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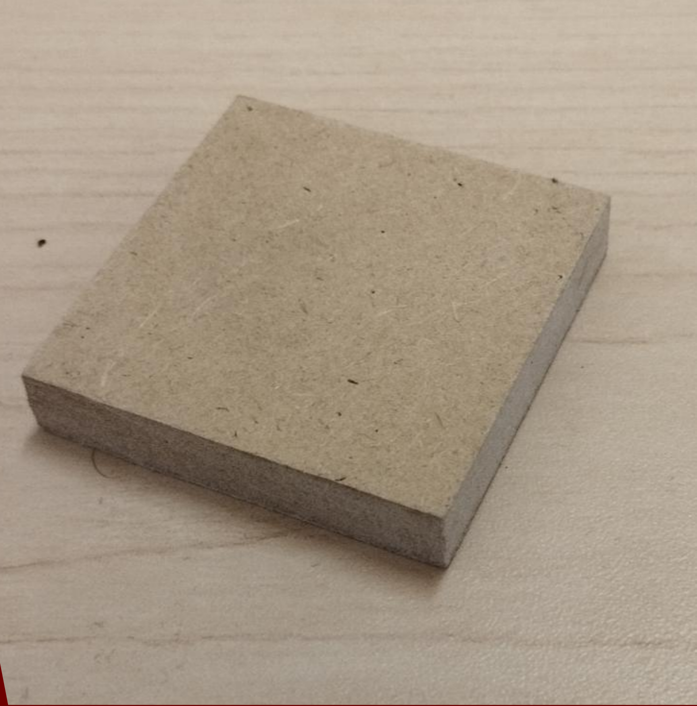
Effect of hydrothermal recycling on the physicochemical properties of fibers recovered from medium-density fiberboard (MDF) waste

Lecoublet Morgan[1*], Rosilei Garcia[2], Alain Cloutier[2] and Ahmed Koubaa[1]

[1] Forest Research Institute, Université du Québec en Abitibi-Témiscamingue (UQAT), Rouyn-Noranda, QC, Canada

[2] Renewable Materials Research Centre (CRMR), Faculty of Forestry, Geography, and Geomatics, Université Laval, Québec, QC, Canada

*Correspondence to: lecm57@uqat.ca



Contextualization

- **In 2023, Canada produced 3.2 million m^3 of wood panels, including 900,000 m^3 of medium-density fiberboard (MDF) [1].**

- Wood panel waste [2]

- **End-of-life management of wood and wood-based materials (North America, United States Environmental Protection Agency [3]) :**
  - Landfill (82 %)
  - Incineration (18 %)
  - Recycling (< 1 %)

Major environmental issues

Contextualization

- Wood panels can be recycled, allowing their components to be reintegrated into a new material manufacturing cycle

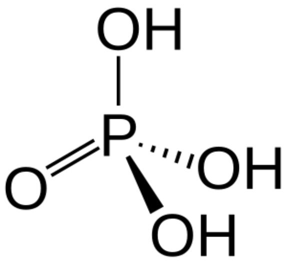
- **Mechanical recycling**
  - ✓ Fast and cheap
  - X Cannot remove UF resin

- **Hydrothermal recycling**
  - ✓ Can remove UF resin
  - X Energy consuming

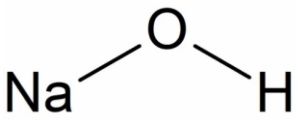
- **Chemical hydrolysis recycling**
  - ✓ Can also remove UF resin
  - X Uses potentially expensive and toxic chemical products

Mechanical recycling

Pressure reactor



Phosphoric acid



Sodium hydroxyde

Aim of the study

Recycle MDF wood panels by hydrothermal recycling

- Technical feasibility
- The recovered fibers will be characterized by several methods

Optimize the recycling parameters for the manufacture of wood-polymer composites

- Influence of time, temperature and concentration of weak mineral acids on recovered fibers properties
- What is the best compromise for recovering the best-performing wood fibers for the manufacture of wood-polymer composites?

Manufacture and optimization of wood-polymer composites produced with recovered fibers

- Technical feasibility
- Multiphysical characterization of composites and process optimization

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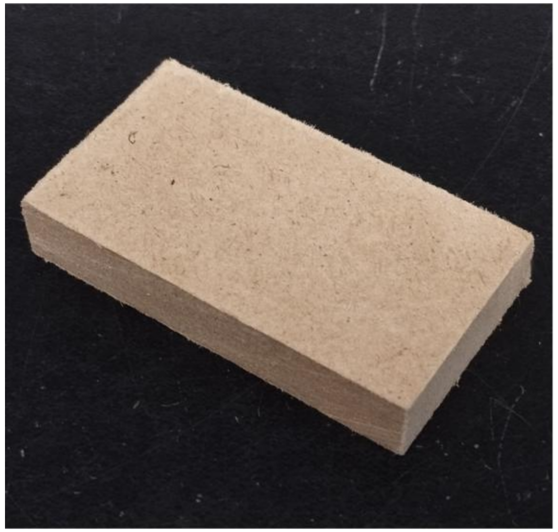
Materials and methods

