

TREATMENT OF DAIRY WASTEWATER USING COAGULATION TECHNIQUES AND MICROFILTER MEMBRANES

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Abstract

Water is one of the most used resources in this planet and the amount of fresh water available for everyday use is a very small percentage. There is a huge water scarcity in many places around the world and apart from this, many of the water sources around the world are contaminated with harmful chemicals and other biologically harmful substances and that is a huge risk for the survival of the aquatic animals and sometimes even for humans and for these reasons, the wastewater should be treated before it is sent out of the industry. This work focuses on the treatment of dairy wastewater as dairy industry being one of the highest consumers of fresh water. This work is performed on a simulant and using acid to agglomerate particles and a membrane filter to filter the wastewater after the coagulated particles are separated by decantation or any similar methods. Various tests were performed before and after the filtration to measure a series of parameters of which turbidity and total suspended solids had significant decrease percentage. The other parameters had a comparatively less percentage decrease with pH of the wastewater remaining almost the same. This proved to be a cost-effective method to treat dairy wastewater.

Keywords: Dairy wastewater, Membrane filter, Turbidity, Total suspended solids, pH

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1. INTRODUCTION

Water is the most widely used resource all around the globe. It plays a vital role in many industries, for cleansing, and most importantly effluent dilution. Due to the overuse of this resource, there is a forced necessity to reduce the consumption of water and to escalate the recyclability of the same in the industrial sector. Amongst all, the food and dairy industry is one of the leading industrial division for water consumption and pollution, containing effluents of organic origin that are hazardous. Organic pollutants normally consist of equal parts of dissolved, colloidal, and suspended substances, while inorganic materials are usually present mainly in solution¹.

Some of the ways to treat dairy wastewater include membrane systems, electrical methods, coagulants. Amongst all, the membrane system has been proven to be the most effective way of

processing dairy effluents. The characteristics of membrane fouling were studied and found that a pH above 9 decreases the membrane fouling. The influence of the pH and the effect of the threshold flux had been investigated using the shear enhanced technique. It was found that the pH is sensitive when it has a value above 8 and the optimum pH value ranges from 7 – 8. Sometimes nano-filter was also used in tertiary treatments of the dairy effluents, one such case was investigated by L.H. Andrade (2014), where Membrane Bio-reactor (MBR) was coupled with a NF. The membrane bioreactor was successful in removing COD, total N, total P, colour but not the dissolved solids. Therefore, NF helped in removing the dissolved solids and after treating the effluent it was found that the Total Solids (TS) content was removed up to 93.1%.

As the NF did not provide better permeate flux and expected water recovery², researchers went on treating the effluent with higher-grade membrane systems such as Reverse Osmosis (RO). In this method, low polluting dairy effluents were processed and removal of 97.8% and 98.2% of COD and conductivity respectively were observed.³ The average permeate flux was found to be 11 lh-1m-2.4 A pilot-scale plan was also proposed with a 90% recovery rate and payback life of 1.6 years.⁴ As the above operation was costlier, the combination of different filtering units was applied and its characteristics were also studied. A combination of UF and NF was studied by Jianquan Luo et al., (2011b) where it was compared with a single NF unit. The characteristics of irreversible fouling were studied and it was found that the two-stage filtration caused only 2.64% of fouling whereas the NF unit caused 59.10%. Other parameters like lactose content, TMP, etc. were measured and operational cost in the former setup was found to be less. More combined filtration models such as micro-filter + nano-filter (MF + NF), MF + RO²⁶ were investigated under different pressures and found that MF + NF yielded a removal of 100%, 96%, 58% and 51% of turbidity, colour, Kjeldahl Nitrogen (TKN) and COD respectively in which MF had a greater part in removing the suspended solids. The coagulation technique can also be used as the main treatment for treating dairy wastewater. One such investigation was performed by Jai Prakash Kushwaha *et al.* (2010a) where they used Poly aluminium chloride, ferrous sulphate, and potash alum for treating the simulated dairy wastewater. Various parameters like pH, COD, Total Solids, Turbidity, Conductivity, etc. were determined and found that this method removed about 66.5% of COD on an average basis.

