Troubleshooting The Solar Hydrogen Activities research Kit

Asia Leong*, Melody Wu, Hannah Park, Vera Qu

ABSTRACT: The SHArK kit is one example of a hands-on research experiment that is used in an outreach program facilitated by the California Institute of Technology. The purpose of the Solar Hydrogen Activities research Kit is to test different combinations of metal oxide solutions for photoactivity using electrolysis. There have been several versions of this kit that have been created and used over time. The current version being SHArK 3.0. Using a green laser and a structure entirely composed of LEGO bricks, this kit is meant to provide an easy way to test metal oxides in search of photoactivity. As it is in its earliest stages, SHArK 3.0 is in the process of troubleshooting. There are a plethora of variables to be tested before the kit is able to be released to the public.

1.INTRODUCTION

Fossil fuels are a very limited resource on our planet today. [2] The Earth is in desperate need of materials that are cheap, in abundance, and sustainable. As fossil fuels are used, damage is done to the environment and the supply of this energy source decreases. [2] A metal oxide such as iron(III) nitrate is a very good example of a photocatalyst that is earth abundant and shows signs of photoactivity. By using tools such as the SHArK kit, people are able to specifically test these metal oxides and their ability to produce photocurrent.

The Solar Hydrogen Activities research Kit was meant to be used as a simple and cost effective way for students to become engaged in the exploration of metal oxides that have the ability to exhibit photoactivity in aqueous solution. [5] The first two versions of SHArK, were efficient but rather complicated to use as the first version utilized a system of complex gearing. [1]The second version of SHArK was a closer design to the SHArK 3.0, but the biggest differences lie in the updates in software. SHArK 3.0 is a much more compact and straightforward kit and, in theory, is much easier to use. Linear actuators, attached to the laser, are held up by a LEGO structure and controlled by the National Instruments MyRIO electronics box. The sample plate, containing thin films of metal oxides, is held in a sample well that places the solutions directly in front of the laser. The laser then proceeds to move in concentric circles allowing the laser to search the entire plate for photoactivity.

In February of 2016, SHArK 3.0[1] was revealed. This new, compact version of the kit has yet to be released to the public as it is currently in beta-testing. As of right now SHArK still has several problems and variables that need to be tested and narrowed down to sufficiently fix the kit and release it to the public. For example, the kit is hyper-sensitive to sodium hydroxide contamination and the slightest drop on either the alligator clips or the copper tape on the sample plate, can lead to electronic interference with the kit. This phase of troubleshooting the kit is essential in order to both improve the kit and lessen the problems that could occur in the future.

2. METHODS

2.1 Setting Up The Kit

The SHArK Kit [3] (Figure 1**)** supplied the user with a usb containing a manual with detailed instructions on how to build the kit and how to use it. The instructions began with the construction of the support legs of the structure. Then the instructions proceeded to detail how to assemble the moveable piece at the top of the structure that held the laser.The X and Y actuators were attached to the kit following the manual's instructions. The laser was placed in the ring attached to the Y actuator.

Once the structure was assembled, the USB containing SHArK 3.0's software was plugged into a Windows laptop. This provided all the database information as well as the software that actually controlled the laser's movements.

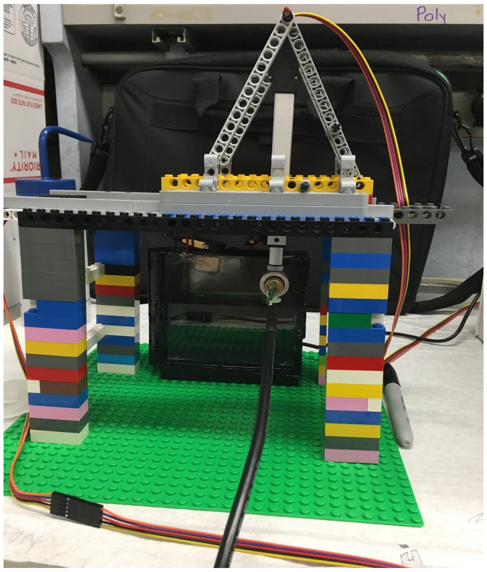

Figure 1: The SHArK Kit.

