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NRC-CNRC

**Industrial Materials
Institute**

Formulation and Processing of PP- Flax Fiber Composites

M-T. Ton-That, J. Denault, E. Patenaude and W. Leelapornpisit

***Bioplastics 2006
27th-29th September 2006
Montreal, Quebec, Canada***



National Research
Council Canada

Conseil national
de recherches Canada

Canada

Outline

- ❑ Flax structure and properties
- ❑ PP composites from flax
 - Compression
 - Injection
 - Extrusion-Injection
- ❑ Conclusions



Flax types

❑ Flax

- Fibers, Shives, Mix of shives & fibers, Dust (Biolin)
- Fiber treatment: non, water, soap and NaOH 1% (50°C, 2 h)



❑ Polymer matrix

- Injection grade PP Profax 1274 (Basell)

❑ Coupling agent: maleic anhydride (MA) grafted polypropylenes

- Epolene-43 (Mn = 9,100; ~4.81 wt% of MA) (Eastman Chemical)
- Epolene-3015 (Mn = 56,000; ~1.5 wt% of MA) (Eastman Chemical)
- Polybond 3150 (Mn = 330,000; ~0.5 wt% of MA) (Chemtura)

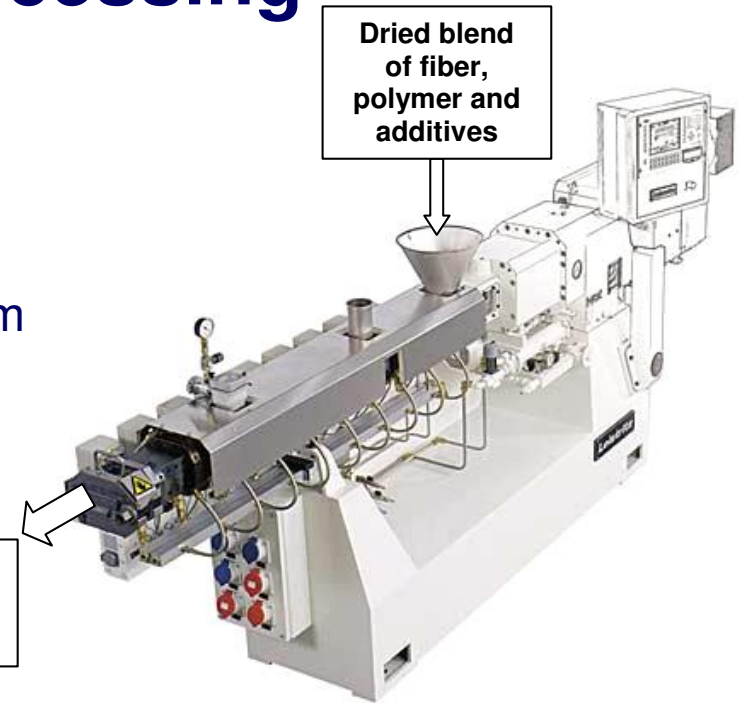
❑ Additives: CaO

Processing

☐ Extrusion

○ Standard TSE, $T=190^{\circ}\text{C}$, 10kg/h, 200rpm

☐ Injection molding; $T=190^{\circ}\text{C}$

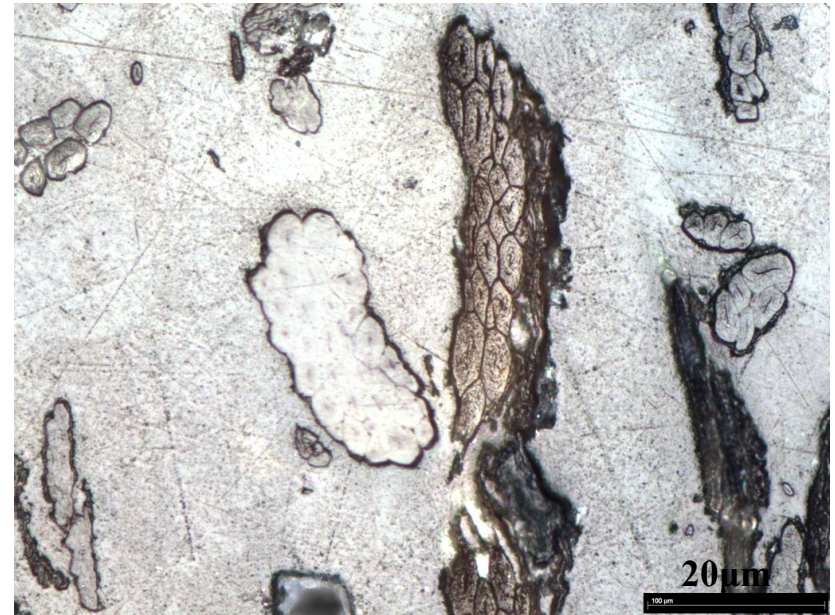


☐ Mat fabrication

☐ Compression molding



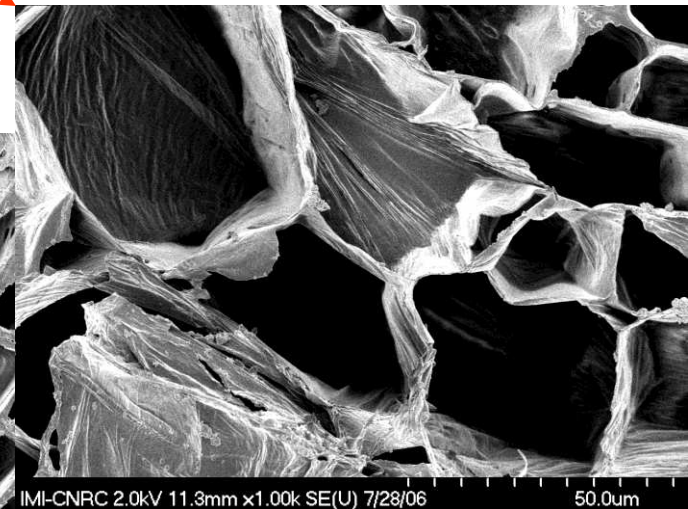
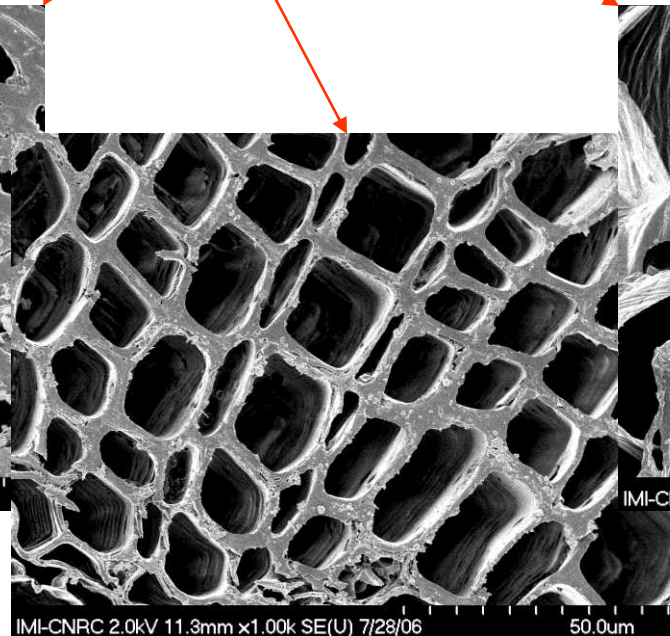
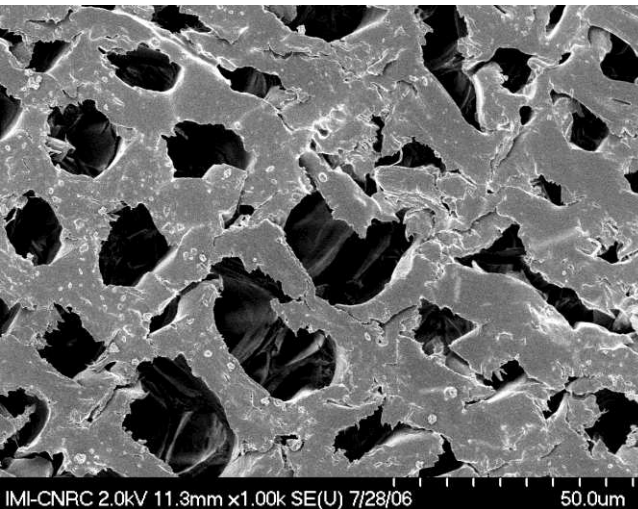
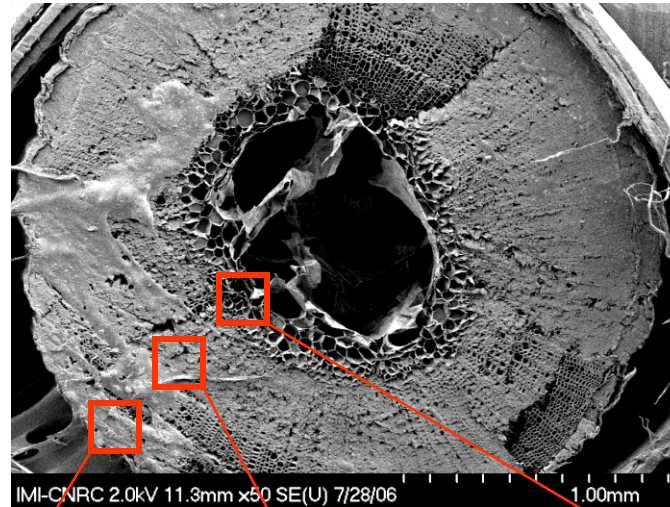
Flax fiber structure



- ❑ Single fiber cells and multiple fiber-cell bundles at different sizes can be observed: very inhomogeneous
- ❑ Fiber cell cross-section is not a regular circle

