Development and Characterization of Methacrylated Silk Fibroin (Sil-MA) for Biomedical Applications

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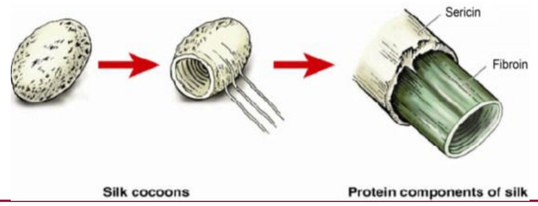
Introduction

- Silk Fibroin (SF) derived from *Bombyx Mori* cocoons are becoming more prevalent for biomedical applications due to its:
  - biocompatibility
  - mechanical properties
  - biodegradability
  - promising clinical applications
- When SF is chemically modified with glycidyl methacrylate (GMA), it becomes a more compatible bioink (Sil-MA).
- **This research refines the synthesis processes of SF and Sil-MA to enhance reproducibility for future biomedical applications.**



Methods

- SF synthesized to ensure that the proper characteristics were being obtained
- Sil-MA synthesized from same sample batches for better comparisons of the data
- 9.3M LiBr solution made fresh for every dissolution step
- Dialysis timeline for SF and Sil-MA vary
- Fourier Transform Infrared (FTIR) Spectroscopy utilized to examine the bonds present in the solution post-dialysis

Synthesis of SF

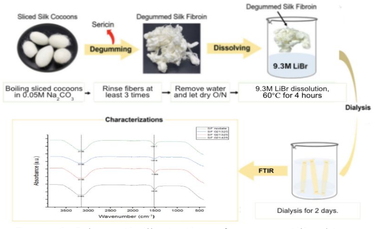


Figure 1. Schematic illustration of process utilized to synthesize silk fibroin.

Synthesis of Sil-MA

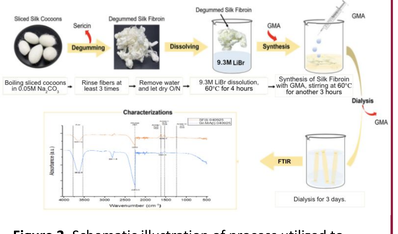


Figure 2. Schematic illustration of process utilized to synthesize methacrylated silk fibroin (Sil-MA).

Results

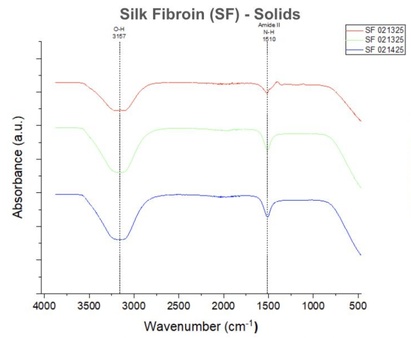


Figure 3. First three gelatinous samples analyzed via FTIR. The only amide band present is Amide II which can indicate excessive water absorption near Amide I.

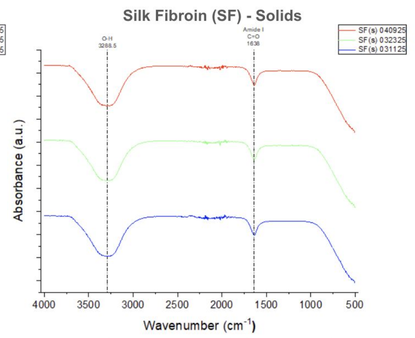


Figure 4. After pH was titrated, three more gelatinous samples were analyzed via FTIR. The only amide band present is Amide I which suggests a high β-sheet content.

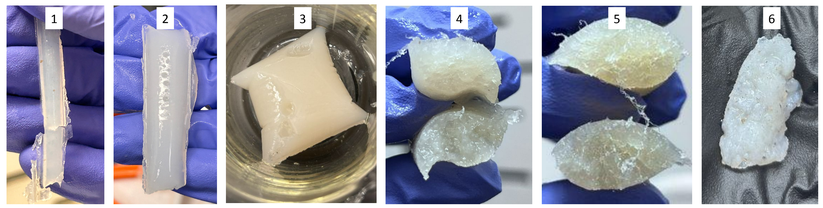


Figure 5. Post-dialysis gelatinous outcomes. Batches 1, 2, and 3 were analyzed in Figure 3. The texture was rubbery and dense-feeling. Batches 4, 5, and 6 were the outcomes when the pH was titrated to basic and analyzed in Figure 4. The texture was softer and more squishy, yielding easily to pressure. It was also not completely gelatinous, where liquid was also extracted.

