

Investigation of Bio-ethanol Steam Reforming over Cobalt-based Catalysts

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PDP-6

Overview

Timeline

- ❖ Start Date - May 1, 2005
- ❖ End Date - April 31, 2009
- ❖ 40% Complete

Budget

- ❖ Total project funding
 - \$1,145,625 (DOE)
 - \$299,715 (OSU cost share)
- ❖ Funding received in FY05
 - \$100,000 (DOE)
 - \$10,458 (OSU Cost share)
- ❖ Funding received in FY06
 - \$185,000 (DOE)
 - \$147,987 (OSU cost share)
- ❖ Funding received in FY07 (to date)
 - 290,473 (DOE)
 - \$25,037 (OSU)

Barriers

- ❖ A. Fuel Processor Capital Costs
- ❖ C. Operation and Maintenance
- ❖ D. Feedstock Issues

Partners

- ❖ NexTech Materials, Ltd. - Catalyst manufacturing scale-up
- ❖ PNNL - Economic analysis and feasibility considerations, deactivation studies



Objectives and Prior Work Summary

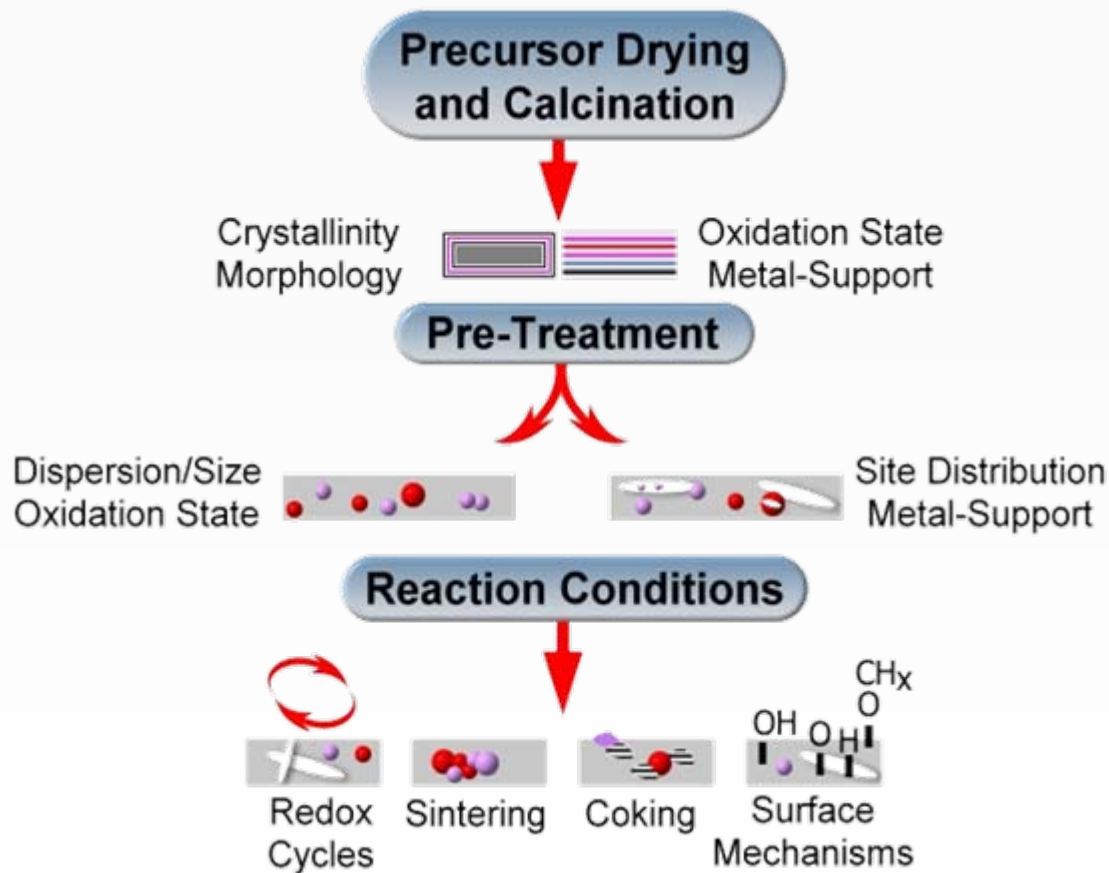


- ❖ **Overall:** To acquire a fundamental understanding of the reaction networks and active sites in bio-ethanol steam reforming over Co-based catalysts that would lead to
 - Development of a precious metal-free catalytic system which would enable
 - Low operation temperature (350-550°C)
 - High EtOH conversion
 - High selectivity and yield of hydrogen
 - Minimal byproducts such as acetaldehyde, methane, ethylene, and acetone
 - Understanding of the catalyst deactivation and regeneration mechanisms
 - Low cost for commercialization.
- ❖ **June 2005-Sep 2006**
 - Launching the project, setting up the experimental systems and establishing the experimental protocols
 - Investigating the effect of synthesis parameters on catalyst performance
 - Testing activity of the initial catalyst system.
- ❖ **FY06:**
 - Understanding the competing reaction networks
 - Identifying active sites during reaction
 - Optimizing catalyst pretreatment parameters



