

Pretreatment and Elimination of Toxicants from Industrial Waste Water Treatments

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Received: 23/7/2010

Abstract: Olive mill waste water (o.m.w.w) generated by the olive oil extracted industry is a great pollutant due to its high organic load. It is dependent upon the type of olive oil production process used. In this study the traditional & continuous process of oil extraction evaluated. Also the applicable & alternative methods for treatment of waste water dealing with aphysiochemical consideration of the properties. The results indicated that the Total Soluble Solids (T.S.S), Volatile Soluble Solids (V.S.S), Fixed Solids (F.X), Chemical Oxygen Demand (COD) and Biological Oxygen Demand after 5 days) (BOD₅) were more significantly higher in continuous mills than in traditional mills, but the value of (T.ph.s) Total polyphenols was slightly higher in continuous mills than in those traditional mills, the use of (PAC) powdered activated carbon achieved a highly significant removal % of T.ph.s, T.S.S, V.S.S, F.S, COD & BOD₅ arranged in a descending order as in the following: 60%, 50%, 50%, 50%, 43% & 22 % respectively. In case of treatment with a mixture of (2:1)% of (NaOH+Na₂CO₃), the percent of removal of T.ph.s, T.S.S, V.S.S, F.S, BOD₅ & COD were highly significant & arranged in a descending order as in the following: 60%, 40%, 40%, 40%, 30% & 25% respectively. And by using a mixture of (NaOH+Na₂CO₃) at a rate of (3:1) proved a more highly significant removal for the T.ph.s, T.S.S, V.S.S, F.S, BOD₅ & COD. These values of removal were arranged in a descending order as in following: 75%, 50%, 50%, 50%, 47% & 40% respectively. The use of FeSO₄.7H₂O was more effective in lowering the values of T.S.S, V.S.S & T.ph.s than FeCl₃.6H₂O & were arranged in a descending order: 40%, 40% & 30% respectively. While the use of FeCl₃.6H₂O was too more effective in decreasing value of F.S, COD and BOD₅ 68%, 50% & 30% respectively. Also it can be mentioned that the increase of H₂O₂ concentration to 5 ml at 2.5 gm of FeSO₄ improved organic matter degradation, however excessive amounts of oxidant to 10 ml has no or even a slight adverse effect on performance due to H₂O₂ induced radical scavenging. It is very common that the national & international goal for the control of water pollution is the elimination of pollutants discharge into navigable waters. Also the industry improvements often finds that it is more economic to treat waste water for reuse than discharge into Environment.

Keywords: Olive mill waste water, T.S.S, V.S.S, Fixed Solids, Chemical Oxygen Demand

INTRODUCTION

The olive mill waste water (o.m.w.w) which generated by the olive oil extraction industry has a great pollutant impacts due to its high organic load (Balice & Cera (1984), and also the phytotoxic & antimicrobial phenolic substances present resisting the biological degradation (Saez *et al.* (1992) & Paredes *et al.* (1986). The Mediterranean countries are mostly affected by these serious environmental problems, where they are responsible for 95% of the world wide olive oil production. These countries are producing more than 11 million tons of olives/year & about more than 1.7 million tons of olive oil are extracted. (Aktas *et al.* (2001) olive oil is extracted mainly in two ways, a traditional process by (a classical, pressing) and a continuous process by (centrifugation). Accordingly a comparative study screened the differences between the 2 types of oil extraction i.e by a traditional & continuous mills in North Sinai. In this study traditional batch and continuous batch of olive oil waste waters originated from these processes were evaluated. On the other hand the treatment of o.m.w.w systems are numerous and fall in large categories (I) bioremediation (In-vitro & In vivo), (II) thermal processes (incineration, pyrolysis, gasification), (III) evaporation (IV) advanced oxidation processes (digestion-oxidation & ozonation) (V) coagulation/flocculation/precipitation & distillation (Jenman (2007). Accordingly the present work was

conducted to study the treatability of some physiochemical parameters & some chemical oxidative processes on treatment of (o.o.m.w.w).

