



Integrated Process for the Treatment of Olive Oil Mill Waste Water (OMW)

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Abstract In this paper a description of a new integrated process for treatment of contaminants of olive oil mill waste water (OMW) under atmospheric pressure and room temperature. This process involves two main stages. The first stage include pre-ozonation of the raw OMW and primary sedimentation process, followed by electrocoagulation using electrolytic cell supplied with an Iron polls. Then suspended lime solution was added and the solids separated by settling in a precipitating tank. The optimum conditions for the first stage such as pH, current intensity, polls metal species and lime quantity were defined. This stage reduces mainly 58.7 % of COD, 86 % of total phenols, 69.9 % of color and 90.9 % of TSS. In the second stage the resulted (OMW) sample driven to an ozone oxidation unit. The oxidation unit include pre-ozonation reactor followed by oxidation process in a Plexiglass rectorin counter verse currents gas liquid phase, then the remaining contaminants was oxidized in advanced catalytic ozonation reactor filled with the catalyst, followed by adsorption on activated carbon column, and the clean disposable water was collected in a storage tank. Optimum conditions for the second stage such as time, ozone dose, pH value and the best catalytic system were determined. Using this integrated wastewater treatment process, OMW can be treated economically and the majority of parameters such as, COD, phenolic compounds, color, turbidity, and TSS can be removed up to 96-100 % using the best studied catalyst systems (Fe₂O₃+CuO/Clay).The treatment cost of (OMW), is clearly less than any other known and published methods.

The obtained treated clear OMW is appropriate to the sewage and agriculture discharge standards declared of Water Pollution Control Regulations based on Environment European EEQs and specifications of UTN and the Syrian Environmental Law No: 50/2002. The suggested integrated process and unit is small in size, and can be installed and carried in a moving vehicle and can be transferred from mill to another. It is suitable for a small, medium and large mills, in particular those geographically faraway dispersed.

Keywords Olive Mill Wastewater; Electrocoagulation; Catalytic Ozonation; AOPs, Ozone, Catalyst

1. Introduction

Mediterranean countries are the main producers of olive oil in the world, they contribute about 95-97 % of the global production, and as a result, an enormous volumes of waste water of olive processing are produced ranging between (10-30 million m³) every season [1,2]. OMW is the most dangerous waste for the environment. There is an archeological evidence of the effects of this waste has damaged environments over the Mediterranean coast for thousands of years [3,4]. OMW disposal created economical and technological challenge being seasonal process and due to large geographical spread of the mills and to its high content of organic matter and high concentrations of toxic phenols and multi-phenols [4,5]. Therefore global legislation in these countries prevent direct disposal of



(OMW) in rivers, lakes and valleys [6]. European Union produces 80.2 % of global production of olive oil, but the technology of treating (OMW) is still unsatisfactory [3]. The actual plan of European Union is to move from classical methods of OMW treatment by evaporation of the water in an open pools to suitable solutions as substitute complying with European new legislation EEQs [7] and specifications of UTN [6], which helps to create a friendly environment. Syrian government has issued the Environmental Law No. 50 of 2002 and the Law of Cleanliness No. 49 of 2004 which prevented disposal of (OMW) in water streams or agricultural land [8]. Resolution 190 issued by the Syrian Minister of Agriculture in 2007 allowed the spraying of OMW on the soil at the same rates and conditions allowed by Italian law No. (574/96). Since small and medium olive presses do not have the physical capability to deal with (OMW), the stresses of environmental law will lead presses to close.

Treatment studies of olive mill waste water are continuing all over the world, but none of the published technical studies in the literature allows a comprehensive, effective, and economical process to provide standards discharge for (OMW) [9].

Abbreviations:

COD= chemical oxygen demand; EC= electrocoagulation; OMW= olive mill wastewater; TSS= total suspended solids; TOC= total organic carbon; TPh= Total Phenols; TN_b= Total Nitrogen Bounded; pH= Acidity degree; OMW_{EC+Ca}= Electrocoagulation + Lime; OMW_{O₃+EC+Ca}= preozonation +Electrocoagulation +lime; λ= Specific electrical conductivity; AOPs= Advanced Oxidation Processes.

The problem of OMW disposal, to a large extent, has been drastic due to poor coordination among the producers of olive oil. Every country has its own legislation and regulations. There is a need for a unified strategy among the producers of olive oil in the world. It is therefore necessary to develop effective, and economic processes to dismantle the contaminants. For example, systems based on the generation of highly reactive oxidizing free radicals, called Advanced Oxidation Processes (AOPs) are adequate [10,17].

