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Research Article

Treatment of Vegetable Oil Industry Wastewaters with Coagulation-flocculation Methods

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ABSTRACT

The vegetable oil industry uses high amounts of water as a process and contains high concentrations of various pollutants and compounds resistant to biodegradation. In this study, the coagulation/flocculation process was applied to treat these wastewaters that have a negative effect on the receiving environment. In coagulation-flocculation experiments performed with $Al_2(SO_4)_3.18H_2O$, the highest COD (92%) and Oil-Grease (89%) removal efficiencies were obtained at a coagulant dose of 800 mg Al^{+3}/L. In experiments conducted with $FeCl_3.6H_2O$ coagulant, the highest COD (88%) and Oil-Grease (89%) removal efficiencies were obtained at a dose of 600 mg Fe^{+3}/L coagulant. This study demonstrated the effective use of the coagulation/flocculation process in the treatment of vegetable oil industry wastewater containing high COD and Oil-Grease.

Introduction

Together with recent increases in industrialization activities and changing human needs, dietary habits of people are also changing. There is a tendency in markets from animal-originated oils to vegetable oils, which were thought to be healthier, and such a tendency increased the vegetable oil production of the industry. Worldwide vegetable oil production has increased over the years and is expected to increase further in the upcoming years. About 60% of vegetable oil production is consumed as foodstuff and 32.0% is consumed in biofuel production [1].

Fats constitute a primary component of cell, tissue, and organs; thus, they are considered to be essential nutrients of human body for maintenance of life and regular performance of body functions. Vegetable oils provide energy, fatty acids, and fat-soluble vitamins for human nutrition. Cooking oil is generally produced from oilseed plants such as coconut, palm, peanut, rapeseed, soybean, olive, cottonseed, and sunflower [2]. During the production of cooking oils, significant amounts of different wastes are generated, including oilseeds, wastewater, organic solid wastes (i.e., seeds and husks) and inorganic residues. Oily parts of seed shells are generally used in feed industry and the remaining parts are used as fuel in production facilities [3].

A significant amount of wastewater is generated in production of vegetable oils, especially during the processing of oil. Such wastewaters may result in serious environmental problems when they were not treated properly [4]. If the oily wastewater is not treated properly, it can cause health problems for living organisms. Presence of oil in water will reduce the oxygen concentration of water and such a case then increases mortality rates of fish, mammals and birds and may harm aquatic organisms [5], [6]. Oil can accumulate in sewer lines and pipes and emit an unpleasant odor, causing conditions to move towards anaerobic conditions. Accordingly, resultant corrosion and clogging of pipes prevent proper operation of wastewater treatment plants [7]. In terms of water consumption of vegetable oil industry, 0.5-0.75 tons of wastewater is generated per ton of fresh fruit during palm oil production [8]. In olive oil production, 1.2 – 1.8 m^3 wastewater is generated per ton of olive [2]. Also in small-scale soybean oil industry, about 80 - 100 tons of wastewater are generated daily [9].

Vegetable oil industry wastewaters have high chemical oxygen demand (COD), biological oxygen demand (BOD),

*This study was produced from the first author's doctoral thesis.

total dissolved solids (TDS), total suspended solids (TSS), oil, grease, phosphate, and sulfate contents [10]. Therefore, when discharged directly into receiving environment, they cause rapid oxygen depletion in the receiving environment and irreversible damage to aquatic life [2]. These wastewaters should be so treated as to meet the discharge criteria. Wastewaters generated during production mostly occur at various stages during oil production/processing, including removal of sticky substances, acid removal, deodorization, and neutralization [11], [12]. Quite a large amount of wastewater is also produced in vegetable oil refining, acidification of soap stock, filter backwashing and equipment washing processes [13]. Besides, volume of wastewater may vary based on processing technology and types and quality of raw materials used during processing [14].

The (BOD)/(COD) ratio is an important parameter used in selection of proper wastewater treatment method and assessment of biological processes as an efficient treatment alternative. While a BOD/COD ratio greater than 0.6 is recommended for aerobic or anaerobic processes, such a value is around 0.2 for edible vegetable oil industry wastewaters [15], [16]. Since sunflower oil wastewaters contain high quantities of lipids, inhibition of microorganisms is an important problem in biological treatment of these wastewaters [17].

Generally, physico-chemical, biological, and electrochemical methods are used in treatment of vegetable oil industry wastewaters [18], [19], [20], [21], [22]. The difficulty in treatment of vegetable oil industry wastewaters comes from the distribution of oil droplets and surface-active substances. These are defined as dissolved oils and free oil droplets and emulsions [23]. The basic color of emulsions is white. If the emulsion is concentrated, the Tyndall effect scatters light and causes the color to shift to blue. If it is intense, the color shifts to yellow.

