Synthesis of TiO₂ by Hydrolysis/Electrochemical to Reduce Hazardous Disinfecting Materials
Adrian Nur ¹*, Arif Jumari¹, Nazriati Nazriati² and Fauziatul Fajaroh ²

¹ Departement of Chemical Engineering, Universitas Sebelas Maret, Jl. Ir. Sutami 36A, Surakarta, Indonesia
² Departement of Chemistry, Universitas Negeri Malang
E-mail: *adriannur@staff.uns.ac.id (Corresponding author)

Abstract. The TiO₂ photo-catalysis can be used for the purpose of disinfectant purpose. In this work, the TiO₂ prepared by hydrolysis-electrochemical method was used to produce disinfectant to replace and reduce conventional material disinfectant. The synthesis of TiO₂ was occured at constant voltage of 10 V for 2.5 hours under constant stirring and room temperature. The product of synthesis was analysed by scanning electron microscopy, energy dispersive X-ray spectrometry, and X-ray diffractometer. The performance of disinfectant was done with inactivation of bacteria E coli in solid media. The phase of TiO₂ particle produced shows anatase and rutile phase. The TiO₂ resulted from hydrolysis/electrochemical method can be used to reduce HCl for disinfectant. The results of testing disinfectant for inactivation of bacteria E.coli in solid media show that disinfectant from HCl/TiO₂ is the most efective to inactivation of bateria E. coli. Treatment with ultraviolet rays resulted less number of bacteria than sunlight.

Keywords: TiO₂, electrochemical, hydrolysis, disinfectant
1. Introduction

The use of conventional material disinfectants such as alcohols, aldehydes, and chlorine compounds are less effective to inactivate some bacteria, especially \( E. \ coli \) [1]. These materials are environmentally unfriendly and possibly containing toxic materials. The \( \text{TiO}_2 \) powder has been known to have photo-catalyst properties [2]. The \( \text{TiO}_2 \) photo-catalyst can be used to disinfectant purpose because \( \text{TiO}_2 \) is a non-toxic substance that is widely used in products like toothpastes and cosmetics [3, 4]. There are several methods to produce \( \text{TiO}_2 \) such as hydrothermal method, sol gel, precipitation, solid state, and solvothermal [5–7]. In this work, \( \text{TiO}_2 \) was produced from \( \text{TiCl}_4 \) by hydrolysis and electrochemical [8, 9]. Using the electrochemical, the phase of particle can be controlled by the current, time, and concentration of reactant [10–13]. The \( \text{TiO}_2 \) resulted by hydrolysis-electrochemical methods was used to produce disinfectant to replace and reduce conventional material disinfectant.

2. Experimental

2.1. Synthesis of \( \text{TiO}_2 \)

The solution of 100 mL for electrolysis consists of 0.1 M \( \text{TiCl}_4 \) (Merck) and 50 % ethanol (Merck). The electrolysis was done in a glass as an electrolysis cell. The cell had two carbon electrodes with 3 cm of distance between of electrodes. The dimensions of electrodes were \( 5 \times 2 \times 0.25 \) cm. The carbon electrodes were immersed in solution at a depth of 2 cm and connected to DC power supply (Zhaoxin PS-3005D). The synthesis of \( \text{TiO}_2 \) was done at constant voltage 10 V for 2.5 hours under constant stirring and room temperature. Fig 1 shows the schematic diagram of the experimental set up. Electrosynthesis resulted the suspension. This suspension was aged at 48 hours. The particles resulted after filtration was dried at 150°C for 2 hours, washed 2 times, and dried again at 60 °C for 6 hours. The particles then were used to disinfectant production.

![Fig. 1. The schematic diagram of the experimental set up](image.png)

The scanning electron microscopy (Inspect S40, FEI) was used to observe the morphologies of the particles and the energy dispersive X-ray spectrometry (EDX) analysis. The X-ray diffractometer (Shimadzu 6000) was used to observe the X-ray diffraction pattern of the particles.

2.2. Disinfectant Preparation

The disinfectant was resulted from 500 mL of aquadest, 20 grams of oxalic acid (Merck), V mL HCl 37% (Merck), 5 grams of NaOH (Merck) and m grams of \( \text{TiO}_2 \) particles resulted from electrolysis. The quantity various of HCl and \( \text{TiO}_2 \) were shown in Table 1.
Table 1. The Quantity Various of HCl and TiO\textsubscript{2} to Disinfectant Production.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Quantity of HCl 37%</th>
<th>Quantity of TiO\textsubscript{2}</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>10 mL</td>
<td>10 grams</td>
</tr>
<tr>
<td>2</td>
<td>0 mL</td>
<td>20 grams</td>
</tr>
<tr>
<td>3</td>
<td>20 mL</td>
<td>0 grams</td>
</tr>
</tbody>
</table>

2.3. Disinfectant Performance Test

Testing disinfectant was done with inactivation of bacteria *E. coli* in solid media. Before testing disinfectant, all materials and equipments were sterilized. Bacteria were bred and incubated for 24 hours at 37 °C. Each sample of disinfectant was added to the bacterial suspension. This suspension was grown on a solid medium in petri dishes. There were two treatments for the bacteria on solid medium that was given sunlight and ultra violet rays (Table 2). The number of bacterial colonies counted with colony counter count.