In previous studies, ozone was used in the treatment of (OMW) as a step prior to the stage of biological treatment to make (OMW) more susceptible to disintegration [9]. Recent studies in the framework of cooperation between the European Union countries have developed catalytic oxidation technology using hydrogen peroxide as an oxidant in addition to heterogeneous catalyst of iron and copper loaded on zeolite. The results showed that TOC is reduced by 22%, and multi-phenols by 48% after four hours of reaction at 90 °C [10,11].

Free radicals are effective in the treatment of (OMW) process and produced via meeting of both ozone and hydrogen peroxide [11,17], or at presence of solid heterogeneous catalyst or hydroxyl ions, therefore degradation of almost total certain persistent stubborn pollutants, which can be found in (OMW) are ensured by this mechanism. The most important acids are parahydroxy benzoic acid, phenolic acids, caffeic acid and ferulic acid [8].

Electrolytic treatment was used also to reduce organic substances and phenols and to ensure coagulation of fine particles [2,14,15].

The use of solid catalysts in advanced oxidation processes enhanced reactions rate due to adsorption of organic pollutants on its surface and increase the concentration of radicals at the surface, leading to the creation of hydrophobic-phylic property which generates a local booster increasing reaction rates [13,18].

2. Aim of the Study

The study aimed to create a new technique for complete treatment of (OMW) and to reduce the load of organic matter to values that allow it to be disposed to agricultural lands, lakes or rivers, or to be re-used. The results obtained shows that the specifications of the treated and produced water by this method at ordinary temperature and pressure are compatible with the standards of current discharge of European and Syrian criteria. The suggested integrated process and unit is small in size, and can be installed and carried in a mobile vehicle and can be transferred from mill to another. It is suitable for a small, medium and large mills, in particular those geographically faraway dispersed. The cost of operation and treatment of suggested technological process of (OMW), is far cheaper than any other published or known methods.



3. Experimental

An integrated technological process was designed for complete treatment of OMW at atmospheric pressure and ordinary temperature, and as a result of many experiments, tests and experience. The best results obtained which includes two main stages and several steps in each will be presented:

Table 1: Characteristics of OMW resulting from the hydraulic olive presses

Indicator	Sample Wash water	Overflow water	Water of olive press	OMW _{raw}	Units
COD	12	129	145	60	g /L
Total phenols	0.522	6.49	9.7	2.26	g/L
TOC	2.20	25.95	22.40	14.90	g/L
pH	5.93	4.81	4.80	5.74	-
λ	3.26	19.08	17.98	8.88	mS/cm

3.1 Samples of black OMW_{raw} were collected from storage tank in a modern hydraulic olive press and most spread units at season time, in Homs city-Syria. This sample represent three added main streams of olive press(water resulting from the sap of olives, effluent from the overflow tank, and water resulting from olives washing). Also samples of each stream was collected too. The results of OMW analysis shown in table(1).

3.2 In the first stage

samples of OMW_{raw} were treated by physio-chemical process, which include:

3.2.1 Settling of suspended solid particles in a primary sedimentation tank and pre-ozonation process using recycled ozone gas remaining in the gaseous mixture outlet of ozonation unit.

3.2.2 Contaminant decomposition and electrocoagulation process using laboratory electrolytic cell supplied with four Iron electrodes and designated (OMW_{O₃+EC}) as shown in fig(1).

3.2.3 Lime treatment of (OMW_{O₃+EC}) in a clarifier tank by adding suspended lime solution to adjust pH to a value (11.0-11.5), and the formed sludge's was precipitates and falls down to the bottom of the clarifier where it's drown out, fig(2).

3.2.4 The transparent overflow water of the clarifier was transferred and deposited in a storage tank and designated (OMW_{O₃+EC+Ca}). The main parameters of this resulted water were determined and shown in tables(2, 3).

