EFFECT OF Fe$^{2+}$ CONCENTRATION ON DEGRADATION OF MALACHITE GREEN OXALATE AND NAPHTHOL BLUE BLACK BY PHOTO-FENTON SYSTEM

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INDUSTRIAL CHEMISTRY PROGRAMME
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2005
DECLARATION

I declare that this thesis is the result of my own research except as cited in references. This thesis has not been accepted for any degree and is not concurrently submitted in candidature of any degree.

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The effects of Fe$^{2+}$ concentration on photo-degradation of MGO and NBB by photo-Fenton system (Fe$^{2+}$/H$_2$O$_2$/UV) have been investigated. The experiment was conducted in a 400 mL cylindrical photo-reactor and the change in dyes concentration were monitored by measuring absorbance spectrophotometrically at $\lambda = 620$ nm and at $\lambda = 618$ nm, respectively. Results indicate that the degradation increased with Fe$^{2+}$ concentration. Comparatively, the degree of NBB degradation was higher than MGO. The highest degradation degree of NBB was obtained at 85.1% while MGO achieved the highest degree of degradation at 74.8%.
ABSTRAK

Kesan kepekatan Fe\(^{2+}\) dalam foto-degradasi MGO dan NBB oleh sistem foto-Fenton (Fe\(^{2+}/H_2O_2/UV\)) telah dikaji. Experimen dijalankan dengan menggunakan 400 mL reactor-foto berbentuk silinder dan perubahan kepekatan pewarna dianalisis secara spektrofotometri dengan mengukur nilai penyerapan masing-masing pada \(\lambda = 620\) nm dan pada \(\lambda = 618\) nm. Keputusan kajian menunjukkan degradasi meningkat dengan kepekatan Fe\(^{2+}\). Secara perbandingan, NBB dinyahwarnakan dengan lebih berkesan daripada MGO. NBB memperolehi peratus degradasi tertinggi pada 85.1\% manakala MGO mencapai peratus degradasi tertinggi pada 74.8\%.
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LIST OF SYMBOLS AND ABBREVIATIONS

\( \text{H}_2\text{O}_2 \) Hydrogen peroxide

\( \text{MGO} \) Malachite Green Oxalate

\( \text{NBB} \) Naphthol Blue Black

\( \text{Fe}^{2+} \) Ferrous ion

\( \text{Fe}^{3+} \) Ferric ion

\( \text{UV-Vis} \) Ultraviolet-Visible

\( \text{D}_{\text{Abs}} \) Degradation Degree

\( \lambda \) Wavelength

\( \lambda_{\text{max}} \) Maximum wavelength

\( \varepsilon \) Molar absorptivity

\( R^2 \) Scatter plot regression coefficient

\( k \) Rate constant

\( v \) Frequency

\( T \) Transmittance

\( P \) Intensity of Light

\( c \) Velocity of Light \((3.0 \times 10^8 \text{ ms}^{-1})\)

\( E \) Energy

\( h \) Planck constant \((6.63 \times 10^{-34} \text{ Js})\)
CHAPTER 1

INTRODUCTION

1.1 An Overview of the Textile Dyes Wastewater Treatment

The textile industry is one of the largest industries in Malaysia which grows drastically from a traditional based to a modern based industry. Yet, it is one of the major polluters to the environment due to the usage of dyes (Azmi et al., 1998). Over 700,000 tons of approximately 10,000 types of dyes and pigments are produced annually worldwide, in which about 20% are assumed to be discharged as industrial effluent during the textile dyeing processes (Azbar et al., 2003).

Effluents from the dyeing and finishing processes are known to contain strong colour; high amounts of surfactants, heavy metals and chlorinated organic; dissolved solids; fluctuating temperature; high and unstable pH; and high chemical oxygen demand (COD) (Arslan and Balcioglu, 1999; Cisneros et al., 2002). Hence, textile waste effluents are one of the wastewater that is not easily amenable by chemical or biological treatment (Moh et al., 2003). In addition, the problem of coloured effluent has been a major challenge and an integral part of textile effluent treatment as a result
of stricter environmental regulations (Azbar et al., 2003). Conventional wastewater treatment techniques (biological, physical and chemical processes) such as flocculation by chemical reagent and activated carbon adsorption are insufficient and ineffective, for the fact that complete removal of colour, dissolved solids, and refractory chemicals is not possible by this method, unless accompanied by advanced treatment techniques. The development of new treatment process, which is more effective in eliminating the dyes from wastewater, is necessary in term of decolourization as well as mineralization (Ince and Tezcanli, 1999; Moh et al., 2003).