MATERIALS AND METHODS

Samples collection:

Sample collection of o.m.w.w samples were obtained from the evaporation ponds of local olive oil mills including the traditional mill & continuous mill from Kosyma & El-Arish respectively. The volume of each sample was 5 litre per treatment collected in a screw cap brown glass bottles and transported immediately to laboratory and kept in the dark at 4°C until analysis and treatment. Five replicates were used for each treatment per tested parameter.

Analysis: All chemicals used were of analytical reagents grade obtained from BDH or sigma chemical companies. Physical and chemical experiments as well as determinations of all tested parameters were carried out according to those recommended in 20th Ed. APHA 1998 (American Public Health Association Washington, DC.) by using An Ion Chromatography IC Model DX 500 Chromatography.

RESULTS AND DISCUSSION

The average values for typical parameters of o.m.w.w samples obtained from classical (traditional) and continuous (centrifugal) mills are presented in table (1).

The results in table (1) showed that the T.S.S, V.S.S, F.S, COD and BOD₅ were more significantly higher in continuous mills than in traditional mills, but the value of T.ph.s was slightly higher in continuous mills than in those traditional mills. These results were in agreement with the results obtained with (Esera *et al.* (2001) & Blika *et al.* (2009)). It is well known that the specific water demand of the continuous olive mill is more than 3 times higher than that of the traditional olive mills. (Abd.Elkrim & Shaheen (2007), so this will consequently led to adilution effect of all tested parameters, but consuming much higher amounts of wasted water and also that type of continuous mills were very familiar in most of countries and citizens. So for that reasons various physiochemical methods have been proposed for pretreatment of continuous o.m.w.w in laboratory.

Effect of Adsorbant (PAC) powdered activated carbon:

The powdered activated carbon used was a wide spread used in the waste water treatment industry. The

data presented in table (2) demonstrated that the use of PAC achieved a highly significant removal % of T.ph.s, T.S.S, V.S.S, F.S, COD & BOD₅. These removal % were arranged in a descending order as in the following: 60%, 50%, 50%, 50%, 43% & 22% for the tested parameters as in the following T.ph.s, T.S.S, V.S.S, F.S, COD & BOD₅ respectively. These results were in agreement with the results obtained by (Nony *et al.* (1975) concluding that filtration and adsorption system achieved a clean up of 100,000 fold of 2-Acetylene amine fluorine (2-AAF) from waste water. Also (El-Dib & Badway (1978) reported that rate of adsorption increased by time of contact and adsorption capacity of activated carbon was enhanced in the presence of a mixture of aromatic compounds. More over (Lambert *et al.* (1996) mentioned that the high percent of dye removal in U.K textile industry effluents exhibited by activated carbon. Recently, (Bertin *et al.* (2004) proved that GAC reactor exhibited COD & polyphenolic compounds depletion yields by 62% & 78% respectively.

Table (1): physiochemical characteristics of o.m.w.w types.

Tested parameters	lional milit Trad	inuuous mill Cont
PH	4.3±0.17	4.5±0.18
T.S.S (gm/l)	48.2±1.9	75.6±3.02
V.S.S (gm/l)	36.7±1.5	61.4±2.5
F.S. (gm/l)	11.5±0.96	14.2±0.57
COD (gm/l)	75.7±3.03	114.3±4.6
BOD ₅ (gm/l)	42.9±1.7	71.5±2.9
T.Ph.s (gm/l)	4.83±0.19	5.99±0.2

o.m.w.w Olive mill waste water, T.S.S Total soluble solids, V.S.S Volatile soluble solids, F.S Fixed solids, COD Chemical Oxygen Demand, BOD₅ Biological Oxygen Demand after 5 days, T.ph.s Total polyphenols

Table (2): Effect of powdered activated carbon treatment on physiochemical characteristics of o.m.w.w

