCESSING OF FIELD COAGULATION



### 2.3 Composition of Rubber factory effluents:

The variations of pH of effluent from different types of factories are mainly due to the different in quantity of acid used. The high total solid content, mainly dissolved organic solids are responsible for high oxygen demand. Another important characteristics of rubber effluent are the significant level of nitrogen mainly in ammoniacal form derived from the preservative used. Therefore, the two potential pollutants in the effluents are organic carbon and ammonical nitrogen.

### 2.3.1 Effluent treatment:

Many methods of effluent treatment have been described. These are derived into three basic types.

i) Physical treatment

ii) Chemical treatment

iii) Biological treatment.

#### i). Physical treatment:

In physical methods, the impurities are separated from effluent without any chemical change. The important methods are filtration, sedimentation and reverse osmosis. In filtration, the effluent is allowed to pass through a filter bed. In sedimentation, the force of gravity act on particles heavier than water and causes them to settle whereas reverse osmosis is a membraneous process where concentration of dissolved substances tends to increase on one side of the membrane.

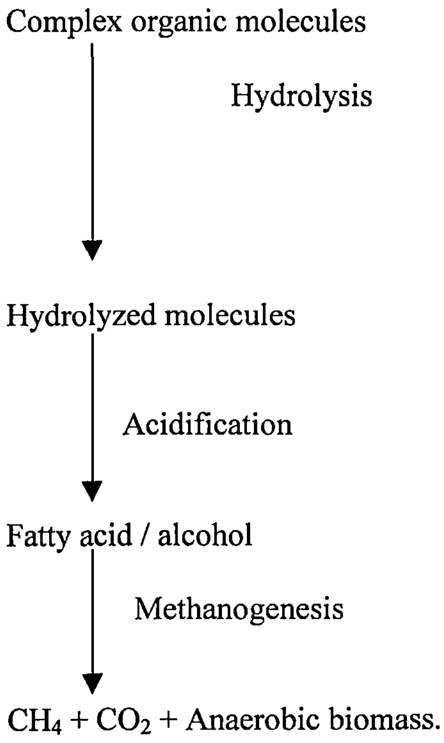
#### ii) Chemical treatment:

In chemical treatment, the impurities in effluents are separated by chemical means. The important processes are precipitation, flocculation, ion- exchange, and oxidation and reduction reactions.

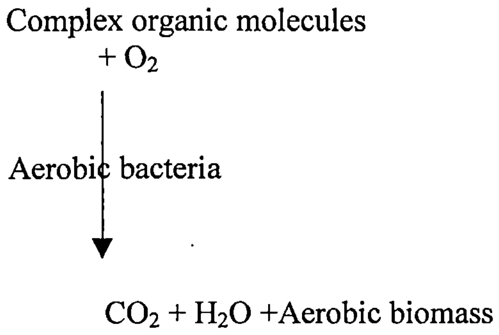
### iii) Biological treatment:

In the biological methods, the principle of breakdown of organic matter by microorganisms is made use of when the effluent contains sufficient dissolved oxygen, complex organic compounds break down into simple compounds such as carbon dioxide and water by the action of aerobic bacteria and the process is described as **Aerobic**. In the absence of dissolved oxygen, anaerobic bacteria bring about this reaction and the process is known as **Anaerobic.**

**Anaerobic**



**Aerobic**



### 2.3.2 Anaerobic Vs Aerobic treatment:

The following table gives a comparative statement of the salient features of both aerobic and anaerobic treatments.

| | Aerobic systems | Anaerobic systems |
|---|---|---|
| Treatment time | Short | Long |
| COD removal | 90-98 % | 70-75% |
| Energy consumption | high | low |
| Energy production | no | yes |
| Bio solids production | high | low |
| Nutrients (N/P) removal | high | low |
| Space requirement | high | low |
| Discontinuous operation | difficult | easy |
| Capacity required | less | high |

### 2.3.3 Anaerobic treatment system:

Anaerobic treatment is characterized by biological conversion of organic compounds (COD) into biogas mainly methane 70-85 volume percentage and carbon dioxide 15- 30 volume percentage with traces of hydrogen sulphide.

In the absence of oxygen many different groups of anionic bacteria "work" together to degrade complex organic pollutants into methane and carbon dioxide (biogas). The microbiology is more complex and delicate than in case of aerobic process, where most bacteria "work" individually.

**Anaerobic Reactions:**

$$\textbf{COD} \longrightarrow \textbf{CH}_4 + \textbf{CO}_2 + \textbf{Anaerobic biomass}$$

For effective biological treatment of industrial effluent a high biological treatment capacity is required. The capacity of biological treatment systems is determined by

- Biomass amount (concentration, volume etc)
- Biomass activity of the biomass.

### 2.3.4 Aerobic treatment system:

Aerobic processes use bacteria that need oxygen to live. During aerobic treatment (air) oxygen is supplied to oxidize the COD into carbon dioxide and water. Aerobic biological process produce new biological mass (bio solids).

**Aerobic reaction:**

$$\textbf{COD} + \textbf{O}_2 \longrightarrow \textbf{CO}_2 + \textbf{H}_2\textbf{O} + \textbf{Aerobic biomass.}$$

Aerobic breakdown depends on adequate supply of oxygen. In aerobic pond, the supply of oxygen is obtained from the photosynthetic activities of algae cells. The conditions required for the growth and survival of the algae and bacterial cells are tolerable pH range, sunlight and adequate availability of carbon, nitrogen and phosphorous. In the oxidation ditch system; supply of oxygen is achieved by mechanical aeration.

### 2.3.5 Activated sludge process:

It is the most frequent and widely applied aerobic technology for the treatment of industrial effluent. The technology is based on an aerated reactor with suspended flocculent aerobic sludge, mixed by aerators supplying the necessary oxygen. Besides compressed air or oxygen, mechanical aerators spraying the water into the atmosphere can be used for oxygen supply. An external gravity clarifier for sludge separation subsequently follows the aerated basins. The decanted aerobically purified effluent is discharged, while the settled aerobic sludge is returned to the aeration basin. Excess aerobic sludge is often dewatered and put to land fill. Use of activated sludge systems allows for effluent discharge to rivers and lakes.

### 2.3.6 Anaerobic treatment:

The digestion of organic matter is carried out in the absence of air in a deep tank. In this process, anaerobic bacteria convert organic solids to methane, carbon dioxide, hydrogen sulphide, ammonia; etc. for smooth reaction the pH must be controlled to near neutral. The degradation of the organic matter takes place in two stages. The first stage involves the acid forming bacteria which hydrolysis the waste into shorter chain organic compounds, especially organic alcohols, acids and ammonia. In the second stage, methanogenic bacteria convert these intermediate products into gases. Biogas plants are based on the anaerobic technology, which serve dual purpose of reducing environmental pollution and generating energy.

Many methods of effluent treatment are being practiced in the rubber processing industries. The treatment can be divided into primary, secondary and tertiary stages. The purpose of primary treatment is to recover small pieces of rubber and to remove materials, which hinder subsequent biochemical reactions. Secondary treatments remove the dissolved organic matter and thereby reduce BOD. All the secondary treatments are mainly biological processes and bacteria are the major organisms that carry out oxidation. They consume the dissolved organic matter as food and thus remove it from the effluent.

The effectiveness of treatment depends on the bacterial population, pH, temperature and nutrients. It is highly affected by extremes in pH and the presence of toxic chemicals like ammonia, hydrogen sulphide etc. The common biological treatment is activated by sludge process, oxidation ditch, oxidation pond and anaerobic digestion.

Tertiary treatment is applied to the waste for the removal of dissolved inorganic solids and bacteria and sometimes for the removal of final traces of organics. The treated effluent is detained in the settling tanks to allow the suspended solids and sludge to settle. Theses tanks are usually constructed using bricks, with a conical bottom for easy settling and removal of sludge. Water from the settling tank is filtered in sand, filters to obtain clear water. With the promulgation of water (prevention and control of pollution) Act 1974, the industries are required to treat wastes to the degree as fixed by the pollution control board, before discharging them into any water body or disposing them on land.

## 2.4 PCB regulations on environment protection:

The types of bacteria found in rubber effluent are coliform, srtreptococci and E-coli. Most of the constituents of effluents can act as substrates for the growth of these microorganisms, thus generating a high BOD and an objectionable order. Therefore, indiscriminate discharge of effluents to natural streams and rivers is objectionable. Govt. of India have enacted pollution control act in 1974 and most of the State Governments have already taken steps to implement the provision of this act. So all the rubber processing factories in the country are now legally compelled to build up facilities to ensure that effluents let out to public out ways conform to specifications prescribed by the water pollution and prevention control authorities. The specification parameters and their limits for the treated effluent are shown in table 2.1.

| S.No | Parameter | Unit | Limit | |
|---|---|---|---|---|
| | | | Inland surface water | On land irrigation |
| **Centrifuging and creaming units** | | | | |
| 1 | pH | | 6 to 8 | 6 to 8 |
| 2 | B.O.D (3 days at 27o c) | mg/l max | 50 | 100 |
| 3 | C.O.D | mg/l max | 250 | *** |
| 4 | Oil & Grease | mg/l max | 10 | 20 |
| 5 | Total kjeldhal nitrogen (as N) | mg/l max | 100 | *** |
| 6 | Ammonical nitrogen ( as N) | mg/l max | 50 | *** |
| 7 | Sulphide ( as S) | mg/l max | 2 | *** |
| 8 | Total Dissolved Solids (TDS) | mg/l max | 2100 | NP** |
| 9 | Suspended Solids (SS) | mg/l max | 100 | 200 |
| **Crepe & crumb units** | | | | |
| 1 | pH | | 6 to 8 | 6 to 8 |
| 2 | B.O.D (3 days at 27o c) | mg/l max | 30 | 100 |
| 3 | C.O.D | mg/l max | 250 | *** |
| 4 | Oil & Grease | mg/l max | 10 | 20 |
| 5 | Total kjeldhal nitrogen (as N) | mg/l max | 50 | *** |
| 6 | Ammonical nitrogen ( as N) | mg/l max | 25 | *** |
| 7 | Sulphide ( as S) | mg/l max | 2 | *** |
| 8 | Total Dissolved Solids (TDS) | mg/l max | 2100 | NP** |
| 9 | Suspended Solids (SS) | mg/l max | 100 | 200 |

Waste water generation m3/t of rubber max

** Not prescribed in case effluent is used for rubber plantation of their own.

In other cases suitable limit, as may be prescribed by state board.

*** Not specified.

**Rubber ( synthetic)**

| S.no: | parameter | Unit | Inland surface water |
|---|---|---|---|
| 1 | pH | | 5.5 to 9 |
| 2 | BOD (3 days at 27o C) | mg/l max | 50 |
| 3 | COD | mg/l max | 250 |
| 4 | Oil & Grease | mg/l max | 10 |
| 5 | Color | mg/l max | Absent |

## 2.5 Biological effluent treatment:

### 1. Objectives of biological treatment:

The overall objectives of the biological treatment of domestic wastewater are to

- Transform (i.e., oxidize) dissolved and particulate biodegradable constituents into acceptable end products.
- Capture and incorporate suspended and non settlelable colloidal solids into biological floc or bio film.
- Transform or remove nutrients; such as nitrogen and phosphorous.
- In some cases, remove specific trace organic constituents and compounds.

The objective is to remove or reduce the concentration of organic and inorganic compounds from industrial wastewater. Because some of the constituents and compounds found in the industrial wastewater are toxic to microorganisms. Pre treatment may be required before the industrial wastewater can be discharged to a public out ways.

## 2. Role of microorganisms in Effluent treatment:

The removal of dissolved and particulate carbonaceous BOD and the stabilization of organic matter found in effluent is accomplished biologically using a variety of microorganisms, principally bacteria. Microorganisms are used to oxidize (i.e., convert) the dissolved and particulate carbonaceous organic matter into simple end products and additional biomass, as represented by the following equation for the aerobic biological oxidation of organic matter.

$$\textbf{(organic matter)}\ \nu_1 + \nu_2\,\mathrm{O_2} + \nu_3\,\mathrm{NH_3} + \nu_4\,\mathrm{PO_4^{3-}} \xrightarrow{\text{micro organism}} \nu_5\,\textbf{( new cells)}$$

$$+ \nu_6\,\mathrm{CO_2} + \nu_7\mathrm{H_2O}$$

Where, $\nu_i$ ---- the stoichiometric co- efficient.

Oxygen, ammonia and phosphate are used to represent the nutrients needed for the conversion of the organic matter to simple end products (i.e., $CO_2$ and water)

## 2.6 Process Outline for Aerobic Biological Treatment:

The raw effluent discharged from various sections of the factory is first passed through the rubber traps to remove the floating rubber particles. From the rubber trap the effluent is send to the equalization tank, to keep the characteristics of the combined effluent uniform. From the equalization tank the effluent is pumped at a uniform rate to the mixing box provided at the end of the mixing channel, where reagent solutions at adequate dosage are added to get the same thoroughly mixed with the effluent. This results in the coagulation of suspended particles and precipitation of a major portion of the dissolved organics and colour bearing materials. While the effluent is flowing through the mixing channel, flocculation of the coagulated / precipitated particles takes place. This slurry carrying the flocs is then allowed to enter the primary settling tank where the flocs are allowed to settle. By this process, more or less complete removal of colour and suspended particles as well as 50 % reduction in BOD and COD are obtained. The sludge settled at bottom of the settling tank is periodically drained to the sludge drying beds for further dewatering and drying. The dried cakes can be periodically removed from the drying beds used for land filling/soil conditioning.

The clear liquid overflowing from the primary settling tank is then send to the aeration tank and subjected to activated sludge process in order to biochemically oxidize the dissolved organics. Nutrients in the form of DAP and Urea are added for perfect biosynthesis. An MLSS of 4000 mg/l is to be maintained in the aeration tank to provide enough bacterial growth. The clear overflow from the aeration tank is to sent to the secondary settling tank and allowed to settle.

The over flow from the secondary settling tank is the treated effluent and meets the standard stipulated by KSPCB for discharge into river/ irrigation. A portion of the sludge settling at the bottom of the hopper bottom tank is returned to the aeration tank to maintain the required MLSS. Excess sludge from the secondary settling tank is drained to the sludge drying beds and dried along with the primary sludge.

### 2.6.1. Unit sizes, Specifications and functions of Equipments:

#### 1. Rubber trap

Size : 20M X 1.4M X 1M

The function of rubber trap is to remove the floating rubber particles.

