Production of cellulose nanowhiskers

Novel materials derived from the low-cost and renewable resources have drawn significant interest to the materials scientists with the growing environmental concern of end-of-life disposal challenges. Materials obtained from renewable bioresources could be the alternative to petroleum based synthetic products due to their advantages of potentially relatively low cost, environmentally friendly nature, easy availability, renewability, and nontoxicity. Cellulose is the most abundant natural polymer on the planet.

The production of cellulose nanowhiskers (CNWs) to be used as nano-reinforcements in PLA-PHB blends started in month 1.

The first aim of the task was to isolate cellulose from wheat straw (WS) in order to obtain microcrystalline cellulose to be used to obtain CNWs by acid hydrolysis. Cellulose nanowhiskers can be modified to achieve a better dispersion in the PHB matrix.

Besides renewability and biodegradability, the production of cellulosic fibers in nano-dimensions adds promising properties, such as high mechanical performance, hydrophilicity, broad chemical modification, formation of versatile semi-crystalline fiber morphologies, large surface area and low density. Therefore, nanocellulose could cover many application fields.

Currently, cellulose nanowhiskers are mainly produced by acid hydrolysis/heat-controlled techniques, with sulfuric acid being the most utilized acid. Extraction of the crystals from cellulose fibers involves selective hydrolysis of amorphous cellulose regions, resulting in highly crystalline particles with source-dependent dimensions, for example, 5–20 nm x 100–500 nm for plant source CNWs. Sulfuric acid hydrolysis grafts negatively charged sulfate half-ester groups onto the surface of the particles, which prevents aggregation in aqueous suspensions due to electrostatic repulsion between particles.

For microcrystalline cellulose isolation from wheat straw, the WS was submitted to a lignocellulosic fractionation process. This was basically a two-step process with a first acid hydrolysis followed by an alkaline hydrolysis. The first step was carried out either as the most conventional dilute acid hydrolysis, or as a less conventional autohydrolysis process. Autohydrolysis was tested at three different temperatures to select the best conditions for cellulose recovery.

Figure 1: Berghof high pressure reactor (BTC-3000, Germany) used for the autohydrolysis of wheat straw

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Development of New Competitive and Sustainable Bio-Based Plastics

The production of CNWs was carried out by acid hydrolysis. In an acid treatment method, yield of nanocellulose depends on the composition of the lignocellulosic biomass and the reaction conditions such as acid concentration, time and temperature. Optimization of experimental parameters is required to obtain maximum yield and to preserve the nanocellulose morphology.

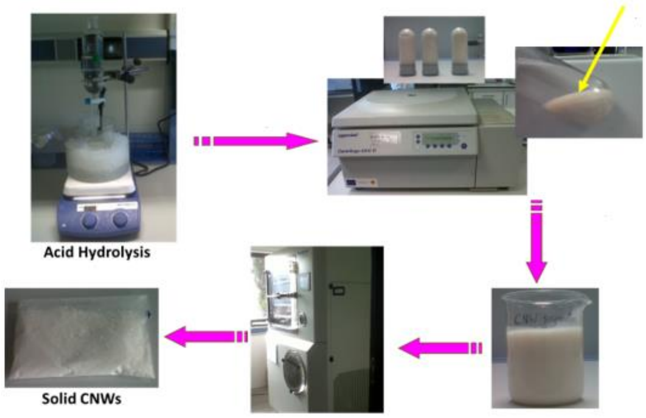

Figure 2: Steps of CNWs production from MCC at lab scale

TEM micrographs revealed that the particles generated were in the nanometer range. The typical diameter of cellulose nanowhiskers is around 2-20 nm, but there is a wide length distribution from 100 to 600 nm and even 1 µm in some cases. In this case, the diameter of CNWs was around 8-15 nm and the length was between 200 and 350 nm approximately. Based on the results obtained at lab scale, a pilot plant design is proposed to produce CNWs from MCC.

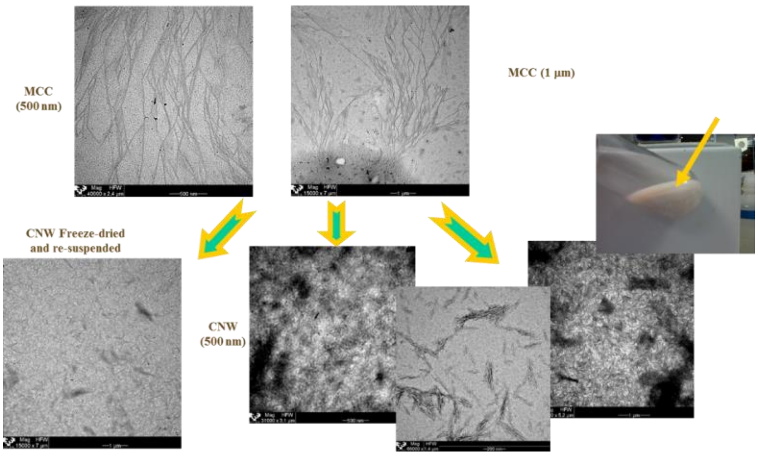

Figure 3: Examples of TEM micrographs of MCC and lyophilized CNWs

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