Tested parameters	Time of contact		
	2 days	6 hrs.	Zero hour
PH	4.5±0.18	6.6±0.26	7.3±0.3
T.S.S (gm/l)	75.6±3.02	37.8±1.5	30.2±1.2
V.S.S (gm/l)	61.4±2.5	30.7±1.2	24.56±0.98
F.S (gm/l)	14.2±0.57	7.1±0.2	5.6±0.2
COD (gm/l)	114.3±4.6	65.2±2.6	54.9±2.1
BOD ₅ (gm/l)	71.5±2.9	55.7±2.2	43.7±1.7
T.Ph.s (gm/l)	5.99±0.2	2.99±0.11	3±0.12

o.m.w.w Olive mill waste water, PAC (1 gm/l of powdered activated carbon), T.S.S Total soluble solids, V.S.S Volatile soluble solids, F.S Fixed solids, COD Chemical Oxygen Demand, BOD₅ Biological Oxygen Demand after 5 days, T.ph.s Total polyphenols

Table (3): Effect of a mixture (NaOH+Na₂CO₃) treatment on physiochemical for different rates and times characteristics of o.m.w.w

Tested parameters	Time of contact		
	Zero hour	6 hrs.	2 days
	Raw sample onl	Treated sample (NaOH+Na ₂ CO ₃), (2:1)	Treated sample (NaOH+Na ₂ CO ₃), (3:1)
PH	4.5±0.18	8.9±0.35	9.5±0.038
T.S.S (gm/l)	75.6±3.02	45.36±1.8	37.8±1.5
V.S.S (gm/l)	61.4±2.5	36.8±1.4	30.7±1.2
F.S (gm/l)	14.2±0.57	8.5±0.34	7.1±0.3
COD (gm/l)	114.3±4.6	85.7±3.4	68.6±2.7
BOD ₅ (gm/l)	71.5±2.9	50.1±2	37.9±1.5
T.Ph.s (gm/l)	5.99±0.2	2.4±0.09	1.49±0.05

Time of contact 6 hrs., 2 days, o.m.w.w Olive mill waste water, T.S.S Total soluble solids, V.S.S Volatile soluble solids, F.S Fixed solids, COD Chemical Oxygen Demand, BOD₅ Biological Oxygen Demand after 5 days, T.ph.s Total polyphenols

In case of treatment with a mixture of (NaOH+Na₂CO₃) at 2 rates (2:1)% & (3:1)% where the volume of NaOH was 2 & 3 times of wt. of Na₂CO₃. The results in (table 3) indicated that firstly, in case of a mixture of (2:1)% of (NaOH+Na₂CO₃), the % of removal of T.ph.s, T.S.S, V.S.S, F.S, BOD₅ & COD were highly significant & arranged in a descending order as in the following: 60%, 40%, 40%, 40%, 30% & 25% respectively. 2-ndly by using a mixture of (NaOH+Na₂CO₃) at a rate of (3:1) proved a more highly significant removal for the T.ph.s, T.S.S, V.S.S, F.S, BOD₅ & COD. These values of removal were arranged in a descending order as in following: 75%, 50%, 50%, 50%, 47% & 40% respectively. The obtained results were in agreement with the results proved by (Zouboulis *et al.* (1996). where the separation of toxic metals, precipitates which were produced by employing either the conventional hydroxide (adding NaOH) or the carbonate-enhanced (adding NaOH+Na₂CO₃) strictly PH controlled precipitation. The necessary treatment time tested was usually 3-4 hrs. in order for the remaining conc.s of toxic metals to reach the environmental safety limits. Also (Hassan (1981) showed that it is possible to desilicate the black liquor of paper water waste up to 95% by using either quick lime, flue gas or sod. bicarbonate as a precipitating agent. Recently other studies by (Hafidi *et al.* (2004) showed that all tested oils that a dilute NaOH solution 20% allows the highest flux through the membrane i.e about 3 fold the flux resulting from 40% NaOH. Also (Lagoudianki *et al.* (2003) demonstrated that different amounts of Ca(OH)₂ were used in treatment of o.m.w.w for different intervals & an important reduction in waste water pollutants.

