Institución Universitaria SALAZAR Y HERRERA

QUID 2017, pp. 605-611, Special Issue N°1- ISSN: 1692-343X, Medellín-Colombia

STUDY ON THE COMBINED COAGULATION-FLOCCULATION METHOD IN WASTEWATER TREATMENT OF RAISIN FACTORY

(Recibido el 15-06-2017. Aprobado el 04-09-2017)

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RESUMEN: La contaminación del agua afecta la salud y la calidad de los recursos limitados de agua limpia y daña gravemente el desarrollo social y económico de la comunidad a largo plazo, además de contribuir a la propagación de muchas enfermedades diversas y peligrosas. Por lo tanto, es esencial reciclar las aguas residuales industriales y las aguas residuales, especialmente en países con escasez de agua o falta de agua. Las aguas residuales de la fábrica de pasas fueron tratadas por un método combinado de coagulación y floculación en este estudio. Posteriormente, este método calculó la concentración y el tipo de coagulante y el pH de las aguas residuales en las que se observó la mayor eliminación de la Unidad Nefelométrica de Turbidez (NTU) y de los Sólidos Suspendidos Totales (TSS). Entre los coagulantes, el sulfato de hierro, que tuvo la mayor tasa de remoción en su concentración y pH más altos, fue considerado como el mejor coagulante para las aguas residuales de la fábrica de pasas.

Palabras clave: Coagulación y floculación, Alúmina (sulfato de aluminio), Cloruro férrico, Sulfato de hierro, Demanda química de oxígeno (DQO), Unidad nefelométrica de turbidez (NTU)

ABSTRACT: Water pollution affects health and quality of limited clean water resources and greatly damages social and economic development of community in the long term in addition to contributing to spread of many diverse and dangerous diseases. Therefore, it is essential to recycle industrial wastewater and sewage especially in countries with water scarcity or lack of water. Wastewater of raisin factory was treated by a combined coagulation-flocculation method in this study. Afterwards, this method calculated concentration and type of coagulant and pH of wastewater in which the highest removal of Nephelometric Turbidity Unit (NTU) and Total Suspended Solids (TSS) was observed. Among coagulants, Iron sulfate ,which had the highest removal rate at its highest concentration and pH, was considered as the best coagulant for wastewater of raisin factory.

Keywords: Coagulation and flocculation, Alum (Aluminium sulfate), Ferric chloride, Iron sulfate, Chemical Oxygen Demand (COD), Nephelometric Turbidity Unit (NTU), Total Suspended Solids (TSS)

1. INTRODUCTION

Continuous and easy access to clean drinking water is one of the key indices of development and welfare in any society;and it is more than 99 percent in developed communities.Unfortunately, according to statistics onpollutant sourcesin some developing countries,there is a severe shortage of continuousaccess to clean water resources for the public.Meanwhile, interaction between human and his industrial waste with environment is one of the problems in clean water supply.Furthermore, the increasing urbanization has led to producedlarge volumes of solid waste and sewage leading to considerable environmental problems and consequences for contamination of water resources.Millions of people die or become patient annually due to the unclean water.According to statistics of the World Health Organization (WHO), 80% of diseases are water related in developing countries [1].In addition to contributing to spread of many diseases, water pollution also affects health and quality of limited clean water resources leads togreat damages on social and economic development of community.Therefore, it is essential to recycle industrial wastewater and sewage especially in countries withwater scarcity orlack of water.Water is one of the strangest phenomena of creation consisting of two elements of hydrogen and an element of oxygen which aregas at normal temperature. [2] Hydrogen is an element which burns while oxygen is necessary for burning.Most earth surface is covered with water.About 1.4 billion cubic kilometers of Earth's surface is coveredwith seas, oceans, and icy mountains.Human being uses about 1% of all water on earth as surface water (streams, rivers and lakes) or groundwater (springs and wells) [3].

1.1. Need for water treatment

On the one hand, the increasing water demand due to population growth, technology development, rising living standards and health of communities, and on the other hand, misuse of available resources and limitation of available water resources have caused problems in mostcountries of world.[4, 5] Surface and subsurface water resources, which are used by humans, are always exposed to different contaminants due to exposure to air and soil.Subsurface water is quantitatively the most important water resource after oceans and polar ices.Lakes, rivers and atmospheric humidity are in the next ranks respectively [6].

