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*Original Research*

# Limits of Physicochemical Treatment of Wastewater in the Vegetable Oil Refining Industry

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## Abstract

This paper presents results of investigations on physicochemical treatment of vegetable oil refinery wastewater (VORW), namely acid and technological wastewater. The study has shown that VORW has a varying high pollution load (organic matter, sulfates, phosphates and chlorides) and the removal of these pollutants from acid wastewater was more effective than that from technological wastewater; the removal of suspended solids and solvent extractables was relatively higher than that of $BOD_5$ and COD. The use of calcium chloride and alum as coagulants increases the concentration of chlorides in the final treated wastewater. The results also showed that physicochemical treatment processes significantly influence the relative biodegradability of the organic matter in the wastewater.

**Keywords:** physicochemical treatment, wastewater, removal of sulfates and phosphates, biodegradability, pollutants

## Introduction

Many technological processes are carried out in order to obtain refined vegetable oils from seeds. The main processes include pretreatment of oilseeds, manufacturing, refining and modification of oils. During these processes by-products and wastes are formed. The operating conditions and processes carried out influence the amount and characteristics of the by-products and wastes formed. The wastewater varies both in quantity and characteristics from one oil industry to another. The composition of wastewater from the same industry also varies widely from day to day [1, 2]. These fluctuations may also be attributed to different types of oils processed. Considerations to be made for the treatment of oily wastes have been outlined [3, 4, 5]. The authors have discussed types of physical, chemical and biological methods used for the oily wastewater treatment. However, despite the use of these methods, disposal and waste treatment still remain major challenges in the fats and oils industries. One of the contributing factors as to why such wastewaters are difficult to treat is the complexity of their sources. Sources, among others, include heat exchangers, barometric condensors, gas scrubbers, cleaning of greasy floors and equipment, leakages, process water from the refining plant and soapstock splitting effluents. Also, pesticides used in the vegetable oilseed growing have been found in vegetable oil processing industrial wastes. Moreover, vegetable oil refinery wastewater is known to contain chemical compounds like phenol, heavy metals from bleaching earth, catalysts used in the hydrogenation process, oxidizable substances and fats and oils. Because of quantity and characteristic variations and complexity, wastewater treatment to meet the desired effluent standards is complicated, and the choice of methods of wastewater treament depends on many local conditions and, therefore, cannot be standardized.

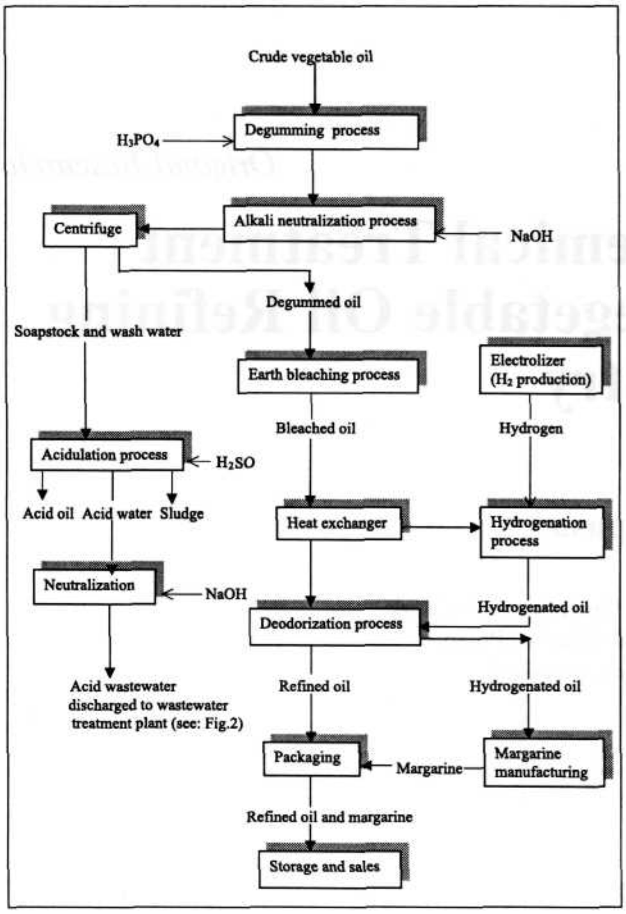


Fig. 1. Simplified schematic diagram of vegetable oil refining processes: sources of vegetable oil refinery wastewater (acid and technological wastewater).

