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PhotoChitosan® Rheology Study

Abstract: *The following study was conducted to assess the rheological behavior of PhotoChitosan® at two different concentrations with varying contents of LAP photoinitiator using the Elastosens rheometer. PhotoChitosan® (PC) powder was reconstituted at 1% and 1.5% and mixed with LAP photoinitiator at 0.1%, 0.2%, and 0.3% for each material concentration. Samples were crosslinked under 100% intensity exposure of 405 nm UV light for 10 minutes. Results show that as the concentration of PC and LAP increases, both the maximum shear storage modulus and the rate of photocrosslinking increases.*

Materials

Name/Description	Part Number	Lot Number	Degree of Methacrylation
PhotoChitosan®	5428	9456	3.9%
LAP	85073-19-4	BRCLAP-I-0324-01	-
DPBS	21600-044 1x50 L	2814720	-

Results

Figure 1 below shows a comprehensive graph of the rheological curves of PhotoChitosan® (PC) at 1% and 1.5%, with varying LAP concentrations of 0.1%-0.3%. The vertical dotted lines at minutes 5 and 15 represent the start and end of UV photostimulation respectively at 100% intensity. Results show that as the concentration of PC increases, both the maximum shear storage modulus (G')—or rigidity of the material—and the rate of photocrosslinking increase. Increasing the concentration of LAP at a fixed PhotoChitosan concentration also increases both G' and rate of photocrosslinking.

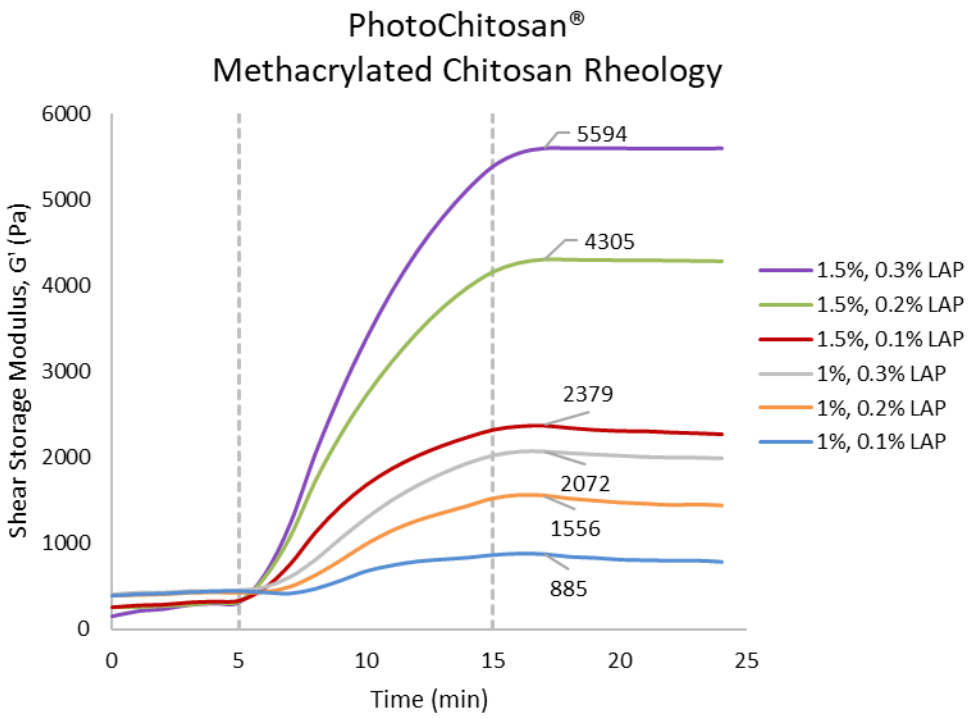

Figure 1. Compilation of viscoelastic curves of photocrosslinked PhotoChitosan® at concentrations 1% and 1.5%, and varying LAP concentrations of 0.1%, 0.2%, and 0.3%. Dotted lines indicate the start and end of 405 nm UV light photostimulation.

