# PHOSPHORUS RECOVERY FROM WASTE WATER BY USING MAGNESIUM AND CALCIUM

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## ABSTRACT

Phosphate rock ore reserves are estimated to be completely used up in upcoming 50 to 100 years. For phosphorus fertilizer production phosphate rock ore is the single source around the world. The agriculture production will be negatively affected if this phosphate rock ore reserves are exhausted. At present there is no alternative phosphorus source. Waste water treatments facilities consist of phosphorus are directly disposed into the environment, and it leads to pollution problems. Struvite and hydroxyapatite precipitation system is the process of recovering phosphorus from waste water treatment and it leads to decrease the deficiency of future scarcity of phosphorus. In present project, it was discussed about assessment of recovery of phosphorus from waste water treatment plant through struvite and hydroxyapatite precipitation and determination of potential for the recovered material to serve as a satisfactory material for phosphate rock substitute. Phosphates are the chief sources of nutrients in waste water, and it leads to eutrophication of water bodies. By using conventional technologies for the removal of these nutrients from waste water is a major challenge, hence different processes are developed for the removal of these nutrients. Phosphorus in nature is not infinite, which will be running out in about 50 to 100 years. Therefore, phosphorus recycling is an important issue as well as challenge for researchers all around the world.

In present project it was investigated that phosphorus recovery from chemical precipitation technology by the precipitation of calcium ammonium phosphate (hydroxyapatite) or magnesium ammonium phosphate (struvite) which is a valuable product and nutrient fertilizer for agriculture production. By using this method not only recovery of nutrients like phosphorus and ammonia, it also minimizes the eutrophication.

**Keywords:** Magnesium Ammonium Phosphate (MAP), Hydroxyapatite (HAP), Batch Reactor, Dairy wastewater, Crystallization, Biochemical Oxygen Demand (BOD), Chemical Oxygen Demand (COD).

## INTRODUCTION

Global population was reached 7 billion on October 2011. The present Global population was estimated to reach 7.5 billion as on January 2017 and it may increase in between 8.4 to 10.8 billion by the year of 2050. This is probably result in increasing the requirement of resources like Food, water, energy and infra structure and at the same time it will also increases the production of waste with increasing the GDP per capita. The production of phosphorus was peaked already as per 2011, leading to probable global shortage of phosphorus by 2045. Peak phosphorus use will occur in upcoming 30 years and it was estimated that at the present rates, reserves will be exhausted by next 75 - 100 years [11]. For the agricultural production Phosphorus (P) is essential and it was mined from the phosphorus rock. Phosphorus (P) is a very important constituent of each breathing cell or organism along with it was furthermore significant for the development of bones and teeth. Phosphorus contained in the foodstuff and it was mainly uptake by the plant life (as of fertilizers and usual earth Phosphorus content) for the duration of agricultural production, in addition to animals (in the appearance of animal nourish), and Phosphorus was supplementary in the form of foodstuff additives (example- process food and snacks). At the same time as the global population has been greater than before, Phosphorus will be progressively more used in the foodstuff creation and conservation system, furthermore other purpose like detergents production. Therefore, reserves of phosphate rock exert an important require on worldwide. For the developing (like India) and developed (like U.S.A.) countries there will be an increasing pressure on phosphate availability since human beings needs foodstuff for continued existence. Therefore, it is very important to initiate performance of 'waste' as a precious source rather than simply throwing away. In natural water the phosphorus was typically synchronized by microbes accordingly therefore phosphorus availability and environment necessities will be balanced. On the other hand, if the Phosphorus input was exceeding the eco-system utilization rate, then finally problem of increasing concentration of phosphorus will be appeared. Surplus of phosphorus will stimulate the algae growth in lakes and ponds. Excess phosphorus will be in twirl decreasing oxygen consideration and it lead to cause eutrophication of surface water bodies. If the concentration of phosphorus was increased it will also result in higher water treatment costs. Recycling of phosphorus is significant one as the phosphorus is nonrenewable constituent. Due to lack of phosphorus recovery technology, massive amount of phosphorus was lost yearly. Therefore, it is inevitable finding many ways to use phosphorus more efficiently as well as finding ways to recycle the phosphorus. Present methods for the removal and recovery of phosphorus which were used in the wastewater management amenities encompass several strong drawbacks. Current waste water management system will cause dissimilar environment harms and in addition to restrictions of phosphorus recycling.

Present work studies on the dissimilar aspects of Struvite and/ or hydroxyapatite precipitation technology which is the best alternative for recovery of phosphorus from waste water.

