Evaluation of Aging of Hydroxyapatite Sol-Gel Precursor Solutions Via Viscosity Measurement for Control of Conformal Orthopedic Implant Coating Uniformity and Thickness

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Introduction

There exists an unmet need in replacing metal internal implants with biocompatible implants which allow for improved osteointegration and supports natural bone regeneration. Hydroxyapatite (HA), a mineral from bones, can be made from HA precursors via sol-gel synthesis. The HA sol-gel process can be used for coating of orthopedic implants and as a key component for metallic alloy replacement.

The **aging process** in sol-gel synthesis plays a critical role in the dip coating process that determines the **microstructure, porosity, and homogeneity** of implant coatings. There are no coherent studies that **dictate the optimal time between when the sol-gel is made and at a desired gelation state to ensure a proper coating.** The goal of this project is to measure the **viscosity** of different HA sol-gel concentrations over **time** to assess its potential to serve as a **key parameter** in the **dip coating** process.

Background

Theory: **Newtonian fluids** have a constant viscosity independent of shear rate, while **non-Newtonian fluids** have variable viscosity that changes when stress is applied. A **cone and plate viscometer** is used to measure the viscosity of non-Newtonian fluids. When the cone is rotated at constant speed (ω), viscosity measurements are obtained at uniform values of shear rate and shear stress across cone diameter.