The first step in treatment of vegetable oil industry wastewaters is the separation of free oils as dispersed, emulsified, and soluble ones. In this step, generally physical treatment methods such as precipitation, flotation and centrifugation are used. Free oils of wastewater can be removed with simple grease traps. However, the main problem in wastewater is the removal of emulsion oils. Following the breakout of emulsion, physical processes such as flotation or coagulation-flocculation can be efficient [24]. The emulsion should be broken to remove the emulsified oil. This breaking process can be realized through physical, chemical, electrical, and biological methods [25]. To overcome the problems frequently encountered in treatment of sunflower oil wastewaters, it is necessary to apply new treatment approaches such as chemical and electrochemical treatment methods. Among these methods, coagulation-flocculation widely used in treatment of sunflower oil wastewaters [26]

The coagulation-flocculation is a two-stage conventional wastewater treatment method in which cationic inorganic metal salts are used as coagulants and long-chain nonionic or anionic polymers are used as flocculants [27]. Addition of oppositely charged electrolytes to the system can lead to a reduction in net surface charge of the oil droplets and consequently to a reduction in repulsion potential between the oil droplets. Aluminum sulfate, FeCl3, is among the most used [28], [29]. Since aluminum or iron salts used in the process have a good coagulation effect on negatively charged oil droplets, coagulation/flocculation can effectively remove majority of the suspended oil and emulsified oil [30], [31]. Deposition of oil droplets is realized through attraction by Van der Waals forces. Some other mechanisms such as compaction of the layers, inter-particle bridging and scavenging contribute to the flocculation process. The oil droplets in the flocs are then removed by sedimentation or flotation. Several coagulants are widely used in oily wastewater treatment. These coagulants are either inorganic (e.g., aluminum sulfate, poly-aluminum chloride, poly-zinc silicate) or synthetic organic polymers (e.g., polyacrylamide derivatives). Choice of coagulants depends on chemical properties of oily wastewater. Use of abrasive coagulants can result in large amounts of hazardous sludge. Alternatively, some natural coagulants have also been used in oily wastewater treatment due to their safe and environment-friendly nature [32], [33].

In this study, optimum conditions were determined for the coagulation-flocculation process, which is used in the treatment of vegetable oil industry wastewater that is difficult to biodegrade.

2. Material and method

2.1. Material

Wastewater samples were taken from an oil factory dealing with soybean oil, cottonseed oil, sunflower oil, corn oil and anhydrous oil production in Kahramanmaraş province of Turkey. Currently, there is a treatment plant operating actively in the facility. The treatment plant performs chemical treatment as to meet the discharge standards to the canal. Samples were taken from the entrance of treatment plant. Samples were transported in 30-liter high density polyethylene (HDPE) buckets. Wastewater samples were stored under laboratory conditions (at +4 ^{0}C) without any pre-treatments. Minimum and maximum values of the wastewater samples are provided in **Table 1.**

Table 1: Analysis values for vegetable oil industry wastewater used in present study.

Parameters	Wastewater Values	Discharge standards 2-h Composite Sample [34]
COD (mg/L)	11503	160
Oil-Grease (mg/L)	2810	60
pH	11.7	6-9
TSS (mg/L)	613	-
EC (µS/cm)	2210	-
Temperature (^{0}C)	80	-

2.2. Chemicals and reagents

For COD analysis, potassium dichromate (Merck 99.5%) was used as disintegration solution and silver sulfate (Merck) sulfuric acid solution (Merck 95-97%) and mercury chloride (Merck) were used as acid solution. Carbon tetrachloride (Sigma Aldrich 99%-FTIR) was used for oil grease analysis. Isopropyl alcohol (Merck) was used for FTIR spectrophotometry diamond cleaning.

2.3. Analytical methods

The pH, electrical conductivity, COD, oil-grease and TSS measurements were performed before and after the experiments with the use of standard methods. COD measurements were made in accordance with Standard Methods (5220-D). In COD analyses, Hach-Lange DR-3900 spectrophotometer was used. Prepared samples were read spectrophotometrically at 600 nm [35].

