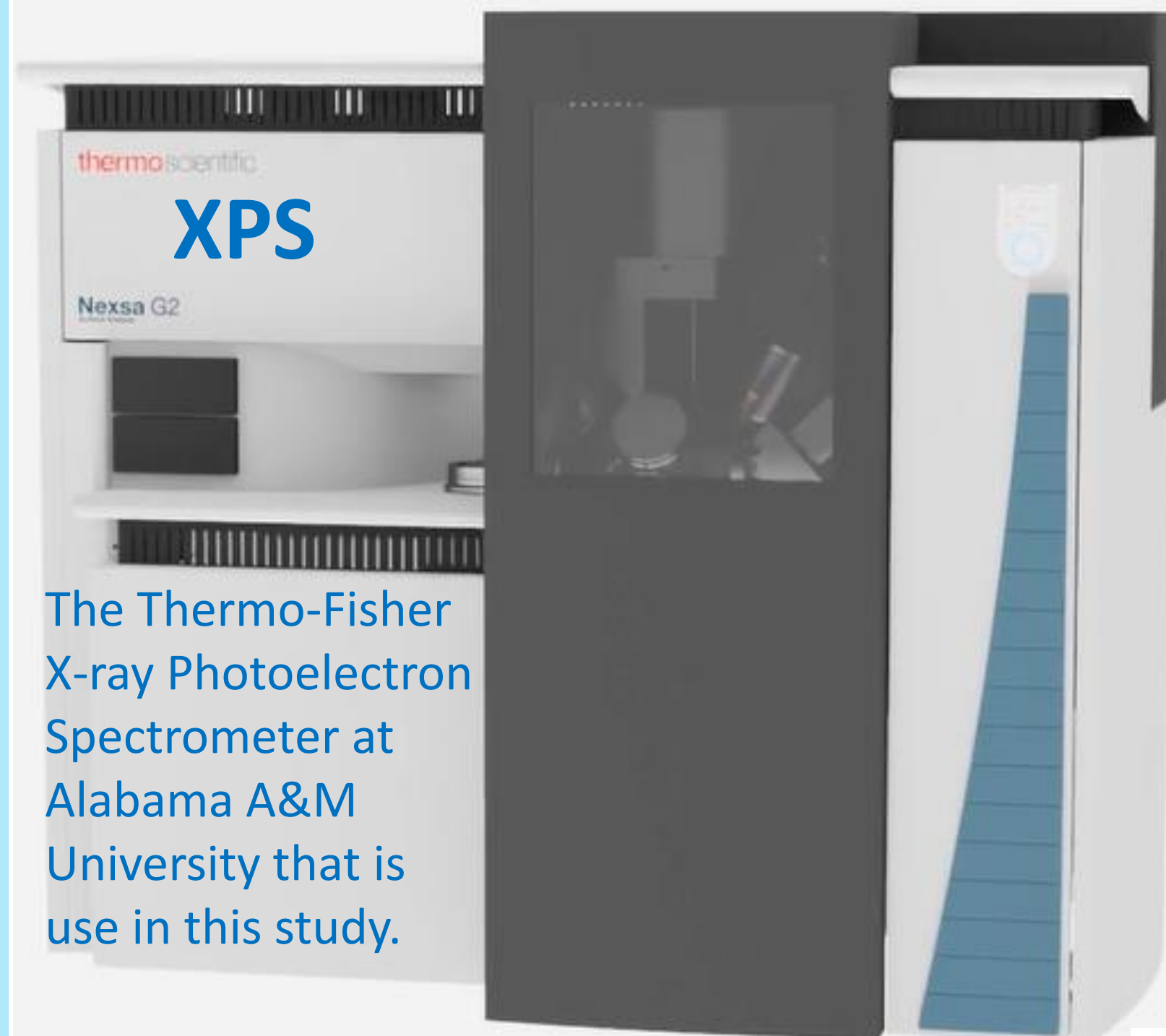


Dehydration of Calcium Sulphate Dihydrate using X-Ray Photoelectron Spectroscopy(XPS) and Raman Characterization

Alexander Egariyewe, M.Drabo,, A. Kassu, Matthew Edwards S. Egariyewe, STEM DAY Spring 2022, Department of Physics, Mathematics & Chemistry, Alabama A&M University