#### 2. Equalisation Tanks

No.off : 2

Size of each tank : 7.7M X 3.25M X 2M

The function of equalization tanks is to collect the effluent from different sections and to keep the characteristics of the combined effluent uniform

#### 3. Effluent Transfer Pumps

Nos off : 1 + 1 standby

Motor HP : 1 HP

Type : Semi-open impeller, self priming
Non-clogging, centrifugal

Materials of Construction : C I

Make : Stork/ Kirloskar

Effluent transfer pump is provided to pump the effluent to the mixing box. A re-circulation loop is provided to control excess effluent flow to the mixing channel than the desired rate.

## 4. Mixing channel

No. of channels : 4

Size of each Channel : 7M X 0.25M X 0.25M

The combined effluent from the equalization tank is pumped at a uniform rate into the mixing box of the mixing channel. Here 10 % lime solution is dosed to raise the pH of the effluent to the range of 9.5 to 10.0. The lime solution is prepared in the lime-mixing tank provided with a mechanical stirrer. The dosage of lime required for raising the pH may vary time to time. The effluent mixed with lime is allowed to flow through the channels, in the mixing box. There are four channels provided of which the first two channels are baffled. The reagent and lime gets thoroughly mixed with the effluent in the baffled channels. The pH of the effluent after chemical dosage should be in the range of 8 –9 . This results in the precipitation of the suspended solids and a major portion of dissolved organic substances. The precipitated particles will form and grow in size as it flows through the last two channels of the mixing channels. The dosage of reagent is fixed according to the characteristics of the raw effluent and may vary from time to time.

## 5. Primary Settling Tank

Size : 7M X 3M X 2.5M

The slurry form the mixing channel is allowed to flow to the primary settling tank where the flocculated sludge is allowed to settle. By this 50 % reduction in BOD and COD are obtained.

The clear overflow from the tank is sent to an aeration tank and subjected to activate sludge process.

## 6. Aeration Tank:

Nos.off : 1

Size : 10M X 10M X3M

HP of aerator : 15 HP

Type :slow speed. Fixed type with
Reduction gearbox and motor

Operation : Continuous

MLSS required : 4000 mg/litre

The secondary treatment can be considered satisfactory, if the slurry in the aeration tank is coffee brown and the settlelability of suspended particles is fast, when kept in a measuring cylinder. The black colour of the effluent and poor settlelability of suspended particles show destabilization of the process. Healthy bacterial sludge will settle fast and occupy nearly ¼ of total volume of the liquid body in 30 minutes. If the volume occupied by the settled sludge is more than 1/3, the MLSS is more than 4000 mg/l. when such a situation arises, some sludge from the sludge re-circulation loop should be drained to the drying beds by opening the valve in the pipe line leading to the drying beds from the sludge re-circulation loop.

Re-circulation of Bio sludge :} 30% to 50% of the effluent

from secondary settling tank } flow rate.

**Nutrients Used**

The quantity of nutrients required when $20M^3$ / Day of effluent is handled is,

1) Urea : 6 kg

2) Factamphose : 3 kg

100 litres solution of DAP and Urea are prepared in separate drums fitted with outlet valves. The flow rates are set in such a manner to flow over a 24-hour duration into the overflow channel of the primary settling tank.

## 7. Secondary Settling Tank

Size : 3.5M X 3.5M X 1.5M SWD With a cone
of depth 2.5M ending at a square of
0.6M X 0.6M

The over flow from the aeration tank is allowed to settle in the hopper bottom settling tank. From the hopper bottom settling tank the sludge is drained to the sludge sump and a portion of the sludge is to be re-circulated to the aeration tank to maintain the required MLSS concentration of 4000 mg/l. Excess sludge is to be drained to the sludge drying beds for dewatering along with the primary sludge.

## 8. Re-circulation Pump

No.off : 1+1 standby

Motor HP : 1

Type : semi-open impeller, priming, centrifugal

Material of Construction : C I

Make : stork/kirloskar

Re-circulation pumps are provided for maintaining a proper re-circulation ratio of sludge from the secondary settling tank

## 9. Sludge Drying Beds

No.of beds : 3

Size of each bed : 8M X 3M X 1.2M

The sludge settling at the bottom of the primary settling tank is drained to the sludge drying bed. Sludge is taken in one bed at a time to a height of 0.3m from the bed surface. Once this height is reached, the sludge is taken in the next cell. The sludge collected in one cell is allowed to dry for at least 3 days and then removed. The removed sludge is to be dumped in an area ear marked for sludge dumping. The area is to be made un-pregnable with LDPE lining. The excess sludge from the hopper bottom-settling tank is also drained to the sludge drying beds for further dewatering along with the primary sludge.

### 2.6.2. Water Run:

1. Empty all the tanks and clean them thoroughly.
2. Keep all the valves in the system in close position.
3. Fill the equalization tank with water upto the maximum water level.
4. Put the switches in the MCC in the 'ON' position. Check the rotation of each pump in the effluent treatment plant by operating the local push button starter. If any pump is found rotating in the opposite direction, switch off the switch in the MCC and interchange the terminal box connection of the motor and start the pump again to recheck.
5. Fill up the casings of the self-priming pumps with water.
6. Open the suction line valve of the effluent transfer pump and put the local push button starter in the ON position.
7. As the pump starts running, open the discharge line valve.
8. The water will flow to the mixing channel and from there to the primary settling tank by gravity.
9. Fill up the primary settling tank with water. Check the drain line to the sludge drying beds.
10. Overflow from the primary settling tank will flow to the aeration tank.
11. Fill the aeration tank with water and run the aerator.
12. Fill up the hopper bottom-settling tank with water.
13. Open the suction line valve of the re-circulation pump of the secondary system.
14. Start the pump and open the valve in the discharge line to the inflow point of the aeration tank.

15. Once this re-circulation loop is checked, closes the valve and opens the valve in the line sludge drying beds.

16. Once all the tanks are checked for visible leaks, pumps are to be checked for proper functioning and all the lines are to be checked and cleared.

17. Keep all the tanks filled with water.

**2.6.3. Start up Procedures:**

1. Keep all the valves in the closed position.

2. As the effluent starts collecting in the equalization tank, start the pump, keeping the discharge valve open. Adjust the re-circulation valve to get the required flow rate, and pump the effluent to the mixing channel.

3. Dose 10 % lime solution to the mixing box of the mixing channel and raise the pH of the effluent t0 9.5-10.0. Dose reagent solution at the end of first channel to get the pH of the effluent between 7.0 to 7.5.

4. Effluent flows to the primary settling tank by gravity. Coagulated particles get settled in the tank.

5. The overflow from the primary settling tank is allowed to flow to the aeration tank.

6. The sludge settling at the bottom of the primary settling tank is transferred to the sludge drying beds by opening the valve at the bottom of the primary settling tank.

7. Load aeration tank as instructed in annexure.

8. The overflow from the aeration tank is, send to the hopper bottom settling tank and the suspended particles are allowed to settle. A portion of the sludge settling at the bottom of the hopper bottom settling tank is returned to the aeration tank for maintaining the MLSS and excess sludge is drained to the sludge drying beds for dewatering and drying along with the primary stage.

9. The overflow from the hopper bottom-settling tank is the treated effluent.

### 2.6.4. Safety:

1. Gloves, Goggles and acid proof aprons are to be worn while handling the reagents.

2. If the persons are entering any tank or pit for cleaning or maintenance purpose, advance safety clearance from the Safety Department of the company is to be obtained. Safety department should rule out the presence of any noxious gases like carbon monoxide or hydrogen sulphide in the pit and ensure sufficient oxygen percentage. If noxious gases are present or oxygen is deficient, air should be blown in to the tank/pit by a portable compressor for sufficient duration before persons are allowed to enter.

3. Periodic inspection should be carried out by competent persons to ensure the good condition of electrical switchgear fittings and cabling. M.C.C should be provided with dry powder and carbon dioxide fire extinguisher of sufficient capacity

### 2.6.5. General procedures to be adopted for stabilizing the activated sludge process:

The following procedures are to be adopted for stabilizing the Activated sludge process.

1. Prepare 5 % cow dung slurry of fresh cow dung.

2. Sieve the slurry thoroughly to remove hay, grass or any other impurities, which might lead to the clogging of pumps and pipeline.

3. The aeration tank should be filled with the cow dung slurry up to the total volume.

4. Fill up the hopper bottom-settling tank with cow dung slurry up to ¾ of the total volume.

5. Start the aerator in the aeration tank.

6. Start the sludge recycling pump to pump back the cow dung slurry from the hopper bottom-settling tank to the aeration tank.

7. Add slightly excess quantity of nutrients than prescribed during start up. The nutrients are dissolved in water and added for 24-hour duration.

8. Keep the system running for 24 hours.
9. On the third day, take 10% of the primary overflow into the system.
10. Do not take the total 10% of the primary overflow in one stretch into the system to avoid shock loading. Spread it over a period of 24 hours.

**2.6.6. I) Start up procedures for Aeration tank:**

1. Fill the aeration tank with fresh water.
2. With aerator under operation, add fresh cow dung as slurry through a sieve of around 3-4 mesh till the concentration reaches a level of around 4000 mg/litre (7-8 tonnes of fresh cow dung).
3. Continue the aeration for 3 days at least 12 hrs a day without adding any effluent.

**II) OPERATION**

1. Feed the effluent into the aeration tank after treatment with lime to pH 8-9 and settling in the primary setting tank at the following rate.

| Days | Quantity of Effluent | Feed time (hrs) | Aeration time (hrs) |
|---|---|---|---|
| 4-10 | 25 | 4 | 12 |
| 11-20 | 50 | 8 | 15 |
| 21-30 | 75 | 12 | 18 |
| After 30th | 100 | 16 | 21 |

2. Add DAP (fertilizer grade) at the rate of 350 gm / litres effluent daily in the aeration tank.

3. At two hours interval, pump the settled sludge from the bottom of the primary settling tank in to the sludge drying bed after closing the outlet valve at the bottom of the sludge drying bed.

4. Occasionally scrape down the solids settled on the conical sides of the primary settling tank using a flat paddle into the bottom before pumping the sludge.

5. Clear supernatant liquid when formed in the sludge drying bed is to be siphoned out to the outside drain.

6. Similarly at 3 hrs intervals, pump the settled sludge from the bottom of the secondary settling tank back into the aeration tank.

7. Occasionally scrape down the solids settled on the conical sides of the secondary settling tank using a flat paddle into the bottom before pumping the sludge.

8. Daily check the settlability of the sludge and MLSS in the aeration tank as follows.

a) **Settlelability**

Take a grab sample from the aeration tank and transfer 100ml into a 100 ml measuring cylinder and note the follows.

1. Volume of the settled sludge at 30 minutes settling.
2. Clarify of the supernatant liquid.

**b) MLSS**

The test sample at (a.) is filtered through a preheated and reweighed filter paper and transfers quantitatively all the sludge into the filter paper. Wash the sludge with distilled water allow to dry at 100 ± 5°C cool and weigh.

**MLSS, mg/L =Wt. of sludge in mg X 10**

9. When the aeration tank is under proper working the effluent in the aeration tank become straw in colour (middy colour) and there will be quick settling when the aeration is stopped.

10. When MLSS is above 4500 mg/litre in the aeration tank divert the sludge from the secondary settling tank to the sludge drying bed.

11. When on of the sludge drying bed is physically filled with settled sludge, divert the sludge to the next bed.

12. Allow the sludge to dry for around 20-30 days (sludge cracks) remove manually for land filling purpose.
13. The treated effluent coming out of the secondary settling tank is diverted into the tertiary tank after adding (aluminium) alum in the drain between the secondary and tertiary settling tanks.
14. The clear supernatant liquid is then collected in the final holding pond where it is used for irrigation.
15. Bottom sludge from the tertiary settling tank is pumped into the sludge drying bed once in a shift.
16. At two hours interval, pump the settled sludge from the secondary settling tank into the aeration tank to maintain MLSS (except night).
17. When MLSS is less than 3000 mg/litre in the aeration tank, add required quantity of cow dung.
18. Aeration tank pH level should be 7.5 to 8 maintained.

# CHAPTER - 3

## 3. Experimental Technique

### 3.1 Chemical precipitation technique:

An aqueous solution of the chemical reagent consisting of inorganic salts and polyelectrolyte is well mixed with the effluent in adequate doses. The inorganic salts present coagulate the colour bearing material existing in solution in a hydrophilic colloidal state and precipitate a portion of the other dissolved organics. The Polyelectrolyte as well as metallic ions having `more than two valencies flocculate the coagulated and precipitated fine particles by forming bridges between them. The flocculating effect thus enhances the settling rate of the particles as well as the sedimentation and thickening of the sludge at the bottom of the settling tank / clarifier. In this process along with the removal of colloidal and suspended particles, Substantial quantity of dissolved organics are also removed partly by precipitation and partly by adsorption to the new surface created by coagulation/ precipitation.

By this technique about 60% reduction in BOD and COD are obtained without spending any energy. While conventional secondary process for BOD removal is highly energy oriented.

### 3.2 Investigations on the use of various flocculents / coagulants in the treatment of effluent from rubber processing factories.

**Chemical precipitation for improved plant performance:**

Chemical precipitation involves the addition of chemicals to alter the physical state of dissolved and suspended solids and facilitate their removal by sedimentation. In the past, chemical precipitation was often used to enhance the degree of TSS and BOD removal. In current practice, chemical precipitation is used.

1. As a means of improving the performance of primary settling facilities.
2. As a basic step in the independent physical-chemical treatment of wastewater.

