Brewery Effluent Sludge Characterization and Dewatering to Increase Potential Water Recycling Capabilities

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Abstract*—In this study various coagulants and flocculants were tested for optimum removal of COD from brewery wastewater. The coagulants used were ferric chloride and an organic coagulant mixture obtained from the brewery company. The flocculants used were a cationic flocculant (Genesys' genefloc, polyquaternary amine), an anionic flocculant (Senfloc 5210), a non-ionic flocculant (Senfloc 5330) and a cationic acrylamide copolymer in aqueous dispersion (Brewery flocculant) for comparison. The organic coagulant mixture in aqueous solution was more effective than the ferric chloride at the same optimal dosage of 25 ppm. The ferric chloride and organic coagulant mixture used alone achieved COD removal efficiencies of 32.7% and 48.5% at a pH of 5.65 respectively while their performances for turbidity removal at pH 5.65 were 63.3% and 91.9% respectively. The best results were achieved when using the ferric chloride in combination with the cationic acrylamide copolymer in aqueous dispersion (Brewery flocculant) at an optimum dosage of 30 ppm and when using the organic coagulant mixture in aqueous solution in combination with the cationic acrylamide copolymer in aqueous dispersion (Brewery flocculant) at an optimum dosage of 10 ppm. When using ferric chloride in combination with the cationic acrylamide copolymer in aqueous dispersion (Brewery flocculant) and the organic coagulant mixture in aqueous solution in combination with the cationic acrylamide copolymer in aqueous dispersion (Brewery flocculant), COD removal efficiencies of 79.6% and 78.2% at a pH of 5.85 respectively was achieved, while they exhibited similar performance for turbidity removal at pH 5.85 of 99.7%. The optimization tests carried out in this study have allowed to improve the understanding of turbidity and COD removal mechanisms and achieved lower dosages of coagulants and flocculants for better treatment, therefore suggesting a more economical approach. Comparing the achieved results with the standard disposal values, it can be seen that the results gathered in this study complied to the values of the standard disposal values. This means that the quality of the treated water in this study complies with the environmental protection laws and will enable the brewery to dispose the treated water into the environment and municipal sewer systems.

Keywords**—** Brewery wastewater, coagulation, flocculation, COD, turbidity, cationic flocculant, anionic flocculant, non-ionic flocculant

I. Introduction

Water is a scarce resource not only in South Africa, but in many parts of the world [1-14]; therefore, the brewery industry like other industries are forced to consider waste water recycling as a method to save water, the environment and cut down costs on pre-treatments for discharge to municipal sewer systems. The brewery industry like any other industry is subject to tight government legislation including environmental protection laws, making discharge routes for wastewater more difficult, as routes to the sea and rivers are not options anymore.

Brewery wastewater comprises of high concentrations of organic matter and suspended solids. The organic matter comes from the use of kieselguhr, wort, surplus yeast and trub. Other components are labelled pulp, washing and cleaning reagents as well as grease and oil from machinery [15-18]. Liu and Feng (2003) reported that extracellular polymeric substances (EPS) are one of the main pollutants of brewery effluent sludge. These pollutants make brewery effluent rich in carbohydrates, nitrogen, phosphate, protein and fats. Untreated brewery wastewater, high in nutrients can have dangerous environmental effects, because of the high chemical oxygen demand (COD) [17,19].

The brewery industry produces the most problematic waste and therefor characterization of the brewery effluent is very important. The reason for characterization is to determine the impact of the wastewater on the environment and if the pre-treatment method used in the brewery is effective [15]. For the purpose of this study four parameters will be considered namely: COD, turbidity, pH and zeta potential, in agreement with standard methods. COD is a measurement of the ability of water to use oxygen during the decomposing process of organic matter [20, 21]. When COD levels are high, it indicates a high amount of oxidizable organic matter in the water, which will deplete dissolved oxygen (DO) levels. Reduced DO levels will

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stimulate anaerobic conditions which are detrimental to higher life forms in the aquatic ecosystem [22]. The COD in wastewater is very high (2000 – 6000 mg/L) [16,23,24]. Turbidity is a measurement of the murkiness of the water and an indication whether the treatment method was efficient or not. Turbidity of 5 NTU/JTU or less is the guideline for drinking water [25]. A high turbidity in water reduces light availability for photosynthesis of plants and increases the temperature of the water, causing an increase in hydrogen sulphide concentration, which is poisonous to aquatic life [17]. The pH determines whether the water is acidic or alkalis [26]. A pH value of between 2 and 10 may a different impact in the output of brewery's wastewater processing [27]. Zeta potential (surface charge) can be described as the electro kinetic potential in colloidal systems [28]. If the zeta potential (negative or positive) of the colloids are low, then coagulation and flocculation will take place [29,30].