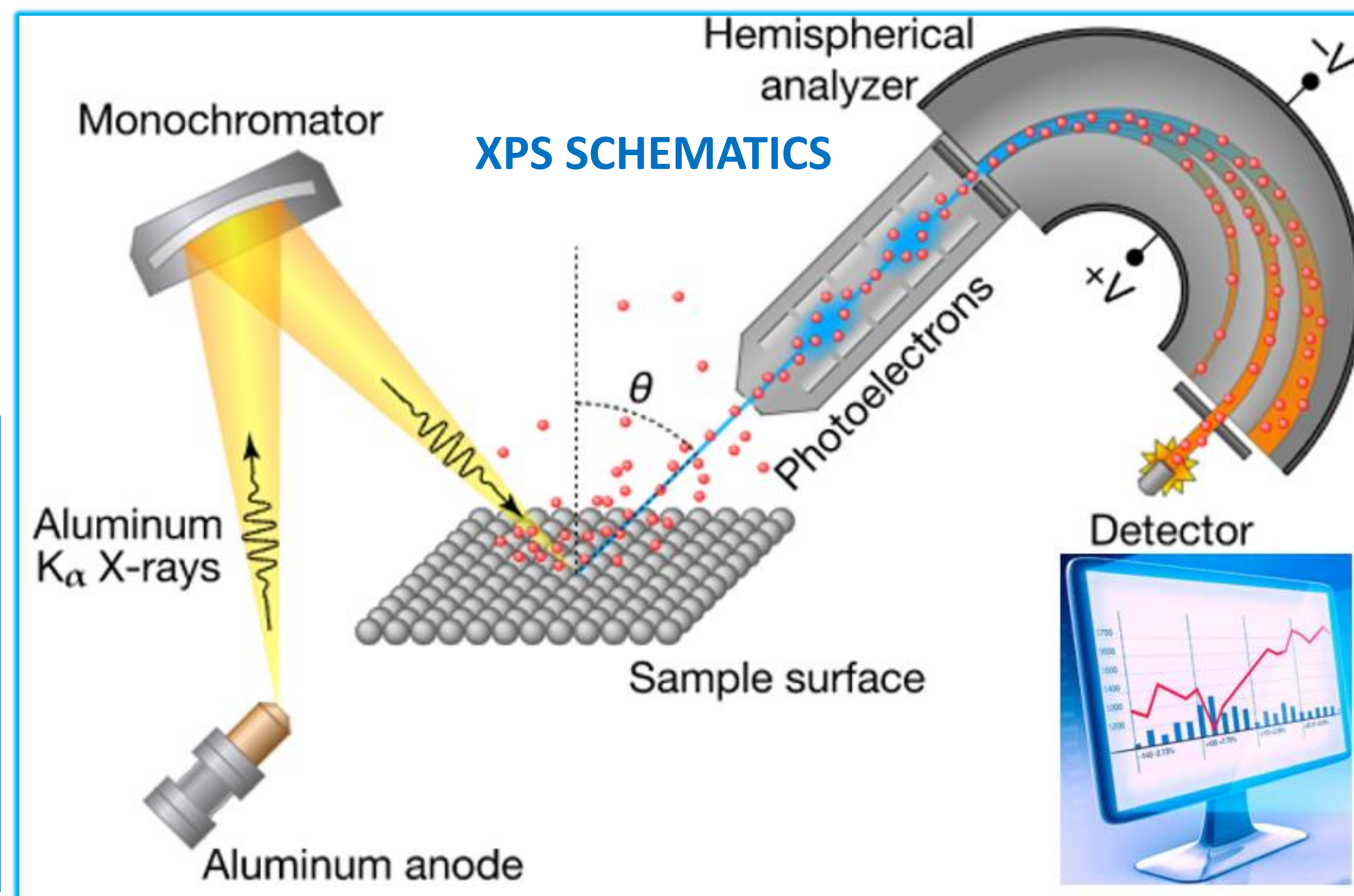
INTRODUCTION

Calcium sulphate dihydrate $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$, known as gypsum, can change to calcium sulphate hemihydrate $\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$, also known as bassanite, when it is put into the furnace over a period. The temperature used was at a constants 140°C for over two hours. The transformation of the gypsum to bassanite is mainly due to the time the raw material $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ undergoes the dehydration process. Staying longer in the furnace could lead to anhydrate of CaSO_4 . When higher temperatures were applied, it also took close to about the same amount of time for transformation to occur. Thus, temperatures higher than 140°C will not have much effect in the process.



