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Assessment of Real Confectionery Industry Wastewater: Characterization and Treatability with Coagulation-Flocculation

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Abstract: It is important to treat the confectionery industry wastewater with appropriate methods due to the high organic pollutants it contains. When these wastewaters are discharged without pretreatment, they cause serious environmental problems such as eutrophication. In this study, real confectionery industry wastewater was taken from Gebze Plastics Organized Industrial Zone (Türkiye). Pollutant concentrations were determined by detailed characterization of the wastewater. Lime, polyaluminium chloride and ammonim persulfate coagulants were added and chemical oxygen demand, total phosphate, orthophosphates, total nitrogen and ammonium nitrogen removal by coagulation-flocculation were investigated. The optimum concentrations of the chemicals used in the study were found to be lime: 0.75 g L^{-1}, polyaluminum chloride: 0.5 g L^{-1} and ammonium persulfate: 0.004 g L^{-1}, respectively; and the optimum pH conditions were found to be lime: 10.9, polyaluminum chloride: 8.1 and ammonium persulfate: 8.1, respectively. In the study, chemical oxygen demand, total phosphate, orthophosphates, and ammonium nitrogen removal efficiencies were ≥86%, and total nitrogen removal efficiency was 43%. Conformity of confectionery industry wastewater, which causes serious environmental problems such as eutrophication in case of direct discharge to the receiving environment, to the sewage discharge criteria after pretreatment was also investigated.

Gerçek Şekerleme Endüstrisi Atıksularının Değerlendirilmesi: Karakterizasyonu ve Koagülasyon-Flokülasyon ile Arıtılabilirliği

Anahtar Kelimeler
Şekerleme endüstrisi,
Gerçek atıksu,
Koagülasyon-flokülasyon

Öz: Şekerleme endüstrisi atık suyunun içerdiği yüksek organik kirleticiler nedeniyle uygun yöntemlerle arıtılması önemlidir. Bu atık sular ön arıtım yapılmadan deşarj edildiğinde ötrofikasyon gibi ciddi çevresel sorunlara neden olmaktadır. Bu çalışmada, Gebze Plastik Organize Sanayi Bölgesi'nden (Türkiye) gerçek şekerleme endüstrisi atık suyu alınmıştır. Atıksuyun detaylı karakterizasyonu ile kirletici konsantrasyonları belirlenmiştir. Kireç, polialüminyum klorür ve amonyum persülfat koagülantları ilave edilmiş ve koagülasyon-flokülasyon ile kimyasal oksijen ihtiyacı, toplam fosfor, ortafosfat, toplam azot ve amonyak giderimi araştırılmıştır. Çalışmada kullanılan kimyasalların optimum konsantrasyonları sırasıyla, kireç: 0,15 g, polialüminyum klorür: 0,1 g ve amonyum persülfat: 0,0008 g olarak; optimum pH koşulları ise sırasıyla, kireç: 10,9, polialüminyum klorür: 8,1 ve amonyum persülfat: 8,1 olarak bulunmuştur. Çalışmada kimyasal oksijen ihtiyacı, toplam fosfor, ortafosfat ve amonyak giderim verimleri ≥%86, toplam azot giderim verimi ise %43 olarak bulunmuştur. Alıcı ortama direkt deşarjı halinde, ötrofikasyon gibi ciddi çevresel risk oluşturan şekerleme endüstrisi atıksuyunun ön arıtımı sonucunda kanalizasyona deşarj kriterlerine uygunluğu da araştırılmıştır.

