Examining the Rheological Properties of 3D Fiber Infused Gels with Silver Nanoparticles

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Introduction

Cellulose is the most abundant, renewable, naturally occurring polysaccharide extracted from various sources, including trees, plants, and cotton. Cellulose hydrogels can be fabricated using various chemical or physical crosslinking methods.[1,2] Hydrogels are composed of three dimensional hydrophilic polymer chain networks and large amounts of water. Hydrogels are highly absorbent and have wide applicability in fields such as drug delivery, tissue engineering, sorbents, contact lenses, and purification.[3] Using the tetramethylpiperidine-1-oxyl (TEMPO) chemical oxidation process, macroscopic cellulose can process into cellulose nanofibrils (CNFs).[4,5] CNFs feature outstanding intrinsic properties due to their high specific stiffness and strength, attractive nanoscale dimensions, high surface areas, and having the ability to undergo chemical functionalization.[3] CNFs in suspension have the same capability to make yield hydrogels macroscopic cellulose. Silver nanoparticles (NPs) can be incorporated into CNFs hydrogels network via chemical reduction.[6] Silver NPs have major applications in the medical fields from diagnostics and antimicrobial agents. Herein, we discussed a green method of making CNF hydrogels infused with silver NPs via a photocatalytic assisted chemical reduction method. The mechanical properties of these hydrogels will be ascertained from rheological measurements.

Procedure

CNFs and $AgNO_3$ solutions

- As received cellulose nanofibrils (CNFs) in suspension (1.10 wt%) with deionized water were used to make CNFs and silver nitrate ($AgNO_3$) solutions. [CNFs were provided by Forest Products Laboratory]
- The density of the suspension was experimentally determined to 0.999 g/mL. Therefore, five 10 mL CNF suspension with molarity of 1 mM, 2mM,3mM, 4mM & 5mM of $AgNO_3$ solutions were made.

Gel Formation

- The solutions were added to well plates -uncovered- to allow gel formation over 24 h and 48 h.

Rheological Studies

- Rheological studies was performed on the Discover HR-2 rheometer to measure rheological properties of storage (G') and loss moduli (G") CNFs/Ag^+ hydrogels after 24 h and 48 h at 25 °C.
- The dynamic modulus of the hydrogel was measured as a function of frequency with a strain rate of 0.5% over frequencies of 0.1 and 100 rad/s.

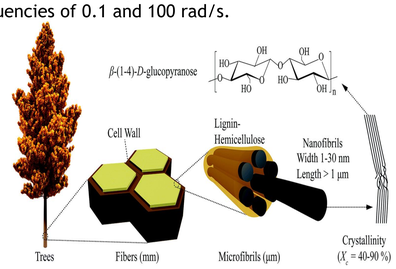


Figure 1. (A) Schematic structure of nanocellulose, starting from its origin to the organic framework found in CNFs (B)Photos of the CNF (C) freestanding gels formed by addition of various amounts of silver nitrate to the CNF

Results and Discussion

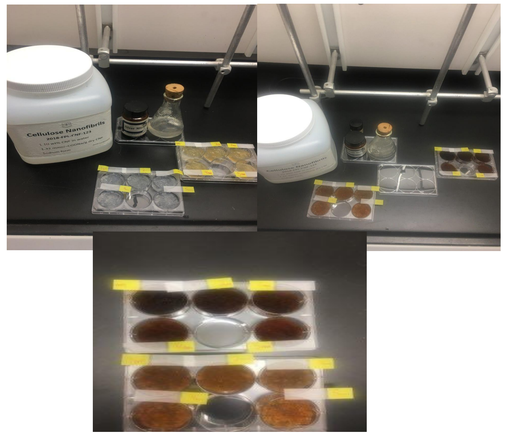


Figure 2. Gels formed with 1 ml of silver nitrate into 1ml, 2ml,3ml, and 4 ml of cellulose solutions. (left) Samples placed in complete darkness and (right)samples placed in direct sunlight.

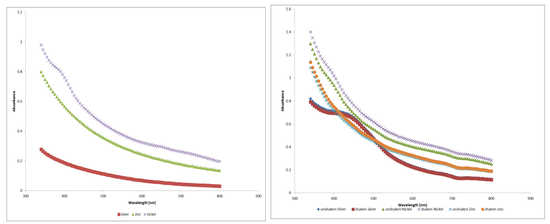
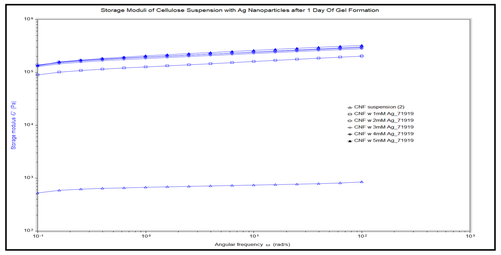


Figure 3. Comparison Study: (l) UV-Vis Spectra of cellulose nanomaterials solutions against Ag^+, Ni^{2+} and Zn^{2+} ions on day 1. (r) UV-Vis of samples 9 days later.