ABSTRACT

Accomplishing hemihydrate and possibly anhydrate from dihydrate is a process that requires both time and temperature. Raman Spectroscopy shows how the dehydration diffusion process affects the compound, while the X-Ray Photoelectron Spectroscopy(XPS) shows how the surface composition of the compound's dehydration process, affect each element.

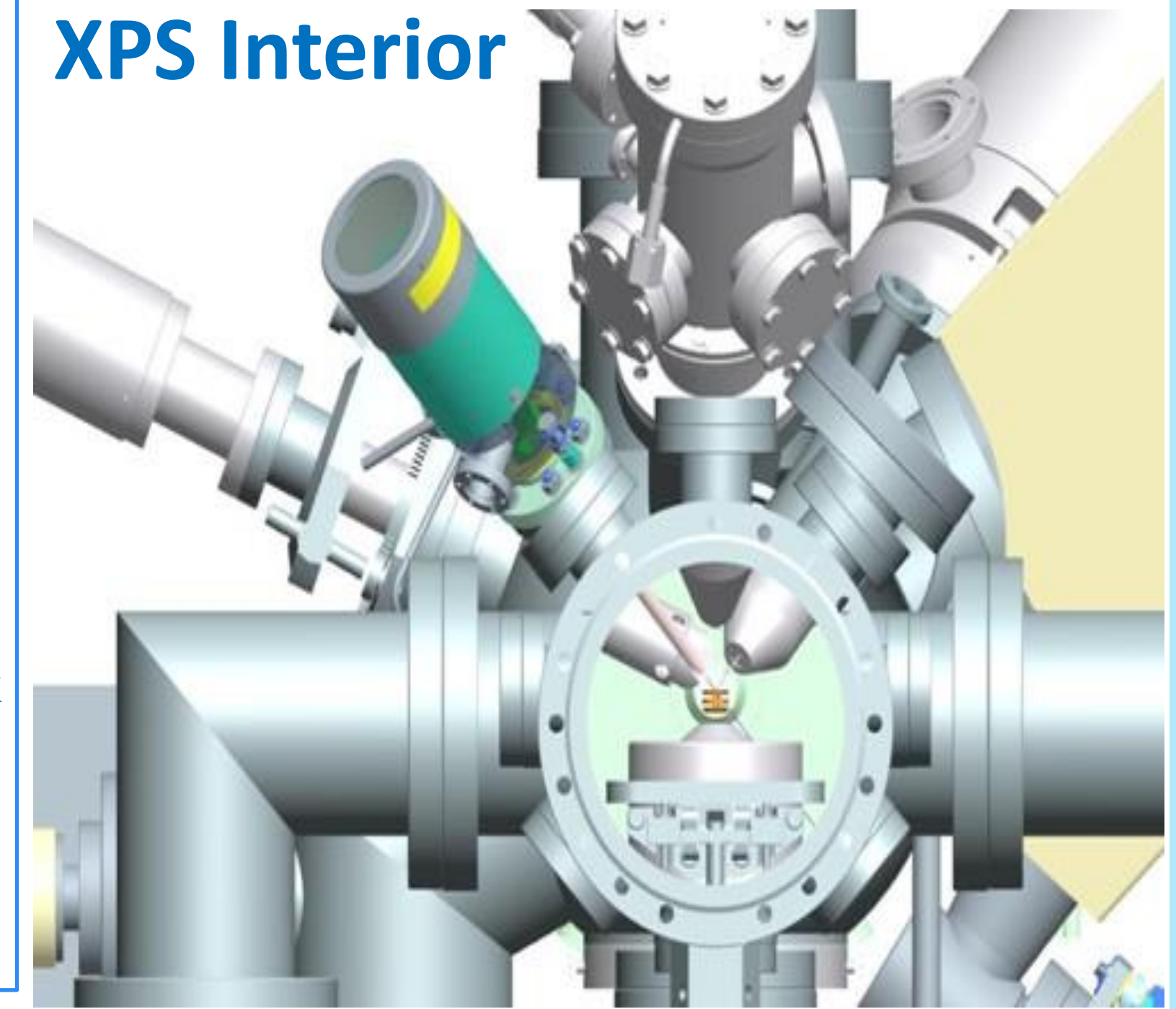


APPLICATIONS

There are several things that it can be used for in the real world. It can further undergo process, that will allow it to be used as a heat shield for thermocouple, to allow temperature reading in places that are extremely hot or that are over 1100°C . Application also extends to protecting planetary reentry vehicle, from burning and to protect the equipment, samples, and collected data, that are on board, from overheating whenever the shuttle is entering a planet from space. All this applications can be attained, because while the temperature is high or low, it will take considerable amount of time for calcium sulfate to undergo a phase change.

