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Wastewater treatment of bottle oil washing water (BOWW) by hybrid coagulation–flocculation and biological process

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ABSTRACT

The efficiency of physico-chemical and biological combined treatment processes of bottle oil washing wastewater (BOWW) was investigated. The coagulation and flocculation process followed by activated sludge treatment was carried out in order to produce a suitable chemical oxygen demand (COD) and oily effluent in conformity with the regulatory standards. Attempts were made in this study to examine the effectiveness of the coagulation and flocculation process using aluminium sulfate and polyelectrolyte (non-ionic preastol) for the treatment of BOWW. The removal of organic matter (COD), oil and color using $Al_2(SO_4)_3{\cdot}18H_2O$ and preastol during coagulation–flocculation process was investigated. Also, the optimum conditions for the coagulation–flocculation process, such as coagulant dosage, polyelectrolyte dosage, and solution pH were investigated using jar-test experiment. Results revealed that in the tested pH range tested, the optimal operating pH was 6.5. Removal percentage of 87, 95, and 97% for COD, oil and color, respectively, and were achieved by the addition of 180 mg/L $Al_2(SO_4)_3.18H_2O$, whereas 87, 99, and 99%, were achieved with the addition of 1 g/L preastol to 180 mg/L $Al_2(SO_4)_3.18H_2O$. The treatment of oily wastewater by an aerobic biological process was conducted using acclimated consortium and lipolytic strain isolated from lipidic effluent. The results of this investigation show that the system is capable of achieving high levels of purity; 93% COD removal, 99% oil removal, and 92% color removal. A comparison between the results shows that the physico-chemical single system was more improved than the physico-chemical and biological combined 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: Oily wastewater; Combined treatment; Aluminium sulfate; Coagulation–flocculation; Biodegradation

1. Introduction

The washing bottle oil processing industry generates a large volume of wastewater estimated at around 8,125 litres per day. This effluent may have negative impacts on water resource and public epuration station of Sfax [1–3]. Reducing environmental polluting load from oily wastewaters and decreasing

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costs are strongly motivating for wastewater treatments [4].

The bottle oil washing wastewater (BOWW) has different characteristics depending on the different storage time. This effluent is characterized by high chemical oxygen demand (COD) and oil concentration. The BOWW gives rise to a major environmental problem related mainly to the difficulty in treating wastewater using biological treatment methods.

Actually, the biological treatment of oily wastewater is not efficient enough to solve completely the oily wastewater problems. This difficulty is related to the long chain of fatty acids antimicrobial activity.

Several techniques for the removal of COD, oil and color from oily wastewater are used. In fact, coagulation–flocculation is a commonly used process in water and oily 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 the performance optimization of coagulant–flocculant, determination of experimental conditions, assessment of pH, and investigation of flocculant addition [5,6].

Aluminum salts have been traditionally used as coagulants for wastewater treatments and color removal. Unfortunately, Al^{3+} chemistry is extremely complex [7]. These compounds undergo a series of hydrolysis reactions; the water molecules that participate in the ionic hydratation sphere are progressively replaced by hydroxyl groups [8]. Significant amounts of non-hydrolyzed Al^{3+} can only be found under low pH. The small size of Al^{3+}, and consequently its hydrophilic character, maintains six water molecules octahedrally oriented around Al^{3+} ion [9].

Aluminium hydroxide precipitates at alkaline pH after hydrolysis. Further increase of pH solubilizes the precipitate by $Al(OH^-)_4$ formation. Actually, a series of multinuclear species like $Al_2(OH)_2^{4+}$ and $Al_{13}(OH)_{34}^{5+}$ are formed as hydrolysis products. Minimum solubility of Al^{3+} is reached at pH 6.5 and hydrolysis precipitation occurs in a narrow pH. Synthetic polyelectrolytes such as preastol typical polymeric flocculants with molecular weights lower than 10^6 are not usually effective. Since many materials carry a negative charge, the positive charge of preastol interacts strongly with negative surfaces to give electric neutrality [10].