1. INTRODUCTION

The confectionery industry (CI) is one of the most widespread and important industries worldwide. Cocoa, sugar, various fats, and emulsifiers are used in the production process, including confectionery and chewing gum products [1–3]. With the development of industrial organisations, pollution sources that cause water pollution have also developed. These industries produce large amounts of wastewater with high levels of pollutants. Confectionery wastewater is considered non-toxic because it does not contain compounds such as heavy metals. The composition and quantity of small-scale CI wastewater varies daily and seasonally. Therefore, it is emphasised that it is not economically and operationally suitable as a raw material. These wastewaters are suitable substrates for microorganisms as they have biodegradable organic matter containing compounds such as high Chemical oxygen demand (COD), nitrogen, and glucose. However, it is risky to discharge wastewater from these industries without pre-treatment [4–6]. Untreated wastewater has many negative effects on human health, flora, fauna, and agricultural lands. CI wastewaters cause eutrophication by increasing phosphorus and nitrogen concentrations in receiving environments and pollute waters that can be used as drinking water [7,8]. Dissolved oxygen concentration in the aquatic environment decreases with the biochemical breakdown of organic pollutants reaching the receiving environment. Thus, anaerobic microorganisms become dominant in the aquatic environment, reducing the self-cleaning capacity of the receiving environment [9]. In order to control water pollution, it is important to treat wastewater with various pre-treatment processes before discharge. Treatment methods such as physicochemical and biological (aerobic/anaerobic) treatment processes [10], membrane technologies [11], adsorption [12], coagulation [13], and electrochemical treatment [14] have been used for the treatment of CI wastewater.

Coagulation-flocculation process has been proven to be effective in wastewater treatment by various studies [15]. Coagulation-flocculation process is generally used to remove suspended solids (SS) and organic pollutants from wastewater. The efficiency of the process is related to the selection of the appropriate coagulant. With lime, which can be used in the process, higher biochemical oxygen demand (BOD) and COD removal (BOD: 34-66% and COD: 32-59%) are achieved under aerobic conditions [16]. Flocculation process is the most widely used method in water treatment due to its ease of operation and economic advantages [17]. Inorganic polymer flocculants added in the flocculation process have advantages such as easy availability and applicability. However, aluminium salts, which are among the coagulants frequently used in the coagulation stage, are among the most widely used inorganic coagulants due to their good treatment performance, easy availability and use, and low cost [18]. It is also reported that polyaluminium chloride (PAC), which is the hydrolysed form of aluminium salt used in present study, is more effective [19].

Adsorption process using activated carbon was applied for the treatment of CI wastewater. After adsorption, BOD and COD were removed with 90% efficiency [20–22]. It has been emphasised that activated carbon is disadvantageous due to its high cost, regeneration, and disposal problems [9]. Electrocoagulation process using zinc electrodes was used for the treatment of sugar industry wastewater. Significant removal of COD (81%), BOD (89%), and total solids content (90%) was achieved [23]. Electrocoagulation process using aluminium anodes and sand filtration process were used for pretreatment of chocolate industry wastewater. Turbidity removal of 96%, colour removal of 98%, and COD removal of 39% were achieved [1]. Turbidity, COD, and BOD removal from sugar industry wastewater using active sugarcane bagasse by coagulation process was investigated. Turbidity, COD, and BOD removal were found to be 68.9%, 64.8%, and 69.9%, respectively [24]. CI wastewater was treated biologically using a pilot-scale filter. COD, nitrate (NO_3-N), and orthophosphates (PO_4-P) removals were found to be 98.9%, 99.5%, and 99.7%, respectively [6].

In this study, real CI wastewater was characterised and investigated by using Lime ($Ca(OH)_2$), PAC, and Ammonim Persulfate $(NH_4)_2S_2O_8$ coagulants with coagulation-flocculation process, COD, total phosphate (TP), PO_4-P, total nitrogen (TN), ammonium nitrogen (NH_4-N), and NO_3-N removal. In addition, SS concentrations in the influent wastewater were also monitored. Studies investigating the removal of COD, TP, PO_4-P, TN, NH_4-N, and NO_3-N parameters, which are important pollution sources in CI wastewater, are limited. The removal of CI wastewater containing high organic pollutants, which discharge without pre-treatment would pose a serious environmental risk, was investigated by coagulation and flocculation process, and the suitability of effluent pollutant concentrations in terms of sewage discharge criteria was investigated.

