Cellulose nanocrystals obtained from cynara cardunculus: lab procedure, SEM analysis, and optical properties

Valentina Coccia, Franco Cotana, Gianluca Cavalaglio, Mattia Gelosia, Enrico Pompili
CIRIAF - CRB Section - University of Perugia, via G. Duranti, 67, 06125
Perugia, Italy. E: coccia@crbnet.it

Keywords: bio-based product, biorefinery, cellulose nanocrystals, residual biomass, steam explosion

Biorefinery purpose aims at designing new virtuous and high-efficiency energy chains, achieving the combined production of biofuels (e.g. bioethanol) and biobased products obtained from by-products and residues. This contribution will present the lab experience carried out by the Italian Biomass Research Centre (CRB) in extracting cellulose nano-crystals (NCC) from a pre-treated (*via Steam Explosion*) fraction of cynara cardunculus i.e. a very common and abundant residual and invasive arboreal variety in central Italy.
A parallel experimental programme is on going for producing bioethanol from some rurally available ligno-cellulosic matrices (Cotana et al. 2014).
The NCC extraction methodology consists of a five step protocol allowing the separation of the nanocrystalline content of cellulose. Such a procedure is literary captured (Oksman et al. 2011, Pirani and Hashaikeh 2013) with the exception of *Step (iv)* that is only CRB Lab experienced and it has been applied for the production of NCC from bio-residual matter (i.e. cynara cardunculus) and as a baseline comparison from the micro crystalline cellulose (MCC). The main protocol phases are mentioned as follows: *(i)* extractives removal from the bioresidue using the Soxhlet apparatus; *(ii)* lignin separation from the cellulose component using basic hydrolysis; *(iii)* the pulp energy bleaching (Marques et al. 2010) with sodium chlorite at controlled pH; *(iv)* acid hydrolysis to deconstruct the cellulose into its two components: the crystalline and the amorphous one; *(v)* ultrasound treatment of the solution and quantification NCC content. In addition, some initial SEM analyses and characterization measurements of the optical properties have been carried out on the obtained NCC glass films.

PREPARATION OF NCC SAMPLES

1 STEAM EXPLOISION PRETREATMENT

pre-treated (*via Steam Explosion*) fraction of cynara cardunculus

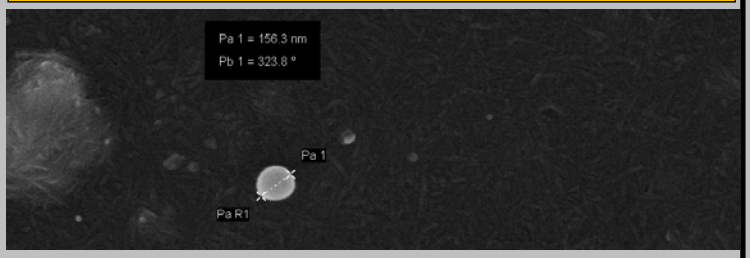
2 LAB EXTRACTION

NCC is typically isolated from cellulose by controlled hydrolysis [1,2]. Amorphous domains are preferentially hydrolyzed, whereas crystalline regions have higher resistance to acid attack. Several steps are necessary to obtain NCC dispersion or solid.

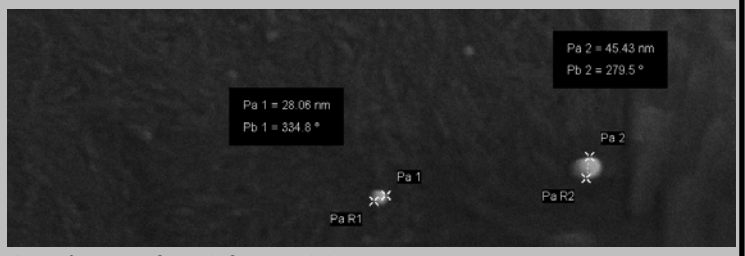
3 NCC GLASS FILM DEPOSITION

Casting deposition

SEM ANALYSIS



SEM image of NCC obtained from cynara cardunculus



SEM image of NCC from MCC

SPECTROPHOTOMETER ANALYSIS

Standard UNI EN 410

Measurement of solar reflectance for glass surfaces

First result *(to be further lab confirmed)*

A measured increase of the total solar reflectance of 30% in the UV range (up to 400 nm) with respect to the reflectance efficacy of a common glass surface.

RELEVANT REFERENCES

1. Cotana F., Cavalaglio G., Gelosia M., Nicolini A., Coccia V., Petrozzi A. (2014). Production of bioethanol in a second generation prototype from pine wood chips. *Energy Procedia*, 45c, 42-51.
2. Marques G., Del Río J.C., Gutiérrez A. (2010). Lipophilic extractives from several nonwoody lignocellulosic crops (flax, hemp, sisal, abaca) and their fate during alkaline pulping and TCF/ECF bleaching. *Bioresource Technology,* 101, 260 – 267.
3. Oksman, K., Etang, J. A., Mathew A. P., Jonoobi M. (2011). Cellulose nanowhiskers separated from a bio-residue from wood bioethanol production. *Biomass and Bioenergy*, 35, 146-152.
4. Pirani, S., Hashaikeh, R. (2013). Nanocrystalline cellulose extraction process and utilization of the byproduct for biofuels production. *Carbohydrate Polymers*, 93, 357– 363.