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Pretreatment of High Organic Load Dairy Industry Wastewater by Chemical Coagulation and Advanced Oxidation Processes

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ABSTRACT

This study was conducted to characterize the dairy industry wastewater and evaluate the efficiency of chemical coagulation and advanced oxidation process (AOPs) as pretreatment techniques. A composite sample was collected from diary industry plant at New Damietta City on March 2018 and characterized for some physico-chemical parameters to check the pollution potential of the effluents. Due to the high content of total suspended solids, chemical coagulation/precipitation process using lime, alum/lime and alum/polyacrylamide was applied for pretreatment of the collected sample and the optimum conditions were determined. Moreover, chemical coagulation using alum/lime followed by AOPs with Fenton's reagent was also assessed. The results showed that the concentration of COD is 112000 mg/L while for BOD is 78000 mg/L. In addition, the removal percentage of oil and grease, COD, total phosphorous (TP) and total Khejdal nitrogen (TKN) by Fenton's reaction after treatment with alum-lime are 86, 85, 99.35 and 99.03 %, respectively compared with that achieved by alum-lime (86.82, 73.11, 91.8 and 54.93 %, respectively). It was concluded that combining chemical coagulation/precipitation and AOPs was effective for the pretreatment of high organic load dairy wastewater.

Keywords: Alum, COD, Dairy effluent, Fenton's reaction, Lime and Physicochemical parameters.

INTRODUCTION

Dairy industry becomes one of the most environmental polluting industries, because of high organic load, large volumes produced, and seasonal variability as well. These industries consume huge quantity of water (Nagappan *et al.*, 2018). The effluents of dairy processing are produced in a discontinuous way with significant change flow rates. The composition and volume of the wastewater generated is affected by the type of product, the manufacture program, design of the processing plant, the water management system applied, and the amount of water being preserved (Sivaprakasam and Balaji, 2019; Kushwaha *et al.*, 2011).

Effluent of dairy industry is characterized by high chemical oxygen demand (COD) due to high organic content of fats, carbohydrates and proteins present in milk (Nagappan *et al.*, 2018). The presence of suspended matter in milk effluent is mainly due to tiny curd found in cheese waste (Joshiba *et al.*, 2019). In addition to large quantities of casein and inorganic salts, dairy wastes also contain detergents and sanitizers that used for washing (Lateef *et al.*, 2013). Decomposition of casein leads to the generation of intense black sludge's and vigorous butyric acid odors (Qasim and Mane 2013). Dumped of the highly nutritive effluent from the dairy industry without treatment into rivers it causes deterioration due to eutrophication (Sabliy *et al.*, 2019).

Pretreatment of dairy wastewater is necessary before biological treatment to remove detergents, oil and grease which increase the formation of filamentous organisms that inhibit the degradation of organic matter through creation scum and foam layers in the aeration tanks (Ying *et al.*, 2002). In addition, adsorption of oil and grease on sludge surface may limit the conveyance of oxygen and soluble substrates to the biomass which leads to reduction in substrate conversion rate (Farizoglu and Uzuner, 2011).

Coagulation/precipitation (C/P) process has been used for wastewater treatment to split up suspended and/or fatty particles because it is easy to operate, cost-effective, and energy saving treatment alternatives. The most widely used coagulants are; lime, alum and iron salt (El-Gohary *et al.*, 2010).

Advanced Oxidation Process (AOPs) is used worldwide for wastewater treatment due to its ability to remove small amounts of hazardous health pollutants. Moreover, AOPs treatment used in treating industrial and municipal wastewater, depending on the production of highly oxidative hydroxyl radical that can break complex toxic molecules in wastewater, thus making them more biologically degradable (Ahmad *et al.*, 2016). The common oxidizing agent is hydroxyl radicals which acts as powerful oxidizing agents, and have enough potential to efficiently destruct pollutants and make wastewater less toxic (Tien and Luu, 2020).

The objectives of this study are to; 1) characterize the dairy industry wastewater to check their pollution potential, 2) investigate the efficiency of chemical coagulation/precipitation process using lime, alum-lime and alum-polyacrylamide as pretreatment technique and 3) evaluate the efficiency of chemical coagulation using alum-lime followed by AOPs for their pretreatment.