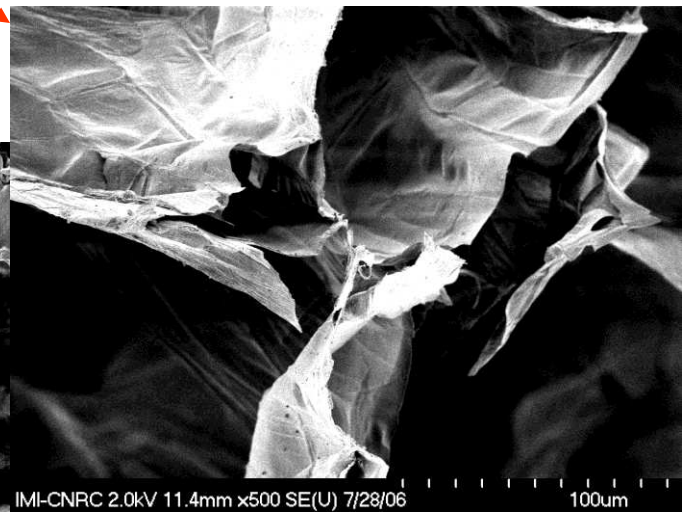
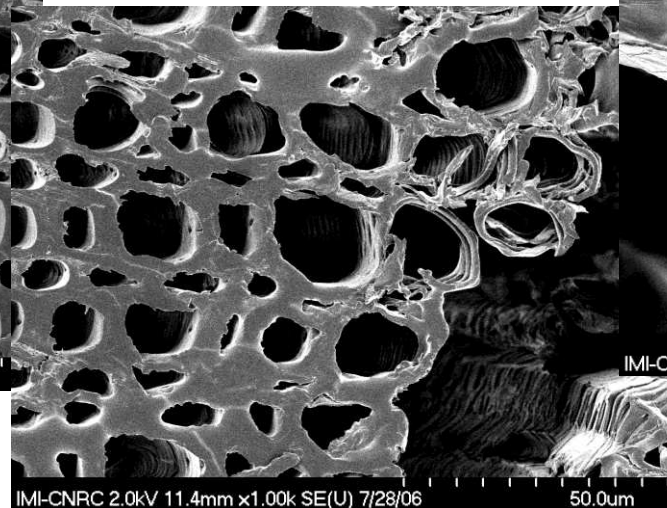
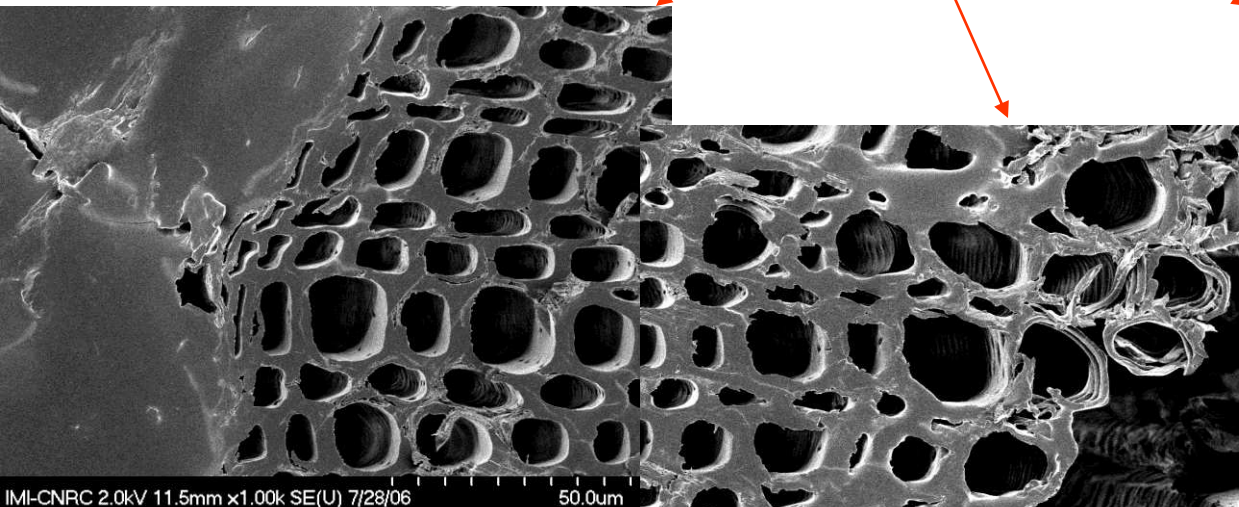
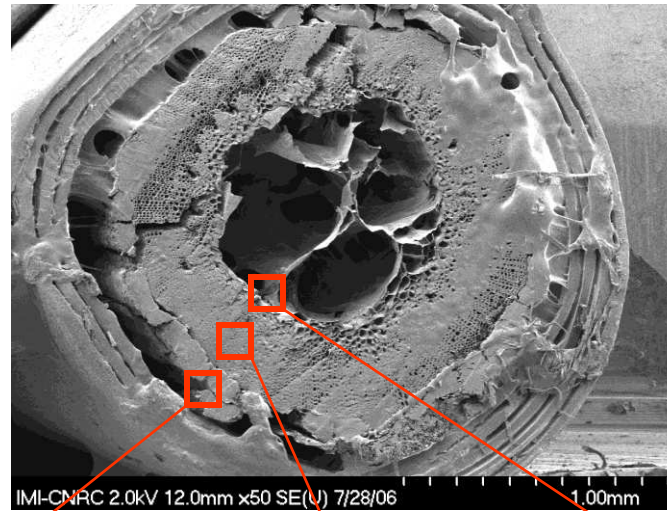
Flax fiber structure

Bottom of the stem

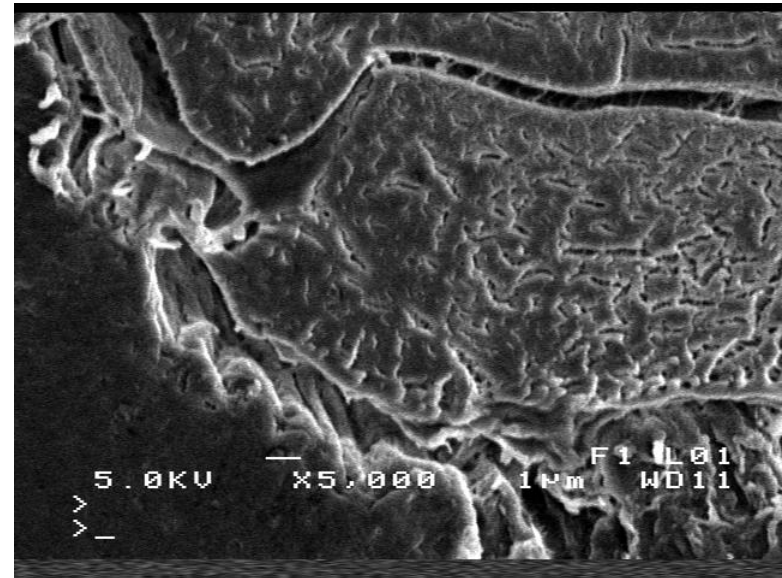
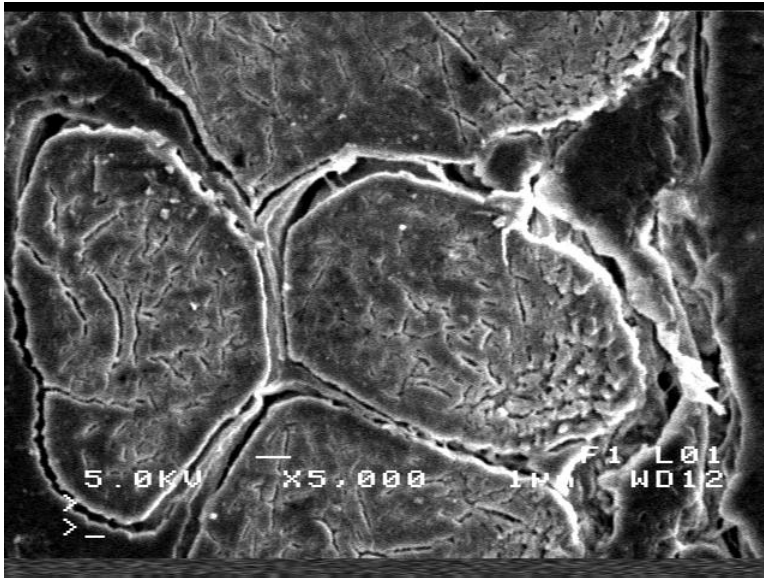


Flax fiber structure

Top of the stem



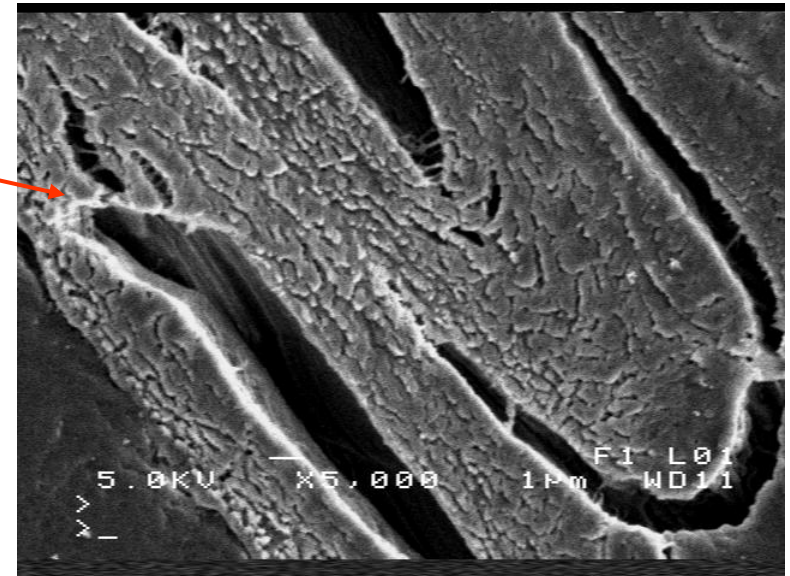
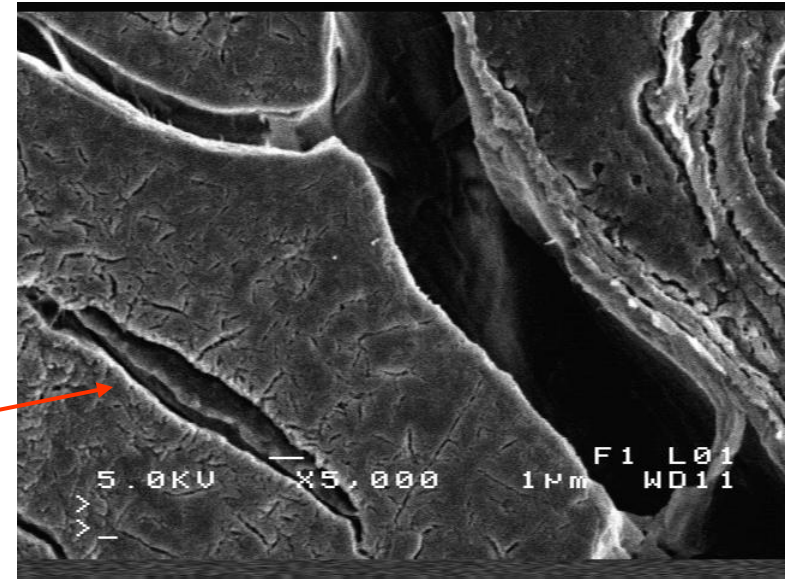
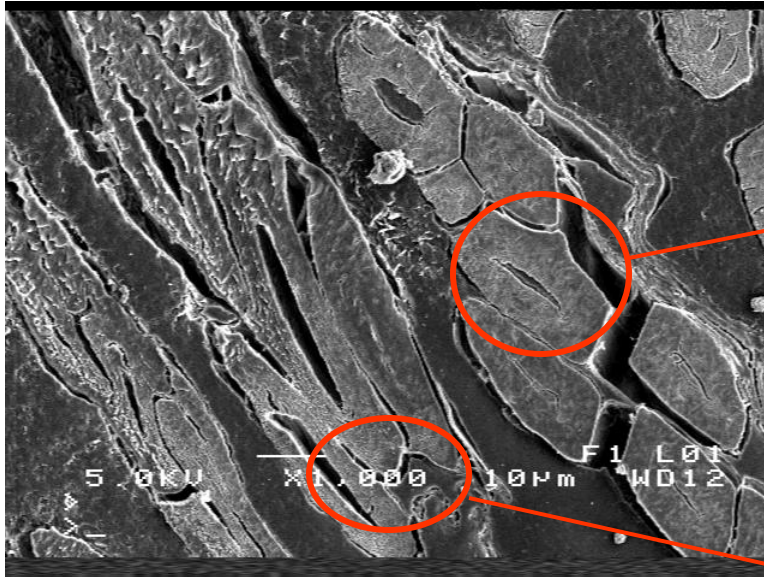
Flax fiber structure



