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CONTINUOUS-FLOW DISINFECTION REACTOR FOR DRINKING WATER USING GEL-DERIVED Ag-TiO_2-SiO_2 CATALYST UNDER UV-C LIGHT

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***Abstract*— Clean water for drinking and cooking has been a serious issue in rural areas of Vietnam, which is mainly due to high-cost treatment of the available water sources in such areas. In order to decrease the treatment cost, gel-derived Ag-TiO_2-SiO_2 thin film coated on glass beads were used as disinfection materials under UV-C light in this work. The photocatalyst was used in a continuous-flow lab-scale reactor to disinfect *E.coli* in water. It was found that the bactericidal activity of this system using the gel-derived Ag-TiO_2-SiO_2 photoatalyst under UV-C irradiation was superior to those using UV-C irradiation alone, a combination of bare glass beads and UV-C irradiation. By using gel-derived Ag-TiO_2-SiO_2thin film, the above reactor was observed to remove 6-log of *E.coli* under UV-C within the hydraulic retention time of 6 minutes.The reactor was also operated with Mekong river water (includes Tien river and Hau river) with the inlet microbial loading of Tien river (after filtering) from 50 – 780 CFU *Coliform*/100mL and that of Hau river (after filtering) from 100 – 260 CFU *Coliform* /100 mL. The optimum flow rate of Mekong river water for *Coliform* disinfection was determined to be 15 mL/min (hydraulic retention time of 9 minutes) which revealed potential application of this photocatalyst in an energy-saving technology for drinking water.**

Keywords*— gel-derived Ag-TiO2-SiO2; E.colidisinfection; UV-C lamp; continuous-flow reactor

I. Introduction

Waterborne pathogens, including viruses, bacteria, and protozoa, are responsible for 3.5 billion cases of diarrhea each year and 1.8 million deaths as a result of contaminated drinking waterall over the world [1]. In Vietnam, chorela diseases are estimated to cause number of mortality rate among children (1,316) each year due to inappropriate sanitation and microbially polluted drinking water. Carrying out National Target Programme on Water Supply and Sanitation in Phase 1 (1998 – 2005) and Phase 2 (2006 – 2011), international support and active participation of people, a considerable part of infrastructure system for rural water supply and sanitation was developed in which remote and poor areas got priorities in investment [2].The rate of population in rural areas of Vietnam having access to water supply and sanitation has increased. The achievements, however, are still very modest, especially for drinking water.

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Of the major types of disinfection treatments in Vietnam, chemical treatment such as chlorination has been enormously applied. However, it is of wide concern that the disinfection methods have serious environmental drawbacks, as chlorine can react with natural organic matter or dissolved organic carbon to form recalcitrant disinfection by-products (DBPs) i.e.,THMs (chloroform, bromodichloromethane, dibromochloromethane, andbromoform) [3, 4]. UV treatment seems more attractive but is limited by the fact that it can only treat surface waters or the regions in close proximity of the surfaces [5]. UV light oxidize some part of DNA only; thus, the natural phenomenon of DNA replication removes the errors after a particular time and the bacteria start to grow again [6]. An additional approach to UVC enhancement is the use of semiconductor photocatalysis which uses light along with a semiconductor material to produce reactive oxygen species (ROS) that destroy organic pollutants in water and inactivate pathogenic microorganisms [7, 8, 9]. Photocatalytic activity of titania has been reported by many researchers [10, 11, 12, 13]. A number of elements have been used as dopants (such as Ag, Fe, Cr, Pd, Cu, and Pt) to modify TiO_2 to improve the TiO_2 photocatalytic activity. Among these ions, Ag^+ is getting more attention than others because of its capability to bind, damage, and alter the functionalities of bacterial cell wall membrane, which are slightly negative [14, 15, 16, 17].

In this work, we report on the continuous-flow disinfection reactor of water using Ag-TiO_2-SiO_2 immobilized on glass beads. The reactor was operated to determine their disinfection activities and effects of flow rate, pH, initial bacterial population under UV-C irradiations. In addition, the purpose of this study was to test if Ag-TiO_2-SiO_2 coated glass beads would give rise to an increase in the disinfection efficiency under UVC irradiation through an on-site study in Mekong Delta. This photocatalysis reactor would be a simple point-of-use method for reducing the risk of using unsafe water.