## STUDY AREA

Dairy industry is considered as one of the important contributors to the water pollution. The wastewater generated from Dairy industries is due to washing of equipment, milk spillage, detergents, and other products of milk waste. Dairy industry effluent is principally biodegradable and dairy industry contains an appreciable quantity of oil and it produces an undesirable odor and can have adverse effect on environment due to high BOD, COD, TSS and nutrients (nitrogen and phosphorus). In addition to the recycling of wastewater contribution, dairy industry wastewater also contains high levels of phosphorus (P). Its recovery from municipal and some of the industries would be beneficial in terms of both resource conservation and pollution reduction.

In the present study, the Phosphorus removal strategy was precipitated by using a divalent cation (Ca or Mg) in contrast to more conventional approaches which uses the addition of aluminum (Al) or iron (Fe) salts. At present reuse options for Fe or Al phosphate materials are limited due to the strong chemical (ionic) bonds formed, however the divalent cation (calcium and magnesium) bonding allows a greater proportion of phosphorus recovery to be available for the support of plant growth. Heritage Dairy industry was located 45 km. from Kakinada city, East

Godavari district, A.P. This project deals with the Phosphorus Recovery from dairy effluent by using Magnesium and calcium. The sample was collected after Equalization tank of three different days in the month of March, April and May 2017.

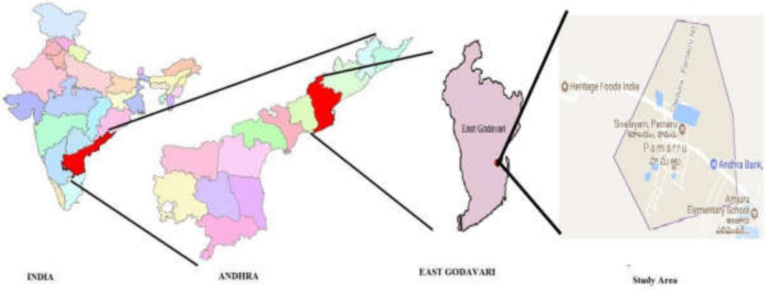


**Figure 1.** Dairy plant

**Figure 2.** Study area

## OBJECTIVES OF STUDY

1. To investigate the potential to recovery of Phosphorus from waste water and thereby protecting as a useful resource.

2. To recognition as well as optimization of operating parameters of Struvite and hydroxyapatite precipitation process for Phosphorus recovery in a laboratory scale system.

## MATERIALS AND METHODS

### Methodology

The setup of laboratory scale experiment was as follows:

1. The precipitation of Magnesium ammonium phosphate and Calcium ammonium phosphate was done by using jar test.
2. With diluted HCL solution, the jars were cleaned and washed with deionized water. At room temperature these jars were allowed for drying.
3. Taken one liter of waste water to each jar.
4. The pH of wastewater (9 to 10) was adjusted by using sodium hydroxide (NaOH) solution.
5. Magnesium chloride (30%) solution for struvite precipitation or calcium chloride (30%) solution for hydroxyapatite precipitation were added into the jar and mixing of wastewater was done with the mechanical mixer until the whole solution of magnesium chloride or calcium chloride was finally added into the waste water.
6. Firstly, rapid mixing was done at 100 rpm for 5 minutes up to the mixing of entire solution.
7. Solution (1000 ml) are allowed for the precipitation of struvite or Hydroxyapatite in a time of reaction up to one hour at a low stirring rate of 60 rpm by using a digital control stabilized Wise Stir jar tester for considerable struvite or Hydroxyapatite precipitation.

8. Solid product was crystallized without disturbance for two hours.

9. By using a vacuum filtration system, filter the mixture is separated from the liquid phase by using a setup of glass vacuum with a vacuum pump (Rocker 400).
10. By using spectrophotometer, the filtrate was analyzed for remaining phosphorus $(PO_4)$ ions.
11. By using what man filter paper No. 42, the precipitate was filtered.
12. Filtrate kept for Desiccator for 10 hours for drying without interfering air.
13. At room temperature finally the filtrate was air dried.
14. Finally precipitated Magnesium ammonium phosphate particles or Calcium ammonium phosphate particles was aggregated and it was finally grown with the sludge which was separated by hand crushing or by sieving with 43 to 63 µm Standard sieve and finally it resulted magnesium ammonium phosphate (struvite) or Calcium ammonium phosphate (hydroxyapatite).
15. Experimental setup is shown in the figure 4.2.

