ALKALI/TX₂ CATALYSTS FOR CO/H₂ CONVERSION TO C₁-C₄ ALCOHOLS

Technical Progress Report for the Period

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ALKALI/TX₂ CATALYSTS FOR CO/H₂ CONVERSION TO C₁-C₄ ALCOHOLS

OBJECTIVE AND SCOPE OF WORK

The objective of this research is to investigate and develop novel catalysts for the conversion of coal-derived synthesis gas into C₁-C₄ alcohols by a highly selective process. Therefore, the variations of catalyst activity and selectivity for the synthesis of alcohols from H₂/CO ≤ 1 synthesis gas for a series of A/TX₂ compounds, where A is a surface alkali dopant, T is a transition metal, and X is a S, Se, or Te, will be determined. The alkali component A, which is essential for C-O and C-C bond forming reactions leading to alcohols, will be highly dispersed on the TX₂ surfaces by using chemical vapor deposition (CVD) and chemical complexation/anchoring (CCA) methods.

The target alcohol space time yields are of the order of 1 kg of alcohol product per kg of catalyst per hour and the target selectivity is > 90% to alcohols. Among the A/TX₂ compounds initially investigated, preference will be given to A = Cs, T = Group VB element (V, Nb, Ta), and X = S or Se. So delimited A/TX₂ catalysts possess the strongest base (Cs), and the CCA preparation method using crown ethers is expected not only to disperse the alkali ions atomically but also to enhance the basicity of their counterions, thus further increasing the specific rates of the base-catalyzed C-O and C-C bond forming reactions.

The benefit of this research rests in providing a scientific foundation and a database for catalytic processes leading to high octane oxygenated fuels such as ethanol/methanol/propanol mixtures, as well as to low olefins obtained by further catalytic dehydration of the C₂-C₄ alcohols. Since the source of carbon can be coal-derived synthesis gas, this research will make a contribution to the technology for high quality clean transportation fuels and for basic chemicals from coal.

ALKALI/TX₂CATALYSTS FOR CO/H₂ CONVERSION TO C₁-C₄ ALCOHOLS

SUMMARY OF TECHNICAL PROGRESS

Catalysts that have been prepared during this quarter include RuS₂, NbS₂, K/MoS₂, and K/Crown ether/MoS₂. The RuS₂ catalysts have been prepared from ethyl acetate solution and from methanol solution using Li₂S and RuCl₃ as the reactants. In both cases, contamination with Li and Cl have been observed. The preparation from methanol solution was found to produce the more stoichiometric RuS₂ after pretreatment in H₂S/H₂. These catalysts have been characterized by HR-ESCA and XRD.

Niobium sulfide has been prepared by two different methods, and the resultant samples are undergoing HR-ESCA, XRD, and elemental analyses. It was shown that preparation via the isopolyanion (Nb₆O₁₉⁸⁻) and (NH₄)₂S does not result in precipitation of NbS₂.

A new batch of MoS₂ was prepared and examined by XRD to verify the desired degree of crystallinity that corresponds to a surface area of 60-65 m²/g. MoS₂ samples were separately surface doped with KOH and KOH/dibenzo-18-crown-6 complex. Low doping levels of 2 mol% K were utilized in the attempt to increase the dispersion of the alkali promoter. Catalytic testing in the temperature sequence of 250, 275, and 250°C resulted in nearly identical alcohol and hydrocarbon synthesis activities and selectivities to be observed. Further characterization of the samples needs to be carried out in order to determine the fate of the crown ether after the catalytic testing.

TECHNICAL PROGRESS

Preparation and Characterization of RuS₂ Catalysts

Preparation of RuS₂ Catalysts Using Ethyl Acetate. The first preparation of a RuS₂ catalyst this quarter involved RuCl₃ and Li₂S reagents [1,2] and used ethyl acetate as the solvent. This sample was repeatedly washed with 15% acetic acid/ethyl acetate solution [3] in an attempt to remove Li and Cl contaminants. The resultant amorphous RuS₂ was subsequently pretreated in 14.8% H₂S in H₂. This was achieved by mixing 0.5-0.75 g of the RuS₂ with Pyrex beads and loading the mixture into a quartz tube (having stopcocks at both ends) under N₂. The gas flow at $\approx$ 1 ml/sec was changed to the H₂S/H₂ mixture, and after 15 min the sample was heated to 125°C. This temperature was chosen to drive off the ethyl acetate (B.P. = 76.5-77.3°C) and acetic acid (B.P. = 116-118°C) used in the preparation and clean-up procedures, and it was maintained for 30 min. The temperature was then increased to 500°C, which was maintained for 1 hr. The sample was then cooled to ambient temperature while maintaining the flowing gas mixture. The resultant crystalline RuS₂ was subsequently removed from the quartz tube in a N₂-filled glove box and sealed in glass tubes (for elemental analysis and X-ray powder diffraction (XRD) analysis) and in glass bottles (for catalytic testing and HR-ESCA analysis).