Approach

Supported Co-catalysts



❖ Preparation

- IWI, SG
- Precursor
- Precursor solution
- Promoter
- Support
- Cobalt loading

❖ Calcination

- Temperature

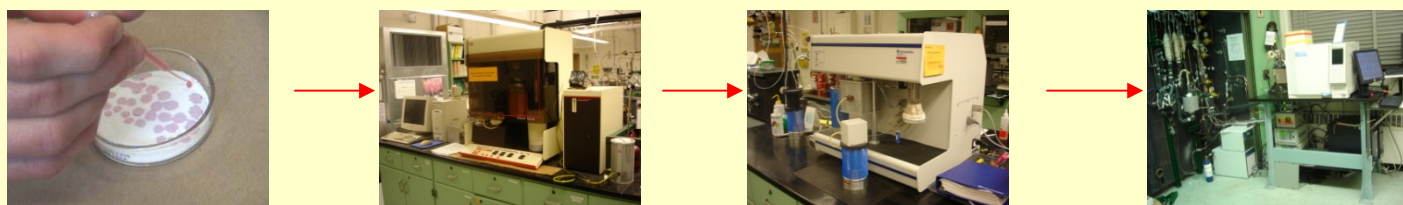
❖ Reduction

- Temperature
- Time

❖ Reaction

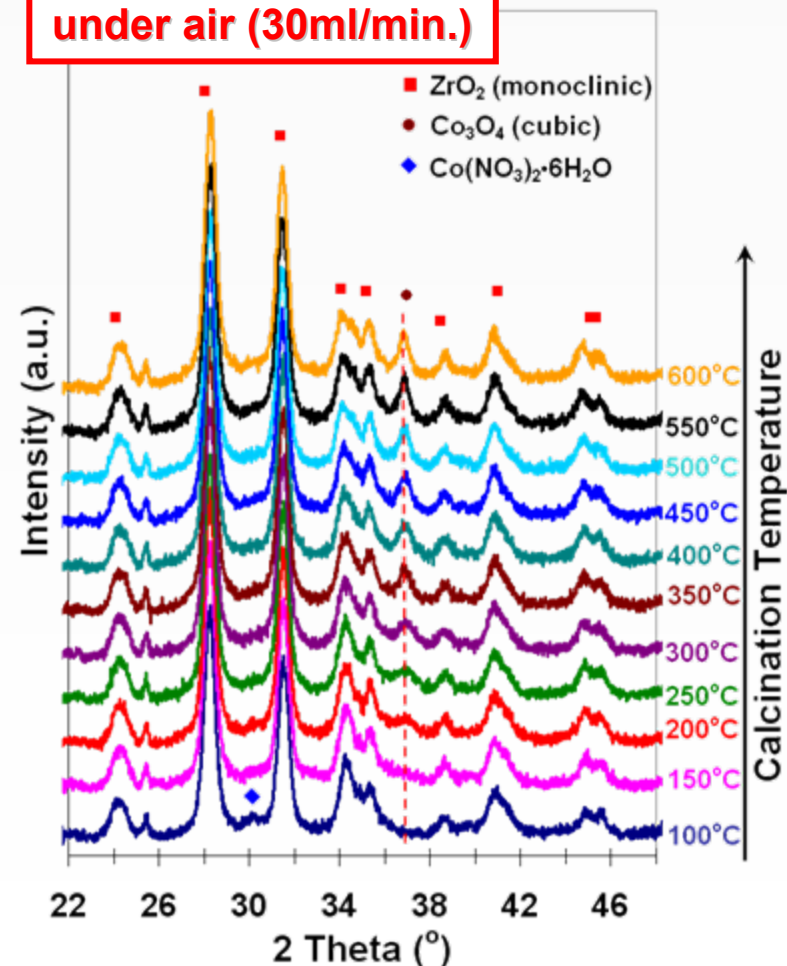
- GHSV
- EtOH:Water ratio
- Temperature
- Oxygen addition
- Deactivation

❖ Deactivation/Regeneration



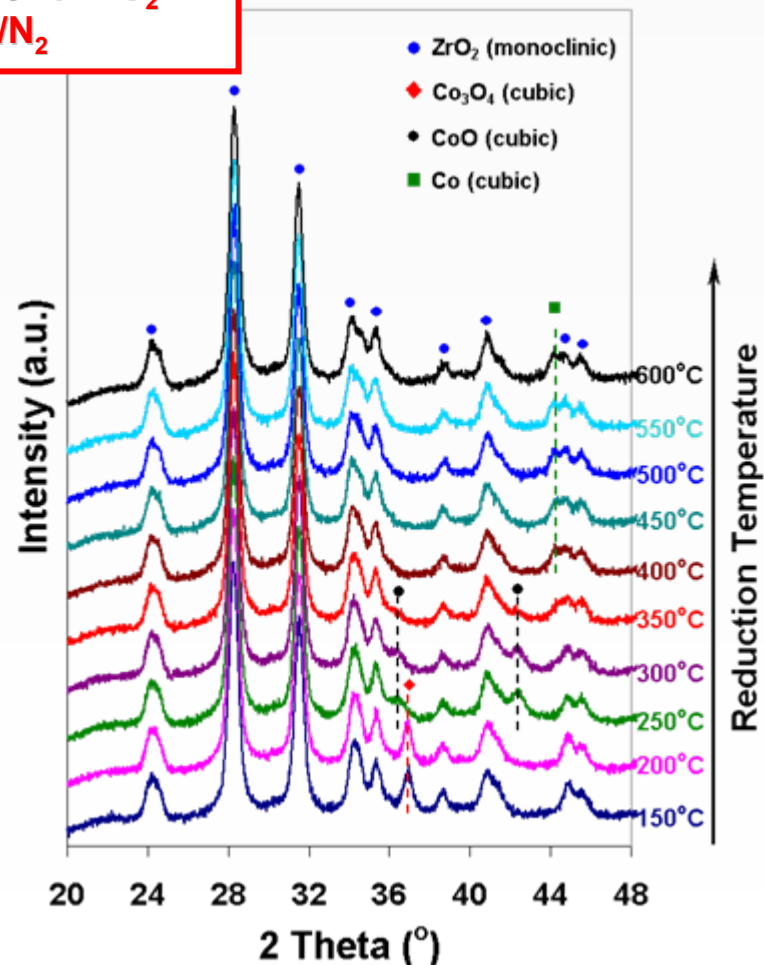
Catalyst evolution: From precursor to active phase

10 wt% Co/ZrO₂ (IWI)
under air (30ml/min.)



The decomposition of the crystalline cobalt precursor and formation of Co₃O₄ during calcination process have been observed.

10 wt%Co/ZrO₂
5% H₂/N₂



Two step reduction, i.e., Co₃O₄→CoO, and CoO→Co, has been confirmed.

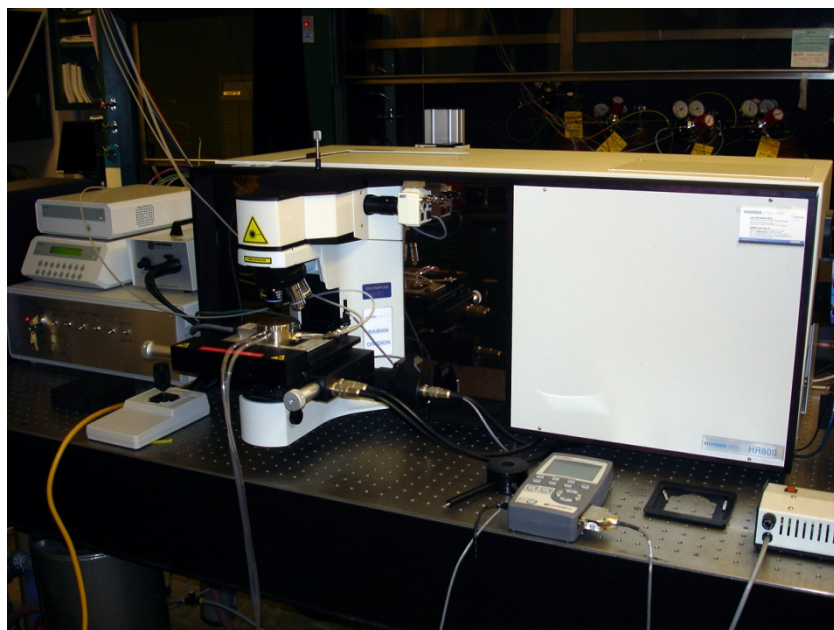
Further Characterization of the Calcination step