**Inorganic chemicals used for coagulation and precipitation process in wastewater:**

| Chemical | Formula | Molecular Wt | Equivalent Wt | Availability | |
|---|---|---|---|---|---|
| | | | | Form | Percent |
| Alum | $Al_2(SO_4)_3.18H_2O$ | 666.5 | ----- | Liquid | 8.5 (Al2O3) |
| Aluminium Chloride | $AlCl_3$ | 133.3 | 44 | Liquid | --- |
| Ferric Chloride | $FeCl_3$ | 162.2 | 91 | Liquid | 20 (Fe) |
| Calcium hydroxide (Lime) | $Ca(OH)_2$ | 56.1 as CaO | 40 | Liquid | 85-99 |

1. .Alum:

When alum is added to wastewater containing calcium and magnesium bicarbonate alkalinity, a precipitate of aluminium hydroxide will be formed. The over all reaction that occurs when alum is added to water may be illustrated as follows.

$$\underset{\text{(Soluble)}}{3Ca(HCO_3)_2} + \underset{\text{(soluble)}}{Al_2(SO_4)_3.18H_2O} \longleftrightarrow \underset{\text{(insoluble)}}{2Al(OH)_3} + \underset{\text{(soluble)}}{3CaSO_4} + \underset{\text{(soluble)}}{6CO_2} + 18H_2O$$

The precipitation reaction given above also occurs with the addition of aluminium chloride. The insoluble aluminium hydroxide is a gelatinous floc that settles, Slowly through the wastewater, sweeping out suspended material and producing other changes. The reaction is exactly analogous when magnesium bicarbonate is substituted for the calcium salt.

### 2.lime:

When lime alone is added as a precipitant the principles of clarification are explained by the following reactions for the carbonic acid and the alkalinity.

$$\underset{\text{(soluble)}}{H_2CO_3} + \underset{\text{(slightly soln.)}}{Ca(OH)_2} \longleftrightarrow \underset{\text{(some what soln.)}}{CaCO_3 + H_2O}$$

$$\underset{\text{(soluble)}}{Ca(HCO_3)_2} + \underset{\text{(slightly soln.)}}{Ca(OH)_2} \longleftrightarrow \underset{\text{(some what soln.)}}{2CaCO_3 + 2H_2O}$$

A sufficient quantity of lime must therefore be added to combine with all the free carbonic acid and with the carbonic acid of the bicarbonates to produce calcium carbonate. Much more lime is generally required when it is used alone than when Ferric chloride / Aluminium chloride is also used.

### 3. Ferric chloride:

Because of the many problems associated with the use of ferrous sulphate, ferric chloride is the iron salt used most commonly in precipitation applications. When ferric chloride is added to waste water, the following reactions take place.

$$\underset{\text{(soluble)}}{2FeCl_3} + \underset{\text{(soluble)}}{3Ca(HCO_3)_2} \longleftrightarrow \underset{\text{(insoluble)}}{2Fe(OH)_3} + \underset{\text{(soluble)}}{3CaCl_3} + \underset{\text{(soluble)}}{6CO_2}$$

### 3.3 Flocculation in wastewater treatment:

The purpose of wastewater flocculation is to form aggregates or flocs from finely divided particles and from chemically destabilized particles. Flocculation is a transport step that brings about the collisions between the destabilized particles needed to form larger particles that can be removed readily by settling or filtration.

Flocculation typically follows rapid mixing where chemicals have been added to destabilize the particles. The destabilization of particles resulting from addition of chemicals is defined as “coagulation”. There are two types of flocculation.

1. Micro-flocculation
2. Macro-flocculation

The distinction between these two types of flocculation is based on the particle sizes involved.

**1.Microflocculation:**

It is the term used to refer to the aggregation of particles brought about by the random thermal motion of fluid molecules. The random thermal motion of fluid molecules is also known as Brownian motion. Micro-flocculation is significant for particles that are in the size range from 0.001 to about 1μm.

**2.Macroflocculation:**

It is the term used to refer to the aggregation of particles greater than 1 or 2 μm. Macro-flocculation can be brought about by

- Induced velocity gradients.
- Differential setting.

Particles can be brought together (i.e., flocculated) by inducing velocity gradients in a fluid containing the particles to be flocculated. As shown in the figure (1) faster moving particles will overtake slower moving particles in a velocity field. If the particles that collide stick together, a large particle will be formed that will be easier to remove by gravity separation.

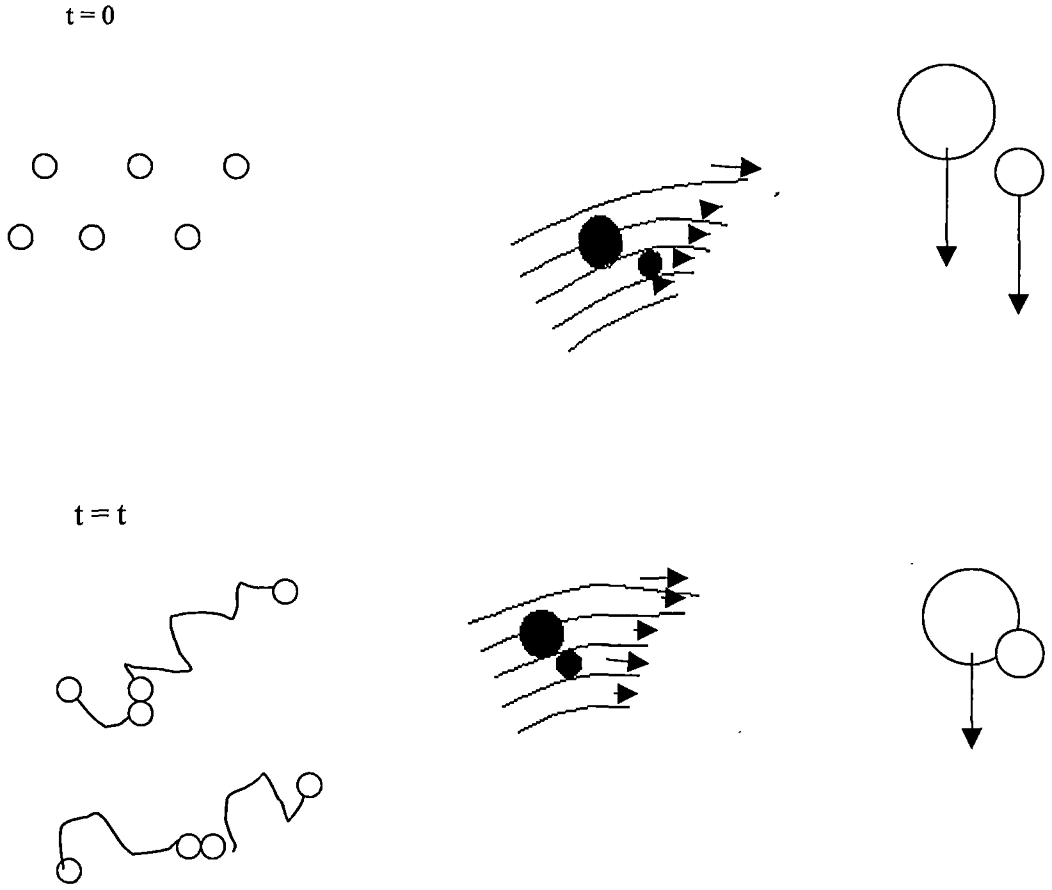
In macro-flocculation by differential settling large particles over take smaller particles during gravity settling. When two particles collide and stick together, a larger particle is formed that settles at a rate that is greater than that of the larger particle before the two particles collided. It should be noted that flocculation brought about by induced velocity gradient is ineffectual until the colloidal particles reach a size of 1 or 2 μm through contacts produced by Brownian motion.

### 3.4 Schematic illustration of the two types of flocculation:

**Fig 3.1.**

**1.Microflocculation** **2. Macro-flocculation**

Brownian motion Velocity gradient Differential settling



**Used for chemicals:**

1. Aluminium chloride
2. Ferric chloride

### 3.5. Fundamentals of chemical coagulation in the wastewater:

Colloidal particles found in the wastewater typically have a net negative surface charge. The size of colloids (about 0.01 to 1μm) is such that the attractive body forces between particles are considerably less than the repelling forces of the electrical charge. Under these stable conditions, Brownian motion, keeps the particles in suspension (i.e., random movement) is brought about by the constant thermal bombardment of the colloidal particles by the relatively small water molecules that surround them. Coagulation is the process of destabilizing colloidal particles so that particle growth can occur as a result of particle collisions.

**Use of Electrolytes:**

Electrolytes can be added to coagulate colloidal suspensions. Increased concentration of a given electrolyte will cause a decrease in zeta potential and a corresponding decrease in repulsive forces. The concentration of an electrolyte that is needed to destabilize a colloidal suspension is known as the critical coagulation concentration (CCC). Increasing the concentration of an indifferent electrolyte will not result in the re-stabilization of the colloidal particles. As with the addition of potential determining ions, the use of electrolytes is also not feasible in wastewater treatment.

### 3.6. Particle Destabilization and Aggregation with Polyelectrolytes:

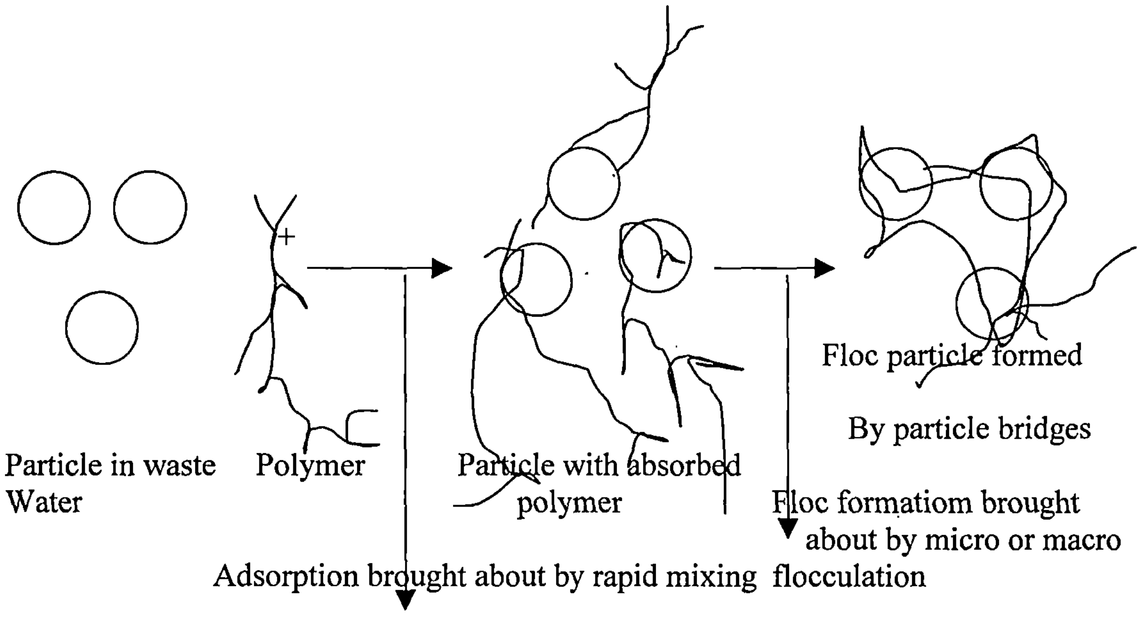
Polyelectrolytes may be divided into two categories they are natural and synthetic. Important natural polyelectrolytes include polymers of biological origin and those derived from starch products such as cellulose derivatives and alginates. Synthetic polyelectrolytes, consists of simple monomers that are polymerized into high molecular weight substances. Depending on whether their charge, when placed in water is negative, positive or neutral these polyelectrolytes are classified as anionic, cationic and non-ionic, respectively. The action of polyelectrolytes may be divided into following three general categories.

1. **Charge neutralization:**

In the first category, polyelectrolytes act as coagulants that neutralize or lower the charge of the wastewater particles. Because wastewater particles normally are charged negatively, cationic polyelectrolytes are used for this purpose. In this application the cationic polyelectrolytes are considered to be primary coagulants. To effect charge neutralization, the polyelectrolyte must be adsorbed to the particle. Because of the large number of particles found in wastewater, the mixing intensity must be sufficient to bring about the absorption of the polymer on to colloidal particles. With inadequate mixing, the polymer will eventually fold back on itself and its effectiveness in reducing the surface charge will be diminished, further, if the number of colloidal particles is limited, it will be difficult to remove them with low polyelectrolyte dosage.

**Definition sketch for inter-particle bridging with inorganic polymers:**

**Fig: 3.2.**



### 2. Polymer Bridge Formation:

The second mode of action of polyelectrolyte is inter-particle bridging. In this case, polymers that are ionic and non-ionic become attached at a number of adsorption sites to the surface of the particles found in the wastewater. A bridge is formed when two or more particles become adsorbed along the length of the polymer. Bridged particles become intertwined with other bridged particles during the flocculation process. The size of the resulting 3-dimensional particles grows until they can be removed easily by sedimentation. Where particle removal is to be achieved by the formation of particle-polymer bridges, the initial mixing of the polymer and the wastewater containing the particle to be removed must be accomplished in a matter of seconds. Instantaneous initial mixing is usually not required as the polymers are already formed, which is not the case with the polymers formed by metal salts. As noted above, the mixing intensity must be sufficient to bring about the adsorption of the polymer onto the colloidal particles. If inadequate mixing is provided, the polymer will eventually fold back on itself, in which case it is no possible to form polymer bridges.

### 3.Charge neutralization and polymer bridge formation:

The third type of polyelectrolyte action may be classified as a charge neutralization and bridging phenomenon, which results from using cationic polyelectrolytes of extremely high molecular weight. Besides lowering the surface charge on the particle, these polyelectrolytes also form particle bridges.

## 3.7. Polyelectrolytes:

Flocculation is agglomeration of the very tiny micromized particles into large units. An efficient flocculation programme is essential for the success of any modern process. Industry polymers of acrylic monomers are being used all over the world for quick solid-liquid separation in acidic, alkaline and neutral mediums. Acrylic polymers are non-corrosive to metals usually found in process equipments and are more efficient than traditional inorganic flocculants. Significant cost reduction can be achieved by process industry by selective and efficient use of flocculants.

Synthetic polyelectrolytes are acrylamide and acrylic acid polymer and copolymers having a very high molecular weight. These polyelectrolyte carry ionic charges and in aqueous media tightly bound and compact polymer molecular starts unwinding and opening. In this state, ionic charges present in polymer attracts suspended fines in media. Result is fines getting agglomerated forming what is known as macroflocs. Soon macro-flocs form a mud like which rapidly starts dropping as flocculation proceeds. Result is heavy and compact sludge settling in lower parts of tank and very clear supernatant liquor.

### 3.7.1. Laboratory trials:

Weight about 0.5 gm to 1.0 gm of the flocculation as per the effective range of pH for treatment of the effluent. Add water to make 0.5 % solution of flocculent stir at slow speed till all flocculants is completely dissolved (overnight soaking of the flocculent in water helps for the fast dissolution). Take the measured quantity of the representative sample of effluent and add measured amount of 0.5 % flocculent solution with slow stirring for couple of minutes. Transfer this in a glass measuring cylinder to observe settling rate of flocculated solid/sludge against the untreated representative sample of effluent.