The chemical limitations of conventional chemical oxidation techniques can be overcome by the advanced oxidation processes (AOPs). AOPs have been successful in degrading most of the organic compounds present in polluted water. In AOP, the hydroxyl radicals (•OH) are generated in solution and responsible for the oxidation and mineralization of the organic pollutants to water and carbon dioxide. Various advanced oxidation processes such as O3/UV, O3/H2O2, UV/H2O2 and Fenton’s reagent (Fe2+/H2O2) has been applied in wastewater treatment (Ghaly et al., 2000). Among these processes, Fenton’s reagent has given a promising and attractive treatment method for the effective decolourization and degradation of dyes (Malik and Saha, 2002). However, the Fenton reaction has two limitations, large production of iron sludge and slow reduction of ferric ions by H2O2 in their application (Lee et al., 2002).

The photochemical advanced oxidation processes including photolysis and photocatalysis are gaining increasing importance for degradation of most of the organic pollutants (Ghaly et al., 2000). The photo-Fenton process (Fe2+/H2O2/UV) involves the hydroxyl radical formation in the reaction mixture through photolysis of
hydrogen peroxide (H\textsubscript{2}O\textsubscript{2}/UV) and Fenton reaction (Fe\textsuperscript{2+}/H\textsubscript{2}O\textsubscript{2}) (Shyh et al., 1999). This illumination of Fenton system by UV-Vis was suggested to overcome the limitations of Fenton reaction (Lee et al., 2002). Photo-Fenton process represents an efficient and cheap method for wastewater treatment, and produces more hydroxyl radicals in comparison with Fe\textsuperscript{2+}/H\textsubscript{2}O\textsubscript{2} or UV/H\textsubscript{2}O\textsubscript{2} system, thus promoting the rate of degradation of various organic pollutants (Ghaly et al., 2000). The key features of the Fenton system are believed to be its reagent conditions (i.e. concentration of Fe\textsuperscript{2+}, Fe\textsuperscript{3+} and H\textsubscript{2}O\textsubscript{2}) and the reaction characteristics, which are the pH, temperature and the quantity of organic and inorganic constituents. Because these parameters determine the overall reaction efficiency, it is important to understand the mutual relationships between these parameters in terms of hydroxyl radical production and consumption (Neyens and Baeyens, 2002).

1.2 Objectives

The objectives of this study are:

a. to evaluate the effects of Fe\textsuperscript{2+} towards the degradation of Malachite Green Oxalate and Naphtol Blue Black using photo-Fenton system (Fe\textsuperscript{2+}/H\textsubscript{2}O\textsubscript{2}/UV).

b. to compare the degradation of Malachite Green Oxalate and Naphtol Blue Black dye using photo-Fenton system.
1.3 Scope of Research

The scope of the research is to examine the effect of Fe\(^{2+}\) in the degradation of a basic dye solution and an acid dye solution using photo-Fenton system consists of Fenton-reagent method with UV light (Fe\(^{2+}/\text{H}_2\text{O}_2/\text{UV}\)). This experiment is carried out in lab-scale using cylindrical photo-reactor (Duran's Pyrex, 400 mL capacity) with the presence of UV irradiation. Two types of simulated textile effluent are prepared from Malachite Green Oxalate salt and Naphtol Blue Black salt, respectively as the organic pollutant. This research has studied three main operational parameters (dye concentration, Fe\(^{2+}\) ion concentration and \text{H}_2\text{O}_2 concentration). The degradation of each dye was obtained by the analysis of samples withdrawn from the reservoir periodically by UV-Visible spectrophotometer. Graphs of Abs against reaction time and also ln Abs against reaction time will be plotted to evaluate the degree of degradation and the kinetic rate order of the chemical reactions.
CHAPTER 2

LITERATURE REVIEW

2.1 Colour and Light

The visible region is a small part of the electromagnetic spectrum, and it is the region of wavelengths that can be seen by the human eye, that is, where the electromagnetic radiation (light) appears as a colour. The visible region consists of colours ranging from violet, beginning at around 380 nm, to red, ending at approximately 780 nm (Christian, 2004; Pavia et al., 2001; Shugar and Ballinger, 1996).

