# Amorphized Cellulose as Filler for Innovative Ecocomposites

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## ABSTRACT

In this work the results of effects induced by a dry ball milling process on cellulose structure, morphology and properties are reported, together with results on the preparation of new polycaprolactone (PCL) based composites prepared with unmodified and ball milled cellulose.

ATR-FTIR technique allowed to qualitatively define cellulose structural changes occurred as a consequence of the ball milling process. The analysis of WAXD spectra revealed that cellulose undergoes to a progressive amorphization during the ball milling process. The cellulose crystallinity index decreases from 0.53 (neat cellulose) to 0.15 (after 60 min ball milling). The mean size of the cellulose crystallite domains was also calculated according to the Scherrer equation, showing a reduction from the original value of about 4.0 nm up to about 3.4 nm after 30 min ball milling.

As an effect of the cellulose amorphization, the amount of absorbed water of cellulose conditioned at 25°C and 50% RH increases with increasing the milling time, varying from 7.3 wt% for untreated cellulose up to 11.6 wt% for the cellulose sample ball milled for 60 min. This behaviour confirmed that water can be easily absorbed by the amorphous fraction, whereas crystalline domains are less accessible to water molecules.

Another interesting consequence of the ball milling treatments is that this process strongly influences the morphology of the cellulose. The average length of the fibres decreases from an original value of about 200 μm up to a final size, after 60 min ball milling, of about 12 μm. The original fibrous morphology of the cellulose is modified to a quasi-circular shape.

For PCL/cellulose composites, cellulose reinforcement was found able to induce a progressive increase of the Young's modulus of PCL with increasing the filler loading. Nevertheless this effect is less marked for composite samples reinforced with ball milled cellulose, either because of its amorphous structure, either because of its reduced aspect ratio. On the contrary, a significant decrease of the tensile strength was recorded for the composites, thus indicating a low interfacial adhesion between the cellulose filler and the PCL matrix.

An interesting result was found by evaluating the elongation at break, because the composite reinforced with ball milled cellulose, also at 30 wt% filler loading, still keeps a noticeable deformability, higher than 300%.

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In order to improve the polymer/filler interfacial adhesion, a modified PCL was prepared and used as compatibilizing agents for the composites realized with unmodified and amorphized cellulose. The addition of the compatibilizing agent was proved to be effective to induce a relevant increase of the stress at yield in the compatibilized composites. This improvement was confirmed by SEM analysis carried out on cryogenically fractured samples.