In recent years, techniques using electricity to treat wastewater are widely used, like that of electro-chemical⁵, electrocoagulation⁶, electro-flocculation⁷, and electro-fenton⁸ process.

Here, the present research focuses on the treatment of dairy wastewater using micro-filtration membrane. The detailed discussion of

the preparation of the effluents and the experimentations are presented below. The analysis of various parameters about the quality of water was done and their results are discussed.

2. Materials and methods:

2.1 Preparation of Simulant

Pasteurized fresh toned milk was taken to be the source of our simulated effluent. The milk was diluted to a ratio of 1:1 with demineralized water, and a solution of 5L volume was prepared. The demineralized water used for the dilution was collected from the deionization plant constructed at Technological Innovation Centre Lab in SVCE College. Then to the sample, 50 ml of acetic acid was added and mixed thoroughly. Within minutes, agglomeration of proteins started, and the sample was left overnight for separation of solid particles and the water. The next day, the sample was decanted carefully and the water present in the solids was recovered using vacuum filtration setup. The water decanted was used as the effluent for the experiment and the solids particles were discarded.

2.2 Experimental Setup

A bench scale membrane experiment was performed using a Microfilter of pore size 5 microns. The dimensions of the membrane have been summarized in table 1. The membrane was inserted the casing and was filed properly to avoid any leaks through the cap of the module. The simulated wastewater was pumped into the membrane using a Diaphragm pump under moderate to high pressure conditions. The pressure generated by the pump was up to 5.5 bars (80psi). The process was carried out at room temperatures with a flow rate of 1.6 Lpm.

2.3 Membrane Conditioning and Cleaning

Prior to the experiment, the microfilter membrane was soaked in deionized water for 24 hours at 25⁰C. After soaking process, the membrane was washed with water for a period of half an hour to maintain the transport properties. After the experiment, the membrane was immersed in a hot water bath of 40⁰C for a period of 6hrs. and washed again with deionized

water available at room temperature (approximately 25°C) to promote the reusage of the membrane for the process.

3. RESULTS AND DISCUSSION

The synthetic wastewater synthesized had a pungent smell, turbid appearance and a very low pH. Earlier to filtration, the wastewater had high values of Total Suspended Solids (TSS), Total Dissolved Solids (TDS), COD, Turbidity etc., After filtration with a microfilter of pore size 5 µm, a clear distinction between the before and after filtration samples were observed. The other parameter results pertaining to the quality of water were presented and discussed briefly. Each experiment was done for three trials for concordance.

3.1 pH and conductivity

3.1.1. Analysis and Results:

When it comes to membranes, pH is considered to be one of the important parameters as it contributes to the membrane fouling. The effect of pH and membrane fouling extent changes for the different type of effluent used.¹ Hence, pH could be used as good method to control the membrane fouling.³ In the current work, pH of the sample was measured using the pH meter (Ecotestr; model: pH 2). It was found that there is no reduction in pH of the initial and the final sample when treated with the membranes. But there was a significant reduction in the pH after adding acetic acid to the sample. The pH reduced from 6.8 to 4.6 upon the addition of weak acid.

Similarly, conductivity was measured using conductivity meter (Ecotestr; model: EC low). Upon filtration, a 10% reduction in the values of conductivity was observed in the treated effluent. Conductivity as a factor does not hinder the membrane fouling because there is no physical effect of particle integration inside the membrane due to the ions causing conductivity.

3.2 Total Dissolved Solids and Total Suspended Solids

3.2.1 Analysis:

The analysis of TDS and TSS were done by gravimetric method. In this method, a porcelain crucible and Whatmann's filter paper of size 42

were taken and their initial weights were noted down. Then, the sample was filtered using vacuum filter where Whatmann filter paper was used as a filtering medium. After filtration, for TSS, the filter paper was kept in oven at 103-105°C until it gets dried and was cooled using desiccator. The final weight of the filter paper was noted down. Similarly, for TDS instead of the filter paper, 20 ml of filtrate which was collected during the vacuum filtration is taken and kept in oven at 103-105°C till it gets dried. The hot porcelain is cooled using desiccator and the weight of the crucible is noted down.