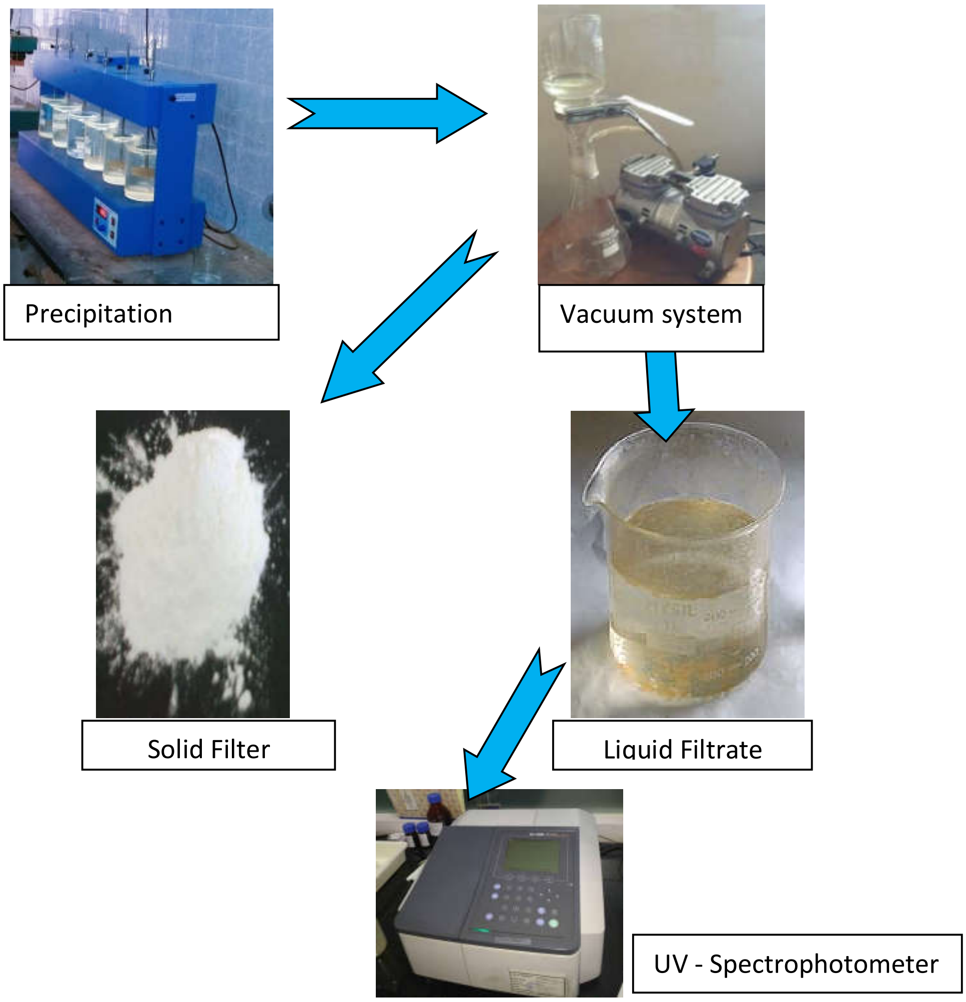


**Figure 3**. Experimental and analytical setup for struvite and hydroxyapatite precipitation

**Analytical Methods**

The raw and treated samples was collected and analyzed. In this study, the samples were analyzed for pH, TSS, COD, BOD, Phosphates and Ammonia.

### Sampling of Dairy Waste Water

The sample was collected in plastic cans of capacity of one liter and it is labeled and stored in cool place (at $4^0$c). The cans were thoroughly rinsed twice with raw sample. The waste water samples were collected for each set of experiment for analysis; one sample was collected before conducting the jar test and another one sample was collected after precipitation of magnesium ammonium phosphate or calcium ammonium phosphate.

### Precipitation of Struvite and Hydroxyapatite

The precipitation of Magnesium ammonium phosphate and Calcium ammonium phosphate was done by using jar test. The jars were cleaned with the help of diluted solution of HCL and washed with deionized water. These jars were dried at room temperature. The jars were filled with dairy wastewater and increased pH to 9 with the help of sodium hydroxide solution for optimum removal of phosphorus in the form of various compounds.

### Precipitation of magnesium ammonium phosphate (Struvite)

After increasing the pH of Dairy wastewater by 9 by using sodium hydroxide solution, 30% solution of Magnesium chloride was added into the jar and mixing of wastewater was done with the mechanical mixer till the whole magnesium chloride solution was added finally into the wastewater. The Hydraulic retention time was maintained by one Hour. After completion of one hour HRT entire solution were set for 2 hours for formation of struvite precipitate at the base of Jar. By using what man filter paper No. 42, the precipitate was filtered and it is kept in Desiccator for ten hours. Ultimately filtrate was air dried at room temperature. Finally precipitated Magnesium ammonium phosphate particles were aggregated and it was grown with sludge which was finally separated by hand picking or by sieving with 43 to 63 µm Standard sieve and finally it was resulted magnesium ammonium phosphate (struvite).

