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Synchrotron X-ray Scattering Study of the Formation of Polyoxomolybdate Nanostructures

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Beamline(s): X27C, X3A2

Introduction: Nanostructured materials have attracted intense interest from various disciplines due to their unique size-dependent properties [1]. The general strategy to synthesize nanostructured materials is to employ pre-organized surfactants or block copolymers as structuring templates [2,3]. We have successfully synthesized a primitive cubic polyoxomolybdate structure [4] with a lattice constant of 5.2 nm by mixing a PEO-containing polymer with $\text{MoO}_2(\text{OH})(\text{OOH})$ aqueous solution. X-ray scattering experiments were performed to characterize the polyoxomolybdate nanostructures and to investigate their formation mechanism.

Methods and Materials: By centrifugation, various PEO-containing polymers were mixed thoroughly with the $\text{MoO}_2(\text{OH})(\text{OOH})$ precursor solution to form a yellow and transparent gel. This original yellow polymer gel finally changed into a dark blue polymer gel. The evolution between the original and final gels was monitored by both SAXS and WAXD techniques.

Results: Typical SAXS patterns of the original and final polymer gels are shown in figure 1. The original P-104 (55 wt%) gel is a two-dimensional hexagonal structure, which is different from the primitive cubic structure of the final product (right part of curve B). Polymer gels certainly did not play a role as a structuring template. The structure of the polymer gel was changed (left part of curve B), or even destroyed in some cases, indicating strong interactions between polymer and inorganic compound. The color change from yellow to dark blue implied that molybdenum was chemically reduced from valence VI to a mixed valence (VI/V) by the PEO blocks.

Conclusions: Primitive cubic polyoxomolybdate nanostructures were synthesized by mixing various PEO-containing polymers with a molybdenum precursor solution. Instead of being a structuring template, the polymer gel stabilized and reduced the molybdenum compound forming polyoxomolybdate nanostructures.

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References:

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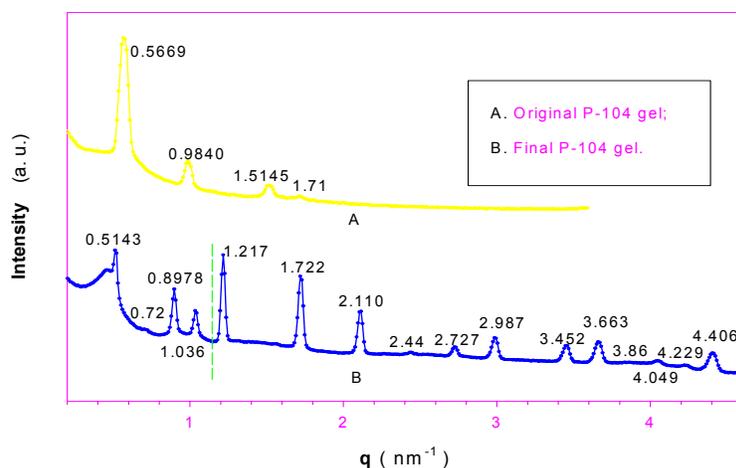


Figure 1. SAXS patterns of original and final P-104 (55wt%) gels.