

New Silicon-Containing Composite Materials for Tackling Reactive Oxygen Species in Disease

Bella Minick and Jeffery L. Coffey, Ph.D.
Texas Christian University
Department of Chemistry and Biochemistry



I. Introduction

Reactive Oxygen Species (ROS) are associated with a broad spectrum of diseases, ranging from bone loss to cancer. One strategy to combat ROS is to treat sources of such species in the body with materials capable of generating hydrogen and reacting with ROS to neutralize it. This project involves incorporating an H₂-generating material known as Calcium Disilicide (CaSi₂) into membranes of another H₂-generating material known as porous silicon for tandem antioxidant drug delivery. Porous silicon (pSi) is an important substrate in drug delivery as its nano-network of pores allows controlled loading of drugs. Our approach centers on the use of spark ablation to deposit CaSi₂ into the pSi. Both porous silicon and CaSi₂ are nontoxic and can be resorbed over time in vivo.

Sample Characterization / Evaluation:

- Scanning Electron Microscopy (SEM) - morphology
- In situ Energy Dispersive X-ray Spectroscopy (EDX) - % Ca
- Criterion of highest CaSi₂ loading % is used to determine the conditions for most efficient addition of CaSi₂ into the membrane.
- We have successfully incorporated CaSi₂ into pSi membranes.
- Current experiments are attempting to measure the amount of hydrogen produced synergistically to improve the performance of pSi to treat in situ ROS production.

II. Experimental

- To prepare CaSi₂/pSi, a piece of pSi membrane is fixed to substrate with a small drop of nail polish, and CaSi₂ powder is added.
- A capillary tube is placed on the pSi and spark ablated with a high-voltage Tesla coil, causing Si atoms on the porous membrane to vaporize along with CaSi₂ and the mixture resettles upon cooling.