This work presents the results of investigations on the physicochemical treatment of vegetable oil refinery wastewater. The refinery (mentioned herein as the company) uses chemical and physical methods for the refining of oils (rapeseed, sunflower, soybean, palm, and hydrogenated). The processes involved are summarized in Fig. 1. On average, the refinery generates 700 $m^3$ of wastewater daily, which includes acid wastewater (80 - 120 $m^3 \cdot d^{-1}$) and technological wastewater (470 - 600 $m^3 \cdot d^{-1}$). The acid wastewater is that stream coming from the soapstock splitting process, whereas the technological wastewater is that stream originating from all the factory's process installations and equipment. In view of the present emphasis placed on environmental pollution control, new legislations and regulations, and changes in economic factors, the company installed a physicochemical wastewater treatment plant. The process flow diagram with sources of wastewater discharged is shown in Fig. 2. The principal parameters of concern are pH, oil and grease (solvent extractables), suspended solids and organic constituents expressed as 5-day biochemical oxygen demand ($BOD_5$) and chemical oxygen demand (COD). Other wastewater components of concern are sulfates, phosphates and chlorides. Hence, the aim of the wastewater treatment plant, like any other, is to reduce the pollution level prior to discharge to the environment to avoid breaking the ecobalance. And the objective of this study was to evaluate the physicochemical treatment process removal of the mentioned parameters and to assess the biodegradability of the wastewater.

## Materials and Methods

The investigations were carried out on fresh collected untreated and treated wastewater samples. Sampling locations are shown in Fig. 2 as A, B, C and D for technological, acid, intermediate and final treated wastewater, respectively. Technological and acid wastewater are the influents into the wastewater treatment plant. The intermediate wastewater is the stabilization tank effluent, thus a mixture of technological and treated acid wastewater and wastewater from the centrifuge, whereas the final treated wastewater, collected from the effluent of the dissolved air floatation (DAF) process, which is the last step in the wastewater treatment process, is an effluent from the treatment of the intermediate wastewater. All measures were taken to ensure obtaining representative samples. Sampling and preparation were done according to standard procedures [Polish Standard: BN-64/6125-11]. Three samples of each of the wastewater were collected on different days each week for five weeks. Samples were analyzed on the same day of collection. When this was not possible, they were preserved at 4°C following instructions as given by standard procedures. Analytically pure reagents were used in all experiments. The following methods were used to determine the parameters: 5-Day BOD test (without seeding), $BOD_5$; open reflux method, COD; extraction with petroleum ether, solvent extractables; drying at 105°C,

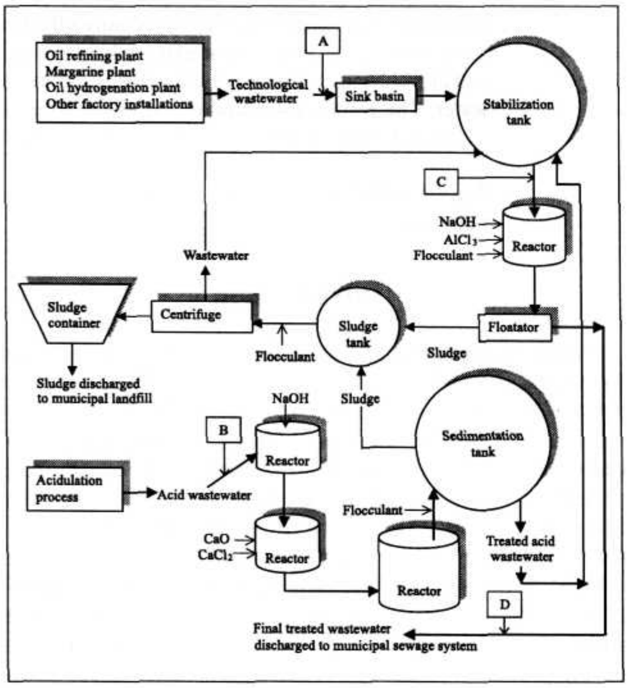


Fig. 2. Simplified schematic diagram of the physicochemical treatment system for the vegetable oil refinery wastewater. Sampling locations: A, technological wastewater; B, acid wastewater; C, intermediate wastewater; D, final treated wastewater.

suspended solids; gravimetric method, sulfates; vanadomolybdophosphoric acid colorimetric method, phosphates; and argentometric method, chlorides. All measurements were carried out according to the *Standard Methods for the Examination of Water and Wastewater* [6].