II. Materials and methods

A. *Preparation of Ag-TiO_2-SiO_2 nanomaterials coated glass beads*

Ag-TiO_2-SiO_2 thin films coated on glass beads was prepared by sol-gel method. The detailed procedures of preparation of Ag-TiO_2-SiO_2 nanomaterials (Ag-TiO_2-SiO_2) were mentioned

in our previous research [18]. Briefly, four solutions were prepared: the first solution, S1, was prepared by mixing a mixture of ethanol (Prolabo, France) and *iso*-propanol (Prolabo, France) (solvent mixture, volume ratio of ethanol and *iso*-propanol = 1:1) with water and nitric acid; the second one, S2, included the solvent mixture, water, and Tetra-ethylorthosilicate (TEOS, 98%, Merck, Germany); the third solution, S3, was a mixture of the solvents and *tetra-iso* propyl orthotitanate (TTIP, Merck, Germany); and the last one , S4, included the solvent mixture, water, nitric acid (Merck, Germany) and silver nitrate ($AgNO_3$, Xilong, China) (mol ratio of silver to TiO_2 is 1%). The above solutions were then mixed and refluxed. The obtained sol-gel solution was hydrothermally treated in a lab-made autoclave at 150°C for 10 h. Solvents were then removed at 50°C using a vacuum evaporation to collect an aerogel. The aerogel was dried in an oven at 105°C for 2 h and then calcined at 550°C. The TiO_2 : SiO_2 weight ratio was controlled at 90:10 [19].

The Ag-TiO_2-SiO_2 thin films were prepared by a conventional dip-coating technique. Firstly, glass beads (0.4-0.9 mm) were washed by ultrasonication and acetone. They were then dried at 110°C in an oven for 1 h. Secondly, glass beads were dipped in sol gel Ag-TiO_2-SiO_2 and then dried at 110°C in an oven for 2 h and subsequently calcined at 550◦C in a muffle furnace for 2 h.

Crystallizes of catalyst were measured by using X-ray diffraction (Rikagu, Cu Kα). The surface morphology and thickness of thin films were obtained by using Scanning electron microscopy (SEM, JEOL JMS-7410F, Japan). The composites produced were characterized by UV–vis spectroscopy (Origin Jasco). The mean particle sizes can be calculated by the Scherrer's equation:

$$D = \frac{0.9\lambda}{\beta\cos\theta}$$

where λ =0.1541 (nm), β is the half-diffraction angle (rad), θ is the half-peak width (degree), and D is the diameter of the crystalline particle (nm) [20].

B. Performance of continuous-flow disinfection reactor

2.1. Reactor

The continuous-flow disinfection reactor used during disinfection tests was a glass tube coupled with a UVC lamp (Philip) as shown in Figure 1a. The illuminated volume for the model configuration with and without glass beads were 150 mL and 630 mL, respectively. Figure 1b shows the Ag-TiO_2-SiO_2 coated glass beads packed in the glass tube with design parameters. The photocatalytic activities of the films were evaluated on their bactericidal activities against *E.coli* in the continuous-flow disinfection reactor packed with a 1.75cm-layer of the catalyst coated glass beads. Water with 10^6 CFU *E.coli*/mL was pumped at different flow rates into the reactor and flowed through the catalyst coated glass beads. Samples of effluent water were collected and analyzed for *E.coli* concentration. Disinfection activities of the catalysts were tested under UV-C (11W, Phillip).

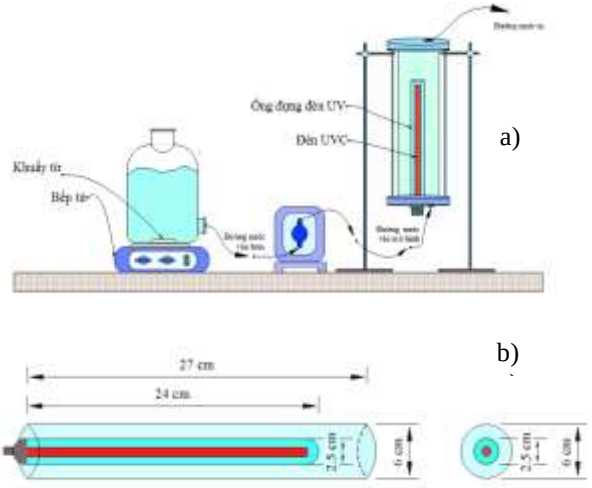


Figure 1: (a) Continuous-flow disinfection reactor used under UVC lamp and (b) UVC reactor parameters

C. Photocatalytic disinfection of E.coli

In order to determine the bactericidal effect of nanoparticles for water purification, water was artificially contaminated by bacterial culture so as to attain final concentration of *E.coli (ATCC 25922)* not less than 10^6CFU/ml. The antibacterial activity of the catalyst against *E.coli* was determined by quantitative evaluation methods. 1 mL of the treated solution was diluted with *sodium chloride* solution (NaCl 0.9%) to suitable bacterial colonies. The diluted solution was then spread on tryptic soy agar (TSA) plates and incubated at 37°C for 24 h before the bacterial colonies were count. Data collected were the average value of three parallel runs or the values showed the best fitting for an exponential reduction.