- ❑ Weak bonding between the fiber cells in the bundle
- ❑ Many defects inside the fiber cell cross-section

Flax fiber structure

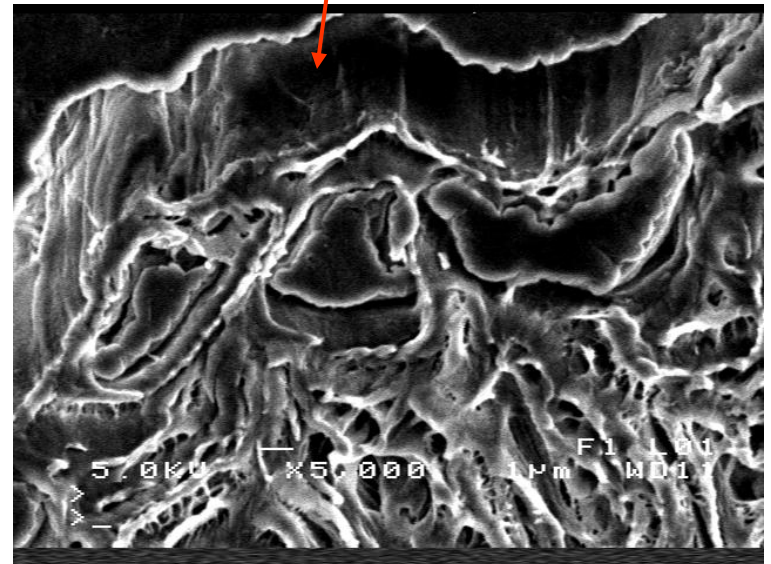
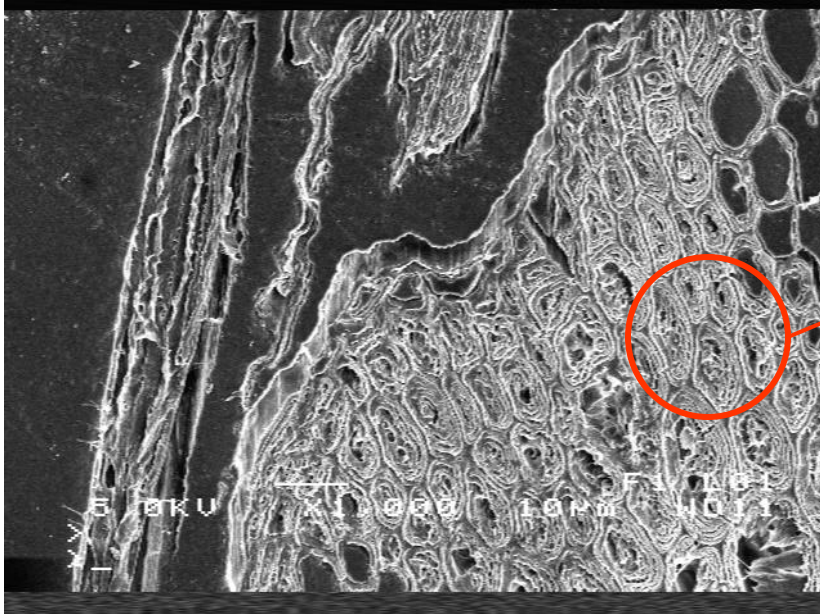
Outer layer of the plant



- Hollow and porous structure can also be observed

Flax fiber structure

Center of the plant

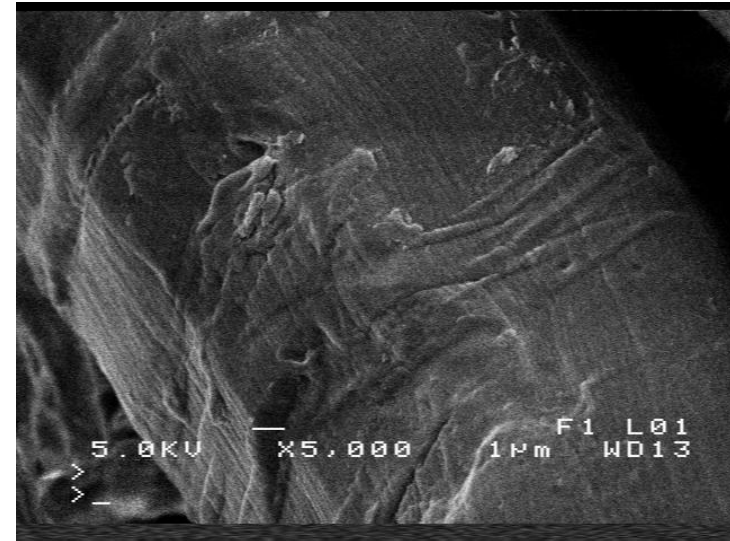


□ Hollow and porous structure

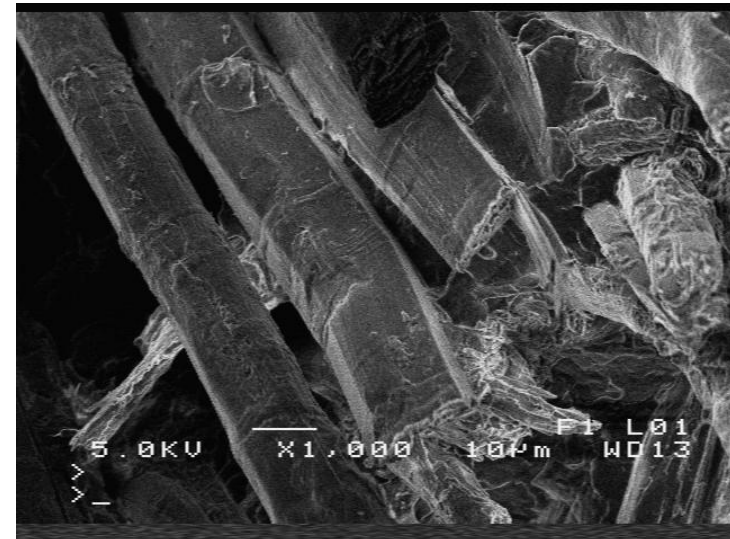
Flax fiber structure



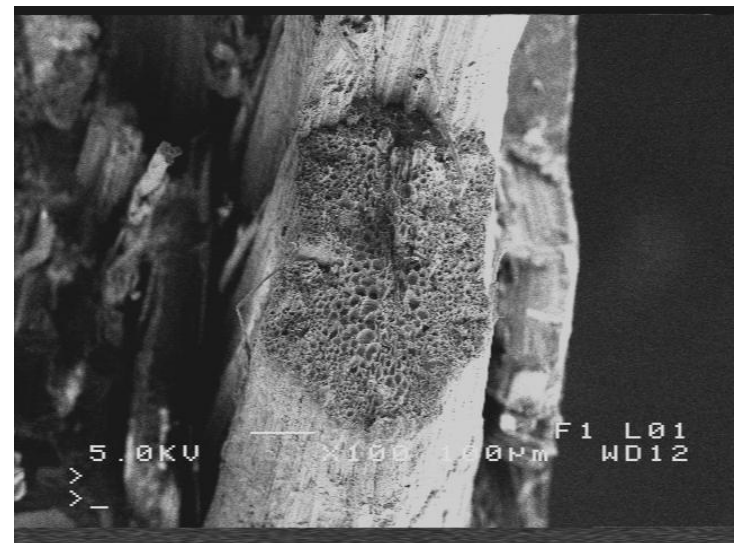
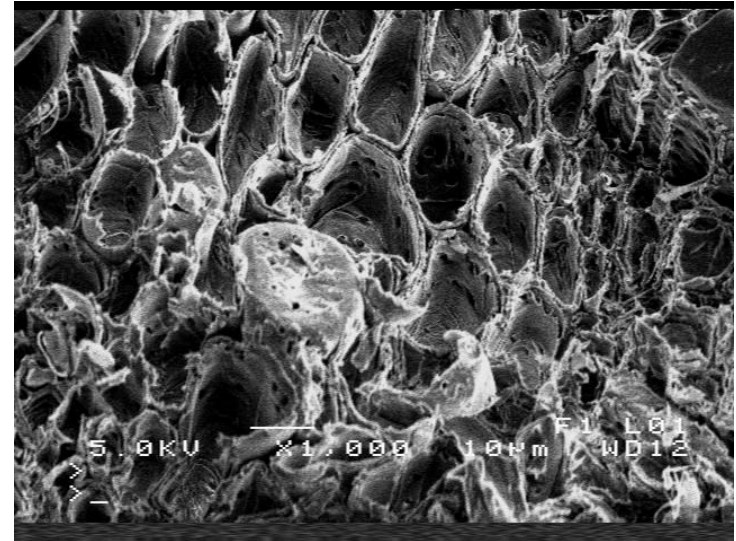
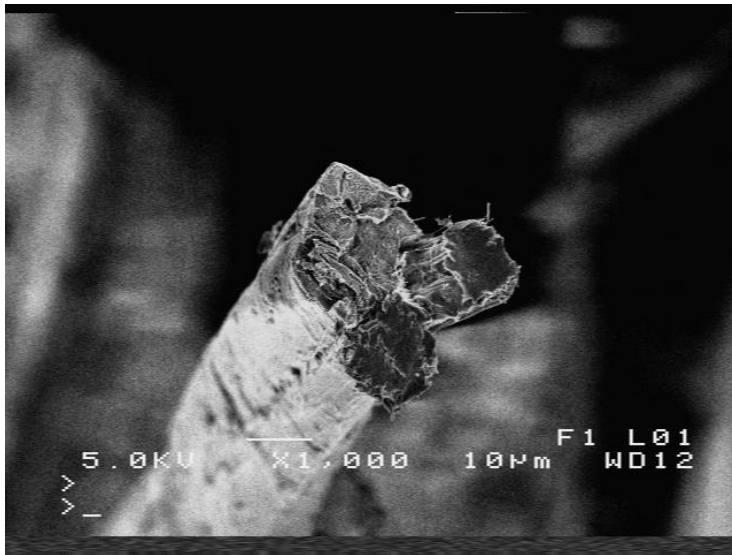
- ❑ Splitting of elementary fiber and the meso structure in the secondary cell wall
- ❑ Fiber with intercellular bonding