MATERIALS AND METHOD

Sampling Site

The wastewater sample was collected from a plant for manufacturing dairy products located in the industrial Zone of New Damietta City, Damietta, Egypt. This plant produces dairy products as milk, cream, hard, soft and cottage cheese, yoghurt, whey powders and butter. A composite (Collection of 12 individual samples taken each 2 hrs., over a 24 hours and mixed together) large quantity water sample was collected on March 2018 in high density polyethylene (HDPE) container that was routinely acid-treated and

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well rinsed with de-ionized water prior to use, dried, and stored with the caps on to prevent contamination.

Physico-Chemical Characterization

The physicochemical characteristics of dairy wastewater sample were analyzed. The studied parameters; pH, Salinity, Electrical Conductivity (EC), Turbidity, Total Dissolved Solids (TDS), (TSS), Dissolved Oxygen (DO), Biological Oxygen Demand (BOD), Chemical Oxygen Demand (COD), Total Phosphorus (TP), Total Nitrogen (TKN) and Oil &Grease, have been determined according to Standard Method (APHA, 2017). The pH of the collected samples was measured directly by pH Meter (HANNA model 211, USA). Salinity, EC and TDS were measured by Digital Portable TDS/ Conductivity meter (HANNA Model 8033, USA). Turbidity was measured by Nephelometric method using Turbidimeter (Aqualytic Al1000, Germany with measuring a range from 0-200 NTU). The total suspended solids (TSS) are determined by the evaporation of a sample after filtration at 105 ºC (Adams, 1991).

Coagulation/Precipitation Process

The coagulants used were lime and alum (aluminum sulfate). Two coagulants aids (lime and polyacrylamide) were used with alum to improve the coagulation process. Lime was used as coagulant and coagulant aide after conversion to milk-of-lime which is suspension (slurry) of Ca $(OH)_2$ in water (Asadi, 2006). The operational parameters such as pH and coagulant dose were optimized. All experiments of chemical coagulation were accomplished using jar test according to Rump (1999). The COD was selected to be the observed parameter, while the other parameters were deter-mined after optimizing all conditions. The percent removal was calculated as following:

$$\% \text{ Removal} = (Ci - Cf)/Ci \times 100$$

Where Ci and Cf are the initial and final concentration, respectively

Advanced Oxidation Process by Fenton's Reaction

The Fenton's reagent was prepared according to Benatti and Tavares, (2012). This reagent consists of H_2O_2 and Fe^{2+} ions, which produce highly oxidative species at lower pH values (Gu *et al*., 2013). The dairy wastewater sample was treated by Fenton's reaction after C/P process with alum-lime. The effects of ferrous ion concentration, volume of hydrogen peroxide and pH on the reduction of COD for dairy industry wastewater were investigated.

RESULTS

Physico-Chemical Characterization

One initial step of result evaluation is to compare the physicochemical analysis of the dairy industry wastewater sample for the current study with the Egyptian Environmental Regulations Law No. 4 /1994 and 44/2000 (Table 1). The results showed that the concentration of COD is 112000 mg/L while BOD is 78000 mg/L. Oil and grease, TP, TKN and TSS are 795.8, 65992, 2243.384 and 113192 mg/L, respectively. The elevated organic load in the characterized sample was mainly due to presence of protein and carbohydrates (Slavov, 2017). Tawfik *et al.*, (2008) studied wastewaters from Arab Dairy Factory and found COD, BOD and TS (3383±1345 mg/L), (1941±864 mg/L), and (831 ± 392 mg/L), respectively. Deshannavar *et al*., (2012) studied wastewater from Dairy effluent and found COD, BOD and TS (1900-2700 mg/L), (1200-1800 mg/L), and (900-1350 mg/L), respectively.

Table (1): Characterization of the dairy industry wastewater sample compared with Egyptian Standards.