Repeat the experiment with variable dosage of 0.5% flocculent solution to get optimum ppm dosage for plant application. The tested dosage is 5ml, 10ml, 15ml, 20ml, 25ml...

### 3.7.2 Optimum Dosage:

The experimentally found that the optimum dosage required is 15ml/1 litre for getting good clarity and high settling rate. In the case of plant operation, the optimum dosage required is depending upon the pH of the raw effluent. The pH required for the effluent is 7.5 to 8.5 before dispatch to primary settling tank.

### 3.8. pH Adjustment in the plant operation:

In a variety of effluent treatment operations and processes, there is often a need for pH adjustment. Because a number of chemicals are available that can be used, the choice will depend on the suitability of a given chemical for a particular application and prevailing economics. Wastewater that is acidic can be neutralized with any number of basic chemicals. Sodium hydroxide (i.e., caustic soda) and sodium carbonate, although somewhat expensive, are convenient and are used widely by small plants or for treatment where small quantities are adequate. Lime, which is cheaper but somewhat less convenient is the most widely used chemical

**PH requirement in the plant operation:**

| After lime addition | After chemical additions |
|---|---|
| 8-9 | 7.5-8 |

# CHAPTER - 4

## TEST METHODS FOR EFFLUENT ANALYSIS: -

### 1.Chemical oxygen demand (COD)

This method determines the quantity of oxygen required to oxidize organic matter in an effluent under specific conditions of oxidizing agent, temperature and time.

Theory: -

The COD test is used for estimating the concentration of organic matter in wastewaters. It is done by heating under total reflux conditions a measured volume of the effluent with a known excess of potassium dichromate, is the presence of sulphuric acid for a period of 2 hours. Organic matter in the sample is oxidised and as a result yellow dichromate is consumed and replaced by green chromic salt. Silver sulphate is added as a catalyst.

$$Cr_2O_7{}^{2-} + 14H^+ + 6e^- \longleftrightarrow 2Cr^{3+} + 7H_2O$$

Measurement is performed by titrating the excess dichromate against Ferrous sulphate using Ferroin indicator (Ferrous phenanthrolein)

If chlorides are present in the effluent, they interfere with the COD test since chlorides are oxidised by dichromate to

$$6Cl^- + Cr_2O_7{}^{2-} + 14H^+ \longrightarrow 3Cl2 + 2Cr^{3+} + 7H_2O$$

This interference is prevented by the addition of mercuric sulphate ($HgSO_4$) to the mixture as $Hg^{2+}$ combines with $Cl^-$ to form mercuric chloride ($HgCl_2$)

$$Hg^{2+} + 2Cl^- \longrightarrow HgCl_2$$

The presence of $Ag_2So_4$ catalyst required for oxidation of straight chain alcohols and acids. If insufficient quantity of $HgSo_4$ is added, the excess $Cl^-$ precipitates the $Ag_2SO_4$ catalyst, thus leading to erroneously low value for the COD test.

$$Ag^+ + Cl^- \longrightarrow AgCl$$

Standard Ferrous Ammonium sulphate (Fe $(NH_4)_2$ $(So_4)_2$. $6H_2O$)

Is used for the titration since standard Ferrous

Sulphate loses strength with age due to air oxidation.
Ferrous ammonium sulphate is oxidised by dichromate as follows

$$Cr_2O_7^{2-} + 14H^+ + 6\,Fe^{2+} \rightleftarrows 2Cr^{3+} + 6\,Fe^{3+} + 7H_2O$$

The red brown colour corresponding to the end point is due to the formation of complex of ferrous ion with phenanthrolein ferrous phenanthrolein complex is formed as soon as all dichromate is reduced to Cr $^{3+}$ (chromic) and therefore further addition of ferrous ammonium sulphate results in the excess of ferrous ion Fe $^{2+}$.

$$Fe^{3+}(C_{12}H_8N_2)_3 + e^- \longrightarrow Fe^{2+}(C_{12}H_8N_2)_3$$

| | |
|---|---|
| Ferric phenanthrolein | Ferrous phenanthrolein |
| (Pale blue) | (Red brown) |

**Reagents: -**

1. Potassium dichromate, 0.1N. Dissolve 4.903g of AR $K_2Cr_2O_7$ in 1 litre of distilled water.

2. Sulphuric acid.

3. Ferrous –1:10- phenanthrolein indicator solution. Dissolve 0.695g of ferrous sulphate, $FeSo_4$ $7H_2O$ in 100 ml of distilled water, add 1.485 g of 1:10 phenanthrolein monohydrate, shake and leave it for 2 days to dissolve completely.

**Ferrous Sulphate solution, 0.1N:-**

Dissolve 27.8g of ferrous sulphate $FeSo_4$. $7H_2O$ in about 500 ml of distilled water, add 25 ml of conc. $H_2So_4$, stir well on cooling make up to 1 litre with distilled water. This solution must be standardized occasionally.

**Procedure:-**

1. Pipette 25 ml of potassium dichromate 0.1 N into a round-bottomed flask of 250 ml or 500 ml.
2. Add 0.4 gm of mercuric sulphate.
3. Add conc. $H_2So_4$ (40) ml, with shaking.
4. Add suitable volume of effluent sample until the colour of the contents in the R.B flask changes slightly from reddish orange to greenish orange.
5. Add a tinge of silver sulphate as catalyst. Fix a suitable reflux condenser to the flask mix the solution well and boil it under reflux for at least 2 hours.
6. After 2 hours of boiling, allow the contents to cool down for about ½ hour and wash the inside of the reflux with some distilled water. Cool the flask further in ice water. Add a few drops (5-6) of indicator and titrate with ferrous sulphate solution (0.1N) until the colour changes to reddish brown that is the end point. Carry out a blank also.

**Calculation: -**

$$\text{COD (mg/litre)} = \frac{(a-b) \times c \times 8000}{\text{Vol of sample taken}}$$

Where a = ml Fe $So_4$ (0.1N) used for blank.

b= ml Fe $So_4$ (0.1N) used for sample

c = normality of Fe $So_4$, 0.1N .

**2. Biochemical Oxygen Demand:**

**Theory**

The amount of dissolved oxygen consumed by chemical and microbiological action. When a sample is incubated for 5 days at 20°C is termed as BOD. The BOD normally gives a rough indication of the organic matter present in the sample.

The oxygen demand of wastewater is exerted by three classes of materials.

- Carbonaceous organic materials usable as a source of food by aerobic organisms.
- Oxidation nitrogen derived from nitrate ammonia and organic compounds, which serve as food for specific bacteria e.g.: -Nitosoamines and Nitrobacter.
- Chemical reducing compounds eg: - Ferrous ion ($Fe^{++}$), sulphites ($SO_3^{--}$) and sulphide ($S^{2-}$), which are oxidized by, dissolved oxygen.

For domestic sewage nearby all oxygen demand is due to carbonaceous organic materials. For effluents subjected to biological treatment, a considerable part of the oxygen demand may be due to nitrification.

**Reagents:**

**1.$MnSo_4$ Solution.**

Dissolved 500gm of $MnSo_4$.H2O in 1 litre distilled water and this would liberate not more than a trace of iodine when added to an acidified solution of potassium iodide.

**2.Alkaline-Iodide-Azide solution:**

Dissolve 500gm of sodium hydroxide in its own weight of distilled water. Allow to stand for some days, during which any carbonate present sinks to the bottom. Siphon out all the clear liquid, and add 150gm of potassium iodide and 10g of sodium azide (dissolved in a small quantity of distilled water) and make up to 1 litre this solution should not give a colour with starch when diluted and acidified.

**3.Sodium thiosulphate solution (0.025N)**

Dissolve 12gm of sodium thiosulphate in 2 litres-distilled water and standardize with potassium dichromate (0.025N)

**4.Starch indicator**

**5.Conc.Sulphuric acid**

**Dilution water:**

As tap water differs very much in their content of inorganic salts, and as most of them are now chlorinated, it is recommended that synthetic dilution water be employed stock solution of the following pure chemicals are required.

a) Ferric chloride : 0.25g $FeCl_2.6H_2O$ in 1 litre distilled water.

b) Calcium chloride :11.0g CaCl2 in 1 litre distilled water.

c) Magnesium sulphate : 10.0g $MgSO_4$ $7H_2O$ in 1 litre distilled water.

d) Phosphate buffer stock solution: Dissolve 34g of potassium phosphate ($KH_2PO_4$) in 500 ml distilled water. Add 175ml NaOH. This should give a pH of 7.2. Add 1.5gm of ($NH_4$) $2SO_4$ and diluted to 1litre.

To each litre of distilled water are added

| | |
|---|---|
| (a) | 0.5 ml solution |
| (b) | 2.5 ml solution |
| (c) | 2.5 ml solution |
| (d) | 1.25 ml solution |

The water should then be well aerated (2hr) using an air pump and subjecting the stream of air through a filler of.

(a) Sodalime (non deliquescent, 4-10 mesh)

(b) Activated charcoal (granular- activated for gas absorption- passed an 18,mesh sieve)

(c) Cotton wool to remove any particle in the air stream

**Preparation of the sample:**

Dilution depends on the strength of the sample. Unless the BOD of the sample is already known approximately, the required degree of dilution will not be known. An approximate dilution figure is estimated from COD determination. After determining the

COD of the sample the approximate dilution can be found out from the following table, assuming the BOD as half of COD.

| **Volume of effluent** | **BOD** |
|---|---|
| 0.01 | 40,000-10,0000 |
| 0.02 | 20,000-50,000 |
| 0.05 | 8,000-2,000 |
| 0.10 | 4,000-10,000 |
| 0.20 | 2,000-5,000 |
| 0.50 | 800-2,000 |
| 1 | 400-1,000 |
| 2 | 200-500 |
| 5 | 80-200 |
| 10 | 40-100 |
| 25 | 16-40 |
| 50 | 8-20 |
| 100 | 9-10 |

**Procedure:**

The BOD bottles of 300ml capacity are fully filled with BOD dilution water and proper volume of sample without air bubbles. A blank should be placed without the sample. The samples should be incubated for 5 days.

To the sample taken from incubator add

1. 2ml of Manganese sulphate solution followed by 2 ml of alkaline iodide-azide solution. Mix well the contents of the bottle by inversion and rotation. The precipitate flocculates and settles at the bottom in 5 to 10 minutes.

2. Add 2ml of Conc.$H_2SO_4$ replace the stopper and mix well the contents by rotation.

When introducing various reagents into the full bottle of sample, the tip of the pipettes in each case should be well below the surface of the liquid replace the stopper carefully after each addition as to avoid exclusion of air bubbles and thoroughly mix the contents by inverting and rotating the bottles several times.

The solution is transferred into the iodine flask and titrate against std. thio using starch as indicator. Blank is also done.

$$\text{BOD mg/litre} = \frac{(a-b) \times c \times 8000}{\text{Vol. Of sample taken}}$$

Where, a -- litre volume for blank

b—litre volume for sample

**3.Total Solids (TS):**

Total solids are the total solid matter obtained when an effluent sample is evaporated in a weighing dish on a steam bath and then dried to constant weight in an oven at100-105 °C.

**Apparatus:**

1. Evaporating dish (100 ml capacity).
2. Measuring cylinder, 100 ml.
3. Steam bath.
4. Oven.

**Procedure:-**

1. Shake the effluent sample thoroughly to homogenize.
2. Measure 100ml of the sample into a weighed crucible.
3. Measure 100 ml of the sample to dryness on a steam bath and complete the drying by heating for one hour in an oven at 100+/-5 °C.
4. Cool is desicator and weigh.

**Calculation:**

$$\text{Total solids (mg/litre)} = \frac{A - B \times 106}{C}$$

Where, A -- Wt. Of crucible + solids (g)

B -- Wt. Of crucible alone

C – Volume of the sample (ml)

### 4..Dissolved solids (DS)

Dissolved solids are the solid matter obtained when an effluent sample after filtration through a what No. 40 filter paper, is evaporated in a weighing dish on a steam bath and then dried to constant weight in an oven at 100 – 105°C.

**Apparatus:**

1. Crucible 100ml capacity
2. Measuring cylinder, 100ml
3. Steam bath
4. Oven
5. Desicator

**Procedure:**

1.Filter the effluent sample through what No: 41 filter paper.

2.Measure 100ml of the sample into a weighed crucible.

3.Evaporate the sample to dryness on a steam bath and complete the drying by heating for one hour in an oven at 100+ 5°C.

4. Cool in desicator and weigh.

**Calculation:**

Dissolved solids (mg/litter) Total solids (mg/litre) $= \frac{(A - B) \times 10^6}{C}$

Where, A -- Wt. Of crucible + solids

B -- Wt. Of crucible alone

C – Volume of the sample (ml)

### 5. Note Inorganic DS:

For inorganic DS, the residue obtained above (3) is ash in the muffle furnace weigh and calculated Inorganic

$$DS\ (mg/litre) = \textbf{Total}\ solids\ (mg/litre) = \frac{A - B \times 10^6}{C}$$

Where, A -- Wt. Of crucible + ash

### 6.Suspended solids (SS)

The difference between total solids and dissolved solids in suspended solids.

Suspended solids = TS - DS

### 7.Total Nitrogen

Suitable volume of the efficient is pipetted out, into the distillation flask, and digested with catalyst mixture and Conc.$H_2SO_4$ and estimated the nitrogen content by micro kjeldahl method. (Refer estimation of Nitrogen by dry rubber)

$$Total\ Nitrogen = \frac{N \times 14 \times 1000 \times TV}{V}\ mg/\ litre$$

Where, N - Normality of $H_2SO_4$.

V - Volume of effluent acid.

### 8.Ammonical Nitrogen:

**Apparatus**

Hoskin's distillation apparatus

Reagents:

1. Sulphuric acid 0.01 N solution

2. Light magnesium oxide.

3. Boric acid solution (2.1)

4. Screened methyl red indicator. Dissolve 0.1g methyl red and 0.05 mg methylene blue in 100 ml ethyl alcohol.

**Procedure:**

1.Pipette into the empty distillation flask required volume of effluent sample (previously neutralized to phenolphthalein if necessary)

2.Dilute to 350ml with ammonia free distilled water.

3.Add 0.25 g of light magnesium oxide plus 2 glass beads to avoid bumping and connect the flask to the splash – head and condenser.