In the experiment of antibacterial activity of the catalyst in Mekong delta river water, *Coliform* membrane filter method was applied. After filtering by sand filter column, an appropriate volume of (100 ml) river water sample was filtered using a membrane filter (Chromocult® Coliform Agar, Merck, Germany) to ensure that no air is trapped underneath and incubate the inoculated dishes aerobically at 35–37 °C in an inverted position for 24 hours. After incubation, examine the plates for the presence of typical colonies of *E. coli* and other *Coliforms*.

III. Results and discussion

A. Characterization of Ag-TiO_2-SiO_2 nanomaterials coated glass beads

Figure 2 shows the X-ray diffractograms of TiO_2-SiO_2 and 1% Ag-TiO2-SiO2 nanomaterials. Anatase (a-TiO_2) which is the favorable structure with better photo-catalytic functional properties than its rutile phase is identified as the primary crystalline phase in both samples with peaks at 2θ of 25.3°,37.8°,48.2°,55.04°, and 62.74° relating to (101), (004), (200), (105), (211), and (204). In addition, the order of descending crystallinity that is observed from the anatase peaks of two XRD patterns is TiO_2-SiO_2< Ag doped TiO_2-SiO_2.

XRD pattern shows no peak related to crystallization of silver particles in the TiO_2-SiO_2 nanomaterials. On the other hand, XRD pattern of the 1% Ag-TiO_2-SiO_2 nanomaterials clearly exhibits peaks at 2θ= 38,1°, 44,5° and 64,3° relating to (111), (200) and (220) diffraction peaks of the metallic silver. Since these peaks are attributed to face centered cubic (fcc) structure of metallic silver, the Ag(111) peak can be overwhelmed in the stronger TiO_2 (004) peak [21].

The mean crystalline size of the TiO_2-SiO_2 and 1% Ag-TiO_2-SiO_2 nanomaterials were calculated ~10.02 nm and 11.35, respectively, based on the peak broadening analysis described by Scherrer's equation for the (101) peak. The crystallite size of Ag-TiO_2-SiO_2 powder tends to slightly increase as compared to TiO_2-SiO_2. These results indicate that the during drying and calcining process Ag+ ions forming on crystal borders or spreading on the surface anatase grains would gradually be neduced to Ag0 is effect to the crystalline size of anatase phase increase [22, 23]. The mean crystalline size of the Ag nanoparticles, was also calculated about 2 - 5 nm on (200) diffraction peak.

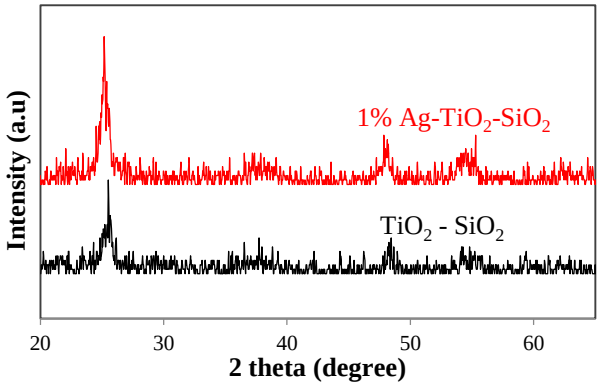
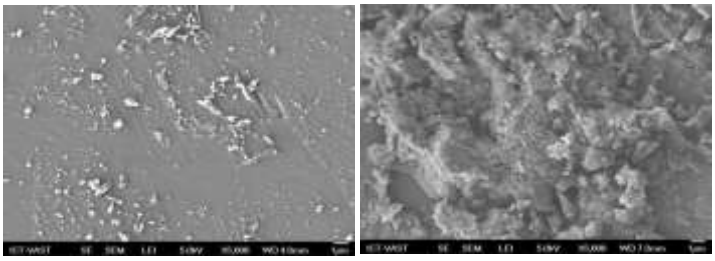


