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Effect of Gelation Route on Pore Architecture, Swelling, Degradation of Silk Fibroin Lyogels

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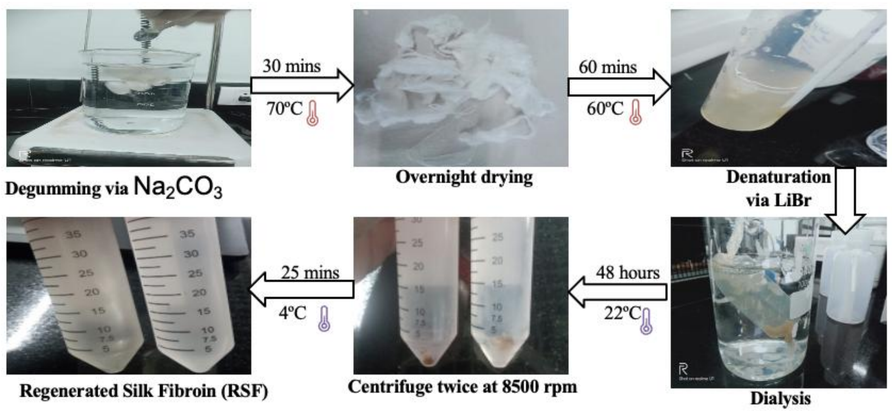
INTRODUCTION & AIM

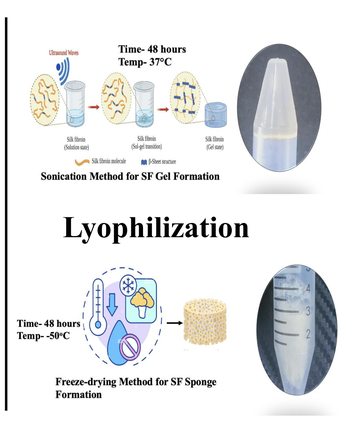
Silk fibroin (SF) is a promising natural biomaterial for biomedical applications due to its excellent biocompatibility, biodegradability, and tunable mechanical properties. Lyogels derived from SF offer interconnected porous structures that can facilitate fluid transport, molecular delivery, and tissue regeneration. However, the influence of gelation methods on the structural and functional properties of SF lyogels remains insufficiently understood.

Aim: To investigate the effect of method of gelation by ultrasonication and self-agglomeration on the structural, physicochemical, and release properties of SF lyogels, and to evaluate their potential for sustained molecular delivery applications.

Method: The lyogels were prepared via two different approaches of gelation, first through ultrasonication for 2mins at 40% amplitude followed by incubation at 37 ºC and second via self-agglomeration facilitated at 4ºC for 2 weeks to form hydrogels. In both cases the SF hydrogels were then frozen at -20ºc for 24 h followed by lyophilisation for 48 h at -50ºc and 0.00 mbar vacuum which can be then rehydrated to get the lyogels.