The pH and EC measurements were made with the use of WTW brand device. Oil analyzes of vegetable oil industry wastewater were made with the use of Agilent Cary 630 FTIR (Serial lot no:MY16160027) device. FTIR spectra were in the range (4000-400 cm^{-1}) and all spectra were adjusted to have a resolution of 4 with 128 scans [36], [37]. These analyses were conducted in accordance with ASTM D7678-11 and ARPA-APPA 75/2011 methods modified with CCL_4 [38], [39]. Measurements were carried out in a windless and humidity-free environment by making a baseline with air. First, crude oil was taken from the factory where vegetable oil was produced, and spectrum scans of this oil were made and the most logical and calibrable one was obtained. peaks and ranges were determined. The 1600-1800 cm-1 region is a distinctive region for vegetable oils. It was stated that the absorption peak in this region explains the C=O ester carbonyl functional group of triglycerides[40]. Stock solutions were prepared with CCL4 in the range of 0-2000 ppm and absorbance values were recorded. Then, a calibration chart was prepared. Since the R2 value of the chart was seen as 0.99, this method was used in oil analysis.

2.4. Method

Coagulation - flocculation

For coagulation – flocculation experiments, 6 jar test setups with mixer were used (**Figure 1**). In coagulation-flocculation treatments, 500 mL wastewater sample was placed into 1000 mL beakers. Iron III Chloride ($FeCl_3.6H_2O$) and Aluminum Sulphate ($Al_2(SO_4)_3.16H_2O$) (Alum) were supplemented as coagulants and optimum pH and coagulant doses were determined based on COD and Oil-Grease removal efficiencies. WTW-brand pH-meter was used to adjust pH while finding optimum pH. Experiments were carried out in FCSL-brand Jar-test device by applying fast mixing at 120 rpm for 2 minutes and slow mixing at 30 rpm for 20 minutes[41]. After settling for one hour, the supernatant was collected for physicochemical analysis. A fixed amount of coagulant (400 mg Fe+3/L and 400 mg Al+3/L) was chosen for both coagulant substances to investigate the optimum initial pH value.

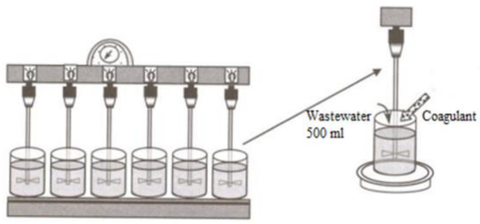

Figure 1. Schematic view of jar-test setup [42]

3. Results and discussion

3.1. Results of coagulation – flocculation experiments

3.1.1. Effect of initial pH on chemical coagulation

In chemical coagulation studies, effects of coagulant dosage and pH on wastewater treatment were investigated [43]. For chemical coagulation, alum ($Al_2(SO_4)_3.18H_2O$ and iron salts $FeCl_3.6H_2O$ were used as coagulant material. Both Al^{+3} and Fe^{+3} based coagulants decompose in solution and also increase polymeric chain reactions to adsorb oil droplets on their surfaces by forming flocs [44] [45]. Initial pH range was selected as 3–10 and COD and Oil-Grease removal efficiencies were investigated at initial pH values. Effects of initial pH on COD and Oil-Grease removal in chemical coagulation are presented in Figure 2.

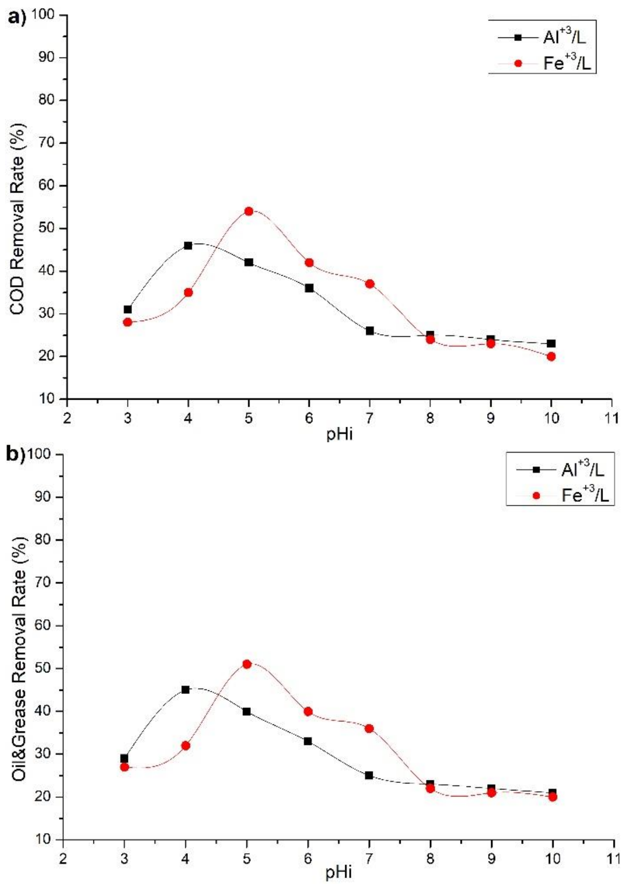

Figure 2: Effect of initial pH on COD (a) and Oil-Grease (b) removal in chemical coagulation. (COD:11503 mg/l; O&G: 2810 mg/l)

