

Subject Area 5.1: Technologies supporting waste disposal and remediation of hazardous wastes

Research Article

Decolourization and Removal of Some Organic Compounds from Olive Mill Wastewater by Advanced Oxidation Processes and Lime Treatment

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Abstract

Background. Olive mill wastewater (OMW) generated by the olive oil extracting industry is a major pollutant, because of its high organic load and phytotoxic and antibacterial phenolic compounds which resist biological degradation. Mediterranean countries are mostly affected by this serious environmental problem since they are responsible for 95% of the worldwide olive-oil production. There are many methods used for OMW treatment, such as adsorption, electro coagulation, electro-oxidation, biological degradation, advanced oxidation processes (AOPs), chemical coagulation, flocculation, filtration, lagoons of evaporation and burning systems, etc. Currently, there is no such economical and easy solution. The aim of this study was to evaluate the feasibility of decolourization and removal of phenol, lignin, TOC and TIC in OMW by UV/H₂O₂ (AOPs). The operating parameters, such as hydrogen peroxide dosage, times, pH, effect of UV and natural sunlight were determined to find the suitable operating conditions for the best removal. Moreover, there is no study reported in the literature related to the use of UV/H₂O₂ and lime together in OMW treatment.

Methods. OMW was obtained from an olive-oil producing plant (Muğla area of Turkey) which uses a modern production process. No chemical additives are used during olive oil production. This study was realised by using two different UV sources, while taking the time and energy consumption into consideration. These two sources were mercury lamps and natural sunlight. Before starting AOPs experiments, one litre of OMW was treated by adding lime until a pH of 7.00. Then, 100 ml was taken from each sample, and 1 to 10 ml of a 30% H₂O₂ (Riedel-deHaen) solution was added. These solutions in closed vessels were laid in the natural sunlight for a week and their compositions and colour changes were analysed daily by UV-Vis spectrophotometer. At the end of the one-week period, they were treated with lime. In this study, the effect of changes in the initial pH, times and H₂O₂ concentrations on removal was investigated. At the end of all experiments, changes in colour, phenol, lignin, TOC and TIC concentrations were analysed according to standard methods.

Results and Discussion. In the samples exposed to natural sunlight and having an H₂O₂/OMW ratio of 3 ml/100 ml, a significant colour removal was achieved approximately 90% of the time at the end of 7 days. When the same samples were treated with lime (pH: up to 7), 99% efficiency was achieved. When phenol and lignin removals were examined in the same concen-

tration, phenol and lignin removal were found 99.5%, 35%, respectively. However, for maximum lignin removal, more use of H₂O₂ (10 ml H₂O₂/100 ml OMW) was found to be necessary. Under these conditions, it was found that lignin can be removed by 70%, but to 90% with lime, at the end of a seven-day period. Rate constants obtained in the experiments performed with direct UV were found to be much higher than those of the samples exposed to natural sunlight ($k^a_{\text{lignin}}=0.3883>>k^b_{\text{lignin}}=0.0078$; $k^a_{\text{phenol}}=0.5187>>k^b_{\text{phenol}}=0.0146$). Moreover, it should be remembered in this process that energy consumption may induce extra financial burden for organisations.

Conclusions. It was found, in general, that colour, lignin, total organic carbon and phenol were removed more efficiently from OMW by using H₂O₂ UV and lime OMW. Moreover, in the study, lime was found to contribute, both initially and after radical reactions, to the efficiency to a great extent.

Recommendations and Perspectives. Another result obtained from the study is that pre-purification carried out with hydrogen peroxide and lime may constitute an important step for further purification processes such as adsorption, membrane processes, etc.

Keywords: Advanced oxidation processes (AOPs); colour; lignin; lime treatment; olive mill wastewater (OMW); phenols; total inorganic carbon (TIC); total organic carbon (TOC)

Introduction

Olive oil is extracted mainly in two ways, by traditional (classical, pressing) and continuous (centrifuging) methods from the pulp of olive fruits obtained by grinding them in stone mills. In the traditional method, the ground olives are pressed in cloth bags and then the liquid mixer is rested in a series of tanks to separate the oil. In the continuous method, the crushed olive fruits are pumped into a three-phase decanter and the impure oil is then centrifuged (İsrailised et al. 1997, Adhoum and Monser 2004).