Pre-treatment of wastewater is aimed at reducing pollutants and removing solids. Coagulation and flocculation are physical and chemical processes and considered as crucial treatment methods for wastewater [31]. This method of treatment has been proven to be very effective over many years and is very cost effective. Coagulation is defined as colloidal suspensions which are destabilized [32-38]. After coagulation has been done, flocculation commences. The flocs are formed, strengthen and enlarged through stirring. This is achieved by the double layer compression (DLVO) theory and cation bridging [24,39]. In accordance to the DLVO theory the total energy of adhesion is the result of the attractive Van der Waals forces and the interaction of the repulsive forces, due to the interpenetration of the electrical double layers [24,40]. Cation bridging uses neutral or like charged EPS to explain flocculation. After charge neutralization the attractive forces become stronger than the repulsive forces as the distance between the particles decreases [24, 40]. Electrostatic and covalent bonding forces mainly take place during flocculation of brewery sludge [41].

This paper aims to characterize and treat brewery effluent sludge through coagulation and flocculation to increase wastewater recycling capabilities within the plant operation.

II. Materials and Procedure

A. Sampling and Preparation

Samples of the brewery wastewater were taken at the discharge point of the treatment plant at Tlokwe Brewery in Potchefstroom, South Africa. Sampling was done using plastic containers with a volume of 25 L and transported from the brewery to the university's laboratory. Testing and characterization of the wastewater before treatment was done, while the water was still fresh and unchanged. The wastewater was analysed and monitored during this time with standard methods. The parameters that were measured with characterization before treatment were: COD, turbidity, pH and zeta potential (surface charge). The containers were covered with black plastic bags after experiments were done to prevent the water changing overnight.

B. Coagulation & Flocculation

The two coagulants that were used are ferric chloride and an organic coagulant mixture in aqueous solution (obtained from brewery) for comparison. The four flocculants that were used are a cationic flocculant (Genesis' genefloc, polyquaternary amine), an anionic flocculant (Senfloc 5210), a non-ionic flocculant (Senfloc 5330) and a cationic acrylamide copolymer in aqueous dispersion (Brewery flocculant – obtained from the brewery) for comparison. Jar tests were conducted using standard procedures. The containers with wastewater were shaken to allow the particles to spread evenly, before filling six 1 L beakers up to 500 mL. A mass of 1 g of the coagulant or flocculant was dissolved in distilled water in a volumetric flask. The dosage for the coagulant or flocculant that was added to the 500 mL wastewater for the jar test was determined by the following formula

$$C_1V_1 = C_2V_2 \quad (1)$$

The optimum dosages of the two coagulants and four flocculants were determined with the same method. A wide range of dosages were chosen namely: 5 ppm, 30 ppm, 60 ppm, 90 ppm, 120 ppm and 150 ppm. After each dosage treatment the turbidity and pH were measured. The turbidity was measured three times to get an average turbidity value. The results according to the turbidity determined a new range of dosages by eliminating some of the dosages in the wide range. This procedure was repeated until an optimum dosage range was determined. The same dosage needs to be done for all coagulant and flocculants involved, before moving on to a next dosage, so that the same conditions apply for that dosage, which makes comparisons and results more accurate. Out of the optimum dosage range which was repeated three times an optimum dosage was determined. The optimum dosage was repeated tree times for accuracy. After each of the three optimum dosage runs, the water was characterized by measuring optimum turbidity, pH and COD. The characterization after treatment versus before treatment determined the performance of the coagulants or flocculants. The jar tests for the coagulants and flocculants were done by adding the coagulant or flocculant and mixing at 220 rpm for 1 min 30 sec. Then the stirring speed was reduced to 40 rpm and the solution was mixed for 15 min. The flocs were then allowed to settle for 30min.

