

Effects of Various Metal Oxides and Deposition Methods on the Performance of Bismuth Vanadate (BiVO_4) as a Photocatalyst

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ABSTRACT: The creation of chemical fuels from solar energy requires a photocatalytic material that is efficient and inexpensive. Bismuth vanadate, BiVO_4 , has been established as a photocatalyst capable of splitting water for creating solar chemical fuels. However, the substance is incapable of transferring of electrons, thus requiring additional substances such as metal oxides to establish a successful process of water splitting and to create the desired product: hydrogen gas. Therefore, the bismuth vanadate solution was layered with different metal nitrate solutions such as $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$, $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, and $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ solutions to enhance its ability to conduct higher photocurrents. Bismuth vanadate samples were synthesized by drop casting or spin coating bismuth and vanadate precursors, followed by an annealing step. After the annealing process, the FTO (Fluorine-doped Tin Oxide) plate was tested for the photocurrents by flashing light and measuring photocurrent in the SEAL (Solar Energy Activity Laboratory) kit. Metal nitrate solutions were layered using drop casting or spin coating. Through manipulating various methods to advance its ability of conducting photocurrent, potential of bismuth vanadate as a promising photocatalyst was proven that would be one of the competent photoanodes for the production of hydrogen gas.

1. INTRODUCTION

The development of a process to convert the planet's greatest renewable source of energy into a form for direct human consumption will be crucial for the future.¹ The sun is a promising source of energy to synthesize carbon-neutral chemical fuels through solar water-splitting.¹ However, artificial photosynthesis, using solar energy to produce hydrogen gas, requires photocatalysts that are earth-abundant and easily scalable.

The Solar Energy Activity Laboratory (SEAL) kit assists the researcher with establishing a setting to test water oxidation catalysts. The kit involves LED lighting and current detecting device to measure the activity of metal oxide catalysts. Once the FTO (Fluorine-doped Tin Oxide) plate that contains the solution drop casted on is submerged into the electrolyte solution inside of the crystallization dish, the spots of solution catalyze the water splitting reaction by absorbing the LED light and emitting electrons.² The LED array unit emits light through 64 spots that are evenly spread across on the box and substitute for sunlight. The current integrator unit detects the flowing electrons that are emitted from the reaction. After the detecting of the current, electrons flow into the graphite counter-electrode and complete the reactions.³

Among various photocatalysts such as titanium dioxide (TiO_2) and tungsten trioxide (WO_3), bismuth vanadate (BiVO_4) is one of the photocatalysts capable of solar water splitting while being economical.⁴ In addition, bismuth vanadate is a direct band gap semiconductor that has the narrow band gap of 2.4 eV, which resides in the visible range (2.4eV-2.5eV) of the electromagnetic spectrum.⁵ Although BiVO_4 performs poorly on transporting electrons and exhibits high electron-hole recombination, its inability to effectively transport electrons can be lessened by combining the chemical with different metal oxides such as nickel, cobalt, and copper.^{1,6}

To improve the performance of bismuth vanadate, the focus of the experiment was on combining BiVO_4 with different metal oxides: nickel, cobalt, and copper.⁵ In this work, the focus was to enhance the photocurrent created during the water splitting mechanism; the enhancing pro-

cess can be done through incorporating bismuth vanadate with different layers of metal nitrate solutions.