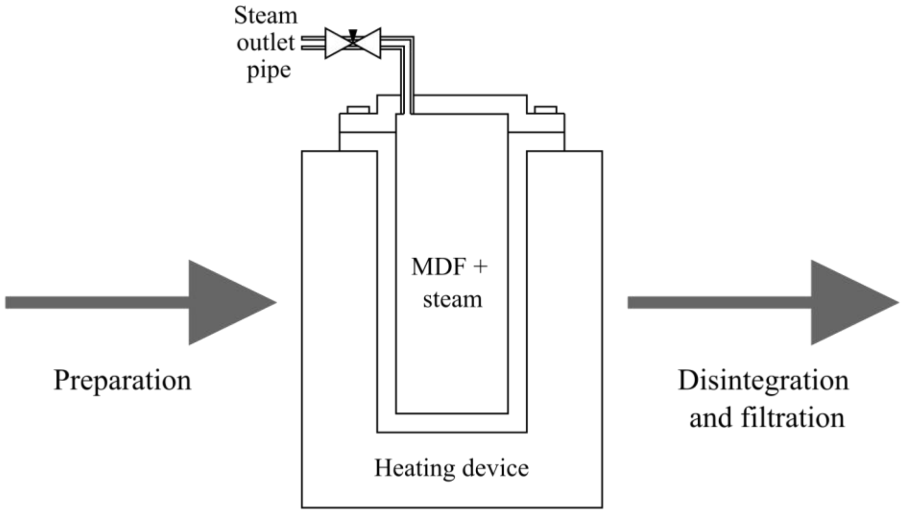
- Use waste MDF as raw material

Thickness	9,8 mm
Density	790 kg·m^{-3}
UF content	Unknow, supposed 10 ~ 12 %
Weight	~ 10 g
Liquid/solid ratio	20/1
Recycling temperature	100 – 200 °C
Recycling time	20 min

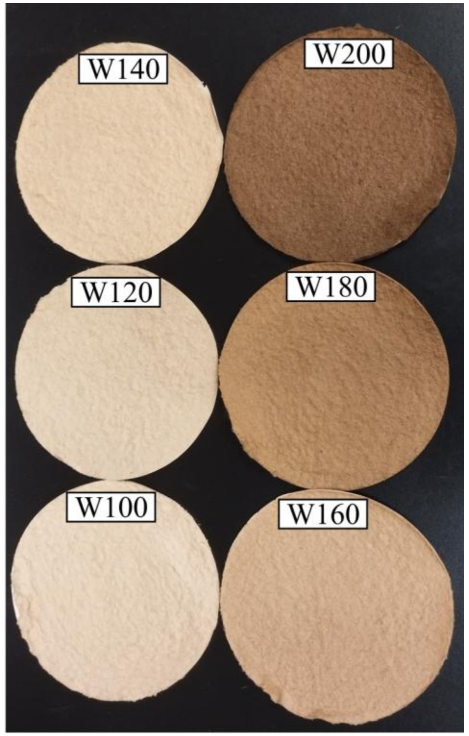
- MDF and recycling parameters



MDF panel



Hydrothermal recovering of TMP fibers from MDF panels



Recovered TMP fibers

- Recycling processing used

Materials and methods

Condition	T (°C)	Pressure (Bars)	Severity degree	Fiber yield (%)
W100	100	> 2	1.30	81.2
W120	120	> 2	1.89	79.6
W140	140	2	2.48	76.6
W160	160	5	3.07	76.3
W180	180	9	3.66	72.7
W200	200	14	4.25	67.9

➨ Produced conditions

➨ **Severity degree: parameter representing the intensity of recycling applied**

$$\text{Severity degree} = Log\ R_0 = t \times e^{\left(\frac{T-100}{14.75}\right)}$$

Materials and methods

- **Morphological characterization**
  - Fiber size distribution
  - Spectrocolorimeter

- Konica Minolta CR-410

- **Thermal characterization**
  - Thermogravimeric analyzer

- TA Q50 thermogravimetric analyzer

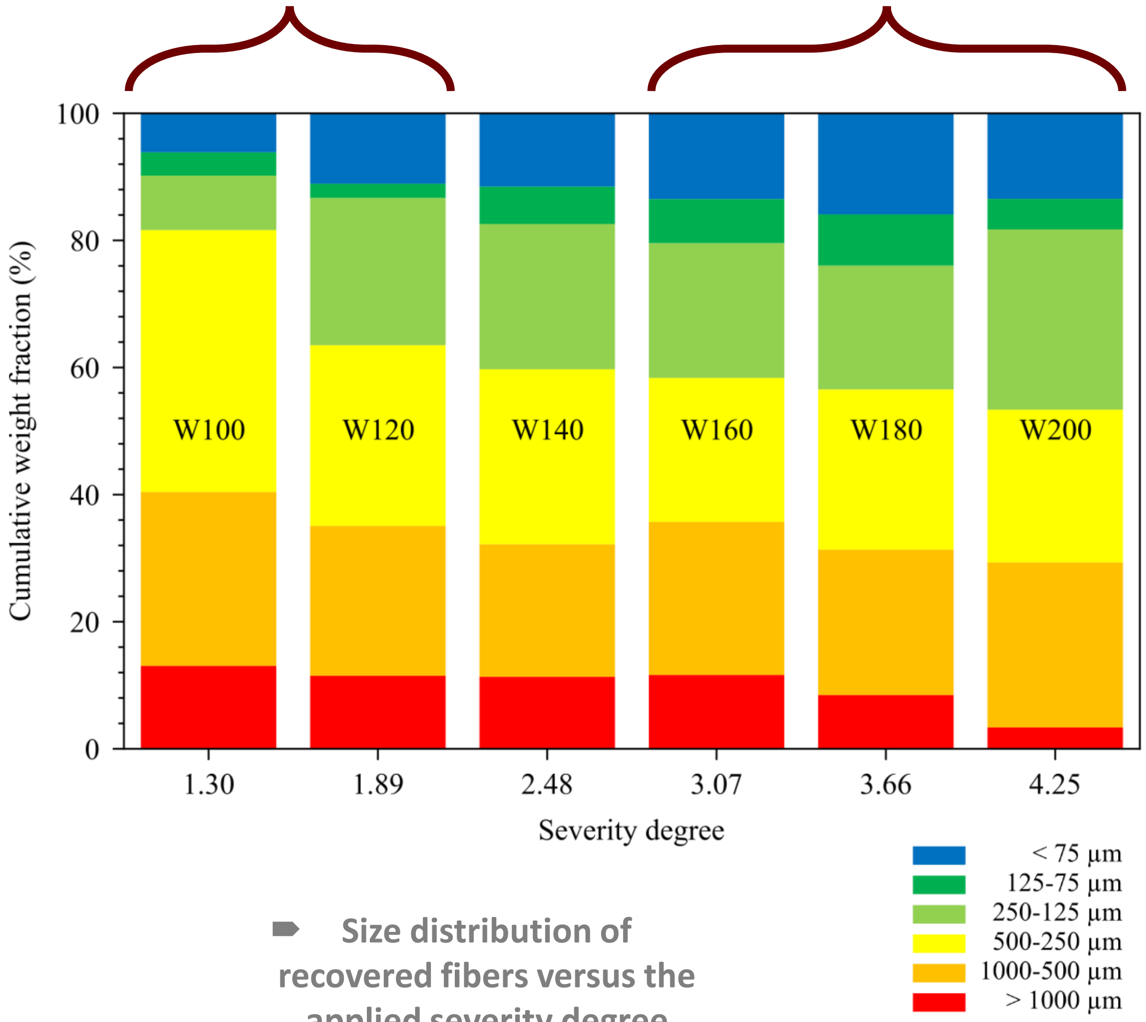
- **Chemical characterization**
  - Nitrogen analyzer
  - FTIR analyzer