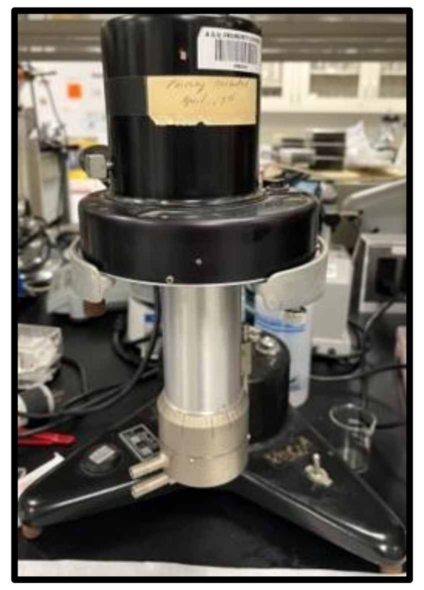

Fig 1. Cone/Plate Viscometer

Shear stress (dynes/cm²) = $\frac{T}{2/3\pi r^3}$

Shear rate (sec⁻¹) = $\omega / \sin\theta$

Viscosity (cP) = shear stress x 100/shear rate

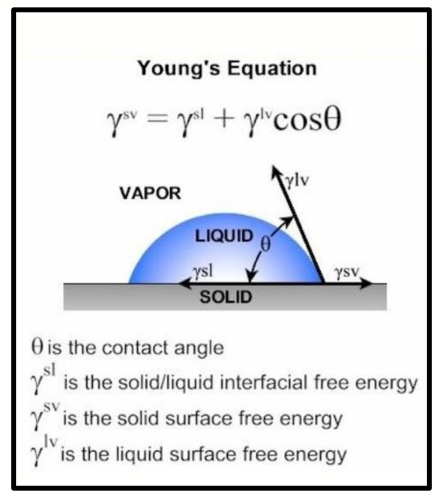

Fig 2. Contact Angle

Methods

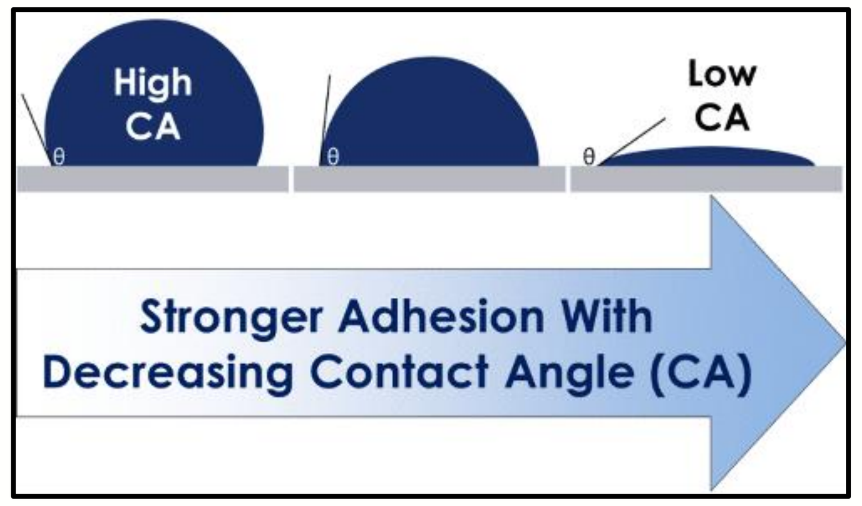

Fig 3. Diagram showing how contact angle dictates adhesion

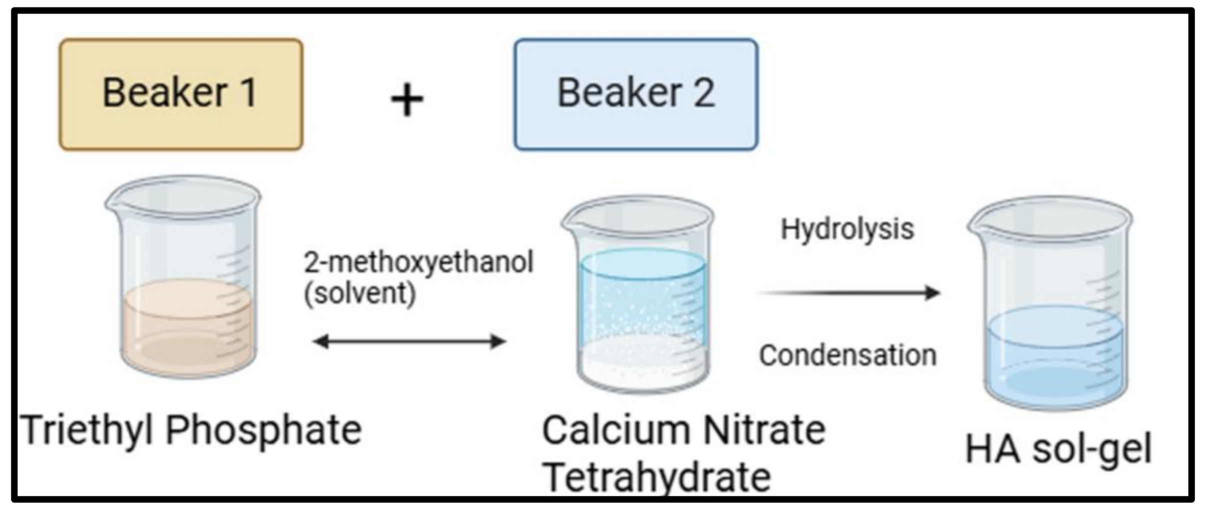

Fig 4. Sol-gel synthesis method

Step 1: Prepare HA Sol-gels
Step 2: Measure pH of Sol-gel Solutions
Step 3: Measure Contact Angle
Step 4: Measure Viscosity (Brookfield Viscometer)
Step 5: Determine Gelation Time Closest to Dip Coating

