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# DEGRADATION OF PHENOL USING SONOLYSIS AND PHOTOLYSIS BY $TiO_2$/RHAC CATALYST AND ANALYSIS WITH SPECTROPHOTOMETER UV-VIS AND HPLC

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**Abstract**

This study focuses on the degradation of phenol in aqueous solutions using photolysis and sonolysis methods. It investigates the impact of catalyst mass, types of light (UV-A, UV-C, and visible light), types of catalyst (RHAC, $TiO_2$, and $TiO_2$/RHAC), and processing time on phenol degradation. The phenol solutions are analyzed before and after degradation using Spectrophotometer UV-Vis and HPLC. The research aims to understand the factors influencing phenol degradation and provide a basis for further studies to enhance the efficiency of phenol removal. Results show significant improvements in degradation percentages of phenol by using $TiO_2$/RHAC as the catalyst. Sonolysis achieves a degradation of 20.82% with a catalyst, which increases to 50.57% with the catalyst. Photolysis achieves a degradation of 29.06%, which rises to 91.99% with the catalyst. The highest degradation percentage is achieved using UV-A light for 5 hours with a catalyst mass of 30 mg of $TiO_2$/RHAC catalyst. HPLC analysis confirms a decreased phenol concentration and the presence of intermediate compounds. The $TiO_2$/RHAC catalyst demonstrates the promising potential for efficient phenol degradation in aqueous solutions.

## INTRODUCTION

Increasing industrial activity has resulted in water contamination, with phenol being a significant hazardous liquid waste. Phenol (**Figure 1**), widely used in various industries, can cause severe health issues and even death at high concentrations. Uncontrolled discharge of phenol and other pollutants into water bodies poses a significant threat to the environment and living organisms. Therefore, it is imperative to address this issue to prevent additional harm and safeguard environmental and human health [1].

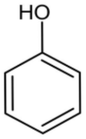

**Figure 1**. Chemical structure of phenol.

Based on the Decree of the Environment Minister of the Republic of Indonesia No. 51/MENLH/10/1995 and Decree of the Health Minister of the Republic of Indonesia No. 907/MENKES/SK/VII/2002, the maximum limits of phenol and its derivatives in waters are 0.5 – 1.0 mg/L and 0.01 mg/L respectively [2] [3]. The methods such as adsorption, reverse osmosis membrane, coagulation/precipitation, and Advance Oxidation-Processes (AOPs) have been made to reduce phenol [4] [5] [6].

AOPs such as sonolysis and photolysis can degrade waste organic compounds into $CO_2$ and $H_2O$ [7], [8]. Sonolysis uses ultrasonic vibrations and light is used by photolysis as a degradation method. Both of these methods produce hydroxyl radicals (•OH) which can react with organic compounds and produce no residue. Sonolysis and photolysis can be combined with a catalyst because the catalyst can increase the production of hydroxyl radicals (•OH) thereby increasing the effectiveness of degradation [9].

Rice husk-activated carbon (RHAC) is a carbon material derived from rice husk and has been activated. According to the Official Statistical News No. 74/10/Th XXV released on October 17, 2022, by Central Statistics Agency (BPS), rice husk is one of the agricultural waste products of 55.67 million tonnes during the rice milling process. The availability of rice husk in Indonesia makes it an economical and easy-to-obtain material [10]. On the other hand, a hetero-catalyst using Titanium Dioxide ($TiO_2$) is one of the potential catalysts in the AOPs method. However, $TiO_2$ still has disadvantages including low adsorption ability, wide band gap, tends to aggregate during the degradation process, and requires a lot of photon energy [11]. Previous studies used several catalysts such as N-doped $TiO_2$, C, N-codoped $TiO_2$ dan C-doped $TiO_2$ in the degradation process of toxic organic compounds (dyes, pesticides, and antiseptics such as phenol) [13] [14] [15] [16] [17] by sonolysis [18] and photolysis [19] as an alternative catalyst.

