

5. CONCLUSIONS AND FUTURE DIRECTIONS

Iron/copper and iron/copper/silicon oxides produced by the simple precipitation pilot plant via mixing aqueous solutions of sodium carbonate and metal salts were acceptable precursors for iron-based F-T catalysts. The precipitation pilot plant runs were short due to the high rate of precipitate production and the relatively slow rate of the available filtration equipment. The best yields of precipitate resulted from presetting the rates of the aqueous feed solutions. Making feed rate changes during a run based on changes in the pH of the blended solution in the reactor didn't work. The feedback loop was too long for this to be successful for the short runs in this work. Although a start was made in determining how reproducible the properties of the precipitates were from run to run more such work would be useful.

A slurry autoclave pilot plant was used to evaluate the catalysts. Catalysts were made from the metal oxide precipitates by adding potassium and activating with either carbon monoxide + hydrogen or carbon monoxide alone, in either case the activation was done before the start of the run in the slurry autoclave reactor. In a few cases potassium was added to the reactor after the catalyst was activated and the evaluation run had begun. In this testing mode the target conversion was not achieved with any of the catalysts evaluated. However, this target was for performance in a slurry bubble reactor and good crossover data between the two reactors are not yet in hand. The target methane + ethane selectivity was achieved.

The best catalyst from the standpoint of high activity and low methane + ethane selectivity resulted from addition of potassium to an iron/copper oxide via potassium carbonate impregnation/calcination followed by activation with carbon monoxide + hydrogen and addition of potassium as a solution of potassium laurate.

Potassium laurate as a solid or in solution was an effective way to add potassium to the catalyst. In addition to allowing preparation of the superior catalyst above its method of use was such that the separate, time-consuming, potassium carbonate impregnation/calcination was eliminated.

There are many experiments that should be done with potassium laurate and other organo-potassium compounds. Foremost are experiments in which solid organo-potassium compounds are added at the beginning of a run (initially potassium laurate) followed by addition of a solution of the organo-potassium compounds (again, initially potassium laurate). Such experiments should have as objectives: determination whether the potassium carbonate impregnation/calcination (above) is required for the superior performance noted when potassium laurate is added during a run.

After completing additional work with potassium laurate, experiments should be performed to determine how other organo-potassium compounds effect the catalyst system. A few tentative experiments were performed during this contract with potassium benzoate and potassium nonylphenolate solutions. However, these were never added to the reactor as solids at the beginning of a run and other organo-potassium compounds were not evaluated. Addition at the beginning of a run would allow determination of whether or not the very small effects produced on the catalyst by potassium benzoate and potassium nonylphenolate are due to the salts being lost to the catalyst system through solubility in the product wax which is periodically removed from the reactor.

Although this is not the case with potassium laurate it could be with other organo-potassium compounds. On the other hand, if the small effect on catalyst performance due to these organo-potassium compounds results from them being unable to promote the iron other experiments are suggested. If this is the case, the anion along with the potassium ion plays a role at the catalyst active site. Many other organo-potassium compounds are available, some of which might be superior to potassium laurate. For instance, dicarboxylic acid salts and salts of acetylacetonate have not as yet been evaluated.

The special method of introduction of potassium during a run as a soluble organo-potassium salt should be extended to the addition of other salts such as those of copper and iron or mixtures thereof.

The most successful run was terminated because of operational problems before all of the planned potassium laurate solution was added. This run should be repeated to confirm that the very high activity is maintained at high levels of added potassium since that is one of the key findings of this work. Furthermore, more potassium laurate should then be added to determine whether the high activity persists at even higher potassium levels which might cause further reductions in the methane + ethane selectivity. Slightly more strenuous conditions might then allow target conversion to be attained at the target selectivity.

Finally, additional experiments should be done in which solutions of potassium laurate are added during the run. Initially the earlier very successful Run 49 (Figures 107 to 113) should be repeated but the addition of the potassium laurate at a different time (initially earlier) to determine whether even better results can be obtained. It is known that the catalyst undergoes changes during the

initial phases of a run, perhaps addition of potassium during this period will produce interesting results.