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### **Greener method for tannin and lignin extraction from the yellow birch bark**

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## Valorization of forest residues into polymer products

### Greener method for tannin and lignin extraction from the yellow birch bark

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- **NRCan:** *Marzouk Benali*

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Norfolk, Virginia, USA



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## Outline

- ❖ Context
  - Bark residue in North America
  - Main chemical composition of bark
- ❖ Overall project description
- ❖ Extraction process
  - Tannin
  - Lignin (preliminary results)
- ❖ Polyurethane (PU) biofoams containing tannin and lignin extracted from bark (preliminary results)
  - Methodology
  - Foaming
- ❖ Biocomposites containing bark residue after tannin and lignin extraction
  - Methodology
  - Mechanical properties
- ❖ Conclusions
- ❖ Future work



## Context

*Bark residue in North America*

(1/2)

Bark is an abundant forest residue in North America  
 ~ 17 million tons /year in Canada  
 ~ 20 million tons /year in USA



~ 40% used for energy production in pulp & paper mills



Bark landfill



Bark incineration

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## Context

*Chemical composition of the bark*

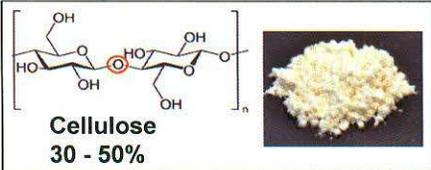
(2/2)