In an attempt to compare the relative efficiency of a suitable and fixed concentration (1gm/l) of some common coagulants used for treatment of o.m.w.w.s. In this experiment designed using two coagulants FeSO₄.7H₂O & FeCl₃.6H₂O were used for treatment of o.m.w.w. The results in table (4) recorded the variations in the relative efficiency of applying the 2 coagulants mentioned before. So the use of FeSO₄.7H₂O was more effective than FeCl₃.6H₂O in lowering the values of T.S.S, V.S.S & T.ph.s and were arranged in a descending order :40%, 40% & 30% respectively. But the use of FeCl₃.6H₂O was too more effective in decreasing value of F.S, COD & BOD₅ and these values were arranged in a descending order: 68%, 50% & 30% respectively. These data were in agreement with the data obtained by (Aly & Badawy (1986) & previous studies by (El-Dib & Badawy (1985), evaluated the efficiency of coagulants AlSO₄ & FeCl₃ for removal of PCB.s from drinking water, where AlSO₄ was more effective in % removal of PCB.s than FeCl₃ & that removal was ranged between 30% to 41% removal. Further (Guida *et al.*, 2004) reported that the coagulation - flocculation process is a versatile method used in drinking water production, industrial & domestic waste water treatment, and a sludge conditioning. The process is mainly based on the use of Al⁺³ or Fe⁺³ salts alone or in combination with calcium salts. Recently, (Jaouani *et al.* (2005) who studied (OME) olive mill effluents treatment by Fe⁺³ coagulation reported the existence of an optimum

coagulant concentration at which treatment efficiency was maximized. Further more & very recently (Ginos *et al.*, 2006) reported that coupling lime or FeCl₃ or FeSO₄ (in the range of several gm/l) with cationic poly-electrolytes (in the range of 200 - 300 mg / l) led to a quantitative T.S.S removal, while COD & T.ph.s removal varied between 10 - 40 % & 30 - 80 % respectively, depending on the materials and the effluent, in question; separation efficiency generally decreased with decreasing coagulant and /or flocculant concentration. On the other hand, (Nandy, *et al.* (2003) & (Eckenfelder (1989) reported that the ferrous sulphate functions over a wide PH range from 3-13, while ferric salts over about PH 4.5 (Sarika *et al.* (2005) & Nandy *et al.* (2003).

The results tabulated in table (5) showed that the increase in volume of FeSO₄ & H₂O₂ until a constant volume of both FeSO₄ & H₂O₂ led to a highly significant removal of T.S.S, V.S.S & F.S with a % of removal equal to 100%, 95% & 5% respectively. Also there was an elimination of COD, BOD₅ & T.ph.s, and the % of removal were equal to 60%, 40% & 85% respectively. On the other hand by doubling the volume of H₂O₂ to be 5 ml & the volume of FeSO₄ was 2.5 gm the results were similar to results when the 2 components has an equal volume 2.5 ml i.e the % removal of T.S.S, V.S.S & F.S were 100, 95 & 5% respectively, but only the % removal of COD, BOD₅ & T.ph.s were better & it has the % of removal equal to 75, 55 & 90% respectively. More over when the case was the opposite i.e the volume of FeSO₄ was 5 gm & volume of H₂O₂ 2.5 ml, the obtained results achieved a very similar results gained when the volume of FeSO₄ was 2.5 gm & H₂O₂ has a volume 1 ml. But when the volume of H₂O₂ was 10 ml & FeSO₄ 5 gm, the results showed that the % removal of T.S.S, V.S.S & F.S were 100, 98 & 2% respectively, but the % removal of COD, BOD₅ & T.ph.s were 50, 30 & 70% respectively. Accordingly it can be concluded that there was a certain specific volume for each component of the Fenton Reagent, i.e FeSO₄ & H₂O₂ to can achieve a best results. Also it can be mentioned that the increase of H₂O₂ concentration to 5 ml at 2.5 gm of FeSO₄ improved organic matter degradation, however excessive amounts of oxidant to 10 ml has no or even a slight adverse effect on performance due to H₂O₂ induced radical scavenging. The gained results were in agreement with the results of many studies in literatures. The chemical oxidation can be economically attractive at low consumption of chemicals, leading then to intermediate products that are less toxic and /or biodegradable (Grigorpoulou & philippopouls (1996) Most common oxidants are chlorine, ferrate, permanganate, ozone & Hydrogen peroxide (Bowers *et al.* (1988) & Eckenfelder (1989). The Fe/H₂O₂ (Fenton) treatment prior to activate sludge plant treatment has been studied for several industrial effluent streams (Bowers *et al.* (1989 & 1991) & landfill leachate by (Anagiotou *et al.* (1993). Further more the overall AOX (Advanced oxidation processes Reduction) for Fe/H₂O₂ treatment was 66 % (Tunkhanen *et al.* 1996). Very recently (Rivas *et al.* (2001) & Ginos *et al.* (2006) reporting that to enhance organic matter degradation, iron-based coagulation was coupled with H₂O₂, thus simulating a Fenton reaction