In certain circumstances, the particles of dispersion may adhere to one another and form aggregates of successively increasing size, which may separate out under the influence of gravity. The nature of the aggregated material may depend on the conditions of its formation, or it may change with time [11,12].

Disposal of oily wastewater is a serious environmental problem due to its high organic loading. In the last few years several attempts to treat oily wastewater have been described in the technical literature [13]. In particular, biological treatments have been suggested [14,15], but they are not so attractive since the waste shows a low biodegradability.

The aim of the present work was to study first, the coagulation–flocculation process efficiency to remove oily wastewater organic load (COD, BOD_5, TSS, TS, VM, O&G, TKN, and Pt) using aluminium sulfate and preastol and also to investigate optimum coagulant and flocculant dosages. Secondly, the efficiency of simple and combined activated sludge treatment with coagulation–flocculation process was studied to reduce oily wastewater pollution.

2. Materials and methods

2.1. Oily wastewater physico-chemical characterization

The oily wastewater sampling was collected from three storage ponds, located at the oil manufacturing industry, for nine months.

Composite oily wastewater was collected, prepared and analyzed [16] for parameters necessary for waste characterization and system design for two months. This effluent was analysed on the same day for total and soluble chemical oxygen demand (COD_T and CODs), biological oxygen demand (BOD_5), total suspended solid (TSS), total solid (TS), volatile matter (VM), oil and grease (O&G), total Kjeldahl nitrogen (TKN), and total phosphorus (Pt) according to the corresponding standard methods [17]. PH was measured with an electronic pH meter Hach, EC 30 Benchtop Model 50100, Loveland, CO, USA. Analytically pure reagents were used in all the experiments. Table 1 shows the different results of wastewater characterization. All the parameters were analysed in triplicate.

2.2. Physico-chemical treatment by coagulation–floccultion

The coagulation–flocculation of oily wastewater was realized by the aluminium sulfate coagulant and the preastol flocculant. Aluminium sulfate coagulant represents a variety of primary coagulants which can be used in a wastewater treatment. One of the earliest, and still the most extensively used, is aluminium sulfate, also known as alum. When alum is added to

Table 1
Oil–wastewater manufacturing industry characteristics

Parameters	Basin 1	Basin 2	Basin 3
pH	12 ± 1.2	11,58 ± 1	11,41 ± 0.8
COD_T (mg/l)	18,894 ± 9.7	18,631 ± 9.5	21,530 ± 8.2
COD_S (mg/l)	11,127 ± 1	11,527 ± 9.4	11,753 ± 9.8
BOD_5 (mg/l)	920 ± 4.9	600 ± 8.6	600 ± 8.5
COD/BOD_5	12.09 ± 1.8	19.2 ± 1.4	19.58 ± 1.9
TSS (g/l)	1.32 ± 0.4	1.46 ± 0.4	3.04 ± 0.5
TS (g/l)	10.37 ± 1.9	12.28 ± 1	12.58 ± 1
VM (g/l)	6.8 ± 0.3	7.8 ± 0.6	7.7 ± 0.5
O&G (g/l)	6.74 ± 0.1	8.54 ± 0.8	7.69 ± 0.6
TKN (mg/l)	Trace	Trace	Trace
NH_4^+ (mg/l)	Trace	Trace	Trace
Pt (mg/l)	Trace	Trace	Trace

COD_T: Total chemical oxygen demand; CODs: Soluble chemical oxygen demand; BOD_5: Biological oxygen demand; TSS: Total suspended solid; TS: Total solid; VM: Volatile matter; O&G: Oil and grease; TKN: Total Kjeldahl nitrogen; and Pt: Total phosphorus.

water, it reacts with water and results in positively charged ions. The ions can have charges as high as +4, but are typically bivalent (with a charge of +2).