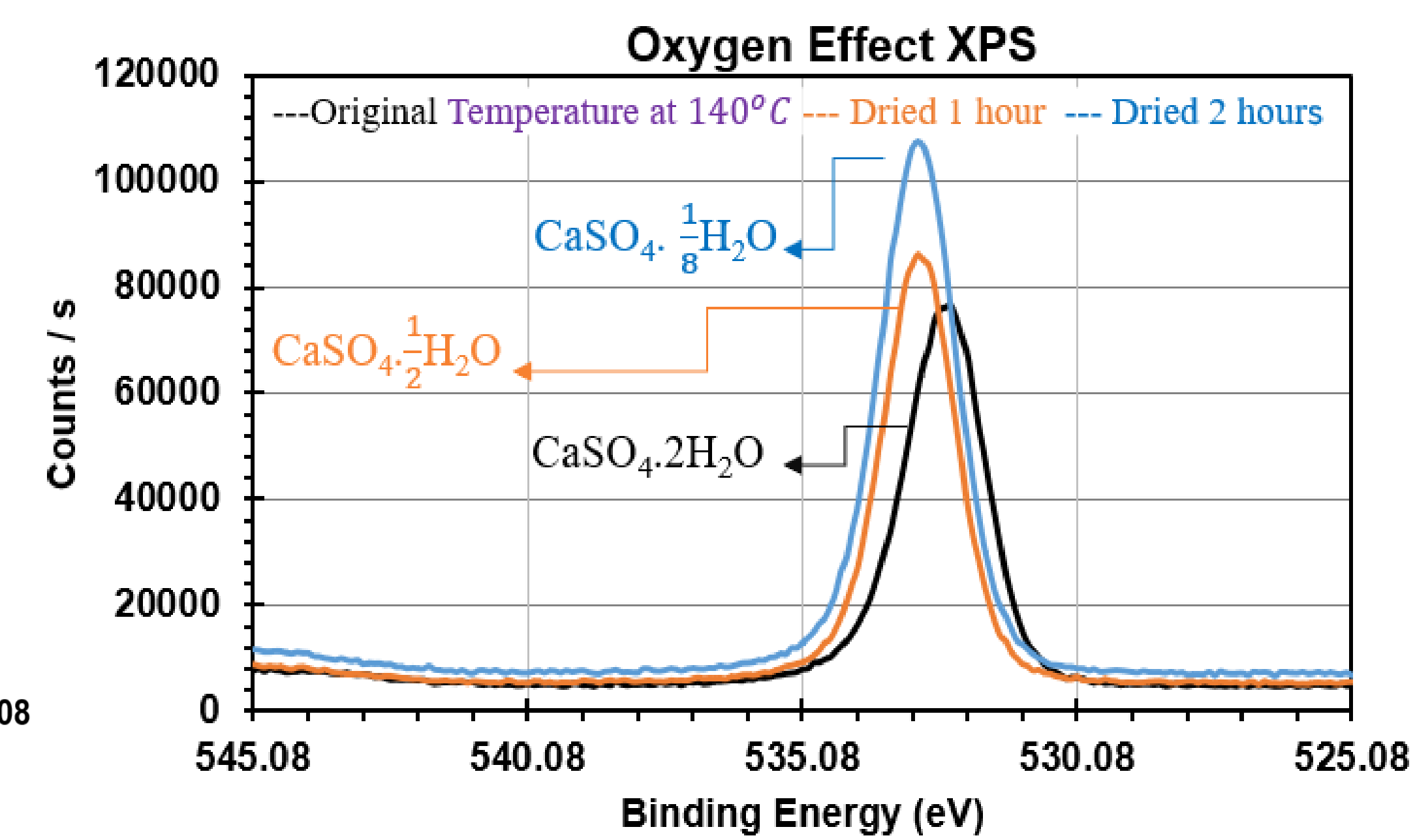