### Precipitation of Calcium ammonium phosphate (Hydroxyapatite)

After increasing the pH of Dairy wastewater by 9 by using sodium hydroxide solution, 30% solution of Calcium chloride was added into the jar and mixing of wastewater was done with the mechanical mixer till the whole Calcium chloride solution was added finally into the wastewater. The Hydraulic retention time was maintained by one Hour. After completion of one hour HRT entire solution were set for 2 hours for formation of Hydroxyapatite precipitate at the base of Jar. By using what man filter paper No. 42, the precipitate was filtered and it is kept in Desiccator for ten hours. Ultimately filtrate was air dried at room temperature. Finally precipitated Calcium ammonium phosphate particles were aggregated and it was grown with sludge which was finally separated by hand picking or by sieving with 43 to 63 µm Standard sieve and finally it was resulted Calcium ammonium phosphate (Hydroxyapatite).

**Figure 4.** Conducting laboratory experiments

**Table 1.** Characteristics of dairy wastewater

| **Dairy wastewater characteristics average** | | | |
|---|---|---|---|
| **S.No** | **Characteristics** | **Units** | **Concentration** |
| 1 | pH | | 6.5 |
| 2 | TSS | mg/l | 1008 |
| 3 | C.O.D. | mg/l | 2876 |
| 4 | BOD | mg/l | 1375 |
| 5 | Phosphates ($PO_4$) | mg/l | 135 |
| 6 | Ammonia ($NH_4$) | mg/l | 66 |

## RESULTS AND DISCUSSIONS

The wastewater generated in Dairy industries is due to washing of equipment, milk spillage, detergents, and other products wastes of milk. Dairy industry effluent is principally biodegradable and dairy industry contains an appreciable quantity of oil and it produces an undesirable odor and can have adverse effect on environment due to high BOD, COD, TSS and nutrients (nitrogen and phosphorus).

In present study not only tested phosphorus and ammonia for phosphorus recovery by using struvite and hydroxyapatite precipitation but also considered different parameters like pH, Total suspended solids, BOD, COD for evaluation of Dairy waste water treatment.

### Characteristics of Dairy Wastewater

**Table 2.** Dairy wastewater characteristics on

| Dairy wastewater characteristics | | | | | | |
|---|---|---|---|---|---|---|
| **S.No** | **Characteristics** | **Units** | **19th March** | **24th April** | **2nd May** | **average** |
| 1 | pH | | 6.5 | 6.4 | 6.6 | 6.5 |
| 2 | TSS | mg/l | 1026 | 988 | 1009 | 1008 |
| 3 | COD | mg/l | 3025 | 2682 | 2923 | 2876 |
| 4 | BOD | mg/l | 1423 | 1308 | 1395 | 1375 |
| 5 | Phosphates ($PO_4$) | mg/l | 138 | 126 | 141 | 135 |
| 6 | Ammonia ($NH_4$) | mg/l | 63 | 69 | 66 | 66 |

### pH Optimization

For precipitation reaction pH is a very important key parameter and it is in the form of Magnesium ammonium phosphate and calcium ammonium phosphate.

**Table 3.** Optimization of pH

| Optimization of pH | | | | | | |
|---|---|---|---|---|---|---|
| **pH** | **Phosphates** | | | **Ammonia** | | |
| | **Initial** | **Final** | **% of Removal** | **Initial** | **Final** | **% of Removal** |
| 8 | 138 | 8.5 | 93.8 | 63 | 7.3 | 88.4 |
| 8.5 | 138 | 7.1 | 94.8 | 63 | 6.9 | 89 |
| 9 | 138 | 6.2 | 95.5 | 63 | 5.2 | 91.7 |
| 9.5 | 138 | 5.9 | 95.7 | 63 | 6.8 | 89.2 |
| 10 | 138 | 5.3 | 96.1 | 63 | 7.6 | 87.9 |
| 10.5 | 138 | 5.1 | 96.3 | 63 | 8.9 | 85.8 |

The optimum pH was calculated by conducting jar test in which different pH of waste water was maintained from 8, 8.5, 9, 9.5,10 and 10.5 with the help of NaOH solution. From the experiment results the recovery of phosphorus was increased by increasing pH condition. At the same time the ammonium concentration was increased up to 9 and then decreased.

It was clearly shows that 95.8% of phosphates and 91.7% ammonium were recovered at pH 9. So that pH 9 was selected for optimum recovery of phosphorus in the form of various compounds of magnesium ammonium phosphates and calcium ammonium phosphates.

Therefore, further tests will be conducted at pH 9.