Olive mill wastewater (OMW) generated by the olive oil extracting industry is a major pollutant because of its high organic load and the phytotoxic and antibacterial phenolic compounds which resist biological degradation. Mediterranean countries are mostly affected by this serious environmental problem, since they are responsible for 95% of the worldwide olive-oil production. In these countries, about 11 million tons of olives are produced per year from which about 1.7 million tons of olive oil is extracted. The seasonal polluting load of olive-oil production is nearly equal to that of

22 million people per year (Saez et al. 1992, Kestiölu et al. 2004, Zanichelli et al. 2006). Therefore, OMW must be treated to remove the phenolic fraction, before being discharged in receiving water bodies or used for irrigation purposes.

The simplest solution applied towards this direction consisted in constructing artificial big ponds into which OMW is stored, awaiting its natural evaporation. However, this method, not only being very slow, but also causes subsequent unpleasant environmental pollution linked to generation of bad odours due to anaerobic activity (Adhoum and Monser 2004).

There are many methods used for OMW treatment, such as that proposed by Kestiölu et al. (2004) who studied the physico-chemical treatment and advanced oxidation processes by means of the ozone or Fenton's reagent in the presence and absence of UV radiation. They showed that the same COD and total phenol removal efficiencies (99% removal for both COD and total phenol) were found to have been given by both H_2O_2/UV and O_3/UV combinations. Another method was recently applied to the treatment of OMW and consists of the application of an integrated centrifugation-ultra filtration system (Turano et al. 2002) allowing an efficient reduction of pollution and a selective separation of some useful product. Traditional physical and chemical techniques, such as flocculation, coagulation, filtration, lagoons of evaporation, the electrochemical treatment of OMW and burning systems also solve the problem, but only partially (İsrailised et al. 1997, Oukili et al. 2001, Schwitzguébel et al. 2002, İnan et al. 2004, Adhoum and Monser, 2004, Uğurlu et al. 2006). In addition, Oukili et al. (2001) have investigated activated clay as adsorbents for removal of organic compounds from OMW, the removal of phenolic compounds have also been investigated effectively by using lime (Aktaş et al. 2001). Curi et al. (1980) have tested the treatment of OMW with a mixture of aluminium sulfate and ferric chloride, calcium hydroxide solution and also acidifying of the waste with hydrochloric acid solution. They have determined the clarifying percent of the wastewater. Calcium hydroxide and aluminium sulphate have also been used besides magnesium sulphate by Tsonis et al. (1989). They have reported that COD value dropped to 20–30% with calcium hydroxide, when it was added until the pH of the waste reached 11. Several biological studies have also been conducted to eliminate the pollution effect of OMW (Hamdi et al. 1992) and the organic content of OMW was oxidized using monopersulfuric acid (Solinas et al. 1992). Lallai et al. (2003) have investigated the biodegradation of phenolic compounds by using aerobic microbial cultures. As a result, there is no such economical and easy solution for removal organic compounds from OMW.

Commonly applied method for removal of COD, colour, phenol and organic compounds from industrial effluents is Advanced Oxidation Processes (AOPs). AOPs are related to the formation of OH radicals, which will accelerate the oxidative degradation of numerous organic compounds dissolved in wastewater. It has been found that AOPs include several processes such as ultraviolet/ozone (UV/O_3), ultraviolet/hydrogen peroxide (UV/H_2O_2), and ozone/hydrogen peroxide (O_3/H_2O_2) (Ranalli 1987, Kestiöglu et al. 2004).

The UV/H_2O_2 process uses ultraviolet radiation to cleave the O-O bond in hydrogen peroxide and generate the hydroxyl radical (Hung et al. 2004, Glaze et al. 1987). The hydroxyl radical can then be scavenged by an organic compound to initiate a radical chain degradation of hydrogen peroxide as in the following reaction:



Hydroxyl radicals produced in either way described above may attack organic molecules by abstracting a hydrogen atom from the molecule (Glaze et al. 1987, Hung et al. 2004, Kestiöglu et al. 2004).

