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Coagulation/flocculation process and sludge conditioning in beverage industrial wastewater treatment

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Abstract

Attempts were made in this study to examine the effectiveness of coagulation and flocculation process using ferric chloride and polyelectrolyte (non-ionic polyacrylamide) for the treatment of beverage industrial wastewater. Removal of organic matter (expressed as chemical oxygen demand, COD), total phosphorus (TP) and total suspended solid (TSS) using ferric chloride and organic polyelectrolyte during coagulation/flocculation process were investigated. Also, the optimum conditions for coagulation/flocculation process, such as coagulant dosage, polyelectrolyte dosage, and pH of solution were investigated using jar-test experiment. The effect of different dosages of polyelectrolyte in combination with coagulant was also studied. The results revealed that in the range of pH tested, the optimal operating pH was 9. Percentage removals of 73, 95 and 97 for COD, TP and TSS, respectively, were achieved by the addition of 300 mg/L $FeCl_3{\cdot}6H_2O$, whereas 91, 99 and 97% removal of COD, TP and TSS, respectively, were achieved with the addition of 25 mg/L polyelectrolyte to 100 mg/L ferric chloride. The volume of sludge produced, when ferric chloride was used solely, was higher compared to the use of combination of polyelectrolyte and $FeCl_3{\cdot}6H_20$. The combined use of coagulant and polyelectrolyte resulted in the production of sludge volume with reduction of 60% of the amount produced, when coagulant was solely used for the treatment. It can be concluded from this study that coagulation/flocculation may be a useful pre-treatment process for beverage industrial wastewater prior to biological treatment.

Keywords: Coagulation; Polyelectrolyte; Optimal conditions; Sludge volume; Beverage wastewater

1. Introduction

Coagulation/flocculation process may be used as a pre-treatment prior to biological treatment in order to enhance biodegradability of the wastewater during the biological treatment [1].

Some of the raw materials (e.g. orange, grape, guava, sugar, phosphates) that are used for production of the beverages may enhance the organic load of the wastewater. An essential feature of wastewater flocculation is the elimination of suspended solids (SS) and as much of the organic materials as possible [2]. To remove SS and organics, a floc forming chemical is needed which can be separated from water by floatation, settling, or adsorption.

Coagulation/flocculation is a commonly used process in water and wastewater treatment in which compounds such as ferric chloride and/or polymer are added to wastewater in order to destabilize the colloidal materials and cause the small particles to agglomerate into larger settleable flocs. Several studies have reported the examination of this process for the treatment of industrial wastewater, especially with respect to performance optimization of coagulant/flocculant, determination of experimental conditions, assessment of pH and investigation of flocculant addition [3,4].

Although several studies have been conducted into the treatment of municipal wastewater through granular activated carbons during which COD and some volatile organic compounds, such as chloroform benzene and carbon tetrachloride were adsorbed to the carbon surface [5,6]. The use of adsorbents [7,8], Fenton's reagent [9], wet oxidation [10], coagulation–electrooxidation [11], advanced oxidation with biological oxidation [12] and coagulation/flocculation process have

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been found to be cost effective, easy to operate and energy saving treatment alternatives [1,13]. Coagulant dosages vary in a wide range aiming at maximum removal efficiency of pollutants using minimum doses at optimum pH [14,15].

$FeCl_3 \cdot 6H_2O$ is a widely used coagulant. It has been used for the treatment of wastewater of industries that are concerned with the production of potato chips [16], soap/detergent [17] and cork processing [18].

The objectives of this study were to simulate coagulation/flocculation process efficiency for beverage industrial wastewater treatment plant with respect to removal of COD, TP and TSS using ferric chloride and non-ionic polyacrylamide and also to investigate optimum coagulant dosages, optimum coagulation pH and effect of polyelectrolyte addition on the coagulation process.

2. Materials and methods

Samples of the wastewater were collected three times weekly for 9 months between 9.00 a.m. and 3.00 p.m. of each sampling day from pipe conveying all the wastewater out of Fumman Agricultural Products Factory that produces juice drink from orange, grape, and guava, etc. Each time a sample was collected, the rate of flow was determined with the aid of a flow-meter. Samples were collected every hour between 9.00 a.m. and 3.00 p.m. to make six samples at the end of each day of sampling; a composite sample was generated by adding together volumes of samples proportional to their rates of flow. The estimated annual outputs of the factory at the time of sampling were 7.73×10^8 kg (773,000 t). The average volume of wastewaters generated daily from the factory was 3.20×10^4 L. The composition of the beverage wastewater is as shown in Table 1.