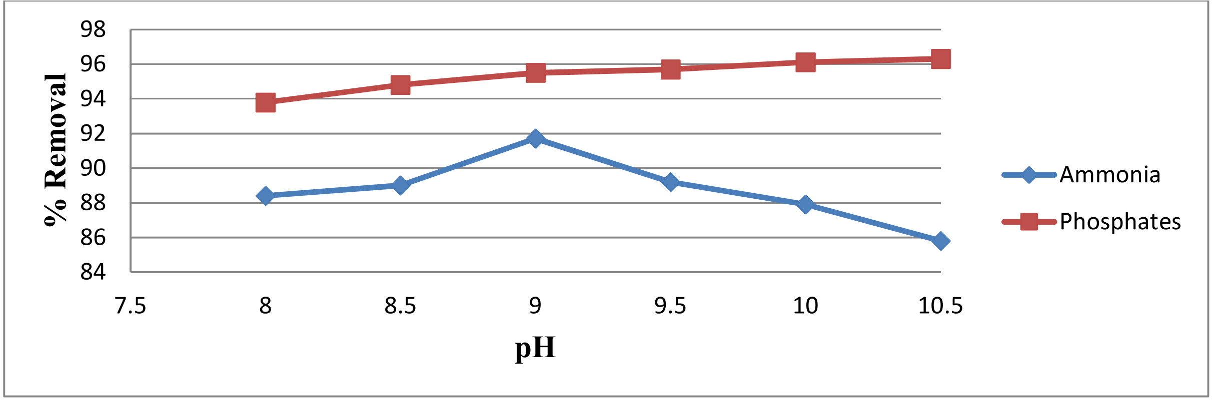
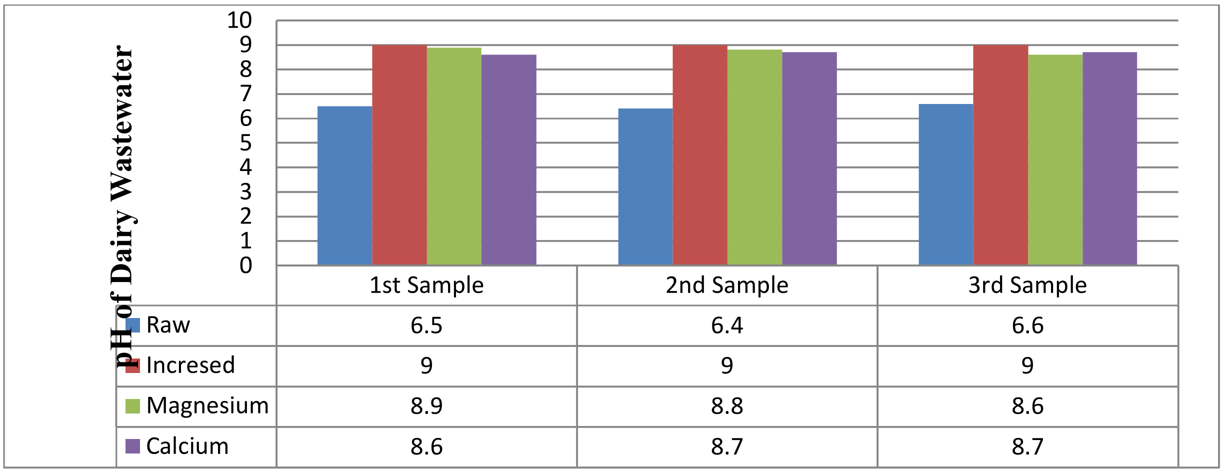


**Figure 5.** Optimization of pH

**Effect on pH when using Magnesium and Calcium**

pH plays a very important role in magnesium ammonium phosphate (Struvite) and Calcium ammonium phosphate precipitation (hydroxyapatite). The Raw dairy waste water of pH was found in the range of 6.4 to 6.6. Initially the pH of dairy waste water increased to 9 with the help of NaOH solution for maximum removal of Phosphorus in the form of various magnesium or calcium compounds. After that 30 percent solution of magnesium Chloride or 30 percent of calcium chloride were added and mixed with raw waste water in this experiment. It shows that when precipitation occurred, the solution of pH was decreased to the range of 8.6 to 8.8 when using magnesium as source, and 8.7 to 8.6 when using calcium as a source from its original range of pH at 9.
The precipitation effect of on the pH is shown in the below figure



| | 1st Sample | 2nd Sample | 3rd Sample |
|---|---|---|---|
| 🟦 Raw | 6.5 | 6.4 | 6.6 |
| 🟥 Incresed | 9 | 9 | 9 |
| 🟩 Magnesium | 8.9 | 8.8 | 8.6 |
| 🟪 Calcium | 8.6 | 8.7 | 8.7 |