### Technological Wastewater Treatment Process

Coagulation, flocculation and dissolved air floatation methods are the critical steps during the technological wastewater treatment process. These methods are intended to remove not only suspended matter, but also organic matter through colloid destabilization and organic adsorption into floes. The wastewater originating from the margarine, oil refining and hydrogenation plants and other factory installations flows into a sink basin and then into a stabilization tank, where it is mixed with treated acid wastewater and wastewater from the centrifuge to obtain a uniform liquor and stabilized flow rate. Coagulant (alum, $AlC1_3$) and flocculating agent are added to the wastewater in special reactors (Fig. 2). Aluminum chloride breaks emulsions by lowering the wastewater's pH, which helps in the coalescence of oil droplets and other organic matter and particles. Sodium hydroxide is also added to adjust the pH to 6-7. The coagulant and flocculant (anionic polymer which causes flocculation of solids and oil droplets resulting in creaming) help in separation of suspended and emulsified fatty matter from the wastewater. This results in the formation of suspensions which are pumped to a floatator where they are finally separated from the rest of the wastewater by DAF process; compressed air is passed through the wastewater at 0.5MPa to release bubbles, to which fine solid particles and oil droplets attach. As the air bubbles rise, suspensions and oils are carried to the top surface of the floatator. The sludge which is formed is removed from the surface of the wastewater by a sludge scraper, and the resulting stream of water is called final treated wastewater effluent; it flows down to the municipal sewage system.

### Acid Wastewater Treatment Process

The treatment process is concerned with the removal of sulfate and phosphate ions, fatty materials, and organic suspensions. The acid wastewater coming from the oil refining plant is mixed with 10% calcium oxide (lime milk, CaO), to adjust the pH to about 6-7, and 40% calcium chloride ($CaCl_2$) as a coagulating agent. The reaction between the added calcium compounds and sodium salts and soaps in the wastewater leads to less soluble calcium soaps, sulfate and phosphate calcium salts. Since the interfacial film changes, emulsion may break and the less soluble calcium salts absorb oily matter and other organic suspensions. In this way, both the calcium salts and the adsorbed matter are removed from the acid wastewater stream. The removal efficiency of these calcium salts is limited by their solubility in water: calcium sulfate about 2500 mg/l; calcium phosphate 100 mg/l. The resulting sludge is pumped to a sludge tank, while the wastewater is pumped to a sedimentation tank. A flocculating agent is added to the wastewater before it flows into the sedimentation tank where the settling and separation of sludge from the wastewater take place. The resulting sludge is pumped to a sludge tank and the resulting waste (called treated acid wastewater) is redirected to the stabilization tank where it is mixed with technological wastewater and wastewater from the centrifuge (Fig. 2).

## Results and Discussions

Vegetable oil refinery wastewater is a complex mixture composed of widely-distributed particle sizes influencing each unit operation of the treatment process. The elimination of the pollution load by physicochemical treatment is affected by many factors such as the characteristics of the organic matter, the nature and concentration of other components, and the design and operation of the treatment facility. As a result, the removal of pollution load may widely vary. The characteristics of wastewater samples are summarized in Table 1. Results presented are for samples taken over a 5 week period. Each week is represented by an average result of three independent samples. These mean $BOD_5$ and COD values confirm that the vegetable oil refinery wastewater has a varying high organic pollution load. The variation of contents of sulfates, phosphates and chlorides was also observed.

