Article

Synthesis of TiO_2 by Hydrolysis/Electrochemical to Reduce Hazardous Disinfecting Materials

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Abstract. The TiO_2 photo-catalysis can be used for the purpose of disinfectant purpose. In this work, the TiO_2 prepared by hydrolysis-electrochemical method was used to produce disinfectant to replace and reduse conventional material disinfectant. The synthesis of TiO_2 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 desinfectant was done with inactivation of bacteria *E coli* in solid media. The phase of TiO_2 particle producted shows anatase and rutile phase. The TiO_2 resulted from hydrolysis/electrohemical method can be used to reduce HCl for desinfectant. The results of testing disinfectant for inactivation of bacteria *E coli* in solid media show that disinfectant from HCl/TiO_2 is the most efective to inactivation of bateria *E. coli*. Treatment with ultraviolet rays resulted less number of bacteria than sunlight.

Keywords: TiO_2, electrochemical, hydrolysis, disinfectant

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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 contaming toxic materials. The TiO_2 powder has been known to have photo-catalyst properties [2]. The TiO_2 photo-catalyst can used to disinfectant purpose because 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 TiO_2 such as hydrothermal method, sol gel, precipitation, solid state, and solvotermal [5–7]. In this work, TiO_2 was produced from $TiCl_4$ 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 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 TiO_2

The solution of 100 mL for electrolysis consists of 0.1 M $TiCl_4$ (Merck) and 50 % ethanol (Merck). The electrolysis was done in a glass as an electrosynthesis cell. The cell had two carbon electrodes with 3 cm of distance between of electrodes. The dimensions of electrodes were 5 × 2 × 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 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.

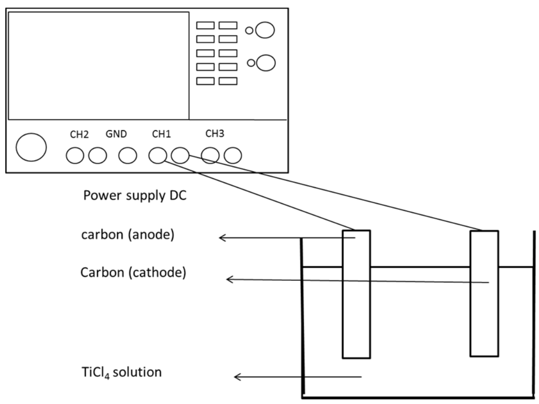

Fig. 1. The schematic diagram of the experimental set up

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 TiO_2 particles resulted from electrolysis. The quantity various of HCl and TiO_2 were shown in Table 1.

Table 1. The Quantity Various of HCl and TiO_2 to Disinfectant Production.

	The quantity of HCl 37 %	The quantity of TiO_2
Sample 1	10 mL	10 grams
Sample 2	0 mL	20 grams
Sample 3	20 mL	0 grams

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.

	Sunlight	Ultraviolet rays
Sample 1a	Yes	No
Sample 1b	No	Yes
Sample 2a	Yes	No
Sample 2b	No	Yes
Sample 3a	Yes	No
Sample 3b	No	Yes

3. Resuld 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:

$$2H_2O \rightarrow O_{2(g)} + 4H^+ + 4e^- \quad (1)$$

$$2H_2O + 2e^- \rightarrow H_{2(g)} + 2OH^- \quad (2)$$

Beside that, the reactions from $TiCl_4$ to TiO_2 was occured:

$$TiCl_{4\,(aq)} + O_{2\,(g)} \rightarrow TiO_{2\,(s)} + 2Cl_{2\,(g)} \quad (3)$$

$$TiCl_{4\,(aq)} + 2H_2O \rightarrow TiO_{2\,(s)} + 4HCl_{\,(aq)} \quad (4)$$

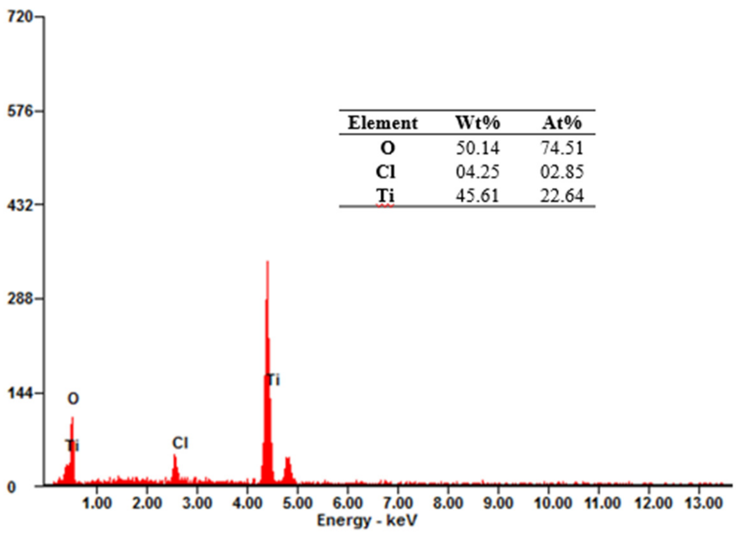

Fig. 2. Energy dispersive X-ray spectrometry (EDX) of TiO_2.