A 4.7 g portion of the amorphous RuS₂ was washed with dry methanol in order to further remove the Li and Cl contaminants (RuCl₃ is sparingly soluble in methanol and the solubility of LiCl is 42.36 g LiCl/100 ml methanol). The procedure was to initially stir the RuS₂ in 300 ml CH₃OH for 2 hr under N₂. After settling for 1 hr, the CH₃OH was decanted and a second 300 ml aliquot of CH₃OH was added to the solid and stirring was maintained for 19 hr. After decantation, the RuS₂ was filtered, washed with three

successive portions of CH_3OH , and dried under suction. The resultant solid was pretreated under 14.8% $\text{H}_2\text{S}/\text{H}_2$ at 500°C as described above.

Chemical analysis of the RuS_2 samples was carried out, and the results are presented in Table 1. The acetate and methanol washed samples are

TABLE 1. Chemical Analyses (Wt%) of the RuS_2 Catalysts.

Element	RuS_2 Amorphous	RuS_2 Acetate Washed	RuS_2 Methanol Washed	Theoretical
Ru	38.83	53.24	61.33	61.18
S	22.29	29.71	33.30	38.82
Li	0.95	1.29*	0.055	---
Cl	8.33	11.40*	<0.16	---

*These values were calculated from a mol ratio of the Ru:Li and Ru:Cl inferred from the ratios obtained for the amorphous sample.

crystalline samples that had been treated with $\text{H}_2\text{S}/\text{H}_2$. It is evident that the methanol washing procedure did result in removal of appreciable quantities of Li and Cl. The crystalline methanol washed sample corresponded to $\text{RuS}_{1.71}$.

XRD and HR-ESCA Examination of the Methanol Washed RuS_2 . The sample was pretreated in $\text{H}_2\text{S}/\text{H}_2$ after the methanol washing treatment, and the XRD pattern showed the sample of RuS_2 (designated as RuS_2 -340) to be very crystalline, as shown in Figure 1. The first large peak centered at $\approx 26^\circ$ is due to the Mylar film covering the sample to protect it from air exposure. As indicated in Figure 1, diffraction peaks corresponding to metallic crystalline ruthenium (Ru^0) are also present in the pattern.

A different portion of this catalyst was examined by HR-ESCA using the SEIKO-SCIENZA ESCA-300 at Lehigh University. The survey scan, shown in Figure 2, indicated that there was no detectable Cl and C (see Figure 3, where the Auger spectral region was used because the Ru 3d lines overlap

with the C 1s line) on the surface of the catalyst. However, significant peaks corresponding to the presence of oxygen were evident. Since RuO_2 was not detected by XRD, the HR-ESCA spectrum indicates that the RuS_2 sample readily picked up oxygen from the environment, although care was taken to prevent air exposure of the sample.

The spectral lines for Ru 3d and S 2p are shown in Figures 4 and 5. Quantitative determination of Ru and S can be achieved by using the peak areas and the Scofield cross-sections to arrive at approximate atomic surface ratios of the atoms comprising the surface. When the binding energy of the elements is in the range of 100 eV apart, a fairly quantitative measure of the surface composition can be obtained. The resultant calculations give a S/Ru atomic ratio of 2.033/1, which is very close to the theoretical ratio of 2/1 for RuS_2 . Although XRD showed the presence of bulk Ru^0 and elemental analysis indicated $\text{RuS}_{1.71}$, these HR-ESCA measurements demonstrate that the surface of the ruthenium sulfide is approximately stoichiometric RuS_2 after the described treatment with $\text{H}_2\text{S}/\text{H}_2$. This also indicates that the sulfiding treatment was not successful in sulfiding the bulk of the catalyst.

Preparation of RuS_2 Using Methanol as the Solvent. Comparison of the previous preparations indicated that using methanol as the solvent should minimize contamination of the sulfide by Li and Cl. Therefore, another preparation of the RuS_2 was carried out using methanol as the solvent and wash solution while eliminating the 15% acetic acid rinse. The technique employed was to slurry 2 g of RuCl_3 in ≈ 300 ml of methanol and stir for 1 hr. To this mixture was added a yellow clear solution containing 1.1 g Li_2S in ≈ 100 ml methanol, and stirring was maintained for 3.5 days. After settling for 4.5 h, the liquid was decanted and the amorphous RuS_2 was filtered and washed four times with 50 ml methanol. Portions of the dried

black RuS_2 were sealed in glass tubes under mild vacuum, while the remainder was pretreated in 14.8% $\text{H}_2\text{S}/\text{H}_2$ at 500°C as discussed previously. The later sample was also sealed in glass tubes after the sulfiding treatment.