LABRAM HR-800 High Resolution Raman Microscope

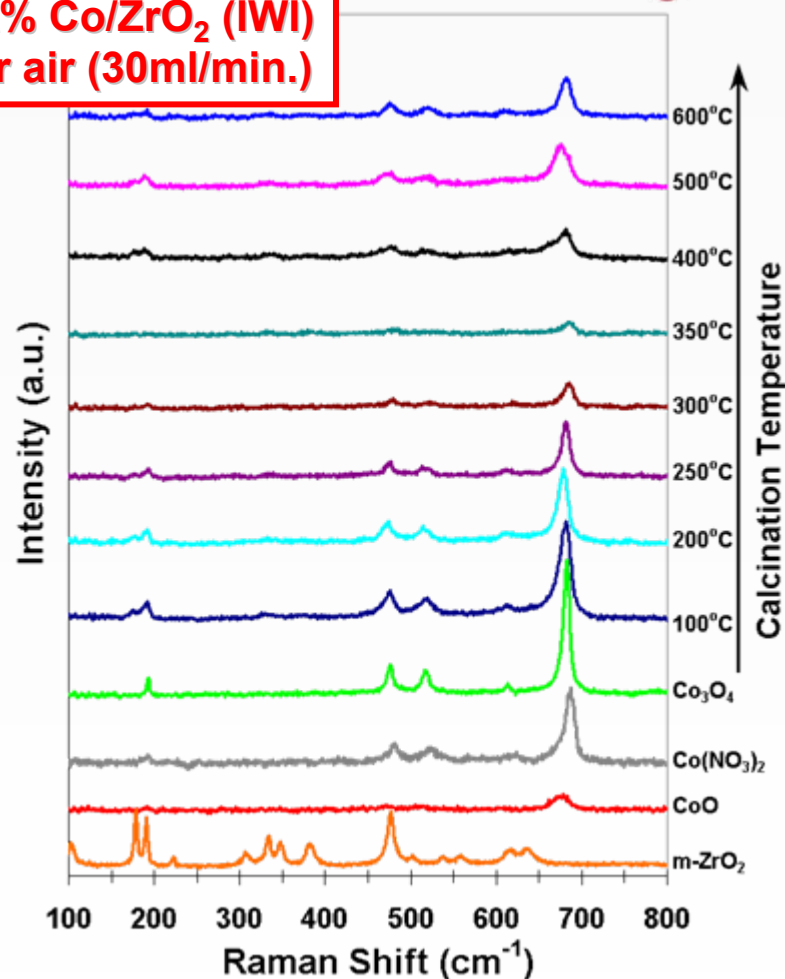
OLYMPUS BX41 microscope

In the current study:

- ❖ Sample is placed in Operando Cell;
- ❖ 50X magnification;
- ❖ Argon ion green laser (514.5nm);
- ❖ 3mW.



10 wt% Co/ZrO₂ (IWI)
under air (30ml/min.)



CoO is an intermediate in calcination of the catalyst precursor before the Co₃O₄ phase forms.

Further Characterization of the Reduction step

❖ Sample:

➤ 10%Co/ZrO₂

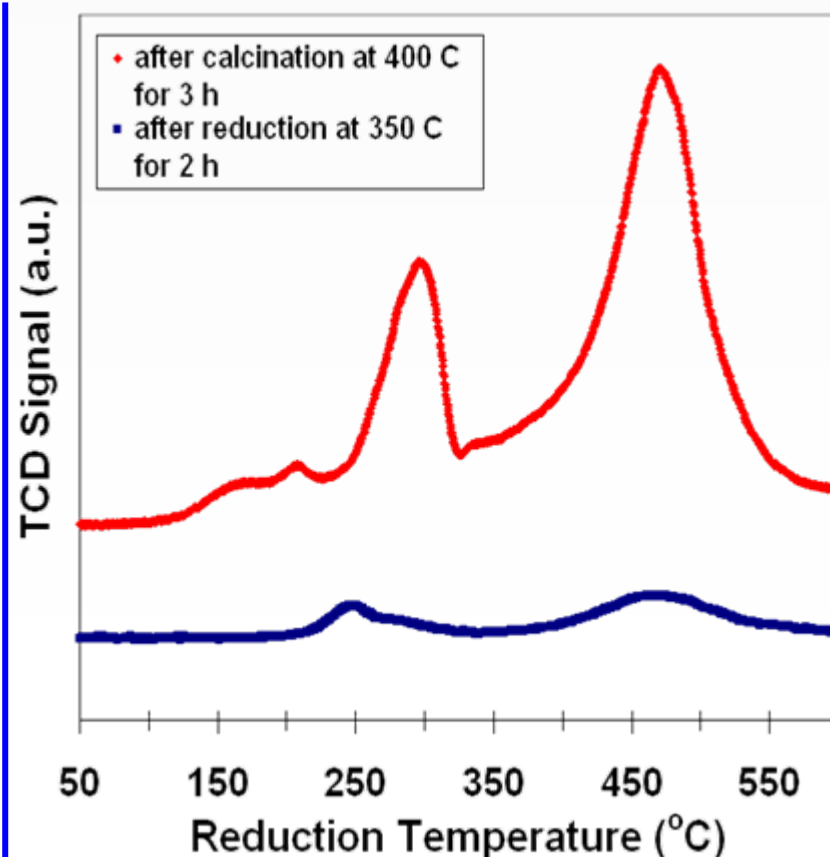
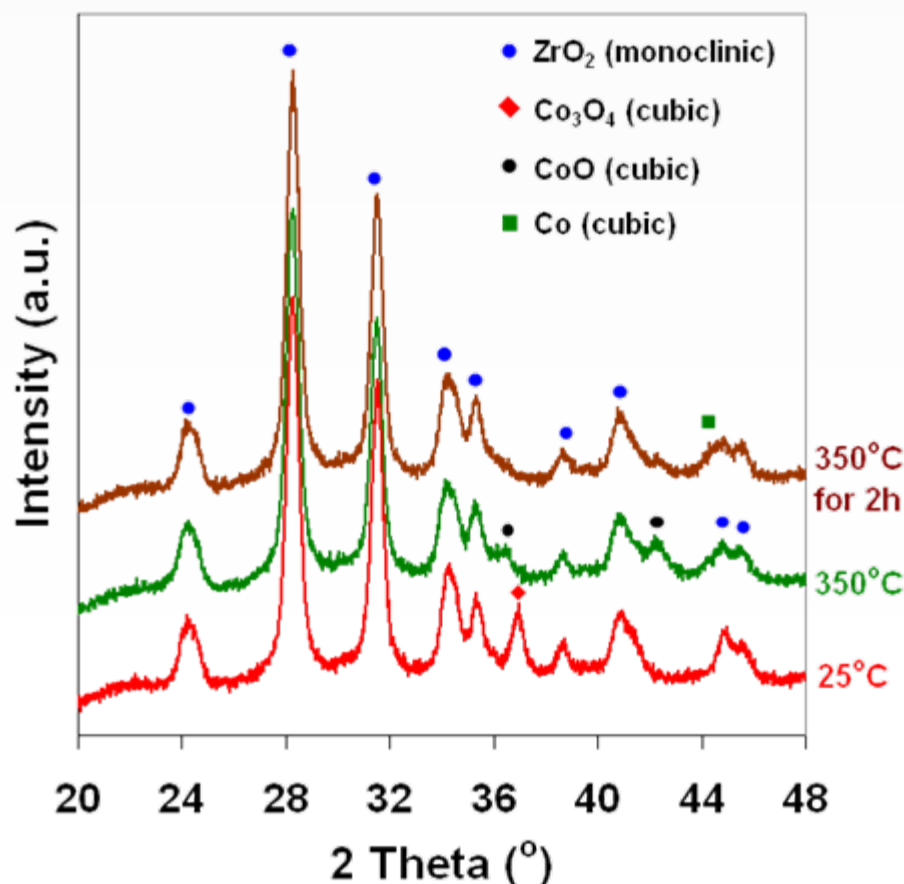
❖ Calcination:

➤ 400°C for 3h;

❖ Reduction:

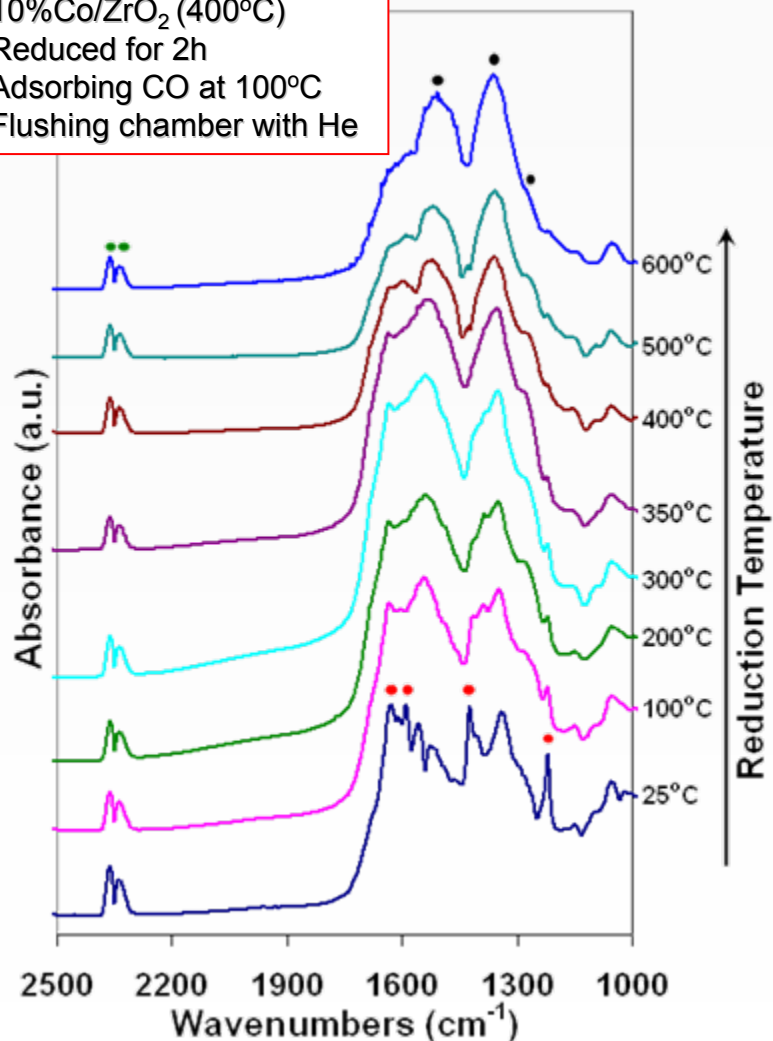
➤ 350°C for 2h under 5%H₂/N₂;

Most of the cobalt oxide has been reduced to metal Co after described activation treatment



In-Situ DRIFTS - CO Chemisorption

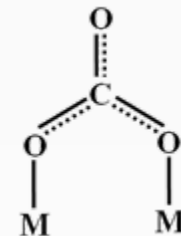
10%Co/ZrO₂ (400°C)
Reduced for 2h
Adsorbing CO at 100°C
Flushing chamber with He



Carbonate formation on the surface correlates well with the metallic surface area.

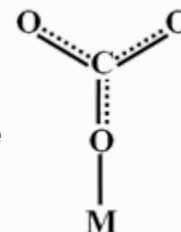
● Bidentate carbonate [1,2]