Water may be drinkable but not pleasant and fresh Apparent, physical and chemical properties of water play significant roles in consumer satisfaction. Natural water usually requires treatment either for drinking or industrial consumption. Water treatment for drinking is both easier and cheaper than water treatment for industrial use.[7]

2. MATERIALS AND METHODS

2.1. Introduction

This research used chemical coagulants to reduce COD, NTU and TSS of raisin factory wastewater.

2.2. Necessary equipment

2.2.1 Laboratory equipment

This research used beaker, Erlenmeyer flask, pipette, Büchner funnel and filter paper for transferring distilled water and weighing materials.

2.2.2 Necessary equipment for analysis of samples

Necessary equipment for analysis of samples is presented in Table 1.

Table 1. *Tools and equipment required for sample analysis*

Cause of deployment	Tools and Equipment
Boil the solution	heater
To straighten	Smoother
To straighten	filter paper
Determine the weight	Scales
Determine TDS	TDS measuring device
Determine PH	PH meter
Determine the temperature	Thermometer
Determine opacity	Turbidity meter
Temperature control	Incubator

3. NECESSARY MATERIALS FOR TESTING

1) The tested raw wastewater of this study consisted of wastewater from raisin factories of Quchan County with relatively high COD, NTU and TSS. Specifications of raisin raw wastewater are presented in Table 2.

Table 2.*Specifications of raisin raw wastewater*

pH of solution	Nephelometric Turbidity Unit (NTU) of solution	Total Suspended Solids (TSS) of solution(mg/L)	Chemical Oxygen Demand (COD)of solution
3.8	720.6	1720	24000

2) Distilled water used for washing laboratory glassware.

3) Sodium hydroxide(NaOH) used to control pH.

4) Coagulant

Alum (Aluminium sulfate) and ferric chloride and iron sulfate were used as coagulants.

4. METHOD

4.1. Coagulation and flocculation

We measured NTU and TSS at concentrations of 20 mg/l, 40 mg/l and 60 mg/l by adding Alum with pH =3.8 to raw wastewater. Jar test device was used for mixing and flocculation. The first step of mixture was performed at 300 rpm for 2 minutes. The second mixture was done at 30 rpm for 30 minutes. At the last step, beaker was left for 30 minutes so that coagulum deposits. The same test was performed with ferric chloride and iron sulfate coagulants. The best concentration, at which the greatest removal occurred, was calculated by evaluation of results. Jar test was then performed for obtained concentrations at pH of 5.5, 7.5 and 9.5.

4.1.1 Turbidity measurement

Turbidity is caused by presence of colloid suspended particles such as sand, mud. A variety of Turbidity meters can be used to determine this parameter .We poured 10 mg/l of sample into the glass tube and shake it completely. After a while, the air bubbles left the container, and then we put it inside the device, displayed number referred to the turbidity. In this test, a WTWTURB355IR device, made in Germany, was used to calculate turbidity. [8]

4.1.2 COD measurement

Chemical Oxygen Demand (COD) is the amount of oxygen used to oxidize the oxidizing organic matter by a strong oxidant. This index refers to both decomposable and non-decomposable organic matter by bacteria.COD can thus be representative of typical organic impurities. COD test is still considered as one of the most important factors in management and design of water treatment plants due to its speed.

5. RESEARCH METHODOLOGY

5.1. COD measurement by titration method

10 cc of Chromates + X wastewater samples + (X-20 distilled water)

Amount of sample depends on its contamination.The more pollution will lead to the less amount of sample.For instance, 1 CC of sample is sufficient for samples with contamination of greater than COD= 10000.Distilled water is added to it, and if we have lower COD sample, the volume of selected sample will be greater and the amount of distilled water will be reduced to the same amount. [8]

There is a need for a control to measure COD by titration; hence, we mix 10 cc of Chromate with 20ccof distilled water in another container without any sample to use this container as a control. Afterwards, we add30 cc of Concentrated Sulfuric Acid and Silver sulfate to both concentrates containers.