C. Measurements

Zeta potential was measured using NanoPlus' zeta/nano particle analyzer and pH was measured using a pH-meter. Turbidity was measured using a turbidity meter and COD was measured using COD reagent vials. The vials were inserted into the reactor and heated for 2 hours at 150^{o}C. The vials were then allowed to cool down to 120^{o}C. The vials were then inverted for mixing and the sample vials were measured with HANNA instruments' HI 83099 COD and Multiparameter Photometer.

III. Results & Discussion

In the brewery industry there are different treatment processes for brewery wastewater which are necessary to reach the water quality standards. The treatment method of coagulation, flocculation and sedimentation are usually done by existing and traditional water treatment systems. This chapter focusses on the results associated to the performance of coagulants and flocculants used for treatment.

A. Characterization of the brewery wastewater before treatment

The characterization of the brewery wastewater before treatment is important as it gives an idea of which coagulant and flocculant will be best to treat the wastewater. In this study 6 samples before the treatment of the brewery were used for experimentation. Figure 1 shows the results of the characterization of the brewery wastewater before treatment.

Sample 1	
Zeta potential:	-10.44 mV
COD:	1233 mg/L
Turbidity:	82.5 NTU
pH:	10.91

Sample 2	
Zeta potential:	-11.38 mV
COD:	2278 mg/L
Turbidity:	254 NTU
pH:	7.05

Sample 3	
Zeta potential:	-7.05 mV
COD:	2009 mg/L
Turbidity:	224 NTU
pH:	5.65

Sample 4	
Zeta potential:	-7.81 mV
COD:	6732 mg/L
Turbidity:	903 NTU
pH:	5.85

Sample 5	
Zeta potential:	-1.4 mV
COD:	-
Turbidity:	5306.7 NTU
pH:	3.52

Sample 6	
Zeta potential:	-2.14 mV
COD:	11980 mg/L
Turbidity:	1593.33 NTU
pH:	4.59

Fig 1: Results of the characterization of the brewery wastewater before treatment

As seen in Figure 1 the zeta potential for all the samples before treatment are negative. The reason for this is that the ionization of the anionic functional groups of carboxylic and phosphate that gives negative charges to EPS, microbial cells and sludge flocs. A zeta potential with a high negative value means that the colloids in the wastewater are electrically stable. This high negative zeta potential value gives stability to the particles, decreasing the coagulation and flocculation ability of the solids. This means coagulation and flocculation tend to not take place. The closer the negative zeta potential value gets to a value of zero the better the coagulation and flocculation ability of the solids in the wastewater. This means that a low negative zeta potential value will have the tendency for coagulation and flocculation to take place. The zeta potential values in Figure 1 ranges from -1 to -11 which is low zeta potential values. This means that coagulation and flocculation will take place. The pH is an important factor which influences the zeta potential. In Figure 1 it can be seen that the lower the pH value, the less negative the zeta potential value. The closer the zeta potential value to zero the better the tendency for coagulation and flocculation to take place. There is also a relation between COD and turbidity values. In Figure 1 it can be seen that the higher the turbidity value is the higher the COD value and also the other way around. In Figure 1 sample 5 does not have COD value, because of challenges encountered at the time of water collection; however, the other parameters are reported for better interpretation of the coagulation and flocculation results.

B. Determination of optimum dosages of the coagulants and flocculants

In this study, 2 coagulants and 4 flocculants were used. EPS and organic particles carry a negative charge (zeta potential) and therefor the cationic (positive) flocculants are likely to give the best results. With some waste waters a coagulant is used to neutralize the particle charges. The only charges remaining in the waste water is the hydrogen and hydroxide ions. If the hydrogen ion concentration is more than the hydroxide ion concentration an anionic flocculant will be more effective and when the hydroxide ion concentration is more than the hydrogen ion concentration a cationic flocculant will be more effective. Therefore, an anionic flocculant, non-ionic flocculant and cationic flocculant will be tested to determine which flocculant will be more effective.