In the present study, it was aimed to investigate the decolourization and removal of some organic compounds (phenol, lignin, TOC and TIC) from OMW by using AOPs method. In addition, constant amount of lime was used for all the experiments after AOPs for increasing the removal efficiency and to prevent pH decreased. Moreover, there is no study reported in the literature related to the use of AOPs and lime together in the OMW treatment.

1 Materials and Methods

1.1 Characterization of the OMW

The wastewater under study was obtained from an olive-oil producing plant (Muğla area of Turkey), which uses a modern production process. No chemical additives are used during the olive oil production. The characteristics of olive mill wastewater used in this work are presented in Table 1.

Table 1: Physico-chemical characteristics of olive mill wastewaters sample

Parameters	Value
Colour	black
pH	5–6
COD ($mg\ l^{-1}$)	6.12×10^5
BOD ($mg\ l^{-1}$)	55.4
Polyphenol (mg/l)	8.0
Lignin (mg/l)	25.5
Density (25°C) (g/l)	0.96
Conductivity ($\mu S\ cm^{-1}$)	7065

1.2 Experimental methods

This study was realised by using two different UV sources, and taking the time and energy consumption into consideration. These two sources were mercury lamps (OSRAM Ultra Vitalux 300 W) and natural sunlight (day and night period, total 24 hours). Initially, one litre of OMW was treated by adding lime until the pH: 7.0. Then, 100 ml from each sample was taken into ten closed vessels to prevent evaporation and 1 to 10 ml of a 30% H_2O_2 (Riedel-de Haen) solution was added to these beakers. These solutions were laid in the sunlight (pane edge in the laboratory) for a week and their compositions and colour changes were daily observed and analyzed. At the end of the one week, they were treated with a

constant amount of lime in such a way that their pH value became 7.0. The operating parameters were selected: hydrogen peroxide dosage, time, pH, effect of UV lamp and natural sunlight (day and night). When only a mercury lamp was used as UV source, the time was limited to 5 hours. At the end of all experiments, changes in colour, phenol, lignin, TOC and TIC concentrations were analysed. All the experiments were performed in duplicate and average values were used.

1.3 TOC, TIC, phenol, lignin and decolourization measurement

The concentration of lignin, TOC, TIC, and phenol in OMW were measured using analysis methods described in APHA Standard Methods (APHA 1995). The dark colour intensity was determined by measuring the sample absorbance at 400 nm (UV-Vis spectrophotometer, Dr Lange).

2 Results and Discussion

2.1 Effect of initial hydrogen peroxide concentration

Much research has evaluated the decolourization of various organic compounds in aqueous solutions by UV/H₂O₂ processes, showing that the H₂O₂ concentration plays a very important role for the generation of hydroxyl radicals to remove organic compounds (Hy et al. 1994, Hy and Chang 2005). OMW was added and a particular amount of H₂O₂ was left under natural sunlight without being subjected to any treatment. The changes taking place in colour, phenol and lignin concentrations of all the samples were analysed daily and the results were plotted as seen in Fig. 1, 2 and 3, respectively.

OMW is strongly coloured related to lignin, tannin and the other high amount of organic compounds. This wastewater contains non-biodegradable product and is known to be a dark red to black effluent (Adhoum and Monser 2004). As

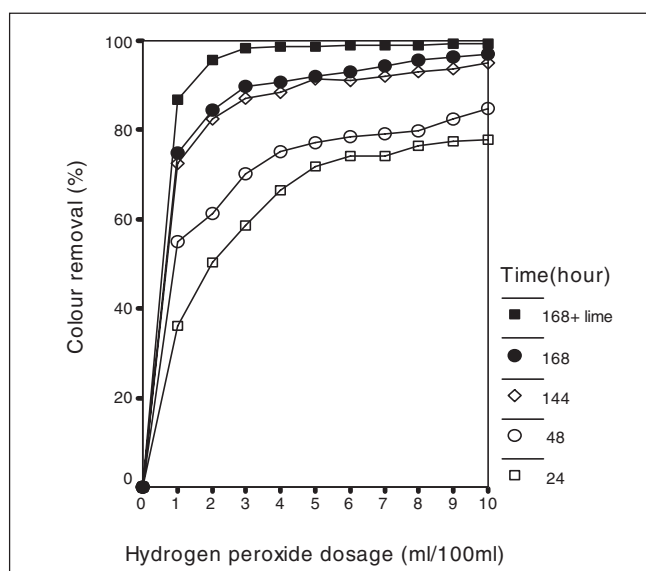


Fig. 1: Effect of H₂O₂ concentration on decolourization in different times and under natural sunlight (Experimental conditions: Temperature, 25°C; initial pH, 7.0)