In this study, $TiO_2$/RHAC was chosen as a catalyst because $TiO_2$ is a photocatalyst to produce reactive compounds when using light and RHAC is an active carbon capable of adsorbing and binding pollutants [20]. In addition, the use of rice husks can reduce waste in agriculture. $TiO_2$ is combined with porous materials such as RHAC to increase surface area, the AOPs process can occur optimally [12]. This research investigates the degradation of phenol through sonolysis and photolysis methods using $TiO_2$/RHAC as a catalyst, aiming to achieve optimal and environmentally friendly phenol degradation. The study examines various parameters including catalyst mass, degradation time, and light types.

## EXPERIMENT

### *Material*

The activated carbon was produced using rice husks from Tapan, Pesisir Selatan, West Sumatra, Indonesia as this region is abundant in paddy fields. Chemicals such as KOH, HCl, $TiO_2$, and $C_6H_5OH$ were obtained from Merck. The pH of the solution was measured using a pH meter, suspended particles were separated from liquids using Whatman filter paper No. 42, and distilled water was utilized as the solvent.

### *Instrumentation*

Fourier Transform Infrared (FTIR) (Unican Mattson Mod 7000 FTIR) and Diffuse Reflectance Spectroscopy UV-Vis (DRS UV-Vis) (SPECORD 210 Plus) were used to characterize the catalyst. Phenol solutions were analyzed by UV-Vis Spectrophotometer (Shimadzu UV-1280) and HPLC (Shimadzu SPD-20A/20AV Series).

### *Procedure*

The rice husks were washed with distilled water and dried at room temperature and 110 °C to a constant mass. The procedure was followed by carbonization at 400°C for 2 hours. The carbon was activated with KOH (1:4). The mixture was stirred until homogeneous, left overnight, and washed with 1 M HCl, and distilled water until the pH was neutral. Activated carbon was oven dried at 110°C, crushed, and sieved using a 45 µm of sieve. This activated carbon was named RHAC.

After RHAC was prepared, the procedure was continued by preparing $TiO_2$/RHAC catalyst. The $TiO_2$/RHAC catalyst (9.5:0.5) was suspended in distilled water, vibrated by a sonicator for 5 hours, and filtered. Then $TiO_2$/RHAC was dried in an oven at 105°C for 24 hours. $TiO_2$/RHAC catalyst was characterized using FTIR and DRS UV-Vis.

Phenol degradation was tested by sonolysis and photolysis, with and without catalyst. The degradation of phenol with photolysis was determined by the optimal mass of the catalyst. Each $TiO_2$/RHAC catalyst of 10, 20, 30, 40, and 50 mg was added to 30 mg/L phenol solution. The solution was irradiated with a UV-A light for 5 hours. The optimal mass of the catalyst was then used to determine the degradation ability at various contact times (1, 2, 3, 4, 5, and 6 hours) by sonolysis and photolysis. Then, the best contact time was then used for the degradation of phenol in various lights types (UV-A, UV-C, and visible light) by photolysis, and catalysts types (RHAC, $TiO_2$, and $TiO_2$/RHAC) by sonolysis and photolysis. The degraded solution was centrifuged for 15 minutes at 2500 rpm and separated by Whatman filter paper no. 42. Absorbance of phenol solution was measured using a Spectrophotometer UV-Vis at 200-400 nm. The degradation percentage was calculated using the equation:

$$\%Degradation = \frac{C_0 - C_t}{C_0} \times 100\%$$

where $C_0$ and $C_t$ correspond to the initial and final phenol concentration.

The highest degradation percentage was determined using High-Performance Liquid Chromatography (HPLC) with a C18 column (250 × 4.6 mm) employing an aquabidest: methanol (65:35) mixture as the mobile phase. The injection volume was set to 10 µL, the flow rate was maintained at 1.0 mL/minute and a UV detector set at 270 nm was utilized. These treatments or methods constituted the analytical setup for the analysis.

## RESULT AND DISCUSSION

### *Modification and Characterization of $TiO_2$/RHAC*

Activated carbon is carbon that has been heated so that it has a strong affinity for adsorbing various materials at 35°C. Activated carbon has a large surface area (300 – 2500 $m^2/g$). Activation can break the hydrocarbon bonds or oxidize the surface molecules so that the carbon pore becomes larger [21].

**Figure 2** is the result of the characterization of the $TiO_2$/RHAC catalyst. $TiO_2$ has a band gap value of 3.2 eV [22]. After $TiO_2$ was combined with activated carbon, the band gap value decreased to 3.1 eV. It assumed that the minerals in rice husk also adsorb when using DRS UV-Vis [23]. This is an advantage because the degradation process can be carried out more efficiently.