4.Place the receiving flask containing 20ml of boric acid solution and two or three drops of the indicator solution below the condenser so that the end of the condenser tube reaches the bottom of the flask.

5.Distill at the rate of 5 to 10 ml per minute until at least 150ml have been collected.

6.Titrate the distillate to a light violet endpoint with standardized 0.01N $H_2SO_4$ using micro-burette.

**Calculation:**

$$\text{Ammonical Nitrogen} = \frac{N \times 14 \times 1000 \times TV}{V} \text{ mg litre}$$

Where, N - Normality of $H_2SO_4$.

V - Volume of effluent latex

TV. – Titre volume.

9. **Free Ammonia:**

10ml of the effluent sample is pipetted out into the distilled flask and distilled in the micro-kjeldhal apparatus. The ammonia evolved is absorbed in the boric acid and titrated with std. $H_2SO_4$. ( 0.01N)

$$\text{Free Ammonia} = \frac{N \times 17 \times 1000 \times TV}{V} \text{ mg / litre}$$

Where, N - Normality of $H_2SO_4$.

V - Volume of effluent acid.

TV - Titre volume

### 10.Sulphate:

In this method, sulphate in rubber effluent is precipitated as Barium sulphate in the presence of hydrochloric acid and determined gravimetrically.

**Reagents:**

1.$BaCl_2$ solution (10 %). Dissolve 10mg $BaCl_2 . 2H_2O$ in 90 ml distilled water.

2.50% HCl.

**Procedure:**

Pipette out 20 ml of the effluent sample dilute with water to 100ml and add 5 ml of HCl. Boil the solution and add 20 ml of $BaCl_2$. The precipitate is again boiled for 1 minute, and cooled. It is filtered through what man no.42 filter paper and ignited in a weighed, clean crucible at 550°C in the muffle furnace. Again weigh of the ash after cooling and calculated the sulphate content.

$$\text{Sulphate concentration mg / litre} = \frac{\text{Wt. Diff} \times 411.5}{\text{Volume of effluent}}$$

### 11.Cholride:

**Out line :**

The sample after neutralization is titrated against silver nitrate solution using potassium chromate.

**Reagents:**

1. Calcium carbonate
2. Std: Nitric acid - 0.1N .
3. Potassium chromate solution

Dissolve 5 gm of potassium chromate with distilled water to make 100 ml. Add silver nitrate solution to produce a slight red precipitate and filter.

**Standard Silver nitrate solution:**

Dissolve 4.791 of silver nitrate dried at 105° + 2°C in distilled water and make up to 1000ml. One millimeter of the solution, which is equivalent to 1mg or chlorides (as Cl). Standardise with a standard chloride solution. The solution shall be kept as the dark.

Use 50 ml of the sample (Filtered if necessary). If the sample is coloured, decolourise with aluminium hydroxide. If the pH of the sample is less than 6.8, add a small amount of calcium carbonate to the sample for neutralization. If the pH is above 10 neutralize with 0.1N nitric acid and then add a trace of Calcium carbonate. Add 1ml of potassium chromate solution and titrate with std: silver nitrate solution with constant stirring until there is perceptible reddish colouration subtract 0.2 ml from the titration figure to allow for the excess of reagent required to form silver chromate.

$$\text{Chloride ( as Cl) mg/litre} = \frac{(\text{T.V} - \text{B}) \times 35.46 \times 1000 \times \text{N}}{\text{V}}$$

**12.Sulphide:**

a. Hydrochloric acid, 6N.

b. Std: Iodine solution 0.025 N: Dissolve 20- 25g potassium iodide, KI, in a little water and add 3.2 gm Iodine. After the iodine has dissolved dilute to 1000ml.

c. Std: sodium thiosulphate solution ( 0.025N)

d. Starch solution.

Pipette out 20ml of Iodine solution to 500ml Iodine flask. Add 2ml of 6N HCl, and add suitable volume of effluent and back titrate with std. thio, using starch as indicator. A blank also should be done.

$$\text{Sulphide mg/ litre} = \frac{400 \times \text{N} \times (\text{B} - \text{V})}{0.025 \times \text{volume}}$$

### 13.Phosphate:

### The vanado molybodo method:

**Ammonium vanadate solution:**

Dissolve 2.5gm Ammonium vanadate in 500ml hot water, add 20 cc. Conc. $HNO_3$ and dilute to 1 litre.

**Ammonium molybdate solution:**

Dissolve 50gm ammonium molybdate in warm water and dilute to 1 litre. Filter before use.

**Procedure:**

A 10 ml of Ammonium vanadate and Ammonium molybdate are added to 50 ml std. Flask and suitable volume of effluent is pipetted and made up to 50ml. Then determine the concentration of phosphate calorimetrically as in the case of fertilizers.

### 14.Oil and grease:

The oils and grease are extracted by an organic solvent. The solvent is distilled off and the weight of the extracted matter is determined.

**Procedure:**

500 ml of the filtered effluent sample is taken in a separating funnel and 50ml of petroleum spirit is added. Acidify it with Conc. HCl separated the ether layer and collected on a dry R.B.flask containing anhydrous sodium sulphate. When this solution becomes clear 40 ml is taken into a weighed beaker dried and evaporated. The Wt of the beaker with residue is determined and calculated the oil and grease content as follows.

$$\text{Oil \& grease} = \frac{\text{Wt. Of residue} \times 1000 \times 100 \times 1000}{500 \times 40}$$

$$= \text{Wt. Of residue} \times 5 \times 10^3$$

# CHAPTER - 5

## 5.1 Present status:

Effluent treatment process is currently adopted in latex centrifuging factories involves the following unit operations.

- Homogenization
- Removal of fine particles of rubber and other suspended particles, if any.
- PH adjustment.
- Primary clarification.
- Aeration.
- Secondary clarification
- Drying of sludge.

The wastewater generated at various stages of latex processing are different in nature.

For example wastewater from barrels, latex reception tanks etc will be mildly alkaline and contains some sludge materials serum from skim coagulation will be highly acidic; pH is the range of 1 – 3. Treatment of the effluent becomes cumbersome due to its variability. Hence all the wastewater generated in a day is homogenized in a compositing pond. Hence the water flowing out of the compositing pond will be of more or less uniform in nature.

A rubber trap is used to remove fine particles of rubber in the effluent. Any floating lighter particles are also removed.

The pH of water coming out of the rubber trap will be acidic, pH being 3 – 4. Aerobic bacteria will not survive at this level of acidity. Hence limewater is mixed in to the waste waster and pH is enhanced to 7-8. During that process some amounts of soluble components are precipitated. Also to sulphate in the wastewater is precipitated as calcium sulphate. The precipitated substances are removed by sedimentation is a primary clarifier. There is some reduction in COD and BOD of the effluent.

The sedimented material is the form of slurry is pumped to drying beds.

The overflow from the primary clarifier goes to aeration tank. Retention period in the aeration tank is 2 – 3 days. In the aeration pond the organic substances present in the wastewater get oxidized, mainly to carbon dioxide and water. The oxidation reactions considerably reduce the COD and BOD loadings of the wastewater. 2-3 days retention is necessary for bringing these parameters down to the level, as specified in the requirements of the pollution control board.

### 5.2 Limitations of the present Methods:

Even though, the methods described above bring quality standards of water, in line with the PCB requirements, the process, as a whole is lengthy and expensive. Some of the limitations of the present method are given below.

#### 1. Slow rate of sedimentation:

The rate of sedimentation of precipitated substances depends on the viscosity of the medium, the higher the viscosity, lower the rate, high concentration of dissolved substances lead to increase in viscosity of the liquid medium. Some of the dissolved substances are precipitated as fine particles and their rate of sedimentation is very low. This causes high retention periods in clarifier.

High retention period causes installation of high tank capacities. This causes higher costs and land requirement.

Longer retention period is the clarifier causes anaerobic degradation of non-rubber substance in the clarifier itself resulting in generation of fowl smelling gases. This causes air pollution.

**High cost of Aerobic Treatment:**

In the present system of effluent treatment, the wastewater reaching aeration pond has substantially high COD and BOD. To reduce the level of these parameters to acceptable limits, long periods of aeration are required. This causes large capacities for aeration tank. Cost of aeration also will be high.

**Pond clarity for treated water:**

Despite the above stated long aeration, the final treated water may not be clear always. Poor clarity is due to the presence some dissolved substances, resisting aerobic oxidation and also due to the presence very minute suspended particles, which do not sediment in the final clarifier.

Even though parameter-wise, the treated water meets the requirements laid down by the pollution control board, an industry is reluctant to recycle the treated water for the factory process.

### 5.3 Scope of the present work:

Effluent treatment system, currently employed by latex processes require high initial investment, higher land requirement and high operating cost. In this circumstance it was thought that if the polluting organic substances present in the water being treated are precipitated with the assistance of an external flocculating agent, the treatment becomes simpler. The duration of effluent treatment and the operating cost would be reduced. Hence a study was under taken on the use of aluminium chloride and ferric chloride as flocculating agents in the treatment of wastewater generated in a latex-centrifuging factory.

### 5.4 Objectives of present day status:

Effluent treatment methods currently followed in latex centrifuging factories are based mainly on aerobic oxidation of the pollutants. After the preliminary stages of removal of suspended impunities, and adjustment of pH, the resultant effluent is aerated. Nowadays electricity is becoming costly and hence aerobic oxidation method is becoming more and more expensive.

The objectives of the present study was to separate the pollutants by precipitation to the maximum extend possible; so that the semi treated effluent is low in level of pollutants. This effluent need to be aerated to lesser extend only, to a reduction in duration of aeration and associated cost.

To achieve this goal, the effects of aluminium chloride, ferric chloride, polyelectrolyte ( copolymer of acrylamide and acrylic acid, a property chemical Sonimer, marketed by M/S Roop Rosayan Industries p Ltd, Mumbai) in reducing the BOD and COD loadings of wastewater are evaluated separately and in combination. The above chemicals are used in presence of lime to enhance the pH. The rate of sedimentation of the precipitated material was studied in the case of each chemical.

Aeration is an important step in processing of wastewater in the conventional process and retension period is 3-4 days. The effect on duration of aeration in final values is reported in the modified treatment method.

# Chapter 6

## 6.1. Effect of flocculating Agents on Precipitation of pollutants from Effluent Generated at Latex centrifuging Factories:

Major effluent generated at a latex centrifuging factory is skim serum. By volume this is about 50-55% of the volume of field latex processed. In addition to this substantial volume of wastewater is generated by way of washing of barrels, tanks, bowl of centrifuging; etc. The composite effluent is subjected to treatment process.

The conventional treatment of effluent involves neutralization acidity by lime addition, followed by aeration. However removal of pollutants by addition of lime was not high. This necessitates longer aeration for oxidation, thus causing higher cost in effluent treatment. Hence use of other chemicals like aluminium chloride $Alcl_3$, ferric chloride $FeCl_3$ and polyelectrolyte (acrylamide and acrylic acid co-polymer) in presence of lime as flocculating agents for bringing in reduction in COD, BOD, total dissolved solids etc were tried.

## 6.2. Experimental:

### 1. Screening of chemicals:

$AlCl_3$, $Fecl_3$, polyelectrolyte mixed sulphates and alum were screened. These chemicals are added to effluent from latex centrifuging factories at 0.5 % concentration. This was followed by addition of lime to pH 7.5- 8.0. The effluent was tested for parameters like pH, BOD, COD, total dissolved solids suspended solids, ammonical nitrogen, total nitrogen; etc. The activities of these chemicals were further examined in presence of polyelectrolyte (synergetic action). The results of preliminary studies indicated that among the chemicals evaluated $AlCl_3$ and $FeCl_3$ gave better performance than others. Hence further studies were limited to $AlCl_3$ and $FeCl_3$. The rate of sedimentation of the precipitated material also was evaluated the effect of pH on sedimentation rate also was studied. The above study was further carried out followed by aeration for 24 hrs. In this study, the effect of lime addition and aeration for 3 days was taken in control.

### 6.3. Results and discussion:

#### 1. Screening of flocculating agents in presence of lime:

Table 6.1 gives the results of the conventional treatment. (Lime addition and aeration for 3 days) table 6.2 to 6.6 gives the results of addition of $AlCl_3$, $FeCl_3$, alum, polyelectrolyte and mixed sulphates respectively in presence of lime. The results in table 6.1 suggest that even though there is substantial reduction in BOD, COD, dissolved solids; etc. the water does not meet the requirements of pollution control board, ie; 3 days aeration is inadequate.

The result in table 6.2 shows the effect of addition of $AlCl_3$ and lime on the level of pollutants in the raw effluent. $AlCl_3$ was added at 0.3 % on the weight of effluent and lime was added in sufficient quantity to enhance the pH to 8.5

Table 6.3-6.6 shows the results for $FeCl_3$, potash alum, polyelectrolyte, and mixed sulphates respectively. Among the chemicals screened, $AlCl_3$ and $FeCl_3$ give better performance compared to others.

When these flocculating agents are added to waste water, the chemical gets uniformly distributed in the effluent. When lime is added the hydroxide of the metal is precipitated. The precipitated metal hydroxide co precipitates some of the proteins and other organic substances by occlusion, leading to a reduction in BOD, COD, etc.

#### 2. Synergetic action of flocculants:

Some preliminary trails were initially on the use of a combination of chemicals in presence of lime. It was observed that the performance of $AlCl_3$, $FeCl_3$, alum and mixed sulphates are improved. When any one of these chemicals were used along with polyelectrolyte, followed by lime. In these cases also pollutants were precipitated and removed by sedimentation. In the actual trails, 0.1% $AlCl_3$ and 0.015% of polyelectrolyte were added. The effect of this addition $AlCl_3$, $FeCl_3$, Alum and mixed sulphate along with polyelectrolyte, followed lime are given in tables 6.7-6.10 respectively. This result shown in table 6.7 suggests that when $AlCl_3$, is added to effluent there is some improvement in BOD, COD removal and a minor improvement removal of ammoniacal nitrogen. However, removal of total nitrogen has increased from 14.2% to 67.5%

The effect of addition of $Fecl_3$ and polyelectrolyte in presence of lime is given in table 5.8. There is a small increase in removal of COD, good improvement in removal of BOD and total nitrogen; however, there is a small reduction in removal of ammonical nitrogen.