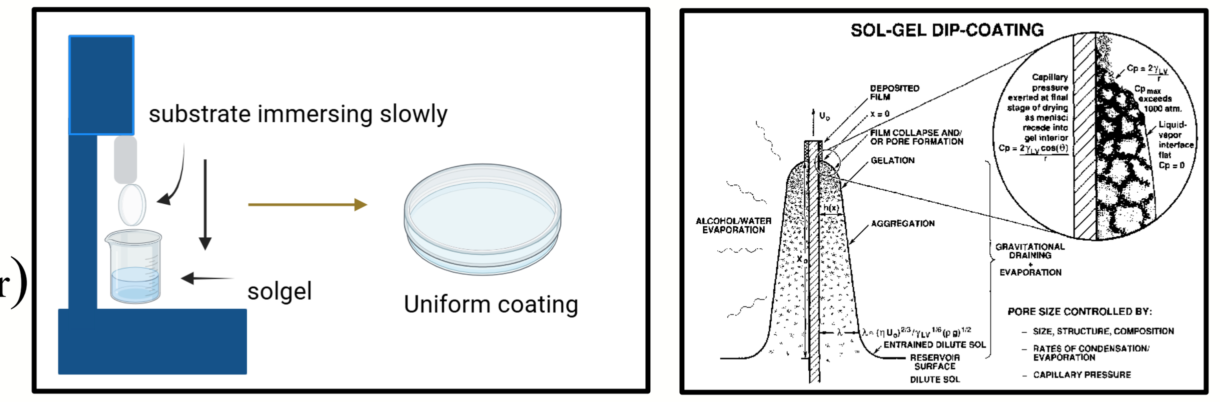

Fig 5. Dip Coating Process - shear rate approximated as the withdrawal velocity (*Vw*) /the film thickness (*h*), or *γ = Vw/h*