**Figure 6.** Effect of magnesium and calcium on pH

**Effect of Magnesium and Calcium on TSS**

In raw Dairy waste water, the total suspended solids concentration was in the range of 988 mg/l to 1026 mg/l. After precipitation of magnesium ammonium phosphate when using magnesium as a source the total suspended solids concentration was decreased to the range of 329 mg/l to 368 mg/l. As in the same manner when precipitation of calcium ammonium phosphate by using calcium as a source the concentration of TSS was decreased to 376 mg/l to 402 mg/l. This was clearly visible that the maximum amounts of colloidal particles were settled at the bottom of the jar. Finally, it was observed that 65.3 % of TSS removal when using magnesium and 61.4% of TSS when using calcium as source.

The effect of precipitation on concentration of Total suspended solids is shown in the below figure.

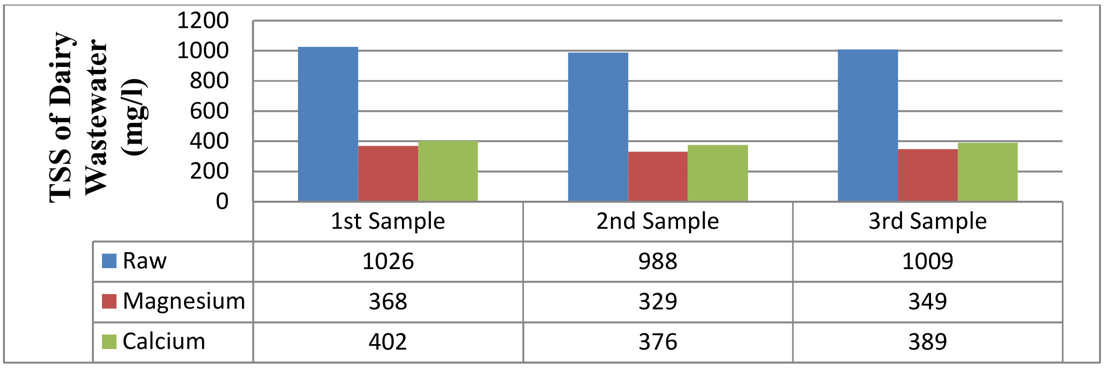


**Figure 7:** The effect of precipitation on concentration of Total suspended solids

### Effect of Magnesium and Calcium on COD

In raw Dairy waste water, the Chemical Oxygen Demand concentration was in the range of 2682 mg/l to 3025 mg/l. After precipitation of Struvite when using magnesium as a source the Chemical Oxygen Demand concentration was decreased to the range of 660 mg/l to 690 mg/l. as in the same manner when precipitation of calcium ammonium phosphate by using calcium as a source the concentration of Chemical Oxygen Demand was decreased to 664 mg/l to 685 mg/l. Finally, it was observed that 76.3 % of COD removal when using magnesium and 76.4% of COD removal when using calcium as source. The wastewater from dairy itself contains bulky number of microbes. These microorganisms consume organic matter of the waste water as food materials. Therefore, it clearly shows that there is sharp decline of COD.

The effect of precipitation on concentration of Chemical Oxygen Demand is shown in the below figure