- ❑ Deformation and structural defects



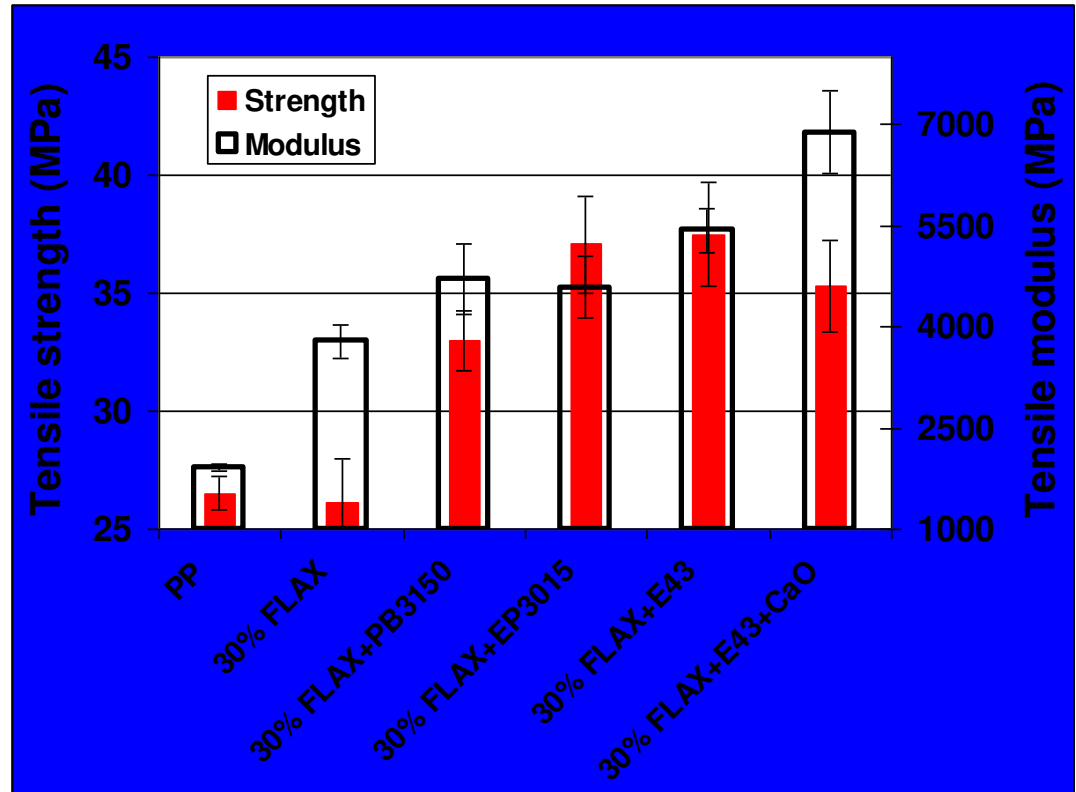
Flax fiber properties



- ❑ Tensile strength: 280 ± 120 MPa
expected: 780 MPa
- ❑ Tensile modulus: 15.8 ± 1.5 GPa
expected: 27.5 GPa
- ❑ Diameter: 10-120 μm
- ❑ Rupture mode is very brittle

Mechanical properties

Compression molding

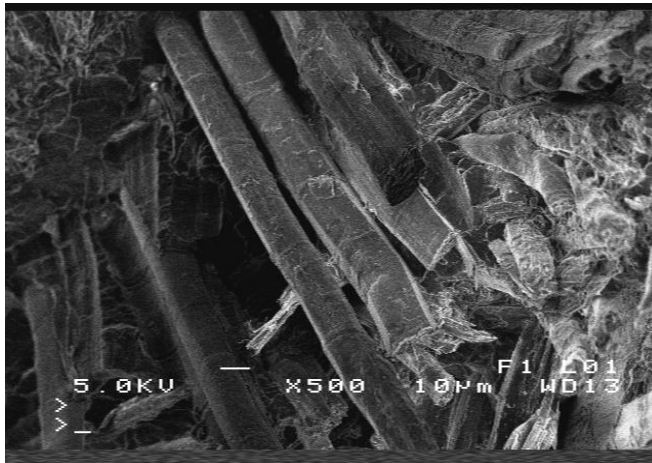
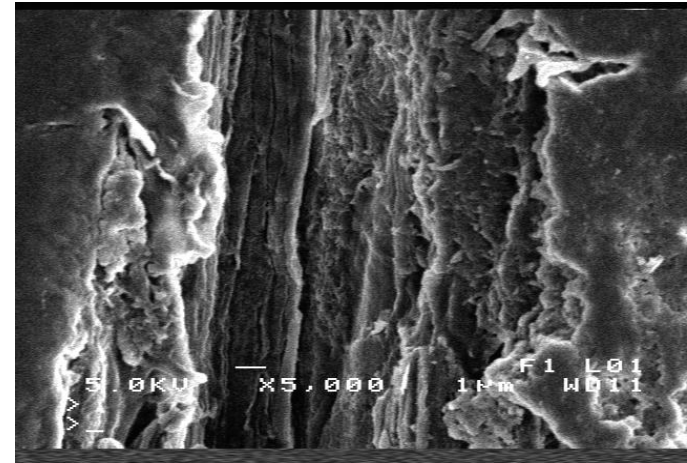
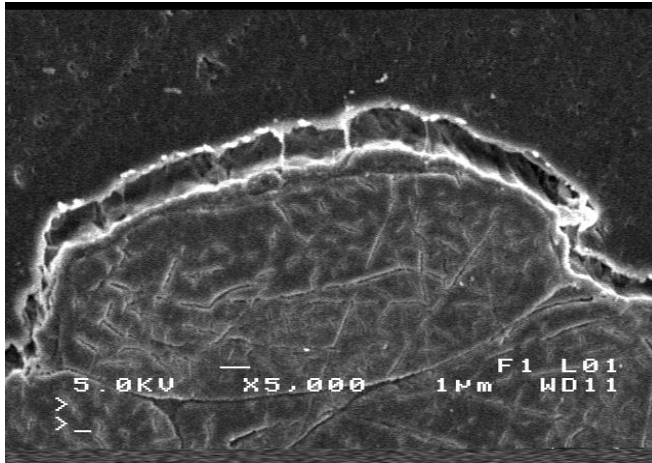


- ❑ Modulus and strength of the composites improve significantly with the presence of coupling agent
- ❑ Type of coupling agent also plays an important role
- ❑ The presence of CaO provide a great increase in modulus

Interface

No coupling agent

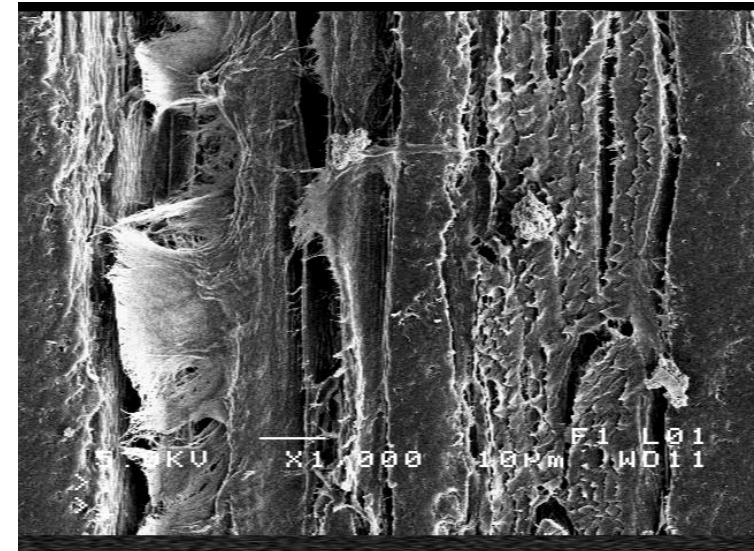
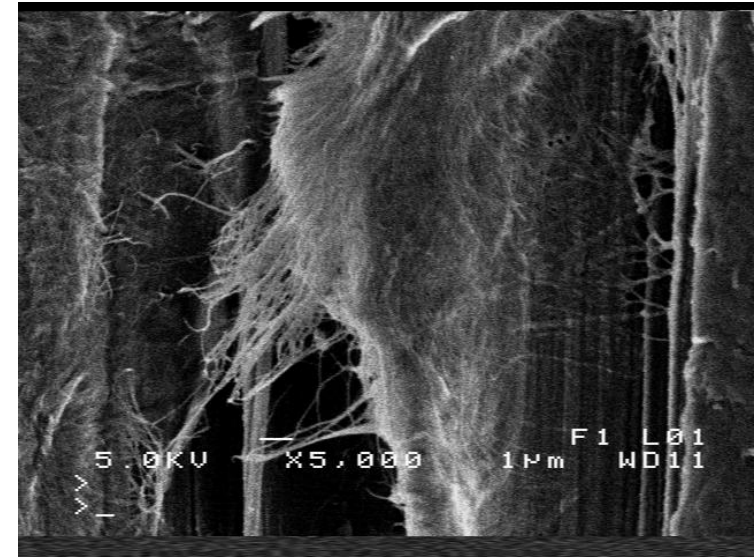
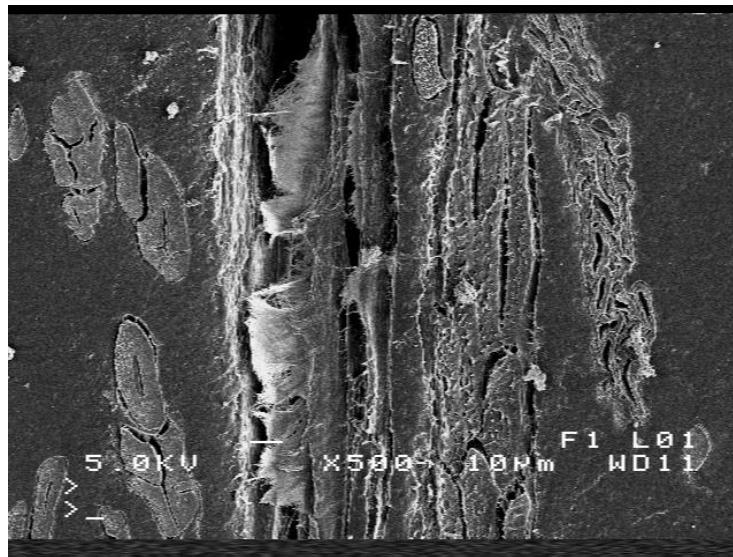
- Very poor interaction between the fiber and the matrix