**Lignin**  
20 - 40%



**Tannin**  
2 - 20%

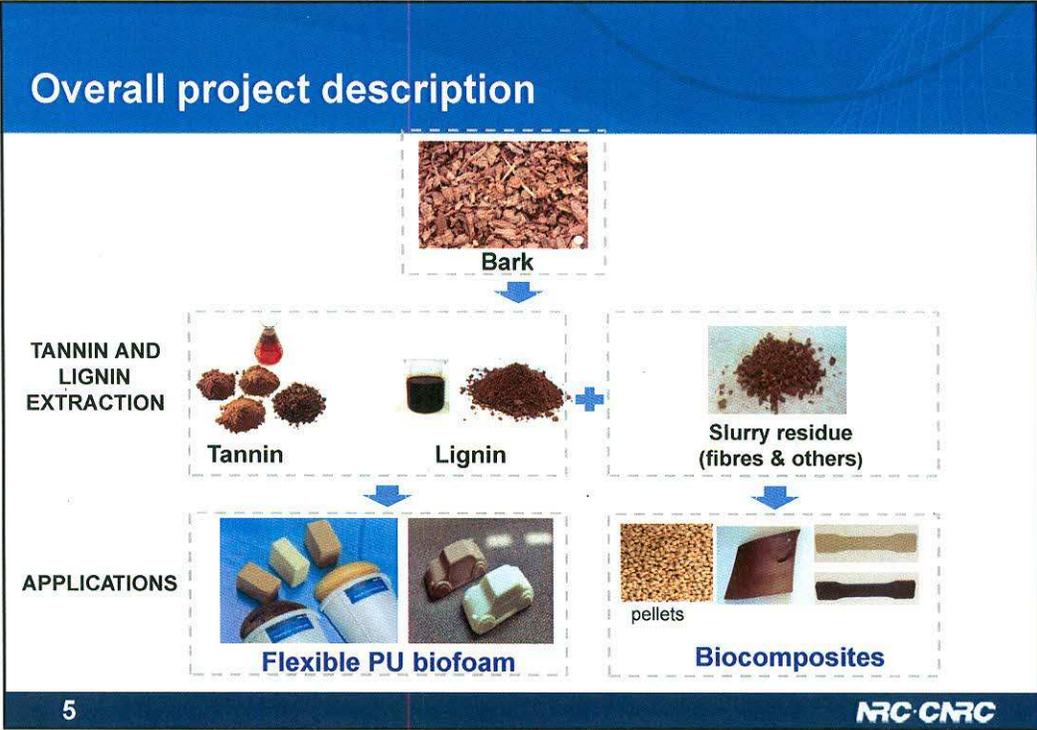


**Cellulose**  
30 - 50%

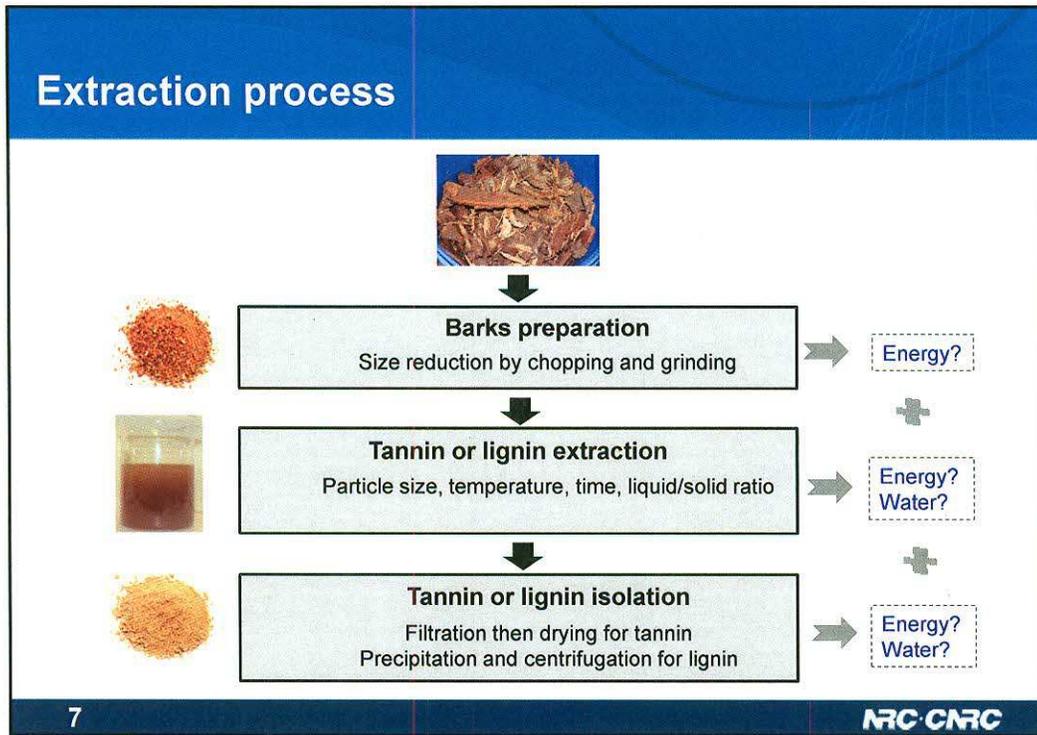
<http://winefolly.com/review/what-are-tannins-in-wine/>

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- ## Specific objective of this presentation
- **Main objective of the project:** To valorize Canadian forest residues into polymer products for industrial applications
  - **Specific objectives of this presentation :**
    - Develop a cost-effective method for tannin and lignin extraction from Canadian yellow birch bark
      - Maximize the tannin and lignin extraction yield
      - Minimize water and energy consumption → minimize operation cost
    - Develop products for industrial applications: flexible biofoams from lignin, tannin and biocomposites from residual cellulose
- 
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# Biomass preparation

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# Raw material

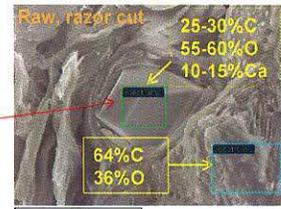
(1/3)

**Fresh yellow birch bark :** From *Coopérative forestière des Hautes-Laurentides, Mont-Laurier, QC*



**Mixture**

- Bark (main constituent)
- Wood sticks
- **Calcium** and other minerals
- Sand
- Dust
- Products of fermentation (alcohol, moisture, etc.)



Bark contains impurities → could affect extraction yield and tannin purity

# Bark size reduction : chopping and grinding (2/3)



Chopping machine



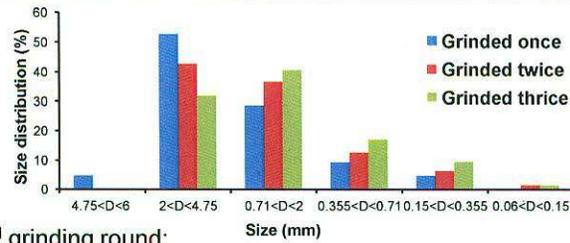
Grinding machine



## Bark size reduction : chopping and grinding (3/3)

### Energy consumption estimation for 1kg dried bark

Energy estimation (Wh/kg)	1 <sup>st</sup> round	2 <sup>nd</sup> round	3 <sup>rd</sup> round
Chopping energy	37	37	37
Grinding energy for each step	144	234	315
<b>Total energy for size reduction</b>	<b>181</b>	<b>271</b>	<b>352</b>
Size : 0.063mm < D < 4.75mm	95%	98%	99%