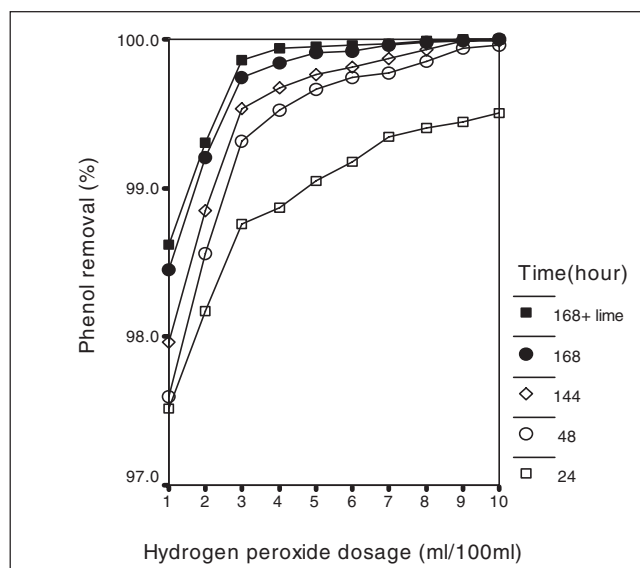


Fig. 2: Effect of H₂O₂ concentration on removal of phenol at different times and under natural sunlight (Experimental conditions: Temperature, 25°C; initial pH, 7.0)

seen in Fig 1, it was observed that significant colour removal was obtained at 3 ml H₂O₂/100 ml peroxide ratios and by adding lime treatment. Moreover, one week can be sufficient for this process. At the end of the seven-day period, colour removal reached 90% at this peroxide concentration and then, after adding lime, approximately 98%.

When Fig. 2 is analysed, it is seen that a fast phenol removal was achieved by increasing times and H₂O₂ concentration. After 3 ml of H₂O₂/100 ml, a plateau is seen in the removal percentage. Phenol removal was observed approximately 100% at the end of one-week by using lime. It was reported that in the process of black water purification performed with H₂O₂ and clay, polyphenols were removed to 95% with H₂O₂ and the use of H₂O₂ has two certain advantages (Oukili et al. 2001). These are:

- A decrease in pH from 7 to 5 because of the production of acetic acid from the polyphenol oxidation with H₂O₂
- An important bleaching of the undiluted OMW

Phenolic compounds were removed to a considerable extent in different H₂O₂ concentrations by increasing time. Moreover, 2 or 3 unit decreases were observed in the pH of wastewater. This result can be explained in the above-mentioned study; that is, decreases of pH have attributed increases in the number of acidic products. As explained before, in all the experiments, certain amounts of lime were used to make sure that the pH was 7.0. It is assumed that the addition of lime makes a great contribution to the neutralisation of acidic products, their sedimentation, and removal from the medium. In addition, the use of lime is of great importance in terms of bringing wastewaters compatible with the discharge standards.

While in low H₂O₂ concentrations, not much lignin removal was observed over time in connection with the increasing

