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Removal of Phenol from Sewage Effluent Using Activated Sludge Coupled with Photo-oxidation Process

Salam K. Al-Dawery*, Sajjala Sreedhar Reddy, Khamis Al Riyami and Zainab Said Nasser

College of Engineering and Architecture, University of Nizwa, Nizwa, Sultanate of Oman

✉ salam@unizwa.edu.om

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Abstract: Treatment of hazardous phenolic substances that are discharged to environment has a significant interest since decades. Biological treatment using freely suspended sludge is the most economical treatment method, but, is considered inactive for treatment of phenolic substances due to the complexity in their chemical structure. The degradation of phenol was experimentally investigated using separate and coupled biological and photo-catalytic processes. The degradation was carried out at different concentrations of phenol from 10 to 100 ppm. The results showed no degradation of phenol using biological treatment, and as expected, the bacterial colony growth was reduced by 50 percent due to lack of nutrient. The results of photo-catalytic process using titanium dioxide (TiO_2) indicated the removal of 80 percent of phenol especially for concentration below 40 ppm. Bacterial colony growth during photo-oxidation test was dramatically reduced and no colony growth was found after 90 minutes after commencing time of experiment. On this basis, the results of using the combined advanced oxidation process with biological treatment, suggested that the photo-catalytic process would be considered as a post-treatment to the biological process.

Key words: Phenol degradation, photo-oxidation, wastewater, activated sludge.

Introduction

Phenol and phenolic derivatives are present in the effluents of many chemical process industries such as resins, plastics, paper manufacturing, petrochemicals, pharmaceutical, etc. (Kulkarni and Kaware, 2013; Sun et al., 2015). Phenol is considered one of the most toxic substances for human being and living organisms even at low concentration; the toxicity level of phenol for human and aquatic life is about 25 mg/l, for microorganism is 200 mg/l and the lethal concentration in blood is 150 mg/100 ml (Marrot et al., 2006; Villegas et al., 2006).

Many national and international bodies have set strict regulations for discharging solution with phenol directly into the municipal sewer systems in order to prevent untreated pollutants from being introduced into the biological treatment plants (EPA, 2014; NPRI Canada, 2017).

There are several processes which have been applied for the phenol removal and aromatic compounds from different industrial wastewaters such as adsorption using activated carbon, extraction, electrochemical and biological treatment, nano-filtration and reverse osmosis (Chung et al., 1998; Znad et al., 2006; Laoufi et al., 2008; Kulkarni and Kaware, 2013).

Many researchers have tried the treatment through enzymatic catalysis, while other researchers have used the isolation of special microorganisms from soil and waste materials (Klibanov et al., 1980; Karam and

*Corresponding Author

Nicell, 1997; Wilberg et al., 2000; Przyblewsk et al., 2006; Mohite et al., 2007; Farhngdoost and Tahery, 2010; Soudi and Kolahchi, 2010). Most of these methods were costly, and lack purification completeness.

Several researchers have investigated the biodegradation of phenol using activated sludge (Bevilaqua et al., 2002; Tziotzios et al., 2005; Marrot et al., 2006; Movahedyan et al., 2009). They studied the effect of adaptation of special bacteria and/or enzyme on phenol degradation. They observed that the use of adopted culture and enzymatic treatments have potentials for the phenol degradation up to concentration of 2.5 g/l.

Unfortunately, many synthetic and naturally occurring organic molecules are hardly biodegradable or non-biodegradable. A potentially viable treatment is the possibility of coupling advanced oxidation process (AOP) with a biological treatment using activated sludge. The activated sludge is the most common process for treating sewage wastewater. This process involves air being introduced into a mixture of wastewater combined with bacteria and protozoa to develop biological flocs which reduces the organic content of the sewage wastewater. In the first process the toxic and/or non-biodegradable compounds would be eliminated, and once the solution biodegradability improved, it can be fed to the biological treatment (Oller et al., 2007; Shanthini et al., 2011).

