

# Degradation of Remazol Yellow FG by Sonolysis and Photolysis with TiO<sub>2</sub>/Active Carbon Rice Husk (TiO<sub>2</sub>/AC) Catalyst and Analysis Using Spectrophotometer UV-Vis

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## Abstract

Remazol Yellow FG is one of the dyes that are often used in the textile industry because it is accessible and reasonably priced. The non-biodegradable dyes produce waste and inhibit sunlight from passing through the water. In this study, we performed Fourier Transform Infrared (FTIR) to characterise the activated carbon of rice husk and TiO<sub>2</sub>/activated carbon rice husk (TiO<sub>2</sub>/AC) using Diffuse Reflectance Spectroscopy UV-Vis (DRS UV-Vis). Remazol Yellow FG was degraded by using sonolysis and photolysis under UV ray ( $\lambda = 254$  and  $365$  nm) and visible ray. We performed the experiments using a variety of variables which consist of catalyst dosage (10–50 mg), contact time (1–6 hours), catalyst type (AC, TiO<sub>2</sub> and TiO<sub>2</sub>/AC), lamp type (365 nm, 254 nm and visible lamp) and initial Remazol Yellow FG concentration (10–30 mg/L) to determine the degradation percentage. We found that the addition of TiO<sub>2</sub>/AC catalyst increased the degradation percentage of Remazol Yellow FG from 6,86% to 52,62% using sonolysis and 8,34% to 95,02% using photolysis. Hence, we concluded that TiO<sub>2</sub>/AC catalyst from rice husk could be an effective catalyst for the Remazol Yellow FG degradation.

## Keywords

Remazol Yellow FG  
Degradation  
Sonolysis  
Photolysis  
TiO<sub>2</sub>/AC

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## 1. Introduction

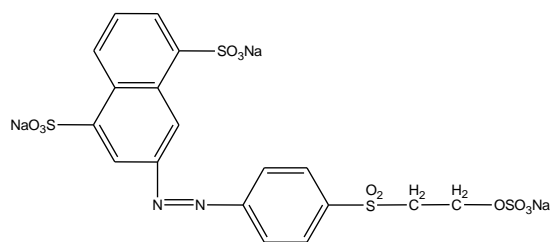
Due to their accessibility and being reasonably priced, the Remazol Yellow FG (Figure 1) is one of the most widely used dyestuffs in the textile industry. The pollution of Remazol Yellow FG in waters inhibits microalgae to get enough sunlight, preventing photosynthesis, and impacting the reduction of the amount of oxygen in the water, which results in an unpleasant odour and bad quality of water. It takes more time and works to completely remove the dyes from water due to the anaerobic degradation of azo dyes yields many hazardous aromatic amines<sup>[1]</sup>.

Several methods can be applied to reduce the contents of dyes waste which are: coagulation<sup>[2]</sup>, adsorption<sup>[3]</sup>, electrocoagulation<sup>[4]</sup> and membrane filtration<sup>[5]</sup>. These methods often cause new problems for the environment such as the emergence of new phases containing pollutants. Additionally, these methods only transfer the dye from the liquid phase to the solid phase and are considered less effective. Besides, photocatalysts might be potential as an alternative method to reduce dye

waste. Photocatalyst was developed using a semiconductor with the advantage of being able to decompose dyes into CO<sub>2</sub> and H<sub>2</sub>O molecules so that they do not harm the environment and could be used repeatedly, and also it is affordable. The process is also relatively short, non-toxic and has a long-term use<sup>[6]</sup>.

One of the most potential semiconductors often used as a photocatalyst in waste treatment is Titanium Dioxide (TiO<sub>2</sub>). TiO<sub>2</sub> is often used as a photocatalyst due to its strong catalytic activity and relatively low cost. However, the TiO<sub>2</sub> has a large band gap, which is 3.2 eV for the anatase phase. Therefore, to reduce the band gap and improve the effectiveness of TiO<sub>2</sub>, the catalyst could be modified with non-metal elements (TiO<sub>2</sub>/Zeolite)<sup>[7]</sup>, transition elements (TiO<sub>2</sub>/NiO)<sup>[8]</sup> and combinations of the two elements (C-N Codoped TiO<sub>2</sub>)<sup>[9]</sup>. Hence, this research investigates on synthesizing activated carbon and characterizing using Fourier Transform Infrared (FTIR) and TiO<sub>2</sub>/AC by Diffuse Reflectance Spectroscopy UV-Vis (DRS UV-Vis). The catalyst was also used in the degradation of Remazol

Yellow FG using sonolysis and photolysis, both were analyzed using a Spectrophotometer UV-Vis.