and this increased COD reduction to about 60%. Also recently (Mantzavinos and Kalogerakis (2005), postulated that AOP may be used either for complete mineralization of all pollutants to

CO₂, water & mineral salts or for the selective removal of the more resistant pollutants used their conversion to biodegradable intermediates.

Table (4): Relative efficiency of two common coagulants used for treatment of o.m.w.w

Tested parameters	Raw sample only	Type of coagulants	
		Treated sample FeSO ₄ .7H ₂ O (1 gm/l)	Treated sample FeCl ₃ .6H ₂ O (1 gm/l)
pH	4.5±0.18	6.3±0.3	4.8±0.19
T.S.S (gm/l)	75.6±3.02	45.36±1.8	56.7±2.2
V.S.S (gm/l)	61.4±2.5	36.8±1.5	52.2±2
F.S (gm/l)	14.2±0.57	8.5±0.34	4.5±0.18
COD (gm/l)	114.3±4.6	91.5±3.7	57.1±2.3
BOD ₅ (gm/l)	71.5±2.9	64.4±2.6	50.05±2
T.Ph.s (gm/l)	5.99±0.2	4.2±0.17	4.8±0.19

m.w.w Olive mill waste water, T.S.S Total soluble solids, V.S.S Volatile soluble solids, F.S Fixed solids, COD Chemical Oxygen Demand, BOD₅ Biological Oxygen Demand after 5 days, T.ph.s Total polyphenols

Table (5): The removal effect of coupling separation with Fenton oxidation agent for treatment of o.m.w.w.s

Conc.s of Fenton Reagent Components**		% Removal of tested parameters					
FeSO ₄ .7H ₂ O:H ₂ O ₂ *(35%v/v)		T.S.S	V.S.S	F.S	COD	BOD	T.Ph.s
1 (gm)	Zero	Zero	Zero	Zero	30%	15%	Zero
2.5 (gm)	100%	100%	90%	10%	45%	35%	65%
2.5 (gm)	100%	100%	90%	10%	55%	40%	75%
2.5 (gm)	100%	100%	95%	5%	60%	40%	85%
2.5 (gm)	100%	100%	95%	5%	75%	55%	90%
5.0 (gm)	100%	100%	90%	10%	50%	35%	60%
5.0 (gm)	100%	100%	98%	2%	50%	30%	70%

**Fenton Reagent → An active oxidative agent.

*H₂O₂ = conc./ H₂O₂ (v/v)35%.

Time of contact 8 hrs. in sunlight.