The technology of advanced oxidation process is considered as a promising and alternative method for treating on-biodegradable organic material. The photo-oxidation process promotes degradation of organic compounds when illuminated with UV light (Al-Dawery, 2013). Another objective of using photo-oxidation process is anti-bacterial growth for purification of water. Thus, due to the unique properties of AOP, many researchers have worked and attempted to enhance the performance of AOP using titanium and iron doping deposition with aid of co-adsorbent such as fiber glass and activated carbon (Yong et al., 2006; Arabzadeh et al., 2016; Al-Dawery, 2016).

Recent emerging technology of oxidation of aromatic compounds such as phenolic groups uses generated sulfate radicals by peroxymonosulfate and persulfate (Wang et al., 2014; Qi et al., 2017). Such method is complex and non-economical for application.

Previous research of coupling advanced oxidation process with a biological treatment and considered AOP as pretreatment to modify the structure of the pollutants by transforming them into more biodegradable intermediates and then using a biological treatment to complete the degradation of the pollutant load (Oller et al., 2007).

Such arrangement may not be effective due to the following three points: the influent to wastewater treatment plant usually consists of suspended materials resulting non-transparent liquid with higher solution turbidity which in turn reduces the transmission of UV light through the solution. The produced hydroxyl radical may pass into the biological process causing death of bacteria useful for organic degradation; as well known, the photo-catalytic process is considered as a strong disinfectant and able to decompose and remove the germs and toxins in water and wastewater.

The AOP process only generates antibacterial effect especially when exposed to UV light; the non-mineralized phenol may also pass into the biological process and exit without the necessary treatment.

The objective of this research work is to develop a hybrid system combining AOP and the biological process in which AOP is used as post-treatment to the biological wastewater treatment. This approach should be considered viable from the economic point of view and with environmentally positive impact.

Material and Methods

Four different phenol solutions concentrations were prepared: 10 ppm, 20 ppm, 40 ppm, 100 ppm; and each was mixed with one litre of municipal wastewater samples. The quantitative analyses of phenol content in the treated was determined using the Folin-Ciocalteu and Folin-Denis methods (Ikawa, 2003). Absorbance measurements were determined using a Shimadzu UV-vis spectrophotometer model UV-1800. Also, GC-MS device was used for the analysis of the final sample treated solution.

Biological Process

Most biological treatment processes consist of aeration process followed by settling tanks with a partially recycled activated sludge stream. In this research work, an alternative design was proposed and constructed; the schematic diagram is shown in Figure 1. The biological reactor consists of a six litres of porous vessel in which the activated sludge was held and only allowing for clear water to leak out, and thus, sludge circulation is not required. The porous tank was used as an inner tank and placed inside a larger plastic tank of twenty litres; the larger tank is used for leaked liquid collection.

An air distributor was fixed inside the porous vessel and used for air purging (supplying oxygen) and mixing

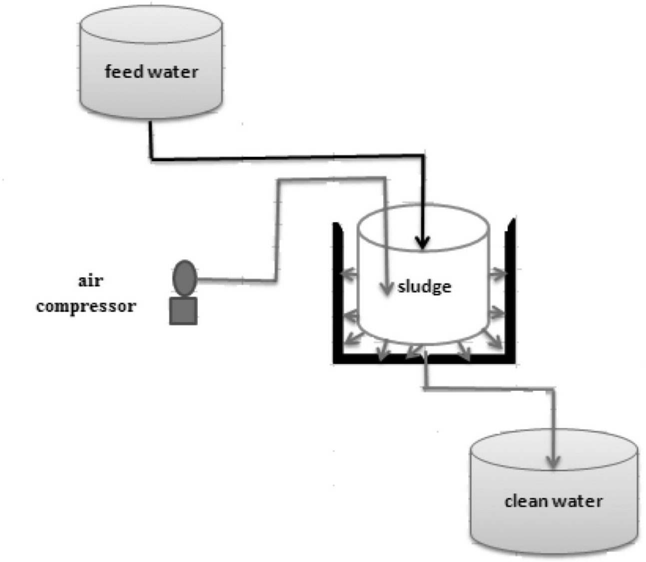


Figure 1: Biological process using porous tank.

purposes. Fresh sludge feed flow rate of 0.72 l/h was adjusted into the biological vessel. Samples of sludge were collected from the aeration basin at Nizwa sewage treatment plant in Sultanate of Oman.