3.3 In the second stage

The treated (OMW_{O₃+EC+Ca})sample was transferred to an advanced ozonation unit which include several steps as shown in fig(3) and as followings:

3.3.1 Pre-ozonation in a plexiglass tube using surpluses of ozone coming from ozonation reactor outlet.

3.3.2 Advanced oxidation process of pre-ozonated sample (OMW_{O₃+EC+Ca})were carried out in two reactors, the first one is ozonation reactor(bubbling column) using ozone gas coming from the ozone generator (total height 2 m; internal diameter 6 cm), the second one is catalytic fixed bed reactor (the bed height 50 cm, and diameter 3.5 cm).Several heterogeneous catalytic systems were prepared and tested, and we showed only the results of the best one which is (Fe₂O₃+CuO/Clay). Also solid suspended fluidized titanium oxide catalyst in powder form was used also(Merck, TiO₂ 2.5 g/L) at high pH for comparison.

3.3.3 The oxidation was carried out in semi-batch mode with recirculation of the OMW_(O₃+EC+Ca), i.e. the water was circulated over the reactor, while fresh ozone containing oxygen stream was continuously fed into the bottom of ozonation reactor. Both gas and treated OMW were fed into the ozonation reactor in co-current up flow. It is important to consider that during catalytic ozonation processes the pH of reaction medium decreases due to formation of acidic species. Therefore, it is necessary to adjust the pH of the treated water sample to a value around 9-10 which enables the achievement of the highest efficiency of contaminants abatement, and this done using adequate quantity of sodium hydroxide solution. The main parameters in this stage was determined and are shown in table (5).



3.3.4 Remaining organic and phenolic compounds were adsorbed from water on Merck activated charcoal powder in a liquid fluidized bed reactor.

3.3.5 Samples were taken during the hole process, and all necessary analysis and tests were realized according to a modern and standard methods. TOC analysis were performed using carbon analyzer, which based on infrared gas analyzer (Liqui TOC Elementar). All important figures and results are illustrated in tables (1,2,3 and 5).

3.3.6

3.4 Electrolytic cell treatment of OMW_{Raw}

A sample of pre-ozonated OMW_{Raw} has been exposed to electrolytic treatment using a cell designed in our laboratory and supplied with four iron plates as electrodes, as shown in fig(1). The elemental composition of the iron electrodes had been determined by means of a Spectrometer (Type ICP-Optical Emission Spectrometry), and the results were as follows: (Fe 95.3%; Al 2.3%; Cr 0.17%; Mn 1.87%; C 0.25%; Si 0.08wt.%). The electrolytic cell process was used to decompose contaminant substances and for coagulation of small solid particles which enhanced by bubbling the surpluses of ozone outlet of catalytic ozonation unit. The electrically treated water was driven to a settling tank to separate solid precipitates and the transparent water over flow was taken to further treatments. In another experiments lime $Ca(OH)_2$ as suspension was added to adjust the pH to a value of (11-11.5) using digital pH-meter, and to precipitate formed salts during electrical coagulation process, inclusive formed undissolved ferric and ferrous hydroxides. The transparent water was driven to an advanced catalytic ozonation unit. Table (2) summarizes the properties of OMW_{Raw} , and treated OMW by EC, lime, and EC+Ca .

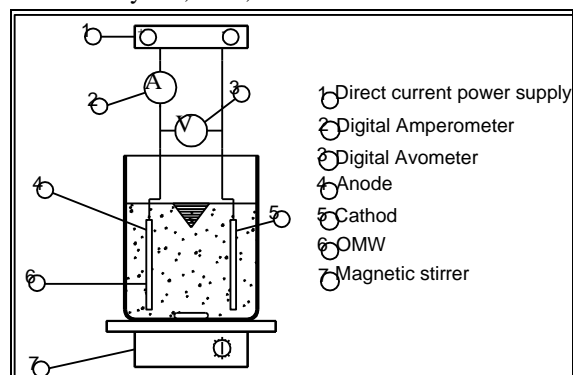


Figure 1: A Schematic diagram of Electrolytic Cell

Table 2: Characteristics of OMW_{Raw} and treated OMW (EC= electrocoagulated, and EC+Ca = electrocoagulated + precipitated with lime, and precipitated with lime only).