Praestol flocculants from Ashland are a clean solution for optimal liquid–solids separation in a variety of industrial BOWW. Praestol flocculants are synthesized from organic, macromolecular, and water-soluble polyelectrolytes that are based on polyacrylamide. Preastol is commonly known as a flocculant or cohesion agent, cationic, anionic, nonionic, in the molecular weight of between 400–1,800, and is white or slightly yellow powder. Preastol is a linear polymers, it is a viscous transparent solution besides being non-toxic and non-corrosive.

Physico-chemical treatment studies were carried out using a jar-test apparatus [18,19]. Composite samples were rapidly mixed first for 3 min at 150 rpm and then slowly for 25 min at 30 rpm before sedimentation. The pH value of 1 L wastewater sample was adjusted to pH in the range of 5–7.5 by using 1.0 M hydrochloric acid (HCl). In the first part of phase 1, the treatment of composite samples was conducted using aluminium sulfate $Al_2(SO_4)_3$, 18 H_2O with concentrations varying from 10 to 240 mg/L. After rapid mixing for 2 min at 200 rpm, 1 g/l of preastol was added 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 TS by using the standard methods [20], so that the effect of the coagulant dose could be studied. Optimum chemical dose corresponding to the maximum removal of COD, oil and color was determined by conducting "Jar Test". Temperature of wastewater sample during coagulation–flocculation was recorded by a thermometer.

The results were analyzed by one-way Analysis of Variance (ANOVA) and Tukey's test ($p < 0.05$) using MINITAB v 10.51 for Macintosh. All tests were carried out in triplicate.

2.3. Sedimentation test

After flocculation, water at the BOWW enters a very large tank. The water velocity is reduced and there is no turbulence at all. This process allows the floc in the water to settle out of solution. This step is done prior to filtration because the colloid particles will pass through a filter. Sedimentation is an important step, since passing the floc through the filters would clog and damage the filter media.

2.4. Biological treatment

2.4.1. Acclimatization and start-up of the bacterial biomass

In case of biological method, domestic wastewater was collected from Sfax and then its microorganisms were fed with a gradually increasing dosage of oily wastewater. During acclimation, pH, COD, and oil and grease concentrations were regularly measured to check the degree of acclimation. The acclimation phase was considered to be over after about 84 days, when steady biomass growth was observed for a specific quantum of COD and oil and grease removal.

2.4.2. Biodegradation study

After the acclimation of suspended biomass, the series of 250 ml Erlenmeyers shaker flasks was subjected to a varying biomass and oil and grease concentration under varying batch periods of three day. Each flask contained 25 ml lipidic effluent added to peptone, yeast extract, carbon, and nitrogen source and K_2HPO_4 (phosphorus source) at different concentrations. Cultures were grown in 500 ml flasks containing 200 ml of medium at a constant temperature (37˚C) in a shaker (200 rpm). An appropriate nutrient balance was provided by keeping the BOD/N/P ratio at 100/5/1.

All the parameters as in acclimation were measured. The performance of the biological treatment was expressed in terms of removal percentage of COD and color over a certain period.

3. Results and discussions

3.1. Oily wastewater physico-chemical characterization

The oily wastewater characterization studies of 24 h composite samples at different times during the study period indicated that effluent contained high organic load and oil content. However, phosphorus and nitrogen were deficient.

The effluent was characterized in terms of pH (8.2–13), total COD (14,461–21,530 mg/L), soluble COD (COD_s) (7,002–11,753 mg/L), BOD_5 (600–920 mg/L), Oil and grease (5,350–8,540 mg/L), TS (1,320–3,040 mg/L), NTK (3.1–4.03 mg/L), TP (0.13–0.15 mg/L), and phenol (0.0030–0.0037 mg/L). Due to the wide variation in the effluent characteristics, necessary oily wastewater treatment to meet the required effluent standards is also quite complicated. Physico-chemical coagulation–flocculation for colloidal pollutants followed by biological processes for dissolved organic matter is commonly used. Coagulation–flocculation method, an optimal method, was reported by other authors [21] to treat oily wastewater characterized by 280–360 mg/L COD, 1,800–2,500 mg/L TS, 1,500–2,000 mg/L TSS, and 300–700 mg/L oil and grease. The conventional biological processes require a COD to BOD ratio less than 2.6 for an adequate treatment. Nevertheless, oily wastewater from industrial plants has a COD/BOD ratio around 19, which necessitates suitable pre-treatment before the biological step.