2. MATERIAL AND METHOD

2.1. Sample collection

The wastewater used in the experiments was obtained from the CI in Gebze Plastics Organised Industrial Zone (Turkiye). The flow rate of the wastewater treatment plant where chemical treatment is performed is 15 m^3. The plant has a rapid mixing tank where the coagulation process takes place, and then a flocculation tank where the wastewater is conveyed for flocculation process. CI wastewater was taken from the inlet of the plant. Samples were taken twice a week, and a total of 4 litres composite sample was formed. The raw wastewater taken from the plant inlet was stored in the refrigerator (4 °C) before the experiments.

2.2 Coagulation-flocculation jar test procedure

Coagulation-flocculation study was carried out under laboratory conditions (24 °C) using a jar test device. During the research, 200 mL of wastewater volume was used in each trial set. Each coagulation experiment was started by adding coagulant to the reactor containing wastewater, and then the appropriate initial pH was adjusted using HCl and NaOH. In the coagulation step, the reaction time was limited to 3 minutes (mins), and the process was continued at a stirring speed of 120 rpm [15]. The flocculation process was carried out at a mixing speed of 40 rpm with a contact time of 15 mins [15]. The precipitation step was determined as 45 mins, and at the end of the process, samples were taken from the upper phase and analyzed for SS, COD, TP, PO_4-P, TN, NH_4-N. Optimum pH values and coagulant doses to be used in CI wastewater were determined by Jar test. After the jar test, samples were taken from the upper phase, and outlet COD concentrations were measured. The removal efficiencies of pollutant parameters in the influent wastewater were determined by using the values in the third jar test, which had the lowest effluent COD concentration and the lowest chemical consumption. For coagulation-flocculation in CI wastewater, 5% $Ca(OH)_2$, 17% PAC, and 0.1% $(NH_4)_2S_2O_8$ were used. The properties of the chemicals used are presented in Tables 1.

Table 1. Properties of the chemicals used

	Coagulants and Chemicals				
	HCl	**NaOH**	**PAC**	**$(NH_4)_2S_2O_8$**	**$Ca(OH)_2$**
Concentration	37%	45%	%17	0.1%	5%
Density (g/cm^3)	1.180	1.32	1.4	0.75	2.21
Mole (g/mole)	36.5	40	97.46	228	74.09

2.3. Characterization

The conductivity and pH were measured using a pH meter and a conductivity device. COD (K-7365), TP and PO_4-P (TR-TP), TN (TR-TN), NH_4-N (LCK303), NO_3-N (LCK339), and nitrite (NO_2-N) (LCK341) were carried out with Hach test kits. Suspended Solids (SS) were determined according to Standard Methods [25].

3. RESULTS AND DISCUSSION

The concentrations and pH values of the chemicals used in the three sets in the study were given in Table 2.

Table 2. Coagulants used in the study, coagulant doses and pH values (in 200 mL volume)

Coagulants	Doses (g)			pH values		
	Set 1	Set 2	Set 3	Set 1	Set 2	Set 3
$Ca(OH)_2$	0.17	0.18	0.15	11.3	11.1	10.9
PAC	0.18	0.2	0.1	7.9	8.2	8.1
$(NH_4)_2S_2O_8$	0.0011	0.0009	0.0008	7.9	8.2	8.1

The concentration and consumption amount of chemicals used in Set 3 were also presented in Table 3. PAC, one of the aluminum salts, was used in the study at a pH value of 8.1. In the coagulation-flocculation process, neutral pH values are given as the first species that provide the sweeping mechanism for aluminum hydroxide precipitates. When the surface charge of colloids is zero at neutral pH values, the sweeping mechanism or charge neutralization occurs.

The content and pH value of the wastewater directly were affected the coagulation-flocculation process [26]. It has been stated that when aluminum salts are used in an oily wastewater, high pH values increase the negative charge of hydroxide (OH^-) and negatively affect the performance of the process [27]. At the optimum polyaluminum chloride silicate dosage and pH 7, COD and turbidity removals were found to be 94.4% and 94.1%, respectively [28].

Table 3. Consumption of coagulants according to set 3

Coagulants	Concentration	Density(d) (g cm^{-3})	Consumption (g L^{-1})
PAC	%17	1.4	0.5
$(NH_4)_2S_2O_8$	%0.1	0.75	0.004
$Ca(OH)_2$	%5	2.21	0.75

An effective volume of 200 mL was used in the present study. The concentrations of the chemicals used in this volume were $Ca(OH)_2$: 0.15 g, PAC: 0.1 g and $(NH_4)_2S_2O_8$: 0.0008, respectively. CI wastewaters have different characterizations according to the product and process. The wastewater characterization studies are summarised in Table 4.