Figure 2: XRD patterns of TiO_2-SiO_2 and 1%Ag-TiO_2-SiO_2 powders prepared by Sol-gel method and calcined at 400°C

The surface morphology of 1%Ag-TiO_2-SiO_2 films prepared by sol-gel method and dipped coating on glass beads (0.4 – 0.9 mm) were examined by SEM (see Figure 4). Fig. 4a and 4b shows morphologies of the bare glass beads and Ag-TiO_2-SiO_2 porous films coated on glass beads. It can be estimated that Ag-TiO_2-SiO_2 films has large specific surface area compared to those of another. The film was directly obtained on surface of glass beads using deposition process by heat treatment at 400°C. The films had light white yellow color and the films thickness was about 0.919 -1.17μm, relating to 1.826– 2.194g Ag-TiO_2-SiO_2/ 1 liter of glass beads.

a) b)

Figure 3.Glass beads before (a) and after (b) dip-coated with Ag-TiO_2-SiO_2



a) b)

Figure 4. SEM images of (a)bare glass beads (GBs), (b) glass beads coated Ag-TiO_2-SiO_2

B. Continuous-flow lab-scale disinfection reactor

In this work, Ag-TiO_2-SiO_2 was immobilized onto glass beads using a dip coating method. The photo-reactor was a custom-built stirred tank reactor which had excellent mixing and good mass transfer properties. The catalyst was irradiated from the inside through the glass to excite the photo-catalyst on the surface.

1) Effect of flow rate on photocatalysis effectiveness

Flow rate experiments were carried out to determine the effect on E.coli suspended in saline solution in reactor configurations under UVC lamp (only UVC, bare glass beads and glass beads coated Ag-TiO_2-SiO_2) (see Figure 5a). For inactivation experiments conducted under UVC lamp, there were no significant decrease in the bacterial activity when the flow rate reduced from 153 mL/min to 25 mL/min. The reduction rate of E.coli colonies by bare glass beads slightly enhanced and less than only UV-C due to the adsorption of bacteria cells onto glass bead surfaces and prevent UVC light transfer through water. In particular, glass beads coated Ag-TiO_2-

SiO_2 exerted stronger bactericidal effects than bare glass beads and only UV-C under UV-C lamp. Further, it is observed that

the photokilling activities of glass beads coated Ag-TiO_2-SiO_2 achieved 6log-reduction unit while 4log-reduction of E.coli population for the reactor exposed to UV-C irradiation with the flow rate at 25 mL/min (hydraulic retention time were 6 minutes and 25.2 minutes, respectively). With the same retention time (6 minutes), the reactor operated without catalyst under UVC exposure got only 5% and glass beads coated Ag-TiO_2-SiO_2 got nearly 100%. These results show that there is a significant difference in the antibacterial activities with and without Ag-TiO_2-SiO_2. Besides, artificial water contaminated by E.coli (ATCC 25922) culture (10^6 CFU/ml) was disinfected at optimum flow rate of 25 mL/min.