When polychromatic light (white light), which contains the whole spectrum of wavelengths in the visible region, is passed through an object, the object will absorb certain wavelengths of the light within that range, leaving the unabsorbed wavelengths to be transmitted. These residual transmitted wavelengths will be seen as a colour. This is complementary to the absorbed colours (Pavia et al., 2001; Christian, 2004; Fritz and Schenk, 1987).

Table 2.1 illustrates the relationship between the wavelength of the light absorbed by a substance and the colour perceived by an observer. These wavelengths
are taken from a study originating at the National Bureau of Standards (Ewing, 1985; Christian, 2004; Fritz and Schenk, 1987).

Table 2.1 Colours in Different Wavelength Regions

<table>
<thead>
<tr>
<th>Absorbed Colour</th>
<th>Wavelength of Light Absorbed (nm)</th>
<th>Observed Colour</th>
</tr>
</thead>
<tbody>
<tr>
<td>Violet</td>
<td>400-465</td>
<td>Yellow-green</td>
</tr>
<tr>
<td>Blue</td>
<td>465-482</td>
<td>Yellow</td>
</tr>
<tr>
<td>Greenish Blue</td>
<td>482-487</td>
<td>Orange</td>
</tr>
<tr>
<td>Blue-Green</td>
<td>487-493</td>
<td>Red-Orange</td>
</tr>
<tr>
<td>Bluish Green</td>
<td>493-498</td>
<td>Red</td>
</tr>
<tr>
<td>Green</td>
<td>498-530</td>
<td>Red-Purple</td>
</tr>
<tr>
<td>Yellowish Green</td>
<td>530-559</td>
<td>Reddish Purple</td>
</tr>
<tr>
<td>Yellow-Green</td>
<td>559-571</td>
<td>Purple</td>
</tr>
<tr>
<td>Greenish Yellow</td>
<td>571-576</td>
<td>Violet</td>
</tr>
<tr>
<td>Yellow</td>
<td>576-580</td>
<td>Blue</td>
</tr>
<tr>
<td>Yellowish Orange</td>
<td>580-587</td>
<td>Blue</td>
</tr>
<tr>
<td>Orange</td>
<td>587-597</td>
<td>Greenish Blue</td>
</tr>
<tr>
<td>Reddish Orange</td>
<td>597-617</td>
<td>Blue-Green</td>
</tr>
<tr>
<td>Red</td>
<td>617-780</td>
<td>Blue-Green</td>
</tr>
</tbody>
</table>

2.1.1 UV-Visible Absorption Spectroscopy

The ultraviolet, UV and visible light region extends from 200 nm to 780 nm. Beer’s Law explains the relationship between the absorption of UV-Visible radiation and the concentration of the analyte in a solution (Harvey, 2000). The following equation is the common form of Beer’s Law, where Absorbance, A of the solution can be obtained:

\[ A = \log \left( \frac{P_0}{P} \right) = \varepsilon \, b \, l \] (2.1)
Where,

\[ P_0 = \text{Intensity of incident light (at a particular wavelength)} \]
\[ P = \text{Intensity of transmitted light through a sample} \]
\[ b = \text{molar concentration of absorbing species (mol/L)} \]
\[ l = \text{layer thickness or length of the sample cell (cm)} \]

The absorbance is directly proportional to the concentration. The constant, \( e \) is called the absorptivity and is dependent on the wavelength and the nature of the absorbing species (Pavia et al., 2001; Fritz and Schenk, 1987; Ewing, 1985; Christian, 2004). Beer's law valid only for low concentration of analyte. At higher concentration, individual particles of analyte no longer behave independently with each other (Harvey, 2000). The ratio \( P / P_0 \) is equivalent to transmittance, \( T \), where as:

\[ \log T = -A \quad (2.2) \]

Thus, the absorbance can be measured experimentally by determining the ratio of the incident to emergent intensities taking the logarithm.