Parameter	Treatment methods of OMW				Units
	OMW_{Raw}	EC	$Ca(OH)_2$	EC + $Ca(OH)_2$	
λ	8.88	12.94	12.80	11.33	mS/cm
pH	5.74	8.27	11.90	11.50	-
Fe	29.00	254.00	-	12.30	mg Fe/L
COD	60.00	49.00	46.40	30.30	g/L
COD _{remov.}	-	11.00	13.60	29.70	g/L
COD _{conv.}	-	18.33	22.66	49.50	%
TPh.	2.44	2.38	0.88	0.92	g/L
TPh _{conv.}	-	2.37	64.02	62.35	%
TN _b	1.14	0.71	-	0.26	g TN _b /L
TSS _{conv.}	-	42	-	78.13	g/L
Color x100	265	465	-	105	Pt Co
TOC	14.90	10.31	13.10	7.74	g/L
TOC _{conv.}	-	30.81	12.00	48.08	%



Table 3: Characteristics of OMW in 4 cases (raw; pre-ozonated; pre-ozonated + electrocoagulated; and pre-ozonated + electrocoagulated +precipitated with lime).

Water Sample	pH	COD	COD _{Conv.}	TPh.	TPh _{conv.}	TSS	TSS _{conv.}	Color	Color Abatement %
		g/L	%	g/L	%	g/L	%		
OMW _{Raw}	5.50	60.00	-	2.82	-	3.30	-	40600	-
OMW _{OutO3}	4.90	51.00	15.00	1.65	41.49	2.90	12.12	28400	30.05
OMW _{OutO3+EC}	7.40	42.12	29.80	2.16	23.50	4.80	-	46500	-
OMW _{OutO3+EC+Ca}	11.80	24.80	58.70	0.40	86.00	0.30	90.9	12200	69.90

3.5 Catalysts preparation

Several types of catalyst were prepared, studied and characterized and used in this research mainly metal oxides deposited on different supports, such as clay, bentonite, and kaoline. Also pure titanium oxide powder was used as catalyst.

The clay was brought from Aleppo northern city in Syria and we found it as the best material to be used in this research, and it is well known as adsorbent materials and mainly composed of alumo-silicate and some calcium carbonate beside some impurities (table 4). The clay samples were finely powdered and then washed by distilled water and masticated with some dilute HCl and formulated as extrudate (rods or pillared), then let to be dried at room for 24 hours, hence dried at 105 °C. The samples was calcined in an electric furnace according to a programmed temperature which raised in the rate (0.5°C /min.) up to a 700 °C and maintained for 3 hours, then cooled by the same rate to room temperature, and then used as catalyst support. It is chemical composition shown in table (4).

The catalyst was prepared by loading the clay support with metals salt using wet impregnation process by (utilising) appropriate concentrations of metal ions in an aqueous solution of nitrate salts (Fe(NO₃)₃·9H₂O, Cu(NO₃)₂·3H₂O). After impregnation process was completed the catalyst was dried in an oven then followed by calcination in an electric furnace and the temperature was raised in the rate (0.2 °C /min.) to achieve at last 300 °C and holded for 2 hours, and then cooled in the same way, the metals is then present in a metal oxide form.

To determine textural and chemical composition of prepared catalyst, samples were degassed at 200 °C under a nitrogen atmosphere then BET surface area were determined using nitrogen adsorption/desorption analysis at a constant temperature(-196 °C), by Gemini Instrument (2370V5.01 model). Both catalysts TiO₂ powder and pillared clay samples was analyzed by x-ray instrument and patterns were obtained using a (Metorex 920 XRT-single sample probe). The main characteristics of the support and prepared catalyst are showed in Table (4).

Table 4: Characteristics of the clay support and catalysts

Material	BET surface area m ² g ⁻¹	Composition %								
		Fe ₂ O ₃	TiO ₂	Al ₂ O ₃	K ₂ O	MgO	CaO	SiO ₂	CuO	P ₂ O ₅
Clay,Support	207.7	10.1	3.45	14.67	0.12	0.67	2.87	51.36	0.89	0.90
(Fe ₂ O ₃ +CuO)/Clay	209.95	11.2	3.66	15.2	0.15	0.58	2.43	51.9	1.47	0.92
TiO ₂ powder	530.2	-	100	-	-	-	-	-	-	-

3.6 Ozonation pilot plant

Experiments were carried out at atmospheric pressure and ordinary temperature, using laboratory pilot plant shown in figure (2), which operate almost in semi-batch system. The OMW_{Raw} collected in storage tank (1) and pumped to a primary settling tank where small pieces of solid sediments separated at the bottom of the tank (2). The overflow water driven to an electrolytic cell(4) supplied with four Iron electrodes (3) and DC current was applied, where chemical changes took place and small fine solid particles coagulated. The water flows to a clarifier (5), and lime solution was added to adjust and control the pH around the value (11-11.5), where calcium carbonate was formed,



and then the water overflows and driven from the clarifier to a trap to separate possible small and fine particles and the water then stored in a storage tank (7).