In addition of polyelectrolyte along with alum in presence of lime gives some improvement in removal of BOD, COD and total nitrogen. A marked reduction in ammonical nitrogen is achieved.

However the use of polyelectrolyte along with mixed sulphate in presence of lime gives only poor performance than that of mixed sulphate when used alone is shown by the results in table 6.6

From the above results it can be concluded that addition of polyelectrolyte is small quantities to $Alcl_3$ or $Fecl_3$ gives good performance in reducing the level of BOD, COD, total nitrogen and ammonical nitrogen, than when $Alcl_3$ or $Fecl_3$ are used alone.

### 3. Effect of aeration on $AlCl_3$ – polyelectrolyte – lime treated effluent:

The raw effluent was treated with $Alcl_3$ – polyelectrolyte mixture, followed by lime, when by a major portion of the pollutants in wastewater was precipitated. After sedimentation, this semi treated effluent was subjected to aeration for different periods, in aerobic bacteria. The results obtained in the reduction of COD and BOD is shown in figures 1 & 2 respectively. The nature of both the curves suggests that there is an induction period of about 12 hrs, during which period there is no any appreciable reduction. Beyond 12 hrs period, reduction is very rapid up to about 30 hrs and beyond 30 hrs, the reductions may is very negligible, and COD and BOD levels have reached low levels.

Even though, air is mechanically incorporated into wastewater, appreciable reduction is observed from 12 hours onwards. This is believed that the microorganisms inoculated into the water and their activity reaches only after this induction period. 30-hrs aeration is sufficient to bring down. BOD and COD levels to meet the requirements of the pollution control board.

### 4. Effect of aeration on Fecl$_3$ - polyelectrolyte – lime treated effluent:

The results obtained on the reduction in COD and BOD due to aeration of Fecl$_3$ - polyelectrolyte – lime treated effluent are presented in the figure 3 and 4. The found on reduction in BOD and COD are similar to that of Alcl$_3$ - polyelectolyte – lime system. The reduction in both BOD and Cod during the initial hours is small; reduction is highest in 12 – 24 hours period and further reduction is small.

The reduction in BOD and COD of the wastewater by aeration is due to oxidation of dissolved / suspended pollutants mainly to $CO_2$ and $H_2O$ and small amounts of oxides of sulphur and nitrogen. Oxides of carbon, nitrogen and sulphur mostly escape into the atmosphere.

A comparison between Alcl$_3$ - polyelectrolyte –lime and Fecl$_3$ - polyelectrolyte-lime systems suggest that the former is better in performance and 24 – 30 hrs aeration can reduce BOD to levels below the requirements of Pollution Control Board.

## 1. Conventional Effluent processing:

The conventional effluent treatment is more costly because of the long duration in the aeration (more than 3 days) required to fulfil the PCB norms.

1. Lime + Aeration (3days)

**Table: 6.1**

| Parameter | Raw effluent | Treated effluent (aeration for 3 days) |
|---|---|---|
| pH | 1.7 | 7.8 |
| COD | 11340 | 2985 |
| BOD | 7640 | 1710 |
| DS | 23770 | 14480 |
| SS | 1970 | 6300 |
| $S^-$ | T | T |
| $TN_2$ | 1250 | 880 |
| $AN_2$ | 1090 | 750 |
| Oil and grease | 7.0 | 7.0 |

## 2. Addition of individual chemicals:

### i) Addition of $AlCl_3$:

The following treatments are carried out in the study.

1. $AlCl_3$ addition + Lime + No aeration

Screening of $AlCl_3$ as flocculating agent in presence of lime

**Table : 6.2.**

Results:

| Parameter | Raw effluent | After addition of $AlCl_3$ + lime | Reduction in parameters (%) |
|---|---|---|---|
| pH | 2.9 | 8.55 | - |
| COD | 6682 | 2762 | 58.7 |
| BOD | 3136 | 1341 | 57.2 |
| DS | 14022 | 10992 | - |
| SS | 143 | 141 | - |
| $S^-$ | 2.5 | T | - |
| $TN_2$ | 834 | 716 | 14.2 |
| $AN_2$ | 764 | 480 | 37.2 |

**3. Addition of $FeCl_3$:**

The following treatments are been carried out in the study.

1.$FeCl_3$ addition + Lime + No aeration

Screening of $Fecl_3$ as flocculating agent in presence of lime.

**Table: 6.3**

Results

| Parameter | Raw effluent | After addition of $Fecl_3$ + lime | Reduction in parameter (%) |
|---|---|---|---|
| pH | 2.80 | 8.00 | - |
| COD | 3927 | 1360 | 65.4 |
| BOD | 1260 | 560 | 55.6 |
| DS | 6520 | 44440 | - |
| SS | 360 | 310 | - |
| $S^-$ | T | T | - |
| $TN_2$ | 684 | 423 | 38.2 |
| $AN_2$ | 570 | 200 | 64.9 |

**4. Addition of alum:**

The following treatments are been carried out in the study.

1. Alum addition + lime + No aeration.

Screening of alum as flocculating agent in presence of lime

Results:

**Table : 6.4**

| Parameter | Raw effluent | After addition of alum + lime | Reduction in parameter (%) |
|---|---|---|---|
| pH | 3.5 | 8.05 | - |
| COD | 5471 | 2937 | 46.0 |
| BOD | 2420 | 1325 | 45.0 |
| DS | 12495 | 9653 | - |
| SS | 786 | 93 | - |
| $S^-$ | 0.6 | T | - |
| $TN_2$ | 1725 | 847 | 50.8 |
| $AN_2$ | 792 | 765 | 3.4 |

**5. Addition of polyelectrolyte:**

The following treatments are been carried in the study.

1. Polyelectrolyte addition + Lime + no aeration.

Screening of polyelectrolyte as flocculating agent in presence of lime:

**Table 6.5**

Results

| Parameter | Raw effluent | After addition of polyelectrolytes + lime | Reduction in parameter (%) |
|---|---|---|---|
| pH | 3.0 | 6.75 | - |
| COD | 17655 | 6344 | 64.1 |
| BOD | 8270 | 4290 | 57.8 |
| DS | 30002 | 17196 | - |
| SS | 6637 | 1140 | - |
| $S^-$ | 0.09 | T | - |
| $TN_2$ | 2393 | 916 | 61.7 |
| $AN_2$ | 2345 | 1384 | 41.0 |

**6. Addition of mixed sulphates:**

The following treatment are been carried out in the study.

1. Mixed sulphated addition + lime + no aeration

Screening of mixed sulphates as flocculating agent in presence of lime:

**Table : 6.6**

Results:

| Parameter | Raw effluent | After addition of mixed sulphates + lime | Reduction in parameter (%) |
|---|---|---|---|
| pH | 5.05 | 7.5 | - |
| COD | 2447 | 982 | 60.0 |
| BOD | 1300 | 741 | 43.0 |
| DS | 9381 | 2932 | - |
| SS | 640 | 101 | - |
| $S^-$ | 93 | 0.05 | - |
| $TN_2$ | 814 | 418 | 48.7 |
| $AN_2$ | 749 | 619 | 17.4 |

## 7. Evaluation of synergetic action of flocculating agents

$Alcl_3$ + Polyelectrolyte + lime

**Table: 6.7**

Results:

| Parameter | Raw effluent | AlCl3 + polyelectrolyte + addition of lime | Reduction in parameter (%) |
|---|---|---|---|
| pH | 2.6 | 7.55 | - |
| COD | 11454 | 3846 | 66 |
| BOD | 5150 | 1638 | 68 |
| DS | 39267 | 15061 | - |
| SS | 3385 | 591 | - |
| $S^-$ | 1.7 | T | - |
| $TN_2$ | 5317 | 1726 | 67.5 |
| $AN_2$ | 2.404 | 1449 | 40 |

## 8. Evaluation of synergetic action of flocculating agents:

$FeCl_3$ + polyelectrolyte + lime

**Table : 6.8**

Results:

| Parameters | Raw effluent | $FeCl_3$ + polyelectrolyte + lime | Reduction in parameters (%) |
|---|---|---|---|
| pH | 2.2 | 8.85 | - |
| COD | 9090 | 2644 | 70.9 |
| BOD | 4680 | 1220 | 73.9 |
| DS | 55355 | 37223 | - |
| SS | 784 | 81 | - |
| $S^-$ | 26 | T | - |
| $TN_2$ | 3061 | 1326 | 56.7 |
| $AN_2$ | 1294 | 635 | 50.9 |

**9. Evaluation of synergetic action of flocculating agents**

Alum + polyelectrolyte + lime

Result:

**Table : 6.9**

| Parameters | Alum + polyelectrolyte + lime | | Reduction in parameters ( % ) |
|---|---|---|---|
| | Raw effluent | Semi | |
| pH | 5.3 | 6.7 | - |
| COD | 14350 | 5442 | 62.1 |
| BOD | 9590 | 4478 | 53.3 |
| DS | 30524 | 12700 | - |
| SS | 973 | 81 | - |
| $S^-$ | 19 | T | - |
| $TN_2$ | 1944 | 849 | 56.3 |
| $AN_2$ | 3102 | 1612 | 48 |

**10. Evaluation of synergetic action of flocculating agents**

Mixed sulphates + Polyelectrolyte + lime

**Table: 6.10**

Results:

| Parameters | Mixed sulphates+ polyelectrolyte + lime | | Reduction in parameters (%) |
|---|---|---|---|
| | Raw effluent | Semi | |
| pH | 3.9 | 6.15 | - |
| COD | 18125 | 8121 | 55.2 |
| BOD | 9680 | 7874 | 18.7 |
| DS | 59187 | 16191 | - |
| SS | 6639 | 6305 | - |
| $S^-$ | 38 | T | - |
| $TN_2$ | 3360 | 1341 | 60.1 |
| $AN_2$ | 4315 | 3250 | 24.7 |

**Effect of duration of aeration on flocculent treated effluent in Alcl$_3$ + Polyelectrolyte + lime system:**

**Table: 1**

| Time | COD | BOD |
|---|---|---|
| 0 | 5268 | 4925 |
| 12 | 4924 | 4760 |
| 20 | 1191 | 507 |
| 24 | 544 | 233 |
| 30 | 352 | 150 |
| 32 | 102 | 11 |
| 34 | 60 | 17 |
| 36 | 49 | 38 |

**Effect of duration of aeration on flocculent treated effluent in Fecl$_3$ - polyelctrolyte-Lime system.**

**Table: 2**

| Time | COD | BOD |
|---|---|---|
| 0 | 5460 | 5030 |
| 12 | 5062 | 4826 |
| 20 | 1206 | 547 |
| 24 | 6035 | 252 |
| 30 | 276 | 172 |
| 32 | 160 | 126 |
| 34 | 82 | 56 |
| 36 | 36 | 22 |

# Chapter – 7

## 7.1. Effect of flocculating system on Rate of sedimentation of sludge:

The speed with which the precipitated sludge sediments in the clarifier is very important in deciding the size of clarifier and the retention period. Studies were carried out by using systems $Alcl_3$ - polyeletrolyte – lime and $Fecl_3$ – polyelectrolyte – lime. The results obtained are given in Figure 5 and 6. At equal concentrations of a flocculating chemicals, it is seen that the overall rate of sedimentation is higher for $AlCl_3$, – polyelectrolyte- lime system. However, as time proceeds, the difference in the rate narrows down. The higher rate of sedimentation in the Alcl3 system is due to the low viscosity of clear serum after precipitation of a higher level of pollutants, compared to that in the Fecl3 system. This was verified by actual measurement of viscosity of the clear serum in the two cases and the viscosity data are presented in the table 7.1 below.

**Viscosity of semi treated effluent:**

**Table : 7.1**

| System | Viscosity (cps ) |
|---|---|
| **$AlCl_3$, – Polyelectrolyte – lime** | **19.0** |
| **$Fecl_3$ - Polyelectrolyte – lime** | **22.0** |

**Rate of sedimentation of sludge:**

$AlCl_3$ + polyelectrolyte +lime system

**Table : 7.2**

| Time (min) | Reduction in depth (cm) |
|---|---|
| 0 | 0 |
| 2 | 1.4 |
| 4 | 5.2 |
| 6 | 8.2 |
| 8 | 10.6 |
| 10 | 12.5 |
| 12 | 13.7 |
| 14 | 15.0 |
| 16 | 15.8 |
| 18 | 16.6 |
| 20 | 16.9 |
| 22 | 1.3 |
| 24 | 17.6 |
| 26 | 17.8 |
| 28 | 18.1 |
| 30 | 18.2 |

**Rate of sedimentation of sludge:**

Fecl$_3$ + polyelectrolyte + lime system

**Table: 7.3**

| Time (min) | Reduction in depth (cm) |
|---|---|
| 0 | 0 |
| 2 | 1.3 |
| 4 | 2.8 |
| 6 | 4.3 |
| 8 | 5.9 |
| 10 | 8.1 |
| 12 | 9.9 |
| 14 | 11.8 |
| 16 | 13.3 |
| 18 | 14.9 |
| 20 | 15.8 |
| 22 | 16.5 |
| 24 | 17.0 |
| 26 | 17.3 |
| 28 | 17.5 |
| 30 | 17.7 |

### 7.2. Effect of pH on the rate of sedimentation of sludge:

In order to standardise the optimum amount of lime necessary to cause precipitation and sedimentation of sludge in presence of $AlCl_3$ – polyelectrolyte –lime and $FeCl_3$–polyelectrolyte – lime. The pH of the effluent being treated was varied by adding different quantities of lime. The data obtained from the former system is given in the figure 7 and the later in figure 8. It is observed that for both the systems, an increase in pH of the aqueous phase causes a reduction in the rate of sedimentation. Increase in pH is due to addition of higher amounts of lime. It is believed that addition of higher amounts of lime ($Ca(OH)_2$) causes an increase in the ionic concentration in the aqueous phase and consequently can increase in viscosity. This causes reduction in the rate sedimentation of precipitated materials.

The result further suggests that at pH near to neutral, the rate of sedimentation is higher for the $AlCl_3$ – polyelectrolyte – lime system. At higher pH's the rates of both the systems are almost comparable.

From the economic point of view, the cost of effluent treatment can be maintained at a minimum by limiting the addition of lime ( ie, low pH ) and increase in rate of sedimentation ( again at low pH) ie, by maintaining the pH of the effluent being treated, at about 7.5 – 8.0.