C. Turbidity and pH

The turbidity of the sorghum brewery wastewater is increased by the organic substance namely sorghum malt. The main causes of turbidity in the brewery wastewater are rinsing of filters and the cleaning of the brewing process equipment. The turbidity of the wastewater before treatment ranged from 200 NTU – 6000 NTU. The wastewater discharged from the brewery needs to be within a specified pH range. The wastewater produced by the brewery has low pH values due to the sorghum malt that is added and the souring stage. The pH decreases when the live fermenting sorghum beer is unsold and given back to the brewery for discharge. All the samples taken at the brewery was before any treatment was done on the wastewater which means before sodium hydroxide was added. This means samples were taken at pH value between 4 and 6. The pH had to be constant otherwise the experimental results will fluctuate and give inaccurate results. The optimum dosage for both coagulants was determined at pH value between 4 and 6. The optimum dosage ranges for both coagulants were determined using wastewater with a pH value of 5.65 before treatment. All the other experimental results were done using wastewater with a pH value between 4 and 6. Fig. 2 and Fig 3 represent the optimization dosage ranges of both the coagulants.

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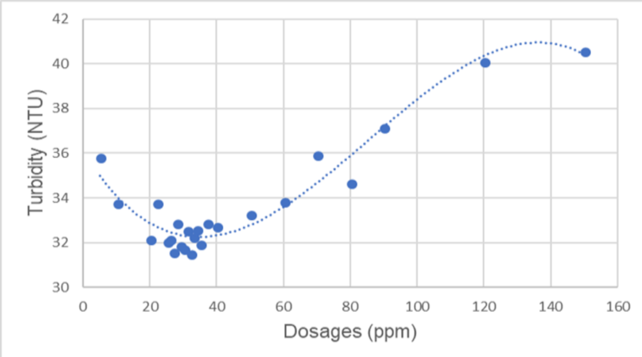

Fig 2: Determination of optimum dosage range of the coagulant ferric chloride

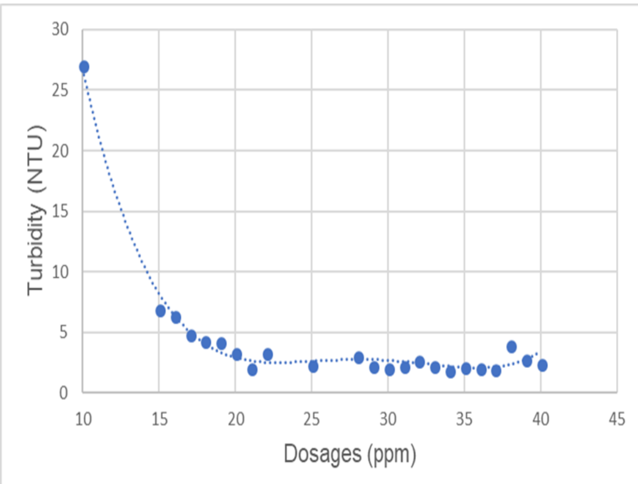

Fig 3: Determination of the optimum dosage range of the organic coagulant mixture in aqueous solution

After determining the optimum dosage ranges for both coagulants, the optimum dosage was determined with consideration of the optimum dosage ranges using the same sample for comparison. Each dosage in the optimum range was repeated 3 times for accuracy. Fig 4 and Fig 5 represent the determination of the optimum dosages of both the coagulants.