Table 2. The Treatment on Bacteria in Solid Media.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Sunlight</th>
<th>Ultraviolet rays</th>
</tr>
</thead>
<tbody>
<tr>
<td>1a</td>
<td>Yes</td>
<td>No</td>
</tr>
<tr>
<td>1b</td>
<td>No</td>
<td>Yes</td>
</tr>
<tr>
<td>2a</td>
<td>Yes</td>
<td>No</td>
</tr>
<tr>
<td>2b</td>
<td>No</td>
<td>Yes</td>
</tr>
<tr>
<td>3a</td>
<td>Yes</td>
<td>No</td>
</tr>
<tr>
<td>3b</td>
<td>No</td>
<td>Yes</td>
</tr>
</tbody>
</table>

3. Result and Discussion

The homogeneous solution was resulted from above composition of solution. When the electrodes was connected to DC power supply, gas bubble appeared at the surface electrodes. The reaction of water oxidation and reduction resulted oxygen and hydrogen gas according these reaction:

\begin{align*}
2\text{H}_2\text{O} & \rightarrow \text{O}_2(\text{g}) + 4\text{H}^+ + 4\text{e}^- \quad (1) \\
2\text{H}_2\text{O} + 2\text{e}^- & \rightarrow \text{H}_2(\text{g}) + 2\text{OH}^- \quad (2)
\end{align*}

Beside that, the reactions from TiCl\textsubscript{4} to TiO\textsubscript{2} was occured:

\begin{align*}
\text{TiCl}_4(\text{aq}) + \text{O}_2(\text{g}) & \rightarrow \text{TiO}_2(\text{s}) + 2\text{Cl}_2(\text{g}) \quad (3) \\
\text{TiCl}_4(\text{aq}) + 2\text{H}_2\text{O} & \rightarrow \text{TiO}_2(\text{s}) + 4\text{HCl(\text{aq})} \quad (4)
\end{align*}
After electrolysis, the white precipitate was resulted. This precipitate was investigated with EDX measurements. The EDX pattern was shown in Fig. 2. The peaks of Ti, O and Cl elements appeared in the EDX pattern. The product of electrolysis contains TiO₂ with Cl as impurity from residual reactant. Fig 3 shows the XRD pattern from particle resulted. The XRD pattern shows that the particle was TiO₂ in rutile and anatase phase. The existence of rutile phase in the XRD pattern is evidenced from presence of the (110) peak at 27.3° (2θ) (JCPDS No 75-1753). The anatase phase can be identified from its (110) anatase peaks located at 25.3° (2θ) (JCPDS No 84-1286).

The results of testing disinfectant for inactivation of bacteria *E. coli* in solid media show in Table 3. From Table 3, the number of bacteria in sample 1 is less than the samples 2 and 3. Its indicate that disinfectant from HCl and TiO₂ is the most effective to inactivation of bacteria *E. coli*. Comparisson of sample 2 and 3
shows that TiO₂ is better inactivation of bacteria than HCl. But the best desinfectant is from HCl and TiO₂. The TiO₂ resulted from electrosynthesis can be used to reduce HCl. From samples 1, 2 and 3, treatment with ultraviolet rays resulted less number of bacteria than sunlight.

### Table 3. The Results of Testing Disinfectant.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Treatment</th>
<th>Number of bacteria</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample 1a</td>
<td>HCl + TiO₂</td>
<td>Sunlight</td>
</tr>
<tr>
<td>Sample 1b</td>
<td>HCl + TiO₂</td>
<td>No</td>
</tr>
<tr>
<td>Sample 2a</td>
<td>TiO₂</td>
<td>Yes</td>
</tr>
<tr>
<td>Sample 2b</td>
<td>TiO₂</td>
<td>Yes</td>
</tr>
<tr>
<td>Sample 3a</td>
<td>HCl</td>
<td>Yes</td>
</tr>
<tr>
<td>Sample 3b</td>
<td>HCl</td>
<td>No</td>
</tr>
</tbody>
</table>

### 4. Conclusion

In this work, the particle of TiO₂ was produced from TiCl₄ by hydrolysis and electrochemical. The phase of TiO₂ particle is anatase and rutile. The TiO₂ resulted from electrosynthesis can be used to reduce HCl for desinfectant. Treatment with ultraviolet rays resulted less number of bacteria than sunlight.

### References