**Figure 1.** Chemical structure of Remazol Yellow FG

## 2. Materials and Methods

### 2.1. Materials

Rice husks were obtained from Batang Betung, Basa Ampek Balai Tapan, Pesisir Selatan, West Sumatra, Indonesia. Potassium Hydroxide (KOH), Hydrochloric Acid (HCl), and Titanium Dioxide ( $\text{TiO}_2$ ) were purchased from Merck (Darmstadt, Germany), Remazol Yellow FG, pH paper, Whatman filter paper no 42, and distilled water.

### 2.2. Synthesis

#### 2.2.1. Synthesis of Activated Carbon

Rice husks were rinsed with distilled water, dried and ground using a grinder. The rice husk was oven-dried at  $110^\circ\text{C}$  and carbonized at  $400^\circ\text{C}$  for 2 hours. The carbon size was filtered using a  $45\ \mu\text{m}$  filter. Carbon activation by adding 2 M KOH (1:4). Mixture was stirred until homogeneous and left overnight. Then, Activated Carbon was calcined at  $400^\circ\text{C}$  for 2 hours and washed using 1 M HCl (up to pH 7). Activated carbon was dried again in the oven at  $110^\circ\text{C}$ .

#### 2.2.2. Synthesis of $\text{TiO}_2$ /Activated Carbon ( $\text{TiO}_2$ /AC) Catalyst

$\text{TiO}_2$  and activated carbon were weighed (9.5:0.5), suspended in distilled water and sonicated for 5 hours. Later, the mixture was filtered and dried in an oven at  $105^\circ\text{C}$  for 24 hours until the size was equal to  $90\ \mu\text{m}$ .

#### 2.2.3. Characterization of Catalyst

The activated carbon was characterized using Fourier Transform Infra-Red (FTIR) and the  $\text{TiO}_2$ /AC catalyst was characterized using Diffuse Reflectance Spectroscopy UV-Vis (DRS UV-Vis). FTIR (PerkinElmer Frontier C90704 Spektrum IR Version 10.6.1) was used to analyse functional groups of the activated carbon and DRS UV-Vis (SPECORD 210 Plus) was used to identify the band gap energy of the catalyst.

### 2.2.4. Degradation of Remazol Yellow FG

Degradation of Remazol Yellow FG was evaluated by photolysis (under UV lamp (365 and 254 nm) and visible lamp) and sonolysis with  $\text{TiO}_2$ /AC catalyst. Some parameters of degradation were studied such as catalyst dosage (10, 20, 30, 40 and 50 mg), contact time (1, 2, 3, 4, 5 and 6 hours), catalyst (activated carbon,  $\text{TiO}_2$  and  $\text{TiO}_2$ /AC) and lamp variations (UV and visible lamp) and initial Remazol Yellow FG concentration (10, 15, 20, 25 and 30 mg/L). The absorbance was measured at 414 nm using a spectrophotometer UV-Vis. All the degradation percentage in various parameter were calculated as follow:

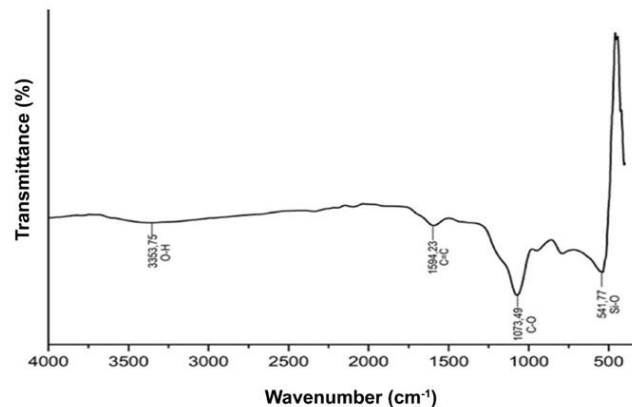
$$\% \text{Degradation} = \frac{C_i - C_o}{C_o} \times 100\% \quad (1)$$

where,  $C_i$  and  $C_o$  are the initial and final concentrations, respectively

## 3. Results and Discussion

### 3.1. Characterization of C and $\text{TiO}_2$ /AC Catalyst

The functional groups on the surface of rice husk activated carbon (AC) were studied using FTIR. The spectrum of activated carbon was presented in Figure 2.