2. EXPERIMENTAL SECTION

2.1 Production of bismuth vanadate solution: slurry and combination methods. Preparation of bismuth vanadate ($\text{BiVO}_4 \cdot 5\text{H}_2\text{O}$) was performed through the slurry method and the combination method. For slurry method, glycerol and triton X were used as solvents to dissolve $\text{BiVO}_4 \cdot 5\text{H}_2\text{O}$. To obtain the 0.05M bismuth vanadate solution, 0.40g of bismuth vanadate was mixed with 25mL of glycerol in a 25mL volumetric flask. With triton X, 0.40g of BiVO_4 was added to a 25mL volumetric flask. The solution inside the volumetric flask was swirled after five drops of triton X added to the volumetric flask, and the process of adding triton X and swirling the solution was repeated until the solute dissolved in triton X. After solid bismuth nitrate dissolved in triton X, water was used to fill up to the 25mL mark on the volumetric flask. A different approach of producing bismuth vanadate solution was also attempted due to the insolubility of bismuth vanadate solid in water: combination method. For combination method, bismuth nitrate ($\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$) and sodium metavanadate (NaO_3V), a vanadium salt, was combined to create a 0.05M solution and was annealed at 500°C for 5 hours. The spot casted plate was annealed at 500°C for 5 hours in a kiln at atmospheric pressure.

2.2 Mixing and layering methods. To achieve the main purpose of obtaining higher photocurrent of bismuth vanadate as a photocatalyst by combining with other metal nitrate solutions, mixing and layering methods were executed. Creation of metal nitrate solutions required $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ solids. To create 0.1M $\text{Co}(\text{NO}_3)_2$ solution, 0.46g of $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ was added measured out and added to a pre-washed clean 25mL volumetric flask. After the solute was added, 25mL of deionized water was added. The parafilm sealed the opening of the flask and the solution was swirled until completely mixed. 0.456g of $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ was consumed for 25mL of 0.1M nickel (II) nitrate solution. After 0.456g of nickel (II) nitrate hexahydrate was measured and transferred into the 25mL volumetric flask. 25mL of water was added. The solution was

thoroughly mixed by putting on the parafilm and swirling. 0.456g of $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ was added for the 0.1M copper (II) nitrate solution. After measuring out 0.456g of copper (II) nitrate solid, the solid was transferred from the weighing paper to a clean 25mL volumetric flask. Then, 25mL of deionized water was added to the 25mL volumetric flask and the solution was mixed thoroughly after sealing the opening of the volumetric flask with the parafilm. Metal nitrate solutions were diluted from 0.1M to 0.05M to be mixed with 0.05M BiVO_4 solution for mixing method. Diluted 0.05M metal nitrates were contained in three separate beakers, and bismuth vanadate solution was added to each metal nitrate solution to create the mixed solution of bismuth nitrate and metal nitrate. A 3 in. by 3 in. FTO plate was divided into three sections (vertical orientation) and a solution of bismuth vanadate combined with each metal nitrate was spotted on each section of the FTO plate. The alignment of the spots was the checkerboard pattern, which displayed gap of one spot between each spot (Fig. 1).

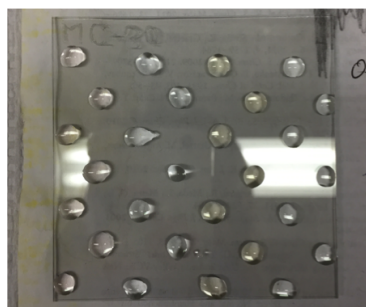


Figure 1. Example of the checkerboard pattern

Layering the metal nitrate solutions onto the bismuth vanadate solution required two annealing processes. After spotting bismuth vanadate solution on the entire plate, following the checkerboard pattern, the plate was annealed at 500°C for 5 hours. After one cycle of annealing, the plate was vertically divided into three sections, equivalent to the division of plate during the mixing method, and each metal nitrate solution was spotted in checkerboard pattern on top of the bismuth vanadate droplets. For both mixing and layering method, the concentrations of solutions tested were brought to 0.05M and 0.025M of bismuth vanadate and metal nitrate solutions by diluting the solutions.