**Rate of sedimentation at pH 7.6, 8.1, 8.8 in Alcl3 – polyelectrolyte – lime system:**

**Table :7.4**

| **Time** | **Reduction in depth at pH** | | |
|---|---|---|---|
| | 7.6 | **8.1** | **8.8** |
| **0** | **0** | **0** | **0** |
| **5** | **12.2** | **8.4** | **4.3** |
| **10** | **16.8** | **12.8** | **8.9** |
| **15** | **20.4** | **15.3** | **12.6** |
| **20** | **22.7** | **16.8** | **15.0** |
| **25** | **25.1** | **18.0** | **16.5** |
| **30** | **26.9** | **18.4** | **17.4** |

### 7.5 Rate of sedimentation at pH 7.5, 8.2. 9.0 in Fecl₃ – polyelectrolyte – lime system

Table 7.5

| Time | Reduction in depth at pH | | |
|---|---|---|---|
| | 7.5 | 8.2 | 9.0 |
| 0 | 0 | 0 | 0 |
| 5 | 10.6 | 6.4 | 4.1 |
| 10 | 19.8 | 12.9 | 8.0 |
| 15 | 22.6 | 16.6 | 12.3 |
| 20 | 23.7 | 18.2 | 14.7 |
| 25 | 24.8 | 19.4 | 16.2 |
| 30 | 25.4 | 20.1 | 16.8 |

# Chapter –8

## 8.1 Factory scale studies:

Based on the results obtained in the laboratory scale studies conducted above, factory scale trails where conducted at 3 factories. The treatment followed on addition of $AlCl_3$ – polyelectroyte as flocculating agents; lime for causing precipitation at pH 8.0 followed 24 hours aeration. The properties of the raw effluent before treatment and after treatment are given in table 8.1.The values reported are the mean of 3 determinations.

The values obtained for various parameters on treated effluent clearly suggests that $AlCl_3$ – polyelectrolyte – lime treatment of followed 24 hrs aerations bring the parameters well with in the limits prescribed by the Kerala state pollution control board.

**Results of Factory scale studies**

**Table : 8.1**

| Parameter | A | | B | | C | |
|---|---|---|---|---|---|---|
| | BT | AT | BT | AT | BT | AT |
| PH | 3.8 | 7.1 | 4.0 | 6.7 | 4.7 | 7.3 |
| COD | 4252 | 185 | 17144 | 49 | 11454 | 162 |
| BOD | 3968 | 46 | 15350 | 13 | 5150 | 22 |
| $TN_2$ | 1837 | 62 | 3301 | 32 | 2212 | 78 |
| $AN_2$ | 1446 | 39 | 2612 | 28 | 1404 | 42 |
| TS | 13608 | 1369 | 41213 | 1480 | 39267 | 1965 |

**Where, COD – Chemical Oxygen Demand**

**BOD – Bio chemical Oxygen Demand**

**$TN_2$ - Total Nitrogen.**

**$AN_2$ - Ammonical Nitrogen**

**TS - Total solids**

**BT – Before Treatment**

**AT – After treatment**

### 8.2. Cost aspects on effluent treatment:

The cost aspects of effluent treatments also are evaluated. The expenditure for treating 100 $M^3$ of effluent on conventional method and the present modified methods are given in table 8.2. The results suggest that the overall expenditure on effluent processing is lower in the modified method.

**Table:8.2**

| Item | Expediture for 100$M^3$ waste water (Rs) | |
|---|---|---|
| | Conventional method | Modified method |
| Labour | 450 | 350 |
| Chemicals | 150 | 250 |
| Electricity | 800 | 200 |
| Total | 1400 | 800 |

FIGURE : 1/2

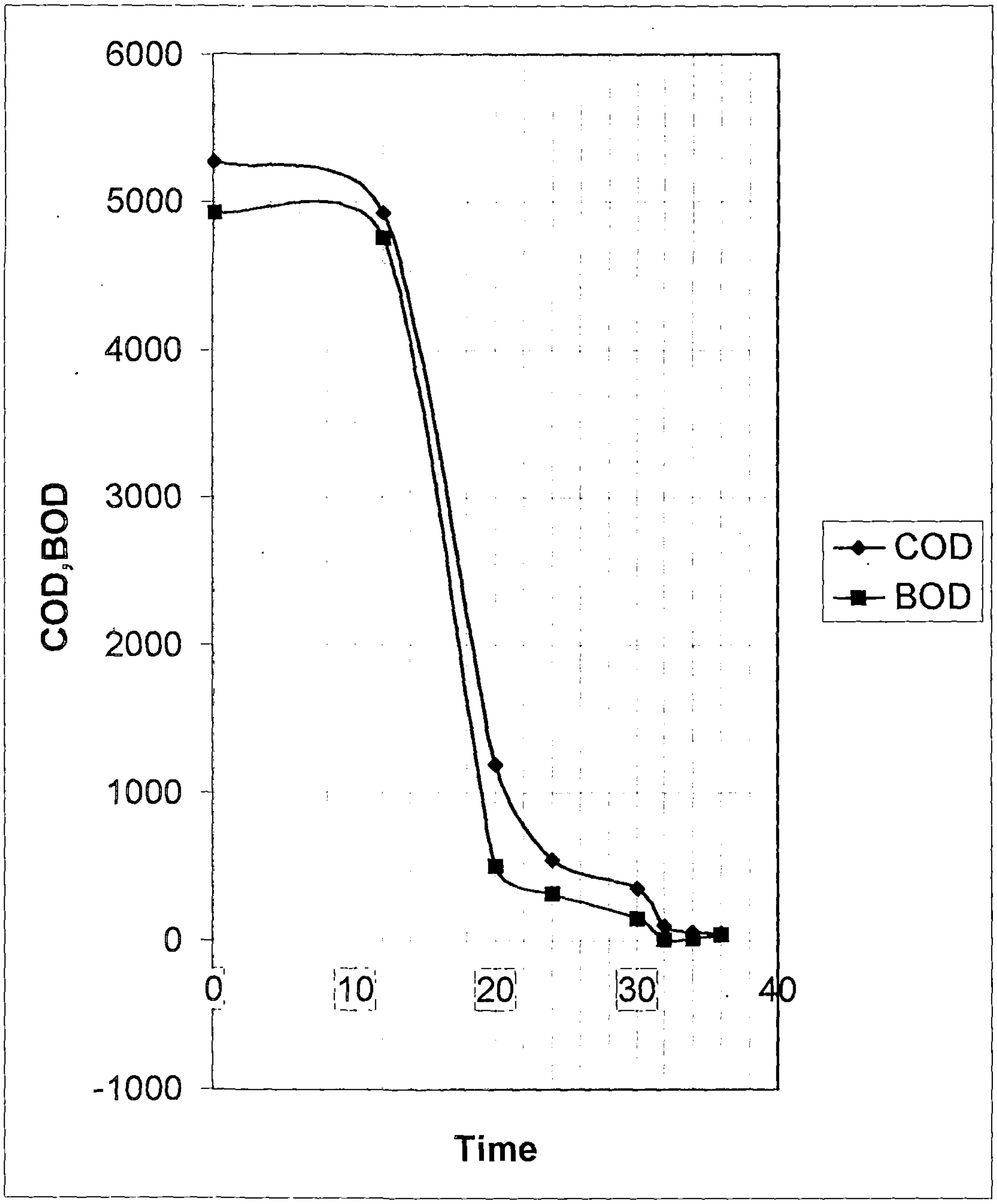

6000
5000
4000
3000
2000
1000
0
-1000
COD,BOD
0
10
20
30
40
Time
COD
BOD

FIGURE: 3/4

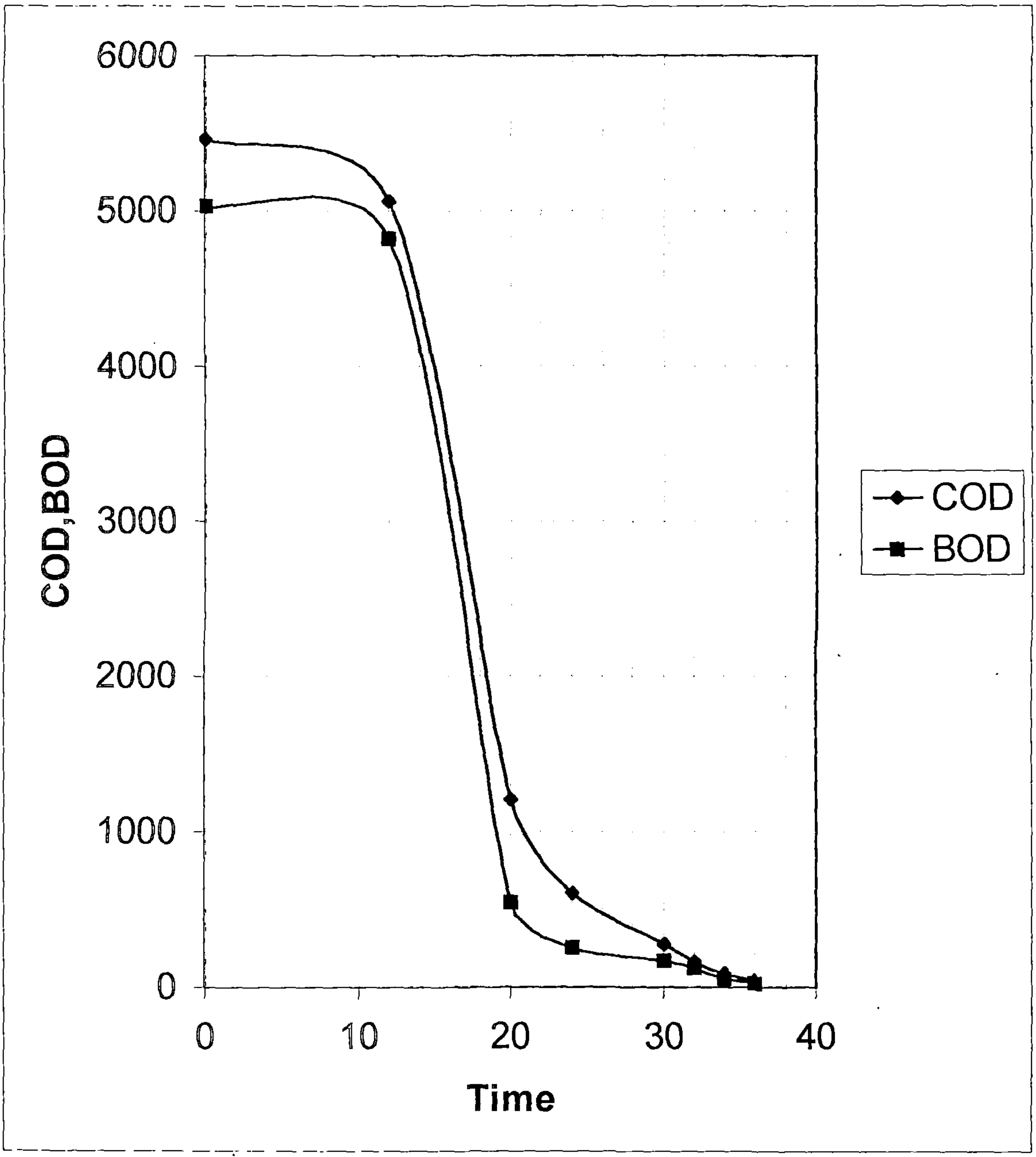

6000
5000
4000
3000
2000
1000
0
COD,BOD
0
10
20
30
40
Time
COD
BOD

FIGURE: 5/6

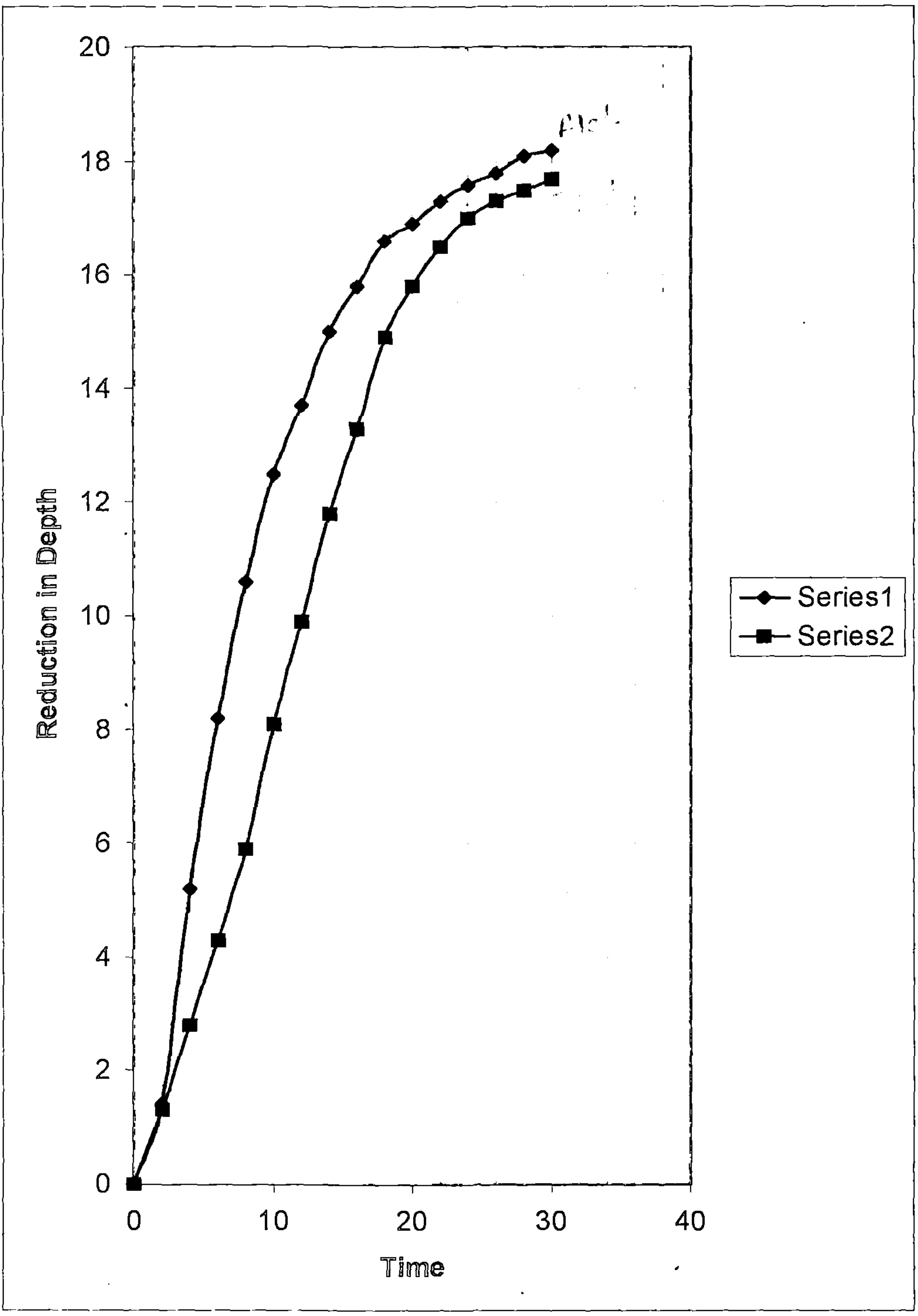

Reduction in Depth
20
18
16
14
12
10
8
6
4
2
0
0
10
20
30
40
Time
Series1
Series2