### Comparison of the Technological Wastewater and the Final Treated Wastewater Effluent

The main pollution load in the technological wastewater are organic matters responsible for high biochemical and chemical oxygen demands, suspended solids and solvent extractables. From these results, $BOD_5$ organic

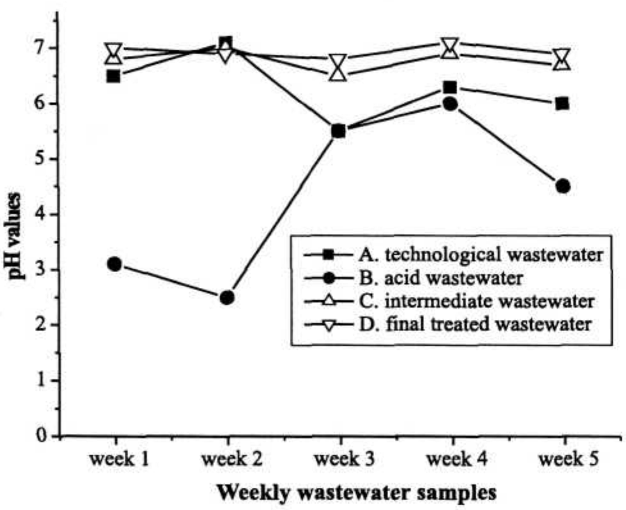


Fig. 3. Variations of wastewater pH values. Each of the results shown is represented by a mean value of 3 samples taken each week for 5 weeks.

Table 1. Physical and chemical characteristics of wastewater.

| Parameters* | Weekly wastewater samples** | | | | Pollutant removal [%]*** | | |
|---|---|---|---|---|---|---|---|
| | A | B | C | D | A' | B' | C' |
| **$BOD_5$, mg $O_2$/l** | | | | | | | |
| week 1 | 903 | 1350 | 1093 | 677 | 25.03 | 49.85 | 38.06 |
| week 2 | 700 | 3736 | 2133 | 539 | 23.00 | 85.57 | 74.73 |
| week 3 | 387 | 3600 | 1933 | 290 | 25.06 | 91.94 | 85.00 |
| week 4 | 725 | 2590 | 1585 | 544 | 24.97 | 79.00 | 65.68 |
| week 5 | 543 | 3230 | 1804 | 407 | 25.05 | 87.40 | 77.44 |
| **COD, mg $O_2$/l** | | | | | | | |
| week 1 | 1686 | 3300 | 2130 | 1349 | 19.99 | 59.12 | 36.67 |
| week 2 | 1266 | 7490 | 3629 | 1013 | 19.98 | 86.48 | 72.09 |
| week 3 | 689 | 8000 | 3384 | 551 | 20.03 | 93.11 | 83.72 |
| week 4 | 1379 | 4354 | 2387 | 1102 | 20.09 | 74.69 | 53.83 |
| week 5 | 977 | 5873 | 2691 | 782 | 19.96 | 86.68 | 70.94 |
| **SS, mg/l** | | | | | | | |
| week 1 | 1200 | 2538 | 727 | 360 | 70.00 | 85.82 | 50.48 |
| week 2 | 904 | 5350 | 746 | 228 | 74.78 | 95.74 | 69.44 |
| week 3 | 491 | 5333 | 388 | 98 | 80.04 | 98.16 | 74.74 |
| week 4 | 911 | 3003 | 606 | 155 | 82.99 | 94.84 | 74.42 |
| week 5 | 719 | 3670 | 451 | 144 | 79.97 | 96.08 | 68.07 |
| **SE, mg/l** | | | | | | | |
| week 1 | 800 | 4750 | 614 | 320 | 60.00 | 93.26 | 47.88 |
| week 2 | 603 | 4300 | 516 | 211 | 65.01 | 95.09 | 59.11 |
| week 3 | 345 | 3877 | 23823 | 104 | 69.86 | 97.32 | 99.56 |
| week 4 | 692 | 2896 | 462 | 187 | 72.98 | 93.54 | 59.52 |
| week 5 | 481 | 3178 | 352 | 144 | 70.06 | 95.47 | 59.09 |
| **Sulfates, mg/l** | | | | | | | |
| week 1 | 11800 | 1298 | 1049 | 792 | 1.00 | 93.29 | 24.50 |
| week 2 | 20600 | 2287 | 1728 | 1158 | 1.03 | 94.38 | 32.99 |
| week 3 | 18600 | 1302 | 1204 | 1180 | 0.00 | 93.66 | 1.99 |
| week4 | 20971 | 2097 | 1494 | 877 | 1.46 | 95.82 | 41.30 |
| week 5 | 10966 | 1097 | 1003 | 909 | 0.11 | 91.71 | 9.37 |
| **Phosphates, mg/l** | | | | | | | |
| week 1 | 702 | 6500 | 416 | 211 | 69.94 | 96.75 | 49.28 |
| week 2 | 600 | 6520 | 378 | 186 | 69.00 | 97.15 | 50.79 |
| week 3 | 550 | 3870 | 306 | 154 | 72.00 | 96.02 | 49.67 |
| week 4 | 645 | 4599 | 362 | 193 | 70.08 | 95.80 | 46.69 |
| week 5 | 455 | 6982 | 332 | 146 | 67.91 | 97.91 | 56.02 |
| **Chlorides, mg/l** | | | | | | | |
| week 1 | 750 | 543 | 598 | 742 | 1.07 | –36.65 | –24.08 |
| week 2 | 602 | 509 | 555 | 596 | 1.00 | –17.09 | –7.39 |
| week 3 | 520 | 680 | 306 | 690 | –32.69 | –1.47 | –125.49 |
| week 4 | 700 | 701 | 700 | 665 | 5.00 | 5.14 | 5.00 |
| week 5 | 770 | 680 | 726 | 800 | –3.90 | –17.65 | –10.19 |