Parameter	Unit	Value	Executive regulations44/2000 for drainage sewer network	Law 4/94 for drainage on coastal environment
pH	-	3.65	6-9.5	6-9
Total Dissolved Solids (TDS)	g/L	45.9	-	2000
Electrical Conductivity (EC)	mS/cm	67.7	-	-
Salinity	%	46.4	-	-
Turbidity	NTU	2190	-	-
BOD	mg/L	78000	600	60
COD	mg/L	112000	1100	100
Oil and grease	mg/L	795.8	100	10
Total Phosphorous (TP)	mg/L	65992	-	10
Total suspended Solids (TSS)	mg/L	113192	800	60
TKN	mg/L	2243.384	-	-

Pretreatment Processes

Two pretreatment schemes were applied for dairy industry wastewater sample under investigation; a) coagulation/precipitation and b) coagulation followed by advanced oxidation process (Fenton's reaction). The fundamental action of coagulants is to flocculate suspended particulates into larger ones that can be extracted by sedimentation or flotation. Two distinct mechanisms may explain coagulation process; neutralization of anionic colloids by cationic hydrolysis products, and incorporation of impurities in an amorphous hydroxide precipitate, sweep flocculation (Loloei *et al*., 2019).

It is found that removal percent of COD at zero coagulant dosage is 35-43%, this due to plain sedimentation, the suspended solids or settable fraction, are removed from the solution by gravitation to form sludge under near quiescent conditions on the bottom of the container. This method also removes settleable organic and inorganic materials and thus reduces the organic load (Gregory and Duan, 2001).

Fenton reaction has received great attention in domestic and industrial wastewater treatment. The main advantage of the Fenton process is the complete destruction of contaminants to stable compounds (Chatterjee *et al*., 2015 and Baroudi *et al*., 2012). The main parameters that affect Fenton's reaction are pH, iron (II) concentration, H_2O_2 volume, initial pollutant concentration and presence of other ions (Zahrim *et al*., 2011 and Aguilar *et al*., 2005).

Coagulation/Precipitation Process Using Lime

The results of coagulation/precipitation process using lime were presented in Figure 1. The influence of lime dosage (0 to 2 g/L) on coagulation process was critically examined (Figure1a). It is found that 1g/L lime dose is appropriate for COD reduction (57.14 %). At a higher dose of lime (2 g/L), undesired rise of pH (11.7) is observed due to the partial dissolution of lime into sample solution which need further neutralization with acid before disposal (Abdul-Majeed and Oleiwi, 2015). Lime was used as a coagulant at lower dose (0.6 g/L) for the color removal of dye from industrial wastewater (Kiely, 1997). The influence of pH (4.76 - 9.36) of wastewater sample on coagulation process at the adopted lime dose (1 g/L) was examined (Figure 1b). The optimum pH is 8.29 for maximum COD removal efficiency. It was reported that lime has many advantages such as adjusting solution pH to the optimum value and improving sludge settleability and stability (Georgiou *et al*., 2003).

(a) **(b)**

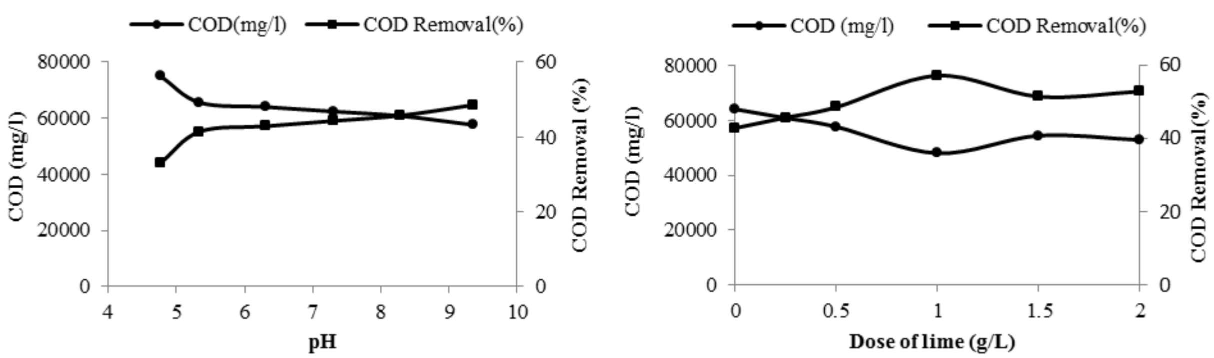


Figure (1): Coagulation/Precipitation process using lime (a) Determination of optimum lime dose, (b) Determination of optimum pH at 1.5 g/L lime dose.

