A New Chemical Approach to Developing Aerogels from Cellulose

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• Background
• Objectives
• Methods – NFCs Isolation and Modifications
• Methods – Aerogels Preparation
• Results – Aerogels Characterization
• Conclusions
Aerogels: Highly porous materials possessing low solids content and are considerably lightweight.

- Storage media for gases
- Cargo carrier (catalysis)
- Cellular therapeutics
- Filtration
- Heat and Sound Insulators
Renewable Resources

⇒ Hyaluronic Acid
⇒ Fibrin
⇒ Agarose
⇒ Heteropolysaccharides
⇒ Cellulose

Employ the most abundant renewable carbon resource found on the planet

High Biocompatibility
Intrinsic cellular interactions
Biodegradable
Low toxicity byproducts

Background

Cellulose

Employ the most abundant renewable carbon resource found on the planet
Objectives of this work

1) To produce aerogels from nanofibrillated cellulose (NFC) by direct water removal by freeze-drying

2) Modify NFCs using TEMPO and/or sodium tetraborate (borax)

3) Characterize the novel aerogels according to their morphology and rheological properties
**Isolation of Cellulose Nanofibers**

**Eucalyptus urograndis** (chips) → Grinding → Sawdust of eucalyptus urograndis → Extraction (Acetone 12 hours) → Extractive free-sawdust → Delignification → Holocellulose → Hemicellulose Removal (KOH 24%, R.T.) → Cellulose Fibers → High pressure homogenizer → Cellulose Nanofibers
NFC Modification

Nanofibrillated Cellulose (NFC) Suspension

TEMPO-mediated oxidation
1) DO = 0.1
2) DO = 0.2

Dialyze for 3 days then centrifuge

Centrifuge suspension

Borax (Bx)
1) Bx:Cellulose 0.09:1
2) Bx:Cellulose 0.18:1
3) Bx:Cellulose 0.36:1

TEMPO-mediated oxidation + Bx

DO = 0.1
Bx:Cellulose 0.09:1
Bx:Cellulose 0.18:1
Bx:Cellulose 0.36:1

DO = 0.2
Bx:Cellulose 0.09:1
Bx:Cellulose 0.18:1
Bx:Cellulose 0.36:1
Results – NFCs Modification

A – NFC non-oxidized
B – NFC oxidized DO=0.1
C – NFC oxidized DO=0.2

\[ DO = \frac{162 \times C \times (V_2 - V_1)}{w - 36 \times C \times (V_2 - V_1)} \]

where C is the NaOH concentration (mol L\(^{-1}\)), \( V_1 \) and \( V_2 \) are the initial and final amount of NaOH in L based on the plateau of the curve, w (g) the weight of the oven-dried sample.
Aerogel Production

Non-modified NFC suspension → Modified NFC suspension → Centrifugation → Rapid Freezing (Liquid Nitrogen) → Vacuum Freeze-Dry → Aerogels
Results – Morphology: Bx effect

Non-oxidized
Non Borax addition

Non-oxidized
Bx:Cell 0.09:1

Non-oxidized
Bx: Cell 0.18:1

Non-oxidized
Bx: Cell 0.36:1
Results – Morphology: TEMPO effect

Non-oxidized
Non Borax addition

DO = 0.1
Non Borax addition

DO = 0.2
Non Borax addition
Results – Morphology: TEMPO + Bx effect

DO = 0.1
Non Borax addition

DO = 0.1
Bx:Cell 0.18:1

DO = 0.2
Non Borax addition

DO = 0.2
Bx:Cell 0.18:1
## Results – Density

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</table>
Results – Rheology

Non-borax addition

Bx:Cell 0.09:1

Bx:Cell 0.18:1

Bx:Cell 0.36:1

Stress (Pa)

Strain

DO = 0.0

DO = 0.1

DO = 0.2
Conclusions

- Aerogels were successfully produced using NFC as starting material followed by vacuum freeze-drying;
- TEMPO-mediated oxidation showed greater improvements for the strength and homogeneity (morphology) of the final aerogels;
- TEMPO + Bx does not significantly enhance the characteristics of the aerogels;
- Bx by itself tended to reduce the density of the aerogels;
- Likewise, Bx decreases the homogeneous nature of the morphology when used on non-oxidized NFCs.
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