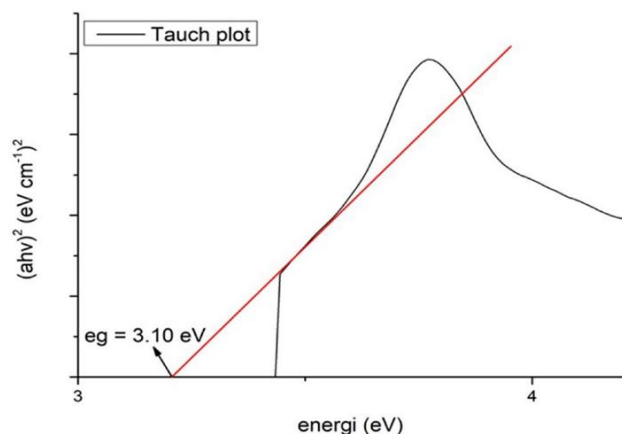


**Figure 2.** FTIR spectrum of rice husk activated carbon (AC)

The O-H stretching group absorption band was located at wavenumber  $3353\ \text{cm}^{-1}$ , while the C=C stretching group absorption band was located at wavenumber  $1594\ \text{cm}^{-1}$ . The C-O group absorption region was indicated at wavenumber  $1073\ \text{cm}^{-1}$ , and Si-O absorption from the silica in the rice husk was indicated at wavenumber  $541\ \text{cm}^{-1}$ . All wavenumber results are appropriate with Khan et al.<sup>[10]</sup>. According to the FTIR spectrum, the Carbon is still containing a functional group after activation. Remazol Yellow FG

degradation may be supported by the functional group on AC.

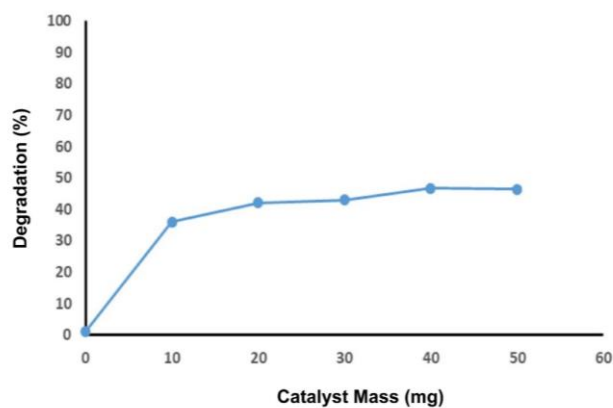
Figure 3 exhibited the DRS UV-Vis spectra of the  $\text{TiO}_2/\text{AC}$  catalyst.  $\text{TiO}_2/\text{AC}$  catalyst was analyzed using DRS UV-Vis to determine the band gap energy<sup>[11,12]</sup>. Carbon atoms can be doped into  $\text{TiO}_2$  to replace some O or Ti atoms. The dopant C could reduce the distance between the valence band and the conduction band of  $\text{TiO}_2$ . As the result, less energy is needed to excite electrons from the valence band to the conduction band<sup>[13]</sup>.



**Figure 3.** DRS UV-Vis spectrum of  $\text{TiO}_2/\text{AC}$  catalyst

According to the touch plot method based on DRS UV-Vis measurements, the  $\text{TiO}_2/\text{AC}$  catalyst has a 3.10 eV band gap. The measurement is carried out to figure out the band gap value of the material, so that it may be determined which light can be degraded later on. Theoretically, the band gap of anatase  $\text{TiO}_2$  is 3.2 eV. But, according to the results, the band gap was decreased after being doped with AC.