The action mechanism of lime (Ca $(OH)_2$) is removing calcium ions and phosphorus from wastewater sample, in addition to any suspended solids. The lime reacts first with the natural alkalinity to form calcium carbonate that enhances suspended solids removal (Al-Asmar, 2006), and then calcium ions combine with the orthophosphate presents (pH 10.5) to generate insoluble and gelatinous calcium hydroxyapatite

$$Ca\ (HCO_3) + Ca\ (OH)_2 \rightleftharpoons 2CaCO_3 \downarrow + 2H_2O \quad (1)$$

$$5Ca^{2+} + 4\ OH^- + 3HPO_4^{2-} \rightarrow Ca_5(OH)(PO_4)_3 \downarrow + 3H_2O \quad (2)$$

The alkalinity of wastewater is important in determining the exact chemical dosage of lime rather than the amount of phosphate present to avoid the large quantities of produced lime sludge which need disposal (El-Gohary and Tawfik, 2009).

Cagulatioon/Precipitation Process Using Alum

The results of C/P process using alum were presented in Figure (2). The influence of alum doses on the COD removal efficiency of wastewater samples were investigated (Figure 2a). The COD removal percentage increased from 35.7 to 48.5% with increasing alum dose (0 to 1.5 g/L). Adsorption and charge neutralization is the predominant mechanism for COD removal at low alum doses where at high doses the sweep floc coagulation by enmeshment in the aluminum hydroxide precipitate prevail (Amud and Alade, 2006). Thus, the optimum alum dose that achieved maximum removal of COD was 1.5 g/L. A higher alum dose (5 g/L) was effective in reducing COD level of the collected dairy effluent (Jopson, 2004). A reduction percentage (23.4) for COD was achieved after treatment of sweet whey, major pollutant in dairy effluent, by aluminum sulphate and sodium alginate (Laine and Cheng, 2007).

The effect of pH on the COD removal efficiency of dairy wastewater sample was examined (Figure 2b). The COD removal increased from 25–71.4% by increasing pH from 3.61 to 9.39. Thus, 9.39 were selected as the optimum pH in the subsequent work. The COD removal percent of dye containing wastewater by alum at pH 5 was 68% (El-Gohary and Tawfik, 2009).

Two coagulants aids (lime and polyacrylamide) were used with alum to improve coagulation process

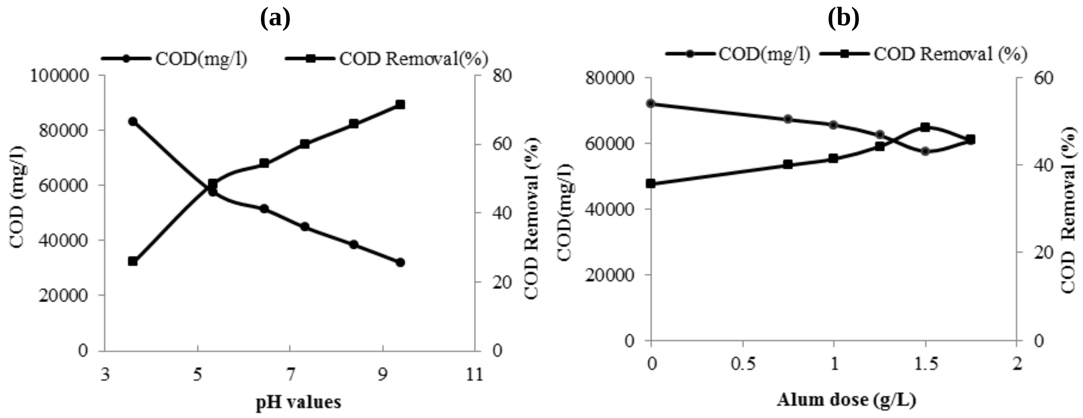


Figure (2): Coagulation/Precipitation process using alum (a) Determination of optimum dose of alum, (b) Determination of optimum pH at 1.5g/L alum dose.