The greatest COD and Oil-Grease removal efficiency of $Al_2(SO_4)_3.18H_2O$ coagulant was achieved at pH 4 and the highest COD and Oil-Grease removal efficiency of $FeCl_3.6H_2O$ coagulant was achieved at pH 5. At constant 400 mg Fe^{+3}/L and 400 mg Al^{+3}/L coagulant quantities, the highest COD removal was 46% and the highest Oil-Grease removal was 45% for $Al_2(SO_4)_3.18H_2O$ coagulant; the values were respectively calculated as 54 and 51% for $FeCl_3.6H_2O$ coagulant. It was reported that micelle-based emulsion was broken at pH range of 4-5 and the coagulant material absorbed the pollutant more easily [46].

3.1.2. Effect of coagulant dosage on chemical coagulation

Amount of coagulant flocculant is an important parameter in coagulation-flocculation process. Coagulant dosage can technically be defined as the amount of substance added at maximum removal efficiency [46]. In chemical coagulation process, effects of coagulant quantity on COD and Oil-Grease removal efficiencies were investigated at dosage range of 100-900 mg/L, experiments were carried out at fixed values at pH 4 for Al2(SO4)3.18H2O and at pH 5 for FeCl3.6H2O and the results are present in Figure 3.

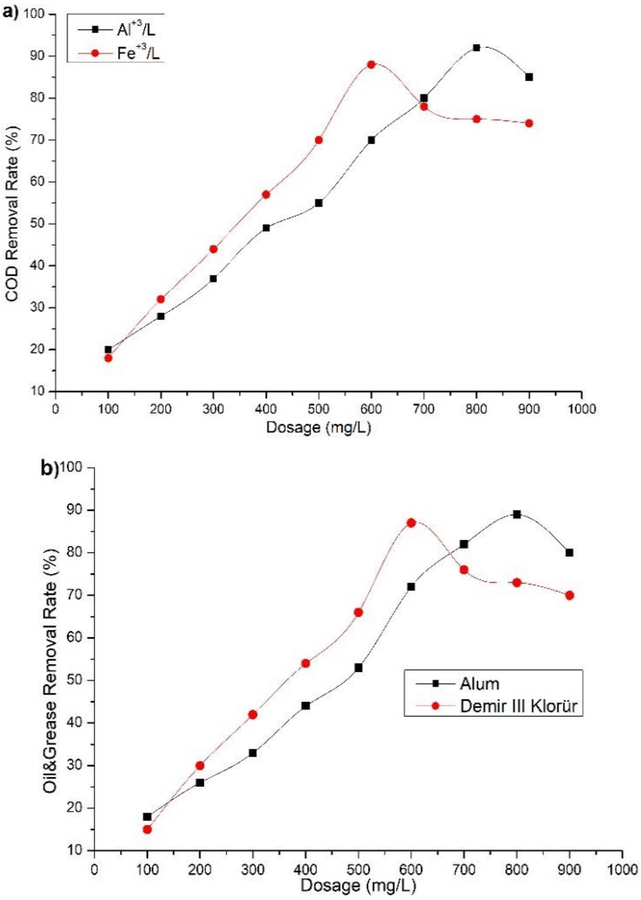

Figure 3: Effect of coagulant dosage on COD (a) and Oil-Grease (b) removal in chemical coagulation (COD:11503 mg/l; O&G: 2810 mg/l)

For $Al_2(SO_4)_3.18H_2O$ coagulant, the highest COD removal (92%) and Oil-Grease removal (89%) was achieved at 800 mg Al^{+3}/L dosage. For $FeCl_3.6H_2O$ coagulant, the highest COD removal (88%) and Oil-Grease removal (89%) was achieved at 600 mg Fe^{+3}/L dosage.