3.2.2. Results:

The tests for the amount of Total Suspended Solids has shown an average reduction of 60% compared to the simulant before filtration. This evidently shows that usage of microfilters for dairy effluents are effective in removal of larger size particles. Similarly, the Total Dissolved Solids present in the effluent has been reduced by an average of 10%. This inference can be explained by accounting for the particle sizes of the dissolvable solids present in the water. A conclusion can be drawn that the values of TDS and TSS encounter changes upon incorporation of microfilters in treatment of dairy effluents.

3.3 Chloride Content

The amount of chloride present in the sample was detected using Mohr's method. The reagents potassium chromate indicator, standard sodium chloride solution (0.0141 N) and standard silver nitrate solution (0.0141 N) that are used for the analysis were prepared freshly.

3.3.1 Preparation of Standard Silver Nitrate Solution (0.0141 N):

AgNO₃ and NaCl was bought from SRL chemicals and AgNO₃ solution was prepared by dissolving 0.2395 g in 100 ml of distilled water. The standardization of AgNO₃ was done using standardized NaCl solution.

3.3.2 Preparation of Potassium Chromate (K₂Cr₂O₄)

Indicator:

To a small amount of distilled water, 5 g of (K₂Cr₂O₄) was dissolved and standardized AgNO₃ solution (0.0141 N) was added until red precipitate is formed. Mixing was performed using the magnetic stirrer, and after mixing, the

solution was allowed to stand for 12 hours. The solution was then filtered and the filtrate obtained was diluted to 100 ml using deionized water.

3.3.3. Analysis:

50 ml of sample was taken and diluted to 100 ml. Since the sample was not colored, Al(OH)₃ was not added to the solution. The pH of the solution was checked and maintained at 7-8 by adding acid or alkali to the sample. Then, 1ml of K₂Cr₂O₄ indicator was added and titrated against standardized AgNO₃ solution until reddish brown precipitate is obtained. The procedure was repeated for the blank and the chloride amount was calculated using

$$\text{Amount of chlorides in mg/L} = \frac{(V_1 - V_2) \times N \times 35.46 \times 1000}{V} \quad (1)$$

where V – volume of sample; V₁ – volume of AgNO₃ consumed for the sample; V₂ – volume of AgNO₃ consumed for the blank; N – normality of AgNO₃

$$\text{Reduction in Cl}\% = \frac{Cl_i - Cl_f}{Cl_i} \times 100 \quad (2)$$

where Cl_i – amount of chlorine before filtration; Cl_f – amount of chlorine after filtration.

3.3.4. Results:

High amount of chlorides in water leads to the formation of hypochlorites and hypochloric acid which eventually leads to bladder cancer when consumed. Chloride content in wastewater also serves to be one of the effective parameters in creating membrane fouling. Higher concentrations of chloride higher the membrane fouling. Increasing the concentration of polyaluminium chloride up to 100 mg/L could cause lower surface charge, which lead to dense deposit layer with less porosity on the membrane surface and accelerated membrane fouling tendency (Bing Wu et al., 2009). Hence it is required to reduce the chlorine content present in water. In this work, the chloride content for the initial sample was found to be 335 ± 5 mg/L and for the filtered sample it was 314 ± 2 mg/L. The average reduction rate was found to be 5.00 ± 2 % implying that there is no role of microfilter in reducing the chloride content effectively.

3.4 Sulphates:

The amount of sulphates present in the sample was detected using colorimeter (Sky Technology India, model STI-403). The reagents like conditioning agent and standard sulphate solution that are used for the analysis were prepared freshly.

3.4.1 Preparation of Standard Sulphate

Solution:

Anhydrous sodium sulphate was bought from SRL chemicals. 1.379 g of anhydrous Na₂SO₄ was thoroughly mixed with 1000 ml of distilled water.

3.4.2 Preparation of Conditioning agent:

Glycerol, Isopropyl Alcohol and concentrated HCl were bought from SRL chemicals. Solution 1 was prepared by dissolving 7.5 g of NaCl in distilled water in a separate beaker. Solution 2 was prepared by adding 5 ml Glycerol, 3 ml conc. HCl, 10 ml of IPA and mixed well in a beaker. Solution 1 and Solution 2 was mixed thoroughly in a magnetic stirrer to obtain the conditioning reagent.