Frequent samples were collected during operation for determining the phenol concentration and bacterial growth in the activated sludge. In respect to biological measurement, bacterial colony forming units (CFU) was determined in the microbiology lab at the University of Nizwa.

Photo-Catalytic Process

Photo-reactor was constructed from a coated glass flat plate (53 cm × 28 cm) with titanium dioxide and used as the solar photo reactor. Titanium dioxide type P25, Degussa AG (Germany) was selected. This catalyst has a particle size of 20 nm with a surface area of 50 m^2/g and composed of both phases 80 % anatase and 20% rutile.

Coating procedure can be summarised as follows: first, four pieces of glass surfaces were scratched using a sharp cutter, then the glass plates were cleaned using distilled water and then washed using 0.5 N NaOH solution in order to increase the OH- group on the glass surface. At the same time, the solution of the TiO_2 was prepared by dissolving 7.5 g TiO_2 in one litre distilled water. The coated glasses were dried using an electrical oven at 105 °C for one hour. In order to enhance the adhesion of TiO_2 on the glass surface, the dry coated glasses were placed in an electrical furnace at 500 °C for 4 hours. The final coated glass surfaces are shown in Figure 2. It can be seen that most of the glass surface was covered by titanium dioxide, and due to manual coating, a very small portion was slightly covered. However, the achieved results indicated an acceptable level of coating.

The four coated glass plates were joined together as one piece and placed on the top of steel frame with an angle of 45 degree. Perforated tube was placed on the top side of the glass for the distribution of solution on the glass surface. All units of the photo-catalytic reactor were connected by a set of tubes, centrifugal pump. A systematic assembly of the reactor is presented in Figure 3.

The process of heterogeneous photo-oxidation permits the destruction of the organic materials and suggests that the photo-oxidation of organic materials involves several intermediate steps depending upon the generation of both radicals; oxyl $^{\cdot}O_2$ and hydroxyl $^{\cdot}OH$ (Saien and Soleymani, 2007; Al-Dawery, 2017).

Basically, this type of AOP depends upon the excitation of the titanium dioxide particles surfaces by illumination using UV light. This action induces two types of carriers on the surface of TiO_2; the electron-hole pairs (e^-, h^+). These carriers produce the free oxyl and hydroxyl radicals that are able to perform a secondary reaction with the organic materials and resulting minerals and carbon dioxide. In addition, the presence of oxygen in the solution retrieves the electron and prevents the recombination of electron-hole reaction.

Figure 2: Coated surface of glass with TiO_2.

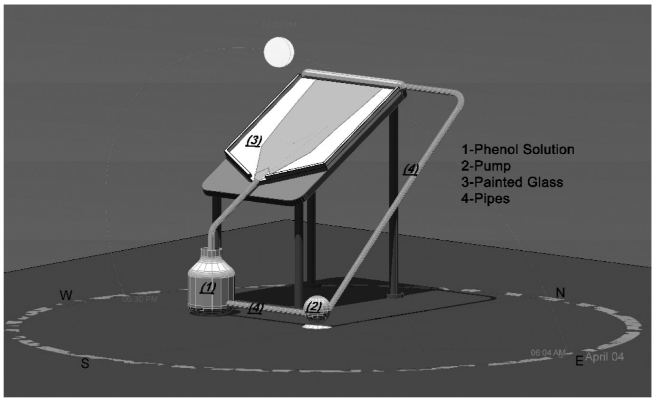


Figure 3. Systematic assembly of photo-catalytic reactor.