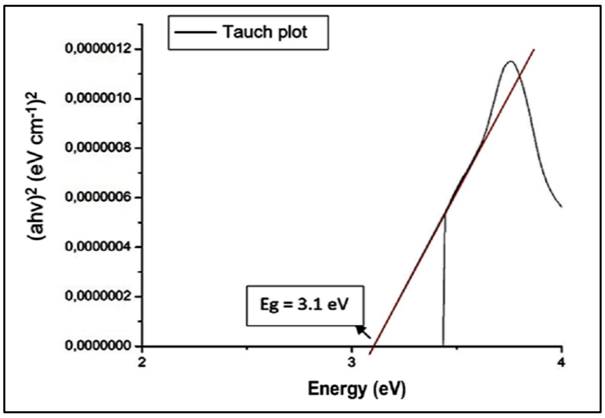

**Figure 2.** Absorption plot of $TiO_2$/RHAC catalyst in DRS UV-Vis.

Functional group analysis on the RHAC surface was tested using FTIR. The results of the FTIR analysis of RHAC are shown in **Figure 3**. The spectrum shows that the absorption at wave number 3353.75 $cm^{-1}$ is the absorption of the OH group for carboxylic acids. The wave number 1594.23 $cm^{-1}$ is the absorption band region of the C=C stretching group of aromatic compounds. The C–O group shown at wave number 1073.49 $cm^{-1}$ and wave number 541.77 $cm^{-1}$ is the fingerprint region of rice husk activated carbon.

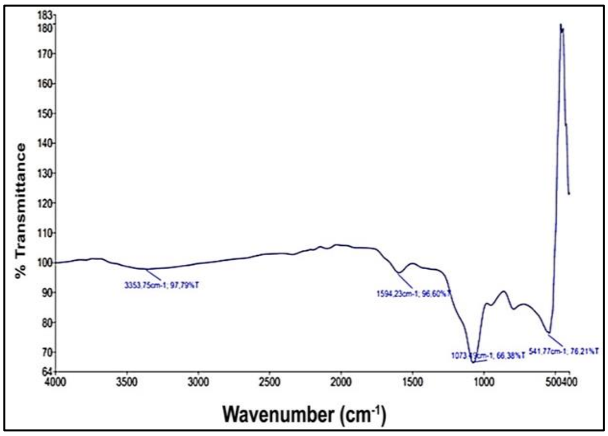

**Figure 3.** FTIR spectrum of RHAC.

The absorption of phenol solutions with concentrations of 10, 20, 30, 40, and 50 mg/L was measured using a UV-Vis spectrophotometer at a wavelength of 200-400 nm. The calibration curve of the phenol solution can be seen in **Figure 4**. The wavelength that shows the maximum absorption peak of the phenol solution is 270 nm. The regression equation obtained is $y = 0.0143x + 0.0145$ with a coefficient of determination ($R^2$) = 0.9996. This $R^2$ value indicates that the curve has good linearity so that it can be used to determine the concentration of phenol in degradation.

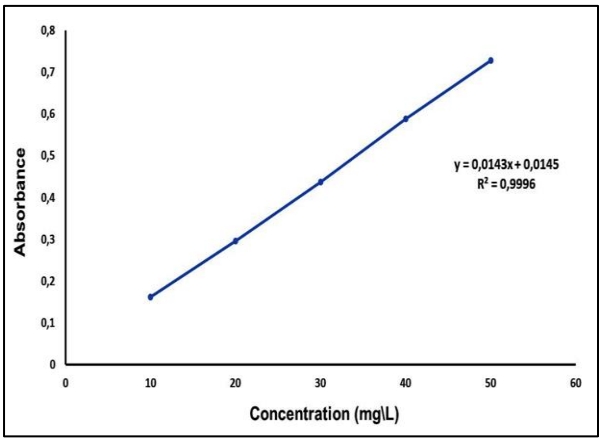

**Figure 4**. Calibration curve of phenol solutions.

Determination of the optimal mass of $TiO_2$/ RHAC catalyst for phenol degradation by photolysis was carried out using various catalyst masses of 10, 20, 30, 40, and 50 mg. The results of phenol degradation at various catalyst masses are shown in **Figure 5**.