Interface

With coupling agent

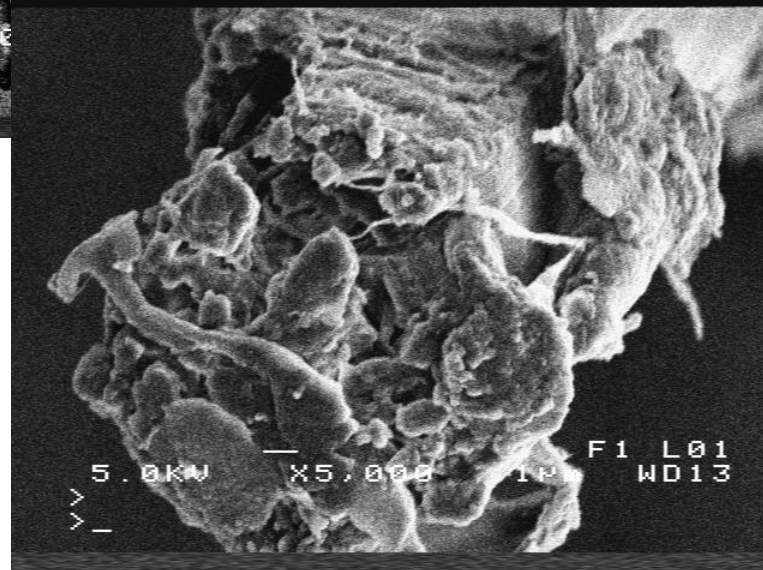
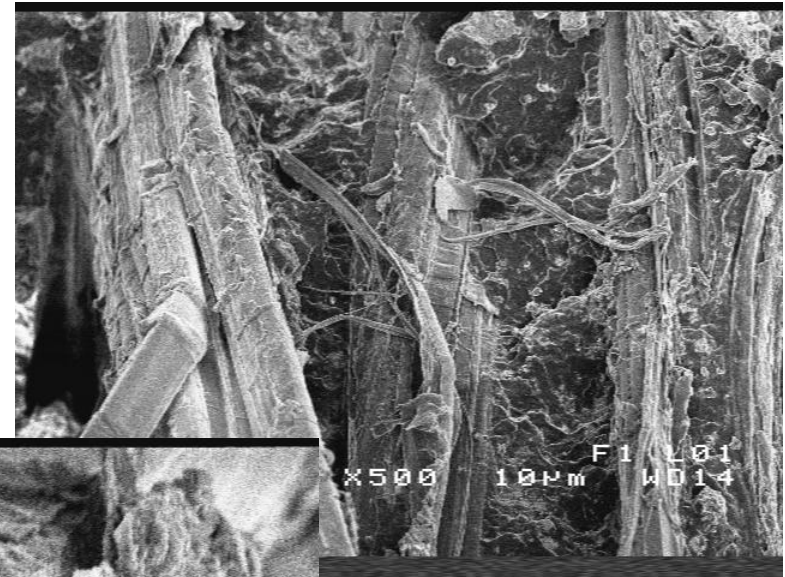
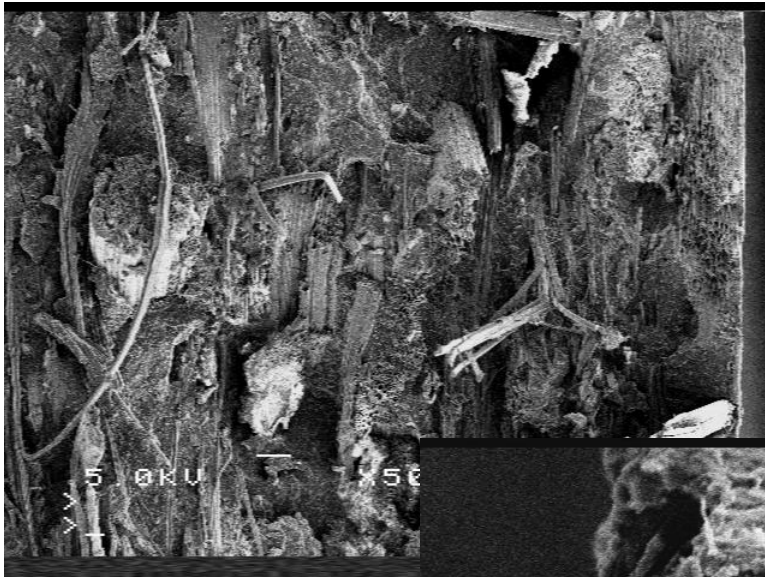
- Great improvement at the interface



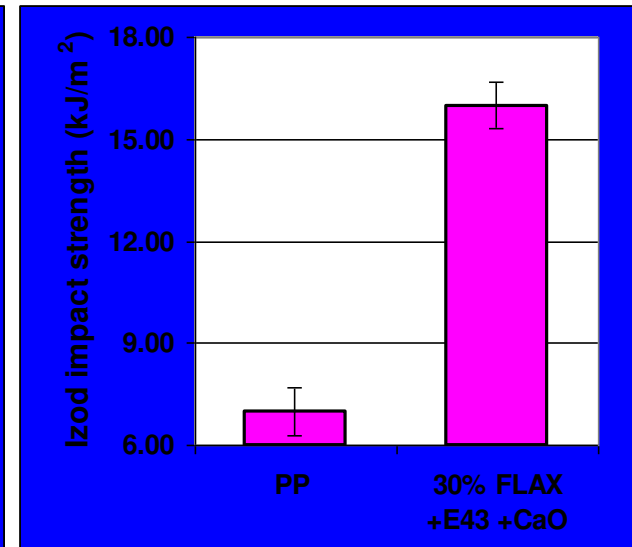
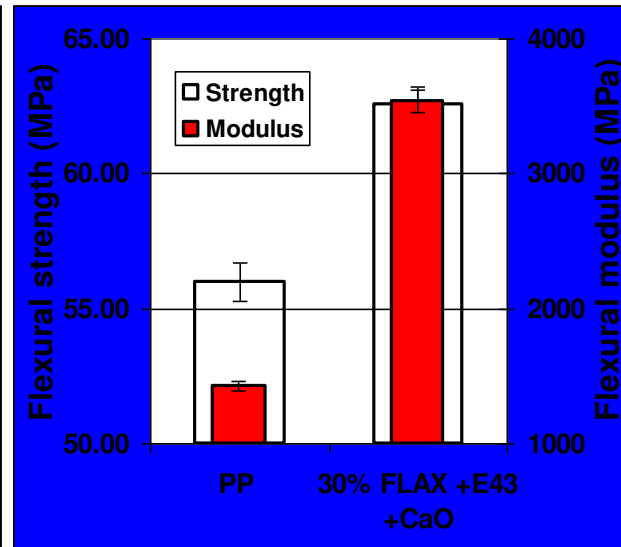
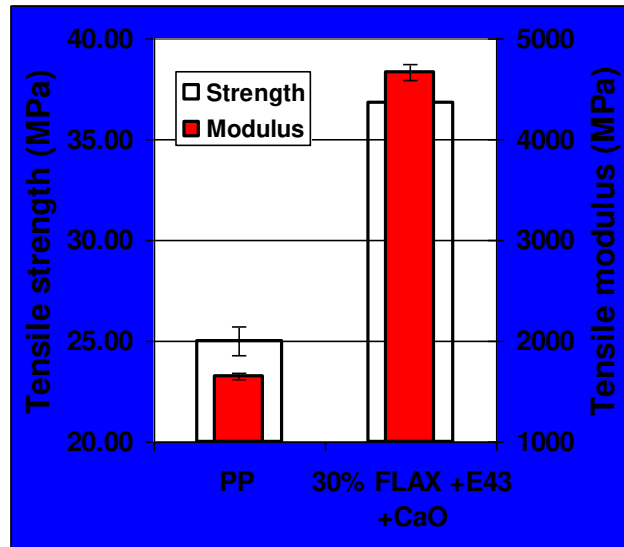
Interface