2.1. Sample analyses

Each time a sample was collected, the temperature, pH and dissolved oxygen (D.O.) were determined at the spot of sampling with a good grade Celsius thermometer (Zeal, England), portable pH meter and portable D.O. meter (model 9071 $D.O_2$ meter, Phillips, England), respectively.

Table 1
Characteristics of beverage industry wastewater

Parameter	No. of samples	Mean	Minimum	Maximum
pH	80	7.24	7.02	7.66
TSS (mg/L)	40	1620	367	2940
Conductivity ($\mu S\,cm^{-1}$)	40	2995	1460	3740
Alkalinity (mg $CaCO_3$/L)	40	371	270	465
COD (mg/L)	40	1750	620	3470
BOD_5 (mg/L)	40	894	728	1745
Total phosphorus (mg P/L)	40	89.5	62.4	100.2
Orthophosphate (mg P/L)	40	41.2	25.5	70.6
Nitrate–nitrogen (mg N/L)	40	28.4	8.3	62.5
Total iron (mg $Fe^{2+} + Fe^{3+}$/L)	40	2.4	1.2	4.5

BOD_5: Biochemical oxygen demand at 5 days.

2.2. Experimental

1. 100, 300, 500, 750, or 1000 mg $FeCl_3 \cdot 6H_2O$ was added to 1 L wastewater sample. After rapid mixing for 2 min at 200 rpm and slow mixing for 30 min at 60 rpm, the liquid was clarified for 1 h, then, the supernatant was withdrawn from a point located about 2 cm below the top of the liquid level of the beaker to determine the COD, TP and TSS by using standard methods [19] so that the effect of coagulant dose could be studied. The raw and treated samples were repeatedly analyzed in order to validate/evaluate the produced results and the analytical errors were less than ±5%. All chemicals used for the analytical determinations were of analytical grade.
2. The pH value of 1 L wastewater sample was adjusted to pH in the range of 7–11, respectively, by using 1.0 M H_2SO_4 or 1.0 M NaOH, after the addition of 300 mg/L $FeCl_3 \cdot 6H_2O$ to the sample. After stirring and clarifying as described in (1) above, the supernatant was withdrawn to determine the COD, TP and TSS, so that the effect of pH on coagulation could be studied.
3. Pre-determined optimum value of $FeCl_3 \cdot 6H_2O$ (300 mg/L) was added to the wastewater sample. After rapid mixing for 2 min at 200 rpm, the pH was adjusted to the pre-determined value by using 1.0 M H_2SO_4 or 1.0 M NaOH. Different concentrations (5, 25, 45, 65, 85, or 100 mg/L) of polyelectrolyte were added and the liquid was mixed slowly for 30 min at 60 rpm, the supernatant was withdrawn as earlier described to determine the optimum polyelectrolyte that enhances coagulation. The pH was again measured after the optimal polyelectrolyte dose had been determined. After the withdrawal of supernatant, the volume of wet sludge produced was determined from the sludge level on the bottom of the jar-test beakers.

3. Results and discussion

3.1. Effects of coagulant dose on coagulation

The results of the effects of different dosages of ferric chloride as sole coagulant on the removal of COD, TP and TSS from the wastewater are as presented in Fig. 1. The tests were conducted at the pre-determined optimum pH of 9.

It is clear from the results that COD, TP and TSS removals increased substantially as the dosage of ferric chloride increases. It was observed that removal of TSS increased linearly up to 300 mg/L. However, TSS increased in a diminishing fashion with increasing doses of ferric chloride above 300 mg/L. This may be as a result of re-suspension of solids at this concentration [16]. Furthermore, the high concentrations (>300 mg/L) of the coagulant confer positive charges on the particle surface (a positive zeta potential), thus re-dispersing the particles [20]. For COD and TP, the removal efficiencies increased rapidly up to 73 and 97%, respectively, with the use of 300 mg/L dose of ferric chloride, whereas between 300 and 500 mg/L the removal of COD and TP increased slowly. Addition of the coagulant above 500 mg/L caused the removal efficiency to appear constant. At this point it is important to note that optimum dose of ferric

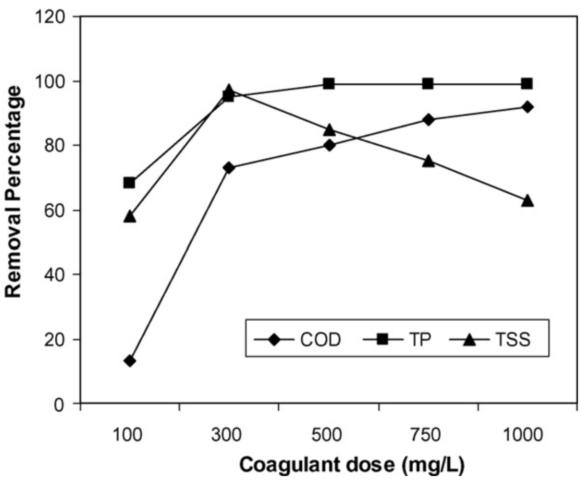

Fig. 1. The effect of coagulant doses on the removal of COD, TP and TSS from the wastewater.

chloride that enhanced maximum removal of COD, TP and TSS was 300 mg/L.