Initially, investigations were performed to evaluate the performance of aluminium sulfate and synthetic polyelectrolytes (preastol) as conditioning chemicals for oily wastewater and to analyze the effects of the dose on emulsion restabilization and colloidal charge.

3.2. Physico-chemical treatment by coagulation–floccultion

3.2.1. PH effect

Emulsion oily wastewater containing 200 mg/L aluminium sulfate, 0.75 g/L preastol was used to study the pH effect by adding HCl. The pH of the original emulsion was nine. Considering the relevance of pH on the hydrolysis products of aluminium sulfate and the colloidal charge of the system, different experiments were designed to analyze the pH effects [22]. Fig. 1 shows that optimal pH was brought to 6.5 values with HCl, because the minimum solubility of Al_3^+ occurred at this pH and aluminium hydroxide precipitated. COD, oil and color removal percentages were of 87, 67, and 97%, respectively. According to Tzoupanos and Zouboulis [22], treatment of oily wastewater by coagulation flocculation show an optimal removal percentage at pH around 6.5.

$$Al^{+3} \longrightarrow Al(OH)^{2+} \longrightarrow Al(OH)_2^+ \longrightarrow Al(OH)_3 \longrightarrow Al(OH)_4^-$$

A treatment setup comprising aluminium sulfate and preastol at 200 and 750 mg/L, respectively, was selected to examine the effect of different pH solution on COD, oil and color removals in the wastewater. These effects are shown in Fig. 1. It was observed that the optimum pH value of 6.5 enhanced substantially the contaminants removal. Increasing pH to 6.5 markedly deteriorated the quality of the wastewater. In the pH range of 5–6.5, COD, oil and color levels decreased and pH increased. The pH is a factor that affects the coagulation process, because the addition of metallic cation (in this case Al^{3+}) automatically decreases the wastewater pH, which may cause further reduction in the contaminants removal. The need to employ a higher dose of coagulant may pose health hazard as a result of residual quantities of excess chemical additives. The excess residual coagulant, when discharged into surface water, interferes with fish survival and growth [23].

3.2.2. Coagulant dose effect

The aluminium sulfate concentrations ranging from 10 to 240 mg/L were applied, obtaining maximum removal percentage. Fig. 2 showed the decrease of COD, oil and color, however, sulfate aluminium concentration increased. It is evident from this figure that the removal percentage of COD, oil and grease, and color increased when the chemicals were added in higher concentrations. The increasing aluminium sulfate dose led to a COD and oil decrease due to aluminium hydrolysis; hydrolysis products with positively charged ions like $Al(OH)^{++}$ and $Al_8(OH)_{20}^{4+}$ can be absorbed on negative particles reducing the charge and changing the sign of the charged particles [24].

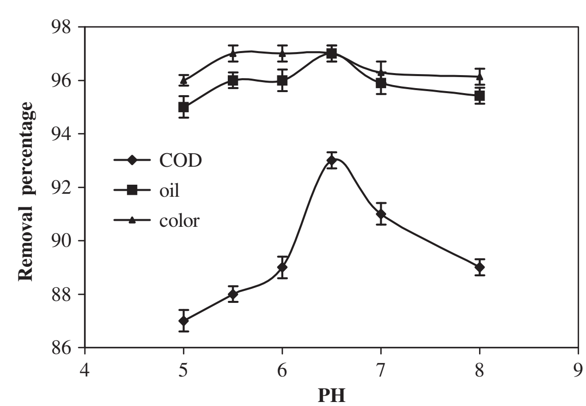

Fig. 1. Removal percentage of COD, oil and grease, and color according to pH of oily wastewater.