- Shimadzu IR-Tracer-100

Morphological analysis – size distribution

- **Global reduction in fiber size with the applied severity degree**

- **Two notable transitions**
  - W100 - W120 : The severity degree applied achieves complete separation of the TMP fibers previously bonded by UF resin.
  - W160 - W200 : Potential degradation of lignocellulosic matter, allowing a better individualization of wood fibers.



Size distribution of recovered fibers versus the applied severity degree

Morphological analysis – spectrocolorimetry

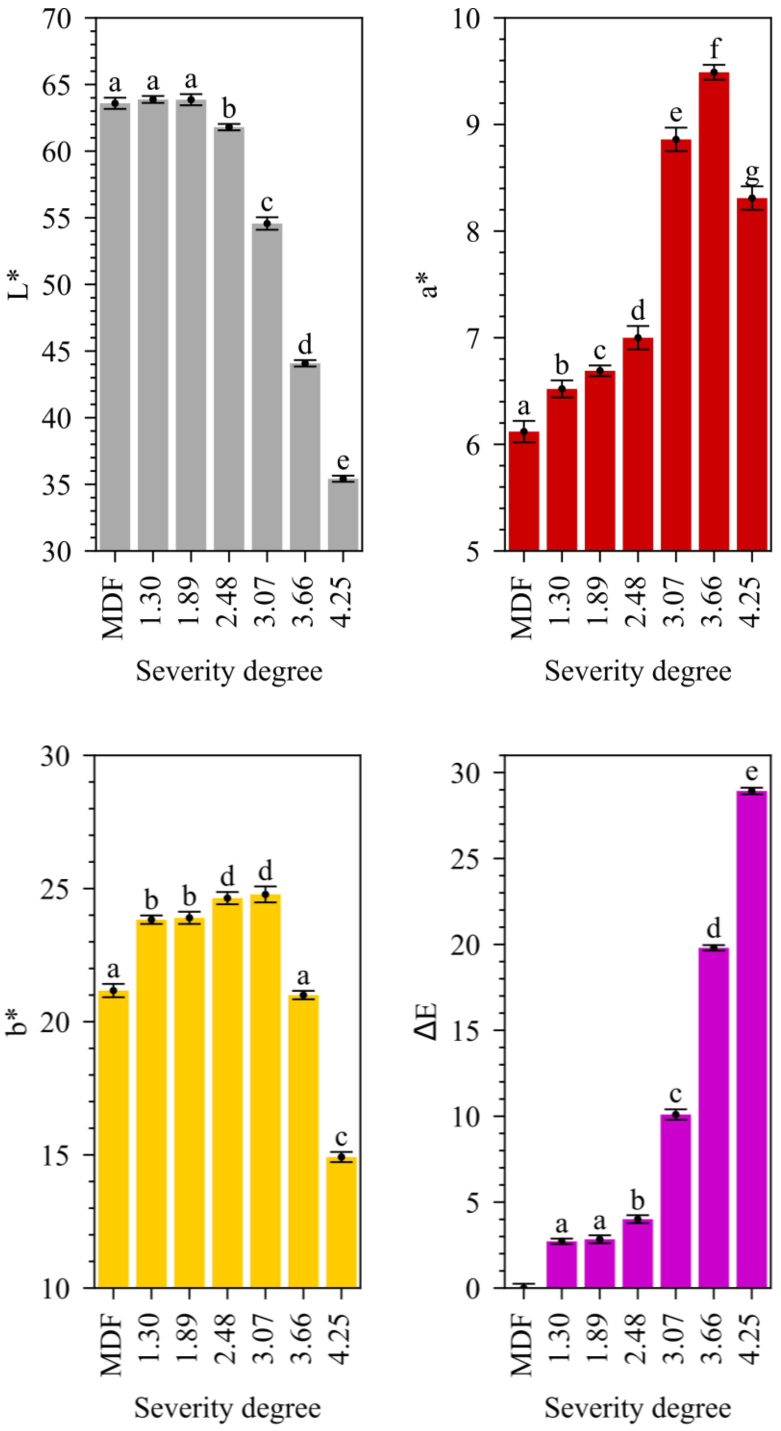
- **Color difference ΔE**

$$\Delta E = \sqrt{(L_2^* - L_1^*)^2 + (a_2^* - a_1^*)^2 + (b_2^* - b_1^*)^2}$$

  - Quantifies the difference between two colors (raw MDF as reference)
  - If ΔE < 5, can be considered as barely visible