Figure 2: An Iron(III) Nitrate and Copper(II) Nitrate plate.

2.2 Making The Plates

Each plate (Figure 2) was created using drop pipetting. Varying volumes of metal oxide solutions were pipetted onto an FTO plate. The plates were then placed onto a hot plate at 65 degrees Celsius in order for the solutions to dry. Initially all of the metal nitrate solutions (iron(III), zinc(II), nickel(III), and copper(II) nitrate) were made at the concentration of 0.05 M . However after observing that some of the spots were "flaking off", after being kilned, the concentration of the iron(III) nitrate and copper(II) nitrate were diluted to 0.04 M.

2.3 Scanning The Plates

To perform a scan with SHArK 3.0, a 3-D printed sample well is used to hold the sample plate and electrolyte solution (sodium hydroxide). As a part of the troubleshooting process, different variables in the scans were tested. Before starting a scan, the FTO plate on the inside of the sample well was cleaned with isopropyl alcohol and left to dry. Once it was completely dry the sodium hydroxide was poured to fill half of the sample well. Before placing the plate into the solution, copper tape was attached to the top of the FTO side of the sample plate. This variable was tested throughout the experiment. Smaller pieces of copper tape were used to ensure that sodium hydroxide did not touch the copper tape which could lead to "noisy" scans. After attaching the copper tape, the sample was placed into the well and sodium hydroxide was added until it was just below the copper tape. Once this was completed, the leads were attached to both the sample well and sample plate. The black lead was attached to the sample and the red lead (alligator clip) was attached to the FTO side of the sample well plate.

The sample well was then placed in front of the laser, underneath the LEGO structure. The raster was started and the scan began. [3]A basic scan lasted approximately 55 minutes and applied no voltage. An advanced scan took approximately two hours and voltage was able to be applied.

3.Results

Through the entirety of troubleshooting the SHArK Kit, a plethora of variables were tested to figure out what exactly worked best.

FTO plate was cleaned with isopropyl alcohol on both the FTO side of the plate and the front. This was seen to make minor improvements on the quality of the scans.

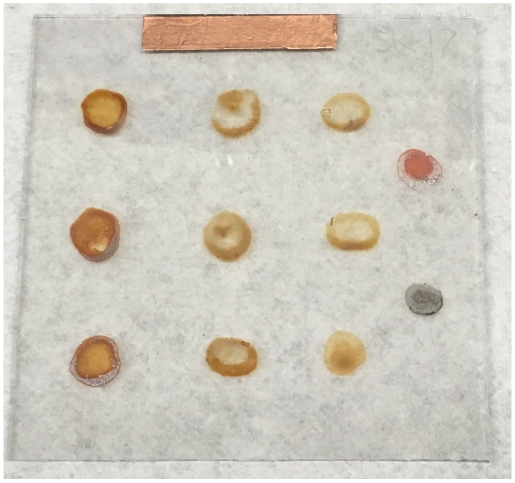

Figure 3: The smaller piece of copper tape is shown at the top of the sample plate.

3.1 Copper Tape

Initially the plates were scanned using pieces of copper tape that covered the entirety of the top of the plate. After experiencing a few of our noisy scans, sodium hydroxide interference was considered as a possible source of the error. Plates were tested using smaller pieces of copper tape, no more than 1 inch, to minimize the amount of sodium hydroxide that could contaminate the tape.(Figure 3) It was found that this method had little effect on the results of our scans.

3.2 Solutions

The concentrations of the solutions began at 0.05 M and were observed to have issues on the hot plate and in the kiln. The spots came out with small bits that appeared to come off when placed into the sodium hydroxide solution. The solutions were diluted to 0.04 M which seemed to improve the quality of the spots.

3.3 Cleaning the Sample Well

One variable explored was the cleaning of the holder's FTO plate (Figure 4) before beginning the scans of the day. Some black residue was noticed on the top edge of the plate where the copper tape goes. Prior to any scanning at the beginning of a day, the FTO plate was cleaned with isopropyl alcohol on both the FTO side of the plate and the front. This was seen to make minor improvements on the quality of the scans.