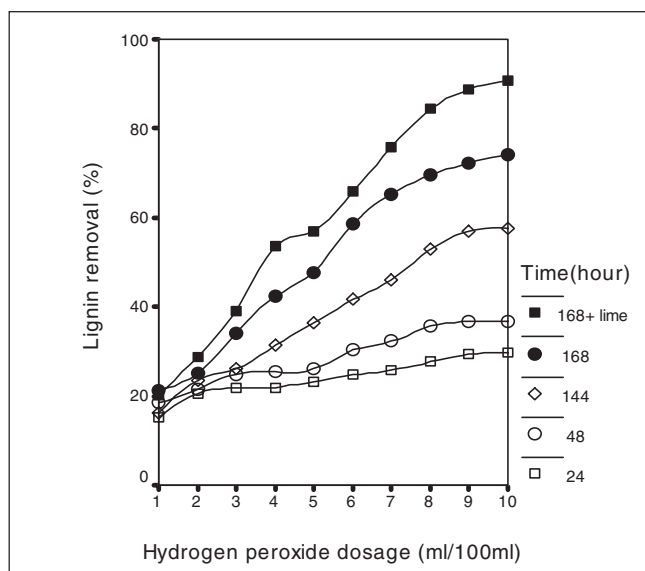


Fig. 3 Effect of H_2O_2 concentration on removal of lignin at different times and under natural sunlight (Experimental conditions: Temperature, 25°C; initial pH, 7.0)

peroxide amount, increases became very remarkable over time (see Fig. 3). Furthermore, it was seen that more lignin removal could be achieved when H_2O_2 treatment was followed by lime treatment. When Fig. 3 is analysed, it is seen for maximum lignin removal, that the use of a high amount of H_2O_2 could be necessary. When the ratio of H_2O_2 was 1/10, a 70% removal at the end of the seven-day period became 90% when lime was applied. All of the parameters, as can be seen later, the least efficiency was achieved with lignin. Lignin, which is a principal plant constituent and heterogeneous aromatic polymer interdispersed with hemicelluloses and occurs surrounding microfibrils. The effluent colour is primarily due to lignin and its degraded products, which are chemically stable, resistant to biological degradation and are intractable to separation by conventional treatment methods (Hy et al. 1994, Eismann et al. 1997). Even in very high peroxide concentrations, the fact that lignins were not being completely removed could be related to its being more resistant to radical reactions than phenol and other organic substances.

TOC and TIC values in olive black water are one of the most important indicators of pollution. When TOC and TIC concentrations are examined, it is seen, depending on an increasing peroxide concentration, which TOC decreased while the TIC concentration increased (Fig. 4). In OMW samples exposed to natural sunlight, TOC concentration ran out when 5 ml H_2O_2 /100 ml was used at the end of the 3-day period. Moreover, when H_2O_2 + lime were used, the TOC concentration at the end of this period was observed to reach its minimum level, while TIC was observed to reach its maximum (5.05 mg/l) (see Fig. 4). Solinas et al. (1992) was reported that there was a close connection between TOC change and H_2O_2 concentration. In the study, it was also reported that the removal of the organic matters can be directly related with H_2O_2 and OH radicals.

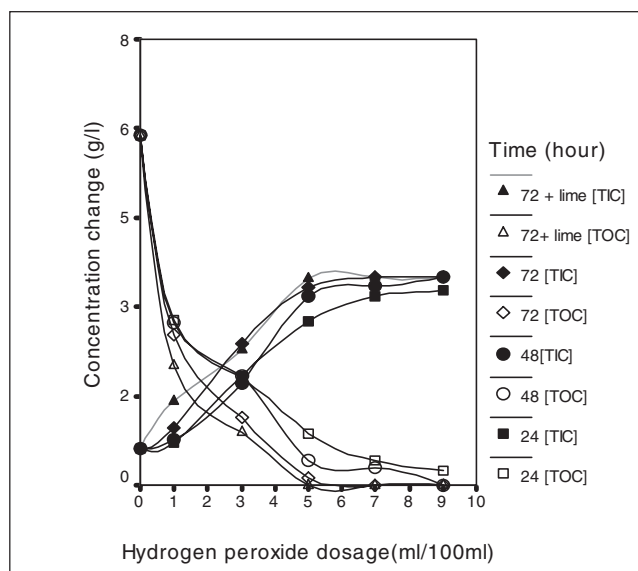


Fig. 4 Effect of H_2O_2 concentration on removal of TOC at different times (Experimental conditions: Temperature, 25°C; initial pH, 7.0)

2.2 The Direct effect of UV/ H_2O_2

In OMW samples directly exposed to UV/ H_2O_2 , the changes in phenol and lignin concentrations were investigated. The results obtained from this investigation were plotted in Fig. 5. It is seen that in the first 60 minutes, 80% phenol removal was achieved when UV+lime was used, but removal was increasing slowly by times. In addition, it can be seen that the use of lime made a great contribution to the removal of both compounds and at the end of a 3-hour period, while 100% phenol removal was achieved; for 90% removal of lignin, 4 hours were required.