With coupling agent and CaO

□ Good interface



Mechanical properties – Injection molding

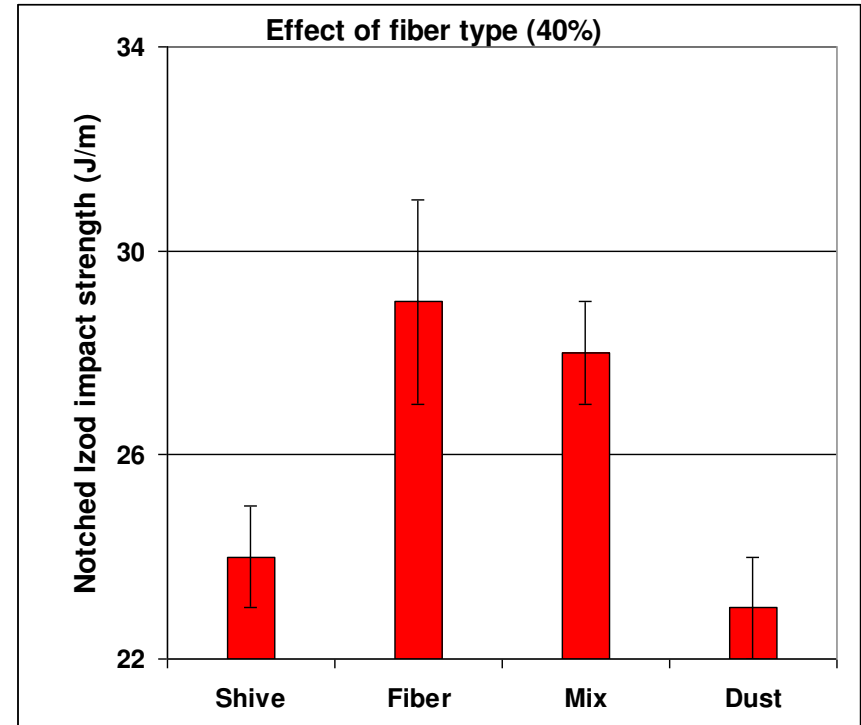
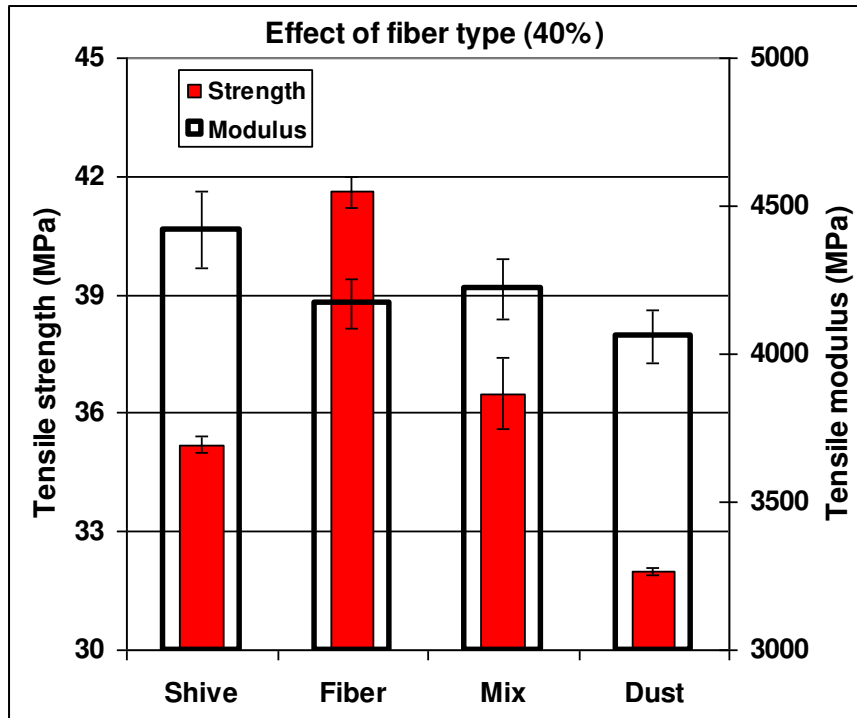


- ❑ Properties equivalent to wood-PP composites
- ❑ Process not optimized
 - ✓ Grinding
 - ✓ Extrusion
 - ✓ Injection



Various Flax types

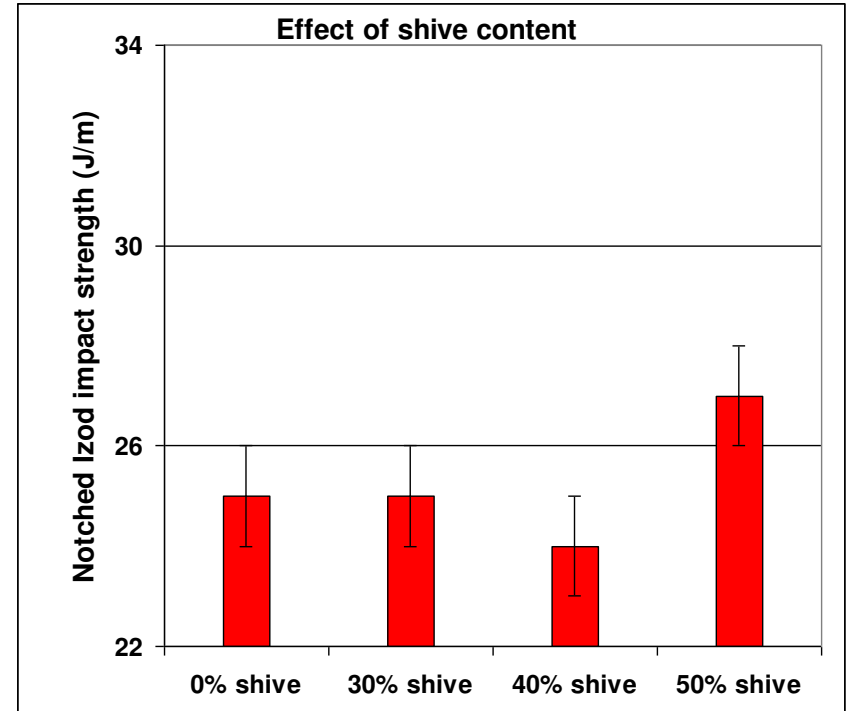
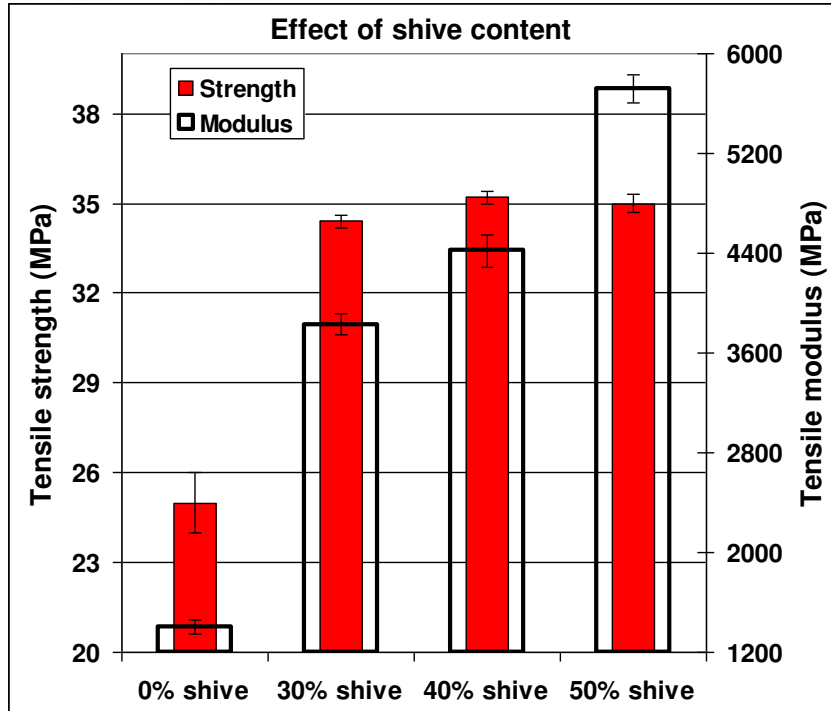
Extrusion-injection



- ❑ All types of flax, even dust, provide a significant improvement in strength and modulus (neat PP: 25MPa and 1400MPa, respectively) → **very positive!**
- ❑ The longer the reinforcement length, the greater the tensile strength and impact strength as the result of high aspect ratio (although fiber length has been reduced significantly during extrusion and injection)

Flax shive PP composites

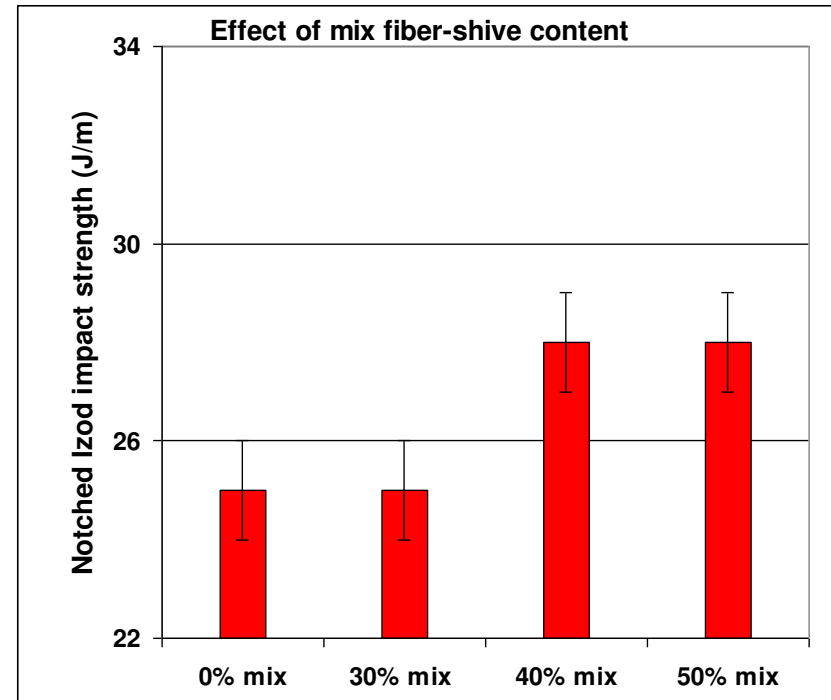
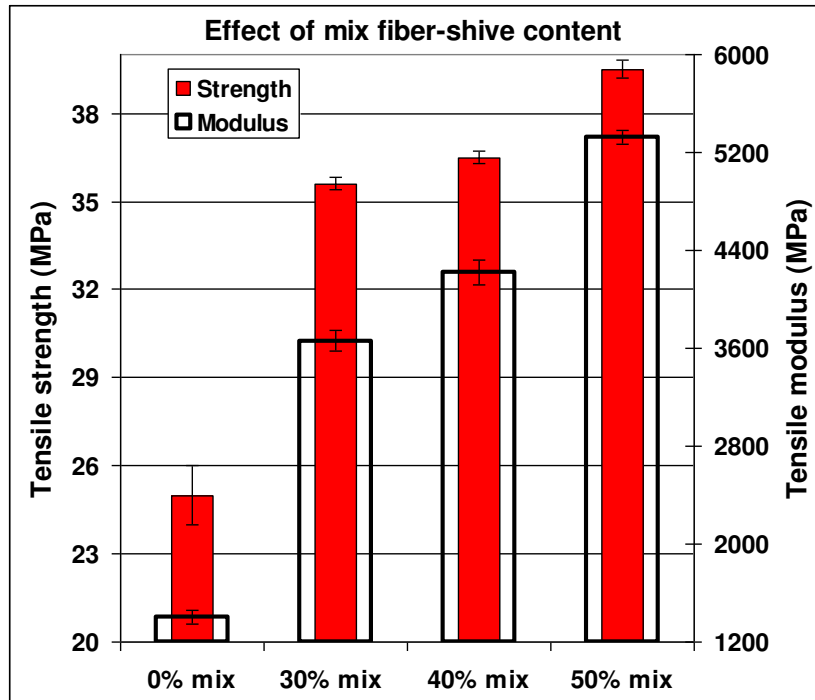
Extrusion-injection



- ❑ The presence of shives increases significantly the tensile strength (>35%) and modulus (180-300%) without affecting the impact strength
- ❑ The increase of shive content in the composites also increases linearly the tensile modulus but not the tensile strength