### 3.2. Effect of Catalyst Dosage



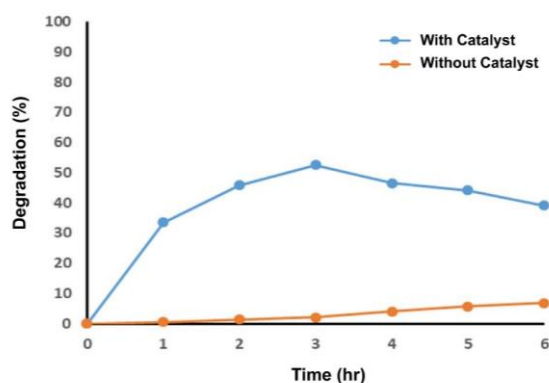
**Figure 4.** Effect of Catalyst Mass on Remazol Yellow FG Degradation with the condition: catalyst dosage of 10-50 mg, contact time of 1 hour, with initial Remazol Yellow FG concentration of 30 mg/L.

Figure 4 illustrates the effect of  $\text{TiO}_2/\text{AC}$  catalyst dosage towards the degradation of Remazol Yellow FG. The amount of  $\text{TiO}_2/\text{AC}$  catalyst of 10 mg to 40 mg increased the degradation percentage from 36.07% to 46.70%. But, with a 50 mg dosage the degradation percentage was decreased. This phenomenon occurs due the higher catalyst dosage would increase the suspension turbidity and diminish the amount of light to reach active surfaces<sup>[14-16]</sup>. Based on the results, the optimum catalyst dosage was 20 mg.

### 3.3. Effect of Contact Time

Figure 5 illustrates the contact time for degradation of Remazol Yellow FG using sonolysis with and without the addition of  $\text{TiO}_2/\text{AC}$  catalyst. Based on the results, the removal efficiency of the catalyst during the degradation with sonolysis enhanced very quickly during 1–3 hours, then it proceeded very slowly after 3 hours. We presume that the de-performance of degradation after 3 h is related to the excessive amount of  $\bullet\text{OH}$  and the reduced active sites of the catalyst. The high amount of  $\bullet\text{OH}$  may lead to competition with each other and the active sites become more saturated during the degrading Remazol Yellow FG after 3 hours. Our experiments yield similar results to Agarwal 2016<sup>[17]</sup>. It is also important to characterise the process of degradation in terms of kinetics and then the optimum time for treatment using sonication can be selected.

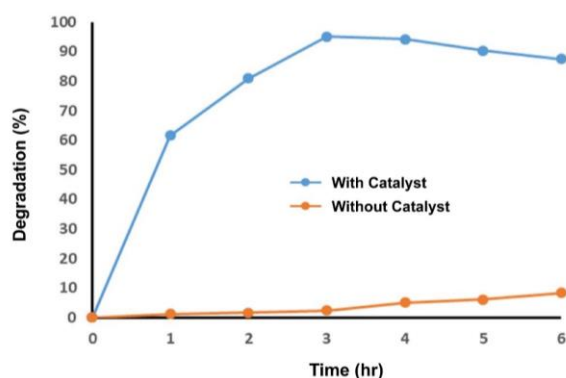
Sonolysis using a catalyst is more capable of degrading Remazol Yellow FG. The additional catalyst in sonolysis has been proven to increase the amount of  $\bullet\text{OH}$ . This phenomenon occurs in the following steps: (1) in sonolysis  $\bullet\text{OH}$  is produced through the nucleation, growth and collapse of microbubbles, and (2) the catalyst is able to provide active sites in cavitation by acting as a nucleation site<sup>[18-20]</sup>.



**Figure 5.** Effect of time on Remazol Yellow FG Degradation by sonolysis with the condition: catalyst dosage

of 20 mg, contact time of 1-6 hours, and initial Remazol Yellow FG concentration of 30 mg/L.

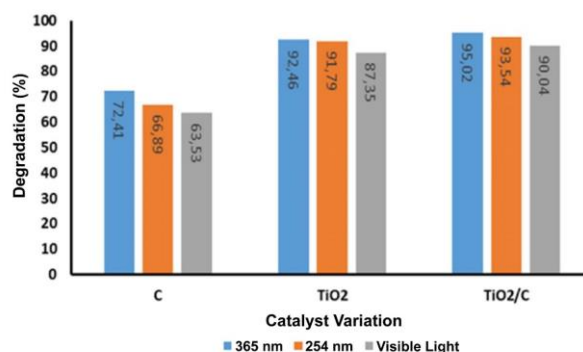
Figure 6 shows the increase in irradiation time also increases the Remazol Yellow FG degradation percentage. The reaction rate is significantly increased during 1–3 hours and reaches its maximum performance at 3 hours (95.02%). However, after 3 hours, the performance slightly decreased. The reaction rate decreases with irradiation time due to the competition between the reactant and the intermediate product. Additionally, the slowing process of degradation after a certain time limit is also affected by the difficulty in converting N-atoms of dye into oxidized nitrogen compounds<sup>[21]</sup> or the short life-time of photocatalyst because of active sites deactivation by strong by-products deposition<sup>[22,23]</sup>.