In all the parameters investigated, more removal of phenol and lignin was observed with lime in the samples both exposed to natural sunlight and direct UV in laboratory con-

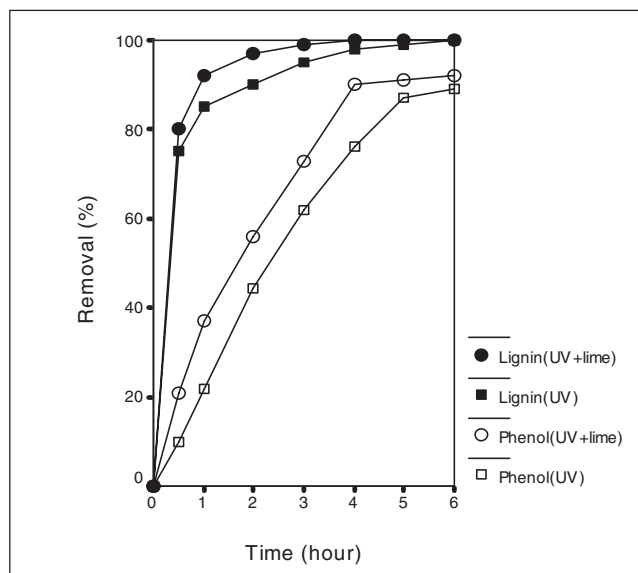
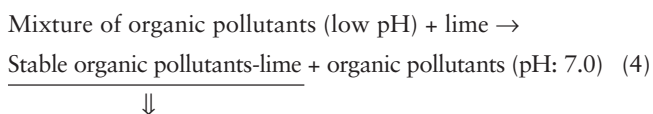


Fig. 5 Effect of time on removal of lignin and phenol (Experimental conditions: Temperature, 25°C; initial pH, 7.0; H_2O_2 /OMW ratio, 3 ml/100 ml)

ditions. Previous study proved that pure lime ($\text{Ca}(\text{OH})_2$) could effectively remove the colour, phenol and the other organic compounds in different wastewater (Aktaş et al. 2001). In another study, it was stated that in the experiments where UV and H_2O_2 are used together, possible reactions of organic substances in aquatic medium due to radical reactions can be as follows (Claze et al. 1987, Carey 1990, Keskinoglu et al. 2004):



In our work, 2 to 3 unit decreases were observed in the pH of wastewater (depending on H_2O_2). This situation can be explained through the emergence of the above-mentioned acidic products in aquatic medium. As stated before, a suitable amount of lime was used after all the experimental procedures (pH: 7.0). Acidic products appearing in Reaction 3 were predicted to have the following interaction with lime.



When direct UV was applied to wastewaters with different H_2O_2 concentrations, the obtained results were plotted in Fig. 6. for phenol and lignin removal. As expected, while the amount of H_2O_2 increased, the amount of removal increased as well. When 4 ml/100 ml and lime were used, about 98% phenol removal was achieved. In the same H_2O_2 concentration, lignin stability was observed when 90% removal was achieved. Moreover, in all the wastewater samples treated with H_2O_2 and lime, a significant amount of increase in the removal was achieved.