1631, 1591,
1425, 1220 cm⁻¹



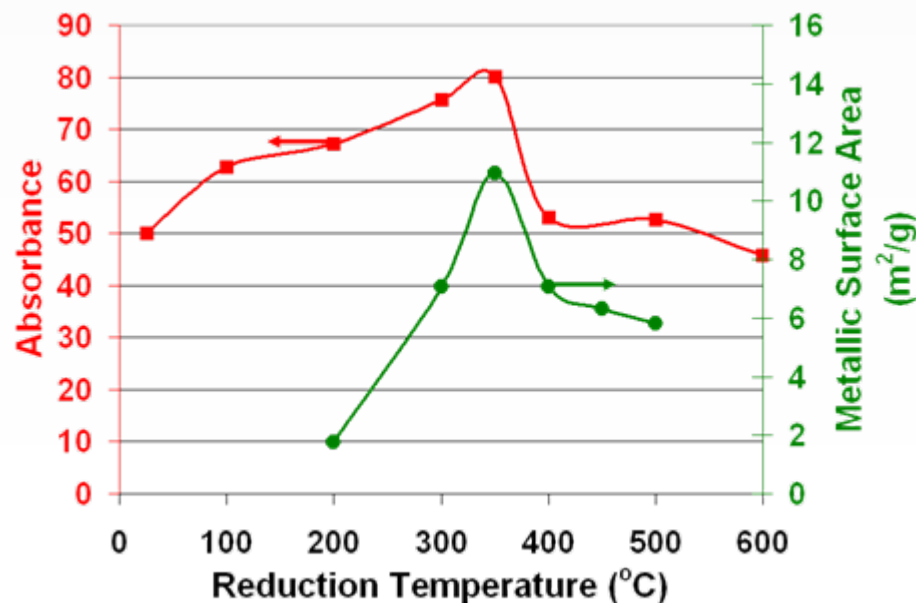
● Adsorbed CO₂

2368, 2343 cm⁻¹
O=C=O stretching



● Monodentate carbonate

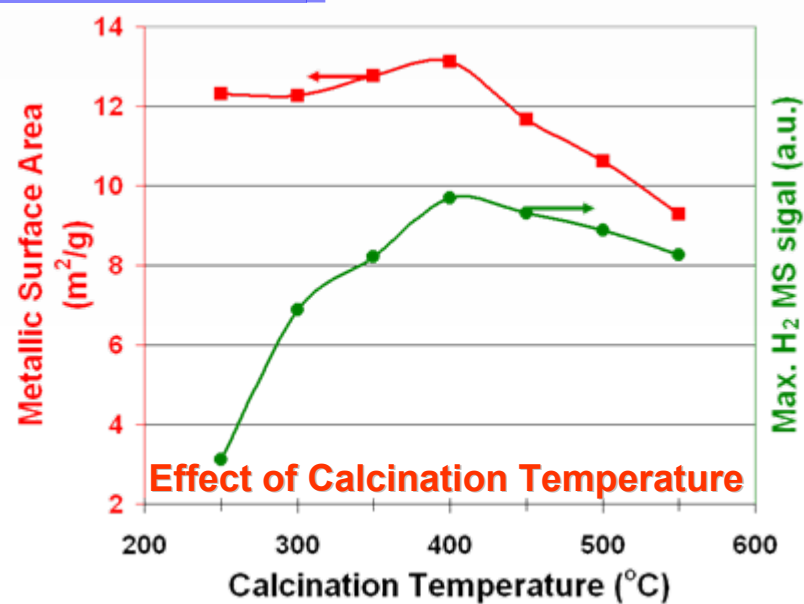
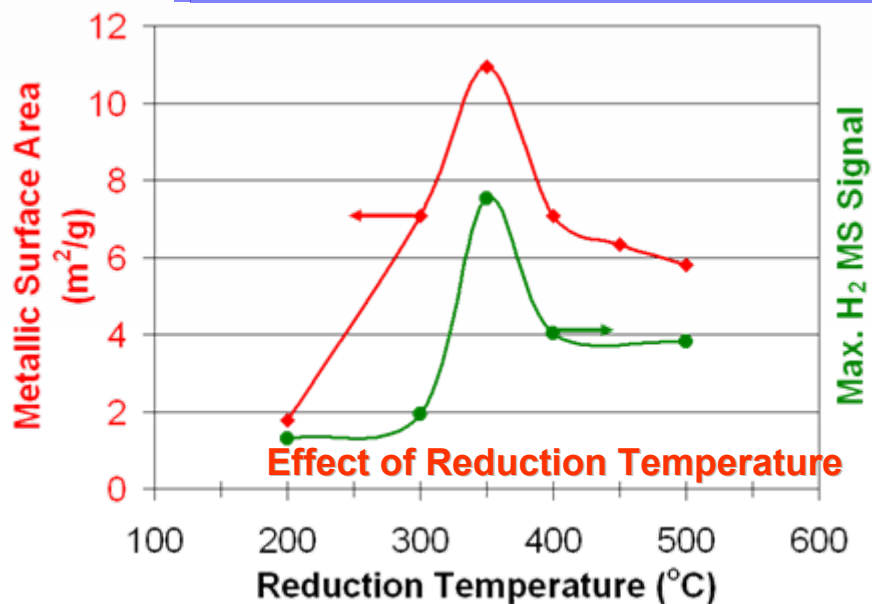
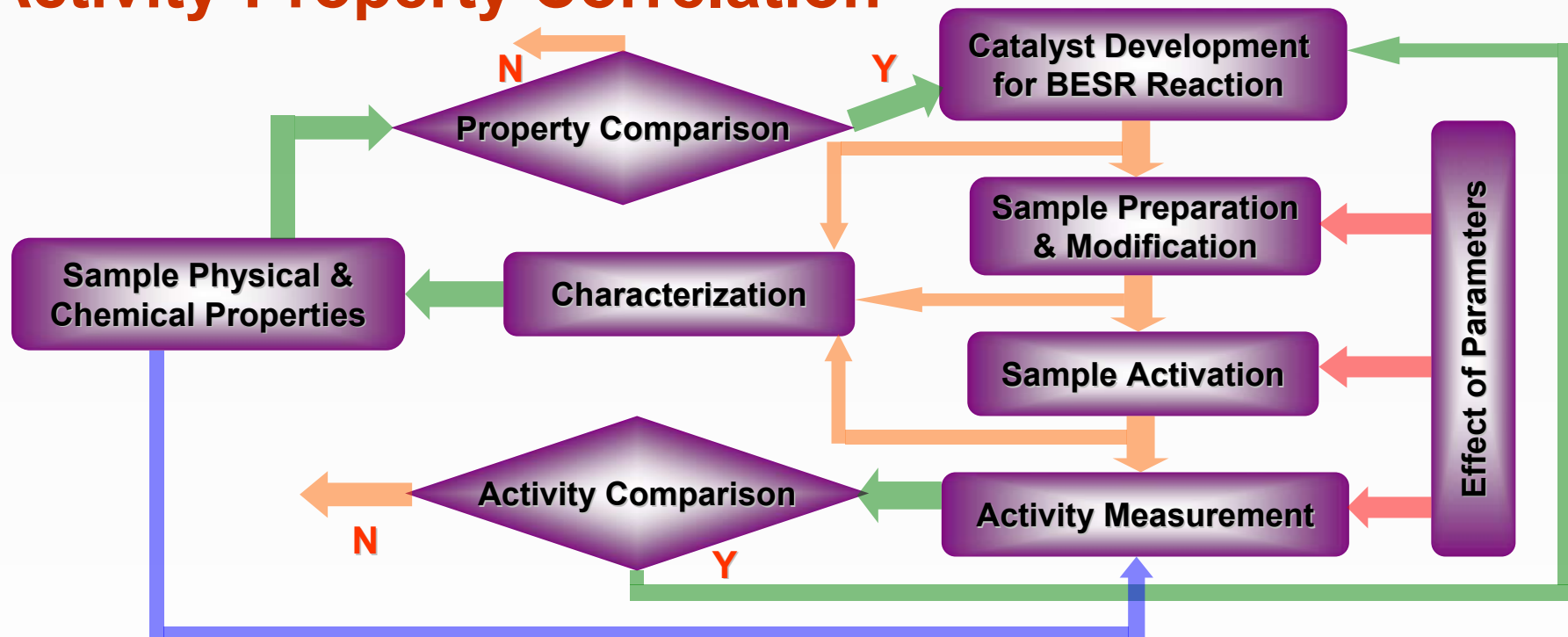
1530, 1373 cm⁻¹