The mechanism is schematically suggested and shown in Figure 4. The two main steps of the photo-oxidation process are:

1. Initiation reaction of the photo-catalytic process:

$$TiO_2 + hv \rightarrow h^+ + e^- \quad (1)$$

$$h^+ + H_2O \rightarrow {}^{\cdot}OH + H^+ \quad (2)$$

$$e^- + O_2 \rightarrow {}^{\cdot}O_2^- \quad (3)$$

$$O_2^- + H_2O \rightarrow {}^{\cdot}OH \quad (4)$$

2. Process of the degradation of organic materials

$$\text{Substrate} + {}^{\cdot}OH \rightarrow CO_2 + \text{minerals} \quad (5)$$

$$\text{Substrate} + {}^{\cdot}O_2^- \rightarrow CO_2 + \text{minerals} \quad (6)$$

The treatment time in photo-oxidation process must be as short as possible in order to avoid high consumption of energy. However, there are other factors that are significantly affecting the oxidation process, such as solar light intensity, catalyst concentration, organic material concentration, pH and temperature. In order to ensure an efficient photo-oxidation treatment process, these factors should be carefully optimized (Al-Dawery, 2016; Chen et al., 2017).

Several experimental tests were conducted using polluted wastewater with different phenol concentrations. The solution was circulated at a flow rate of 0.333 litre/minute and running over the coated glass surface. The coated glass surface was exposed to the UV sunlight during tests in order to provide the suitable energy for exciting the particles of the titanium dioxide.

During the operation, solar UV light was frequently measured. The UV radiation available during experiments at a 365 nm wavelength was ranging from 1.89 to 2.2 mW/cm^2.

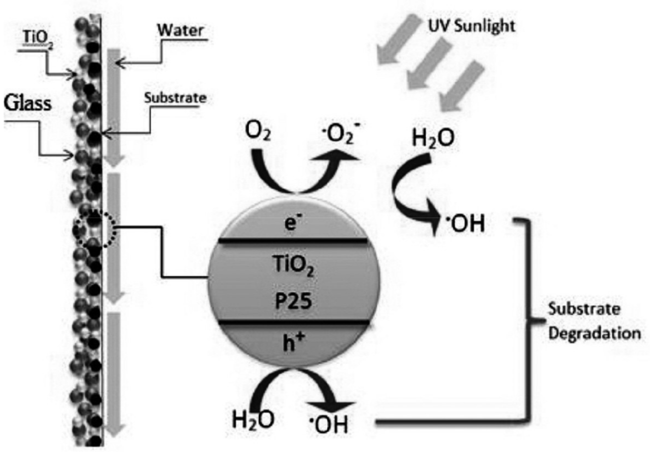
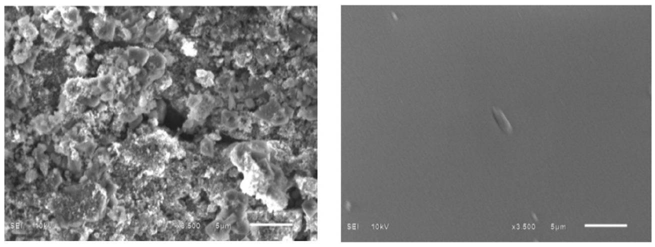


Figure 4: The mechanism of photo-catalytic activities of TiO_2.

Although the coated glass was not completely composited with the titanium dioxide, the two materials (glass and titanium dioxide) were attached with each other and TiO_2 was adhered on the glass surface. According to the SEM images, the TiO_2 matrix present prismatic particles like crystal and, it can be observed, rises in the size of the particles. This may be due to aggregation of the particles of TiO_2 on the glass surface. The morphologies of the pure glass and coated glass with TiO_2 were scanned using Scanning Electron Microscope and shown in Figure 5.

Comparisons between images of pure glass and coated glass indicated that the fine TiO_2 particles were sufficiently integrated on the glass surface.