Elemental analysis of the H_2S pretreated sample was carried out, and the results are shown in Table 2. This analysis yields composition

TABLE 2. Chemical Analysis (Wt%) of the RuS_2 Catalyst Prepared Using Methanol as the Solvent and then Pretreated with 14.8% $\text{H}_2\text{S}/\text{H}_2$ at 500°C .

Element	RuS_2	
	Methanol Prepared	Theoretical
Ru	57.65	61.18
S	38.29	38.82
Li	1.81	---
Cl	0.95	---

corresponding to $\text{RuS}_{2.094}$, which is much closer to the expected stoichiometry than was the ruthenium disulfide formed with ethyl acetate as the solvent. However, it is apparent that appreciable quantities of Li and Cl are still present as contaminants.

XRD analyses of the samples again demonstrated that the initial sample was completely amorphous while the $\text{H}_2\text{S}/\text{H}_2$ treated sample was not highly crystalline. The latter sample was appreciably less crystalline than the ethyl acetate solution preparation described earlier (see Figure 1). In the XRD pattern of the poorly crystalline sulfided catalyst prepared from the methanol solution, it was not evident that Ru^0 was present in addition to the expected RuS_2 . The phenomenon of Ru^0 being present in some samples and not present in others will be further investigated in the future.

Preparation of NbS_2 Catalysts

The objective of this research was to prepare NbS_2 in as close to a 1:2 stoichiometric ratio as possible. A study of the literature on this topic

indicated that NbS_2 tends not to form as an isolated compound but prefers to form in conjunction with other phases having the formula of $\text{Nb}_{1+x}\text{S}_2$. It was indicated that preparations carried out in a quartz tube at temperatures greater than 900°C become contaminated with NbO and Nb_5Si_3 . The presence of multiphase products and contaminants in existing methods of preparation, e.g. sintering of mixtures of Nb and S, has led to seeking a method of synthesizing pure NbS_2 by precipitation.

In the current research, commercial samples of " NbS_2 " were obtained, and it was found by elemental analysis that the stoichiometry of these samples was close to $\text{NbS}_{1.55}$. Pretreatment of a purchased sample with 2.04% H_2/N_2 caused loss of sulfur than resulted in a stoichiometry of $\text{NbS}_{1.46}$.

Preparations of niobium sulfide have now been carried out using NbCl_x ($x = 4$ or 5) and Li_2S with either tetrahydrofuran (THF) or methanol as the solvent. All of the preparations, with the exception of the isopolyanion preparation, were carried out using dry box techniques.

Preparation of NbS_2 from NbCl_4 and Li_2S in THF. A 24.99 g (65.98 mmol) portion of $\text{NbCl}_4 \cdot 2\text{THF}$ was weighed and placed in a 600 ml beaker, 240 ml THF was added, and the solution was continuously stirred while 6.35 g (131.96 mmol) Li_2S was added. The solution turned from yellow-orange to black and a mild effervescence was observed. A final 80 ml portion of THF was added, and the solution was stirred overnight in the N_2 -filled glove box at ambient temperature.

After stirring for 16 hr, the solid was allowed to settle for 45 min and the solution was vacuum filtered. The collected solid was washed with 12.25% acetic acid/THF solution (95 ml/680 ml). The dried black powder was pretreated in 15% $\text{H}_2\text{S}/\text{H}_2$ at 500°C for 1 hr, and then samples were sealed for subsequent XRD, HR-ESCA, and elemental analyses.

Preparation of NbS₂ from NbCl₅ and Li₂S in Methanol. To 50 ml of methanol was added 10.0 g NbCl₅ (some bubbling occurred), and the solution was added to 250 ml methanol containing approximately 5 g of Li₂S. The resultant solution turned dark brown (again some bubbling occurred) and was stirred for 24 hr in the N₂-filled dry box. After filtering, washing three times with 50 ml portions of methanol, and drying, the product was a yellow-brown solid.