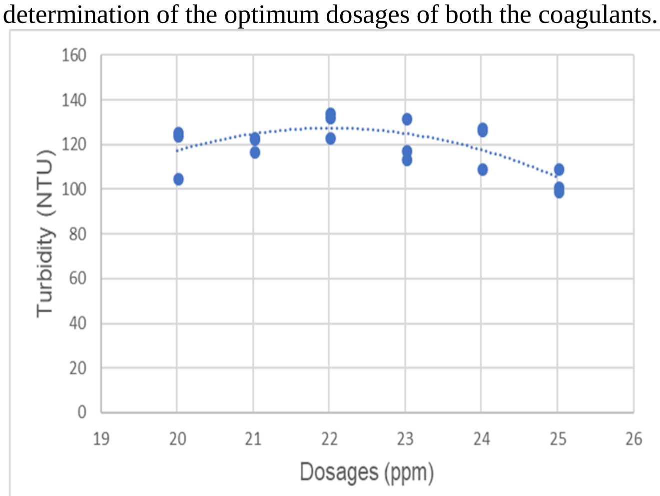

Fig 4: Determination of optimum dosage of the coagulant ferric chloride

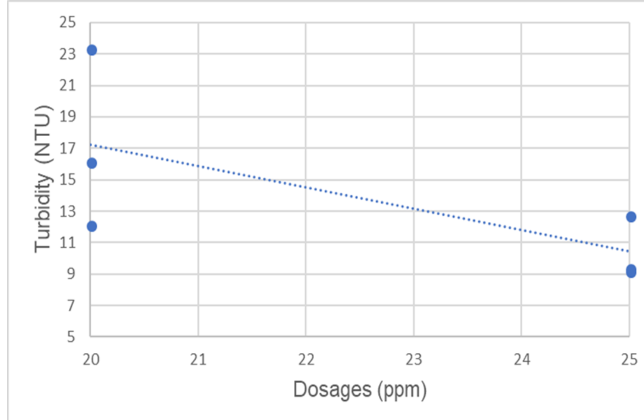

Fig 5: Determination of optimum dosage of the organic coagulant mixture in aqueous solution

The optimum dosage range for the organic coagulant mixture in aqueous solution was 20 ppm to 25 ppm. 25 ppm dosage was also taken as the optimum dosage at the organic coagulant mixture in aqueous solution then a better comparison of the effectiveness could be done between the two coagulants as they have the same optimum dosage. The optimization process of the flocculants was done by adding the optimum dosage of the coagulants first and then the dosages of the flocculants. The reason for this was to get the best combination of results with coagulant and flocculant. The optimization process was done with the same sample for accurate experimental results. The sample that was used for the optimization process of the flocculants was Sample 4. Fig 6 to Fig 13 represent the determination of the optimum dosage of the flocculants when used with both coagulants.

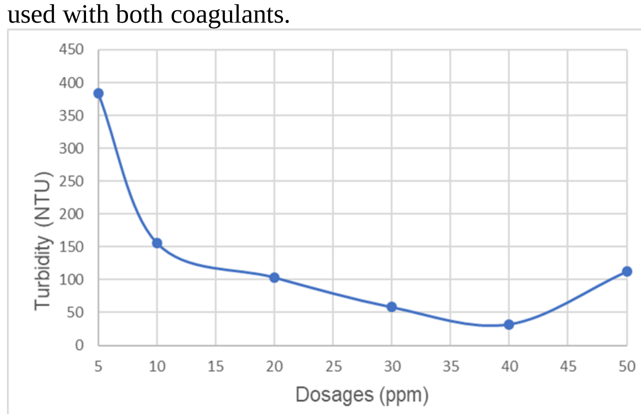

Fig 6: Determination of the optimum dosage of the cationic flocculant (Genesys' genefloc, polyquaternary amine) when used with the coagulant ferric chloride

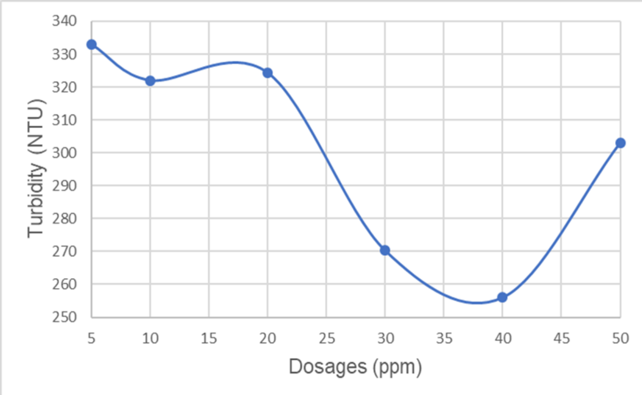

Fig 7: Determination of the optimum dosage for the anionic flocculant (Senfloc 5210) when used with the coagulant ferric chloride

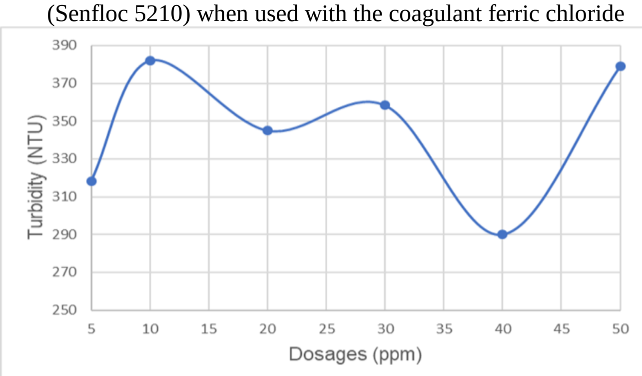

Fig 8: Determination of the optimum dosage for the non-ionic flocculant (Senfloc 5330) when used with the coagulant ferric chloride