Note: Take the container under the poring cold water while adding acid because it is an exothermic reaction.

Note: Before adding acid, the prepared solution is Terra cotta due to chromate.After adding acid, if color of solution is dark green, it means that the COD of wastewater is high and sample has high volume, so this solution is no longer usable, and we should prepare a new solution with less wastewater sample.

After preparing the sample solution and control, we throw 3 to 4 glass bullets inside both solutions in order to prevent explosion.Then, we put sample on a heater under the hood; and cold water in distillation state should be boiled at a temperature of 100°C to 150°C for 2 hours. Water flow should not be interrupted.After 2 hours and cooling, we add Freon indicator to sample(3 drops)and then stir it.

Then, we start with boiled solution and titration sample, and we stir solution while adding Titrant solution to sample and control. Little by little, the sample becomes green, and then blue, and when the solution becomes red by the first drop, we read the number.Do the same step for control solution.We call the control number by "A" and sample number by "B".

$$A-B=(S)\,. \tag{1}$$

Now we calculate amount of COD by the following formula.

$$COD = \frac{F \times S \times 8000}{V} \tag{2}$$

V: Sample volume picked up at the beginning of test.

F: Factor check (Normality)

5.2. Preparation of normal titration solution (Titrant) 25%

$$FAS \Rightarrow (NH_4)_2 Fe(So_4)_2 .6H_2O \tag{3}$$

5.3. Ferrous ammonium sulfate hexahydrate

We weigh 98 g of FAS, and transfer this amount to a 1000-ml Volumetric flask, and then add water (e.g.up to 300 CC). We measure 200CC of Concentrated sulfuric acid by a Graduated cylinder and transfer it into Volumetric flask, and finally increase volume by distilled water to volume of 1000 after dissolution. [8]

Now, we titrate $k_2cr_2o_7$ 10cc + Freon indicator + pureConc$H_2So_4$30 ml with Titrant solution.

F × amount of consumed Titrant = 0.25 × 10cc

5.4. Preparation of Freon

We transfer 1.485 g (1,10-Phenanthroline monohydrate) or 695 mg of (FeSO4.6H2O) to100 ml Volumetric flask afterdissolution.

5.5. Preparation of concentrated sulfuric acid 95-98%

For half a liter of this acid, we weigh5.06 g of AgoSO4 and pour on it.

We weigh AgoSO4 on paper.

5.6. Preparation of Normal Potassium Dichloramate 25% ($K_2Cr_2O_7$)

We weigh 259 g of this matter on a wax paper (or on an Aluminum foil). We transfer this amount to 1000 ml Volumetric flask, and finally we increase volume by distilled water.

Note: Potassium dichromate is water absorbent.We heat it up in oven at a temperature of 105 °C for 2 hours. Then, we put it in Desiccator to reach the ambient temperature.Finally, we weigh it.

6. TSS MEASUREMENT

In order to measure the TSS, we weigh a filter paper and place it inside the Büchner funnel.We pick up 50 ml of filtered wastewater by pipette, pour on the filter paper, and measure its weight after filtering, and then subtracts the obtained weight from weight of filter paper. Therefore,the amount of TSS is calculated in 50 ml of treated wastewater. [8]

7. DISCUSSION AND CONCLUSION

In this paper, we investigated the obtained results of coagulation and flocculation tests on raw wastewater of raisin factory, and then calculated the best coagulant, pH, and concentration of coagulant in which the rates of NTU and TSS removal are obtained.

7.1. Raw wastewater specifications

The rate of NTU was first obtained by Turbidity meter in order to calculate the removal percentage of NTU, TSS and COD in wastewater of raisin factory.Afterwards,TSS was calculated using filter paper and Büchner funnel; and COD was measured by titration.Figure 1 shows these specifications.