The intensity of UV sun light at the site of the University of Nizwa was measured for six weeks and at different day times. The data are shown in Table 1 which indicated that the UV intensities at this site are ranging between 1.3 to 2.2 mW/cm^2. This UV light intensity could be considered suitable for required illumination level for the titanium dioxide and strong enough for photo-catalytic operation.



(a) Coated glass, X3500 **(b) Pure glass surface, X3500**

Figure 5: Images of pure and painted glass surface with TiO_2.

Results and Discussion

Four experiments have been carried out in order to examine the performance of bioreactor and activated sludge for treating wastewater containing phenolic compound. The degradation was carried out at different concentrations of phenol from 10 to 100 ppm. The results are presented in Figure 6.

From Figure 6, it can be seen that there was no reduction in phenol concentrations. This means that the biological treatment was not sufficient for the removal of phenol compound. However, the fluctuation in the results was due to the instrumentation error.

In order to show the effect of phenol solution on the bacterial growth, several samples were collected from

Table 1: UV measurement at the site of the University of Nizwa

Date	*Time*	*UV sun light intensity at 365 nm scale (mW/cm^2)*
10/June	9:20 am	1.34
	1:20 pm	1.88
	3:20 pm	0.953
11/June	8:40 am	1.025
	1:00 pm	2.19
	3:25pm (cloud)	0.89
12/June	8:50 am	1.011
	12:00 noon	2.05
	3:00 pm	1.063
13/June	9:00 am	1.161
	12:15 pm	1.793
	2:30 pm	0.943
16/June	11:30 am	1.971
	2:00 pm	2.214
	3:30 pm	1.274
17/June	10:00 am	1.37
	1:45 pm	2.197
	3:40 pm	1.222
18/June	8:45 am	1.032
	12:00 noon	1.934
	3:00 pm	1.45
19/June	10:00 am	1.632
	1:00 pm	1.935
	3:30pm (cloud)	0.415
20/June	8:30 am	1.39
	1:30 pm	1.955
	3:00 pm	1.692
23/June	10:10 am	1.882
	1:30 pm	2.237
	3:20 pm	1.636
24/June	9:00 am	1.671
	12:30 pm	2.019
	3:30 pm	1.288
25/June	10:00 am	1.779
	1:00 pm	1.68
	4:10pm (cloud)	0.226
03/July	9:00 am	1.515
	11:30 am	2.211
	2:45pm (cloud)	0.229
07/July	9:30 am	1.473
	1:00 pm	1.937
	3:45pm (cloud)	0.717
18/July	9:15 am	1.441
	1:30 pm	1.933
	3:00 pm	1.303

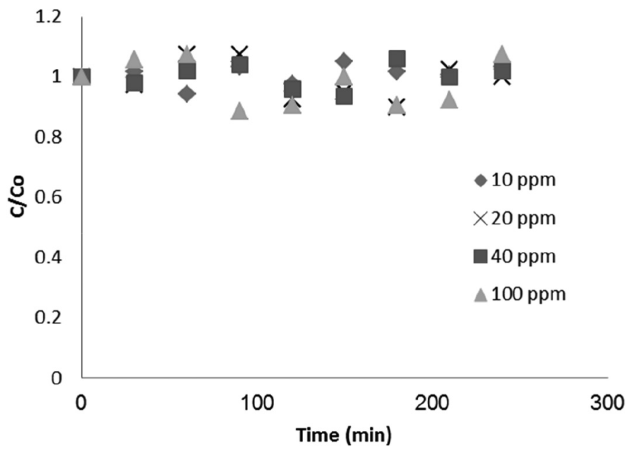


Figure 6: Non-degradation of phenol using biological process.

the bioreactor and examined. Four types of species were found: *Bacillus*, *Streptococcus*, *Escherichia coli* and *Pseudomona*. Types of colonies formed are shown in Table 2. The bacterial growth analyses during experiments for phenol concentration 20 ppm and 100 ppm are presented in Figure 7. It can be seen that the bacterial growth declined during the experiment due to the death of bacteria for lack of nutrients and may be the phenol compound acted as inhibitor of bacteria growth. In both tests, the bacterial colony growth was reduced by almost 50%.