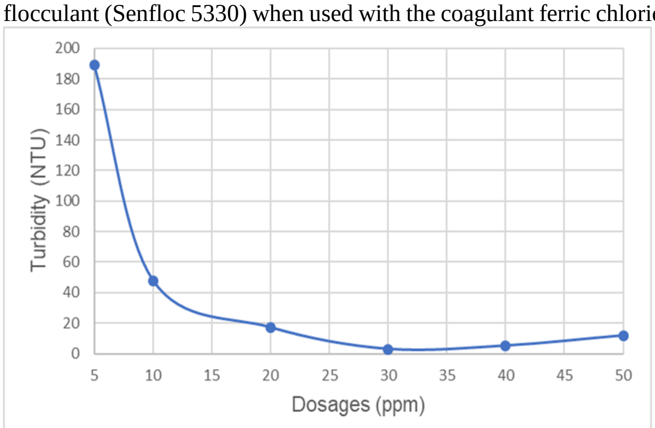

Fig 9: Determination of the optimum dosage for the cationic acrylamide copolymer in aqueous dispersion (Brewery flocculant) when used with the coagulant ferric chloride

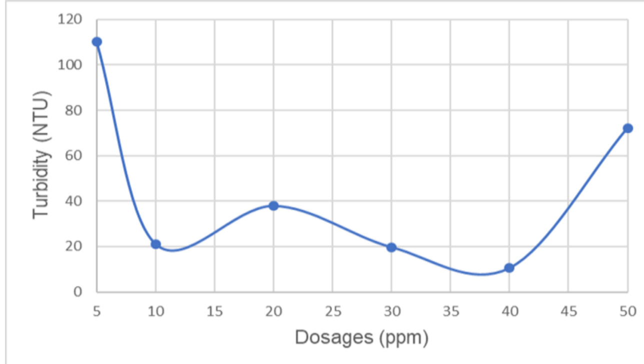

Fig 10: Determination of the optimum dosage for the cationic flocculant (Genesys' genefloc, polyquaternary amine) when used with the organic coagulant mixture in aqueous solution

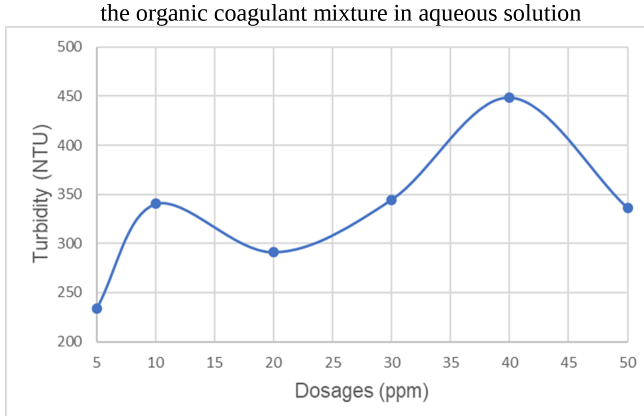

Fig 11: Determination of the optimum dosage for the anionic flocculant (Senfloc 5210) when used with the organic coagulant mixture in aqueous solution

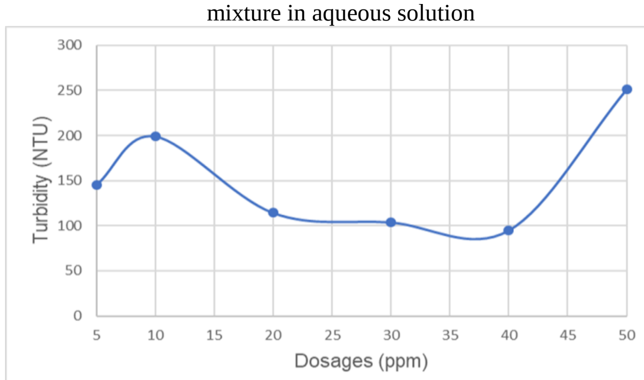

Fig 12: Determination of the optimum dosage for the non-ionic flocculant (Senfloc 5330) when used with the organic coagulant mixture in aqueous solution