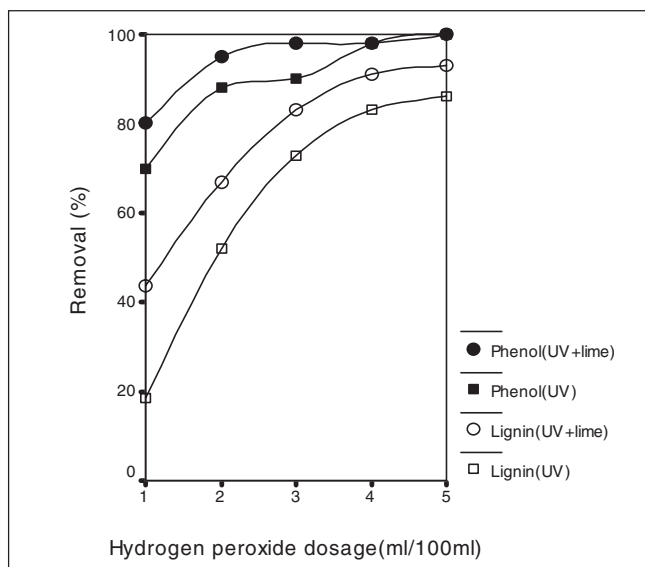


Fig. 6: Effect of initial dosage of H_2O_2 on removal of lignin and phenol (Experimental conditions: Temperature, 25°C; initial pH, 7.0; time, 2 hrs)

2.3 The effect of pH

In general, raising pH results in reducing organic molecule degradation rates by a UV/ H_2O_2 process so that H_2O_2 generally dissociates into water and oxygen rather than hydroxyl radicals under an alkaline condition (Ersoy et al. 1998). In the experiments, the initial suspension pH was adjusted to 1, 3, 5, 7, 9 or 11 by adding HCl and NaOH. Then, it was exposed directly to UV light by using constant $\text{H}_2\text{O}_2/\text{OMW}$ ratios (3 ml/100 ml) for 2 hours. At the end of this time period, the samples were taken and then they were treated with lime so that the pH was 7.0. Phenol and lignin removal values before and after lime treatment were plotted in Fig. 7. At very low and high pH values, removal efficiencies were low. The most suitable range of pH values for phenol and lignin removal was found to be between 6 and 9, and, at this range, 85% lignin and 95% phenol removal were achieved. In the same studies aiming to investigate the removal of dye molecules with UV/ H_2O_2 , the pH range of 7.2–8.9 has been reported to be very efficient (Hy and Chang 2005). In another study, at different pH ranges, TOC removal with the UV/ H_2O_2 system in dye substances has been analysed, and it has been reported that the maximum removal has been achieved in the pH range of 7–9. Again, in the same study, it has been stated that outside of the pH range of 3.3–10.6, the lowest TOC removals have been achieved (Solinas et al. 1992). Our study confirms the above-mentioned results, as the efficiency here was found to be low at very high and low pH values.

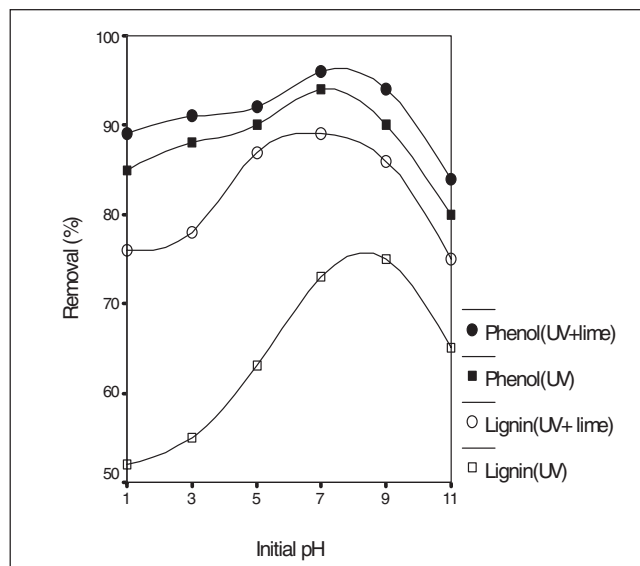


Fig. 7: Effect of initial pH on concentration of lignin and phenol (Experimental conditions: Temperature, 25°C; time, 120 min; $\text{H}_2\text{O}_2/\text{OMW}$ ratio, 3 ml/100 ml)

2.4 Kinetics of process

The decolourization, phenol, lignin, TOC and TIC were conducted using the H_2O_2 process under various operating conditions such as initial hydrogen peroxide concentration, natural sunlight and direct UV light power. In this process, UV and natural sunlight irradiates the hydrogen peroxide to produce the strongest oxidizing free radicals, such as hydroxyl and peroxide radicals, which attack the organics