**Figure 6.** Effect of time on Remazol Yellow FG Degradation using Photolysis with the condition: catalyst dosage of 20 mg, contact time of 1-6 hours, and initial Remazol Yellow FG concentration of 30 mg/L.

### 3.4. Effect of Catalyst and Irradiation

The effect of the irradiation and catalyst used on the degradation of Remazol Yellow FG is presented in Figure 7. Based on the results, the degradation of Remazol Yellow FG using  $\text{TiO}_2/\text{AC}$  catalyst have the highest performance than using  $\text{TiO}_2$  or AC only. The addition of AC to  $\text{TiO}_2$  is able to reduce the band gap below 3.2 eV, therefore maximizing light absorption. This statement is in line with the DRS UV-Vis spectrum discussed earlier, in which  $\text{TiO}_2/\text{AC}$  has a band gap energy of 3.10 eV. Additionally, due to adsorption being the first step in photodegradation and AC having the ability to adsorb organic compounds, the degradation could increase<sup>[24]</sup>.

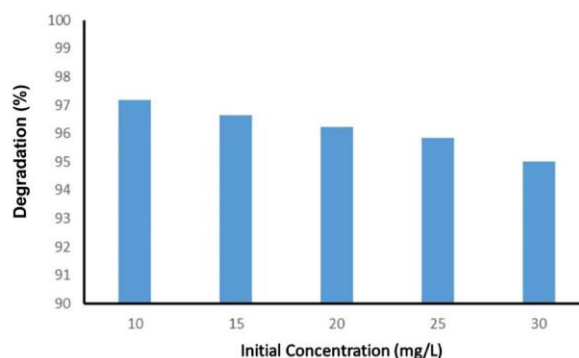


**Figure 7.** Effect of Irradiation and Catalyst on Remazol Yellow FG Degradation with the condition: catalyst dosage of 20 mg, contact time of 3 hours, and initial Remazol Yellow FG concentration of 30 mg/L.

Then, the percentage of degradation of Remazol Yellow FG using various radiation sources showed an insignificant result. UV light gives better degradation results than visible light. This is due to the shorter wavelength associated with a larger photon energy<sup>[25–27]</sup>. The results of this study also show that 254 nm UV light had lower degradation performance than 365 nm UV light, similar to the study by Akyol 2005<sup>[28]</sup>.

### 3.5. Effect of Initial Remazol Yellow FG Concentration

The effect of Remazol Yellow FG concentration was examined using a variety of concentrations ranging from 10 to 30 mg/L in 20 mg catalyst dosage. Figure 8 shows the degradation performance among the concentration tested and resulted in a decreased performance of degradation towards a higher concentration of Remazol Yellow FG. This deperformance event occurred due to the Remazol Yellow FG molecules competing more intensely for  $\text{TiO}_2/\text{AC}$  catalyst. Hence, to increase in the performance of degradation should be accompanied by an increase in catalyst dosage<sup>[29–31]</sup>.



**Figure 8.** Effect of Irradiation and Catalyst on Remazol Yellow FG Degradation with the condition: catalyst



dosage of 20 mg, contact time of 3 hours, and initial Remazol Yellow FG concentration of 30 mg/L.

#### 4. Conclusions

The performance of the  $\text{TiO}_2/\text{AC}$  catalyst for the degradation of Remazol Yellow FG has been successfully investigated. The parameters that affect degradation are compared. Based on the result of our experiment, the degradation of Remazol Yellow FG 10 mg/L using 20 mg  $\text{TiO}_2/\text{AC}$  catalyst is proven highly efficient with photolysis during 3 hours of contact time with UV 365 nm with 97,17% degradation percentage.

#### Conflict of interest

The authors declare no conflict of interest.

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