Table 4. The characterization of the real CI wastewater and comparison with literatüre

Wastewater Type	pH	COD (mg L^{-1})	SS (mg L^{-1})	TP (mg L^{-1})	PO_4 (mg L^{-1})	TN (mg L^{-1})	NH_4-N (mg L^{-1})	NO_3-N (mg L^{-1})	NO_2-N (mg L^{-1})	Literature
Sugar Industry	5.5	3682	790	-	5.9	-	-	-	-	[29]
Sugar Industry	6.7–8.4	1100.3-2148.9	220.3-790.7	-	1.2-9.6	11.1-40.6	0.0-4.2	0.4-0.9	-	[30]
Sugar Cane Industry	7.1	31735	-	-	11.3	61.8	8.1	7.6	-	[15]
Pudding Dessert Wastewater	6.60-7.70	21000-68000	9000-40000	-	50.0-70.0	-	-	-	-	[6]
Chocolate Manufacturing Industry	7.4	3608	-	-	-	-	-	-	-	[1]
Sweet-snacks and Ice-cream Industries	5.6-7.1	8960-11900	-	-	-	78-157	89-120	-	-	[8]
Confectionery Industry	3.83	20025	900	4.5	-	-	-	78	-	[31]
Confectionery Industry	7.5	448	231	10.5	-	-	-	-	-	[32]
Confectionary Wastewater	6.6	8330	1835	11.5	3.70	37	0.25	3.71	0.30	Present Study

The only study in which all of the parameters related to organic pollutants given in the table were monitored is the present study. The pH value of the wastewater in the present study is within the range specified in the literature. While COD concentration varies between 3608-68000 mg L^{-1} in the studies, COD concentration in the present study was found to be 8330 mg L^{-1}, and it is seen that it is compatible with the studies in the literature. SS concentration was found as 1835 mg L^{-1} and it is in parallel with the literature. Studies monitoring TP concentration in CI wastewater are limited.

In the present study, TP concentration was higher than the value given in the literature and was found as 11.5 mg L^{-1}. PO_4-P was measured at different concentration ranges in the studies and was determined as 3.7 mg L^{-1} in the present study. TN, NH_4-N, and NO_3-N concentrations determined in the study were 37 mg L^{-1}, 0.25 mg L^{-1}, and 3.71 mg L^{-1}, respectively. NO_2-N concentration was found as 0.30 mg L^{-1} in the study.

$$\boldsymbol{Removal\,rate}\ (\%) = \frac{\boldsymbol{C0 - C}}{\boldsymbol{C0}} * \boldsymbol{100} \qquad (1)$$

Following the coagulation-flocculation process, pollutant removal was monitored in the samples taken from the upper phase, and the removal efficiencies are given in Figure 1. Removal efficiencies were calculated according to the following Equation (1).

C0: the concentration of pollutants in the influent wastewater sample

C: the concentration of pollutants treated wastewater sample

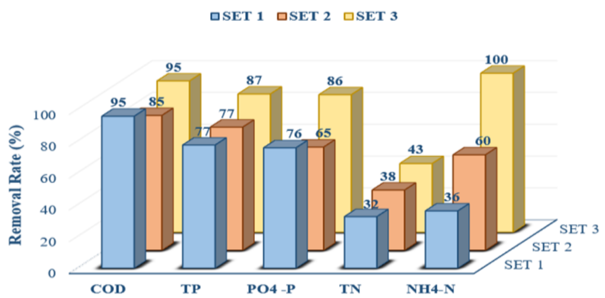

Figure 1. Removal efficiencies of pollutants in all set

When the removal efficiency of pollutants in three sets after the chemicals applied in the Jar test was analysed, the set with the best removal efficiency was Set 3. When the COD removal in Set 3 was analysed, a removal efficiency of 95% was determined. In the literature, it is seen that COD removal efficiencies of CI wastewater by coagulation-flocculation process vary between 56.7% and As seen in Table 5, all of the pollutants detected in this study and included in the Water Pollution Control Regulation supplied the discharge criteria.