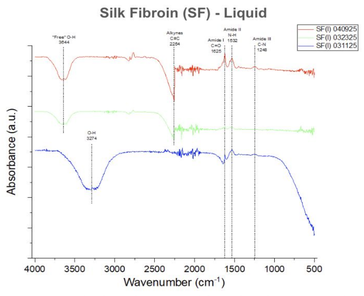


Figure 6. The liquid extracted from batches 4, 5, and 6 were characterized. SF is present proven through the key Amide bands. Titration of the water using NaOH revealed itself in the Free O-H bonds and Alkynes bonds.

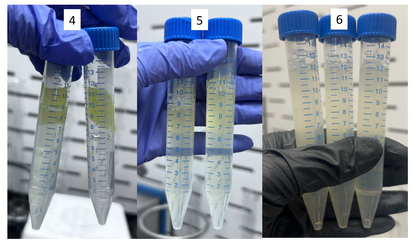


Figure 7. The liquid extracted from batches 4, 5, and 6. Batches 4 and 5 resembled water and were very low in viscosity, whereas in batch 6, had the pale yellow tint, with the viscosity of light syrup. These samples were analyzed in Figure 6.

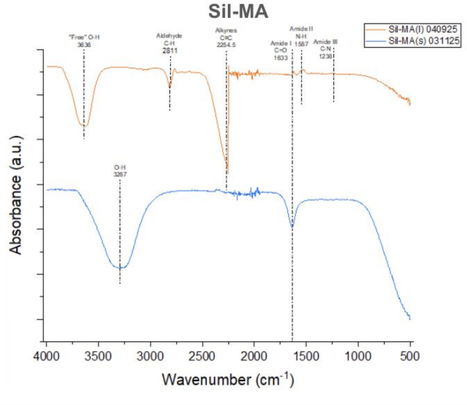


Figure 8. Sil-MA was attempted twice. When looking at the legend, the "l" and "s" in the parentheses represent the state of which the sample was analyzed. Similar to the solids analyzed in Figures 3 and 4, "Sil-MA(s) 031125" follow the same characterization with only the Amide I being present. For "Sil-MA(l) 040925," all the key amide bands confirm that the presence of SF. However, the Alkynes bond reveal that effect of the use of NaOH for titration to make the water more alkaline. The Aldehyde bone reveals that there was some type of chemical interaction between the GMA and the SF, but that specific peak does not confirm the synthesis of Sil-MA.

Discussion

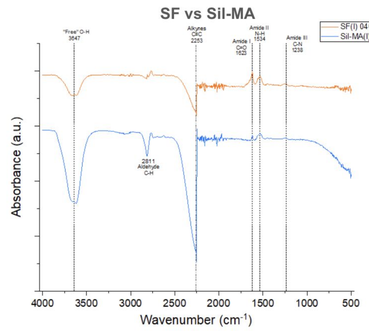


Figure 9. The SF and Sil-MA that are being compared in the FTIR graph were synthesized from the same initial batch of degummed cocoons. The reason it was done like this was to observe how GMA interacted with SF. The distinguishing factor that would have confirmed the synthesis of Sil-MA would be a methacrylate peak demonstrated around 1700 cm^{-1}. Since that particular bond is absent, it confirms that Sil-MA was not sufficiently synthesized. Although Sil-MA cannot be confirmed with this graph, it can be confirmed that there was, in fact, a chemical modification that occurred between SF and GMA, demonstrated through the Aldehyde bond.

- pH and LiBr dissolution should continue to be parameters that are monitored to ensure reproducibility of results
- The liquid extractions demonstrated to be SF via FTIR
- Sil-MA was not synthesized completely as the methacrylate bond was absent in the FTIR

Future Direction

Continue Synthesis of Sil-MA → **Sil-MA + LAP** (photoinitiator) → **3D Bioprint Implant**

References

Read Final Report Here

Watch Video Overview Here

Acknowledgements

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