3.4.3. Analysis:

The colorimeter was switched on and calibrated at 420 nm wavelength using distilled water. After preliminary steps, 10 stoppered standard flasks were taken for analysis out of which 7 is used for standard sulphate solutions (5,10,15,20,25,30,35 mg/l), one for blank and two for the samples. Standard solutions of different concentrations were prepared by adding 5ml of std. sulphate solution in first flask, 10ml in second flask and so on. To analyse samples, 20 ml of samples were poured into the standard flasks and 5 ml of conditioning agent and a pinch of BaCl₂ were added to all 10 standard flasks. The solution is made up to 100 ml by adding distilled water. Once the samples were prepared, 5 ml of each sample was taken in cuvette and subjected for the absorbance measurement at 420 nm in the colorimeter and the results were obtained using the formulae.

$$Y = mX + C \quad (3)$$

$$\text{Amount of sulphate in mh/l} = \frac{X \times 100}{ml(\text{sample})} \quad (4)$$

where, Y – absorbance of the sample; X – concentration of sulphate in mg; m – slope of the calibrated curve

3.4.4. Results:

Presence of Sulphur in the water creates odor and promotes sewer corrosion as sulphates reduce to hydrogen sulphides under anaerobic conditions. Hence, sulphates are of considerable concern when it comes to wastewater treatment. In this work, the sulphate content for the initial sample was found to be 805 ± 5 mg/l and for the filtered sample it was 725 ± 10 mg/l. The average reduction rate was found to be 10.00 ± 2 % implying that there is no role of microfilter in reducing the sulphate content effectively.

3.5 Phosphates:

The amount of phosphorous (mainly orthophosphate) present in the sample was detected using colorimeter (Sky Technology India, model STI-403). The reagents like Ammonium molybdate - antimony potassium tartrate solution, Ascorbic acid solution, H_2SO_4 solution (11N), and standard phosphate solution that are used for the analysis were prepared freshly.

3.5.1 Preparation of Standard Phosphate solution:

0.4393g of KH_2PO_4 was taken and subjected to drying in an oven at $105^\circ C$ for an hour. The pre-dried salt is dissolved in distilled water and diluted to 1000 ml. 100 ml of the prepared solution was made up to 1000 ml to get the standard phosphate solution.

3.5.2 Preparation of Ammonium molybdate-antimony potassium tartrate solution:

4g of ammonium molybdate and 0.1g antimony potassium tartrate was dissolved in 400 ml of distilled water and diluted to 500 ml.

3.5.3 Preparation of Ascorbic acid solution:

60 g of ascorbic acid was dissolved in 800 ml of distilled water and diluted to 1 liter. 2 ml of acetone was added and the prepared solution is stable for two weeks.

3.5.4 Preparation of H_2SO_4 solution (11N):

31 ml of conc. H_2SO_4 (98% pure) was slowly added to 60 ml distilled water approximately. The solution was cooled and diluted to 100 ml.

3.5.5. Analysis:

The colorimeter was switched on and calibrated at 650 nm wavelength using distilled water. After preliminary steps, 12 stoppered standard flasks were taken for analysis out of which 10 is used for standard sulphate solutions (0.01, 0.02 ... 0.09, 0.1 mg/l), one for blank and two for the samples. Standard solutions of different concentrations were prepared. From the prepared samples, 50 ml of solution was taken to which 1ml of 11N H_2SO_4 , 4ml of ammonium molybdate-antimony potassium tartrate solution and 2ml of ascorbic acid solution was added and mixed thoroughly. The procedure is repeated for each and every sample. Once the samples were prepared, 5 ml of each sample was taken in cuvette and subjected for the absorbance measurement at 650 nm in the colorimeter and the results were obtained using the formulae.

$$Y = mX + C \quad (5)$$

$$\text{Amount of Phosphate in mg/l} = \frac{X \times 100}{\text{ml(sample)}} \quad (6)$$

where, Y – absorbance of the sample; X – concentration of phosphorous in mg; m – slope of the calibrated curve.