From the 1<sup>st</sup> to the 3<sup>rd</sup> grinding round:

- Total energy consumed almost doubles
  - No significant difference on particle size while the grinding energy more than doubles
- Grinding once is more cost-efficient

## Tannin extraction

## Tannin extraction : conventional method (1/7)



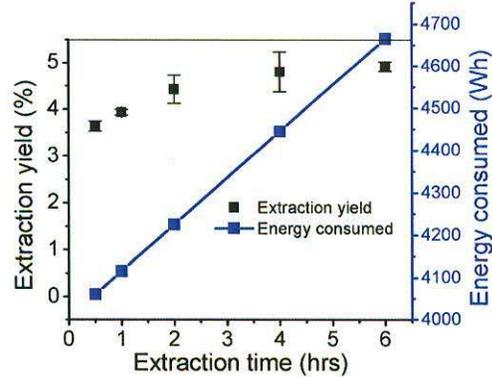
### Extraction parameters

- Demineralized water (pH : 5.72)
- Water/bark mass ratio : 2.5 - 5.0
- Temperature : 60 - 95°C
- Time : 0.5, 1, 2, 4, 6 hrs
- Mechanical stirrer : 100 rpm



Grinded bark

- Effect of the extraction time
    - Greater extraction time: a bit higher yield
    - Greater extraction time: greater energy consumption
- Extraction time not more than 2 hours



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## Tannin extraction : conventional method (2/7)

### Energy consumption estimation for 1kg dried bark using conventional method:

- Grinded bark
- Extraction for 2 h
- Oven-drying to isolate extract

<b>Total energy (Wh)</b>	4,225
<b>Energy consumption distribution (%)</b>	
Chopping	1
Grinding	3
<b>Tannin extraction</b>	<b>19</b>
<b>Tannin isolation</b>	<b>77</b>

- Tannin extraction and isolation are responsible for the major part of the energy consumption in the process .
- Need to reduce :
- Improved method 1 : energy reduction for **extraction step using ultrasound**
  - Improved method 2 : water & energy reduction for **isolation step by reusing extract solutions**

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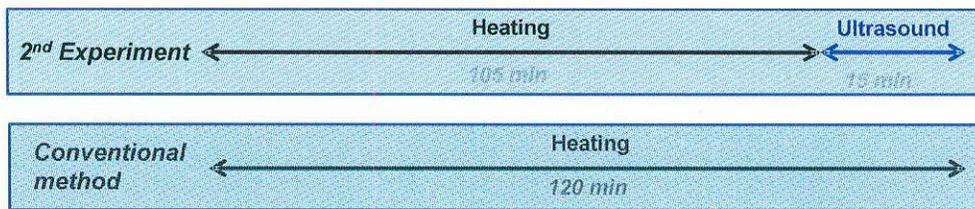
## Tannin extraction : improved method 1

(3/7)

- **Principle:** reduce the extraction time with the aid of ultrasounds to save energy for tannin extraction step

**Extraction parameters**

- Grinded bark
- Water/bark mass ratio : 2.5
- Heating and ultrasonic operating at 95°C



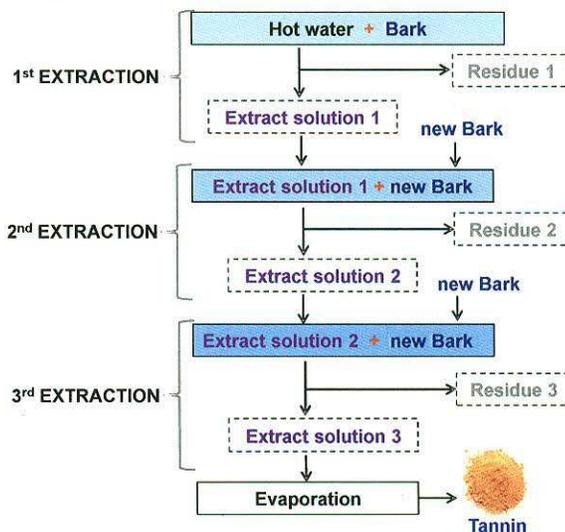
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## Tannin extraction : improved method 2

(4/7)

- **Principle :** re-use extract solution of the first extraction as solvent for the second extraction, and repeat it
- **Potential benefits**
  - Significantly reduce water consumption
  - Significantly reduce energy consumption, especially the water removal step (tannin isolation step)
  - Reduce production cost