3.2. Effects of pH on coagulation

A treatment set-up comprising of only ferric chloride at 300 mg/L was selected to examine the effect of different pH of solution on the removal of COD, TP and TSS in the wastewater. These effects are shown in Fig. 2. It was observed that optimum pH value of 9 enhanced substantial removal of the contaminants. Increasing the pH above 9 markedly deteriorated the quality of the wastewater. In the pH range of 7–9, contaminants level decreased as pH increases.

Coagulation pH as a factor that influences coagulation is important because addition of metallic cation (in this case Fe^{3+}) automatically lowers pH, which may cause further reduction in the removal of the contaminants. The need to employ higher dose of coagulant may pose health hazard as a result of residual quantities of excess chemical additives [21]. The excess residual

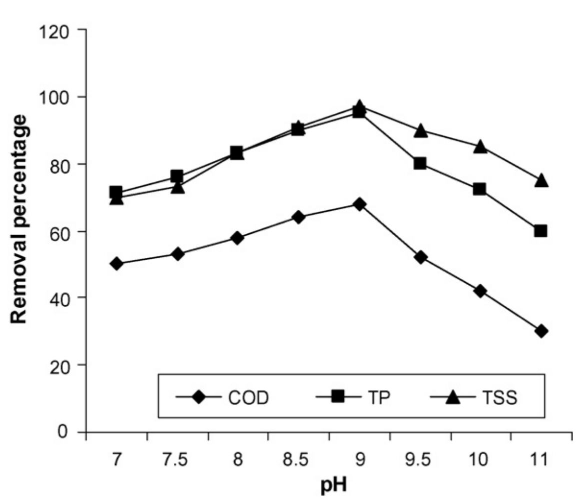

Fig. 2. The effect of different pH on the removal of COD, TP and TSS from the wastewater.

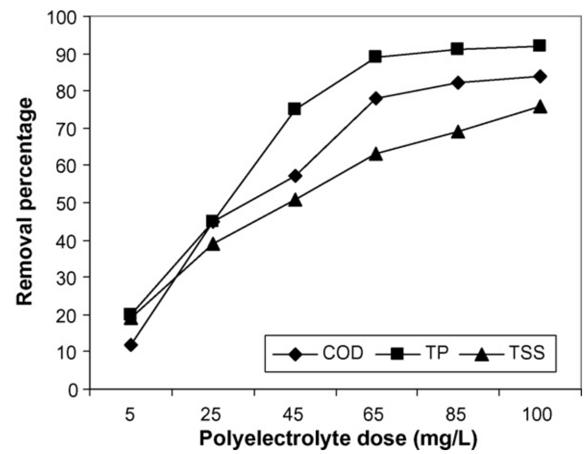

Fig. 3. The effect of polyelectrolyte dose on the removal of COD, TP and TSS from the wastewater.

coagulant, when discharged into surface water, interferes with fish survival and growth [22].

3.3. Effects of polyelectrolyte dose on the removal of the contaminants

Polyelectrolytes act as coagulant aids in the treatment of water and wastewater; they may also be used as primary coagulant for the same purpose [1,23,24]. Many polyelectrolytes are advantageous over chemical coagulants because they are safer to handle and are easily biodegraded [24,25]. The percentage removal of COD, TP and TSS as functions of different doses of polyelectrolyte is as shown in Fig. 3. The dose of polyelectrolyte was varied from 0 to 100 mg/L. The removal of COD, TP and TSS increased with increasing dose of polyelectrolyte.

3.4. Effects of polyelectrolyte on coagulation process

Several studies had reported the use of combination of polyelectrolyte (as a coagulant aid) and chemical coagulant in the treatment of abattoir wastewater [1,19,23,26,27,28]. The dose of polyelectrolyte (non-ionic polyacrylamide) was varied from 0 to 100 mg/L and the ferric chloride dose was made constant at either 100 or 300 mg/L. In order to determine the optimal dose of the polyelectrolyte, the percentage removal of COD, TP and TSS was considered (Tables 2 and 3).