2.3 Production of Bismuth Vanadate Solution:

Usage of Ammonium Metavanadate. Mixing bismuth nitrate and a different vanadium salt, ammonium metavanadate (NH_4VO_3), other than sodium metavanadate also produced bismuth vanadate solution. 0.24g of bismuth nitrate was mixed in 25mL of deionized water in 25mL volumetric flask to create 0.05M bismuth nitrate solution. 0.06g of ammonium metavanadate was combined in 25mL of water in a 25mL volumetric flask to create 0.05M-ammonium metavanadate solution. 0.05M bismuth nitrate solution was combined with ammonium metavanadate solution in a 50mL beaker. Incorporating the checkerboard pattern, the combined solution was spotted on the entire plate; the plate was annealed for 5 hours at 500°C.

2.4 Incorporating Different Solvents. Using bismuth nitrate combined with ammonium metavanadate,

different solvents were tested for the ability to dissolve the compound with efficiency. Therefore, 2M nitric acid, acetone, and 10% acetic acid were used to dissolve the bismuth vanadate compound. 0.24g of bismuth nitrate (0.05M) and 0.06g of ammonium metavanadate (0.05M) were added in one 10mL volumetric flask to obtain 0.05M solution. 10.0mL of 2M nitric acid was added into the volumetric flask. The solution was swirled until partially dissolved. The same concentrations of bismuth nitrate and of ammonium metavanadate were added in a 10mL volumetric flask for producing the solution using 5mL of acetone combined with 5mL water as solvents. 10% acetic acid was used as the third solvent. Acetic acid and water were added into the volumetric flask that contained 0.24g of bismuth vanadate and 0.06g of ammonium metavanadate to produce 0.05M bismuth vanadate solution.

2.5 Spin Coating. Solutions created using 2M nitric acid, acetone, and 10% acetic acid were casted on the plates using the spin coating method. Preparation for spin coating was processed through cutting 3in. by 3in. FTO plate into two smaller plates. Scotch tape covered the top 1.8cm of each small plate. Each plate was placed on the spin coater and performed two cycles. The first cycle was run for 6 seconds at 1000rpm. After the first cycle, the plates were annealed on the hot plate for 10 minutes at 200°C. Plates were taken off from the hot plate after 10 minutes and went through the second cycle for 16 seconds at 1000rpm.

3. RESULTS

3.1 The Slurry Method. The 0.05M solution consisting of BiVO_4 solid with glycerol (plate labeled MC-04) produced the photocurrent of 0.5-0.6 μA (Fig.2). The other plate for slurry method using triton X, MC-06, also produced the photocurrent of 0.5-0.6 μA (Fig.2).

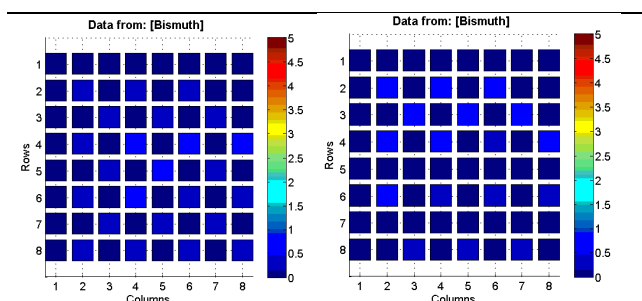


Figure 2. MC-04: adaptation of slurry method for the 0.05M BiVO_4 and glycerol solution (left) / MC-06: adaptation of slurry method for the 0.05M BiVO_4 and triton X solution (right)

3.2 The Combination Method. For the slurry method, BiVO_4 solid was used for the solution. However, bismuth nitrate ($\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$) solid and vanadium salt (In this experiment, sodium metavanadate (NaO_3V) was used) were mixed for the mixing and layering methods. The different molarities of the solution, 0.05M and 0.025M, for combination method (combination of bismuth nitrate and sodium metavanadate) generated the result of 0.7-1.2 μA and 0.5-0.6 μA , respectively, on the same plate (MC-15) (Fig.3). For plate MC-18 (Fig.3), the plate was drop casted using the same method of mixing bismuth nitrate