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 TiO_2 with Cl as impurity from residual reactant.
Fig 3 shows the XRD pattern from particle resulted. The XRD pattern shows that the particle was TiO_2 in rutile and anatase phase. The exixtence of rutile phase in the XRD pattern is evidenced from presence of the (110) peak at 27.3^{O} (2θ) (JCPDS No 75-1753). The anatase phase can be identified from its (110) anatase peaks located at 25.3^{O} (2θ) (JCPDS No 84-1286).

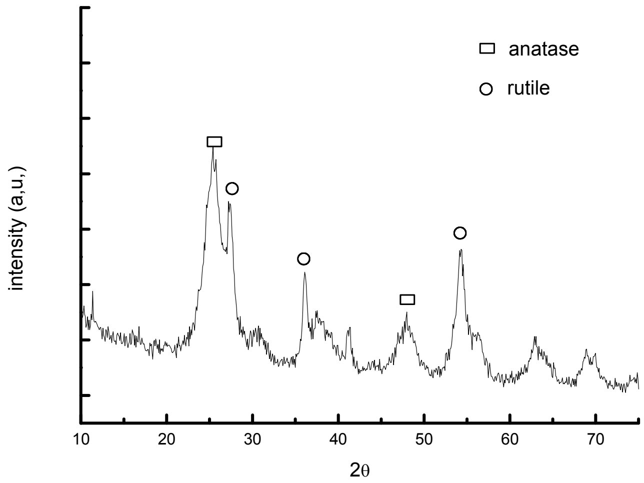

Fig. 3. XRD patterns for particles of TiO_2.

The SEM micrograph of particle resulted show that the particle was agglomeration and sperical shape. The size of agglomeration was about 1 µm (Fig. 4).

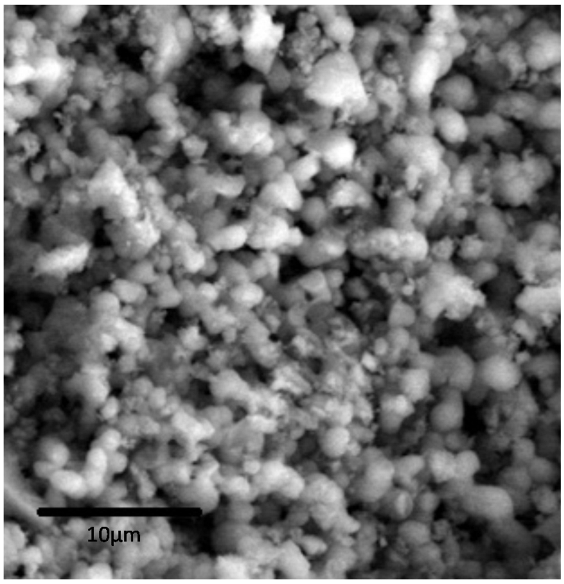

Fig. 4. SEM images of the particles.

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 TiO_2 is the most efective to inactivation of bateria *E. coli*. Comparisson of sample 2 and 3 shows that TiO_2 is better inactivation of bacteria than HCl. But the best desinfectant is from HCl and TiO_2. The TiO_2 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.

Sample		Treatment		Number of bacteria
		Sunlight	Ultraviolet rays	
Sample 1a	HCl + TiO_2	Yes	No	113 ± 12.6
Sample 1b	HCl + TiO_2	No	Yes	101 ± 17.2
Sample 2a	TiO_2	Yes	No	137 ± 25.1
Sample 2b	TiO_2	No	Yes	125 ± 18.3
Sample 3a	HCl	Yes	No	156 ± 32.5
Sample 3b	HCl	No	Yes	145 ± 22.9

4. Conclusion

In this work, the particle of TiO_2 was produced from $TiCl_4$ by hydrolysis and electrochemical. The phase of TiO_2 particle is anatase and rutile. The TiO_2 resulted from electrosynthesis can be used to reduce HCl for desinfectant. Treatment with ultraviolet rays resulted less number of bacteria than sunlight.

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