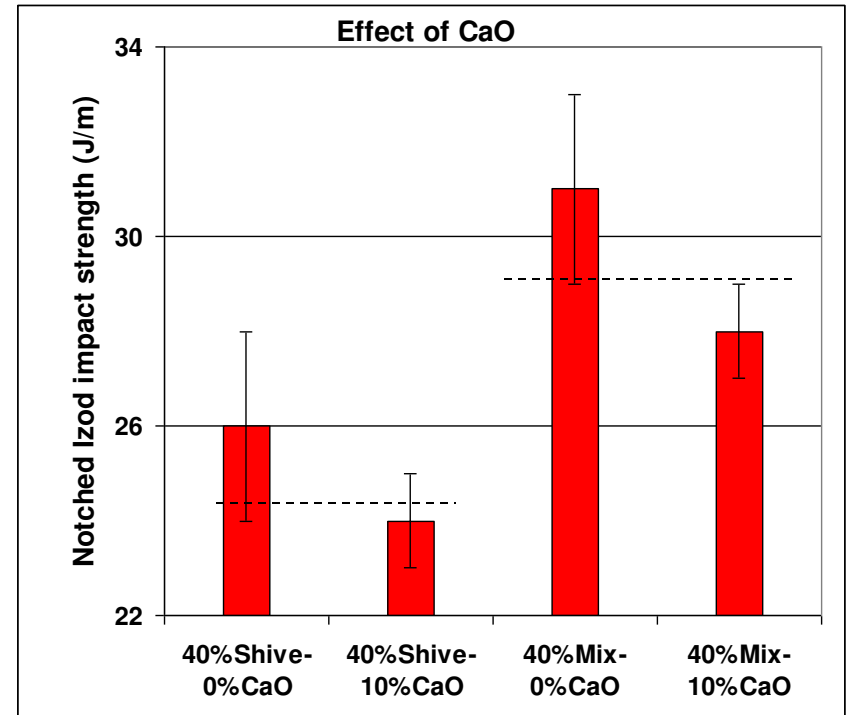
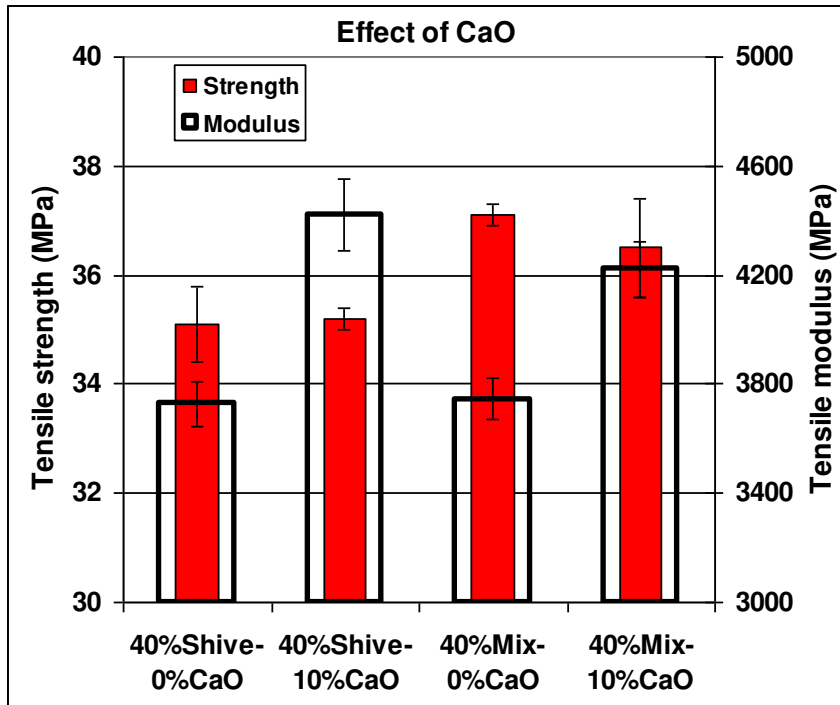
Mix shives-fibers

Extrusion-injection



- ❑ The presence of mixture of shives and fibers increases significantly the tensile strength (>35%), tensile modulus (160-300%) and the impact strength (~12%)
- ❑ The increase of the content of mixture of shives and fibers in the composites also increases linearly the tensile modulus and the tensile strength

Effect of CaO Extrusion-injection



- ❑ CaO improves more than 15% tensile modulus without sacrificing the tensile strength
- ❑ Impact strength was not affected by CaO (as standard deviation is taken into account) and remains the same as pure PP (25 J/m)

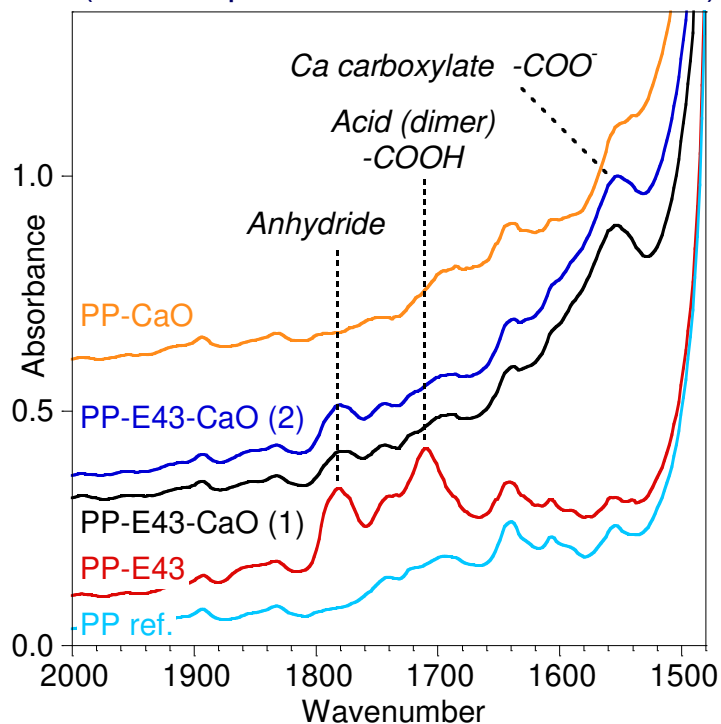
Roles of CaO

□ CaO

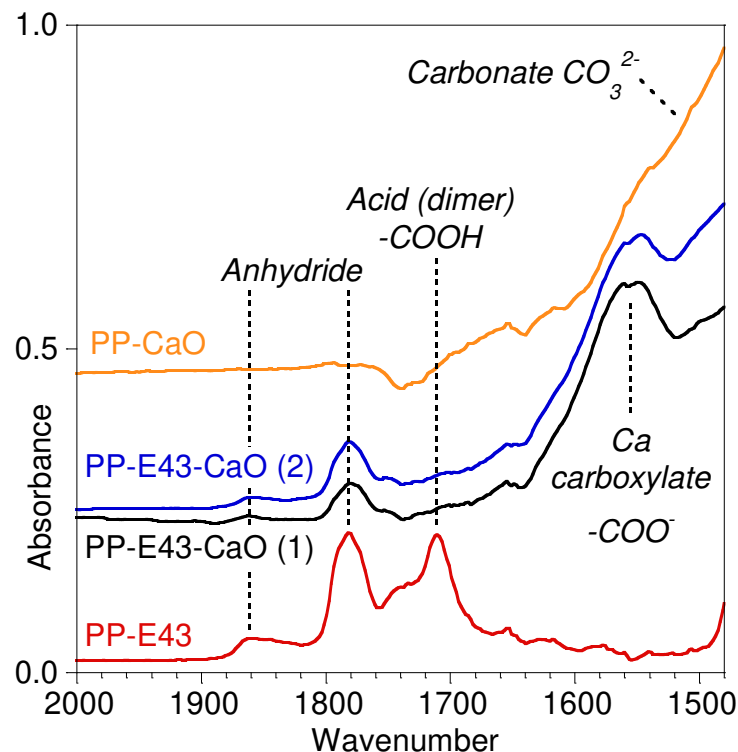
- Absorbs humidity in flax
- Neutralizes acidity in flax
 - minimize degradation during processing
- Reacts with maleic anhydride group of coupling agent
 - Improve interface between flax and PP matrix
 - Increase molecular weight of coupling agent
 - Limit a loss in toughness and impact

Interaction between CaO and coupling agent - FTIR study

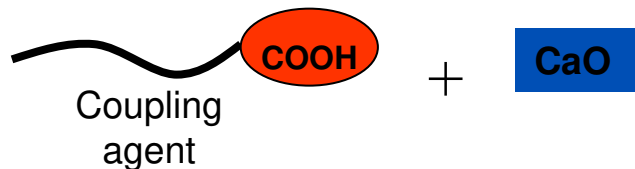
Spectra as measured
(thin hot-pressed films in transmission)



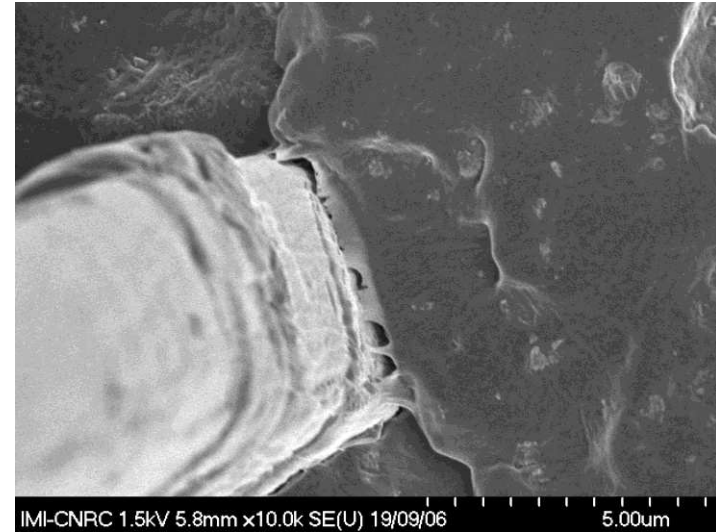
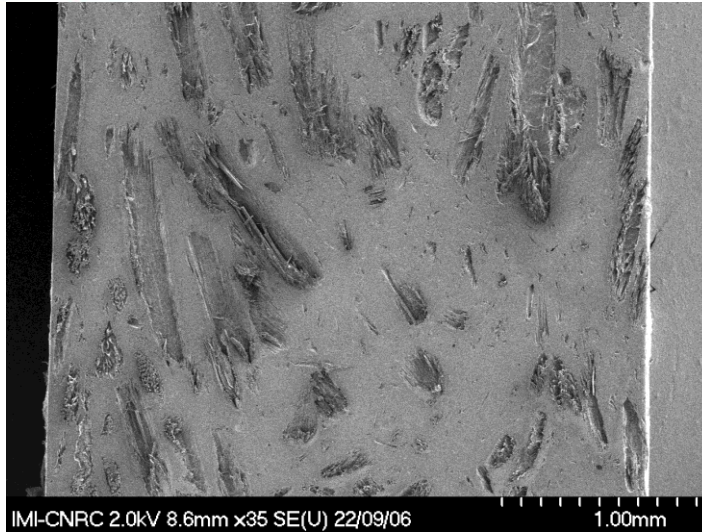
Difference spectra obtained by
subtracting PP ref.