(Figure3). Addition of different polyacrylamide doses (0 to 0.5g/L) to wastewater samples was studied (Figure 3a). The results indicated that the optimum dose of polyacrylamide is 0.05g/L with COD removal percentages of 67.14 %. However, increasing polymer dose above 0.05g/L has no significant effect due to the bridging mechanism (Neyens and Baeyens, 2003). Moreover, the removal of COD decreased to 50% at higher polyacrylamide doses due to the presence of some organic polymer remains in the treated samples (Figure3a). Therefore, the recommended polymer does for the subsequent work was 0.05 g/L. The addition of anionic polyacrylamide increases the flocculation efficiency of the coagulant that leads to increase the settling speed, reduce the amount of coagulant required for the treatment and then lower the cost of the coagulation-flocculation process (Krzemińska *et al.*, 2015). Alum dose 0.5g/L aided with 0.02 g/L of polyacrylamide was applied for treatment of simulated dairy industries wastewater (El-Gohary and Tawfik, 2009).

Different lime doses (0-1.5g/L) at the recommended dose of alum (1.5g/L) have been examined at pH 9.39 (Figure3b). The optimum dose of lime is 0.5g/L with COD removal of 82.8%. Jain *et al.*, (2019) studied the capability of turbidity removal from wastewater of Al-Ahdab Oil fields by alum/lime and found enhancement in the turbidity removal when the ratio of alum and lime (coagulant aid) was 3:1 as compared to alum alone. The addition of alum to water releases hydrogen ions and consequently lowers the pH which retard the formation of an effective floc, Al $(OH)_3$. Therefore, addition of lime can improve the alkalinity and optimize coagulation process (Gregory and Duan, 2001).

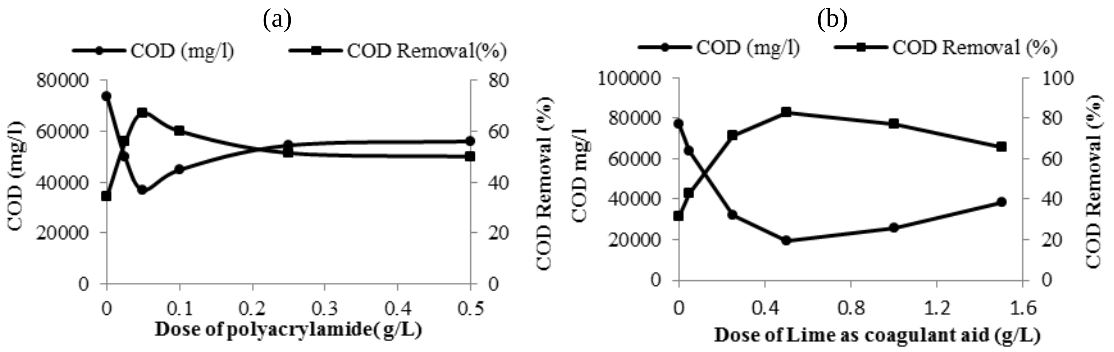
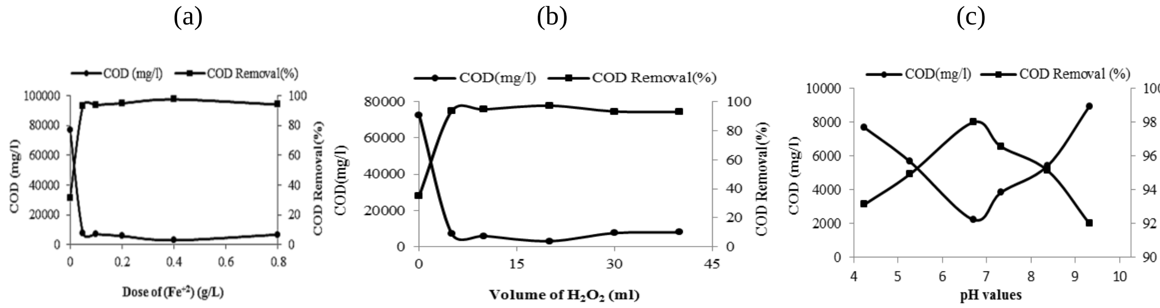


Figure (3): Coagulation/Precipitation process using alum (a) Determination of optimum dose of polyacrylamide, (b) Determination of optimum dose of lime at 1.5g/L alum dose.