PH ranged between 2.1 – 3.6.

o.m.w.w Olive mill waste water, T.S.S Total soluble solids, V.S.S Volatile soluble solids, F.S Fixed solids, COD Chemical Oxygen Demand, BOD₅ Biological Oxygen Demand after 5 days, T.ph.s Total polyphenols.

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المعاملة الأولية وإزالة المواد السامة من مياه المخلفات الصناعية السائلة

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تعتبر المياه التي تنتج من صناعة زيت الزيتون ملوث خطير لأنها تحتوي علي جمل عضوي كبير، ويعتمد نوع المياه علي نوع العملية المستخدمة في إنتاج زيت الزيتون. وفي هذه الدراسة نتناول الطرق التقليدية وطرق الطرد المركزي والطرق التطبيقية و الطرق البديلة لمعالجة مياه المخلفات بالمعاملات الفيزيوكيميائية. وقد وضحت النتائج أن نسبة (المواد الصلبة الذائبة الكلية و المواد المتطايرة و المواد الثابتة و الأكسجين الكيميائي المستهلك و الأكسجين الحيوي المستهلك بعد خمسة أيام) أعلي كثيراً في طرق الطرد المركزي عن الطرق التقليدية، لكن قيمة الفينولات أعلي قليلا في طرق الطرد المركزي عن التقليدية، و أن الكربون النشط حقق نسبة إزالة أعلي في (الفينولات و المواد الصلبة الذائبة الكلية و المواد المتطايرة و المواد الثابتة و الأكسجين الكيميائي و الأكسجين الحيوي) ورتبت هذه النسب في ترتيب تنازلي (٦٠ - ٥٠ - ٥٠ - ٤٣ - ٢٢%) علي التوالي. وفي حالة معاملة (هيدروكسيد الصوديوم + كربونات الصوديوم) بنسبة ١:٢ كانت نسبة إزالة (الفينولات و المواد الصلبة الذائبة و المواد المتطايرة و المواد الثابتة و الأكسجين الحيوي و الأكسجين الكيميائي) كانت عالية جدا ورتبت النتائج في ترتيب تنازلي (٦٠ - ٤٠ - ٤٠ - ٣٠ - ٢٥%) علي التوالي. وعندما كانت نسبة (هيدروكسيد الصوديوم + كربونات الصوديوم) بنسبة ٣ : ١ حققت نسبة إزالة أعلي في (الفينولات و المواد الصلبة الذائبة و المواد المتطايرة و المواد الثابتة و الأكسجين الحيوي و الأكسجين الكيميائي) و رتبت النتائج في ترتيب تنازلي (٧٥ - ٥٠ - ٥٠ - ٤٧ - ٤٠%) علي التوالي. وفي معاملة أخرى كانت كبريتات الحديدوز مؤثرة في خفض نسبة (المواد الصلبة الذائبة و المواد المتطايرة و الفينولات) عن كلوريد الحديد ورتبت النتائج في ترتيب تنازلي (٤٠ - ٤٠ - ٣٠%) علي التوالي. بينما كانت معاملة كلوريد الحديد مؤثرة في خفض نسبة (المواد الثابتة و الأكسجين الكيميائي و الأكسجين الحيوي) ورتبت النتائج في ترتيب تنازلي (٦٨ - ٥٠ - ٣٠%) علي التوالي. عند زيادة تركيز فوق أكسيد الهيدروجين ل ٥ مل عند ٢,٥ جم من كبريتات الحديدوز حسنت إزالة المواد العضوية غير أن الإفراط في كمية المادة المؤكسدة ل ١٠ مل ليس له تأثير عكسي وذلك لأن فوق أكسيد الهيدروجين يمتلك شقوق إزالة. إنه أصبح لأهميه أن الهدف القومي والعالمي للتحكم في تلوث المياه هو التخلص من الملوثات الملقاة في هذه المياه، أيضا التكنولوجيا الصناعية وجدت أن الأفضل اقتصادياً معالجة مياه النفايات لإعادة استخدامها بدلاً من تصريفها في البيئة.