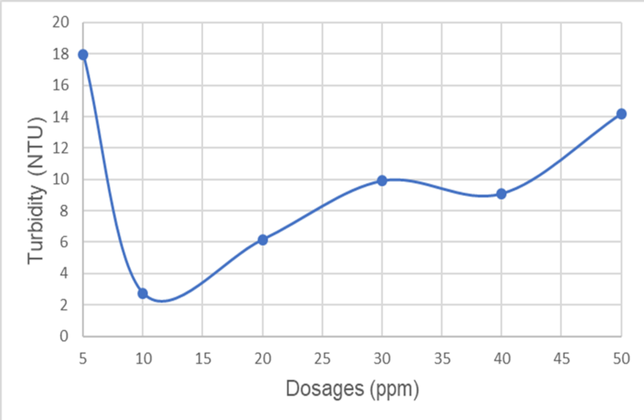

Fig 13: Determination of the optimum dosage for the cationic acrylamide copolymer in aqueous dispersion (Brewery flocculant) when used with the organic coagulant mixture in aqueous solution

The pH before treatment versus the average pH after treatment following the dosage of coagulant or flocculant showed that the pH was relatively constant throughout the experiments which gave acceptable results. This means that pH did not have an influence on the results. The pH values before treatment was between 5 and 6 and remain relatively the same after treatment.

D. *Turbidity and COD removal efficiency of coagulants*

Table 1 represents the turbidity, COD and pH measurements/characterization at the optimum dosages of the two coagulants after treatment.

TABLE I: THE TURBIDITY, COD AND PH MEASUREMENTS/CHARACTERIZATION AT THE OPTIMUM DOSAGES OF THE TWO COAGULANTS AFTER TREATMENT

Sample 3			
Ferric Chloride			
Optimum Dosage (ppm)	Turbidity (NTU)	pH	COD (mg/L)
25	93.56666667	4.81	1356
25	97.03333333	4.77	1430
25	65.16666667	5.7	1385
25	73.23333333	5.08	1241
Average:	82.25	5.09	1353
Organic coagulant mixture in aqueous solution			
25	14	4.89	1011
25	23.66666667	4.81	1167
25	16.86666667	4.78	923
Average:	18.17777778	4.826667	1033.66667

Table 2 represents the turbidity and COD removal efficiencies of both coagulants at optimum dosage.

TABLE II: THE TURBIDITY AND COD REMOVAL EFFICIENCIES OF BOTH COAGULANTS AT OPTIMUM DOSAGE

Coagulants	Optimum Coagulant Dosage (ppm)	Turbidity removal efficiency (%)	COD removal efficiency (%)
Ferric Chloride	25	63.3	32.7
Organic coagulant mixture in aqueous solution	25	91.9	48.5

E. *Turbidity and COD removal efficiency of flocculants when used with coagulants*

Table 3 represents the turbidity, COD and pH measurements/characterization at the optimum dosages of the flocculants.

TABLE III: THE TURBIDITY, COD AND PH MEASUREMENTS/CHARACTERIZATION AT THE OPTIMUM DOSAGES OF THE FLOCCULANTS

Sample 6				
Combination	Optimum Flocculant Dosage (ppm)	Turbidity (NTU)	pH	COD (mg/L)
Ferric + Cationic (Genesys)	40	114.6666667	4.42	4493
Ferric + Brewery Floc	30	4.616666667	4.24	2448
Ferric + Non-ionic	40	237	4.53	5656
Ferric + Anionic	40	230.6666667	4.44	5008
Brewery + Cationic (Genesys)	40	19.36666667	4.56	3698
Brewery + Brewery Floc	10	4.893333333	4.31	2608
Brewery + Non-ionic	40	94.43333333	4.5	4379
Brewery + Anionic	20	193	4.5	4867

Table 4 represents the turbidity and COD removal efficiencies of the flocculants together with the coagulants at optimum dosage.