Table 2: Types of colonies formed

Type of bacteria	*0 min*	*30 min*	*60 min*	*180 min*	*240 min*
Bacillus sp	Yes	Yes	Yes	Yes	Yes
Streptococcus sp	Yes	Yes	Yes	Yes	Yes
Escherichia coli	Yes	Yes	Yes	Yes	Yes
Pseudomonas sp.	Yes	Yes	Yes	Yes	Yes

For treatment of phenol using the photo-catalytic process, several experimental works were carried out to explore the influence and powerful action of TiO_2 especially when illuminated with UV sunlight on degradation of phenol. During experimental work, four different concentrations of phenol solutions were used: 10 ppm, 20 ppm, 40 ppm and 100 ppm. The results of these tests are plotted in Figure 8. The obtained result proved the capability of TiO_2 for degradation of phenol. For phenol concentrations lower than 100 ppm, complete degradations were achieved, while for concentration of 100 ppm, the phenol was decomposed to an acceptable level; longer exposure to sun light will improve its degradation further.

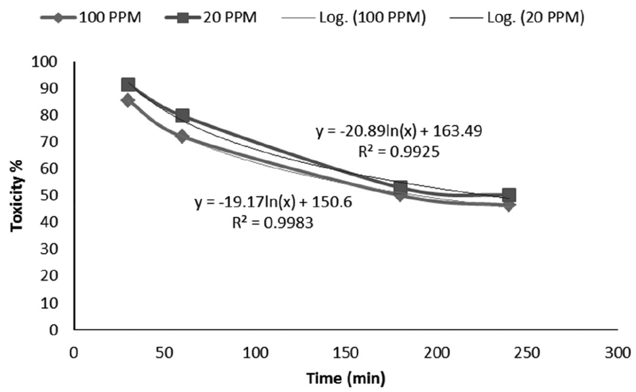


Figure 7: Bacterial colony toxicity using phenol with 20 and 100 ppm.

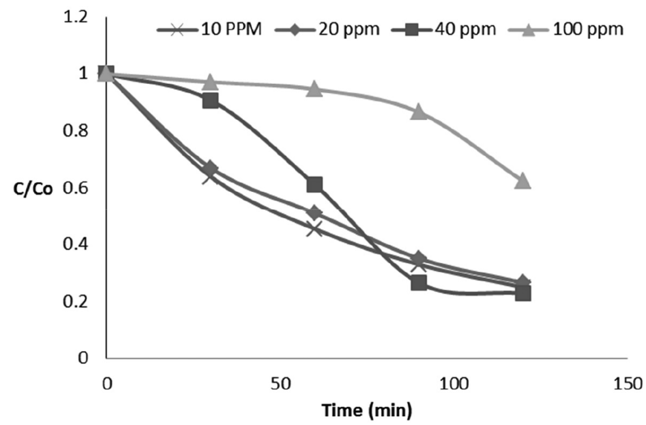


Figure 8: Comparison between phenol degradation using AOP.

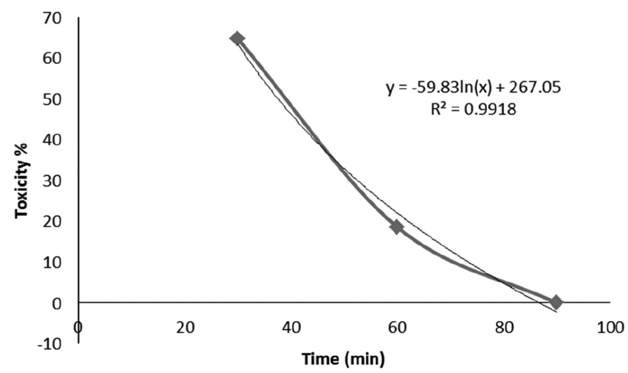


Figure 9: Bacterial colony toxicity when using AOP.