Explanations:
* Each of the results shown is represented by a mean value of 3 samples taken each week for 5 weeks. $BOD_5$ - biochemical oxygen demand; COD - chemical oxygen demand; SS - suspended solids; SE - solvent extractables;
** A - technological wastewater; B - acid wastewater; C - intermediate wastewater; D - final treated wastewater.

***poluutant removal [%] from wastewater X, where X = A,B or C, calculated as: $X' = (X-D)/X \cdot 100$, where $X' = A', B'$ or $C'$

load is only reduced by about 25%, whereas COD is reduced by about 20%. The removal of sulfates, phosphates and chlorides also varied despite the fact that their concentrations in the influent streams were almost similar. These results show that the process removes more of suspended solids and solvent extractables than the removal of $BOD_5$ and COD. This indicates that the physicochemical treatment methods are less efficient in removal of $BOD_5$ and COD, which probably result from dissolved and more stable emulsified organic compounds.

According to the data from Table 1, the removal of the pollutants greatly varies. These variations can be attributed to many factors as mentioned in the Introduction. Furthermore, the removal of the pollutants depends on the effectiveness of the coagulation process; when alum is mixed with water it dissociates to $Al^{3+}$ ions, which hydrate to form $Al(H_2O)_6^{3+}$ that further undergo hydrolytic reactions to form aluminum hydroxide ($Al(OH)_3$) precipitate. $Al(OH)_3$ removes suspended solids, organic matter and phosphates by coprecipitation or adsorption at the surface [7]. However, these mechanisms of pollutant removal only insignificantly influences the reduction in the concentration of sulfates (Table 1). The results show that the concentration of chlorides, unexpectedly, decreased (weeks 1 and 2, Table 1); the mechanisms that caused the reduction of chloride content are unclear and difficult to justify since chloride salts used are soluble. Nevertheless, because the final effluent is resulting from the treatment of intermediate wastewater; therefore, the increase in chloride content is probably due to additional chlorides from alum and the acid wastewater stream, which is earlier treated with calcium chloride. Moreover, water which is used for preparation of reagents may also contribute to the high concentration of chlorides. Van Bens Benschhoten and Edzwald [8] reported that initial OH/A1 molar ratio, temperature, rate of addition of $OH^-$, presence of different anions and aging period of solutions all influence the extent of formation of Al(OH)3. Therefore, the observed variations in pollutant removal could also be due to the factors mentioned. pH should also be taken into account as it affects the efficacy of the coagulant. Omoike and Vanlloon [7] reported that $Al(OH)_3$ exhibits solubility in the pH 6.0 - 6.5 range and above these pH values solubility increases. As shown in Fig. 3, the pH of the wastewater fluctuated appreciably below and above this range. Hence, the pH fluctuations also contributed to variations in degree of pollutant removal even though the level of pollution of the influent wastewater was similar.