($\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$) and sodium metavanadate (NaO_3V) but switching the sides of the molarities. Left side with molarity of 0.05M carried the photocurrent of 0.5-0.7 μA while the right side with 0.025M carried the photocurrent of 0-0.5 μA . Plate MC-16 (Fig.4) presented the mixing method of 0.05M bismuth vanadate (bismuth nitrate and sodium metavanadate) solution with 0.05M metal nitrate solutions by mixing the bismuth nitrate solution with each metal nitrate solution. MC-16 had cobalt II nitrate solution, copper II nitrate solution, and nickel II nitrate solution on the plate (divided into three sections). MC-21C was created to retest the result of MC-16 and adapted the corresponding method as MC-16 with a different vanadium salt: ammonium metavanadate (NH_4VO_3). MC-16 produced the photocurrent of 0.04-0.05 μA ; MC-21C conducted the photocurrent around 0.05 μA (Fig.5). Additionally, the colors of MC-16 and MC-21C transformed from gray and light black from the colors of metal nitrate solutions after 5 hours of annealing process. Mixing method that consisted of bismuth nitrate and ammonium metavanadate as the vanadium salt conducted the average photocurrent of 4.4 μA from MC-31B (Fig.6). Due to the unexpectedly high photocurrent, MC-31C was created to test the exact same solution (Fig.7).

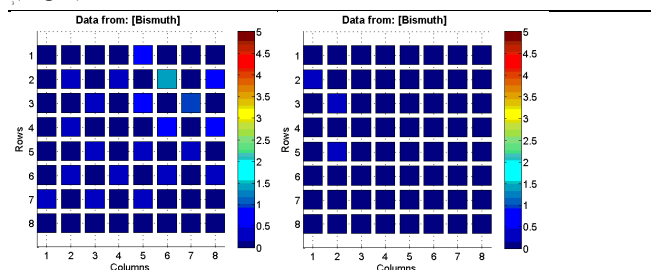


Figure 3. MC-15: combination of $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$ and NaO_3V (left half: 0.025M/ right half: 0.05M) (left) / MC-18: combination of $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$ and NaO_3V (left half: 0.05M/ right half: 0.025M) (right)

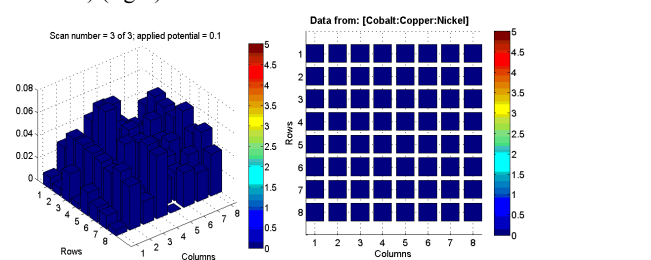


Figure 4. MC-16: 0.05M bismuth vanadate solution (bismuth nitrate and sodium metavanadate) mixed with different 0.05M metal nitrate solutions (Co,Cu, and Ni) (3D and 2D results)

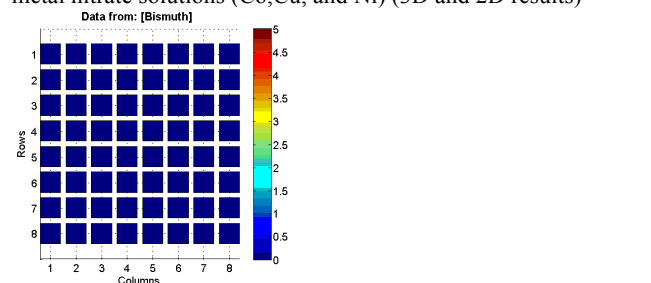


Figure 5. MC-21C: 0.05M bismuth vanadate (bismuth nitrate and ammonium vanadate) solution mixed with various 0.05M metal nitrate solutions (Co, Cu, and Ni)