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## Tannin extraction : comparison

(5/7)

### Energy consumption estimation for 1kg tannin

Extraction parameters

- Grinded bark
- Water/bark mass ratio : 2.5
- Heating at 95°C, 2 h

	Conventional method	Improved method 1	Improved method 2 (extract re-used only once)
<b>Tannin yield (%)</b>	2.3± 0.0	3.5± 0.0	2.4± 0.0
<b>Energy consumption distribution (kWh)</b>			
Chopping	1.59	1.04	1.18
Grinding	6.26	4.11	4.62
Tannin extraction	35.25	22.67	15.99
Tannin isolation	140.61	140.61	56.25
<b>Total energy (kWh)</b>	<b>183.71</b>	<b>168.43</b>	<b>78.04</b>

- Energy consumption of the tannin extraction and tannin isolation step decrease greatly
- Tannin yield is in the range reported in the literature (1.5 – 4.4% of dried bark)

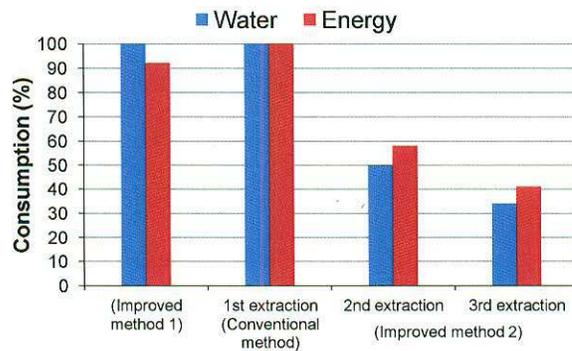
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## Tannin extraction : comparison

(6/7)

### Potential for reduction of water and energy compared to conventional method



Extraction of 2 h	Tannin yield (%)
Conventional method	2.3 ± 0.0
Improved method 1	3.5 ± 0.0
Improved method 2	2.4 ± 0.0

Improved method 2 has a great potential to significantly reduce water and energy consumption for the extraction of tannin from bark

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## Tannin characteristics

(7/7)

Characteristics	Commercial	Extracted bark
Moisture (%)	6	8
Tannin (%)	50	60
Water soluble (%)	97	98

- Similar quality as commercial tannin

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## Lignin extraction Preliminary results

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## Lignin extraction : conventional method (1/3)



Reactor

### Extraction parameters

- Grinded bark: 800 g
- Alkaline solution : 1-3% NaOH
- Alkaline /bark mass ratio : 2.5
- Temperature : 95°C
- Time : 2 hrs
- Mechanical stirrer : 100 rpm



Grinded bark



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## Lignin extraction : conventional method (2/3)

### Based on lignin extraction of 1kg dried bark

- Alkaline solution / bark mass ratio : 2.5
- Extraction for 2 h

NaOH	% (in solution)	1	2	3
	In mass (g)		25	50
Mass of water (g)	Extraction	2475	2450	2425
	Residue washing	3600	3600	3600
	Total water	6075	6050	6025
Energy (Wh)		TBD	TBD	TBD
Lignin yield	% (based on dried bark mass)	3.5 ± 0.2	10.2 ± 0.4	14.8 ± 0.3
	In mass (g)	35	102	148

- Lignin yield increases with the increase of the NaOH concentration
  - Need more sulfuric acid for lignin precipitation
- Conventional method consumes too much chemicals and water
  - Not economical and not ecological

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## Lignin extraction : improved method

(3/3)

### NRC's concept for lignin extraction from bark

**Objective:** Reduce water and energy consumption and the chemicals use

**Strategy:** Reuse the black liquor obtained from the previous extraction for the extraction of new batch to:

- Increase the lignin concentration
- Decrease the amount of water (extraction and residue washing)
- Reduce the chemicals (NaOH and H<sub>2</sub>SO<sub>4</sub>)
- Reduce the energy for the filtration after lignin precipitation

*On-going work*

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## Polyurethane (PU) biofoams containing tannin and lignin extracted from bark

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## Flexible biofoam : approach (1/2)

Tannin/Lignin extracted from yellow birch bark

↓

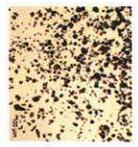
Mix Tannin/lignin with polyol  
(Dispersion/solubility, viscosity)

↓

Develop biofoam formulation  
(Foaming ability and foam properties)