Destabilization is mainly due to the formation of these compounds although hydroxide precipitation has an important role as well.

Destabilization by aluminium salts cannot be completely described neither by the double layer model nor by the bridging model [11]. The double layer model predicts that the concentration of coagulant necessary to destabilize a colloidal is virtually independent of the initial colloidal charge and colloidal concentration; reversion of the colloidal charge and system restabilization cannot occur; electrostatic interactions are predominant and chemical interactions and adsorption are absent; Zêta potential for optimum aggregation is near zero [24].

For COD, oil and color, the removal efficiencies increased rapidly up to 87, 95, and 97%, respectively, with the use of 180 mg/L dose of sulfate aluminium, whereas between 180 and 240 mg/L the removal of COD, oil and color increased slowly. At this point it is important to note that the optimum dose of aluminium sulfate that enhanced maximum removal of COD, oil and color was 180 mg/L. A similar result was observed by Mazumder and Mukherjee [21], where 180 mg/L of sulfate aluminium removed 83% of oil and grease in the oily wastewater.

3.3. Flocculant dose effect

After the optimization of pH and aluminium sulfate doses, pH, and aluminium sulfate concentrations were predetermined to 6.5 and 180 mg/L values, respectively.

Preastol concentration varying between 0.25 and 1.5 g/L were applied. Fig. 3 shows the high decrease of COD, oil and color when preastol was used at a concentration of 1 g/L after decantation. Thus, the removals were 93, 99, and 99% for COD, oil and color, respectively. These results are similar to earlier reports showing removed COD (97%) in the presence of NALCO flocculant [25].

Further increase in the preastol dose did not cause further increase in these removal percentages. This may be due to the formation of a greater number of flocs. Besides, increasing the coagulant dose increased $Al_2(SO_4)_3$ supersaturation, which increased the nucleation rate and hence the floc growth rate. As a result, the suspension of a greater number of flocs was enhanced, and subsequently, the removal of larger amount of organic matter was achieved, due to the availability of larger surface area on which the adsorption of the organic matter took place. On the contrary, low coagulant dose 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 the adsorption of organic matter took place. Thus, the optimum coagulant and flocculant dose is defined as the value above, which there is no significant difference in the increase of removal efficiency with a further addition of coagulant and flocculant [26]. Thus, the optimum $Al_2(SO_4)_3$ and preastol doses that enhanced COD removal were 180 and 1 g/L, respectively. A synergistic effect of preastol–polyelectrolyte combination achieved higher removal percentage of COD, oil and color.

3.4. Sedimentation test

Sedimentation tests were performed immediately after flocculation at different doses. After 20 min sedimentation under gravity, the supernatant was carefully pipetted off and the optical density at 600 nm was measured. Fig. 4 shows the optimal flocculation defined as the 1 g/L preastol dose that gave the steepest decrease in the optical density of the supernatant.

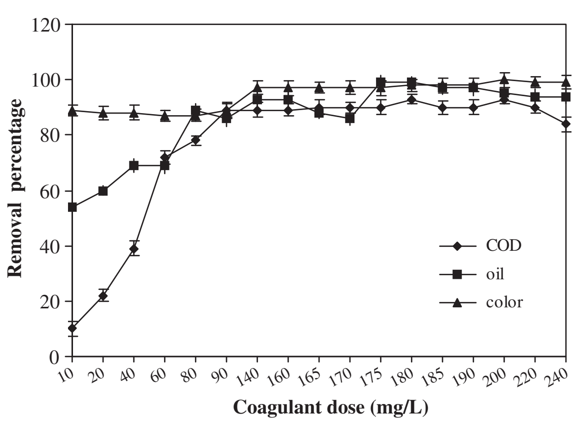

Fig. 2. Removal percentage of COD, oil and grease, and color according to coagulant dose of oily wastewater.