Preparation of NbS₂ from the Isopolyanion Nb₆O₁₉⁸⁻ and (NH₄)₂S. The hexaniobium isopolyanion is a species that is stable in basic solution and can be used as a precursor to other Nb compounds [4]. A mixture of 1.11 g (4.15 mmol) Nb₂O₅ and 2.72 g (8.30 mmol) Cs₂CO₃ was placed in a 10 ml fused quartz crucible and heated at 1000°C for 10 hr. The resulting melt was an off-white gel. The crucible and the melt were placed in a beaker containing 90 ml of NaOH solution at pH 8.33. The melt dissolved to produce the isopolyanion solution.

About 5 ml of the isopolyanion solution was added to 50 ml NaOH solution (pH = 11), to which a 25 ml aliquot of (NH₄)₂S was added. A transparent yellow solution resulted. Lowering the pH with acetic acid destabilized the isopolyanion. However, white Nb₂O₅ was precipitated before the expected NbS₂ was formed. Therefore, the preparation of NbS₂ using this method is not feasible because of non-overlapping pH ranges where the isopolyanion and the sulfiding agent are destabilized.

Preparation and Testing of K/MoS₂ and Surface-Held K/Crown Ether Complexes

We have previously shown that large doping levels, e.g. 10-20 wt%, of alkali salts are needed to produce optimum alcohol synthesis alkali/MoS₂ catalysts [5,6]. It is desirable to develop a method of dispersing the alkali over the surface of the MoS₂ so that much smaller quantities of

alkali can be utilized. Crown ethers contain oxygen or sulfur ligands that complex alkali in the ring or as sandwich complexes, as discussed below. The purpose of examining the use of crown ethers to disperse alkali on the surface of the MoS_2 is to increase the dispersion of the alkali, as well as to increase the basicity of the anion. The expected result in the increase in basicity is that more low molecular weight alcohols ($\text{C}_1\text{-C}_4$) may be formed from $\text{H}_2/\text{CO} \approx 1$ synthesis gas by using crown ethers in this capacity.

The properties of crown ethers make them ideal for catalysis studies because they have relatively high melting points. When the crown ethers are complexed with alkali, the melting point are even higher. In addition, the crowns are very stable molecules and are not very soluble in water or alcohols.

Dibenzo-18-crown-6, where the 18 indicates the number of C and O atoms in the crown and the 6 indicates the number of O atoms, has a crown size that will accomodate a potassium ion in a 1:1 complex, a schematic of which is shown in Figure 6. This particular crown was used in this experimental study mainly because of this 1:1 complexing ability. It would be unfavorable to use a crown of such a size that is would not accomodate the alkali ion in the center of the crown because sandwich complexes may then be formed that contain two crowns to one metal ion [7]. Other stoichiometries may also result, e.g. in the case of CsSCN complexation with dibenzo-18-crown-6 a 3:2 complex is formed.

Preparation of MoS_2 . Using the procedure that was previously described for the preparation of MoS_2 [8], a new sample of MoS_2 was prepared. In a hood, 107 ml of 22% aqueous $(\text{NH}_4)_2\text{S}$ was added to 15 g of $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$ contained in a 800 ml beaker. The solution turned from a dark yellow to a dark red-orange color. The solution was continuously stirred while the temperature was increased to 60°C . To this solution was added 100 ml of

glacial acetic acid diluted to 400 ml with distilled water. Discontinuing heating, the solution was stirred for 3.5 hr. During this time, the solution turned black and a brown precipitate formed. The solution was filtered and the product was rinsed with distilled water. After drying in an oven at 110°C overnight, the product consisted of MoS_3 .

Portions of the MoS_3 were calcined by loading ≈ 2 g into a quartz tube with Pyrex beads. While maintaining a flow of N_2 at a rate of 1 ml/sec, the quartz tube was heated in a furnace at 450°C for 2 hr. After cooling, the black product consisted of MoS_2 . XRD indicated that the sample was poorly crystalline and should have a surface area of $\approx 60\text{-}65 \text{ m}^2/\text{g}$.

Preparation of Dibenzo-18-Crown-6/KOH. About 2 g of dibenzo-18-crown-6 and 0.35 g KOH were dissolved in 140 ml of methanol, and the solution was heated while being continuously stirred. When the volume of solution remaining was ≈ 20 ml, 50 ml of toluene was added and heating was continued. The solution was evaporated to 50 ml and then cooled to room temperature. Large white plate-like crystals formed upon cooling, and the solution was placed in an ice bath for 20 min to ensure complete crystallization. The product was filtered and washed with three 20 ml aliquots of cold toluene, and then the product was placed in a vacuum desiccator to dry.