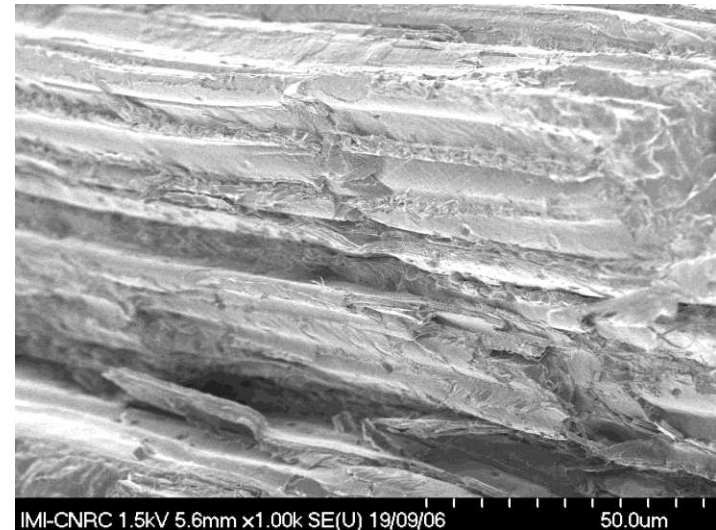
□ Evidence of chemical reaction between coupling agent and CaO



Dispersion and Interface Fiber composites



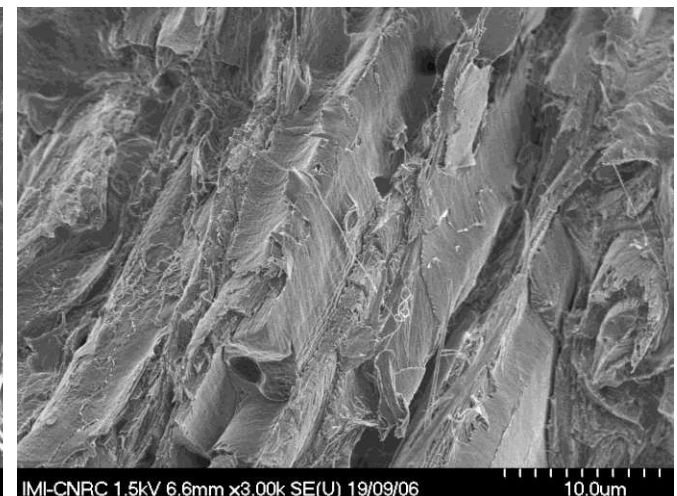
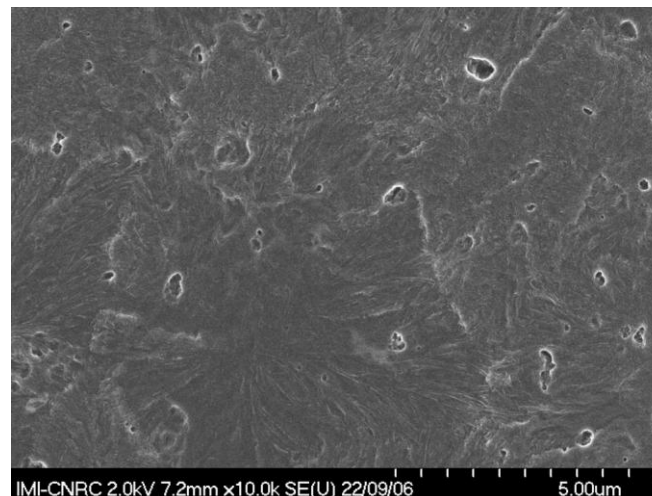
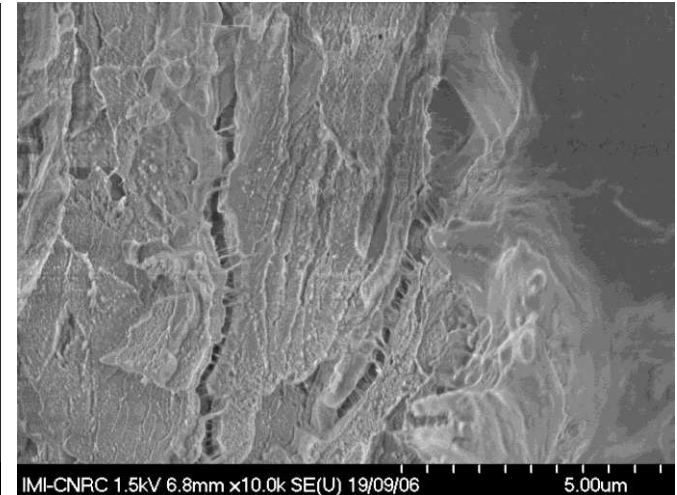
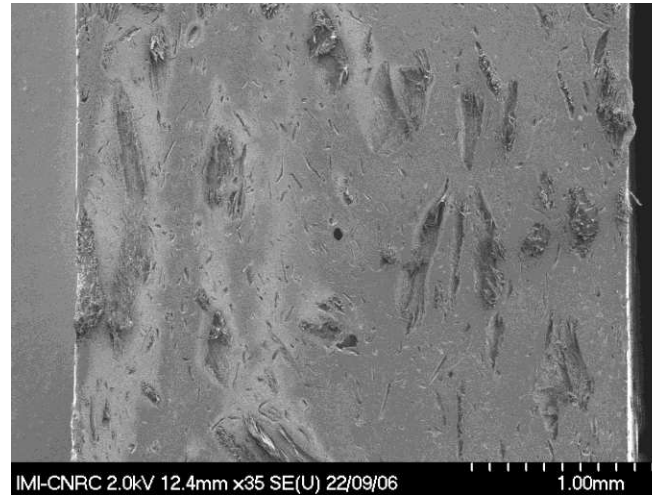
- ❑ Good dispersion and distribution of shives
- ❑ Good shive-matrix interface



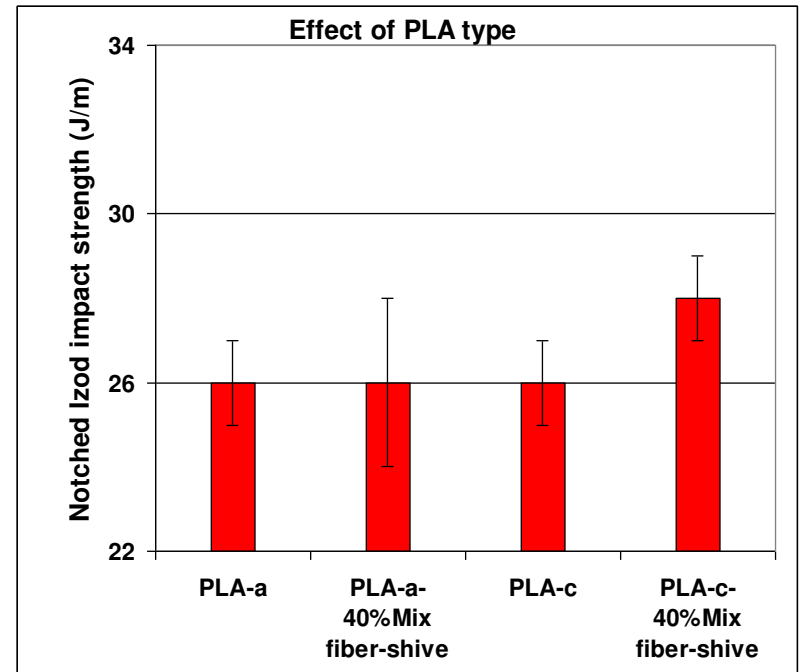
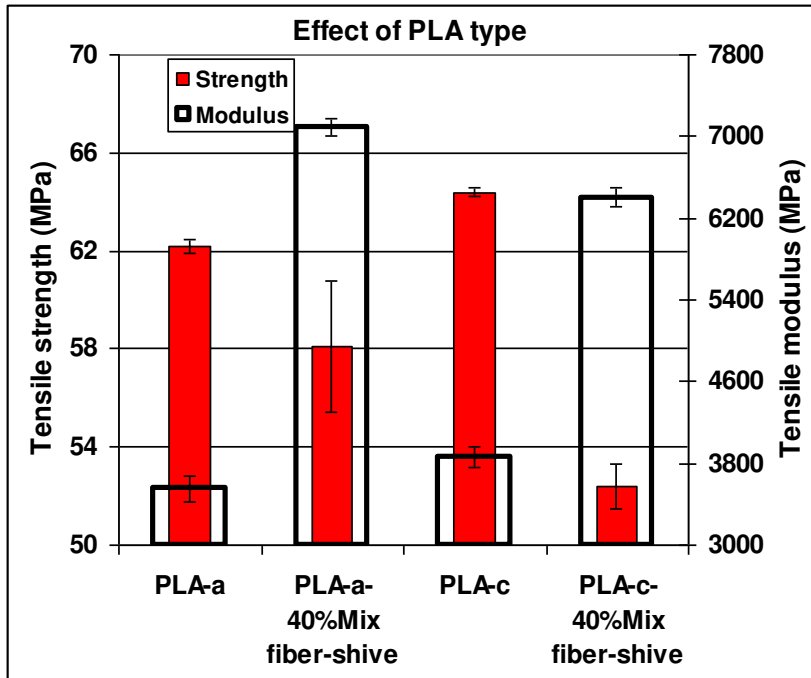
Dispersion and Interface

Mixed fiber-shive composites

- ❑ Very good dispersion of shives and fibers in the matrix and great reduction of fiber length can be observed
- ❑ Crystalline structure of PP matrix remains
- ❑ Very good fiber-matrix interface



PLA-Flax Composites



- ❑ Great improvement in modulus for both crystalline (~70%) and amorphous (~100%) PLAs
- ❑ Noticable reduction in tensile strength
- ❑ No effect on impact strength

Conclusions

- ❑ Flax fibers and shives can improve significantly the polymer performance even without fiber surface treatment.
- ❑ The composite properties are determined by many different factors: flax types, formulation, and processing equipment and conditions.
- ❑ The incorporation of some selective mineral fillers can greatly improves the stiffness and the impact resistance without scarifying the strength.
- ❑ CaO is the most promising candidate for such purpose.
- ❑ PLA-flax composites also show great interest although more work on optimization of formulation and processing conditions must be done.

Acknowledgement

The Government of Canada for the funding via CBIN project