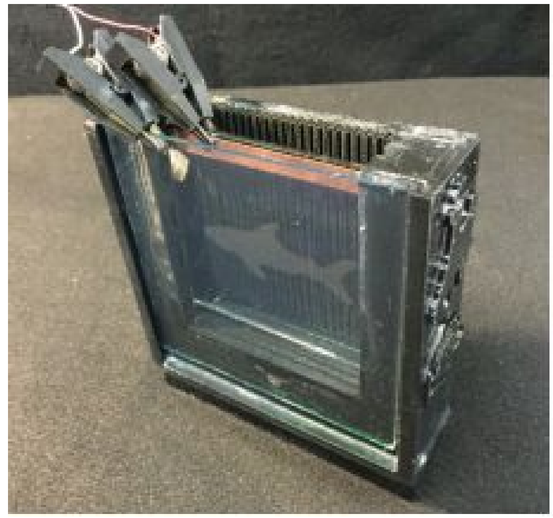

Figure 4: The 3-D printed sample well.

3.4 The Electronics of The Kit

When taking the previously mentioned precautions showed no change in scan noisiness, the electronics of the kit were questioned. The laser began to experience some issues, such as stopping in the middle of scans and vibrating when stopping. Then, a new electronics box (Figure 5**)** was obtained. It turned out significantly greater results than before. There was much less noise and more visible spots.

Figure 5: The new electronics box after being attached to MyRIO

4. Discussion

Most of the improvements (Figure 6**)**that were made came at the end of the research. The dilution of the solutions and the changes in the size of copper tape did not make as much of an improvement in the scans as did the new electronics box or the isopropyl alcohol. In looking closer at the metal nitrate solutions, there was small residue that was noticed floating inside the vials which may have contributed to the lack of results(Figure 7**)**. The copper tape may not have made as much of an improvement because there were other ways sodium hydroxide could have interfered with the scans, such as through the leads. The data collected proved that using the isopropyl alcohol had made some improvement in the scans. Use of the new electronics box made huge improvements in the quality of the scans .

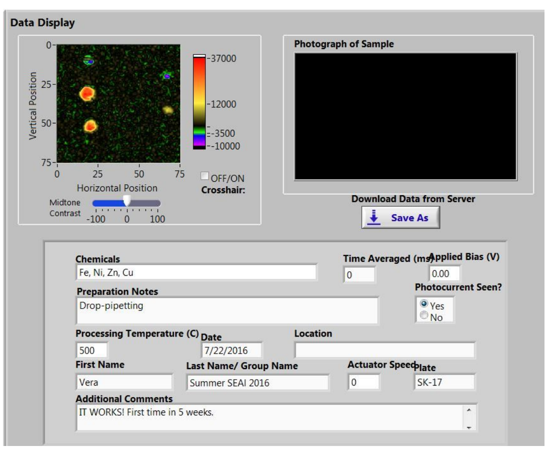

Figure 6: The first scan that worked for us in the SHArK Kit.

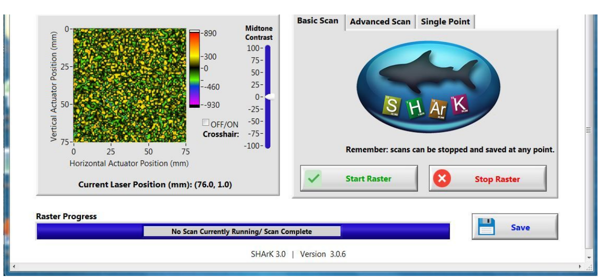

Figure 7: An example of a noisy scan.

Corresponding Author

*asia.leong36@gmail.com

5. References

(1) Background http://thesolararmy.org/shark/background/ (accessed Jul 28, 2016).

(2) Gray, H. B. Nature Chemistry Nature Chem 2009, 1 (2), 112.

(3) The Kit http://thesolararmy.org/shark/the-kit/ (accessed Jul 28, 2016).

(4) Mckone, J. R.; Lewis, N. S.; Gray, H. B. ChemInform 2014, 45 (10).

(5) Rowley, J. G.; Do, T. D.; Cleary, D. A.; Parkinson, B. A. ACS Appl. Mater. Interfaces ACS Applied Materials & Interfaces 2014, 6 (12), 9046.