- **Under a severity degree of 2.48: slight color alteration, but above 2.48, significant variation**

- **Different applications possible :**
  - Low severity degree : wood/polymer composite and MDF
  - High severity degree : cardboard

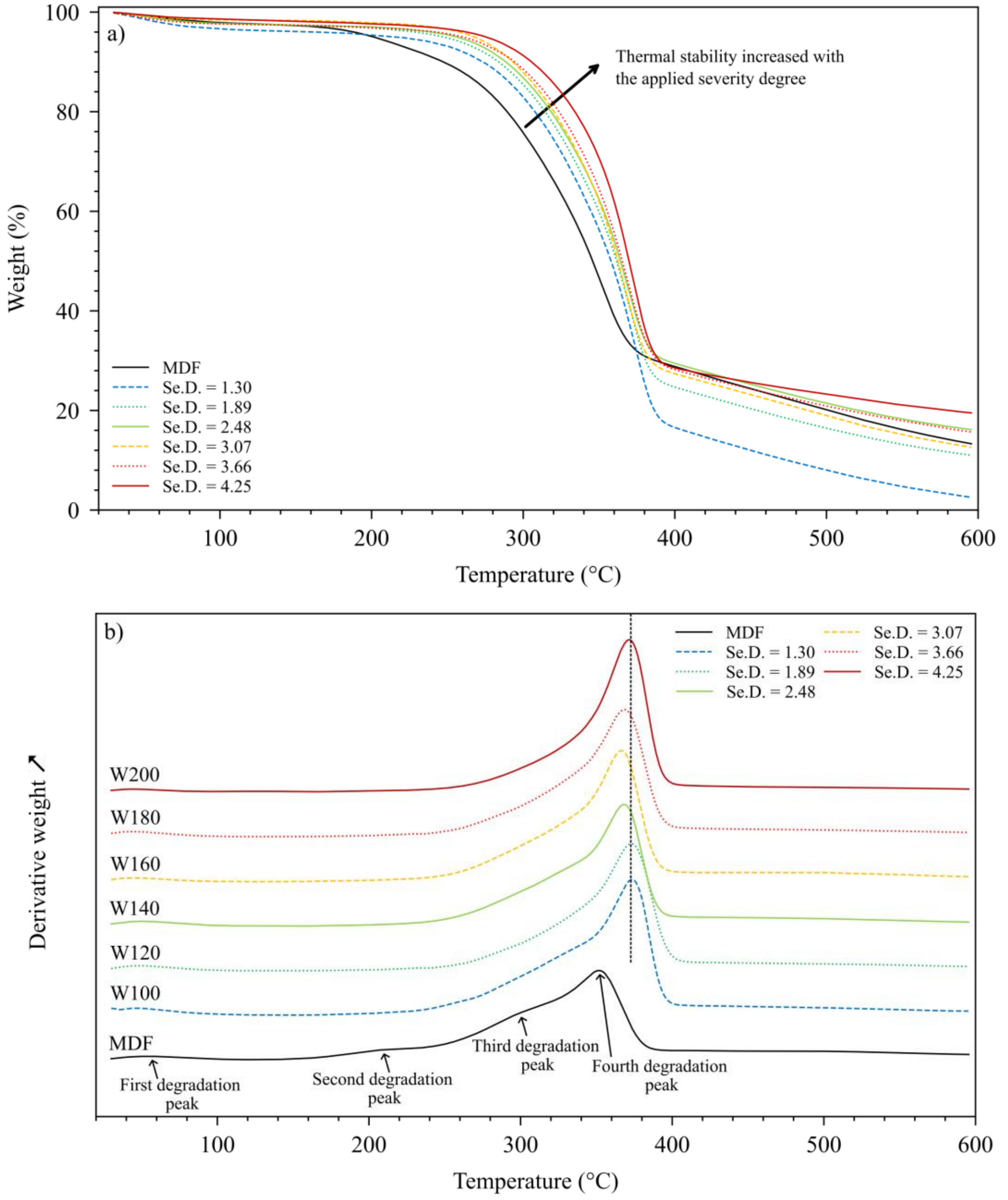


L*, a*, b* and ΔE components of recovered fibers versus the applied severity degree

Thermal analysis – thermogravimetric analysis

- **MDF shows 4 degradation peaks**
  - Water, UF resin hemicellulose and cellulose
  - Lignin slightly visible

- **Increasing the applied severity degree increases the thermal stability of recovered fibers**
  - Loss of UF resin and hemicellulose, components with lower thermal stability than cellulose
  - Results to be confirmed with surface chemical analysis



- **Thermal stability and derivative thermal stability of recovered fibers versus the applied severity degree**