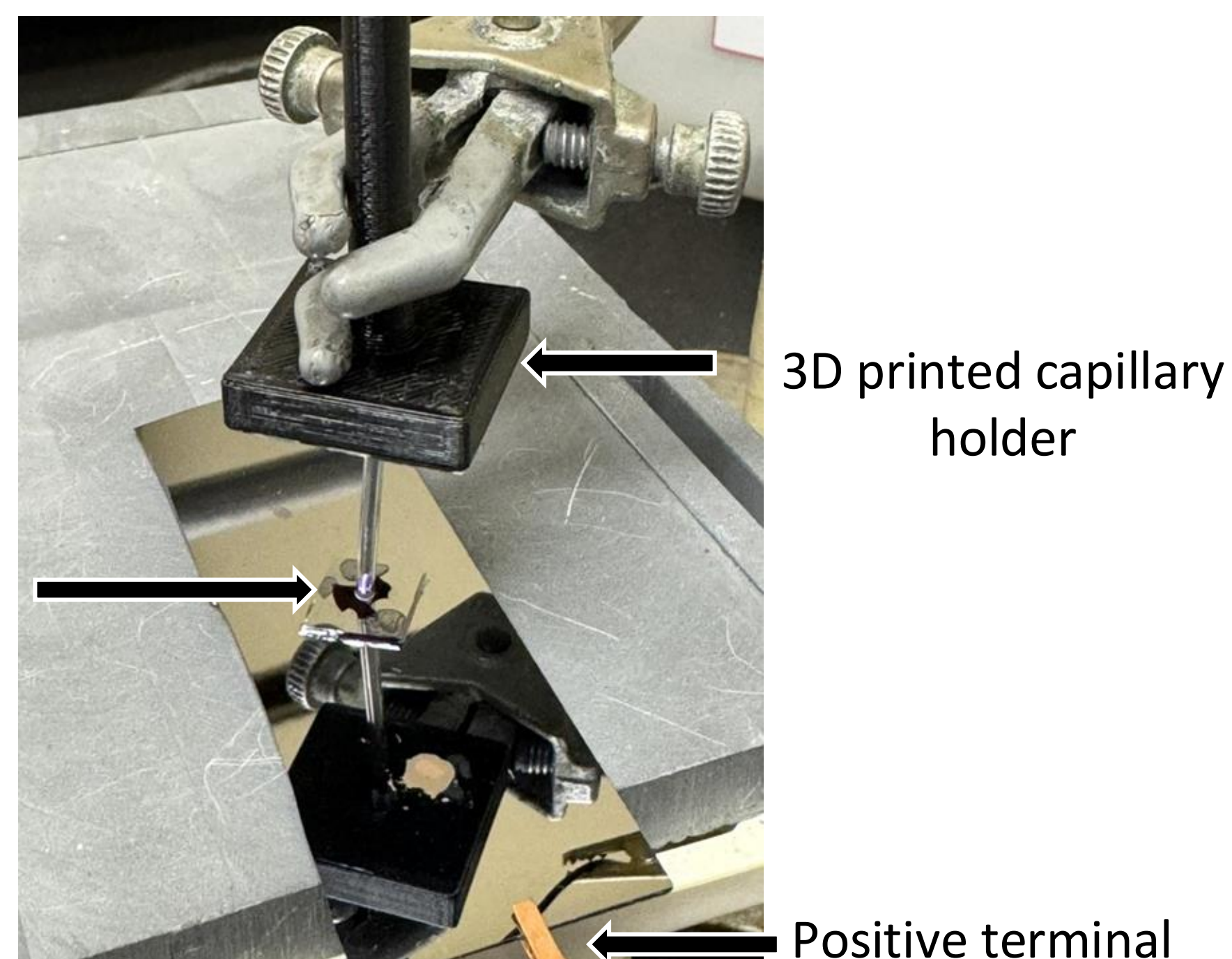


Figure 2- Experimental setup showing the 3D-printed capillary tube holder and spark ablation of CaSi₂ onto the porous silicon (pSi) membrane.

pSi / CaSi₂ Composite Fabrication

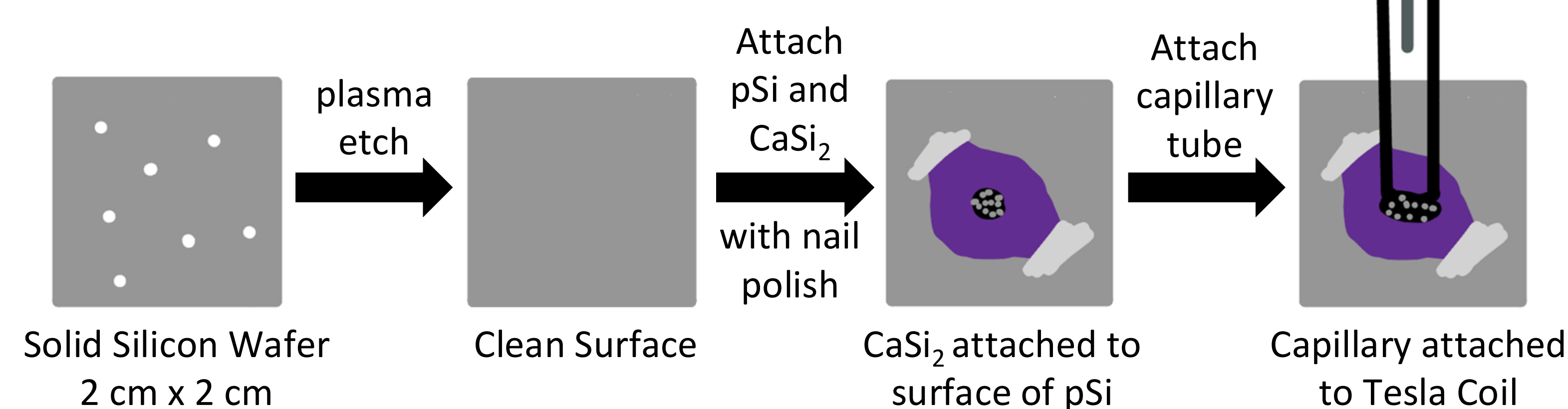


Figure 1- Schematic of CaSi₂ incorporation into porous silicon (pSi) via spark ablation using a Tesla coil.