Ozone was produced by a laboratory generator (20) (CFS-2G, serial number CH-CFS32G1146) at a rate $0.18 \text{ m}^3/\text{h}$ which equal 20.22 g/h of ozone, via electric discharge of oxygen (Purity 99.0 %) supplied from compressed oxygen cylinder(18).

The ozone gas flow was controlled and measured and injected via a porous glass plate (23) located at the bottom of the plexiglass ozonation reactor (12), which enabled small bubbles to be produced. The outlet at the top of ozonation reactor allowed excess gas to flow to the bottom of the pre-ozonation plexiglass reactor (9) and bubbled in counter verse currents of contaminated water ($\text{OMW}_{(\text{O}_3+\text{EC}+\text{Ca})}$) entered at the top of the reactor which supplied from storage (7).

The ozonated water in the outlet at the bottom of reactor (12) pumped to the top of the advanced fixed bed catalytic ozonation reactor (15), then driven to the top of the adsorption reactor charged with charcoal in a fluidized bed column (10 g/L), and then driven to treated clean water storage tank (17).

Unconsumed gaseous mixture in pre-ozonation reactor column (9) flows from the top of the column (9) to the atmosphere after passing through KI solution (25). The pilot plant were equipped with all necessary measurement tools, valves and facilities. Samples were taken and tested periodically during the reaction process, and it is main properties were determined according to standard methods of analysis.

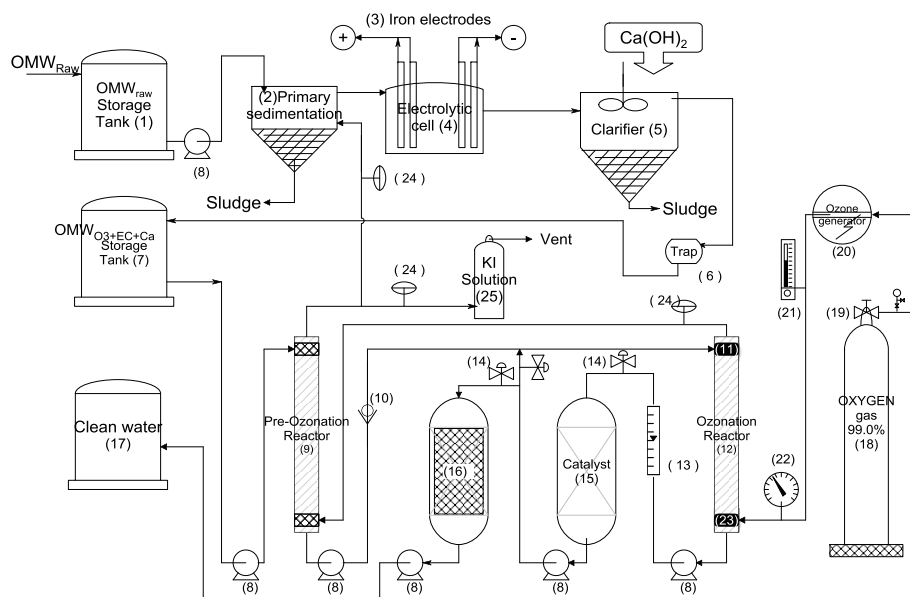


Figure 2: Integrated process pilot plant for the treatment of olive oil mill waste water

1- OMW_{Raw} Storage Tank; 2- Iron electrodes; 3- Primary sedimentation tank; 4- Electrolytic cell; 5- Clarifier; 6-Trap; 7- $\text{OMW}_{\text{O}_3+\text{EC}+\text{Ca}}$ Storage Tank; 8- Pumps; 9- Pre-ozonation glass reactor; 10- Non return valve; 11- Distributer; 12- Ozonation Plexiglass reactor; 13- Flow meter; 14- valves; 15- Catalytic ozonation reactor 16- Activated carbon column; 17- Clean water tank; 18- Oxygen gas cylinder; 19-Needle valve; 20- Ozone generator; 21- Gas Flow meter; 22-Gas counter; 23- Porous diffuser; 24- Diaphragm control gas ; 25- KI solution.