From Table 2, it can be seen that removal of COD reached 70% during the use of 100 mg/L ferric chloride and 5 mg/L polyelectrolyte. The COD percentage increased to 91% when

Table 2
The effects of addition of different doses of polyelectrolyte to 100 mg/L dose of $FeCl_3{\cdot}6H_2O$ on the removal of COD, TP and TSS from the wastewater

Parameter	Dose of polyelectrolyte (mg/L)					
	5	25	45	65	85	100
COD (%)	70	91	93	95	95	96
TP (%)	81	99	99	99	99	99
TSS (%)	74	97	96	94	90	82

Table 3
The effects of addition of different doses of polyelectrolyte to 300 mg/L dose of $FeCl_3 \cdot 6H_2O$ on the removal of COD, TP and TSS from the wastewater

Parameter	Dose of polyelectrolyte (mg/L)					
	5	25	45	65	85	100
COD (%)	84	95	96	96.6	97	98
TP (%)	97	99	99	99	99	99
TSS (%)	88	85	72	63	50	41

the dose of the polyelectrolyte was increased to 25 mg/L, this slowly increased to 93% when the dose of the polyelectrolyte was increased to 45 mg/L. Further increase in the dose of the polyelectrolyte did not cause further increase in the percentage removal of COD. However, COD removal slowly increased to 96% with the addition of 100 mg/L of polyelectrolyte to the coagulation process.

Percentage removal of TP reached maximum (99) with the use of 100 mg/L ferric chloride and 25 mg/L dose of the polyelectrolyte. Addition of polyelectrolyte above 25 mg/L caused no increase in the removal of TP.

For the removal of TSS, maximum removal of 97% was achieved with the use of 100 mg/L ferric chloride and 25 mg/L polyelectrolyte. Further addition of polyelectrolyte above 25 mg/L caused gradual decrease in the removal of TSS. This may be due to the re-suspension of particles at higher doses of the polyelectrolyte.

From Table 3, there was improvement in the removal of COD with the use of 300 mg/L ferric chloride and 10 mg/L polyelectrolyte. The removal of COD linearly increased with increasing dose of the polyelectrolyte above 25 mg/L, until 98% removal was achieved at 100 mg/L polyelectrolyte. This may be due to formation of a greater number of flocs, also, increasing the dose of the coagulant increased the supersaturation of the $Fe(OH)_3$ which increased the nucleation rate and hence the floc growth rate. As a result, suspension of greater number of flocs was enhanced, and subsequently, removal of larger amount of organic matter was achieved, due to the availability of larger surface area on which adsorption of the organic matter took place. On the contrary, low doses of coagulant led to the formation of larger but fewer flocs as a result of faster growth rate relative to nucleation rate, which resulted in a smaller surface area on which adsorption of organic matter took place.

The optimum dose of a coagulant or flocculant is defined as the value above which there is no significant difference in the increase in removal efficiency with a further addition of coagulant or flocculant [23]. Thus, the optimum doses of ferric chloride and polyelectrolyte that enhanced COD removal were 100 and 25 mg/L, respectively. The use of 300 mg/L dose of ferric chloride at different dose of polyelectrolyte increased the removal of COD at all the doses of the polyelectrolyte, hence, this would not be a suitable decisive system for determining optimum dose.

Either 100 or 300 mg/L dose of ferric chloride and different doses of polyelectrolyte achieved between 81 and 99% removal of TP. However, TP did not seem to be a decisive parameter for determining the optimum dose because its removal from the wastewater increased with increasing dose of the polyelectrolyte up to the maximum concentration (100 mg/L) employed. A synergistic effect of ferric chloride–polyelectrolyte combination achieved higher percentage removal of the contaminants (Table 2). The optimum doses of ferric chloride and polyelectrolyte that enhanced higher TSS removal were 100 and 25 mg/L, respectively.

Table 4
Residual concentration of iron ($Fe^{2+} + Fe^{3+}$) (mg/L) as a function of coagulant dose

Coagulant dose (mg/L)	Residual ($Fe^{2+} + Fe^{3+}$) (mg/L)
0	2.40
100	2.07
300	1.83
500	1.23
750	0.52
1000	0.11

3.5. Residual iron test

The residual concentrations of iron ($Fe^{2+} + Fe^{3+}$) in the clear supernatant after treatment are as shown in Table 4.