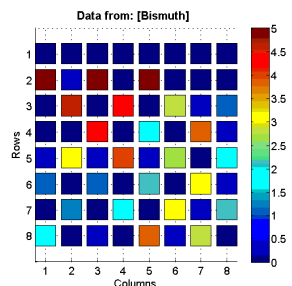


Figure 6. MC-31B: Mixing method using bismuth vanadate (bismuth nitrate and ammonium metavanadate) and metal nitrate solutions (Co, Cu, Fe, Ni)

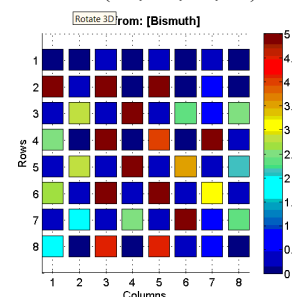


Figure 7. MC-31C: Duplication of plate MC-31B for accuracy of the result due to unexpectedly high photocurrent

3.3 The Layering Method. The second method discussed, the layering method, was performed with plates MC-21B and MC-25B. MC-21B was conducted with bismuth nitrate and sodium metavanadate; MC-25B was conducted with bismuth nitrate and ammonium metavanadate. After one round of annealing with drop-casted bismuth nitrate combined with sodium metavanadate (for MC-21B) or ammonium metavanadate (for MC-25B), metal nitrate solutions were drop casted on top of the existing spots. After the second annealing process, spots of MC-21B turned gray and black. Spots of MC-25B maintained the colors of respective metal nitrate solutions (Fig.8). MC-21B carried the average photocurrent of 0.05 μA (Fig.9) while MC-25B conducted wide range of photocurrents of 0.5-3.0 μA (Fig. 10). Bismuth vanadate solution was acquired through dissolving the combined solutes of bismuth nitrate and ammonium metavanadate. For the solvents, 2M nitric acid, acetone with water, 10% acetic acid, and acetylacetone (2,4-pentanedione). The 0.05M bismuth nitrate solution in 2M nitric acid produced a bright yellow color after annealing (MC-21A). However, the spots turned into light neon green after the testing cycles were performed. The conductivity of the plate was average photocurrent of 2.5-3.0 μA (Fig.11). Solution dissolved in acetone with water created yellow color. Acetone solution was tested in two different methods: drop casting and spin coating. Drop casted plate (right side of MC-25C) had an average photocurrent of 0.5 μA and while the spin coated plate (MC-SCB) produced an average photocurrent of 0.05 μA (Fig.12). Drops on the plate of bismuth vanadate using acetic acid (left side of MC-25C) were orange before annealed and after annealed (Fig.13). The average photocurrent of the plate was 0.5 μA (Fig.14) while the spin-coated plate (MC-SCC) of the same solution produced the average conductivity of 0.05 μA (Fig.15). The bismuth vanadate solution using acetyl-

acetone established green and yellow layers after combining sodium metavanadate with acetylacetone and NaOH (Fig.16). However, the color changed again to dark red and black when bismuth nitrate was added onto the solution, producing odor.

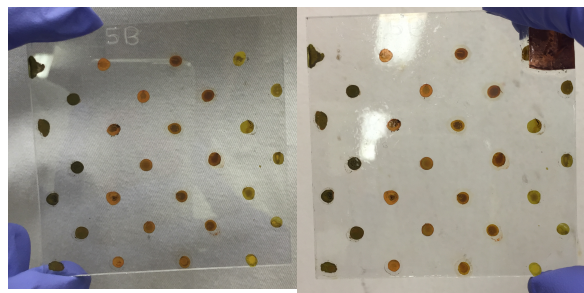


Figure 8. MC-25B: color of the plate did not change after contacting the NaOH solution (left: before testing/ right: after testing (submerged in electrolyte solution))

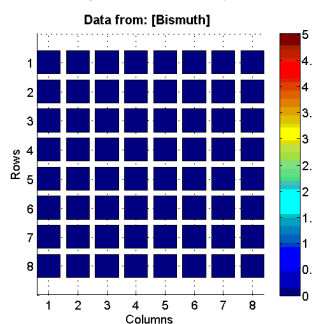


Figure 9. MC-21B: layering method of metal nitrate solutions with BiVO_4 using sodium metavanadate as the vanadium salt

