X-RAY DIFFRACTION ANALYSIS OF MICROCRYSTALLINE CELLULOSE OBTAINED FROM BROUSSONETIA

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Microcrystalline cellulose (MCC) is a product composed of highly ordered crystalline regions of cellulose, typically obtained by removing amorphous regions through acid hydrolysis of alkali-treated or purified cellulose. Due to its high degree of crystallinity, chemical stability, biocompatibility, and mechanical strength, MCC is widely used in pharmaceuticals, the food industry, cosmetics, composite materials, and environmental sorbents. The main objective of this study is to perform a comparative analysis of the crystalline structure of alkali-treated fibers and MCC samples obtained from Broussonetia using X-ray diffraction (XRD). XRD analysis enables the identification of crystalline and amorphous phases of cellulose, evaluation of the degree of crystallinity, and assessment of the effectiveness of the treatment process.

Natural fibers obtained from Broussonetia were selected as the object of the study. Initially, the raw material was mechanically cleaned, ground, and fractionated, and then prepared for alkaline treatment. During the alkaline treatment process, the fibers were treated with a NaOH solution, resulting in the partial removal of amorphous components such as hemicellulose, lignin, and pectin. Subsequently, the material was washed, neutralized, and dried. For the production of microcrystalline cellulose, the alkali-treated fibers were subjected to additional chemical treatment. At this stage, the amorphous regions of cellulose were partially degraded, enriching the relative content of crystalline domains. The obtained samples were then prepared for X-ray diffraction analysis. During XRD analysis, diffraction peaks were observed within the 2θ angle range. Particular attention was paid to the crystallographic planes characteristic of cellulose I structure, namely (101), (10$\bar{1}$), (002), and (040). The X-ray diffractograms of the alkali-treated fibers and MCC samples showed major diffraction peaks at $2\theta \approx$ 14–16°, 22–23°, and 34–35°. These peaks are characteristic of the cellulose I crystalline structure, indicating that cellulose crystallites are preserved in materials derived from Broussonetia. In the MCC sample, the peak corresponding to the (002) plane at around $2\theta \approx 22.5°$ exhibited the highest intensity. The strong and sharp nature of this peak indicates a high crystalline phase content in MCC. In addition, the peaks corresponding to the (101) and (10$\bar{1}$) planes at $2\theta \approx$ 14–16° are more pronounced in MCC compared to alkali-treated fibers. This suggests that, as a result of acid or additional treatment, amorphous components are reduced, leading to an increase in the proportion of ordered crystalline regions of cellulose. In contrast, the diffraction peaks of alkali-treated fibers are relatively broader and of lower intensity, indicating the presence of a certain amount of amorphous phases within the material. These amorphous phases may consist of hemicellulose, lignin, pectin, and disordered regions

of cellulose. At the same time, alkaline treatment does not completely destroy the cellulose structure; rather, the main crystalline lattice is preserved. The peak corresponding to the (040) plane at around 2θ ≈ 34–35° is also more clearly observed in the MCC sample.

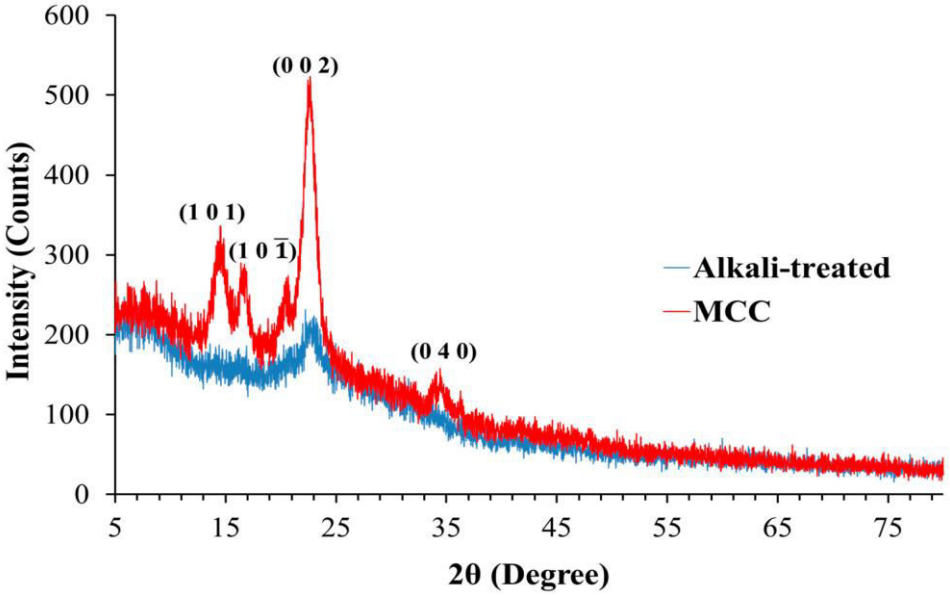


Figure 1. X-ray Diffractograms of Alkali-Treated Fibers and MCC (Microcrystalline Cellulose) Obtained from Broussonetia

This confirms the ordered arrangement of cellulose chains and the formation of crystallites. In alkali-treated fibers, the peak in this region is less pronounced, indicating a lower degree of crystalline ordering. A comparison of the diffractograms shows that the process of obtaining MCC increases the crystallinity of Broussonetia fibers. During this process, the amorphous regions are reduced, and the crystalline domains of cellulose become relatively enriched. As a result, MCC is formed as a material with a high degree of structural order, good mechanical stability, and chemical resistance. The X-ray diffractograms of alkali-treated fibers and microcrystalline cellulose obtained from Broussonetia demonstrate that both materials possess a cellulose I crystalline structure. In the MCC sample, the diffraction peaks—particularly the peak corresponding to the (002) plane—exhibit higher intensity, confirming a higher degree of crystallinity. In contrast, the peaks observed in alkali-treated fibers are broader and of lower intensity, indicating the partial retention of amorphous components. Overall, Broussonetia can be considered a promising lignocellulosic raw material for the production of high-quality microcrystalline cellulose.

References:

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