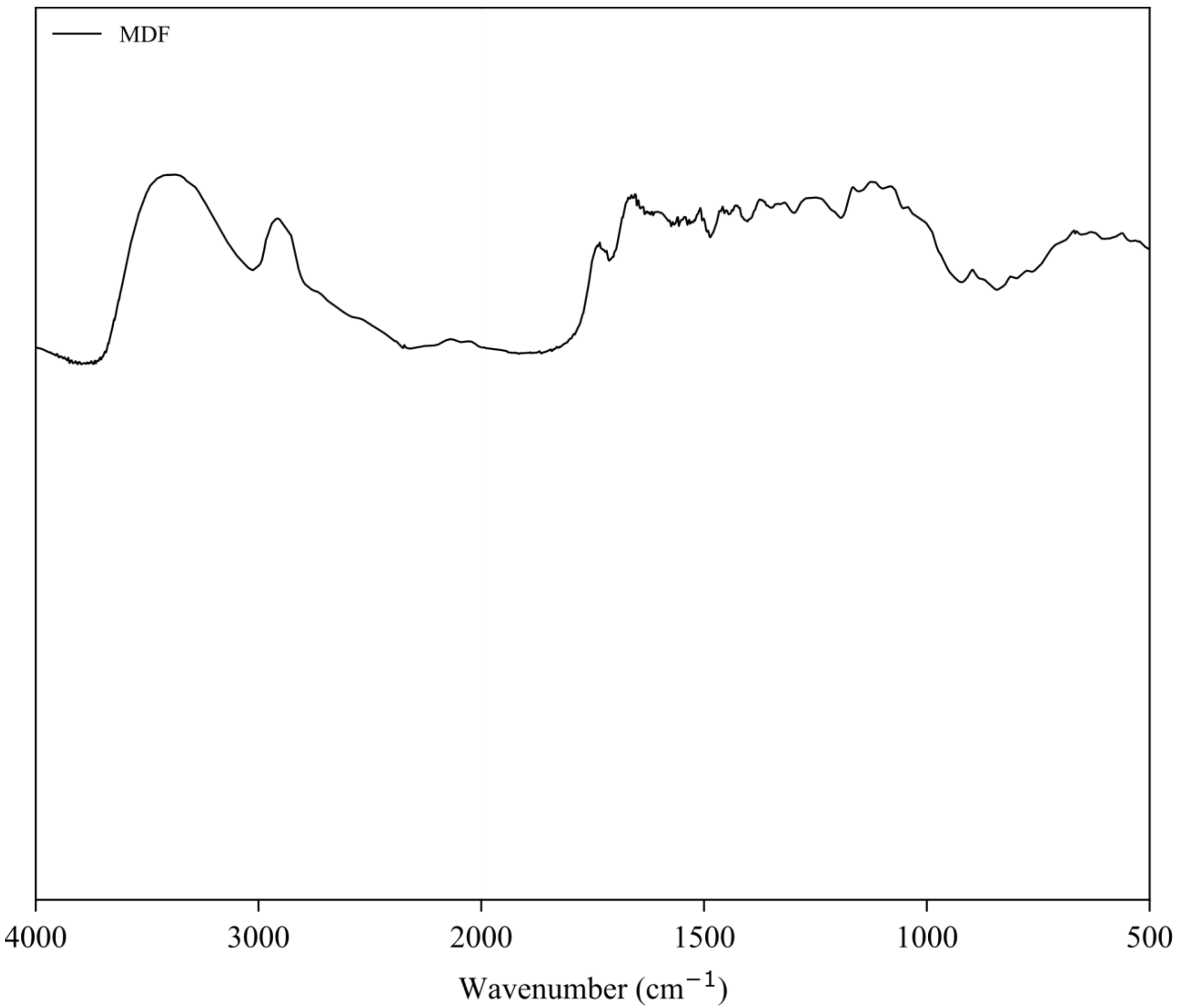
Chemical analysis – nitrogen analysis

- **MDF contains around 12 % of UF**
- **All recycling efficiently removes UF resin**
  - Removed UF resin > 80 %
- **Interesting behavior regarding the effect of severity degree**
  - Decrease of the N content until a severity degree of 2.48, then increase until a severity degree of 4,25
  - Loss of lignocellulosic components ?
    - In accordance with previous tests

Condition	Severity degree	Nitrogen content (%)	UF resin content (%)	Removed UF (%)
MDF	n.d.	3.75 ± 0.05 (a)	12.53 ± 0.15 (a)	n.d.
W100	1.30	0.92 ± 0.05 (b)	3.07 ± 0.17 (b)	– 79.1
W120	1.89	0.60 ± 0.01 (c)	1.99 ± 0.04 (c)	– 87.9
W140	2.48	0.33 ± 0.01 (d)	1.11 ± 0.02 (d)	– 95.0
W160	3.07	0.45 ± 0.06 (cd)	1.50 ± 0.20 (a)	– 91.9
W180	3.66	0.58 ± 0.03 (c)	1.94 ± 0.10 (a)	– 88.3
W200	4.25	0.70 ± 0.02 (d)	2.32 ± 0.07 (a)	– 85.2