Doping of MoS_2 with the K/Crown Ether Complex and with Only KOH. A solution of the KOH/dibenzo-18-crown-6 complex was prepared by adding 0.1041 g (6.504×10^{-4} mol) of the complex to a 100 ml volumetric flask and diluting to the mark with a mixture of 90% toluene/10% methanol. Warm water was then run over the flask to aid in the dissolution of the solute. A separate KOH solution was prepared by adding 0.0415 g (7.411×10^{-4} mol) KOH to a 100 ml volumetric flask and diluting with distilled water. Six 1 g portions of MoS_2 were placed into six 50 ml beakers, and three levels of

doping were carried out using 2 ml, 10 ml, and 20 ml of the two solutions. In a hood, the beakers were gently heated and a N_2 flow was passed over the surfaces of the samples. The samples were evaporated to dryness and then bottled in air for characterization studies and catalytic testing.

Testing of the Catalysts for Alcohol Synthesis Activity. The catalysts that were tested contained the highest dopant levels in both of the series (0.130 mmol K/g of catalyst = 0.021 mol K/mol MoS_2 $\approx$ 2 mol% K). The powdered catalysts (0.75 g) were tested with $H_2/CO = 1$ synthesis gas at 82.6 atm (8.4 MPa) with a gas flow rate of GHSV = 7786 l/kg catal/hr (5.84 l/hr = 97.33 ml/min). The reaction temperature was varied in the range of 250-275°C.

Over both the KOH/MoS_2 and the $K/Crown/MoS_2$ catalysts, hydrocarbons and alcohols were observed as products as shown in Figures 7 and 8. After the reaction sequence of increasing the temperature to 275°C and then decreasing it to 250°C, the yield of hydrocarbons decreased relative to the initial yields. Comparisons of the alcohol yields shown in Figure 9 and the selectivities shown in Figure 10 demonstrate that the $K/Crown/MoS_2$ catalyst was initially less active and less selective toward alcohols than the corresponding KOH/MoS_2 catalyst. However, upon tested at the elevated temperature of 275°C and returning to 250°C, the activities and selectivities of the catalysts were nearly identical. This might indicate a repositioning of the K dopant on the MoS_2 surface at the elevated temperature and perhaps decomposition of the crown ether. The final state of the crown ether has not yet been determined.

Figures 7-9 show that the product yields are rather low and Figures 7-10 demonstrate that methane is the principal products formed under the reaction conditions employed. These observations are not unexpected since the dopant levels of the catalysts were intentionally very low compared with the optimum dopant levels of $CsOCH/MoS_2$ and $KOCH/MoS_2$ catalysts [5,6,9].

It is to be noted that the previously prepared and tested near-optimum 10.36 wt% KOOCH/MoS₂ catalyst corresponds to 18.03 mol% K and 0.22 mol K/mol MoS₂, while the present catalysts contain 2 mol% K. It has also been previously shown [5,6,9] that the undoped MoS₂ catalyst produced hydrocarbons, principally methane, under the same reactions conditions as utilized here and that the yield and selectivity toward hydrocarbons decreases as the alkali content of the catalysts is increased.

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FIGURE 1

FIGURE 2

RUS2-340, sample RUS2-300 & -310 cleaned up with CH3OH.
 RUS2.340 SURVEY LEHIGH 1990-07-20 18: 48

Pass energy	300 eV
Slit width	1.1 mm
Energy step	0.60 eV
Det. height	90 %
Lens mode	transmission
X-ray power	7.4 kW
Rec. time	01:34:32

Binding Energy [eV]

FIGURE 3

RUS2-340, sample RUS2-300 & -310 cleaned up with CH3OH.

RUS2.342 C-AUGER LEHIGH 1990-07-22 11:15

FIGURE 4

RUS2-340, sample RUS2-300 & -310 cleaned up with CH3OH.
 RUS340.001 RU3D LEHIGH 1990-07-22 13:07

FIGURE 5

RUS2-340, sample RUS2-300 & -310 cleaned up with CH3OH.

RUS340.002 S2P LEHIGH 1990-07-22 13:58

FIGURE 6

Dibenzo-18-Crown-6

Dibenzo-18-Crown-6
Complexed with KOH

FIGURE 7

Products from KOH/MoS₂

yield - G/KG cat/hr

FIGURE 8

Products from Crown/KOH

FIGURE 9

Yield comparison KOH VS Crown KOH

—●— KOH alcohol
 —○— KOH alkanes
 —■— Crown/KOH alcohol
 —○— Crown/KOH alkanes

yield = G/KG cat/hr

FIGURE 10

Selectivity Comparison
KOH vs Crown/KOH

END

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