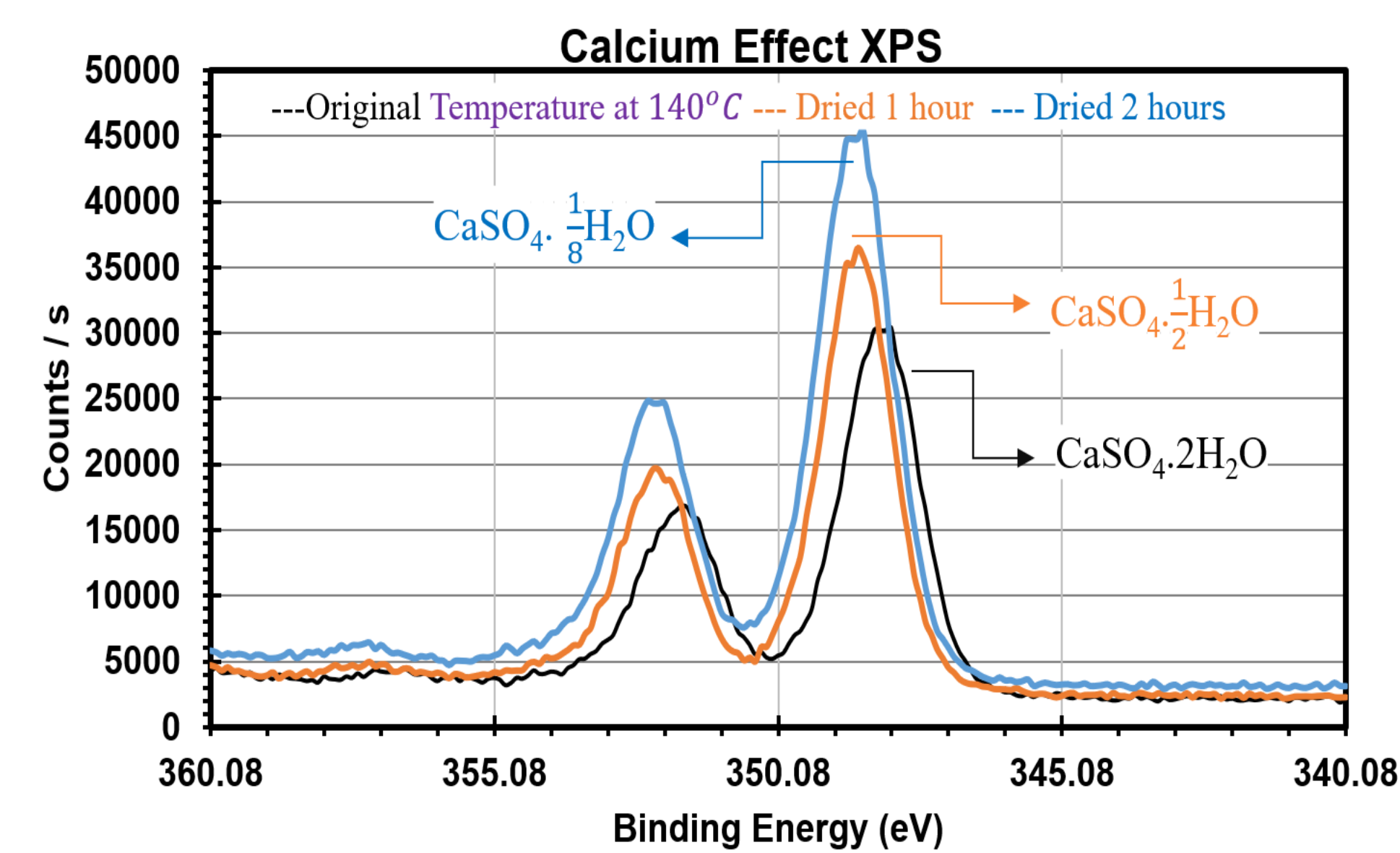