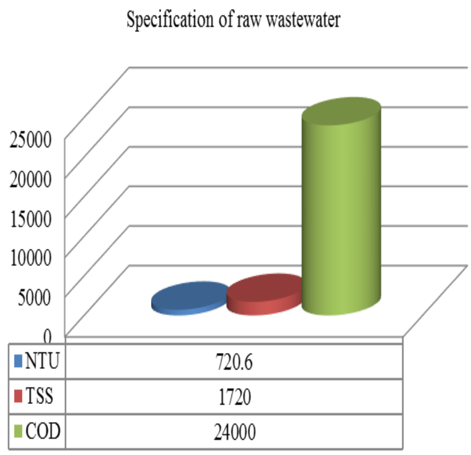

Figure1.*Specifications of raw wastewater*

7.2. Calculation of proper concentration of coagulant at pH= 3.85

We considered three compounds namely Alum, Iron sulfate, and Ferric chloride in order to calculate proper coagulant.All matters were tested at concentrations of 20 mg/l, 40 mg/l and 60 mg/l in order to calculate the most suitable concentration which led to the highest removal percentage.

7.3. Alum (Aluminium sulfate)

Figure 2 shows the removal percentage of NTU and TSS at different concentrations of Alum.The upward trend of graph indicates that the increased concentrations of NTU and TSS will lead to increased removal percentage of NTU and TSS.

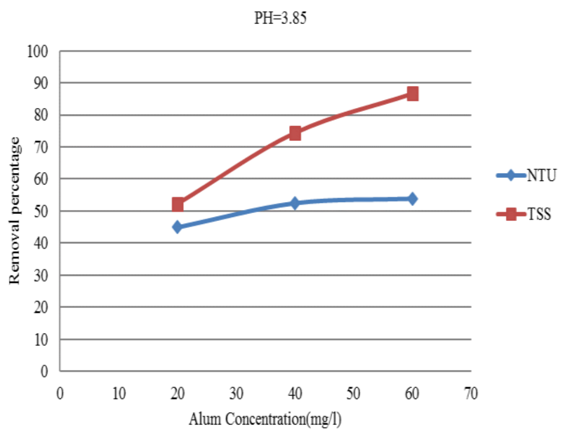

Figure 2.*Removal percentage of NTU and TSS at different concentrations of Alum (Alum concentration)*

Removal percentage of NTU was obtained equal to 331.8 at concentration of 60 mg/l, and removal percentage of TSS was 230 in the same conditions.

7.4. Iron Sulfate

The amounts of NTU and TSS were calculated for different concentrations of Iron Sulfate by exchanging the coagulant matter and performing the test.The increased concentration of coagulant led to the increased removal percentage of NTU and TSS as shown in Figure 3.

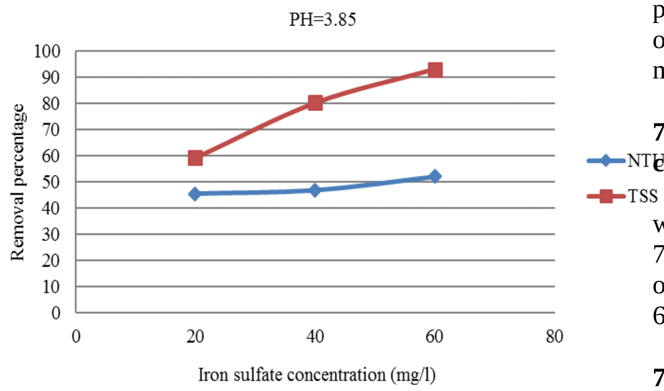

Figure 3.*Removal percentage of NTU and TSS at different concentrations of Iron Sulfate (Iron Sulfate concentration)*

In this test, the removal percentage of NTU was 345.45 at concentration of 60 mg/l, and removal percentage of TSS was equal to 120 in the same condition.The upward trend of graph indicates that the increased removal percentage will lead to increased concentration of Iron Sulfate.

7.5. Ferric chloride

Ferric chloride is another applied coagulant in this test.Like other coagulants, the increased amount of this matter will lead to increased concentrations of coagulation and flocculation, and consequently increased removal percentage of NTU and TSS.Figure 4 shows removal percentage of NTU and TSS at different concentrations of Ferric chloride.