METHOD





RESULTS & DISCUSSION

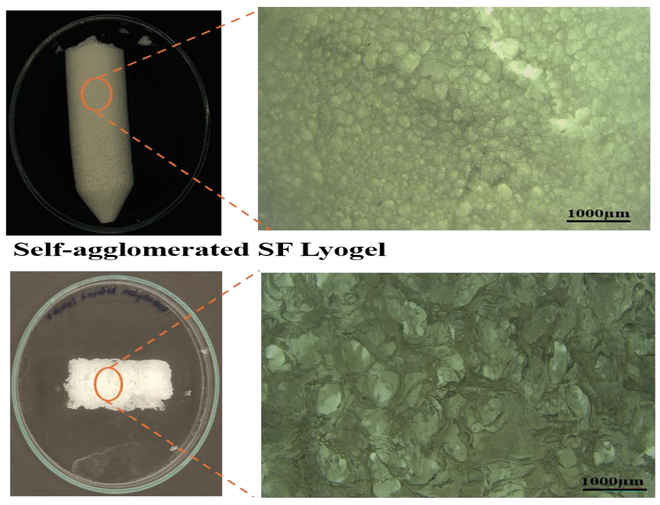


Figure 1: Microscopic images of SF lyogels at 1X magnification

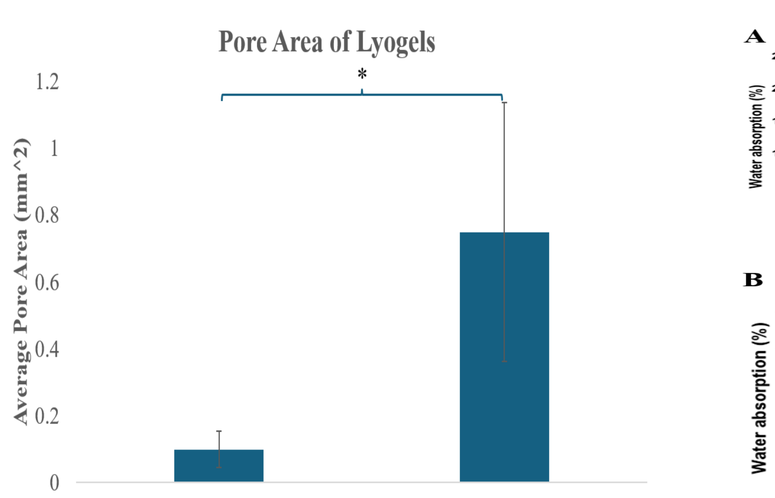


Figure 2: Graph shows the significant difference in the pore area of the SF lyogels

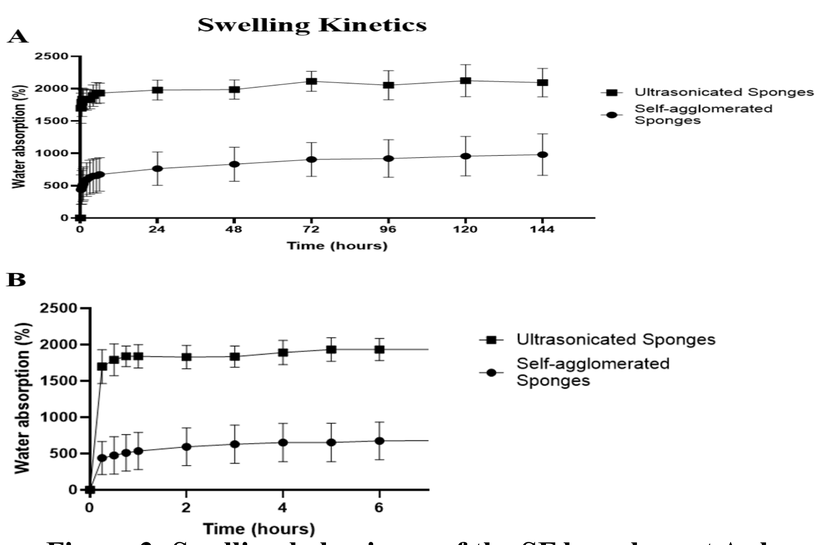


Figure 3: Swelling behaviours of the SF lyogels part A shows the swelling behaviour up to 144 h, Part B is the subset of A focusing on initial 6h behaviour