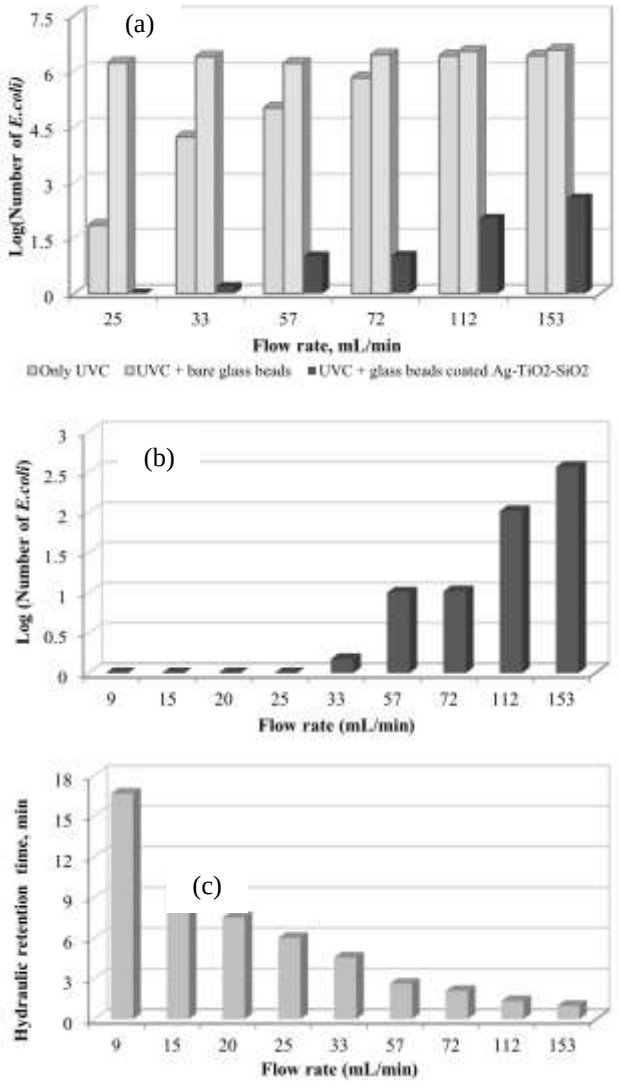


Figure 5: Effect of flow rate on photocatalysis effectiveness: (a) bacterial activities of different materials under UVC lamp; (b) bacterial activities of glass beads coated Ag-TiO2-SiO2; (c) relationship between retention time and flow rate

2) Effect of initial concentration of E.coli on photocatalysis effectiveness

The number of viable bacteria in different water sources may vary. It is thus practicable to investigate the effect of bacterial population on the photodisinfection activities in reactor. Effect of initial bacterial loading on antibacterial activities was concerned in water [24]. At high bacterial loading, the probability of collision between the *E.coli* and catalyst used increase and directly reduces the mass transfer rate at the catalyst surface. At the flow rate of 25 mL/min, 6 log-reduction unit was achieved with glass beads coated Ag-TiO_2-SiO_2 with initial bacterial population 10^6CFU/mL. Therefore, good results in antibacterial activities against *E.coli* were easily achieved with initial bacterial population less than 10^6 CFU/mL.

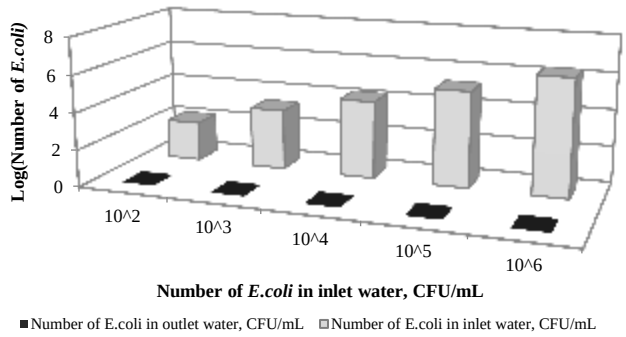


Figure 6: Effects of initial bacterial population on the photocatalytic inactivation of E.coli at 25 mL/min and pH = 6.

3) Effect of pH on photocatalysis effectiveness

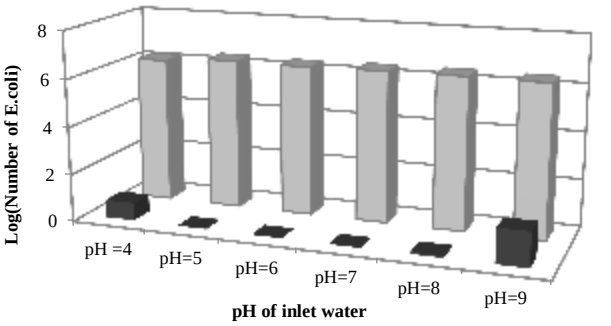


Figure 7: Effects of pH on the photocatalytic inactivation of E.coli at 25 mL/min and initial average bacterial population of 10^6 CFU/mL.

In a semiconductor photocatalytic system, pH take a key role in driving the oxidation reaction and efficiency. The pH can affect the surface charge of the photocatalysts used, and subsequently

the rate of photo-oxidation [25, 26, 27]. To investigate the effects of pH in the photo-disinfection of E.coli by the glass beads coated Ag-TiO_2-SiO_2, a variation of pH 4.0 – 9.0 was simulated in the continuous-flow lab-scale disinfection reactor (see Figure 7). In this instance, the initial pH was adjusted using 0.1M sodium hydroxide and hydrochloric acid, respectively. It is noted that there was a difference in the effectiveness of initial pH in these experiments. The results show that antibacterial activities of glass beads coated Ag-TiO_2-SiO_2 against *E.coli* bacteria were extremely high and independent of initial pH between 5.0 and 7.0. The photo-disinfection activity was significantly reduced at pH 8 and 9, which was very close to PZC (Ag-TiO_2-SiO_2) = pH 7.8. Slowly reduction of *E.coli* was observed at pH 9.0, only 3log-reduction with the flow rate of 25 mL/min. However, it is noted that the Ag-TiO_2-SiO_2 catalyst with high PZC is highly functional under a wide pH range.