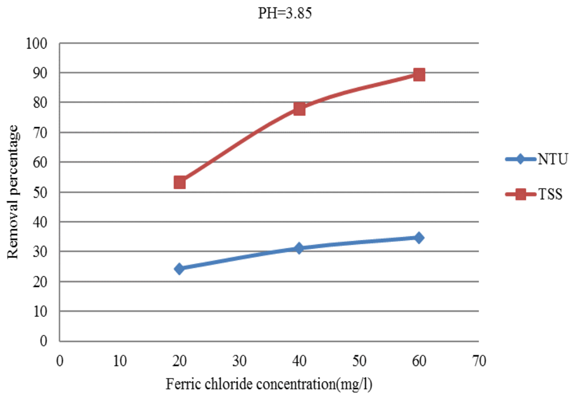

Figure 4.*Removal percentage of NTU and TSS at different concentrations of Ferric chloride (Ferric chloride concentration)*

Removal percentage of NTU was obtained equal to 470.2at concentration of 60 mg/l, and removal percentage of TSS was equal to 180 in the same conditions.Using a coagulant at pH=3.85, the concentration of 60 mg/l was determined as the most suitable concentration leading to the highest removal percentage.It should be noted that removal percentage of TSS was higher than NTU in each of the above mentioned conditions.

7.6. Calculation of proper pH of coagulant at concentration of 60 mg/l

As shown, some amounts of NTU and TSS were removed using coagulants at pH = 3.85.pH of 5.5, 7.5 and 9.5 were tested in order to investigate the effect of pH on removal of NTU and TSS at concentration of 60 mg/l by each coagulant.

7.7. Alum

Figure 5 shows the removal percentage of NTU and TSS at different pH values for Alum.The Upward trend of graph indicates that the increased pH will lead to the increased removal percentage of NTU and TSS.

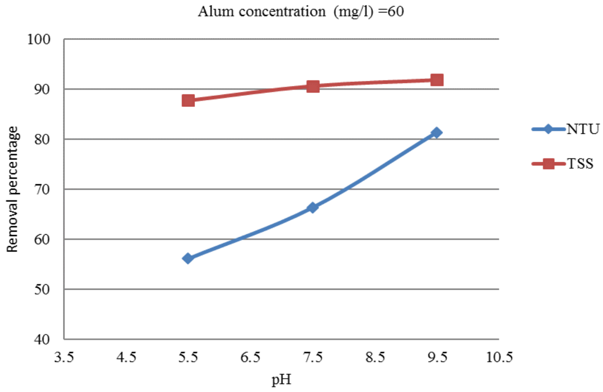

Figure 5.*Removal percentage of NTU and TSS by Alum as the coagulant at different pH of wastewater*

7.8. Iron Sulfate

The highest removal rate was obtained at pH=9.5 using Iron Sulfate at different pH.This value was equal to 29.67 for NTU and 40 for TSS.Increased removal percentage of NTU and TSS is shown in Figure 6, and its upward trend indicates that increased pH will lead to the increase in removal percentage of NTU and TSS.

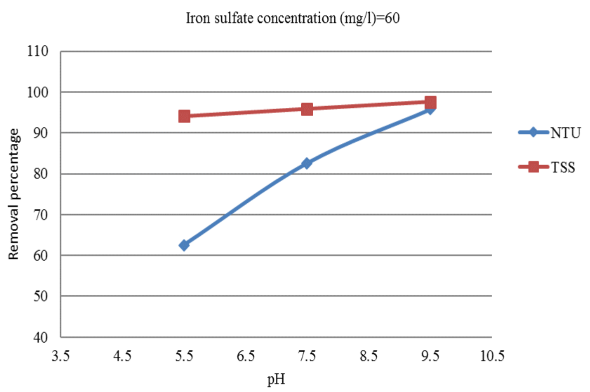

Figure 6.*Removal percentage of NTU and TSS by Iron Sulfate as the coagulant at different wastewater pH*

7.9. Ferric chloride

Coagulation and flocculation using Ferric chloride at concentration of 60 mg/l leads to removal of NTU up to the value of 60.62 and reduces TSS to 60.Figure7 shows removal percentage of NTU and TSS at concentration of 60 mg/l of Ferric chloride.