In respect to the bacterial colony growth, the results indicated that the oxidation process had a larger impact compared to that of using phenol, as shown in Figure 9. The results showed bacterial growth was dramatically reduced and no colony growth was found after 90 minutes after commencing time of experiment. From these results, it can be observed that the titanium dioxide acted as a strong inhibitor for the biological growth and thus cannot be used as pretreatment coupled with biological process. Types of colonies formed during the test are analyzed and presented in Table 3.

Table 3: Types of colonies formed

Type of bacteria	*0 min*	*30 min*	*60 min*	*90 min*
Bacillus sp	Yes	Yes	Yes	No
Streptococcus sp	Yes	Yes	No	No
Escherichia coli	Yes	Yes	Yes	No
Pseudomonas sp.	Yes	No	No	No

Sample from the treated wastewater using photo-catalytic reactor was analyzed using GC-MS device and the result is presented in Figure 10. This result indicated that the phenol was completely degraded and the major produced compound from the phenol degradation is ethynylcyclopropane. The produced material and phenol are both considered toxic and antibacterial (CHEMTREC, 2008). Longer exposure to sun light will further improve the degradation of both components. However, there is no aromatic compound in the ethynylcyclopropane to be oxidized; thus, the degradation of ethynylcyclopropane would be much easier than phenolic compounds.

From the obtained results, it can be concluded that the combination of AOP and biological process for the treatment of bio-refractory materials can be effective if the photo-oxidation process is recommended as a post treatment to the municipal biological wastewater treatment; other arrangement may reduce the efficiency of the biological process and volubility of the activated sludge.

Conclusion

The present study provides an innovative approach of activated sludge and photo-oxidation for the treatment of wastewater containing phenol compounds. The results showed that there was no reduction in phenol concentration during the experimental work; at the same time the bacterial growth declined by 50% due to lack of nutrients. The activated sludge and its bacterial contents did not consume the phenol and thus unable to remove the phenol from the wastewater. The findings proved the capability of painted TiO_2 for the degradation of phenol and complete degradation were achieved when phenol concentration was lower than 100 ppm, while for higher concentration and longer exposure to UV sunlight will improve the phenol degradation. The photo-catalytic process using titanium dioxide (TiO_2) removal of phenol was 80% at phenol concentration below 40 ppm.