[1] Mattos, L.V., Noronha, F.B. J. Catal. 233(2005) 453-463;

[2] Mattos, L.V., Noronha, F.B. J. Power Sources 152 (2005) 50-59; 8

Activity-Property Correlation

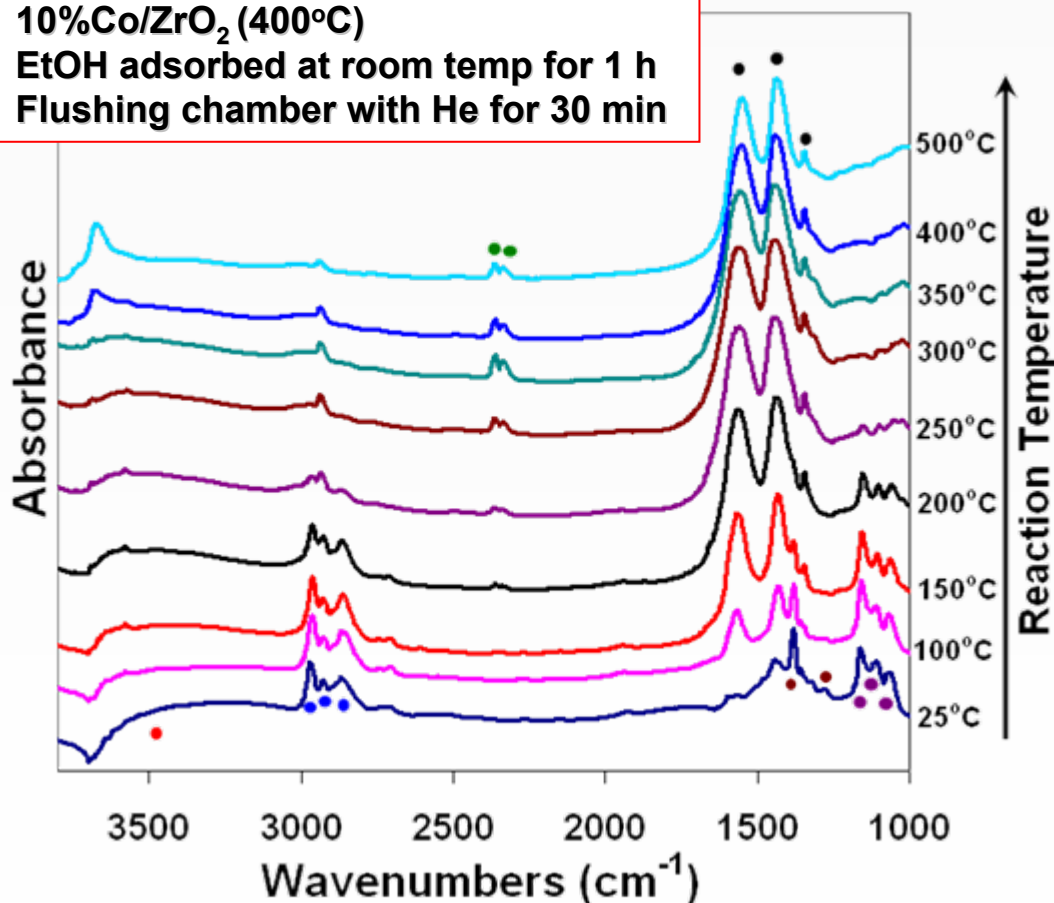


Reaction Network and Intermediates: *In-Situ* DRIFTS-Ethanol TPD

10%Co/ZrO₂ (400°C)

EtOH adsorbed at room temp for 1 h

Flushing chamber with He for 30 min



- Ethanol molecules are adsorbed onto the surface at room temp
- Interaction with OH groups
- Ethoxy, acetate and carbonate intermediates
- CO (characteristic peaks: 2200~2000cm⁻¹) is not observed

● M-OH

3650~3150cm⁻¹, O-H stretching

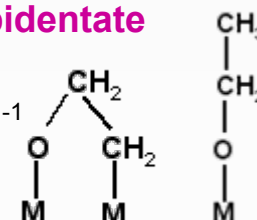
● CH₃- or CH₃CH₂-

2970, 2928, 2867cm⁻¹:C-H stretching

● Monodentate and bidentate ethoxy species [1]

1161, 1110, 1066cm⁻¹

CCO stretching



● Acetates [2]

CH₃COO

1552, 1441, 1346cm⁻¹

● Adsorbed CO₂

2361, 2338cm⁻¹

O=C=O stretching

● Molecularly adsorbed ethanol [3]

1383cm⁻¹: C-H bending

1294cm⁻¹: OH bending

[1] Erdöhelyi, A., et al. Catal. Today, 116 (2006) 367.