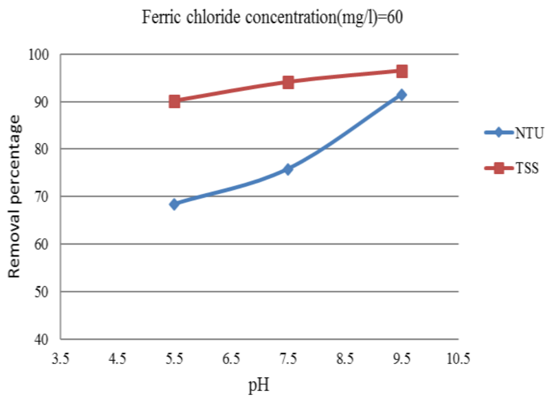

Figure 7.*Removal percentage of NTU and TSS by Ferric chloride as the coagulant at various wastewater pH*

As shown, the increased pH results in an increase in removal percentage of NTU and TSS, as this value becomes maximum at pH=9.5.According to graphs of Sections 3 and 4, the increased concentration of coagulant has a greater effect on TSS, while pH increase has amore significant effect on NTU.

8. DETERMINATION OF APPROPRIATE COAGULANT

8.1. Percentage removal of NTU

Figure 8 shows the removal percentage of NTU using the mentioned coagulants at the concentration of 60 mg/l and pH=9.5.Iron sulfate with removal percentage of 95.8% shows the highest removal percentage of NTU.

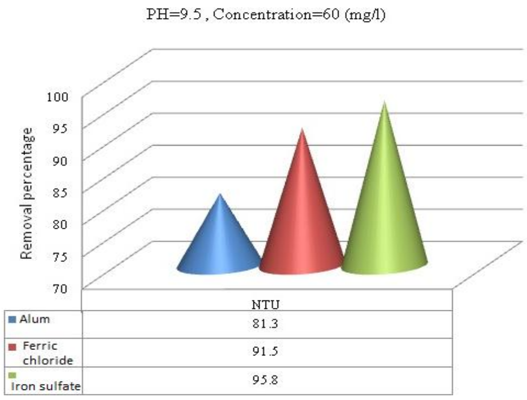

Figure 8.*Removal percentage of NTU using coagulants*

8.1.1 Removal percentage of TSS

Removal percentage of TSS through coagulants is shown in Figure 9.Iron sulfate showed better performance in TSS removal and it was considered as the best coagulant due to the removal percentage of 97.6%.

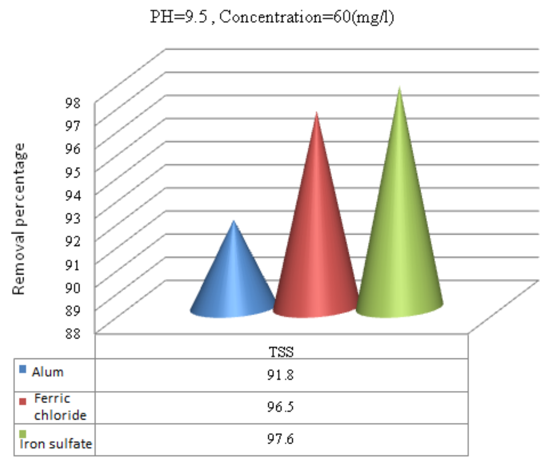

Figure 9.*Removal percentage of TSS using coagulants*

9. DISCUSSION AND CONCLUSION

Using a combined coagulation-flocculation method, this research sought to reduce the rates of NTU, TSS and COD as much as possible in wastewater of raisin factory.The combined method consisted of a coagulation-flocculation step by Alum, Ferric chloride, and Iron sulfate at different concentrations and pH.According to studies, the increased concentrations of coagulants led to the increased removal percentage of NTU and TSS.Furthermore, increased pH led to the increased removal percentage of NTU and TSS.Among all coagulants, Iron sulfate, which had higher removal percentage at the highest concentration and pH, was determined as the best coagulant for wastewater of raisin factory.

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