↓

Prepare prototype



Lignin dispersed in polyol



Lignin soluble in polyol



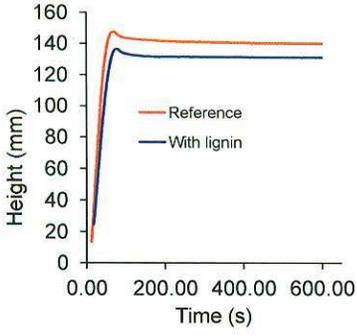
Lignin-polyol mixture flows very well



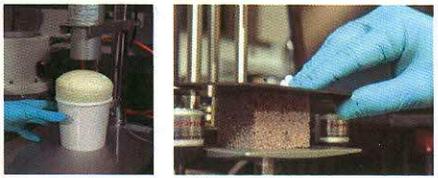
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## Flexible biofoam : foaming (2/2)

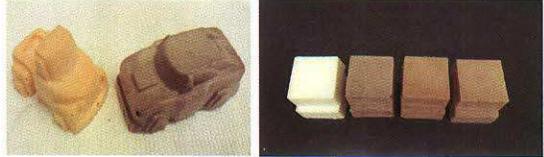
**Evolution of height (mm) vs time (s)**



Time (s)	Reference Height (mm)	With lignin Height (mm)
0.00	0	0
50.00	140	130
100.00	145	135
200.00	145	135
400.00	145	135
600.00	145	135



**Foaming and testing Lignin-PU foam**



**Lignin-PU foam prototypes**

Lignin containing biofoam has similar characteristics as conventional foam in terms of foaming and mechanical properties

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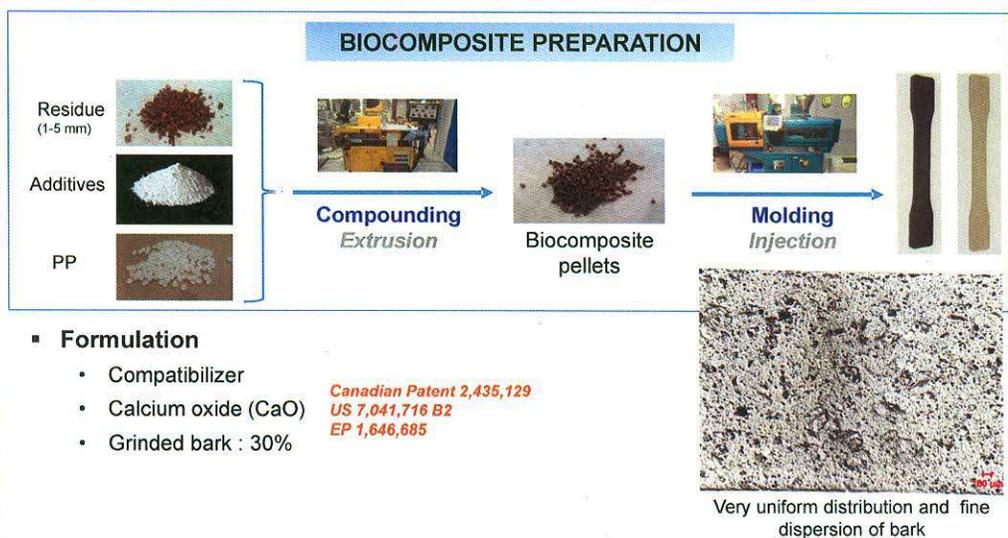
# Biocomposites containing bark residue (cellulose) after tannin and lignin extraction

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## Methodology

(1/3)



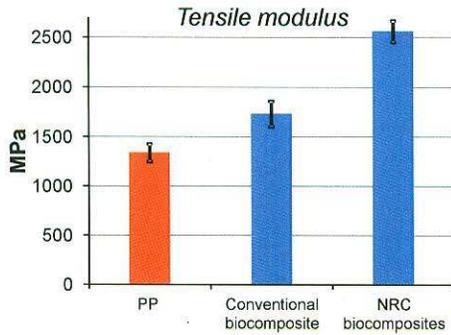
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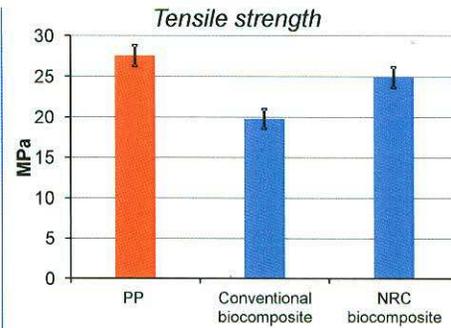
## Biocomposite tensile properties