FIGURE: 7

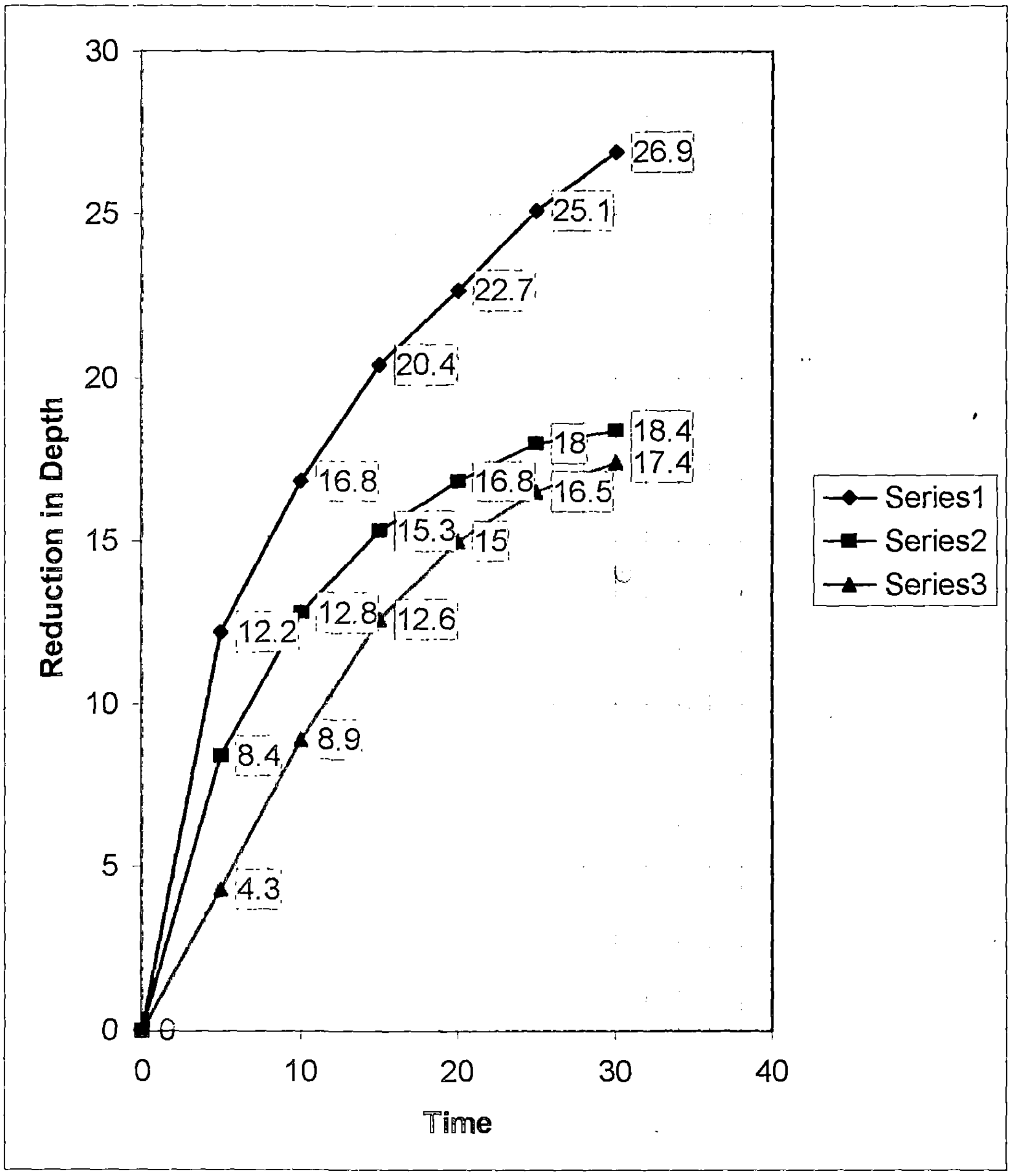
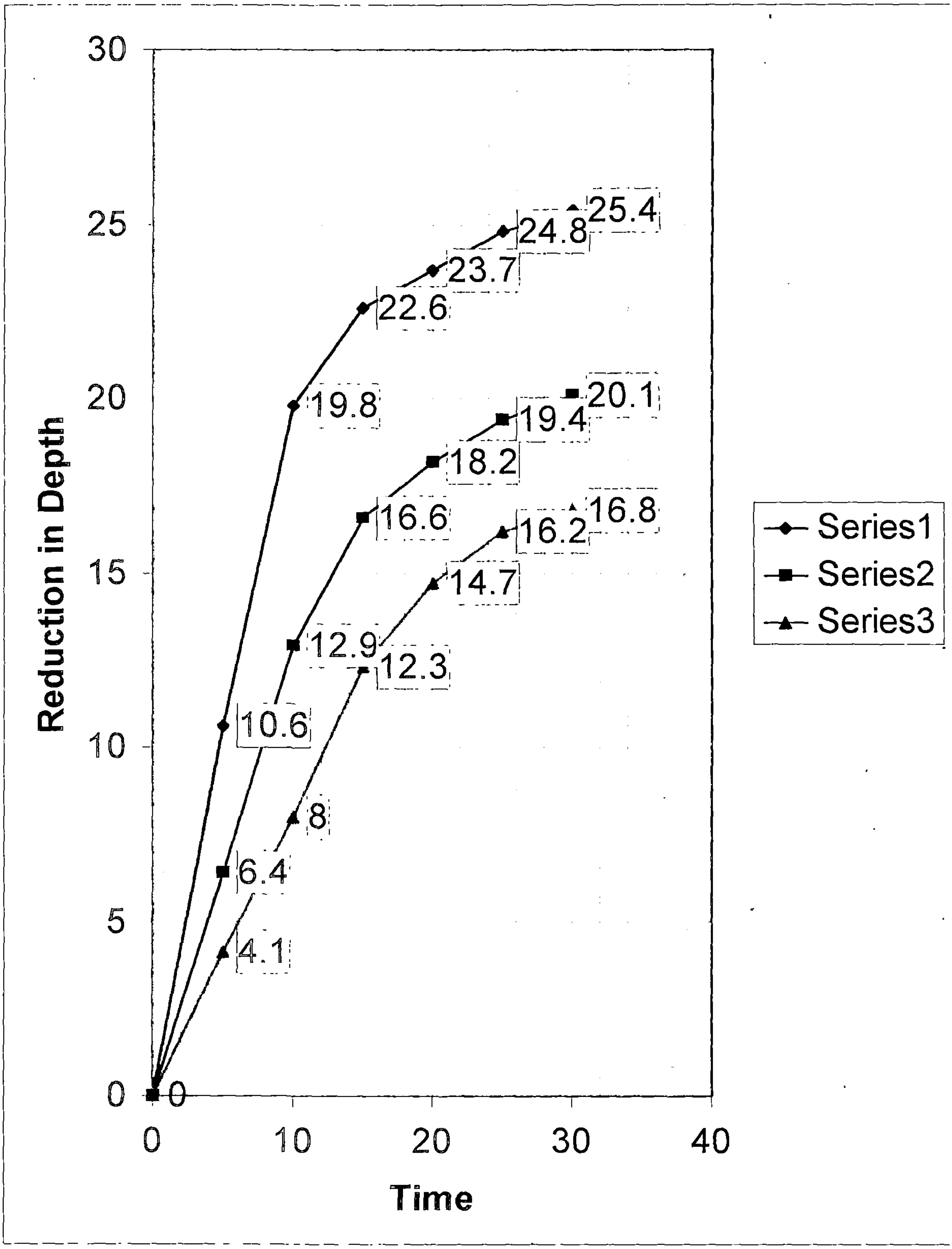

30
25
20
15
10
5
0
Reduction in Depth
0
10
20
30
40
Time
26.9
25.1
22.7
20.4
16.8
12.2
0
18.4
18
16.8
15.3
12.8
8.4
17.4
16.5
15
12.6
8.9
4.3
Series1
Series2
Series3

FIGURE : 8



Reduction in Depth
Time
0
5
10
15
20
25
30
0
10
20
30
40
0
10.6
19.8
22.6
23.7
24.8
25.4
6.4
12.9
16.6
18.2
19.4
20.1
4.1
8
12.3
14.7
16.2
16.8
Series1
Series2
Series3

## CONCLUSIONS:

Major conclusions that are obtained from the study are summarised as follows

- Aluminium chloride and Ferric chloride act as good flocculating agents in presence of lime (Ca (OH2) to precipitate and remove pollutant's present in waste water generated in latex centrifuging factories.
- The activity of $AlCl_3$, $Fecl_3$ are enhanced in the presence of polyelectrolyte .
- Among $Alcl_3$- polyelectrolyte – lime and $Fecl_3$ – polyelectrolyte – lime systems, the performance of the former system is better in removal of pollutants
- Short-term aeration of 24 hours is sufficient for the above effluent being treated to bring the parameters within the specifications of the Pollution Control Board. In the conventional methods, even 72 hours aeration is inadequate.
- Rate of sedimentation of precipitated sludge is higher for $Alcl_3$ – polyelectrolyte – lime system compared to $Fecl_3$ – polyelectrolyte – lime system
- Rate of sedimentation of precipitated sludge decreases with increases in pH. PH about 7.5 to 8.0 is found to be optimum.
- In the factory scale trials, treatment of raw effluent with $Alcl_3$ – polyelectrolyte – lime system ( at pH 7.5) followed by 24 hours aeration brings the final treated effluent with in the specifications of the Kerala State Pollution Control Board.
- The cost of $Alcl_3$ – polyelectrolyte –lime addition, followed by 24 hours aeration system is lower than the convention method.

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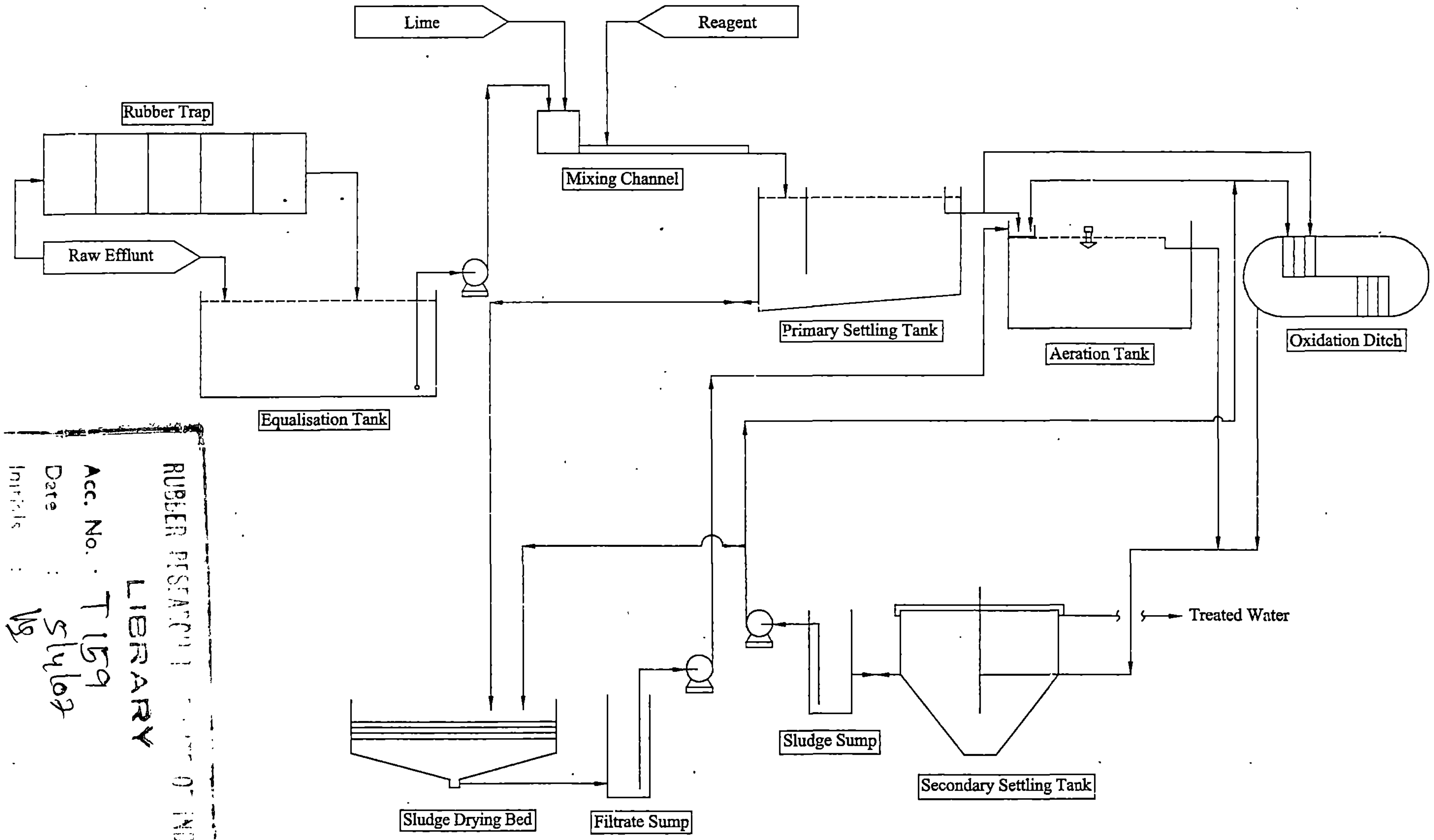

**FLOW DIAGRAM E. T. P.**