III. Results

SEM/EDX Characterization

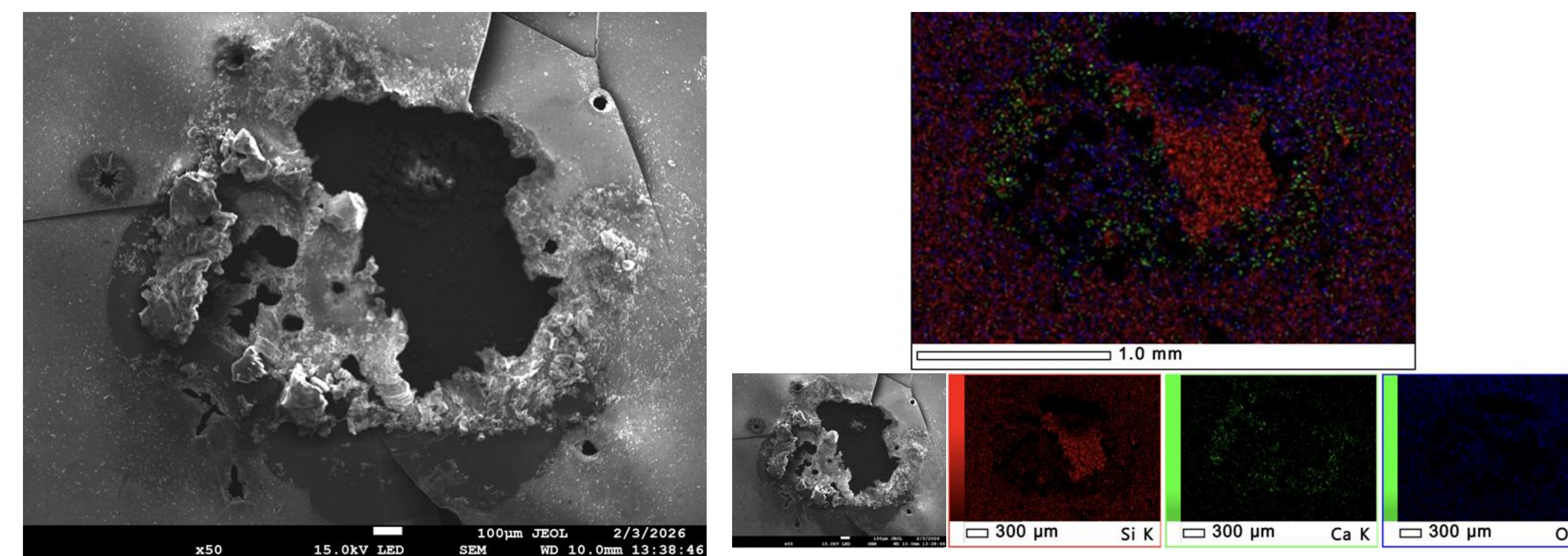


Figure 3- Left: pSi membrane after 1 hr of spark ablation. Right: EDX map showing calcium distribution on the membrane.

- Ca is concentrated around the ablated region in the EDX map, showing localized incorporation in the spark-ablated area of the pSi membrane.