Effects of $Al_2(SO_4)_3.18H_2O$ and $FeCl_3.6H_2O$ dosages on pH values were also investigated (**Table2**). Accordingly, decreasing pH values were observed with increasing coagulant quantities supplemented into the process. Treatment efficiencies increased with increasing coagulant dosages. While the pH value was found to be 4-5 for fixed dosages of 400 mg Fe+3/L and 400 mg Al+3/L, the treatment efficiency increased by 20% with increasing dosages at this fixed value pH. Therefore, the study in **Table 2** was carried out. When the initial pH (pHi) was brought to around 7 with the increased dosage, treatment efficiencies of around 90% were obtained. Such an increase in treatment efficiency could be attributed to separation of the broken emulsion because of reduced system pH by added coagulants. This micellar pollution was removed more easily with decreasing pH. Additionally, pH-dependent change in flock types might have been effective in increased efficiencies. The aim here is to reduce the actual pH of the wastewater, which is 11.7, to a constant pH of 4-5, where the efficiency is highest, instead of adding coagulant. Instead of reducing the pH to 7 and adding the coagulant, the pH of the system spontaneously rises to 4-5 without the addition of acid. It was aimed at reducing it to 5. In this way, less acid was used.

Therefore, for pHi value of 7, a pH value including the optimum values of the process was reached. In a smilar study, coagulation-flocculation membrane process was employed in treatment and re-use of vegetable oil factory wastewaters. Aluminum sulfate was used as a coagulant in process and the turbidity, COD and TOC removal efficiencies were calculated as 100%, 98 and 97, respectively [47]. In another study in which coagulation-flocculation experiments were conducted, iron, alum and lime were used in treatment of vegetable oil industry wastewaters and more than 83% removal efficiencies were achieved for Oil-Grease, TSS and COD removal [48]. Again, in a different study conducted with coagulation-flocculation process, COD, turbidity, and Oil-Grease removal efficiencies were calculated as 96, 92 and 99%, respectively, by the addition of 700 mg/L $FeCl_3 \cdot 6H_2O$ [46].

Table 2. Assessment of $Al_2(SO_4)_3.18H_2O$ and $FeCl_3.6H_2O$ Coagulation Experiments Conducted with the Same Wastewater Samples

		Al^{+3}/L	Fe^{+3}/L
Dosage (mg/L)		800	600
pHi	Influent	7	7
	Effluent	4.02	4.93
Removal Efficiency (%)	COD	92.03	88
	Oil-Grease	89.02	89

Haddaji et al. (2022) reported similar results. In a different real-scale study where vegetable oils were removed, the COD removal efficiency was found to be 79.5% under optimum conditions when 1 gL^{-1} $FeCl_3$ and 1 mLL^{-1}

cationic polymer were used at pH =8 [49]. Almojjly et al. (2018) in a similar oil-grease removal study. It has been reported that COD and oil-grease removal efficiency increases by decreasing pH to 7-8 levels[50] In a similar study by Al-Shamrani et al. (2002), oil removals of up to 99.3% at pH 8 and 99.94% at pH 7 were reported for aluminum sulfate and ferric sulfate, respectively.[41] These results show that when pH values are low, zeta potential values are low and therefore the probability of clotting increases.[41]

4. Conclusion

In coagulation-flocculation experiments with $Al_2(SO_4)_3.18H_2O$ coagulant, the highest COD (92%) and Oil-Grease (89%) removal efficiencies were achieved at 800 mg Al^{+3}/L coagulant dose. In coagulation-flocculation experiments with $FeCl_3.6H_2O$ coagulant, the highest COD (88%) and Oil-Grease (89%) removal efficiencies were achieved at 600 mg Fe^{+3}/L coagulant dose. Coagulation levels and solution pH values decreased with increasing coagulant dosages. Therefore, pHi of 7 was thought to be suitable for the process. Since effluent pH values were within the range of 4-5, they should be increased to meet local discharge standards (6-9). In this system, while the standards were met for COD, standards for Oil-Grease were not met. At this stage, this system can be considered as a pre-treatment option for this facility. If oil leaks that may occur in the production system can be reduced, discharge standards can be fully met. In addition to these evaluations, when we evaluate the coagulation-flocculation treatment process for this business from an environmental perspective; Reducing the amount of water discharged in the process and pollution loads, detecting, and recovering oil leaks can make this method a sustainable method that works with higher efficiency and meets discharge standards.

Ethics committee approval and conflict of interest statement

"There is no need to obtain permission from the ethics committee for the article prepared."

"There is no conflict of interest with any person / institution in the article prepared."

Authors' Contributions

Turna T: Study conception and design, visualization, analysis, and interpretation of data, drafting of manuscript.

Yıldız YŞ: conceived the original idea, supervised the project, critical revision.

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