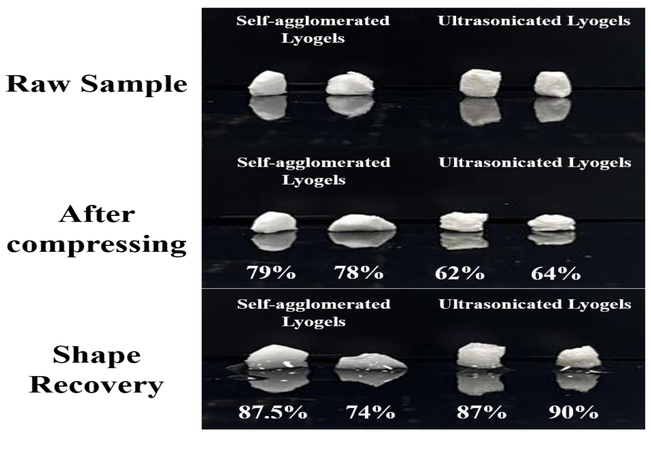


Figure 4: Shape recovery behaviours of the SF lyogels

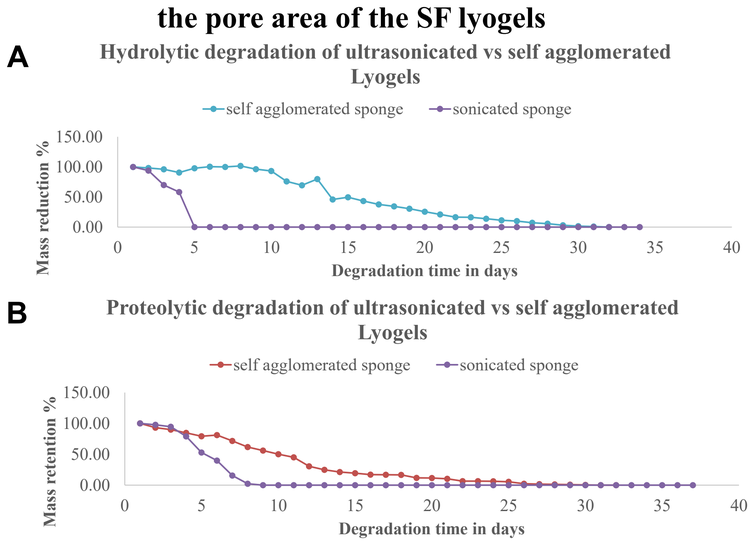
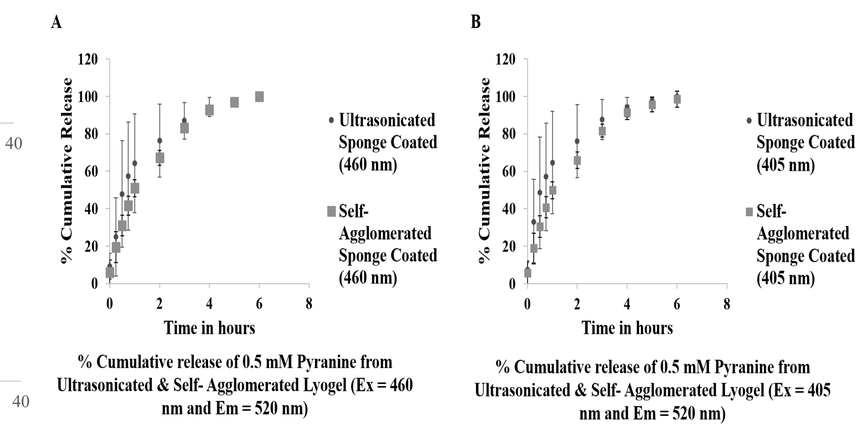


Figure 5: Degradation of the SF lyogels under hydrolytic & proteolytic conditions



% Cumulative release of 0.5 mM Pyranine from Ultrasonicated & Self- Agglomerated Lyogel (Ex = 460 nm and Em = 520 nm)

% Cumulative release of 0.5 mM Pyranine from Ultrasonicated & Self- Agglomerated Lyogel (Ex = 405 nm and Em = 520 nm)

Figure 6: Release profile of the Pyranine dye at 405 nm & 460 nm from SF lyogels via coating method

