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X-ray Powder Diffraction Study of Hydrogel Chitosan Membranes

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Nowadays, natural polymeric materials have become increasingly important due to their abundance and low costs. Of many kinds of polysaccharides, chitin and its derivative chitosan are particularly interesting because of their unique physicochemical and biological properties [1]. The prominent features are closely related to the amino groups of these amino polysaccharides that are structurally similar to a cellulose which possesses only hydroxyl groups. Chitin and chitosan are thus versatile biopolymers of growing importance in biotechnology with many emerging applications in medicine, agriculture ad environment protection [2]. In our study a structural characteristic of hydrogel chitosan membranes is given. The membranes were formed by the phase inversion method. Chitosan of molecular weight of 500, 700 kD and deacetylation degree of 90, 67 % respectively, was used in the experiments. The membrane-forming solutions were chitosan salts in the form of acetate**,** lactate and malate with different polymer concentrations. The amount of solvent was chosen stoichiometrically to the content of amino groups in a chitosan molecule. The structural characteristics were based on the analysis XRD powder diffraction patterns. X-ray diffraction results were obtained using X'PERT PRO powder diffractometer employing CuK*a* radiation. Hydrogel chitosan membranes reveal better ordering than the initial polymer. Pure chitosan exhibits rather amorphous properties.

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Using Preferred Orientation to Resolve Overlapping Reflections

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In 1999, Wessels *et al.* [1] demonstrated the practical viability of the 'texture method' for resolving reflections that overlap in a powder diffraction pattern. More information about the relative intensities of overlapping reflections could be obtained, by collecting synchrotron data on a textured, polycrystalline sample as a function of sample orientation. In contrast to other related approaches, a full texture analysis was used to establish how the crystallites were oriented in the sample. This information was then used to extract a single set of single-crystal-like reflection intensities via a joint refinement procedure using all diffraction patterns (between 5 and 1296) simultaneously.
The data collection strategies for both, reflection and transmission geometries have been described [2]. Both have their drawbacks. The reflection mode experiment requires 3 days of synchrotron beamtime per sample, extreme corrections to the data for higher tilt angles, and a large homogeneously textured specimen. The transmission mode experiment (using an area detector) allows the beamtime to be reduced to *ca* 6h per sample, involves no tilt correction, and requires only a very small sample, but these advantages are gained at the expense of data resolution. In an attempt to improve the resolution of the transmission geometry setup, the experiment has been adapted to accommodate the one-dimensional Si microstrip detector on the powder diffractometer on the Materials Science Beamline at the SLS [3]. This setup has allowed the resolution of the data to be improved both in 2θ (from 0-35° to 0-60°) and in peak width (from *ca* 0.06°2θ to *ca* 0.03°2θ) with an acceptable increase in the amount of beamtime required (*ca* 12h per sample).
New techniques for preparing textured samples have also been explored. In most cases, the polycrystalline material is dispersed in a polymer solution, and then subjected to shear forces. A variety of polymers and shear techniques have been investigated in an attempt to obtain stronger textures with a minimal amount of polymer in the final specimen.
The data analysis software has been modified to accommodate the data from the Si-microstrip detector, and test datasets have been collected and analyzed. The framework structure of the zeolite offretite ($P\bar{6}m2$, a = 13.291 Å, c = 7.582 Å) could be solved from the extracted intensity data without difficulty. Similarly, the vanadate chains in viologen vanadate ($C2/m$, a = 21.232 Å, b = 6.318 Å, c = 3.585 Å, γ = 94.92°) could be found by direct methods using default input. Analysis of the data collected on a silicate of unknown structure is in progress.

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