(2/3)

**Comparison between biocomposites made with bark after tannin and lignin extraction using conventional and new NRC's formulation**



Modulus of NRC's bark biocomposite is much higher than those of the reference PP or the conventional biocomposite



Strength of NRC's bark biocomposite is higher than that of the conventional biocomposite and is approaching the reference

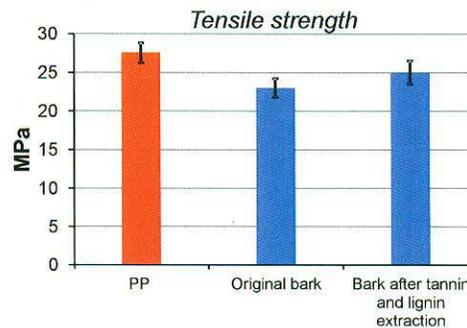
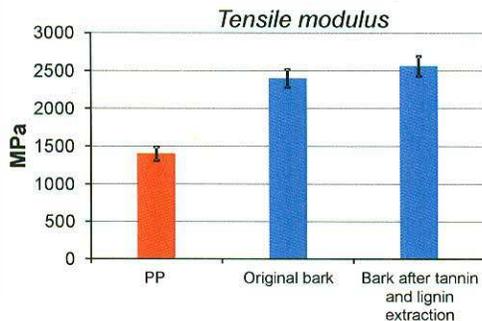
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## Biocomposite tensile properties

(3/3)

**Comparison between biocomposites made with original bark and bark after tannin and lignin extraction**



- No significant difference in mechanical properties between the biocomposites made with the original bark and the ones after lignin and tannin extraction
- Biocomposites with barks after lignin and tannin extraction have much less odor than those with original barks

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## Conclusions

- Effective approach with multiple advantages that can be applicable for tannin and lignin extraction:
  - Maximize the yield while minimizing impurities
  - Minimize water, chemical and energy consumption
  - Minimize operation cost
  - Simplify the process and enable an easy scale-up
  
- The obtained tannin/lignin and bark residue after extraction (cellulose) can be used to formulate flexible PU biofoams and biocomposites, respectively, with good processing characteristics and properties:
  - Tannin or lignin can partially replace petroleum based-polyol in the preparation of PU biofoam
  - Cellulosic residue can replace petroleum-based PP

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## Future work

- Tannin and lignin extraction
  - Validate with other barks having a higher tannin content
  - Combine tannin extraction with lignin extraction
  - Scale-up
- PU biofoam using lignin or tannin
  - Continue to optimize formulation
- Biocomposites
  - Continue to optimize formulation
  - Recyclability and cost analysis on the bark bio-composites

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  - *Éric Patenaude*
  - *Éric Cloutier*
  - *Yves Simard*
  - *Michel Carmel*

**THANK YOU VERY MUCH FOR YOUR ATTENTION !**

## **Greener method for tannin and lignin extraction from the yellow birch bark**

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Bark is an abundant forestry residue in North America (~17million m<sup>3</sup>/year in Canada alone). Currently, the majority of bark is incinerated or landfilled<sup>1</sup>. Bark contains a large fraction of tannin and lignin (up to 50 wt% on a dry basis), which can be utilized as a renewable aromatic chemicals<sup>1</sup> to replace fossil chemicals. The principle objective of our project is to extract tannin and lignin from Canadian bark and convert them into bio-based polymer products in order to support the economic sustainability of the forestry and manufacturing sector.

In this specific paper we present a novel green method for tannin and lignin extraction from Canadian yellow birch bark. The aim is to maximize tannin and lignin yield while minimizing energy and water consumptions and the processing cost. At first, bark was grinded to reduce the size then the tannin was extracted from the grinded bark by hot water without chemicals in the presence and absence of ultrasound energy. Finally, the lignin was extracted from the residue after tannin extraction using alkaline solution.

The yellow birch bark has relatively low tannin content (1.5% to 4.4%) and very lignin high content (~40%). Thus, the conventional tannin extraction process is not very cost-effective for this type of bark. We develop a novel method that consists of re-utilizing the extract solution for a second extraction. The re-utilization of the extract solution permits significantly reduction of energy and water consumption. Water and energy consumption for every step in the process was estimated and minimized in the lab conditions.