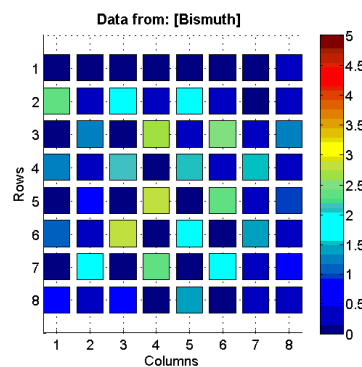


Figure 10. MC-25B: incorporation of layering method using ammonium metavanadate as the vanadium salt for BiVO_4 and metal nitrate solutions layered on top of the bismuth vanadate spot.

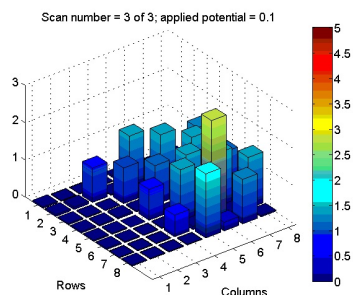


Figure 11. MC-21A: result of bismuth vanadate activity with 2M nitric acid as the solvent.

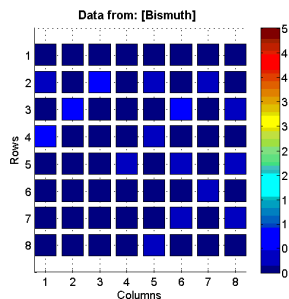


Figure 12. MC-SCB: spin coated plate of bismuth vanadate dissolved in acetone

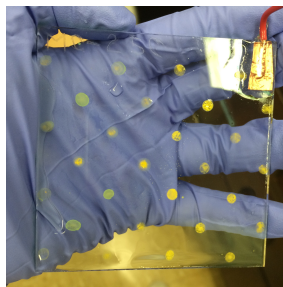


Figure 13. MC-25C: plate after annealed (left half: 10% acetic acid/ right half: acetone)

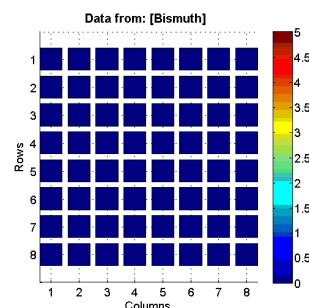


Figure 14. MC-25C: two different solutions of BiVO_4 using 10% acetic acid and acetone as solvents (left half: 10% acetic acid/ right half: acetone)

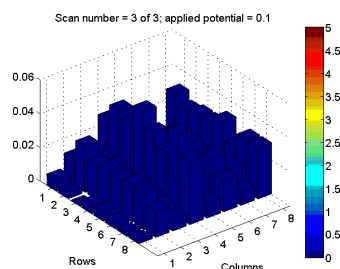


Figure 15. MC-SCC: Spin-coated plate of BiVO_4 using 10% acetic acid as the solvent

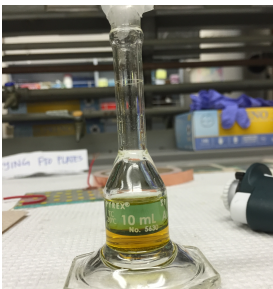


Figure 16. Mixture of sodium metavanadate, acetylacetone, and 0.1M NaOH (green and yellow layers)