TABLE IV: THE TURBIDITY AND COD REMOVAL EFFICIENCIES OF THE FLOCCULANTS WHEN USED WITH THE COAGULANTS AT OPTIMUM DOSAGE

Combination	Optimum Flocculant Dosage (ppm)	Turbidity removal efficiency (%)	COD removal efficiency (%)
Ferric + Cationic (Genesys)	40	92.8	62.5
Ferric + Brewery Floc	30	99.7	79.6
Ferric + Non-ionic	40	85.1	52.8
Ferric + Anionic	40	85.5	58.2
Brewery + Cationic (Genesys)	40	98.8	69.1
Brewery + Brewery Floc	10	99.7	78.2
Brewery + Non-ionic	40	94.1	63.4
Brewery + Anionic	20	87.9	59.4

F. *Turbidity and COD removal efficiency of brewery*

Table 5 represents the results of the characterization of the brewery wastewater after treatment using the brewery's treatment method.

TABLE V: THE RESULTS OF THE CHARACTERIZATION OF THE BREWERY WASTEWATER AFTER TREATMENT USING THE BREWERY'S TREATMENT METHOD.

Sample 6: Quality of water treated at the brewery	
COD:	6732 mg/L
Turbidity:	674.67 NTU
pH:	4,69

The turbidity and COD removal efficiency results of the brewery wastewater treatment are 57.7% and 43.8%. The coagulation and flocculation treatment method used in this study resulted in a massive improvement on the turbidity and COD removal efficiencies compared to the brewery's treatment method. The best results were obtained using the cationic acrylamide copolymer in aqueous dispersion (Brewery flocculant) and cationic flocculant (Genesys' genefloc, polyquaternary amine). This means that when the zeta potential is negative before treatment a cationic flocculant will function the best and when the zeta potential is positive before treatment an anionic flocculant will function the best. The combination of coagulant and flocculant that gave the best results were 30 ppm cationic acrylamide copolymer in aqueous dispersion (Brewery flocculant) used with the ferric chloride, and 10 ppm cationic acrylamide copolymer in aqueous dispersion (Brewery flocculant) used with the organic coagulant mixture in aqueous solution. The acceptable limit of COD level in brewery's wastewater to be discharged in the environment must be lower than 2500 mg/L. If the COD is higher than 2500 mg/L, then the brewery has to pay fines. Depending on how high the turbidity and the COD values are at the beginning of treatment determines also if the ability of the treatment process to reduce the COD lower than 2500 mg/L. The coagulation and flocculation treatment method will help the brewery to save money by not longer paying fines related to disposal of too dirty wastewater. The brewery uses a dosage of the coagulant and flocculant of 80 ppm organic coagulant mixture in aqueous solution and 20 ppm cationic acrylamide copolymer in aqueous dispersion (Brewery flocculant). This study achieved optimum dosage values of the coagulant and flocculant lower than that used by the brewery. Efficient optimum dosages of 25 ppm organic coagulant mixture in aqueous solution and 10 ppm cationic acrylamide copolymer in aqueous dispersion (Brewery flocculant) were determined. This study achieved lower dosage values for the coagulant and flocculant compared to the brewery's values, this will allow to save money. The best results were achieved using 30 ppm cationic acrylamide copolymer in aqueous dispersion (Brewery flocculant) in combination with the ferric chloride. This means that a better combination of coagulant and flocculant was determined. The price of the organic coagulant mixture in aqueous solution is R 27.21 per kg and the price of ferric chloride is R 6.74 per kg, so the ferric chloride coagulant is much cheaper than the brewery coagulant. This means that the combination of 30 ppm cationic acrylamide copolymer in aqueous dispersion (Brewery flocculant) with the ferric chloride which is a different combination of coagulant and flocculant to the one used by the brewery, will result to financial gain and improved water treatment.

IV. Conclusion

The brewery industry is forced by tight government legislation to look at wastewater recycling as a method to save water, the environment and cut down costs on pre-treatments for discharge to municipal sewer systems. Therefore, breweries need to have the correct treatment methods in place. This study was done to observe the effect of coagulation, flocculation and sedimentation on turbidity and COD removal of brewery wastewater. From this paper it can be seen that the characterization of wastewater is necessary for the selection of the suitable type and dosage of coagulant and flocculant. It can also be seen that with the use of the correct dosages, coagulation, flocculation and sedimentation is not only an effective treatment method but also cost effective.

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