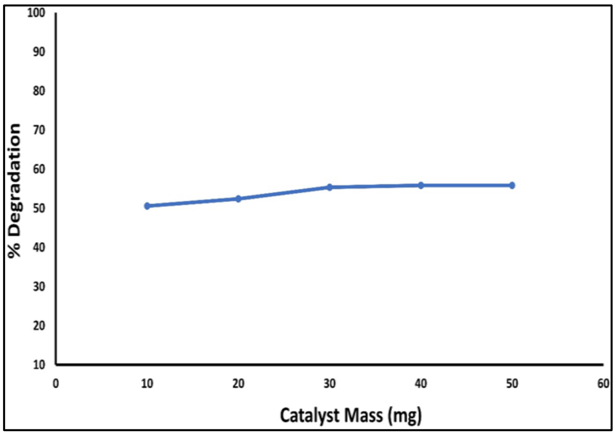

**Figure 5**. Effect of $TiO_2$/RHAC catalyst mass.

The data shows that the percentage of degradation increased significantly at 10 to 30 mg catalyst mass, but slightly decreased at 40 and 50 mg. The amount of $TiO_2$/ RHAC catalyst increasing causes more phenolic compounds to be absorbed and increases the surface active side. The decrease in the degradation percentage occurred because the catalyst made the saturated solution, and turbid which decreased light penetration [13]. The optimal catalyst mass of $TiO_2$/RHAC for 5 hours irradiation times to degrade phenol is 30 mg with a degradation percentage of 55.38%.

### Effect of Catalyst on Phenol Degradation

A comparison of the degradation percentage of phenol by sonolysis and photolysis (UV-A light) using 30 mg $TiO_2$/RHAC catalyst for 1, 2, 3, 4, 5, and 6 hours is illustrated in **Figure 6A** and **Figure 6B**.

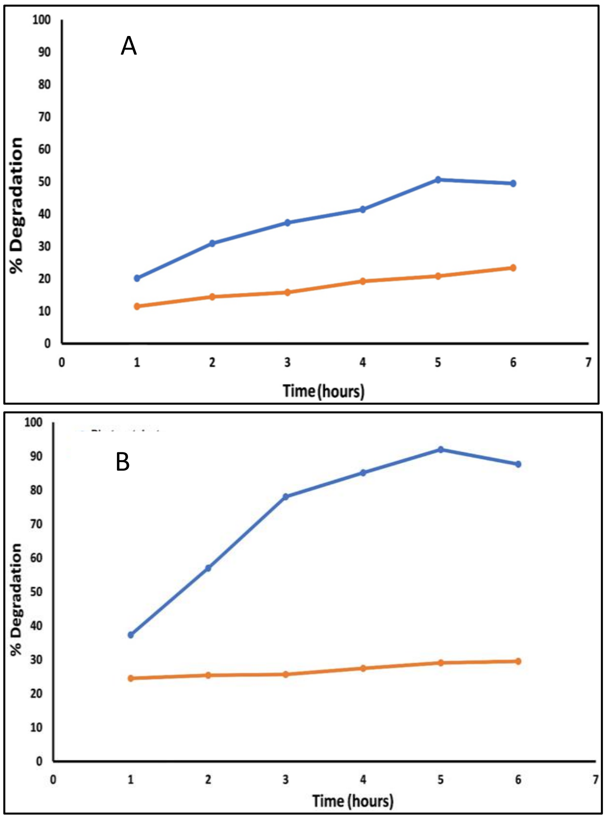

**Figure 6**. Effect of Catalyst and Degradation Methods.

[Phenol]=30 mg/L, time=5 hours, catalyst mass= 30 mg (A) Sonolysis (with and without catalyst); (B) Photolysis (with and without catalyst)

Based on **Figure 6**, the degradation percentage of phenol without and with catalyst increased with increasing time. The increase in the degradation percentage was due to the longer sonolysis time, more hydroxyl radicals (•OH) are produced. However, the degradation percentage of phenol decreased slightly when it was degraded for 6 hours. In the context of phenol degradation through sonolysis, the duration required for effective degradation was 5 hours. Without the presence of a $TiO_2$/RHAC catalyst, the degradation percentage achieved was 20.82%. However, when introducing the $TiO_2$/RHAC catalyst, the degradation percentage increased significantly to 50.57%. Catalysts play a crucial role in the degradation of compounds by facilitating the production of a greater number of hydroxyl radicals (•OH). These hydroxyl radicals are highly reactive and effectively react with organic compounds such as phenol, leading to the degradation process [14]. The presence of the $TiO_2$/RHAC catalyst resulted in an enhanced degradation efficiency.