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Conclusion

The ability to reconstitute PhotoChitosan® at different concentrations and also mix with different photoinitiator concentrations allows for the potential to effectively fine-tune the final photocrosslinked hydrogel stiffness for various applications. PhotoChitosan® provides a stiffness ranging from about 800 – 5600 Pa; manipulating other variables such as temperature, time of exposure, and UV light intensity can potentially provide further variations and fine-tunability to produce a desired hydrogel with distinct rheological properties.

Procedure

The following sample preparation and experimental set up procedures were performed to carry out the study. Briefly, lyophilized PhotoChitosan® was reconstituted at 1% and 1.5% with 20 mM acetic acid at 40°C. Three solutions of LAP were prepared at 50 mg/ml, 100 mg/ml, and 150 mg/ml to mix in with solubilized PhotoChitosan® and obtain final LAP concentrations of 0.1%, 0.2%, and 0.3% respectively (see *"Sample Preparation"* procedure below). Then, the Elastosens rheometer was calibrated, and the experimental parameters set (see *"Elastosens Experimental Set Up"* procedure below). Each experimental group was tested in duplicate sequentially and the test data averaged. The same bulk components, i.e. PhotoChitosan®, acetic acid, and LAP were used for all test groups in this study.

Sample Preparation

1. The corresponding volume of 20 mM acetic acid was added into individual vials of lyophilized PhotoChitosan® to yield concentrations of 1% and 1.5%. They were then gently mixed on a shaker table at 40°C overnight or until fully dissolved.
2. LAP powder was solubilized at 50 mg/ml, 100 mg/ml, and 150 mg/ml in acetic acid and covered in aluminum foil to minimize light exposure.
3. The calculated volume of LAP solution was added to each PhotoChitosan® vial (0.02x of solubilized PhotoChitosan® volume) and further mixed until homogeneous.
4. All samples were stored at 40°C prior to testing.

Elastosens Experimental Set Up

1. The Elastosens was turned on and calibrated once via vibration calibration.
2. The temperature was set to 20°C using the manual temperature control.
3. Once the temperature was equilibrated, a large empty sample cup was inserted in the testing chamber and secured. The same sample cup was used for all test groups in this study.
4. New sample files were created for each group in the following folder: PhotoChitosan.
    a. File format: product name, lot number, concentration and/or test sample description.
        i. E.g. PhotoChitosan 9456 0.1% LAP.

5. The following test parameters were set for all test conditions:
    a. Type – Stiff.
    b. Sample File – Named as noted above.
    c. Test Name – Tester's initials followed by the number of the test run under the same file name (each test was run in duplicate, and the resulting curves averaged).
        i. E.g. the first test ts01, second test ts02.
    d. Custom Information:
        i. Volume – 2 g.
        ii. Oil – No.
        iii. Concentration – 1% or 1.5%.
        iv. Photoinitiator – LAP.

v. Photoinitiator concentration – Varies.
vi. Cup size – Large.
vii. Light intensity – 100%
viii. Exposure time – 10 min.
ix. Temperature – 20°C.

6. The following test sequences were set in the "Measurement Sequences" window for all samples:
    a. Sequence 1: Thermal incubation
        i. Duration: 5 min.
        ii. Step: 1 min.
        iii. Temperature configuration: manual.
    b. Sequence 2: Photocrosslinking
        i. Duration: 10 min.
        ii. Step: 1 min.
        iii. Temperature configuration: manual.
        iv. Photostimulation LED 405nm: 100%.
    c. Sequence 3: Equilibration
        i. Duration: 10 min.
        ii. Step: 1 min.
        iii. Temperature configuration: manual.

7. The sample cup calibrated once prior to testing each group.
8. The sample cup was removed from the machine and placed on a scale to add 2 g ± 0.1 g of sample.
9. The sample-containing cup was placed into the machine and the test started.
10. After the first test was finished, a second test (following the procedure above) was run to obtain a duplicate test under the same file name.
11. The sample was removed from the cup after gelled, and the cup rinsed with milli-Q water and dried to be reused.