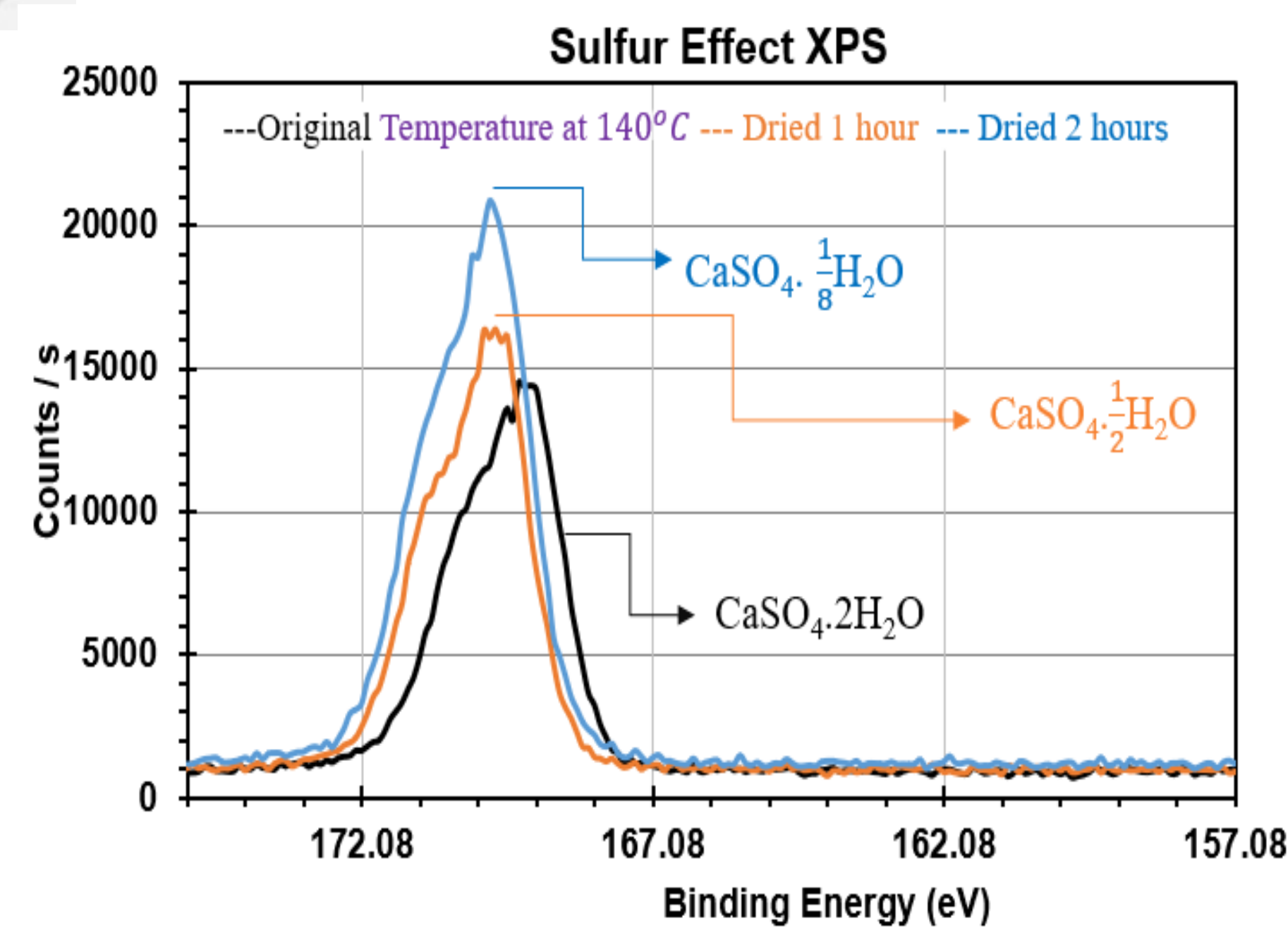
METHODS AND OBJECTIVE