96.1% [33]. In the coagulation process using alum, COD removal efficiency was 96.1%, while when polyaluminum chloride was used, the removal efficiency was 95% [34]. In another study investigating the removal of sugar industry wastewater by coagulation, COD removal was found to be 69-80% when $FeCl_3$ was used as coagulant [35], while COD removal was found to be 82% when Alum was used [13].

In a coagulation study using activated sugarcane bagasse coagulant, COD removal was found to be 64.8% [24]. The COD removals in the present study are in parallel with the studies in the literature. In Set 3, TP was removed with high efficiency, and the removal was found to be 87%. There is no study on TP removal using coagulation-flocculation process in CI wastewater in the literature. In the present study, PO_4-P removal was found to be 86%. Coagulation-flocculation process integrated with nanofiltration and reverse osmosis membrane was used for the treatment of sugarcane industry wastewater. In the coagulation-flocculation process with unshelled and shelled moringa seeds, PO_4-P removal was 44% and 23%, respectively.

When moringa seeds were used after degreasing after treatment with ethanol, PO_4-P removal efficiencies reached 59%. When PO_4-P removal was compared with the literature, higher removal efficiencies were obtained with the coagulants used in present study [15]. In the study, TN was removed to a limited extent compared to other compounds and the removal efficiency was found to be 43%, while NH_4-N was completely removed. Unshelled and shelled moringa seeds and moringa seeds formed after ethanol treatment were also used for TN and NH_4-N removal from sugarcane industry wastewater. TN removals were found to be 31%, 27%, and 41%, respectively. NH_4-N removals using unshelled and shelled moringa seeds and moringa seeds formed after ethanol treatment were found to be 20%, 9%, and 25%, respectively [15]. CI wastewater is aimed to supplied the Gebze Plastics Organised Industrial Zone sewerage discharge criteria. Pollutant parameters and sewerage discharge values of the treated wastewater obtained in Set 3 are given in Table 5.

Table 5. Effluent concentrations and discharge criteria

Parameter	Effluent concentrations	Discharge criteria*
COD (mg L^{-1})	416.5	4000
pH	8	6.5-10
SS (mg L^{-1})	<50	500
NH_4-N (mg L^{-1})	<0.005	-
NO_3-N (mg L^{-1})	<0.005	-
NO_2-N (mg L^{-1})	<0.005	-
TN (mg L^{-1})	21.1	80
TP (mg L^{-1})	1.5	2
PO_4-P (mg L^{-1})	0.52	-

* Discharge criteria: GOIZ discharge criteria *(Water Pollution Control Regulation, Table 25)*

4. CONCLUSION

CI wastewater differs according to the type of product produced and the production process and poses a great environmental risk. Wastewater discharged without pre-treatment threatens aquatic life by polluting surface water, groundwater and soil. In the study, detailed characterization of the wastewater was carried out. The treatability of the wastewater was investigated by coagulation-flocculation process using $Ca(OH)_2$, PAC and $(NH_4)_2S_2O_8$ coagulants and the most effective removal rate was determined in Set 3. There is no study investigating the removal of COD, TP, PO_4-P, TN and NH_4-N using real CI wastewater. The COD removal rate determined in the study coincides with the studies in the literature and it is seen that a higher removal rate is achieved than many studies. No study on TP removal was found in the literature and TP removal in Set 3 was determined with high efficiency. PO_4-P, TN and NH_4-N removal efficiencies were found above the values stated in the literature. Due to the environmental risk of CI wastewater, it is important to increase the studies on pretreatment of wastewater, especially from small-scale establishments. As a result of the study, it was observed that CI wastewater, which poses a serious environmental risk, can be treated by coagulation - flocculation process and using this process discharge criteria are supplied. However, since the disposal of the chemical sludge to be formed after coagulation-flocculation is troubled, the treatability with biodegradable coagulants should be investigated.

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