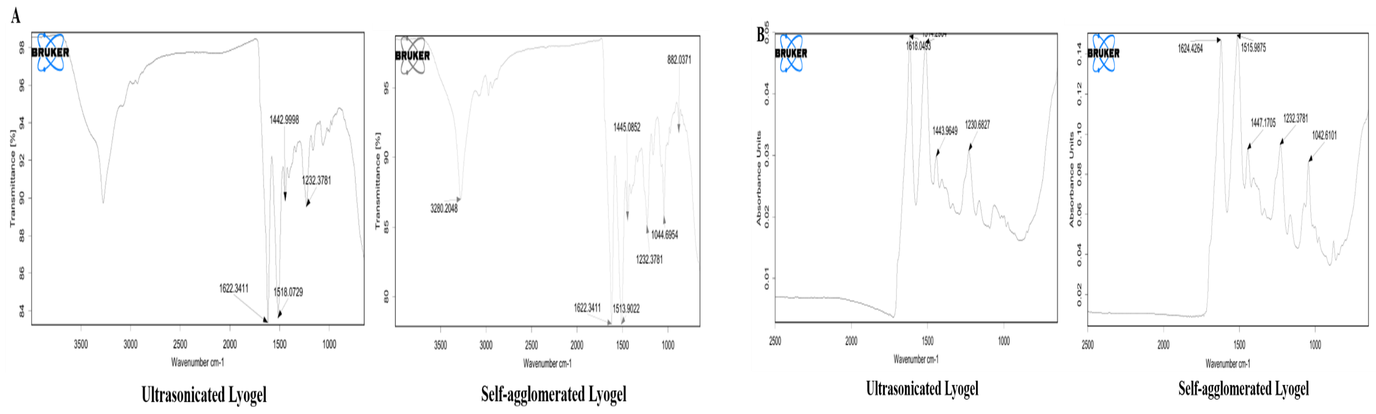


Figure 7: A FTIR; Transmittance & B FTIR; Absorbance: characteristic silk fibroin amide bands: Amide I (1618–1624 cm^{-1}), Amide II (1513–1518 cm^{-1}), and Amide III (1230–1232 cm^{-1}), confirming β-sheet-rich silk II secondary structure.

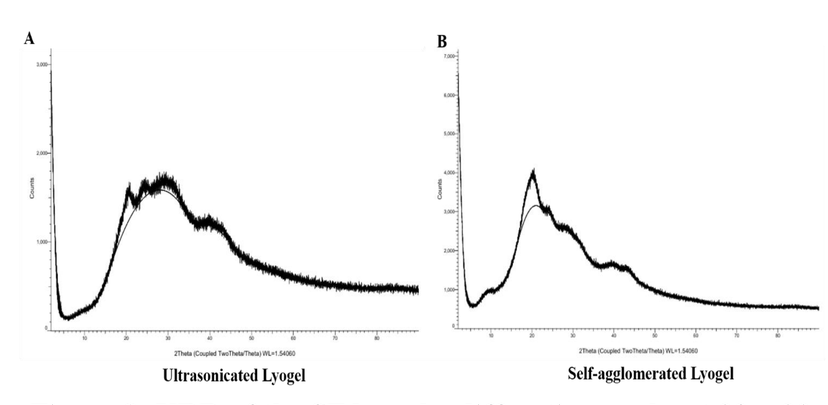


Figure 8: XRD of the SF lyogels; diffraction peaks at 2θ ≈ 20–21° and a shoulder at 24–25°, indicating silk II β-sheet crystalline domains within an amorphous matrix.

CONCLUSIONS

1. Ultrasonication significantly influenced the microstructure and performance of SF lyogels, producing a highly porous network with larger pore areas, enhanced water absorption (597–1113%), superior water retention (87.54%), rapid shape recovery, and faster molecular release (T_{50} = 41 min).
2. Self-agglomerated lyogels exhibited higher β-sheet crystallinity, lower porosity (38.5%), reduced water uptake (168.3%), and slower release kinetics (T_{50} = 64 min).
3. FTIR and XRD analyses confirmed Silk II formation in both lyogels, with greater molecular ordering in the self-agglomerated samples.
4. Overall, self-agglomerated SF lyogels demonstrated superior physicochemical properties and show strong potential as biomaterials for controlled drug delivery and wound healing.

FUTURE WORK/ REFERENCES/ACKNOWLEDGMENT

https://www.nature.com/articles/s41598-024-63061-4
https://sci-hub.se/https://www.sciencedirect.com/science/article/abs/pii/S095965261932503X

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