It was observed that increasing the dose of ferric chloride, decreased the residual iron concentration in the supernatant. This behaviour seems to be inherent to the coagulation/flocculation process [18]. The higher dose of ferric chloride increased supersaturation of $Fe(OH)_3$ thus, increased nucleation rate of $Fe(OH)_3$ which consequently removed the iron in solution more effectively by being adsorbed and/or retained by the precipitate. Increase in the supersaturation of $Fe(OH)_3$ nuclei favours their union and hence more effective coagulation/flocculation.

3.6. Effects on solid production

In general, the amount and the characteristics of the sludge produced during the coagulation/flocculation process are highly dependent on the specific coagulant used and on the operating conditions [23]. The wet sludge volume at the bottom of the jar test beakers after coagulation/flocculation process was used to quantify the volume of sludge generated in this study. The volume (mL/L) of the settled sludge is shown as functions of coagulant type and dose (mg/L) in Table 5.

From Table 5, it can be seen that the volume of sludge produced, reduced considerably with increasing dose of polyelec-

Table 5
Volume of sludge produced during the coagulation/flocculation process

Coagulant type and dose	Volume of sludge
Ferric chloride (100 mg/L)	282
Ferric chloride (300 mg/L)	669
Ferric chloride:polymer (100:10) (mg/L)	170
Ferric chloride:polymer (100:20) (mg/L)	132
Ferric chloride:polymer (100:25) (mg/L)	94
Ferric chloride:polymer (300:10) (mg/L)	578
Ferric chloride:polymer (300:20) (mg/L)	491
Ferric chloride: polymer (300:25) (mg/L)	425

trolyte in the coagulation process. This may be due to non-ionic nature of the polyelectrolyte employed in this study, which has high molecular weight, thus, providing long bridges between small flocs to enhance particle growth. It also has the ability to attract and hold colloidal particles at polar sites on the molecule. Generally, organic polymers generate less sludge than inorganic salts since they do not add weight or chemically combine with other ions in the water to form precipitate. Thus, the sludge produced by the use of ferric chloride in combination with polyelectrolyte was compact and reduced in volume. These findings are in accordance with our previous studies [1,23] and those of others [26,28,29]. The water content of the sludge produced when ferric chloride alone was used and that produced when combination of ferric chloride and polyelectrolyte was used were measured after centrifugation and it was found that combination of ferric chloride and polyelectrolyte produced much water and less sludge than when ferric chloride alone was used.

4. Conclusions

(1) Coagulation/flocculation process was conducted for the treatment of beverage industrial wastewater to achieve maximum removal of COD, TP and TSS. Coagulant dose, polyelectrolyte dose, pH of solution and addition of polyelectrolyte as coagulant aid were investigated and found to be important parameters for effective treatment of beverage industrial wastewater.

(2) Coagulation process using 300 mg/L $FeCl_3{\cdot}6H_2O$ efficiently reduced COD, TP and TSS by 73, 95 and 97%, respectively. Increasing the coagulant dose above 400 mg/L, however, caused TSS to increase in a diminishing fashion. This was as a result of re-suspension of solids at this dose (>400 mg/L). Also, the high concentration (>400 mg/L) of $FeCl_3{\cdot}6H_2O$ confers positive charges on the particles surface (a positive zeta potential), thus, re-dispersing the particles.

(3) Coagulation was enhanced at pH range of 7–9, below which hydrogen ions compete with $FeCl_3{\cdot}6H_2O$ for COD, TP and TSS, resulting in poor removal of the contaminants and above which there can be production of negatively charged organic contaminants on which adsorption is electrostatically hindered. In this case, higher dose of metallic cation (coagulant) will be required; this might pose a health hazard as a result of residual quantities of excess chemical additives, which can also interfere with fish survival and growth in the receiving surface water. The optimum pH for the coagulation process was 9.

(4) Results of the present work indicated that addition of 25 mg/L polyelectrolyte reduced the dose of ferric chloride from 300 to 100 mg/L during which 99, 97 and 91% of TP, TSS and COD removal were, respectively, achieved.

(5) Increasing the concentration of $FeCl_3{\cdot}6H_2O$ enhanced increasing supersaturation of $Fe(OH)_3$ nuclei, thus creating larger surface area on which organic matter can be adsorbed. This also reduced the concentration of residual iron in the supernatant because the iron may have been adsorbed and/or retained by the precipitate $(FeOH)_3$:

$$FeCl_3 + 3H_2O \rightarrow Fe(OH)_3{\downarrow} + 3H^+ + 3Cl^-$$

(6) The combined use of coagulant and polyelectrolyte resulted in the production of sludge volume with reduction of 60% of the amount produced when coagulant was solely used for the treatment.

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