## Comparison of Acid Wastewater and the Final Treated Wastewater Effluent

Although the quantity of acid wastewater is relatively low, the extent of its pollution load is larger than that of technological wastewater as indicated by the 5-day biochemical oxygen demand, chemical oxygen demand and other tests (Table 1). The process removes 50-90% and 60-90% of $BOD_5$ and COD, respectively. The removal is not consistent and it varies considerably in accordance with the variation of the wastewater quantity and characteristics. However, the removal of suspended solids and solvent extractables is more than 90% and it is consistent. Thus, the high values of $BOD_5$ and COD in the final treated wastewater effluent are due to the presence of dissolved or very stable emulsified organic compounds in the wastewater which cannot be removed by physicochemical methods. The removal of sulfates and phosphates is about 95 and 98%, respectively, while the

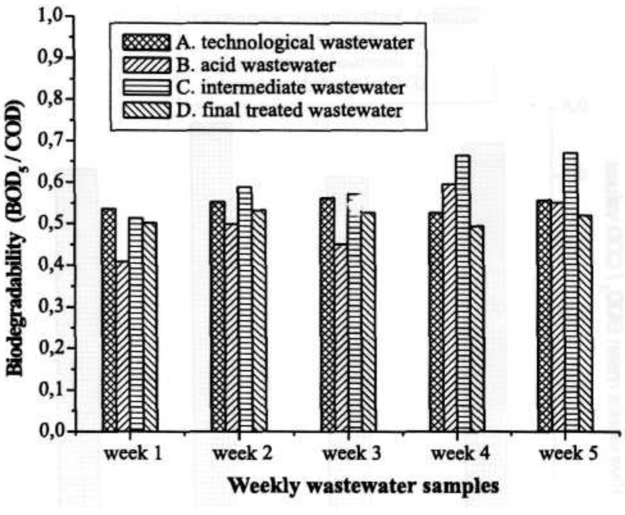


Fig. 4. Relative biodegradability of wastewater. Each of the results shown is represented by a mean value of 3 samples taken each week for 5 weeks.

removal of chlorides is greatly insignificant; increase is predominant over decrease (Table 1).

Some of the reactions taking place in the reactors can be shown schematically as:

$$3Ca^{2+} + 2PO_4^{3-} \rightarrow Ca_3(PO_4)_2$$

$$Ca^{2+} + SO_4^{2-} \rightarrow CaSO_4$$

$$Ca^{2+} + 2RCOO^- \rightarrow Ca\ (RCOO)_2,$$

where $RCOO^-$ – fatty acid ; R – hydrocarbon chain.

Because of the varying concentrations of $PO_4^{3-}$ and $SO_4^{2-}$ ions and other wastewater contaminants, the precipitation of calcium salts is also likely to be affected. As a result, this causes the observed differences in the removal of the pollutants (Table 1). Therefore, excess dosages of calcium ions should be expected to promote the removal of sulfate, phosphate, fatty acids and other pollutants. The mechanism by which these calcium salts remove organic matter and other suspensions is given in the subsection: acid wastewater treatment process.

From the equations shown above, it is evident that the increase in concentration of chlorides in the final treated wastewater effluent was predominant because the acid wastewater is treated with calcium chloride as a coagulant. However, the cause of the decrease in concentration of chlorides observed during week 4 is unclear and difficult to explain.

Generally, these results show that the physical and chemical processes in use are less efficient, particularly in the removal of organic contaminants responsible for $BOD_5$ and COD. However, they are more efficient for removal of suspended solids, solvent extractables, sulfates and phosphates. For the effective removal of organic pollutants, in addition to physical and chemical methods, perhaps a biological treatment process may have to be used to improve the quality of the final effluent.

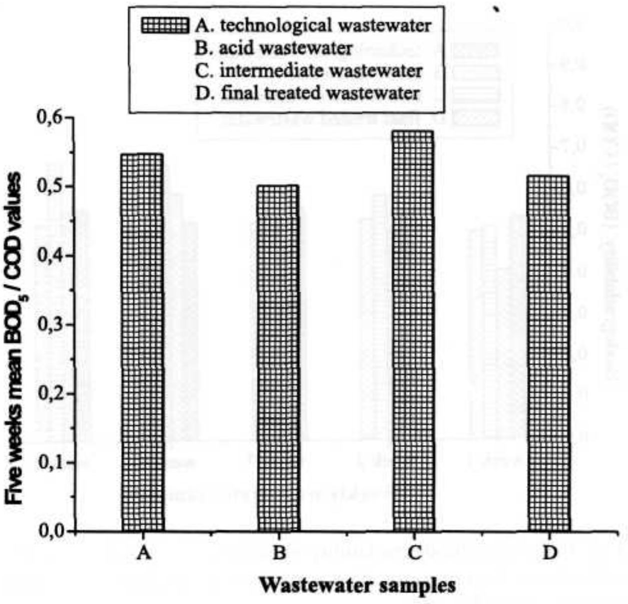


Fig. 5. Five weeks mean $BOD_5$/COD values of wastewater.