C. *Continuous-flow disinfection reactor with Mekong river water*

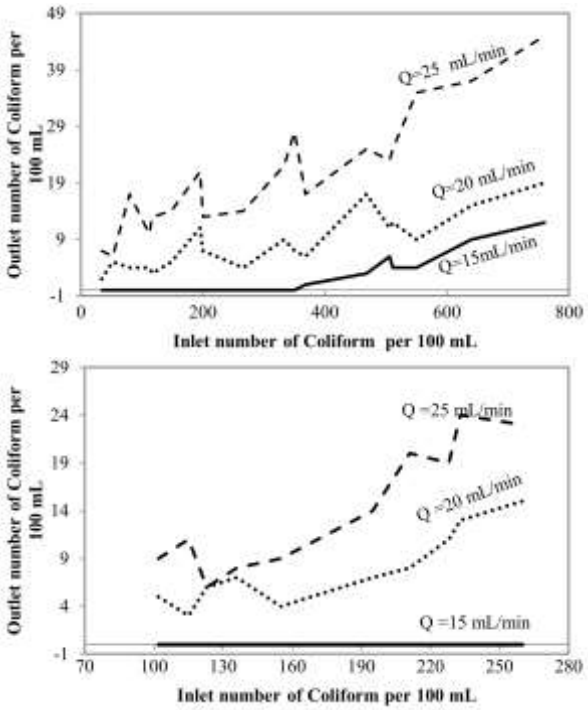


Figure 8:Effects of initial bacterial population on the photocatalytic inactivation of Coliform with Mekong river water: (a) Tien river, (b) Hau river

It is clear from the experiments, that in lab-scale reactor, photocatalyst under UV-C irradiation is more efficient than UV-C alone for the disinfection of artificial water contaminated with *E.coli*. Therefore, it is essential that photocatalytic reactor is tested for disinfection with real water (surface water). The reactor was operated on-site with Mekong river water (includes Tien river and Hau river) which was filtered by a sand filter column. The microbial loading of Tien river (after filtering) was typically 50 – 780 CFU *Coliform*/100mL while that of Hau river (after filtering) was 100 – 260 CFU*Coliform*/100 mL.

With respect to using a photoreactor under UV-C light with Mekong river water, the experiments were carried out with three flow rates (15, 20 and 25 mL/min) according to the optimum flow rate in the lab. In both flow rates (20 and 25 mL/min), the outlet concentration of *Coliform* were not completely inactivated, 2 – 45CFU *Coliforms*/100 mL. The disinfection efficiencies of the flow rate of 25 mL/min achieved approximately 80 – 95% while that of 20 mL/min got 90 – 98%. The optimum flow rate for inactivation with surface water (both Tien and Hau river) was determined to be 15 mL/min, approximately haft that reported for artificial water. However, the outlet concentration of *Coliform* achieved 0 CFU *Coliform*/ 100mL with inlet *Coliform* less than 350 CFU/100 mL (see Figure 8b).

IV. Conclusion

Continuous-flow disinfection reactor under UV-C irradiation is a simple and low cost technique used to disinfect contaminated drinking water. The reactor was filled with glass beads (0.4 -0.9 mm) coated Ag-TiO_2-SiO_2 and has been shown to be effective for inactivation of a wide range of *E.coli* at lab scale. Under the illumination of UV-C light (11W), a 6-log bacterial reduction was achieved in 6 minutes at the optimal flow rate (25 mL/min) of artificial water. In addition, it was found that the Ag-TiO_2-SiO_2 coated glass beads was operable under a wider span of pH conditions, of up to pH 8 when compared to other conventional photocatalyst (pH 6-7).

With the microbial loading of Tien river water of 50 – 780 CFU *Coliform*/100mL and that of Hau river from 100 – 260 CFU *Coliform* /100 mL (after sand filtering), the reactor was able to disinfect the water samples within 9 minutes at the flow rate of 15 mL/min. Following exposure, the water is safe to drink without *Coliform* in case of inlet concentrations of *Coliform* less than 350 CFU/100mL. From this study, it is anticipated that the Ag-TiO_2-SiO_2 system could be applied as an alternative disinfection reactor in rural Vietnam areas.

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