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Direct Synthesis of Nanostructured Oxides by Polyol-Mediated Method

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Abstract – A simple and cheap polyol-mediated method was used to obtain nanostructured particles with enhanced properties. Nanostructured tin oxide and glycolate as well as zinc oxide were prepared by reflux in ethylene glycol media starting from ordinary salts. The as-synthesized tin compounds were tested as electrode in reversible Li-ion batteries and exhibited extremely stable cycling performance with a discharge capacity above 400mAh g-1 for 50 to 100 cycles. The ZnO electrode made with as-synthesized particles showed to be stable structure for 20 cycles.

The tin oxide and tin glycolate nanoparticles were obtained from tin chloride di-hydrate and tin oxalate, respectively. In a typical procedure, the synthesis consists in adding the precursor salt to ethylene glycol followed by refluxing at 195 ^{o}C for about 4-5 hours. Magnetic stirring in air conditions is applied during the entire synthesis. After cooling to room temperature the particles are washed in acetone and separated by centrifugation. The chemical reaction that is supposed to lead to SnO_2 nanoparticles in this case is [1]

$$SnCl_2 \cdot 2H_2O + 1/2O_2 \xrightarrow[195°C]{EG} SnO_2 + 2HCl + H_2O$$

When tin oxalate was used as precursor salt, tin glycolate was obtained after refluxing in ethylene glycol. This compound is formed of hexagonal-shaped, micron-sized tin glycolate particles which, after heat treatment carried out in air at 600-800 ^{o}C for 2 hours, produce a series of tin oxides. Investigation under scanning electronic microscope showed that after the heat treatment the hexagonal-shaped tin glycolate consisted of nanosized tin-based particles (80-120 nm), encapsulated within tin glycolate shell [2].

Zinc oxide nanoparticles with dimensions between 20-30 nm were obtained using the polyol method from zinc acetate precursor salt using the cited typical procedure.

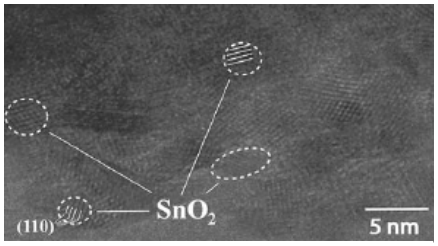

Figure 2: Micrograph (TEM) of tin oxide nanoparticles obtained by polyol-mediated method.

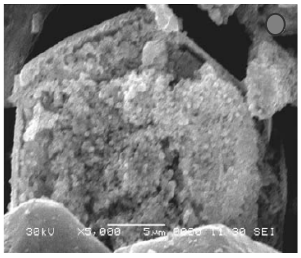

Figure 2: Micrograph (SEM) of tin oxide nanoparticles encapsulated in tin glycolate shell.

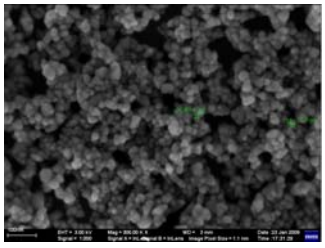

Figure 3: Micrograph (FEG) of zinc oxide nanoparticles obtained by polyol-mediated method.

All these materials were tested as electrodes in reversible Li-ion storage and, although, the tin oxide obtained from chloride presented the highest surface area, retaining 400 mAhg^{-1} beyond 100 cycles, it showed a extremely high first cycle irreversible capacity loss of 72% due to decomposition caused by the direct contact with the electrolyte. The tin oxide encapsulated in the glycolate shell showed to be electrochemically improved since the tin oxide particles are protected from the electrolyte. In this way, it was possible to have about the same specific charge capacity with reduced solid/electrolyte interface formation in the electrode. For zinc oxide nanosized particles the electrochemical tests are in progress.

[1] Ng, S. H., Dos Santos, Dayse Iara, S.Y. Chew, Wexley, D., J. Wang, Dou, S. X., H.K. Liu Electrochemistry Communications **9** (2007) 915.
[2] Ng, S. H., Chew, S., Dos Santos, Dayse. I., Chen, J., J. Wang, Dou, S. X., H.K. Liu Chemistry - An Asian Journal **3** (2008) 854.

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