In addition to sonolysis, phenol was also degraded through photolysis utilizing the $TiO_2$/RHAC catalyst. A comparison of the degradation percentage by photolysis with UV-A light can be seen in Figure 6(B). Utilizing a 30 mg $TiO_2$/RHAC catalyst irradiated with UV-A light, the degradation percentage of phenol significantly increased during photolysis. However, the degradation percentage decreased during the 6-hour photolysis period. Based on the obtained data, it was determined that the optimal irradiation time for phenol degradation through photolysis was 5 hours, resulting in degradation percentages of 29.06% without a catalyst and 91.99% with the presence of a 30 mg $TiO_2$/RHAC catalyst under UV-A light.

Differences in the degradation percentage also occur between sonolysis and photolysis. The degradation percentage by photolysis is greater than the degradation percentage by sonolysis. The activated carbon and UV-A light can affect the degradation process because RHAC can adsorb more phenol and reduce the band gap of $TiO_2$ and UV-A light can generate electrons to produce hydroxyl radicals (•OH) [15]. The UV light used in photolysis is generally more effective to degrade phenol than ultrasonic waves in sonolysis. However, many factors such as pH or temperature cause differences in the degradation percentage. It is still uncertain that the degradation percentage using sonolysis is always smaller than photolysis [24].

### Effect of Catalyst Types on Phenol Degradation by Sonolysis

The degradation of phenol through sonolysis was evaluated by comparing the effects of different catalysts, namely RHAC, $TiO_2$, and $TiO_2$/RHAC. In **Figure 7**, it is evident that the presence of the

$TiO_2$/RHAC catalyst significantly influences the degradation of phenol. The difference in degradation percentage between $TiO_2$ and $TiO_2$/RHAC is 13.5%, while the difference between RHAC and $TiO_2$/RHAC is even more pronounced at 41.65%. These comparative results indicate that the utilization of the $TiO_2$/RHAC catalyst is capable of substantially increasing the degradation percentage of phenol, highlighting its superior effectiveness compared to the other catalysts examined.

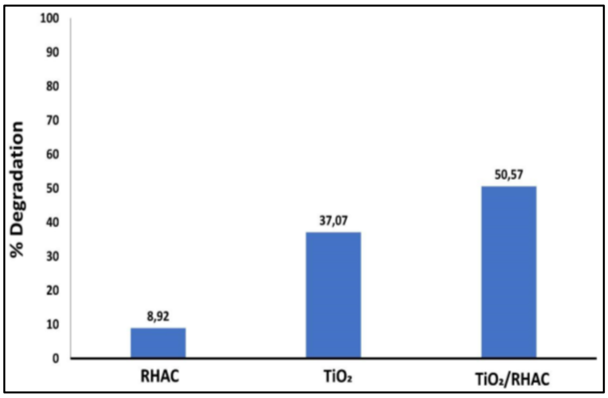

**Figure 7**. Effect of catalyst type.

[Phenol]= 30 mg/L, sonolysis times= 5 hours, catalyst mass= 30 mg.

## Effect of Light Type and Catalyst Type on Phenol Degradation by Photolysis

Phenol degradation through photolysis was assessed using different light sources, namely UV-A light (λ=365 nm), UV-C light (λ=254 nm), and visible light. **Figure 8** illustrates the degradation percentages of phenol under UV-A, UV-C, and visible light as 91.99%, 57.89%, and 52.4%, respectively. Based on these results, UV-A light is the most effective for phenol degradation through photolysis. UV-A light has a longer wavelength and higher energy than UV-C and visible light, enabling deeper penetration into the sample and increased interaction with organic compounds. UV-C light is primarily effective for degrading complex organic compounds, while organic compounds readily absorb UV light compared to visible light [16].