In this respect, $BOD_5$/COD ratios of the untreated and treated wastewater were calculated in order to evaluate the potential biodegradability of the organic compounds in the wastewater. Results are illustrated in Fig. 4. $BOD_5/COD > 0.5$ can be interpreted to suggest that the organic compound is biodegradable [9, 10]. Thus, these results indicate that most of the organic compounds in vegetable oil refinery wastewater should be biodegradable. However, the relative biodegradabilities of the wastewater greatly fluctuated. During the period of these experiments, different oils (rapeseed, sunflower, soybean and palm) were processed. Nevertheless, no special attention was paid to monitoring the relationship between the oil processed and the wastewater characteristics. Other researchers attribute fluctuation of vegetable oil wastewater characteristics to different oils refined [1, 11] and to operating conditions and processes [11]. Further, wastewater characteristics are not only influenced by raw materials and products processed, but also by water usage including washing procedures during and after the production cycles [9], These factors together with those mentioned in the previous sections apparently influenced the observed differences in the $BOD_5$/COD values of each wastewater. According to data in Fig. 4, the $BOD_5$/COD values for technological wastewater were higher than those for acid wastewater. Therefore, acid wastewater, which is characterized by a high rate of oxidizable substances, fats, fatty acids, sulfates and phosphates [12], as well as low pH values (Fig. 3), would be less biodegradable. Intermediate wastewater had higher $BOD_5$/COD values than final treated wastewater. Therefore, the results also suggest that physicochemical treatment processes carried out have both negative and positive influences on the biodegradability of the wastewater; intermediate wastewater had the highest $BOD_5$/COD values, but the values were significantly reduced during the next treatment steps.

The mean $BOD_5$/COD values for the five weeks period are shown in Fig. 5. Although the $BOD_5$/COD value for technological wastewater was the highest during the first week, on average it was found that the intermediate wastewater had the highest $BOD_5$/COD value. Hence, results shown in Fig. 5 suggest that a mixture of technological wastewater and treated acid wastewater may be easily treated by a biological treatment process.

Suspended solids and solvent extractables are used to evaluate wastewater pollution load and treatability. Suspended solids and solvent extractables in vegetable oil refinery effluents may originate from emulsified fats and oils, soaps and oil and fat spillage [11]. The nature of these sources makes them predominantly organic. Hence, these industrial effluents can respond to biological treatment; combined treatment consisting of physicochemical and biological treatment would remove emulsified fats and oils and other organic matter more efficiently [2, 3, 12, 13, 14].

## Conclusion

The effectiveness of the treatment process was different for each parameter monitored ($BOD_5$, COD, suspended solids, solvent extractables, sulfates, phosphates, and chlorides). Reduction of one of these parameters does not guarantee that others have been equally affected. Results show that the removal of suspended solids and solvent extractables was almost consistent and higher than that of $BOD_5$ and COD. It can be affirmed, therefore, that most of the organic contaminants leading to high $BOD_5$ and COD values are due to soluble and stable emulsified organic matter, which the physicochemical treatment system does not remove from both the acid and technological wastewater. It was also affirmed that the removal of sulfates and phosphates from the acid wastewater was efficient, but poor from the technological wastewater. As a result of the use of coagulants (calcium chloride and alum), the concentration of chlorides increased in the final treated wastewater. On the other hand, such processes as coagulation, flocculation and sedimentation are insufficient and produce sludges which are not only difficult to remove, but also a burden to the environment [15, 16]. The physicochemical treatment processes significantly influence the relative biodegradability of the organic compounds in the wastewater. Hence, for effective treatment of vegetable oil refinery wastewater, in addition to physicochemical methods, a biological treatment process would probably improve the quality of the final effluent and ensure the reduction in biodegradable organic matter content.

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