Advanced Oxidation Process (AOP) by Fenton's Reagent

The results of Fenton's reaction application after C/P process with alum -lime were presented (Figure 4). The influence of ferrous ions concentration on COD removal was examined (Figure 4a). Different concentrations of ferrous ions (0 to 0.8 g/ l) and 20 ml of H_2O_2 (30%) were added to 1L of alum-lime treated samples. The optimum dose of ferrous ion was 0.4g/L with 97.4% COD removal. However, increasing the dose of ferrous ion above 0.4g/L, decrease the COD removal. The reduction in COD changed from 35.17 to 97.14% with increasing the concentration of hydrogen peroxide up to 20 % (Figure 4b). Addition of $FeSO_4/H_2O_2$ reduced not only colloids but also soluble contaminants from milk processing effluents; up to 80% of fat removal (initial concentration of 1931 mg/L) was achieved (Slavov, 2017).

The effect of pH (4.25 to 9.32) on Fenton's reaction efficiency was studied (Figure4c). The removal of COD increased from 93.1 to 96.5 % with changed pH from 4.25 to 7.32. Therefore, 7.32 were chosen as the optimum pH for the subsequent work.



Figure(4): Fenton reaction after alum-lime (a) Determination the optimum dose of Fe^{2+}, (b) Determination the optimum volume of H_2O_2 ml at 0.4 g/L Fe^{2+}, (c) Determination the optimum pH at 0.4 g/L Fe^{2+} and 20 ml of H_2O_2 (30%).

Initial pH of 5; 10 mM Fe^{2+}; 100 mM H_2O_2 and 30min reaction time were recommended as optimum operational conditions for olive mill wastewater treatment (Sivrioğlu and Yonar, 2015). Ikechukwu (2012) applied Fenton process to dairy effluents from dairy plant in Bursa City, Turkey and the optimum conditions determined were pH 3.5, 800 mg/L H_2O_2 and 700 mg/L $FeSO_4$ that achieved 74% COD removal.

Comparison of Treatment Processes Effectiveness

The physicochemical analysis of dairy wastewater sample treated by alum-lime (T1) and that treated by Fenton's reaction after alum-lime (T2) and the overall removal percentage were presented in Table (2). The COD % removal was 82.86 and 85 for samples T1 and T2, respectively. One of advantages through adopting advanced oxidation process using Fenton's reaction is the ability to remove mostly colloids as well as soluble contaminants from milk processing effluents (Slavov, 2017). This is obvious from the removal efficiency of oil and grease, TKN and total P (Table 2). The performance of advanced oxidation process using Fenton's reaction is more effective for TKN removal (99.03%) than alum-lime (54.93%).

Table (2): Physico-chemical characterization of the pretreated sample and the removal percentage.

Parameter	Unit	T1*	Removal %	T2**	Removal %	The overall % Removal [a]
pH	-	6.97	-	6.9	-	-
Turbidity	NTU	61	97.21	12.9	78.85	99.41
COD	mg/L	19200	82.86	2880	85	97.43
Oil and grease	mg/L	214	73.11	30	85.98	96.23
Total Phosphorous (TP)	mg/L	5411.12	91.8	35.195	99.35	99.95
Total Suspended Solids (TSS)	mg/L	50774	68.09	26810	47.2	76.3
TKN	mg/L	1011.2	54.93	9.8412	99.03	97.55

T* (1): sample treated with alum- lime
T** (2): sample treated with Fenton's reaction (after alum - lime)
[a] : The overall % removal for the sample after combined treatment with alum-lime and Fenton's reagent .

Removal percent of total-P by alum-lime was 91.8% and 99.35% after Fenton's reaction. Phosphates may be removed as soluble complex through the interaction with aluminum forms, or as insoluble complexes, producing compounds with formulae: Al $(OH)_3$-x(PO_4)x, which can be adsorbed onto the positively charged Al (III) hydrolyzed species, or act as nucleus for the precipitation of Al (III) hydrolyzed products. Moreover, it may be removed by the direct adsorption onto Al $(OH)_3$-products; so-called sweep flocculation or precipitation with insoluble aluminum hydroxide (Jiang and Graham, 1998). Total suspended solids does not mostly affected by Fenton's reaction compared to the coagulation with alum-lime as achieved removal percent of 68.09 and 47.2 for samples T1 and T2, respecttively. The (C/P) process is used to rem-ove solid material, turbidity, heavy metal, colour and organic matter in water and wastewater (Yonar *et al*., 2018).

