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Mechanical properties of bionanocomposites with bacterial cellulose nanofibers and bacterial cellulose whiskers

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Objective

In the present work, bacterial cellulose sheets have been synthesized under standard HS conditions from Acetobacter Xylinum. After disintegration in water, **bacterial cellulose nanofibers** (BCN) have been directly used or hydrolyzed to obtain **cellulose whiskers** (BCW). Characterization of BCN and BCW has been carried out using X-ray diffraction, SEM-FEG & AFM.

These nano-fillers BCN & BCW have then been used at different concentrations to prepare **bionanocomposites** with natural rubber (NR). Mechanical properties have been analyzed and an important improvement of Young's modulus has been observed. For example adding **5% of BCN increased** the tensile modulus of natural rubber **more than 10 times**. A comparison of both bionanofillers is also detailed.

Current work

Introduction

In the last few decades, cellulosic nanocomposites have emerged as an innovative solution to produce advanced materials with improved properties [1]. The strategy consists of adding cellulose nanoscale fillers in various polymer matrices resulting in a mechanical reinforcement and alteration of properties, such as transport. Within the nanocellulose family, we can quote cellulose whiskers or nanocrystals [2], nanofibrillated cellulose (NFC or MFC), electrospun nanofibers and bacterial cellulose nanofibers (BCN) [3], all different in sizes and morphology.
To date, only a few studies have utilized cellulose from bacterial origin for nanocomposites. [4] However due to continuous nature of these fibers with nanoscaled diameter and high crystallinity, they appear as promising candidates for nanocomposites.

In this context, the objective of this work was to assess the impact of various morphologies of bacterial cellulose on the performance of natural rubber.

Material & Methods

Bacterial Cellulose Nanofibres (BCN)

Bacterial cellulose sheets were prepared under static conditions in a Hestrin-Schramm medium from Acetobacter Xylinum (strain 23769 American type culture collection). Purification was performed with the standard cycles in sodium hydroxide and washing in water. The bacterial cellulose nanofibers were used in this form and were also hydrolyzed into whiskers for the preparation and evaluation of two types of nanocomposites.

Bacterial Cellulose Whiskers (BCW)

Acid hydrolysis was achieved at 51°C with 60 wt % s ulphuric acid (pre-heated), for about 60 min, under magnetic stirring. The ²acid /cellulose ratio is 70 ml/g [1]. The suspension was diluted with ice cubes to stop the reaction and washed until neutrality by successive centrifugation,. The dry rate was about 66% of BCW

Microscopy

AFM :The measure was performed using an AFM Multimode (DI, Veeco, Instrumentation Group) in tapping mode with Multi 130 tips
SEM FEG was a Zeiss ultra 55 column Gemini

Films Preparations

Films have been made by solvent casting. BCW whiskers and BCN were placed in a water emulsionof 58% NR which was then cast in a small mold at controlled drying conditions.

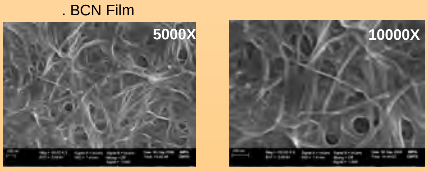
Films characterizations

Scanning Electron Microscopy (SEM): A field emission scanning electron microscope (FESEM), model Quanta 200 FEI, with accelerating voltage of 12.5 kV was used to study LC fibers and MFC surface topography.
Tensile test: When it was possible tensile tests were realised on 3 samples at room temperature whith a speed rate : 0.01 mm/min
Xray diffraction: The measure was performed using a panalytical X'pert pro MPD –ray diffractometer and using a co-filtered Co Kα=1.789A (6.9 Kev)

Results and discussion
BCW & BCN characterisation

SEM –FEG characterisation

. BCN Film

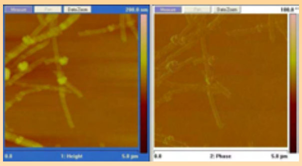


. BCW

Source	L (nm)	D (nm)	L/D
BCN	>1000nm	27 +/10	
BCW	558 +/- 174	22+/-6	23 +/-13

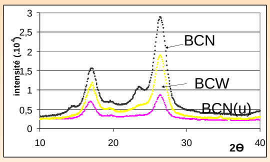
- Proof of nanoscale whiskers
- **Similar diameter** between BCN & BCW

AFM



•Similar values for BCN & BCW

X Ray diffraction



	Cristalinity Index (%)
BCN u	65
BCN	77
BCW	77

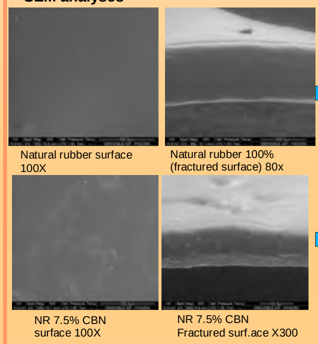
BCNu: BCN unpurified

The BCN cristallinity index is high and was maintained in BCW.

Results and discussion
Nanocomposites

- Nanocomposites films

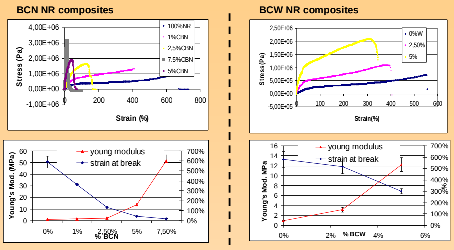
SEM analyses



Homogeneous in mass with teflon mold marks on surface

No important impact of CBN on homogeneity

Tensile test



BCN	εR (%)	σR (MPa)	E (MPa)
0	590 +/- 115	0,81+/- 0,32	1,01 +/- 0,15
1	365 +/- 79	1,17 +/- 0,18	1,66 +/- 0,51
2,5	134 +/- 4	1,57+/-0,13	2,47 +/- 0,26
5	46 +/- 4	1,92 +/- 0,03	13,82 +/- 0,6
7,5	18,17 +/- 5	3,16 +/- 0,06	51,3 +/- 10,9

BCW	εR (%)	σR (MPa)	E (MPa)
0	580+/-137	0,83+/-0,33	0,97+/-0,21
2,5	516+/-120	2,02+/-0,96	3,16+/-0,98
5	301+/-46	2,04+/-0,75	12,21+/-2,78

Concentration=%w/w

Addition of BCN & BCW **increases Young's Modulus** but decreases elongation

BCN and BCW have a similar impact on modulus whereas **elongation is most depressed with BCN** compared to BCW due to entanglement

Conclusion & Perspectives

-This study allows to work on **new source** for obtaining bio-nanoparticules.Their dimension are in the range of other whiskers

-There is no interest to produce BCW because **BCN are already mechanically efficient** except if elongation is expected. Indeed **BCW keep correct elongation** because they can move on each other.

- Film with 100% BCN or BCW and process optimisation possibilities could be considered as next steps

Sample	E_R	s_R	e_R	Ref.
NR-5 wt% CD whiskers	32.7	5.67	0.203	Siqueira et al 2010
NR-5 wt% starch nanocrystals	2.46	1.95	0.915	Angellier et al. 2005
NR-5 wt% palm tree whiskers	16.8	4.84	0.402	Bendahou et al 2009
NR-5 wt% chitin whiskers	10.5	1.76	0.530	Gopalan Nair et al. 2003

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