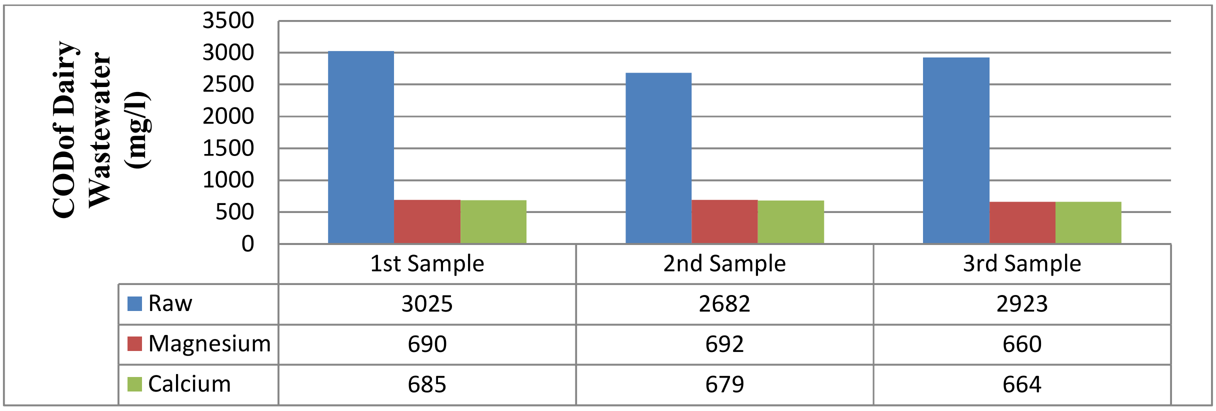


**Figure 8.** The effect of precipitation on concentration of COD

### Effect of Magnesium and Calcium on BOD

In raw Dairy waste water, the Biochemical Oxygen Demand concentration was in the range of 1308 mg/l to 1423 mg/l. After precipitation of magnesium ammonium phosphate, the concentration of Biochemical Oxygen Demand was decreased to in the range of 279 mg/l to 298 mg/l when using magnesium as a source. As in the same manner when precipitation of calcium ammonium phosphate the concentration of Biochemical Oxygen Demand was decreased to 286 mg/l to 302 mg/l by using calcium as a source. Finally, it was observed that 78.5% BOD was removed. The dairy wastewater itself contains bulky number of microbes. These microorganisms consume organic matter of the waste water as food materials. Therefore, it clearly shows that there is sharp decline of BOD.

The effect of precipitation on concentration of Biochemical Oxygen Demand is shown in the below figure

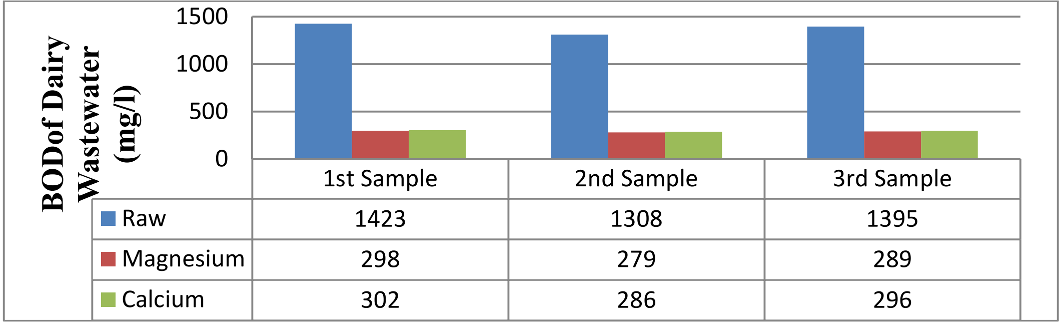


**Figure 9.** The effect of precipitation on concentration of Biochemical Oxygen Demand

### Effect of Magnesium and Calcium on Phosphates

In raw Dairy waste water, the initial concentration of phosphates was in the range of 126 mg/l to 141 mg/l. After precipitation of struvite when using magnesium as a source the concentration of phosphates was decreased to in the range of 5.1 mg/l to 6.3 mg/l. as in the same manner when precipitation of calcium ammonium phosphate by using calcium as a source the concentration of phosphates was decreased to 6.3 mg/l to 7.2 mg/l. Finally, it was observed that 95.62 % of phosphates were removed when using magnesium and 94.92 % of phosphates were removed when using calcium as source.

Recovering of phosphates was not only for recovering of nutrients for agricultural purpose but also useful for the removal of phosphorus from dairy waste water will prevent the way of growing of algal blooms in the receiving water bodies.

The effect of precipitation on concentration of phosphates is shown in the below figure

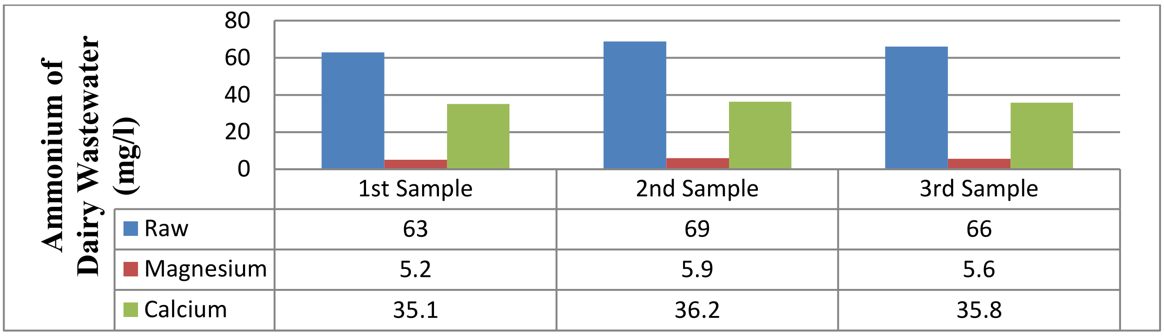
**Figure 10.** The effect of precipitation on concentration of phosphates

### Effect of Magnesium and Calcium on Ammonium

In raw Dairy waste water, the initial ammonium concentration was in the range of 63 mg/l to 69 mg/l. After precipitation of magnesium ammonium phosphate, the concentration of ammonium was decreased to the range of 5.2 mg/l to 5.6 mg/l when using magnesium as a source. As in the same manner when precipitation of calcium ammonium phosphate the concentration of ammonium was decreased to 35.1 mg/l to 36.2 mg/l by using calcium as a source. Finally, it was observed that 46 % of ammonium was removed when calcium is used as source and 91.6 % of ammonium was removed when using magnesium.