Comparing the results of the present study with those published indicated a considerable variation in performance due to differences in treatment systems and operating conditions. Yazdanbakhsh *et al*., (2015) investigated three integrated techniques: coagulation, acid cracking and Fenton-like process to treat Olive oil wastewater with characteristics (COD 55.8 g/L, pH 5.02, BOD5 8.25 g/L and TP 0.44 g/L). Coagulation process with $FeCl_3$ removed 91.2% of COD where acid cracking and Fenton-like process achieved 94% and 98%, respectively. Combined treatment using coagulation/flocculation prior to UV-A LED photo-Fenton for Crystallized-fruit wastewater (COD 35.4 g/L, pH 6.95, BOD 6.6 g/L,TP 0.38 g/L and TSS 1.85 g/L) achieved 80% of COD removal, 99% of turbidity and 95% of total suspended solids (Rodríguez-Chueca *et al*., 2016). Coagulation of two olive mills wastewater, W1 and W2, characteristics (pH, TSS, TP, COD for W1 and W2 were 5.3, 36.7, 3.5 and 61.1 g/L and 5.1, 52.7, 2.5 and 29.3 g/L, respectively) achieved a COD and TP removal between 10–40% where combining coagulation and Fenton oxidation achieved 30–80%, respectively. Ginos *et al*., (2006) proved that the combined processes enhance COD removal to about 60%.

CONCLUSION

This work was adopted for the characterization of dairy industry wastewater and assessment the efficiency of coagulation/precipitation (using lime, alum/lime and alum/polyacrylamide) followed by AOPs (after alum/lime) as pretreatment techniques. The obtained results revealed that coagulation/precipitation process was effective as pre-treatment technique for dairy industry wastewater, and more specifically, for decrease the values of TSS, turbidity and a considerable part of organic matter to levels that facilitate further treatment. The maximum COD removal percent achieved with alum-lime was 82.8% where 67% was obtained using alum-polyacrylamide. Removal percentages of COD, oil and grease, total-P and TKN by Fenton's reaction after treatment with alum-lime were more effective com-pared with that achieved by alum-lime. Therefore, the combination of chemical coagulation/ precipitation and Fenton reaction is substantial for the pretreatment of high organic load dairy wastewater.

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المعالجة الاولية للمخلفات السائلة لصناعة الألبان باستخدام عمليتى التخثر الكيميائي والأكسدة المتقدمة

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الملخص العربـــــي

اجريت هذه الدراسة لتوصيف مياه صرف صناعة الألبان وتقييم كفاءة عمليتى التخثر الكيميائي والأكسدة المتقدمة كمعالجة اولية. تم تجميع عينة مركبة من المخلفات السائلة لمصنع البان بمدينة دمياط الجديدة و ذلك خلال شهر مارس 2018 و توصيف بعض الخصائص الفيزيوكيميائية لهذه العينة للتحقق من مدى التلوث. و نظرًا للتركيز العالي للاملاح الكلية العالقة تم تطبيق عملية التخثر الكيميائي و الترسيب باستخدام الجير، الشبه / الجير و كذلك الشبه / بولي أكريلاميد بهدف المعالجة الاولية للعينة المجمعة. تم دراسة الظروف المثلى لعملية التخثر الكيميائي باستخدام الشبه / الجير متبوعةً بالاكسدة المتقدمة باستخدام كاشف فينتون. أظهرت النتائج أن تركيز الأكسجين الكيميائي المستهلك هو 112000 ملجم / لتر بينما كان تركيز الأكسجين الحيوي المستهلك 78000 ملجم / لتر. كما اوضحت النتائج ان كفاءة عملية إزالة الأكسجين الكيميائي المستهلك ، الزيوت و الشحوم ، الفوسفور الكلى والنيتروجين الكلى باستخدام تفاعل فينتون بعد المعالجة بالشبه / الجير كانت 85 ، 86 ، 99.35 و 99.03 ٪ ، على التوالي مقارنة مع تلك التي حققتها عملية المعالجة بالشبه / الجير 82.86 ، 73.11 ، 91.8 و 54.93 ٪ ، على التوالي. و قد خلصت النتائج إلى أن الجمع بين التخثر الكيميائي / الترسيب و AOPs كان فعالًا كمعالجة أولية لمياه صرف صناعة الالبان ذات الحمل العضوي العالي.