Table 5: Characteristics of $OMW_{O_3+EC+Ca}$ after treatment with $(O_3/(Fe_2O_3+CuO)/Clay)$ catalyst

t min	$(O_3/(Fe_2O_3+CuO)/Clay)$ catalyst									
	COD g/L	COD _{Conv.}	COD _{Ads.} g/L	COD _{Ads.} %	TPh. g/L	TPh _{Conv.} %	TPh _{Ads.} g/L	TPh _{Ads.} %	g O _{3in} /ΔCOD	Cost \$/m ³
0	20.60	-	12.00	41.70	0.37	-	0.08	78.4	-	-
25	12.60	38.80	2.40	88.30	0.04	89.70	0.00	100	0.42	2.40
50	5.40	73.80	0.80	96.00	0.0142	96.70	0.00	100	0.39	4.80
100	1.80	91.3	0.08	99.60	0.00	100.00	0.00	100	0.67	9.60

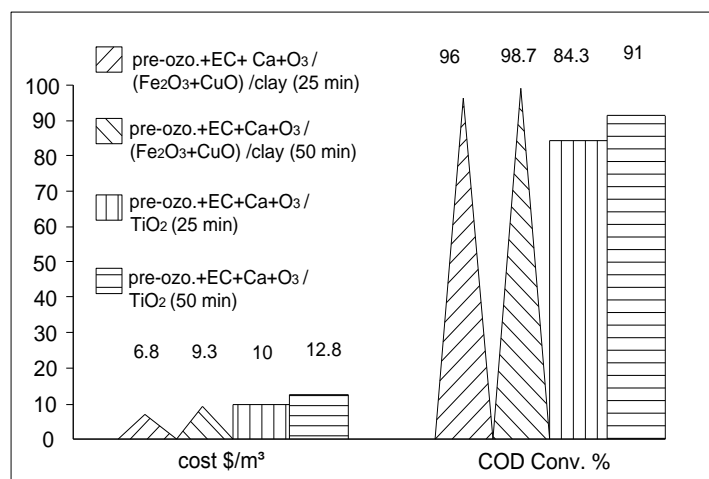


Figure 3: Comparison between COD conversion (%) and the cost of treatment (US\$) using Integrated process for the best different treatment systems

5. Conclusions

5.1 The suggested integrated process shows efficient and economic method for treating of olive mill contaminated water and shows several advantages in comparison with other known process and it is a result of our laboratory study.

5.2 Two steps of treatments were necessary to obtain disposable water. The first step includes pre-ozonation of OMW_{Raw} and sediments separation followed by electrolytic cell coagulation and treatment followed by lime suspension treatment, this stage reduces mainly 58.7 % of COD, 86 % of total phenols, 69.9 % of color and 90.9 % of TSS (table 3). The second step includes advanced oxidation of $OMW_{O_3+EC+Ca}$ using $(Fe_2O_3+CuO)/Clay$ catalyst systems at 25 min followed by adsorption on charcoal, this stage reduces 96 % of COD, 100 % of total phenols and color and TSS (table 5).

5.3 Treated water by this method have the standard specification which allow it to be disposed to public network or used in irrigation, according to the specifications defined by Environment European Quality EEQs [7], and specifications of UTN [6], and Syrian standards, (the standard value COD=2 g/L[8]).

5.4 low operating cost which equal to (6.8\$/m³) which is quite low compared with any other similar process[19].

5.5 Pre-treatment of raw OMW_{Raw} by surplus of ozone (side product in catalytic ozonation unit), shows effective influence in EC process and contaminants decomposition and surplus ozone consumption[20,21].

5.6 Lime precipitation reduces the concentration of TSS and turbidity at high rates, and reduces the total phenols, color, and doubled the reduction percentage of COD and raise the pH to desired value in order to implement ozonation process without additional complications and cost. More than 64% of the total phenols in

sludge's were separated, and this provides an opportunity to recover these compounds in an easy way to take advantages of them in several areas.

5.7 Electrolytic cell treatment was a necessary step to eliminate some contaminants and enable colloidal particles to be coagulated and easily precipitated [18,22,23].

5.8 The prepared catalyst in our laboratory ($O_3/(Fe_2O_3+CuO)/Clay$) and tested in proposed integrated process, was more active and the operating cost is farless at all conditions compared with TiO_2 catalyst[9].

5.9 The suggested integrated process and unit is small in size, and can be installed and carried in a moving vehicle and can be transferred from mill to another. It is suitable for a small, medium and large mills, in particular those geographically faraway dispersed.

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