Results

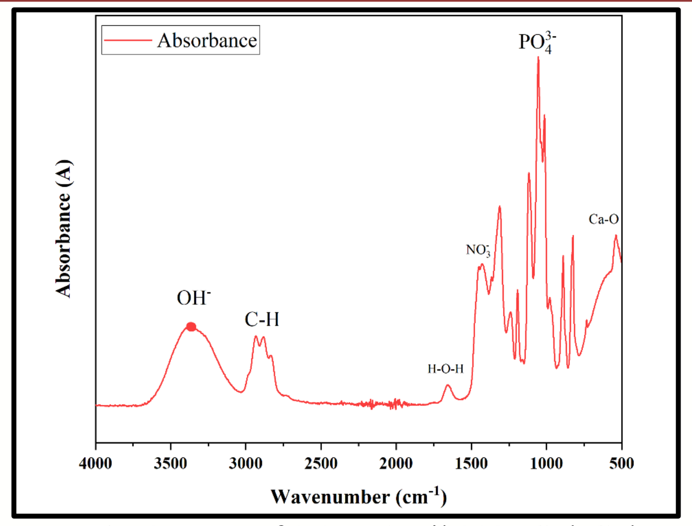

Fig 6. FTIR of 0.1 M April 2025 Sol-Gel

Average Contact Angle: 24˚

Droplet	Contact Angle
1	16˚
2	29˚
3	27˚

Table 1. Contact Angle Measurements

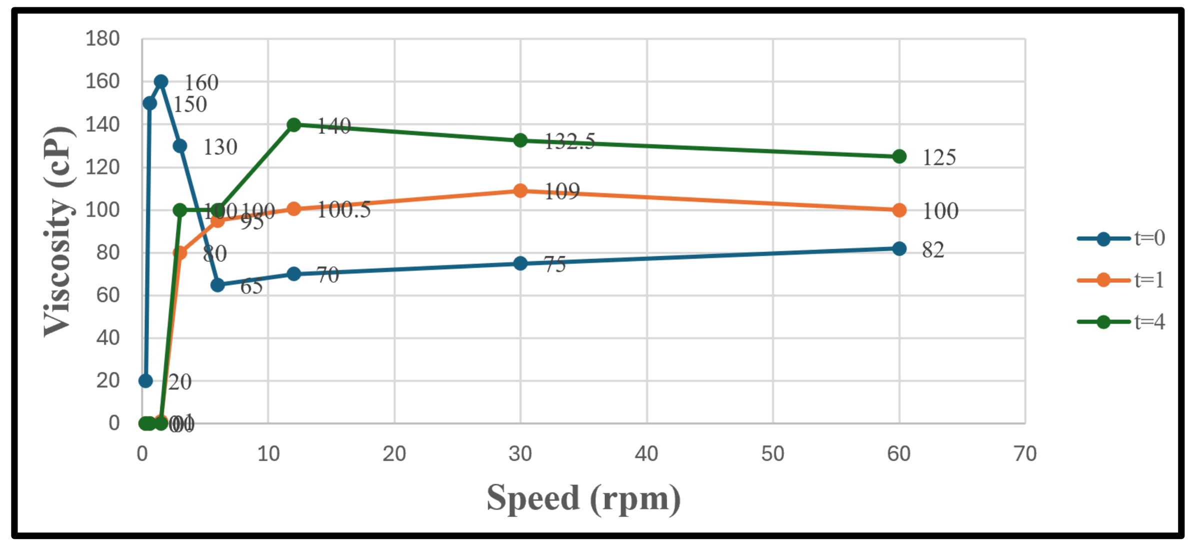

Fig 7. 0.5 M HA Sol-gel Viscosity Measurements vs Time

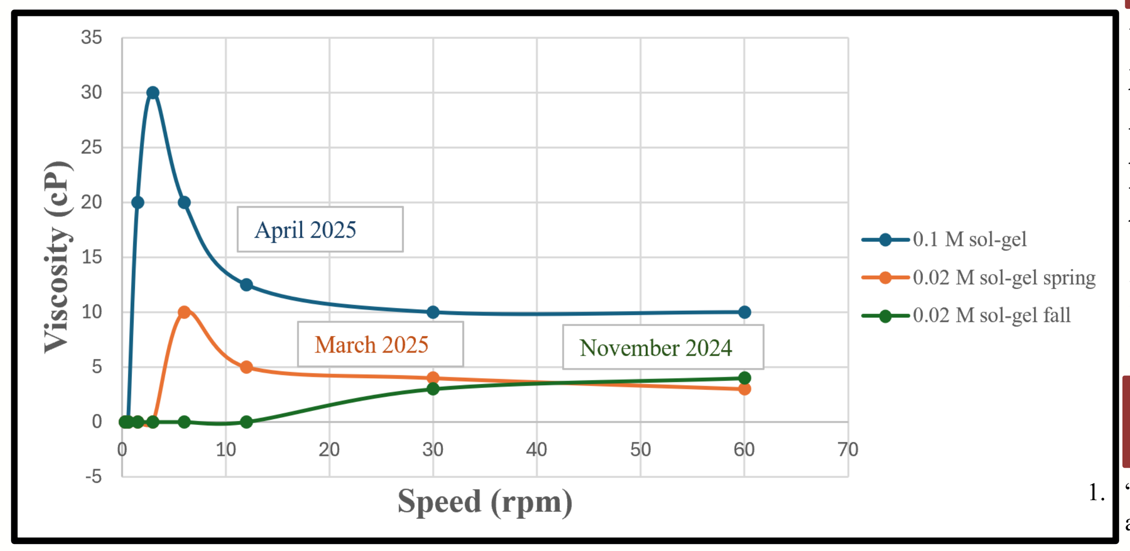

Fig 8. Viscosity Measurements of Aged HA Sol-gels

Discussion

FTIR characterization of the HA sol-gel verifies it contains the chemical signature that is consistent with HA sol-gel precursor components.

The results from contact angle measurements indicate that the HA sol-gels utilized in this study exhibit favorable wetting properties.

Viscosity measurements indicate that the HA sol-gels tested exhibit non-Newtonian, shear thinning behavior.

Increase in sol gel viscosity with increased time (high shear rates at asymptotic limits) indicate that viscosity measurement can serve as an indicator of the gelation state favoring conditions of uniform coatings of desired thickness.

Next Steps

Extend sol-gel viscosity measurements over a designated range of HA sol-gel concentrations.

Determine favorable time point(s) of the sol-gel process to ensure high quality HA films.

Verify optimal gelation time points that can produce consistent HA coating thickness.

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