Recovering of ammonium was not only for recovering of nutrients for agricultural purpose but also useful for the removal of ammonium from dairy waste water will also prevent the emission of greenhouse gases into the atmosphere.

The effect of precipitation on concentration of ammonium shown in figure



| | 1st Sample | 2nd Sample | 3rd Sample |
|---|---|---|---|
| 🟦 Raw | 63 | 69 | 66 |
| 🟥 Magnesium | 5.2 | 5.9 | 5.6 |
| 🟩 Calcium | 35.1 | 36.2 | 35.8 |

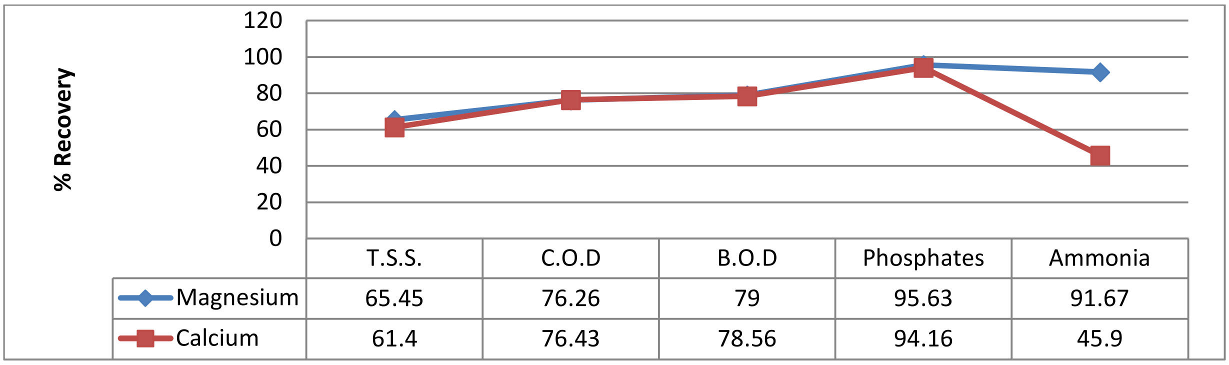
**Figure 11.** The effect of precipitation on concentration of ammonium

### Efficiency of Phosphorus Recovery by using Magnesium and Calcium

Struvite and Hydroxyapatite precipitations lead to increase in pH in all conditions. The Total suspended solids concentration was decreased after treatment. Both the COD and BOD values were also decreased.

The percentage of phosphate undergone precipitated and it was investigated that 91.67 % phosphates were recovered in the form of magnesium ammonium phosphate and 45.9 % of phosphates were recovered in the form of Calcium ammonium phosphate.

Efficiency of Phosphorus Recovery by using Magnesium and Calcium is shown in the below figure



| | T.S.S. | C.O.D | B.O.D | Phosphates | Ammonia |
|---|---|---|---|---|---|
| Magnesium | 65.45 | 76.26 | 79 | 95.63 | 91.67 |
| Calcium | 61.4 | 76.43 | 78.56 | 94.16 | 45.9 |

**Figure 12.** Efficiency of Phosphorus Recovery by using Magnesium and Calcium

## CONCLUSION

1. Struvite (Magnesium ammonium phosphate) and Hydroxyapatite (Calcium ammonium phosphate) precipitation shows to pH increment in all cases.
2. The Total suspended solids concentration was decreased to 65.45% by struvite precipitation and 61.4% by hydroxyapatite precipitation.
3. C.O.D. decreased to 76.26% by struvite precipitation and 76.43% by hydroxyapatite precipitation.
4. B.O.D. decreased to 79% by struvite precipitation and 78.56% by hydroxyapatite precipitation.
5. The phosphorus recovered was 95.63% and 91.67% by struvite precipitation and Hydroxyapatite precipitation respectively from Dairy wastewater.
6. The ammonia was recovered 94.16% and 45.9% by struvite precipitation and Hydroxyapatite precipitation respectively from Dairy wastewater.

Recovery of phosphorus from Dairy wastewater also reduces the Eutrophication.

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