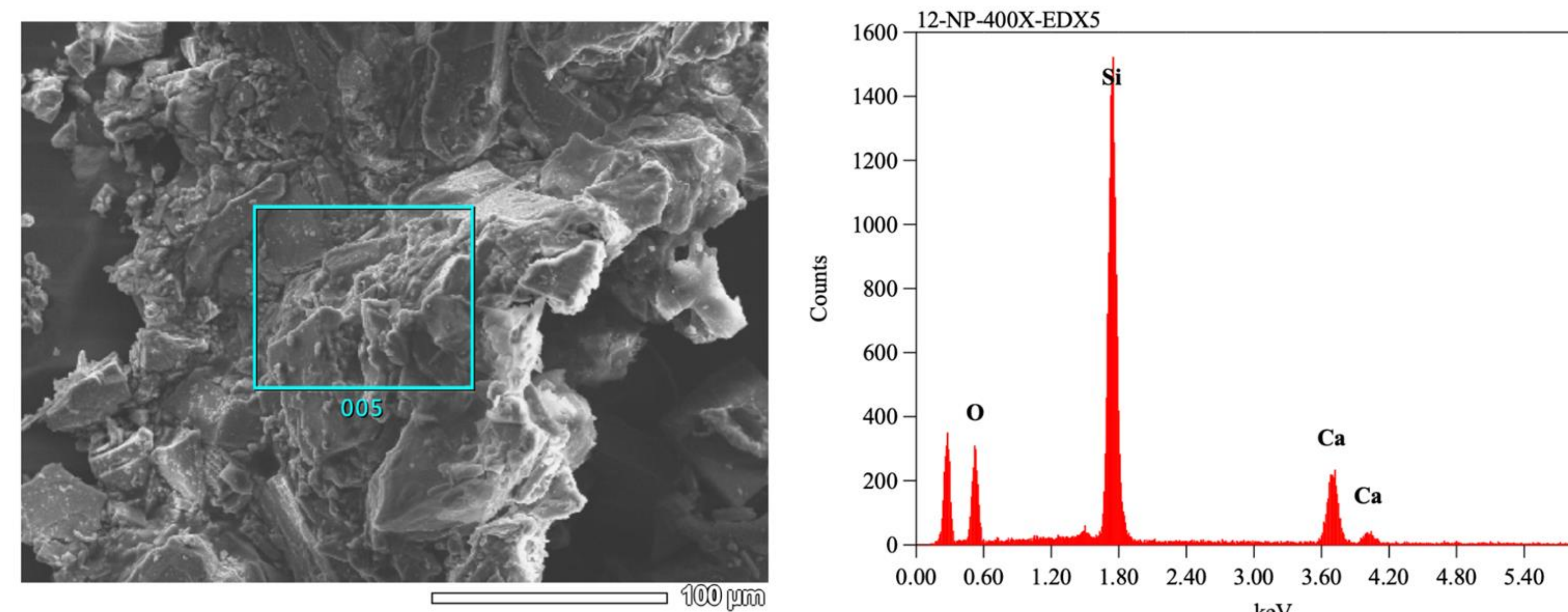


Figure 4- EDX spectrum of a CaSi₂-infused pSi membrane at 400× magnification showing a calcium mass percentage of 17.00%.

- Ca peaks confirm ~17% Ca in the sample.
- Similar morphology to CaSi₂ starting material at the ablated edge shows CaSi₂ incorporation.

IV. Discussion

The Calcium mass percentage in the membranes consistently is between 15–25%, indicating the method is reproducible. These results show that the porous silicon membrane is capable of successfully incorporating CaSi₂.

Table 1- Calcium mass percentage, membrane location, and morphology for four CaSi₂ pSi membrane trials

Trial	Ca % mass at 400x magnification	Where in sample most Ca is found	Morphology of highest Ca region
1	17.00	touching edge of small ablated hole in sample	raised surface, similar morphology to CaSi ₂ starting material
2	22.88	aggregated cluster left of ablated hole	similar morphology to CaSi ₂ starting material
3	19.71	touching edge of large ablated hole in sample	raised surface, similar morphology to CaSi ₂ starting material
4	20.75	aggregated cluster left of ablated hole	similar morphology to CaSi ₂ starting material

V. Future Work

- Focus will be on measuring hydrogen (H₂) generation to evaluate the performance of CaSi₂-infused pSi for treating in situ ROS production.
- H₂ generation from CaSi₂ will be measured while optimizing the experimental conditions.
- Once the method is consistent, the same conditions will be applied to CaSi₂-incorporated pSi membranes to compare hydrogen production and assess any improvement from incorporation.



Figure 5- Hydrogen-rich pen setup measuring H₂ (ppb) from CaSi₂ powder.

VI. References

1. Chen, S.; Yu, Y.; Xie, S.; Liang, D.; Shi, W.; Chen, S.; Li, G.; Tang, W.; Liu, C.; He, Q. Local H₂ Release Remodels Senescence Microenvironment for Improved Repair of Injured Bone. *Nat. Commun.* **2023**, *14*, 7783.
2. Seregin, V. V.; Coffey, J. L. Bias-Assisted in Vitro Calcification of Calcium Disilicide Growth Layers on Spark-Processed Silicon. *Biomaterials* **2006**, *27*, 3726–3737.
3. Seregin, V. V.; Coffey, J. L. Biomaterialization of Calcium Disilicide in Porous Polycaprolactone Scaffolds. *Biomaterials* **2006**, *27*, 4745–4754.

VII. Acknowledgments

TCU Department of Chemistry and Biochemistry; SERC