Chart 1. Significant plates and their properties

	Solution	Method	Average Photocurrent (μA)	Color	Figure #
MC-04	BiVO_4 +glycerol	Slurry (5 hours)	0.5	Light yellow	2
MC-06	BiVO_4 +triton X	Slurry (5 hours)	0.5	Light yellow	2
MC-15	$\text{Bi}(\text{NO}_3)_3$ + NaO_3V	Combination (5 hours)	0.9(0.05M) 0.6(0.025M)	Light yellow to fade white	3
MC-16	$\text{Bi}(\text{NO}_3)_3$ + NaO_3V +metal nitrate solutions	Mixing (5 hours)	0.04-0.05	Dark gray and black	4
MC-21A	$\text{Bi}(\text{NO}_3)_3$ + NH_4VO_3 +2M nitric acid	Combination (2 hours)	2.5-3.0	Bright yellow-turned into neon green after contact with the electrolyte solution	11
MC-21B	$\text{Bi}(\text{NO}_3)_3$ + NaO_3V +metal nitrate solutions	Layering (5 hours)	0.05	Gray and black	9
MC-21C	$\text{Bi}(\text{NO}_3)_3$ + NH_4VO_3 +metal nitrate solutions	Mixing (5 hours)	0.05	Gray and light black	5
MC-25B	$\text{Bi}(\text{NO}_3)_3$ + NH_4VO_3 +2M nitric acid+ metal nitrate solutions	Layering (2 hours)	2.1	colors of individual metal nitrate solution	10
MC-25C	$\text{Bi}(\text{NO}_3)_3$ + NH_4VO_3 + 10% acetic acid/ $\text{Bi}(\text{NO}_3)_3$ + NH_4VO_3 + acetone	Mixing different solvents (2 hours)	0.5	Fade yellow	14
MC-SCB	$\text{Bi}(\text{NO}_3)_3$ + NH_4VO_3 +acetone	Spin coating (2 hours)	0.05	Bright yellow	12
MC-SCC	$\text{Bi}(\text{NO}_3)_3$ + NH_4VO_3 +10% acetic acid	Spin coating (2 hours)	0.05	Uneven spread of yellow	15
MC-31B	$\text{Bi}(\text{NO}_3)_3$ + NH_4VO_3 +2M nitric acid+ metal nitrate solutions	Mixing (2 hours)	4.4	Colors of individual metal nitrate solution	6

4. DISCUSSION

4.1 The Slurry Method. The slurry method for producing 0.05M bismuth vanadate solution was not an appropriate method to create testable solution since glycerol's high viscosity made drop casting ineffective. Triton X was also not the proper solvent for dissolving bismuth vanadate due to its low surface tension and tendency to widely spread on the FTO plate. Solutions using glycerol and triton X both produced relatively low photocurrent of around 0.05-0.06 μA when drop casted and tested on the SEAL kit. Comparing the slurry method and combination method, combination method created plates that conducted better photoactivity.

4.2 The Combination Method and Vanadium Salts. MC-15, a combination method of bismuth nitrate and sodium metavanadate produced an average photocurrent of 0.7-1.2 μA , while the average current for the plates that were made using the slurry method did not exceed the average photocurrent of 1.0 μA . However, bismuth vanadate's performance improved when ammonium metavanadate was added to the solution instead of sodium metavanadate. This result is possible since sodium metavanadate could residual sodium ions even after the plates were annealed. However, ammonium metavanadate does not have this tendency. Bismuth vanadate solution also produced even higher activity when the bismuth nitrate combined with ammonium metavanadate was dissolved in 2M nitric acid. The resulting photocurrent was 2.5-3.0 μA , higher compared to the solution with sodium metavanadate as the vanadium salt (Fig.11). The combination of bismuth nitrate and ammonium metavanadate dissolved in 2M nitric acid properly produced bismuth vanadate according to the SEM

(Scanning Electron Microscopy) data using FEI Nova NanoSEM 450 as the device. From the SEM data, it was discovered that the atomic percentage for vanadium in the solution was 3.08%. The atomic percentage for bismuth was 2.60%. Therefore, the solution consisted of both bismuth and vanadium (Fig.17).