Sample of $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ was put in the ampule. Excessive particles were removed as not to cause contamination. It was put in the furnace after setting it at temperature of 140°C . It was then dried for one hour. Then it was placed in the X-Ray Photoelectron Spectroscopy(XPS) and Raman Spectroscopy for analysis and characterization. It was then put back in the furnace to further dehydrate for another hour. It was taken for second section of analysis and characterization.



RESULTS AND DATA

The results and data collected are shown in these graphs. The Raman Spectroscopy shows that the H_2O molecule, has moved towards a higher Raman shift, after heating the raw material for over 1 hour. This means that $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ has undergone changes, turning into what is now $\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$. While the X-Ray Photoelectron Spectroscopy(XPS) shows that each element on the surface has attained a higher binding energy level (electron volts eV), due to the reduction in H_2O , we can read that there is a binding energy shift in the peak of Calcium, Oxygen, and Sulfur, after one hour.



CONCLUSION

Gypsum undergoes dehydration to accomplish bassanite. The more time it spends under-going the process, the more likely it will turn into the anhydrous state. While it is in the $\text{CaSO}_4 \cdot \frac{1}{8}\text{H}_2\text{O}$ state, it can be reversed, and undergo hydration process towards dihydrate. This has a greater chance of happening at lower damp temperature over a long period of time. The color changes to the sample during dehydration or hydration is almost not noticeable. Only time determines whether it undergoes transformation after it is at temperature of 140°C .

REFERENCE

- [1] Alexandre Merlen, Josephus Buijsters, Cedric Pardanaud. "A Guide to and Review of the Use of Multiwavelength Raman Spectroscopy for Characterizing Defective Aromatic Carbon Solids: from Graphene to Amorphous Carbons." Oct. 2017 <<https://hal.archives-ouvertes.fr/hal-01596681/document>>
- [2] Blomfield Chris "Applications of High Resolution in XPS and AES" KRATOS ANALYTICAL Sep 2021 <<https://slideplayer.com/slide/6612319/>>
- [3] Byjus Education Tech "Chemistry on Calcium Sulfate CaSO_4 " Jan 12, 2022, <<https://byjus.com/chemistry/calcium-sulphate/>>
- [4] Charles Q. Choi "New Spaceship Antenna Prevents Radio Silence During Fiery Re-Entry" Jun 16, 2015, <<https://www.space.com/29675-hypersonic-spacecraft-communications-reentry-tech.html>>
- [5] Engbrecht, Dick C., and Hirschfeld, Deidre A. "Measurement of heat flow through cast slabs of calcium sulfate dihydrate." United States: N. p., 2019. <Web. doi:10.1002/fam.2715.>
- [6] Fincknor M. Miria "Materials for Spacecraft" NASA, Marshall Space Flight Center, Alabama <<https://ntrs.nasa.gov/api/citations/20160013391/downloads/20160013391.pdf>>
- [7] Fine art America "Computer Monitor" Feb 9, 2022, <<https://fineartamerica.com/featured/glowing-computer-monitor-with-graphs-ikon-ikon-images.html>>
- [8] Grimm Groupe "XPS and UPS Background" Aug 15, 2021 <<https://grimmgroup.net/research/xps/background/>>
- [9] James Lambert "Overview of the Compact Integrated Raman Spectrometer (CIRS) for a Europa Lander Mission (415-4)" Jet Propulsion Laboratory Pasadena, CA June 27, 2019, <file:///C:/Users/vladr/Downloads/CL%2319-3609.pdf>
- [10] Monson Chris "SpaceX Red Dragon entering Mars' atmosphere" Vision of SpaceX's Dragon 2 capsule. May 25, 2020. <<https://www.humanmars.net/2020/05/spacex-red-dragon-entering-mars.html>>
- [11] ThermoFisher Scientific "X-RAY PHOTOELECTRON SPECTROMETERS" Oct 7, 2021, <<https://www.azom.com/equipment-details.aspx?EquipID=5143>>
- [12] Voynick Steve "Gypsum and Anhydrite" Rock and Gem, Calcium Sulfate CaSO_4 May 30, 2018, <<https://www.rockngem.com/gypsum-and-anhydrite/>>
- [13] Zimo Sheng, Jun Zhou, Zhu Shu, Yahaya Yakubu, Yun Chen, Wenbin Wang, Yanxin Wang, "Calcium sulfate whisker reinforced non-fired ceramic tiles prepared from phosphogypsum", "Boletín de la Sociedad Española de Cerámica y Vidrio", Volume 57, Issue 2, 2018, Pages 73-78, <<https://www.sciencedirect.com/science/article/pii/S036631751730095X>>

ACKNOWLEDGEMENTS

This work was supported in part by the U.S. Department of Energy (DOE) NNSA MSIPP award number DE-NA0003980, and Office of Defense Nuclear Nonproliferation Research and Development, the DNN R&D (NA-22) and the National Science Foundation (NSF) HBCU-UP Program through award number 1818732

I would like to thank , Dr. Mebougna Drabo, Department of Mechanical Engineering, Dr. Matthew Edwards, Department of Physics, Mathematics and Chemistry, Dr. Aschalew Kassu, Department of Mechanical, Civil Engineering, and Construction Management, and Dr. Stephen Egariyewe, Department of Electrical Engineering, Alabama A&M University, Huntsville, Alabama, USA.

