

CHAPTER IV

EXPERIMENTAL

Materials :

The photocatalyst TiO_2 , used throughout the experiment is TP-2 (product of Fujitan Co., anatase). Its surface area is $17.30 \text{ m}^2/\text{g}$. Dyes include acid black 1, acid yellow 17, new cocine, orange G, tartrazine, acid orange 7, and congo red. The chemical structure of these dye given in Figure 4.1. All solvents and other chemicals were reagent or spectrophotometric grade. They were used without further purification.



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| <p>Acid orange 7</p> <p>$\lambda_{\max} = 507.5 \text{ nm}$, MW = 350</p> <p>C.I. = 15510</p> | |
| <p>Orange G</p> <p>$\lambda_{\max} = 507.5 \text{ nm}$, MW = 452</p> <p>C.I. = 16230</p> | |
| <p>Tartrazine</p> <p>$\lambda_{\max} = 507.5 \text{ nm}$, MW = 512</p> <p>C.I. = 19140:1</p> | |
| <p>Acid yellow 17</p> <p>$\lambda_{\max} = 507.5 \text{ nm}$, MW = 551</p> <p>C.I. = 18965</p> | |
| <p>New cocchine</p> <p>$\lambda_{\max} = 507.5 \text{ nm}$, MW = 604</p> <p>C.I. = 16255</p> | |
| <p>Acid black 1</p> <p>$\lambda_{\max} = 507.5 \text{ nm}$, MW = 618</p> <p>C.I. = 20470</p> | |
| <p>Congo red</p> <p>$\lambda_{\max} = 507.5 \text{ nm}$, MW = 696</p> <p>C.I. = 22120</p> | |

Figure 4.1 Structure of studied azo dyes, λ_{\max} means wavelength at maximum absorption spectra of dyes in this study.

Apparatus and Method:

The apparatus for photocatalytic degradation was shown in Figure 4.2.

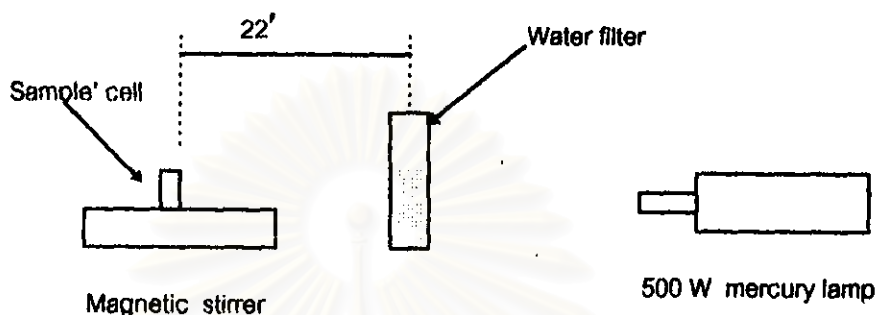


Figure 4.2 Photocatalytic degradation apparatus

The apparatus comprises of a 500 W mercury lamp, water filter with Pyrex glass windows, magnetic stirrer, and glass cell. Infrared ray and UV below 310 nm from light source eliminated by the filter.

Degradation experiment:

1. Study effect of amount of TiO_2 on the degradation of dye

Each sample cell comprises of 25 ml of 2×10^{-4} M of acid orange 7 with various amount of TiO_2 suspended.

2. Study effect of concentration of dye on the photocatalytic degradation

Each sample cell comprises of 75 mg of TiO_2 suspended in 25 ml of various concentrations of dye.

3. Study effect of initial pH of dye solution on photocatalytic degradation

Each sample cell comprises of 75 mg of TiO_2 suspended in 25 ml of various initial pH of 1×10^{-4} M of acid orange 7.

4. Study effect of chemical structure of dyes on photocatalytic degradation

Each sample cell comprises of 75 mg of TiO_2 suspended in 25 ml of 1×10^{-4} M of dye solution.

The cell was covered by a rubber plug with a hole for aeration. The cell was illuminated for specific period. After illumination, the suspension was filtered through a 0.22 μM millipore membrane. The filtrate was subjected to analysis.

Analysis:

1. The disappearance of dye was determined by HPLC with a inertsil ODS-3 column of GL Sciences Inc., and MD-910 multi-wavelength detector of Japan Spectroscopic Co., Ltd. The mobile phase was a mixture of methanol and 0.01 M of tetra-n-butylammonium chloride in ratio 1:3.
 2. The appearance of intermediate aromatic products was determined in same way.
 3. Intermediate organic acids, nitrate ions, nitrite ions, and sulfate ions were determined by IC 7000 series II Ion Chromatographic Analyzer of Yokogawa Analytical System equipped with a Excelpack CHA-E11 Column for organic acids and a Excelpack ICS A44 Column for anions. The mobile phase for the analysis of organic acids was 0.25 mM of Na_2SO_4 and 1 mM of H_2SO_4 and that for anions was 15 mM of H_2SO_4 and mix. 4 mM of Na_2CO_3 and NaHCO_3 .
 4. The appearance of ammonium ions was determined by Jasco 880- PU HPLC chromatograph with a Shodex IC Y-521 ion exchange column and Shodex CD-5 conductivity meter.
- Identification of intermediate and final products was made by comparing the retention time with that of pure sample.
5. The total organic carbon (TOC) eliminated by illumination was determined by TOC 500 Total organic carbon of Shimadzu.