Table 2: Summary of the kinetic and other properties of OMW by UV/H₂O₂ and sunlight (UV/H₂O₂) methods (Initial pH:7.0, H₂O₂/OMW:3 ml/100 ml, 25°C)

Compounds	UV / H ₂ O ₂		Natural sunlight / H ₂ O ₂	
	Rate constant, k^a (hour ⁻¹)	r^2	Rate constant, k^b (hour ⁻¹)	r^2
Lignin	0.3883	0.87	0.0078	0.91
Phenol	0.5187	0.90	0.0146	0.92
TOC	–	–	0.0058	0.91
TIC	–	–	0.0425	0.83

k^a = rate constant by using natural sunlight / H₂O₂

k^b = rate constant by using UV / H₂O₂

instantaneously as soon as the reaction starts to degrade the target compound. Besides, the decolourization rate expression of organic compounds can be simplified as a pseudo-first order kinetics model as follows (Ersoy et al. 1998, Uğurlu 2005).

$$\ln \frac{[C]_t}{[C]_0} = -kt \quad (5)$$

The slope of the plot of $\ln C_t/C_0$ versus time gives the value of rate constant k , min⁻¹. Here, C_0 is the initial concentration in milligrams per litre, and C_t is the concentration value in milligrams per litre at time t . The above equation may be of the type $y=mx$, and a line plot of $\ln C_t/C_0$ versus t indicates the validity of a first order reaction of process (Table 2) ($y= \ln C_t/C_0$, $m= -k$ and $x= t$). Table 2 provides the rate constant (k), respective r^2 values for phenol, lignin, TOC removal and TIC. When the rate constants presented in Table 2 are examined, rate constants in the experiments performed with UV were found to be higher than those of the samples exposed to natural sunlight ($k^a_{\text{lignin}}=0.3883 \gg k^b_{\text{lignin}}=0.0078$; $k^a_{\text{phenol}}=0.5187 \gg k^b_{\text{phenol}}=0.0146$). These results show, in the purification processes carried out by considering the seasonal and climatic factors, that the amount of production and other factors, natural sunlight may play an important role. However, a late emergence of its results is a disadvantage. On the other hand, it was found that to accelerate the procedure, direct UV and lime can be used, but that this induces extra financial burden due to the energy consumption involved in the procedure.

3 Conclusions

When OMW with different H₂O₂ concentrations was exposed to natural sunlight or UV, important changes were observed in colour, lignin, phenol, and TOC values. In the samples exposed to natural sunlight and having peroxide/wastewater ratios of 3 ml/100 ml, a significant rate of colour removal was achieved (90%). When the same samples were treated with lime (pH: up to 7), 99% efficiency was achieved. When phenol and lignin removals were examined in the same concentration, it was found that 90% phenol removal increased to 100% with lime and 30% lignin removal to 40%. In addition, when having peroxide/wastewater ratios of 10 ml/

100 ml, when phenol removal was examined in the same concentration, it was found that the same phenol removal of 100% was obtained for the cases with lime and without lime. However, for maximum lignin removal, more use of H₂O₂ (100 ml H₂O₂/1 L OMW) was found to be necessary. In these conditions, it was found that lignin can be removed 90% by using lime at the end of a seven-day period. On the other hand, in the experiments carried out under direct UV light, maximum lignin and phenol removal was achieved at the end of a five-hour period. However, when the wastewater obtained at the end of the fourth hour was adjusted in such a way with lime that the pH was 7.0, maximum efficiency was achieved (100% for phenol and lignin). Rate constants obtained in the experiments performed with direct UV were found to be much higher than those of the samples exposed to sunlight ($k^a_{\text{lignin}}=0.3883 \gg k^b_{\text{lignin}}=0.0078$; $k^a_{\text{phenol}}=0.5187 \gg k^b_{\text{phenol}}=0.0146$). However, it should be remembered in this process that energy consumption may induce extra financial burden for organisations. When the effect of pH on the removal was investigated, it was found, in general, that efficiency decreased at very low and high ranges. Moreover, in the study, lime was found to contribute, both initially and after radical reactions, to the efficiency to a great extent. Another result obtained from the study is that pre-purification carried out with hydrogen peroxide and lime may constitute an important step for further purification processes (adsorption, membrane processes, etc.).

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