- **Nitrogen analyzer results**

Chemical analysis – surface chemistry by FTIR

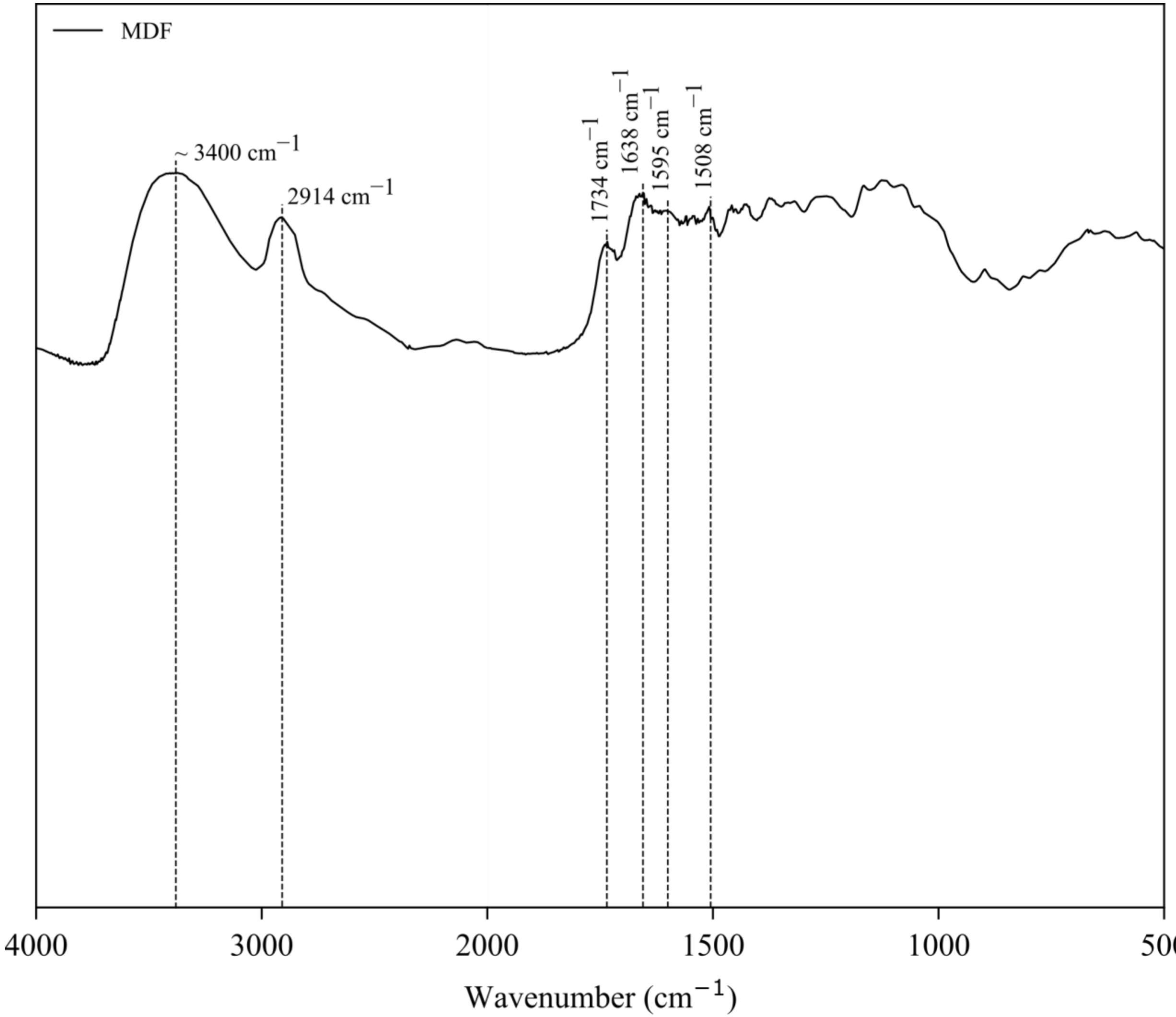


- DRIFT-mode FTIR spectrum of MDF

Chemical analysis – surface chemistry by FTIR

Wavenumber (cm^{-1})	Spectral peak analysis	Reference
3400	–OH stretching of cellulose	[6]
2914	C–H stretching of cellulose	[4,6]
1734	C=O stretching of hemicellulose	[5]
1638	C=O, C–N of primary amide UF, adsorbed water	[3,4,6,7]
1595	C=O of secondary amide UF	[3]
1508	C=C stretching vibration aromatic ring skeleton in lignin, C–N peak of secondary amide UF	[3,5,7]