3.5.6. Result:

Presence of high amount of phosphates in the water is responsible for eutrophication in rivers when let into the rivers without treating. Its presence leads to problems in quality of water such as increased purification costs, decreased recreational and conservation value of impoundments, loss of livestock and the possible lethal effect of algal toxins on drinking water. In this work, the phosphate content for the initial sample was found to be 157 ± 5 mg/L and for the filtered sample it was 114 ± 5 mg/l. The average reduction rate was found to be 30.00 ± 2 % implying that there is minor role of microfilter in reducing the phosphate content effectively.

3.6. Chemical Oxygen Demand:

COD of the sample was analyzed using the titration method. The reagents like conditioning

agent and standard sulphate solution that are used for the analysis were prepared freshly.

3.6.1. Preparation of Standard Potassium Dichromate Reagent:

K₂Cr₂O₇ and (Hg₂)₂SO₄ were bought from Rankem Chemicals. 0.4913 g of K₂Cr₂O₇ was taken and dried in oven at 103°C for 2 – 4 hrs. To the dried K₂Cr₂O₇, 3.33 g of (Hg₂)₂SO₄ was added inside the beaker. 16.7 ml of conc. H₂SO₄ was poured to the mixture and the contents were mixed completely. The hot solution was allowed to cool and left overnight for dissolving. The next day, the contents were transferred carefully to 100 ml standard flask and distilled water was added up to the mark. The prepared solution is stored in brown bottle to avoid interference with sunlight.

3.6.2. Preparation of Sulphuric Acid Reagent:

Ag₂SO₄ was bought from Rankem Chemicals. 1.1 g of Ag₂SO₄ was taken and mixed thoroughly with 100 ml of conc. H₂SO₄. The solution was allowed to stand for 24 hours for complete dissolving. The contents were stored in brown bottle and covered with silver foils to avoid interference with sunlight.

3.6.3. Preparation of Standard Ferrous Ammonium Sulphate Solution:

Ferrous Ammonium Sulphate (FAS) was bought from Rankem Chemicals. The solution was prepared while cooling the vials in order to yield perfect answers. 1.96 g of FAS was taken and mixed with little amount of conc. H₂SO₄ for dissolving purpose. The prepared paste is then mixed with 500 ml of distilled to yield a solution of strength 0.01 N. The strength of FAS is made low to get accurate values of Chemical Oxygen Demand.

3.6.4. Analysis:

COD Digester was switched on half an hour before and temperature of the digester was fixed to 150°C. Meanwhile, 2.5 ml of preserved samples and 2.5 ml of blank were taken in COD vials individually. To the samples and blank, 1.5 ml of Potassium dichromate solution and 3.5 ml of Sulphuric Acid Reagent were carefully added. The solution undergoes exothermic reaction and the vials are very hot. The vials were tightly closed and placed carefully inside the COD digester for 2 hrs. After 2 hours, the

vials were removed carefully and cooled inside a desiccator. The cooled samples were taken in a conical flask and titrated against FAS where ferroin is used as indicator. The titration is continued until the appearance of reddish-brown colour. Similar procedure was followed for all the samples. The consumed FAS volume was noted and COD was quantified using the formula.

$$\text{Amount of COD in mg/l} = \frac{(A-B) \times N \times 8 \times 1000}{V} \times D \quad (7)$$

where, A – Volume of FAS consumed by blank; B – Volume of FAS consumed by sample; N – Normality of FAS; D – Dilution Factor; V – Volume of the sample taken.

3.6.5. Result:

When it comes to wastewater treatment, COD becomes the most important parameter as it quantifies the amount of oxygen required to oxidize the organic matter present in the water. COD is a test which is used to measure pollution of domestic and industrial waste. The waste is measure in terms of equality of oxygen required for oxidation of organic matter to produce CO₂ and water. COD test is one which is useful in pinpointing toxic condition and presence of biological resistant substances (Ajim S. Sutar *et al.*, 2015). If high levels of COD are present, then the amount of Dissolved Oxygen (DO) decreases drastically thereby leading to anaerobic conditions.