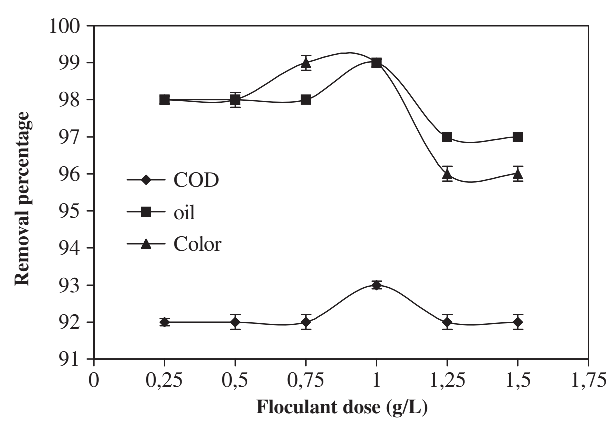

Fig. 3. Removal percentage of COD, oil and grease, and color according to preastol dose of oily wastewater.

3.5. Biodegradation study

To determine the residual soluble COD, a supernatant resulting from chemical treatment with the aluminium sulfate dose of 180 mg/L and the preastol dose of 1 g/L was used to feed the biological treatment of oily wastewater.

According to the evaluation method suggested by Germili et al. [27], 18 capacity biological treatments were twice seeded with the same acclimated mixed culture.

During three days of treatment, the soluble COD concentrations were plotted until slight variation of COD concentration. Figs. 5–7 show the high decrease of COD and color after three days of biological treatment. Thus, the removals were 70 and 99% for COD and color, respectively. In case of a batch period of 24 h, only 50% COD removal was attained and maintained constant to 144 h. A long batch period of 192 h or more is required to achieve a COD removal efficiency of about 70% at the same food to microorganism ratio. These results are similar to earlier reports showing increased COD after biological treatment of lipidic wastewater. Cammarota and Freire [28] showed that COD reached 78% after the biological treatment of wastewater with high oil and grease content.

With biological treatment, a stronger activity of the microorganisms produces metabolites such as organic acids as shown, which contributes to a general acidification. Among these metabolites, since the reactions occur in aerobic conditions, are carbonates and bicarbonates which, like –COOH, can bind Ca^{++} contained in the added lime.

The combined oily wastewater treatment using coagulation–flocculation and biological treatments processes was used in this study. Residual soluble COD values were 93% for lipidic effluents. Oily wastewater has a very high color concentration. The

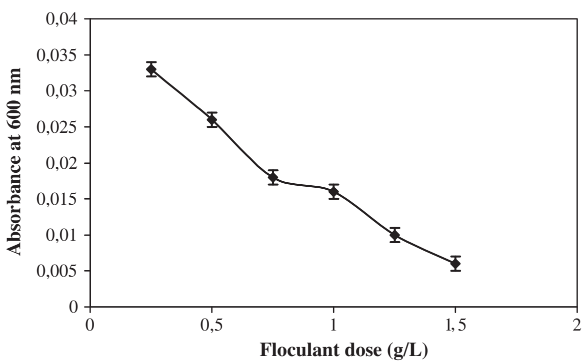

Fig. 4. Optical density measurements in the supernatant after 20 min sedimentation of flocculated suspension according to preastol.

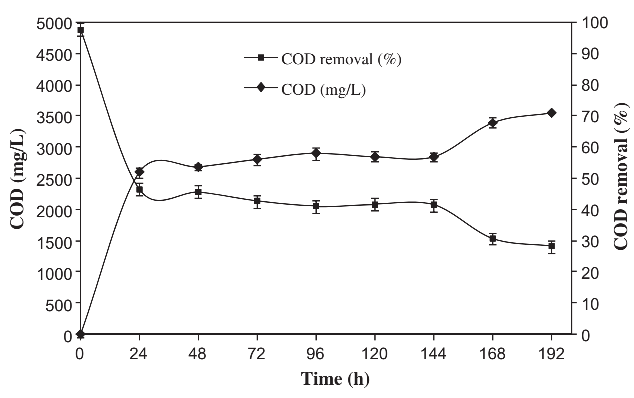

Fig. 5. Evolution of COD and COD removal after biological treatment.

