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Preparation of Multicomponent Metal Oxides Using Nozzle Spray and Microwaves

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Materials. Ammonium heptamolybdate tetrahydrate (99%), ammonium metavanadate (99%), telluric acid (99%), vanadyl sulfate (99%), oxalic acid dehydrate (99%), palladium (II) nitrate hydrate (42% Pd), tellurium dioxide, and nitric acid were purchased from Aldrich Chemicals. Niobium ammonium oxalate (20% Nb) was purchased from H. C. Starck.

Characterization methods. Structural analyses were performed using powder X-ray diffraction (XRD) methods. XRD data were collected on a Scintag XDS 2000 diffractometer with Cu Kα radiation on the powder samples.

The morphologies of the prepared materials were studied using a Zeiss DSM 982 Gemini field emission scanning electron microscope (FESEM) with a Schottky emitter. The sample powder suspension in isopropanol was dispersed on AuPd-coated silicon chips mounted onto the stainless steel sample holders using silver conductive paint.

The isothermal N_2 adsorption/desorption experiments on the powder samples were conducted on a Micrometrics ASAP 2010 accelerated surface area system. Each sample was degassed at 120 °C for 3 hours. Nitrogen gas was used as an adsorbate at liquid nitrogen temperature. Surface areas were calculated by the Brunauer-Emmett-Teller (BET) method.

The elemental analyses were done at the Environmental Research Institute, Storrs, CT, using a Perkin-Elmer Model 140 Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES) instrument equipped with an autosampler.

Preparation. The use of the NMW and INM methods could be extended to the preparation of a large variety of catalysts usually prepared from liquid precursors. In the case of INM, the *in-situ* mixing of precursors should not form solids that could plug the inner tube of the nozzle spray. A preliminary experiment called "*in-situ* mixing test" was done to make different mixtures to prove the general applicability of the technique. Figure S1-a, shows the mixture of Mo-V-Te-Nb-Ox precursor salts. Figure S1-b and Figure S1-c show the mixture of the metal precursor solutions needed to make MnOx and CuO/ZnO/Al_2O_3 materials. The idea was to evaluate the experimental conditions such as speed of injection and metal salt concentrations appropriate for *in-situ* mixing prior to the spraying step.

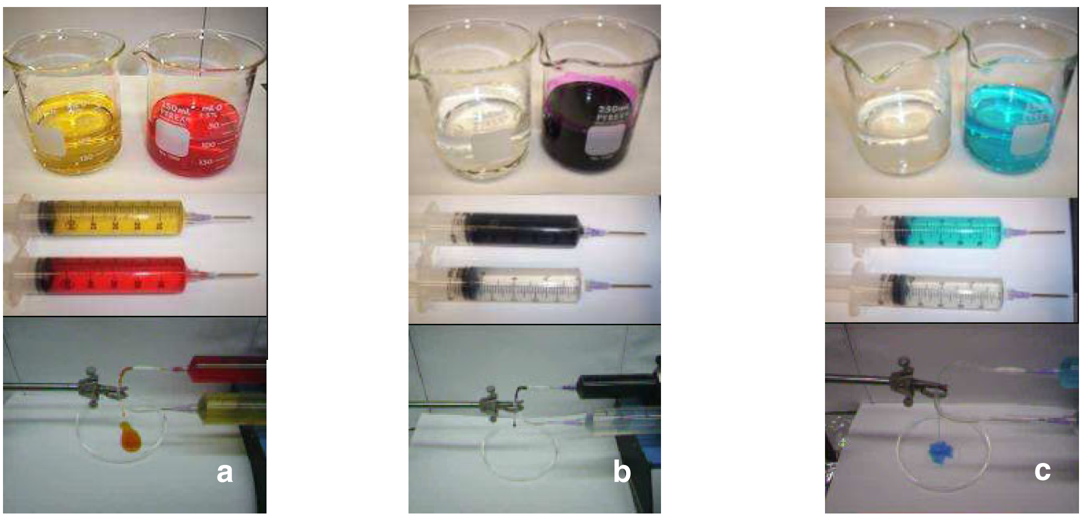

Figure S1. Metal precursors solutions mixed *in-situ.* (a) Mo-V-Te-Nb-Ox precursor (orange opaque gel) prepared from mixing solution A: 1 M Mo + 0.3 M V + 0.23 M Te (orange clear solution) and solution B: 0.17 M Nb + 0.155 M oxalic acid + 0.24 M HNO_3 + 0.01 M Pd (yellow clear solution), (b) MnOx precursor (brown gel) prepared from mixing solution A: 0.2 M $KMnO_4$ (dark purple solution) and solution B: 0.3 M glucose (clear transparent solution), and (c) $CuO/ZnO/Al_2O_3$ precursor (blue gel) prepared from mixing solution A: 0.2 M $Cu(NO_3)_2.2.5H_2O$ + 0.3 M $Zn(NO_3)_2.6H_2O$ + 0.05 M $Al(NO_3)_3.9H_2O$ (clear blue solution) and solution B: 1 M NaOH + 0.15 M Na_2CO_3. (clear transparent solution)

Figure S2. Metal precursors solutions mixed *in-situ*. (a) Mo-V-Te-Nb-Ox precursor (orange opaque gel), (b) MnOx precursor (brown gel), and (c) $CuO/ZnO/Al_2O_3$ precursor (blue gel). Note that the $CuO/ZnO/Al_2O_3$ precursor gel obtained is very viscous. Lower concentrations of salts would have to be used to avoid plugging of the nozzle spray.

The NMW and INM methods are quite versatile. Nano-size clays, layered double hydroxides, zeolites, metal oxides, spinels, porous metal oxides, and other materials can be produced using NMW and INM methods that have novel particle sizes and shapes.