### 2.1.2 Photon Absorption by Molecules

As a result of energy absorption in UV-Visible regions, atoms and molecules will involve in the promotion of electrons in \( \sigma, \pi, \) and \( n \) orbital, which transit from a state of low energy (ground state) to a state of higher energy (excited state) (Pavia et al., 2001). The increase in energy is equal to the energy of the absorbed radiation \( (hv) \). The energy
of a quantum of radiation, called the photon, is related to the frequency or wavelength where:

\[ \Delta E = E_{\text{excited}} - E_{\text{ground}} = h \nu = h \left( \frac{c}{\lambda} \right) \]  

(2.3)

\( E \) is the energy of the photon in ergs and \( h \) is Planck's constant, \( 6.62 \times 10^{-34} \) joule-second (J-s). \( \lambda \) is the wavelength (cm), \( \nu \) is the frequency (s\(^{-1}\) or Hz), and \( c \) is the velocity of light (3 \( \times 10^{10} \) cm/s). It is apparent that the shorter the wavelength or the greater the frequency, the greater the energy (Hollas, 2002; Christian, 2004; Fritz and Schenk, 1987).

The internal energy of a molecule, \( E_i \) (excluding translation energy) can be written as:

\[ E_i = E_{\text{rotation}} + E_{\text{vibration}} + E_{\text{electron}} \]  

(2.4)

The colour appearance of dyes and pigments are basically explainable by the theory of photon absorption in UV-Visible regions, which is a field of quantum chemistry study. The transitions that result in the photon absorption in UV-Visible region are transitions between electronic energy levels (Pavia et al., 2001; Abrahart, 1977).

When an atom or molecule absorbs UV-Visible radiation, it undergoes a change in valence electron configuration. The valence electron in organic molecules and inorganic anions occupied quantized sigma bonding (\( \sigma \)), pi bonding (\( \pi \)) and non-
bonding (n) molecular orbital. There are 4 types of transitions between quantized energy levels account for molecular UV-Visible regions, as shown in Table 2.2 (Harvey, 2000):

Table 2.2 Types of electronic transitions, wavelength range and examples

<table>
<thead>
<tr>
<th>Transition</th>
<th>Wavelength range (nm)</th>
<th>Examples</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\sigma \rightarrow \sigma^*$</td>
<td>$&lt; 200$</td>
<td>C=C, C–H</td>
</tr>
<tr>
<td>$n \rightarrow \sigma^*$</td>
<td>160-260</td>
<td>$H_2O, CH_3OH, CH_3Cl$</td>
</tr>
<tr>
<td>$\pi \rightarrow \pi^*$</td>
<td>200-500</td>
<td>C=C, C=O, C=N, C≡C</td>
</tr>
<tr>
<td>$n \rightarrow \pi^*$</td>
<td>250-600</td>
<td>C=O, C=N, N=N, N=O</td>
</tr>
</tbody>
</table>

All coloured organic compounds contain carbon-carbon double bond, which are formed by $\sigma$ bond and $\pi$ bond. According to Pauli Exclusion Principle, these bonds are molecular orbitals. Each orbital occupied two electrons and have opposite spin (Pavia et al., 2001; Moh, 2002). As UV-Visible radiation is absorbed, $\sigma$, $\pi$ or $n$ electron (in ground state) is promoted to occupy an empty anti-bonding orbital, $\sigma^*$ or $\pi^*$ which occur at higher energy level. Electrons are positioned in non-bonding orbitals (n) when molecule contains lone-paired electrons of nitrogen or oxygen atom. Each of these orbitals has specific energy level. Figure 2.1 below shows the energy level diagram according to the type of molecular orbital (Moh, 2002; Suppan, 1994; Atkins, 2001):
REFERENCES


Roques, H., 1996. *Chemical water treatment: Principles and Practice*. VCH Publisher Inc. USA.