$TiO_2$/RHAC catalyst in photolysis has a high phenol degradation effect. The difference in degradation percentage between $TiO_2$/RHAC and $TiO_2$ is 14.42%. Activated carbon acts as a photosensitizer, absorbing and transferring light to $TiO_2$. This process decreases the band gap value and increases the surface area, resulting in more effective phenol degradation. [25].

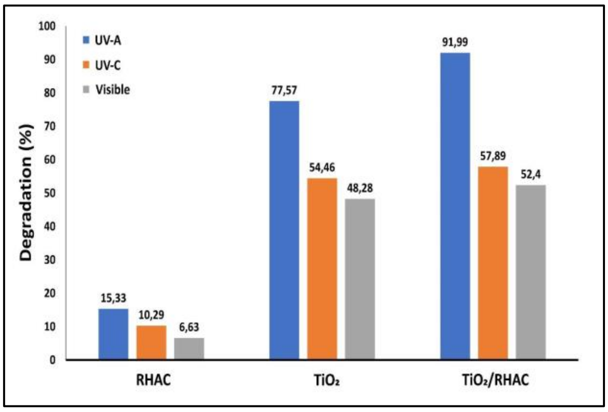

**Figure 8**. Effect of light and catalyst type.

[Phenol]= 30 mg/L, irradiation time= 5 hours (UV-A light), catalyst mass= 30 mg.

## HPLC Analysis

Phenol solutions were treated with photolysis using a $TiO_2$/RHAC catalyst and UV-A light and subsequently analyzed using High-Performance Liquid Chromatography (HPLC) to evaluate the alterations in composition pre-degradation and post-degradation. The HPLC system employed in this study consisted of a 270 nm UV detector, C18 column (250 × 4.6 mm), aquabidest: methanol (65:35 v/v) mobile phase, an injection volume of 20 µL, and flow rate of 1 mL/min.

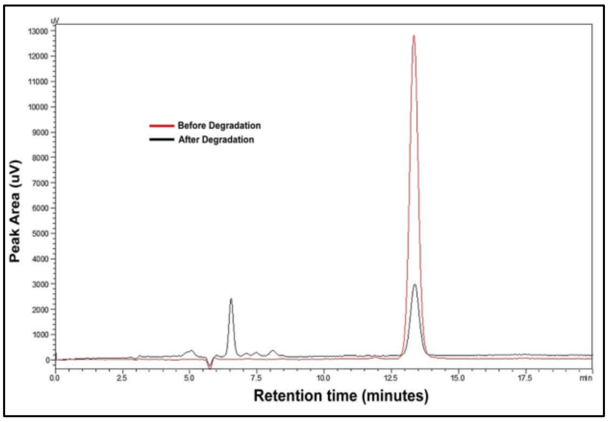

**Figure 9**. Chromatogram of phenol before and after degradation.

[Phenol]=30 mg/L, irradiation time= 5 hours (UV-A light), $TiO_2$/RHAC = 30 mg.

Based on the HPLC chromatogram in **Figure 9**, before degradation, the phenolic compounds only produced one peak at a retention time of 13.365 min with a peak height of 12788. After degradation, the chromatogram produced two peaks at a retention time of 13.365 min and 6.523

with a peak height of 2836 and 2350, respectively. Based on the chromatogram, it can be seen that there is a decrease in peak height after degradation for 5 hours. The formation of a new peak is due to the formation of intermediate compounds resulting from phenol degradation. The existence of a new peak in the chromatogram as phenol degradation intermediate compounds need to be analyzed further.

## CONCLUSION

The experimental results indicate that phenol degradation is influenced by multiple factors, including catalyst mass, degradation duration, catalyst type, and light type. The degradation percentages of 30 mg/L phenol through sonolysis and photolysis using UV-A light for 5 hours were 20.82% and 29.06%, respectively. However, the addition of 30 mg of $TiO_2$/RHAC catalyst significantly increased the phenol degradation percentages to 50.57% in sonolysis and 91.99% in photolysis using UV-A light. The implementation of the $TiO_2$/RHAC catalyst effectively adsorbed phenol compounds and enhanced the generation of hydroxyl radicals (•OH), thereby optimizing the degradation process.

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