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Ceramic Caride Synthesis: Process Optimization of Adsorption and Carburization

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Tungstate and molybdate loading were optimized using identical matrices of experimental conditions. To determine the optimal conditions for molybdate/tungstate adsorption, a statistical design of experiments was developed using DesignExpert 9 software (StatEase Inc.). A series of 30 individual experiments were carried out in order to mathematically model the effects of time, temperature, pH, and initial metal concentration on adsorption. Solution concentration was set at 1000 ppm, 10,500 ppm, and 20,000 ppm, temperature was held at 20°C, 40°C, and 60°C, reaction time was set at 1, 2, 4, and 6 h, and using hydrochloric acid (HCl). Solutions were prepared by adding sodium molybdate/tungstate to 100 mL of deionized water. Sodium chloride (NaCl) was also added to the solution so that a concentration of 0.2 M NaCl was achieved to promote adsorption [3]. A 2.5 g sample of activated carbon was added to each solution and the solutions were agitated to keep the activated carbon suspended in solution. Experiments carried out at 20°C were agitated on an orbital shaker table at 480 rpm, and the experiments carried out at elevated temperatures were agitated at a hot/shake plate at 480 rpm. Following adsorption, the activated carbon was removed from solution via vacuum filtration. The solution was analyzed via ICP-DES using an ICP Thermoscientific ICP 6000 instrument. The ICP-DES results were used to produce a response surface model of the adsorption data, and a mathematical model was generated using DesignExpert 9 to determine optimal adsorption parameters. Adsorption behavior of tungstate and molybdate anions was modeled using a base 10 log relationship. Molybdate adsorption was mathematically modeled using a base 10 log transform and a modified quadratic relationship. Molybdate adsorption onto the activated carbon matrix was expressed as the mass (g) of adsorbed Mo per gram of activated carbon. A three-dimensional response surface model of tungstate adsorption was developed using the experimental design matrix prepared using DesignExpert. Tungstate adsorption was modeled using an inverse square root transform and a modified quadratic relationship.

Optimal conditions for Mo and W adsorption were determined to occur at a pH of 2, a reaction time of 2 h, 20°C, and an initial Mo/W concentration of 18,000 ppm.

Tungstate carburation was synthesized at 950°C under a reducing gas mixture of methane, hydrogen, and carbon monoxide from a precursor material consisting of tungstate anions adsorbed onto an activated carbon matrix. Conversion to the desired tungsten carbide species, WC, was achieved with values exceeding 90%.

Experimental results were used to produce a preliminary model of carburization behavior for the W-loaded precursor. A 2-factorial analysis of the XRD data was used to produce this model, and a power series transform was used in conjunction with a linear relationship to produce the mathematical model for predicting optimal carburization conditions. From this model, it was determined that maximum conversion to tungsten carbide can be achieved through the use of temperatures approaching 950°C, a reaction time of 8 h, and an activated carbon content that does not exceed 20% of the total mass of the sample prior to carburization. Modelling carburization behavior indicated that activated carbon additions greater than 20% of the total sample mass reduced the conversion of the W-loaded precursor to tungsten carbide and may kinetically inhibit this process.

CONCLUSIONS

Tungsten carbide was synthesized at 950°C under a reducing gas mixture of methane, hydrogen, and carbon monoxide from a precursor material consisting of tungstate anions adsorbed onto an activated carbon matrix. Conversion to the desired tungsten carbide species, WC, was achieved with values exceeding 90%. Experimental results were used to produce a preliminary model of carburization behavior for the W-loaded precursor. From this model, it was determined that maximum conversion to tungsten carbide can be achieved through the use of temperatures approaching 950°C, a reaction time of 8 h, and an activated carbon content that does not exceed 20% of the total mass of the sample prior to carburization. Modelling carburization behavior indicated that activated carbon additions greater than 20% of the total sample mass reduced the conversion of the W-loaded precursor to tungsten carbide and may kinetically inhibit this process.

Molybdenum carbide, Mo2C, has been synthesized from the carburization of a Mo-loaded precursor at conversion rates approaching 90%. A preliminary mathematical model of the carburization process has been produced that indicates that this process is primarily thermodynamically driven with high degrees of conversion to Mo2C possible at 850°C. The model may indicate that shorter reaction times and less aggressive gas atmospheres may be more effective than originally expected though further refinement of the model is necessary in order to confirm this possibility. Micrographs of the products carburized at 850°C indicate that the carbide crystals produced by this process are submicron in size and may be suitable for both structural and catalytic applications.

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- **Goals After Graduation:** My work here at Montana Tech has allowed me to be a part of some very interesting research as well as giving me the opportunity to try my hand at teaching some courses. I would like to be able to take the skills I’ve gained here and continue teaching and doing cool science.