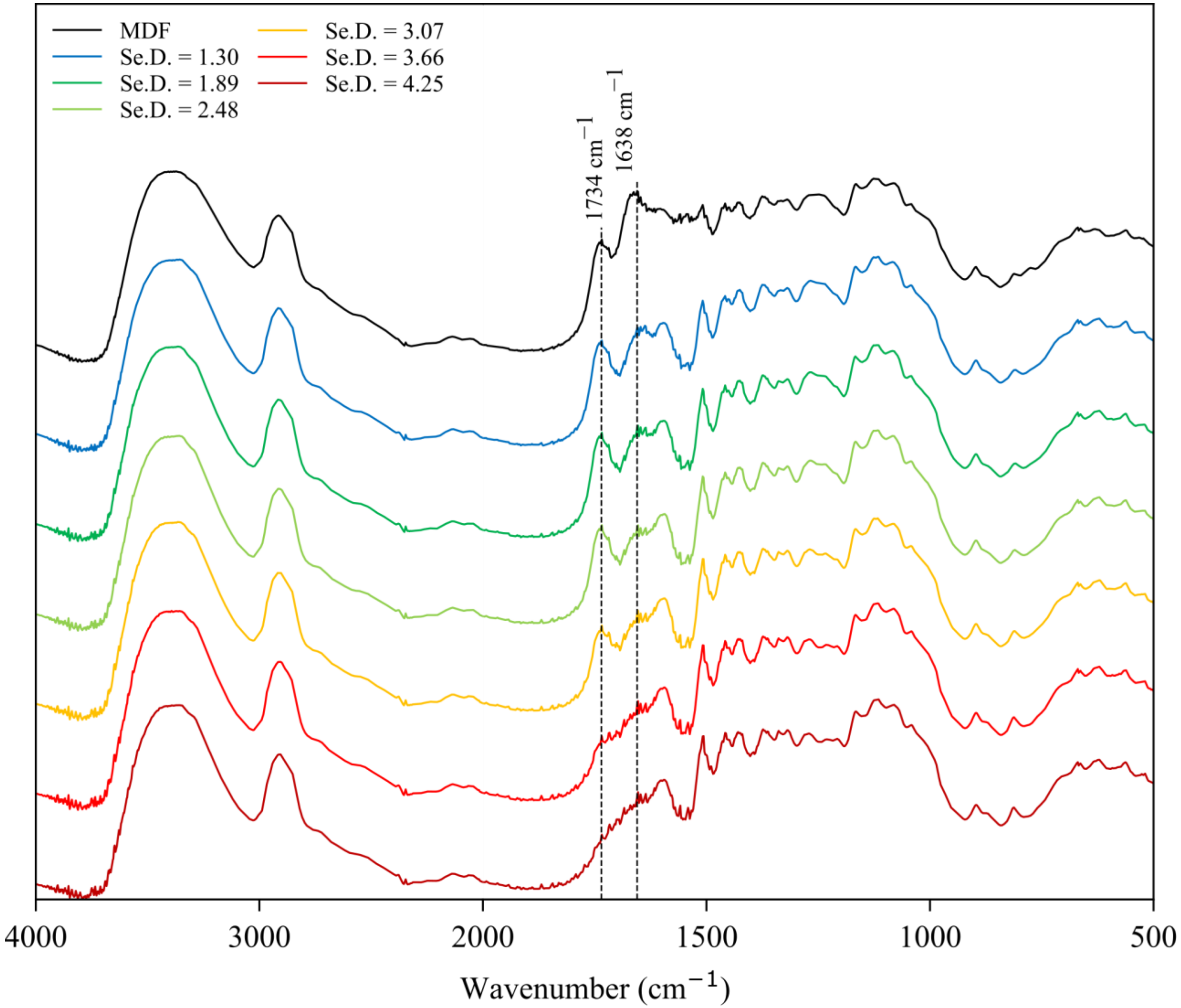
Key MDF peaks



DRIFT-mode FTIR spectrum of MDF

Chemical analysis – surface chemistry by FTIR

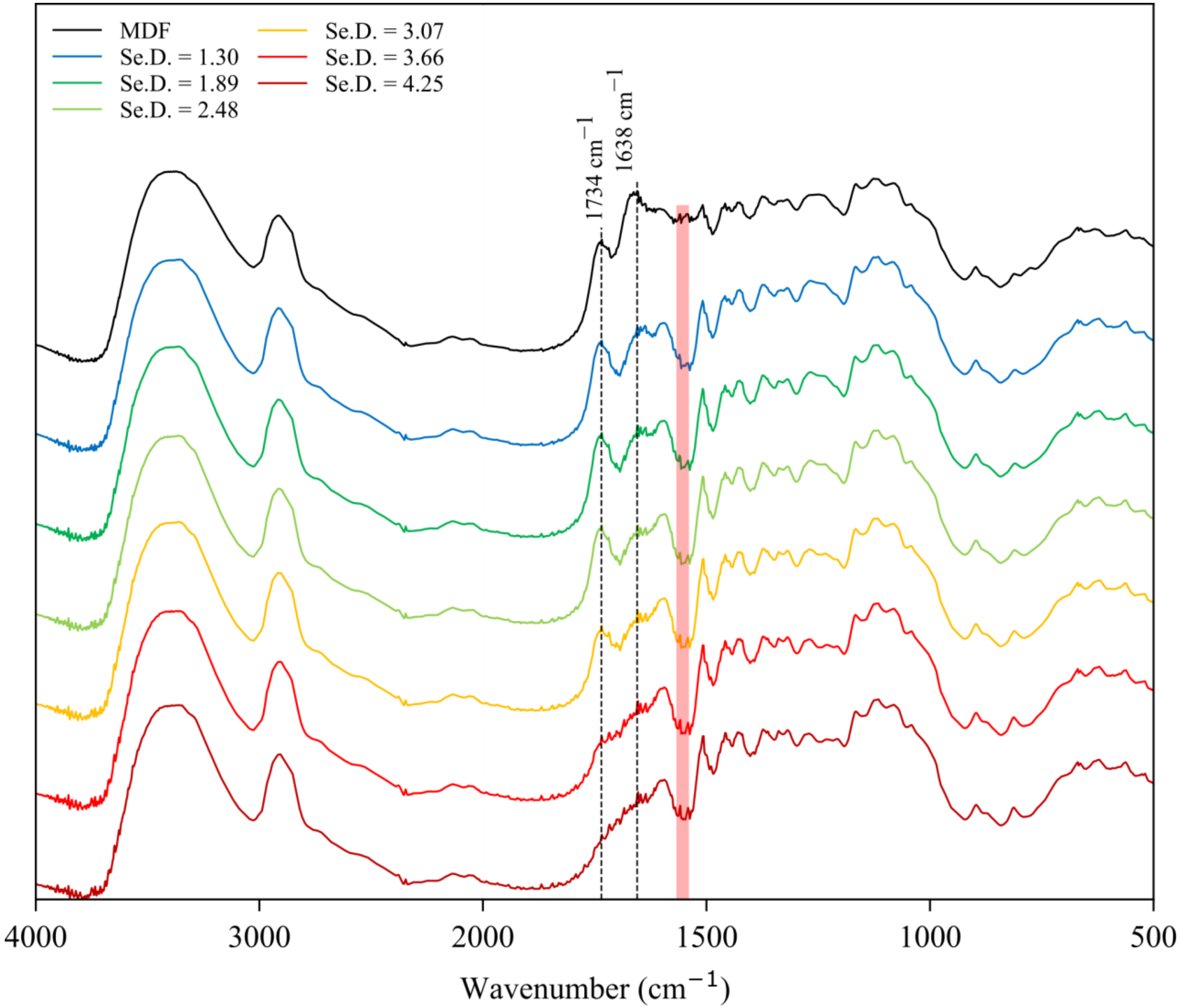
- **The hydrothermal recycling does not appear to chemically affect cellulose and lignin**
  - No significant changes
- **Two peaks were affected**
  - 1734 cm^{-1} – Hemicellulose
  - ~ 1640 cm^{-1} – Lignin + water