Figure 4: Summer Research Experience for Teachers (SRET) Mentor, Dr. Tracy Brown-Fox instructing SRET Fellow Ms. Tiara Davis on proper utilization of the Discovery HR-2 Rheometer to be used for her rheological studies for her research on the gelation of CNF suspension in the presence of silver ions.

Figure 5. Values of storage modulus and loss modulus taken at frequencies of 0.1 and 100 rad/s for CNF suspension and CNF suspensions with 1mM and 5mM, after 2 days of gelation. Observed between 30 -50% weight loss between the CNF/Ag+ solutions and freestanding hydrogels.

	Frequency (rad/s)	G' (Pa)	G" (Pa)
CNF	0.1	526.6	89.57
	100	850.7	58.95
CNF with 1mM of silver nitrate	0.1	76357.9	20253.5
	100	162006	25697.5
CNF with 5mM of silver nitrate	0.1	108813	34358.2
	100	249236	35703.8



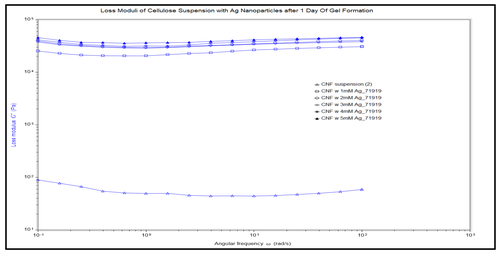
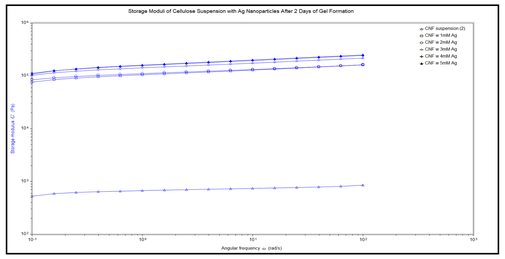


Figure 6:Viscoelastic properties of the CNF suspension and CNF/Ag+ ions: dynamic frequency sweep (25°C) of the gels at a strain rate of 0.5% (top) storage modulus plots, (bottom) loss modulus plots after 1 day of gelation.



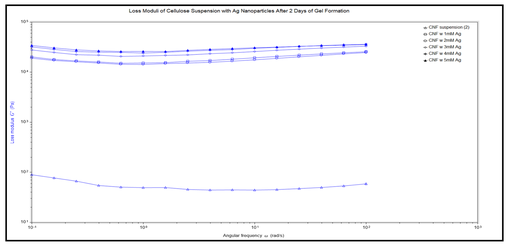


Figure 7:Viscoelastic properties of the CNF suspension and CNF/Ag+ ions: dynamic frequency sweep (25°C) of the gels at a strain rate of 0.5% (top) storage modulus plots, (bottom) loss modulus plots after 2 days of gelation.

Conclusions

- The gel formation is primarily dictated by the interactions of the fibrils carboxylate (COO^-) groups and metal cation interaction of the metal silver ions.
- The storage modulus of CNF/Ag^+ ions increases by orders of 2-3 magnitudes compared to the CNF suspension; indicative of tunable mechanical properties.
- Increasing amounts of silver ions increases the interactions between COO^- groups and Ag^+, therefore yielding higher values of storage modulus.
- The storage and loss moduli CNF hydrogels are more frequency dependent than the CNF/Ag^+ hydrogels.

Figure 8: SRET Fellows, Ms.Tiara Davis is working with a Johnson C. Smith University (JCSU)Student, Dr. Tracy Brown SRET mentor, in the New Science Center at JCSU.

Figure 9: SRET Mentor, Dr. Tracy Brown-Fox, Assistant Professor of Chemistry, SRET Fellow Ms. Tiara Davis, JCSU student researcher, and Dr. Russell discussing SRET research. Dr. Russell was Tiara's former chemistry professor at JCSU.

Figure 10: SRET Fellow Ms. Tiara Davis is demonstrating to a student researcher at JCSU how to properly use the Discover Hr-2 Rheometer.

Classroom Application

Below are a few North Carolina Common Core State Standards that proved applicable during this research:

- Scientific Processes: includes Science as Inquiry
- Understand the properties of matter and changes that occur when matter interacts in an open and closed container: NC- 8.P.1
- Compare physical changes such as size, shape and state to chemical changes that are the result of a chemical reaction to include changes in temperature, color, formation of a gas or precipitate: NC-8.P.1.3
- Explain how the idea of atoms support the law of conservation of mass: NC-8.P.1.4

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