In this experiment, COD content for the initial sample was found to be 41434 ± 50 mg/L and for the filtered sample it was 39840 ± 50 mg/L. The average reduction rate was found to be 3.00 ± 2 % implying that there is no role of microfilter in reducing the COD content effectively. Similar results were obtained by Airton C. Bortoluzzi *et al.*, (2017) where microfilter was used as a pre-treatment filter. Furthermore, the low COD removal could be associated with carbohydrate degradation, mostly from lactose, which could produce lactate ion and ethanol. Those organic

molecules have low molecular weight, thus permeating through the MF membrane.²⁶

3.7 Turbidity

Turbidity of the sample was measured using microprocessor based turbidity meter. The reagents required for the nephelometer were prepared freshly.

3.7.1. Preparation of Hydrazine Sulphate Solution – (Solution 1) and Hexamethylenetetramine Solution– (Solution 2)

Hydrazine Sulphate and Hexamethylenetetramine were purchased from SRL Chemicals. Solution 1 was prepared by mixing 1 g of Hydrazine in 100 ml of distilled water. Similarly, solution 2 was prepared by mixing 10 g of Hexamethylenetetramine in 100 ml distilled water.

3.7.2. Analysis

In 100 ml volumetric flask, 5 ml of Solution 1 and 5 ml of Solution 2 were mixed. The suspension mixture was allowed stand for 24 hours at 25°C and transferred to 100 ml standard flask. The next day, the solution was diluted to the mark and mixed. The turbidity to this suspension is 400 NTU. After preparing standard solution, the nephelometer was switched on and the cuvette containing blank was inserted. The blank was observed for standard readings and set zero button was pressed. Once done with blank, the cuvette containing standard solution was inserted and observed for stable readings. The deviation was calculated and adjusted using the INC/DEC keys available in the instrument. After calibration, the cuvette containing the samples were subjected to analysis and the corresponding NTU values were noted.

3.7.3. Results

It is essential to analyse the turbidity of the water as it indicates the amount of sediments, bacteria etc., present in water. It is required to maintain very low turbidity. In this experiment, turbidity for the initial sample was found to be 500 ± 3 NTU and for the filtered sample it was 35 ± 1 NTU. The average reduction rate was found to be 93.00 ± 1 % implying that there is significant role of microfilter in reducing the

turbidity effectively. 78% reduction in turbidity was observed when operated at 1 bar pressure and 83% reduction was observed when operated at 2 bars.²⁶ The high turbidity removal is due to the bigger size of the particles present in the effluent as most of the turbid creating materials have a size greater than 0.1 μm .

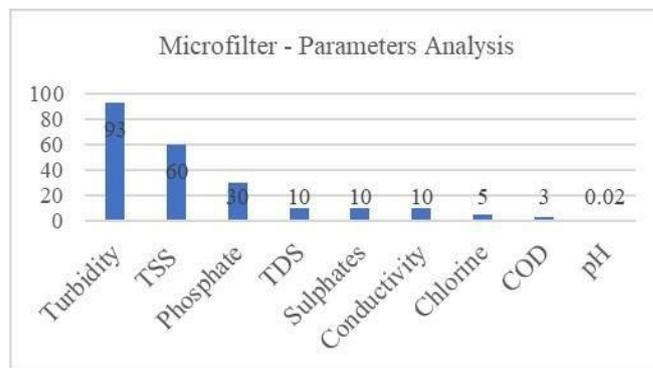


Figure 1. Overview of the effect of removal of parameters when the effluent is subjected to microfiltration process.

4. CONCLUSION

Thus, the present work involved the treatment of simulated wastewater using microfilters. The simulant was treated with acetic acid for agglomeration of particles. The permeate after filtration was analyzed for different water testing parameters to study the filtration efficiency of the process and to check the quality of the water obtained. An overall view of the different parameters is given in figure 4 and following conclusion is obtained

(i) Microfilter membrane is the most suitable for removing turbidity as high turbidity removal is due to the larger size of the particles (greater than 0.1 μm) present in the effluent.

(ii) An average of 60% reduction in the value TSS was observed because of the presence of larger size insoluble particles present in the effluent.

(iii) The membrane has no significant effects in reducing COD, TDS, Sulphates, Conductivity and Chlorine.

(iv) No reduction of pH was observed when the effluent is subjected to filtration.

(v) The reason for the low COD removal could be associated with carbohydrate degradation,

mostly from lactose, which could produce lactate ion and ethanol. Those organic molecules have low molecular weight, thus permeating through the MF membrane.

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