- **DRIFT-mode FTIR spectrum of MDF and recovered fibers**

Chemical analysis – surface chemistry by FTIR

- **The hydrothermal recycling does not appear to chemically affect cellulose and lignin**
  - No significant changes
- **Two peaks were affected**
  - 1734 cm^{-1} – Hemicellulose
  - ~ 1640 cm^{-1} – Lignin + water
- **Presence of a zone of interest in the spectrum**
  - ~ 1550 cm^{-1}, appearing to be resin-related
  - Need to confirm this observation by comparing these results with previously determined nitrogen analysis



- **DRIFT-mode FTIR spectrum of MDF and recovered fibers**

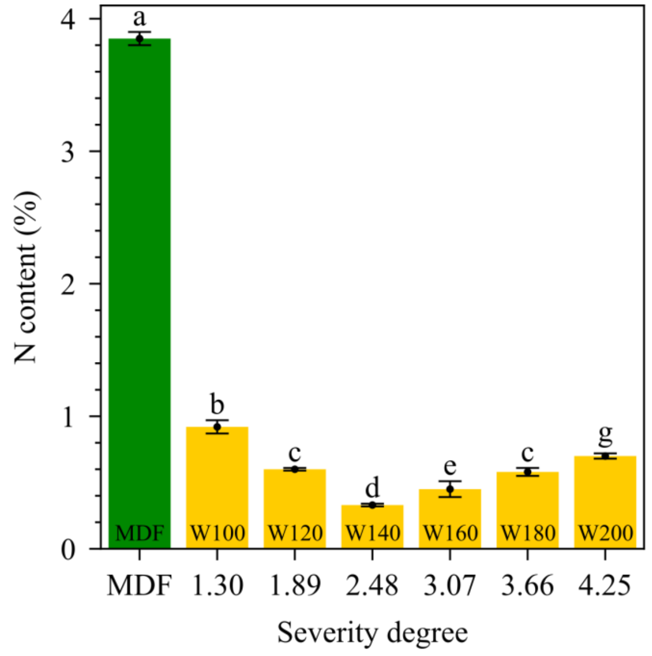
Chemical analysis – comparison

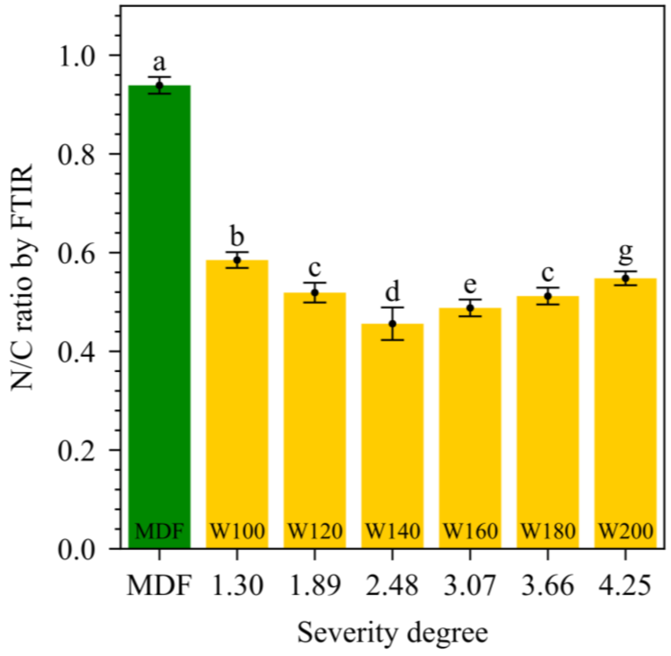
- **Determination of UF resin / cellulose ratio :**

$$Ratio\ N/C = \frac{I_{depression\ @\ 1550\ cm^{-1}}}{I_{cellulose\ @\ 2930\ cm^{-1}}}$$

- **The N/C ratio gives similar values compared to the previously determined nitrogen content.**
  - Same statistical results

- **Infrared analysis can be a fast, non-destructive method for predicting the nitrogen content of our recycled fibers.**
  - More work need to be conducted





Conclusions

- ✓ **Physical analyses showed that the degree of severity had a negative impact on fiber size, but also a positive impact on the thermal stability of the recovered fibers**

- ✓ **Spectrocolorimetric analyses showed that the fibers only began to change color perceptibly at temperatures of 160°C and above**

- ✓ **Chemical analyses confirmed the effectiveness of the treatment applied to remove the UF resin**
  - ✓ UF resin removal efficiency > 80 %

Perspectives and project continuation

- **Short-term: Use of industrial weak acid to improve fiber individualization**
- **Medium-term: Optimization of the recycling process assisted by industrial weak acid to produce wood/polymer composites**
- **Long-term: Manufacture and optimization of wood/polymer composites with the fibers produced**

- Wood/polymer composite prototypes produced with PP and recycled wood fibers treated with an industrial weak acid

THANK YOU

Corepan Bois
Consortium de recherche sur les panneaux composites à base de bois

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