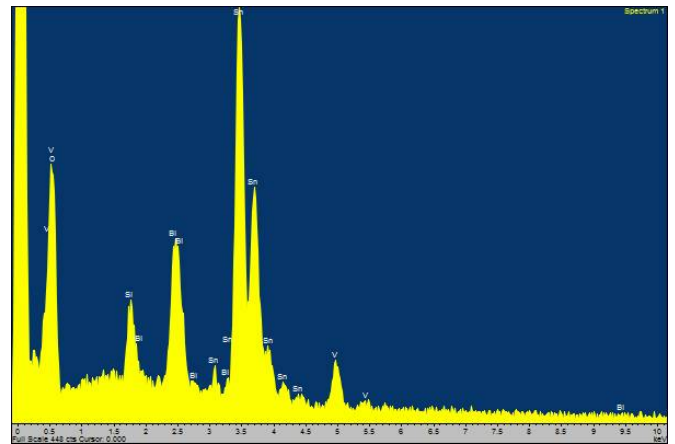


Figure 17. Scanning Electron Microscopy image of MC-21A sample

4.3 Spin Coating. One method attempted to deposit the metal nitrate solutions was spin coating. However, spin coating did not work properly for the SEAL kit since the box of the kit produced 64 spots of LED light to imitate the sunlight while spin coating created a thin layer of bismuth vanadate that was troublesome for detecting activity of the compound.

4.4 The Mixing and Layering Methods. Comparing the mixing and layering method, MC-15 and MC-18 performed low photocurrent of average 0.5 μA while layer-

ing method created average photocurrent of 3.0 μA . One potential reason of the mixing method's low catalytic properities is due to the separate reaction between bismuth nitrate, vanadium salt, and the metal nitrate solutions when the plates were annealed at 500°C. Because the bismuth nitrate and vanadium salt reacted separately with the metal nitrate solutions, bismuth vanadate could not be produced during the annealing process and the spots turned into gray and black colors. Another result of mixing method's poor performance is due to the vanadium salts. The results of the mixing method using ammonium metavanadate were higher average photocurrents than that of the layering method with ammonium metavanadate. Therefore, with ammonium metavanadate as the vanadium salt, it is seen that mixing method obtained better result than that of layering method. Although MC-21B, layering plate with sodium metavanadate, did not produce high photocurrent, the layering method using ammonium metavanadate resulted in a high average photocurrent of 3.0 μA . Experimenting with different solvents to improve activity of drop-cast bismuth vanadate, 2M nitric acid was established to be the most adequate solvent for dissolving bismuth vanadate. The activity of bismuth vanadate could be improved by adding layers of metal oxide since it the process of adding metal oxide layers improves oxygen revolution.

4.5 Time Factors. One of the variables during the experiment was length of annealing process. Although the plates were originally placed in the kiln for 5 hours, the time of annealing changed to 2 hours with the production of bismuth nitrate combined with ammonium metavanadate and 2M nitric acid since it was discussed that excessive annealing time would not assist on producing higher photocurrents during the testing.

5. CONCLUSION

BiVO_4 was tested with different methods and variables throughout the experiment. Although bismuth vanadate was insoluble in water, a different method of combining bismuth nitrate and vanadium salt established more efficient way of producing the solution. Different variables such as different concentrations were measured and tested with various trials. Between 0.05M and 0.025M bismuth vanadate solutions, 0.05M solution performed better activity. Comparison between the mixing method and the layering method projected the result of potential of the mixing method to be more effective than the layering method with

the control of the vanadium salt as ammonium metavanadate. Although mixing method conducted higher photocurrent than other methods during the experiment, the result would be more promising if multiple trials are performed on mixing the metal nitrate solutions onto the bismuth vanadate (bismuth nitrate and ammonium metavanadate) solution dissolved in 2M nitric acid. Metal nitrate solutions have potential to improve the activity of drop-cast bismuth nitrate as an effective photocatalyst. It is also possible to mainly focus on copper and iron as the metal nitrate solutions since they were the metal oxides that enhanced the activity of bismuth vanadate by the most difference.

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7. ACKNOWLEDGMENTS

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