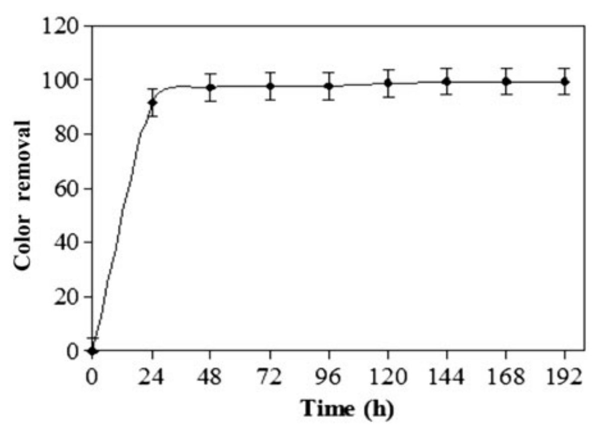

Fig. 6. Removal of color in time after biological treatment.

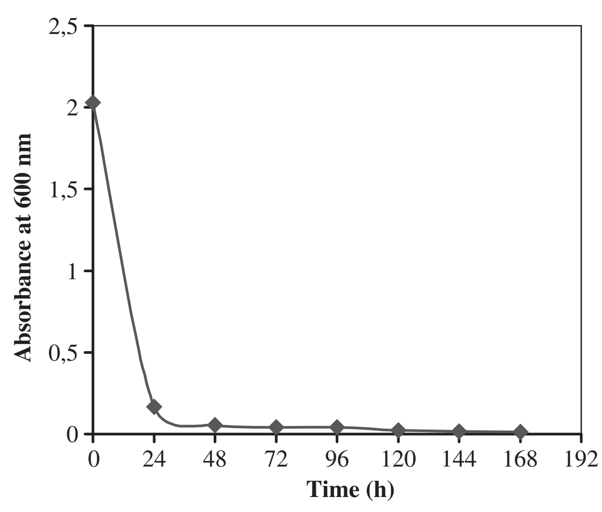

Fig. 7. Evolution of absorbance at 600 nm according to time.

color removal from this effluent was experimentally investigated by using biological treatment. The color removal yield was examined as a result of different times. Data in Figs. 6 and 7 were taken from the lipidic effluent tests with biological treatment.

4. Conclusions

Coagulation–flocculation process may be used as a pre-treatment in order to enhance the biodegradability of the wastewater during the biological treatment. Some of the raw materials that are used for the production of the beverages may enhance the organic load of the wastewater. An essential feature of wastewater coagulation–flocculation is the elimination of suspended solids (SS) and as many of the organic materials as possible. In the present study, the oily wastewater contains both high organic and N loads. A significant amount of COD is in the form of insoluble material, which can be easily removed by chemical methods.

The COD content of oily wastewater is largely correlated with the oil concentration of raw effluent. Oily wastewater can be successfully treated using a combination of physico-chemical and biological methods. Using a pre-treatment process by coagulation and flocculation before biological treatment minimizes operating costs. The physico-chemical treatment processes significantly influence the relative biodegradability of the lipidic effluent. The application of coagulant and flocculant treatment in jar-test laboratory apparatus resulted in higher than 99% reduction of oil content in the oily wastewater. The COD removal was quite 93% and color was 99%. In order to reduce the oil content from an oily wastewater of 3,300 mg/L to less than 30 mg/L, the following optimum parameters have been found: aluminium sulfate: 180 mg/L, preastol: 1 g/L, and pH around 6.5.

The biological treatment of oily wastewater is feasible under batch mode, but it would take at least 192 h to attain 70% COD removal.

Abbreviation

BOWW	—	Bottle oily wastewater
COD	—	Chemical oxygen demand
BOD	—	Biological oxygen demand
TKN	—	Total Kjeldahl nitrogen

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