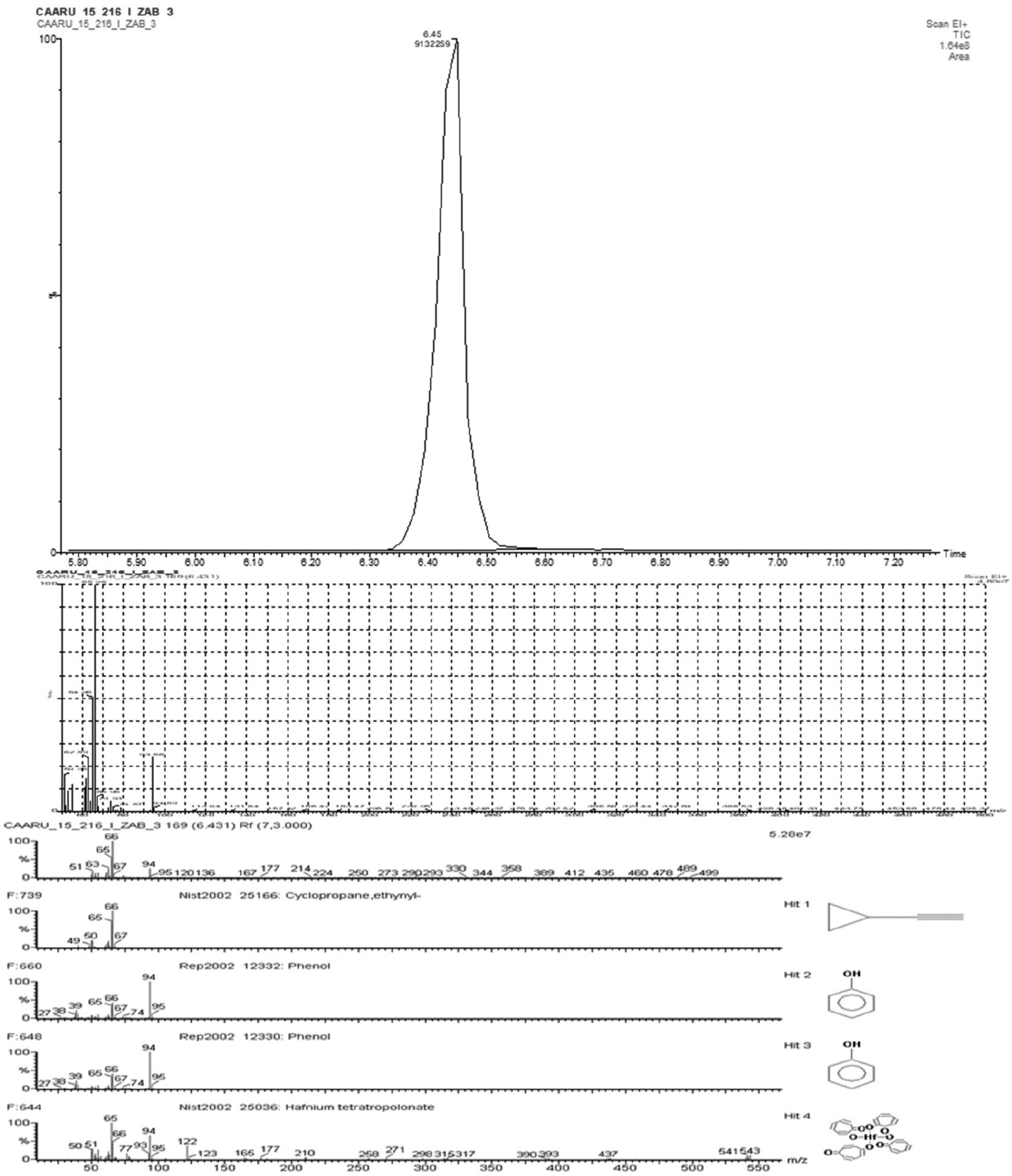


Figure 10: GC-MS result for major produced compound from the phenol degradation using photo-oxidation process with 100 ppm phenol.

Bacterial colony growth during photo-oxidation was dramatically reduced and no colony growth was found after 90 minutes of experiment. Thus, the application of the combination of AOP and biological processes, photo-oxidation process is recommended as a post-treatment to the biological wastewater from industrial wastewater.

Acknowledgement

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Calendar of Events

The IAFOR International Conference on Sustainability, Energy and the Environment - Hawaii 2019
3rd to 5th January 2019
Honolulu, Hawaii, United States of America
Website: https://iicseehawaii.iafor.org/
Contact person: Kiyoshi Mana
Organized by: IAFOR – The International Academic Forum

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21st to 23rd January 2019
Coimbatore, Tamilnadu, India
Website: http://www.psgias.ac.in/wp-content/uploads/2018/10/Brochure-ECWE-2019-B3.pdf
Contact person: Dr.R.Selvakumar

10th International Conference on Environmental Science and Development (ICESD 2019) - EI Compendex, Scopus
13th to 15th February 2019
Milan, Italy
Website: http://www.icesd.org/
Contact person: Ms. Sophia Du
Organized by: CBEES

International Conference on Advances in Water and Wastewater Treatment Technologies (2W2T 2019)
8th to 10th March 2019
Shenzhen, China
Website: http://www.2w2t.net/
Contact person: Eddie

9th International Conference on Environment Science and Engineering (ICESE 2019)
20th to 22nd March 2019
Leuven, Belgium
Website: http://www.icese.org/
Contact person: Mr. Issac Lee
Organized by: ICESE