[2] Guil, J.M., et al. J. Phys. Chem. B, 109 (2005) 10813

[3] Raskó, J., et al. Appl. Catal. A: Gen., 299 (2006) 202

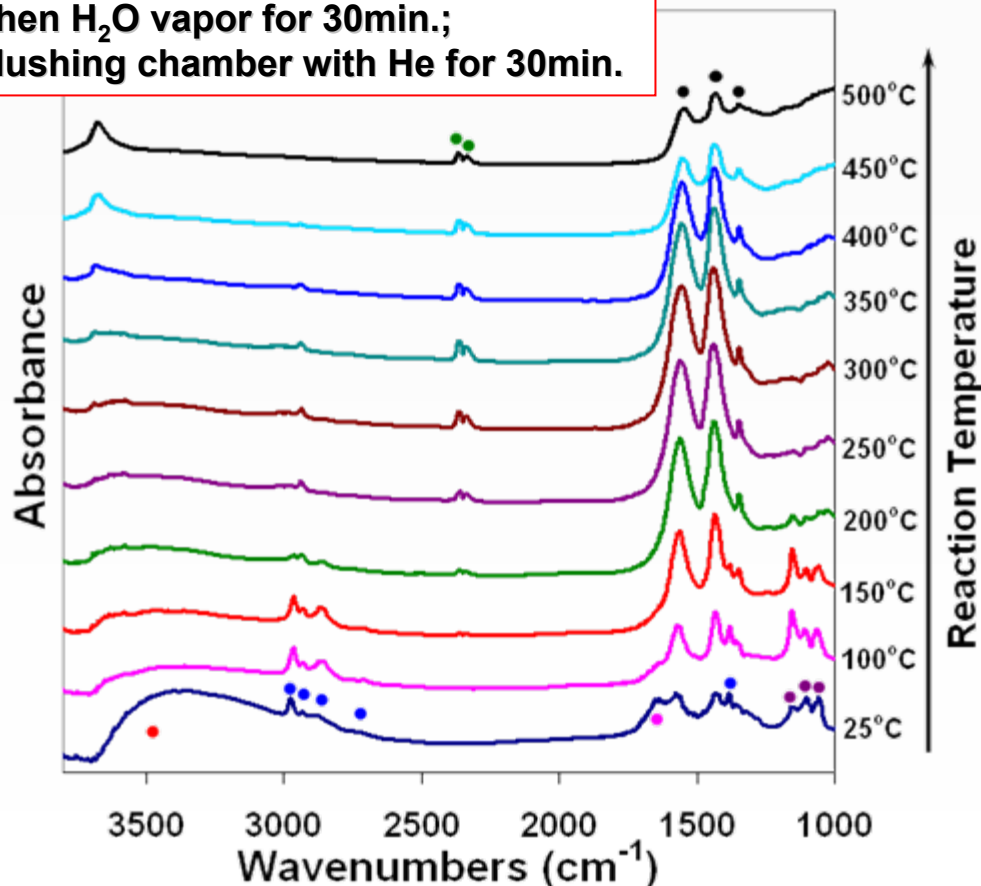
Reaction Network and Intermediates: *In-Situ* DRIFTS - EtOH+H₂O Reaction

10%Co/ZrO₂ (400°C)

Introducing EtOH vapor for 1h;

Then H₂O vapor for 30min.;

Flushing chamber with He for 30min.



● M-OH

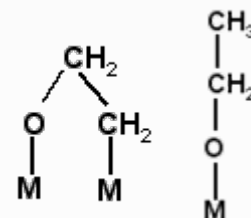
3650~3150cm⁻¹, O-H stretching

● CH₃- or CH₃CH₂-

2962, 2927, 2865 cm⁻¹: C-H stretching
1385cm⁻¹: CH₃- bending

● Monodentate and bidentate ethoxide

1169, 1106, 1063cm⁻¹
CCO stretching



● Acetates

CH₃COO
1569, 1429, 1348cm⁻¹

● Adsorbed CO₂

2361, 2336cm⁻¹: O=C=O stretching

● Molecularly adsorbed H₂O

1654cm⁻¹

- Water and ethanol molecules are adsorbed onto the surface at room temp
- Interaction with OH groups
- Water facilitates formation of ethoxy and acetate surface intermediates at lower temp.,
- CO (characteristic peaks: 2200~2000cm⁻¹) is not observed

Reaction Network and Intermediates:

❖ Sample:

➤ 10%Co/ZrO₂;

❖ Calcination:

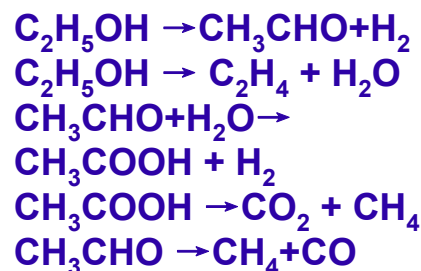
➤ 400°C for 3h;

❖ Reduction:

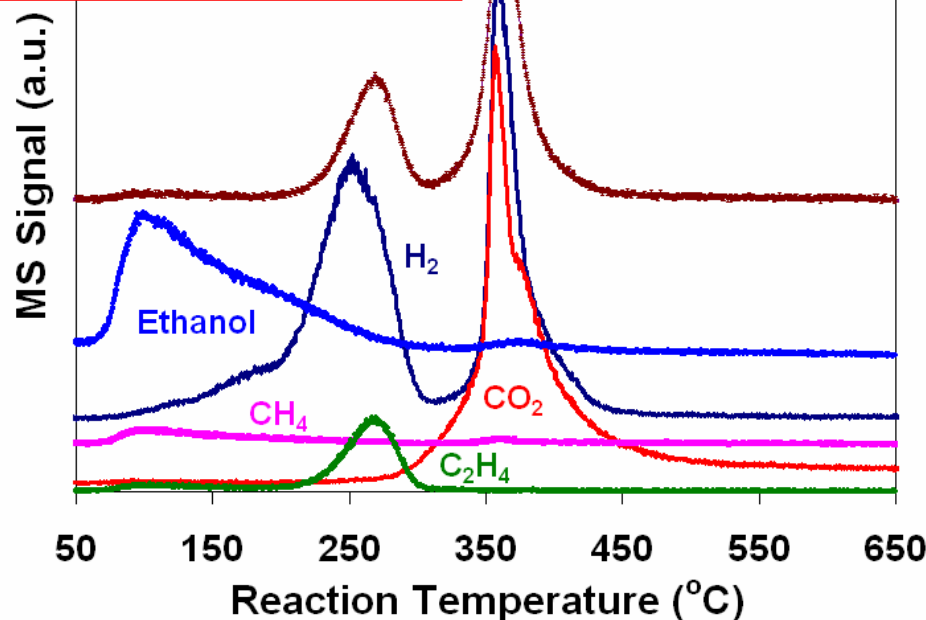
➤ 350°C for 2h;

❖ Before reaction:

➤ Flowing EtOH for 1h;



EtOH TPD



❖ Sample:

➤ 10%Co/ZrO₂;

❖ Calcination:

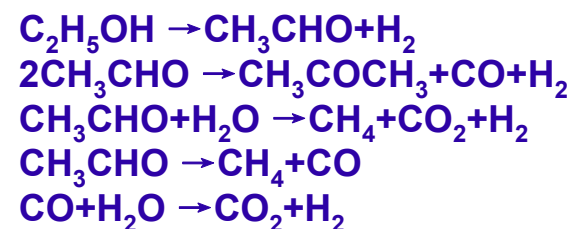
➤ 400°C for 3h;

❖ Reduction:

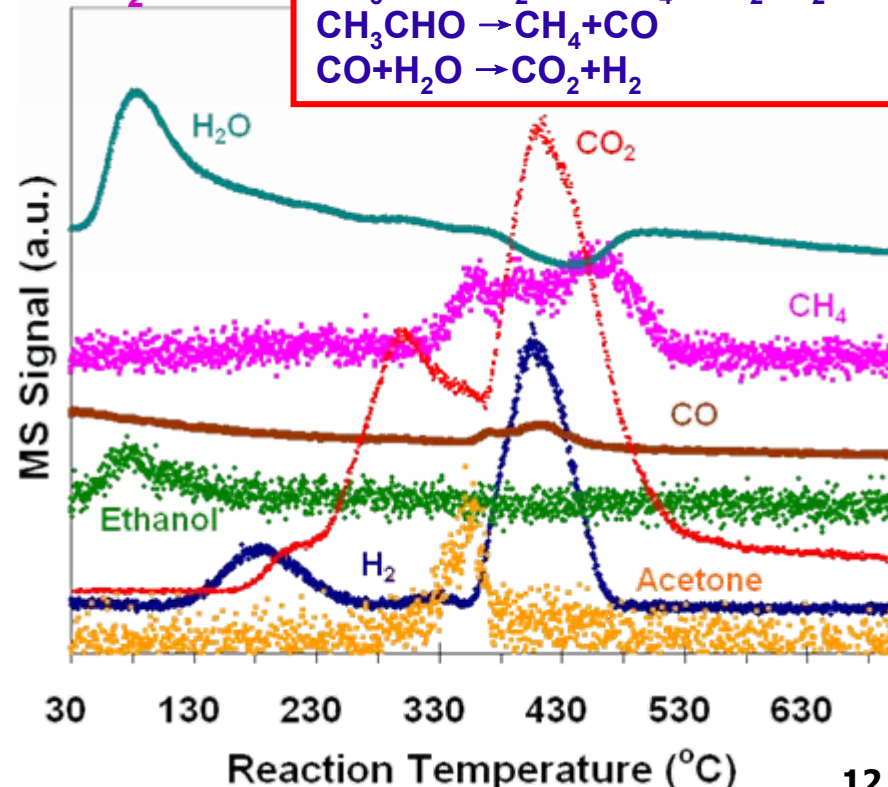
➤ 350°C for 2h;

❖ Before reaction:

➤ Flowing mixture of EtOH:H₂O(1:10) for 1h



EtOH+H₂O TPD

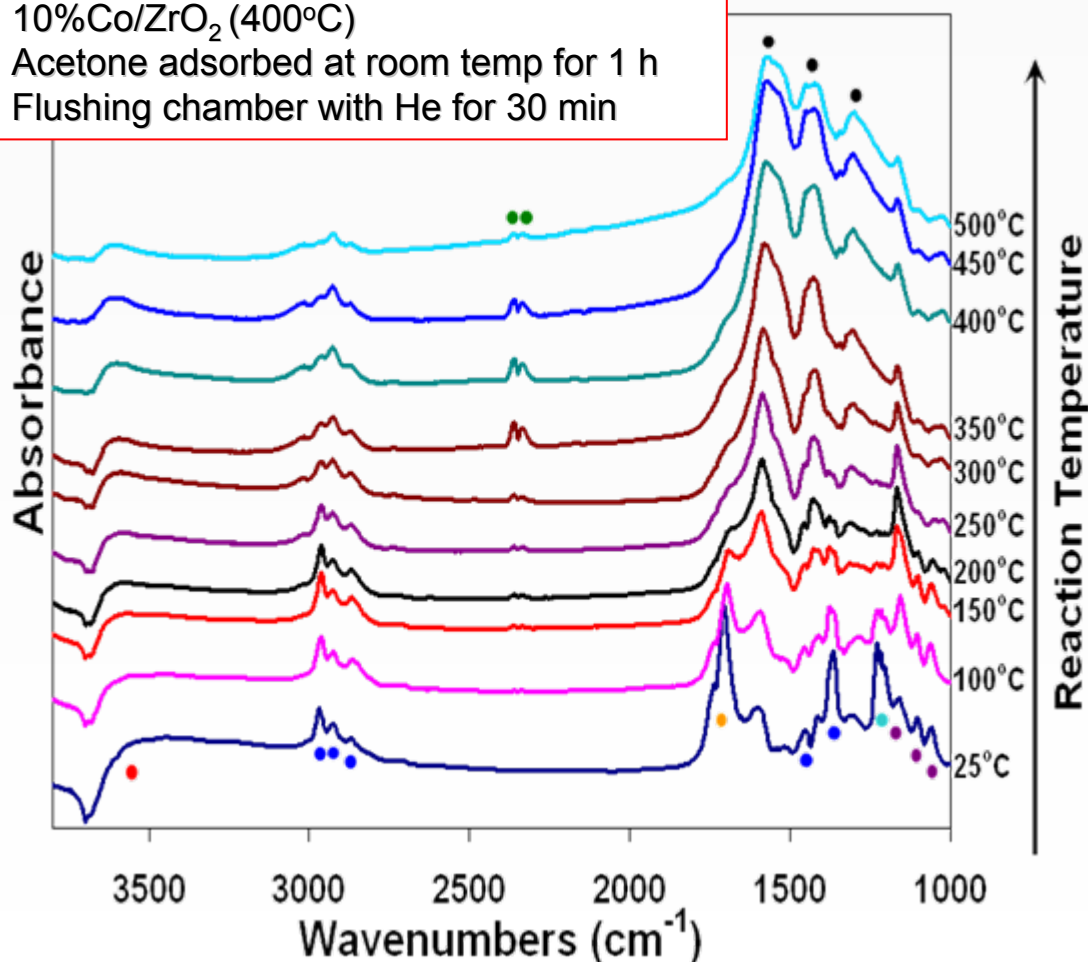


Reaction Network and Intermediates: *In-Situ* DRIFTS-Acetone TPD

10%Co/ZrO₂ (400°C)

Acetone adsorbed at room temp for 1 h

Flushing chamber with He for 30 min



● M-OH

3650~3150cm⁻¹, O-H stretching

● CH₃-

2963, 2927, 2865cm⁻¹:C-H stretching

1456, 1371cm⁻¹: CH₃- bending

● Adsorbed CO₂

2361, 2336cm⁻¹

O=C=O stretching

● Acetates

1580, 1440, 1310cm⁻¹

● Monodentate and bidentate acetoxide

1170cm⁻¹: C-C stretching

1107, 1066cm⁻¹: C-O stretching

● O=C- carbonyl group:

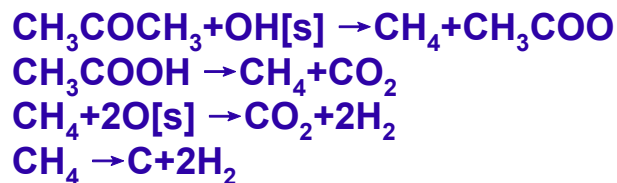
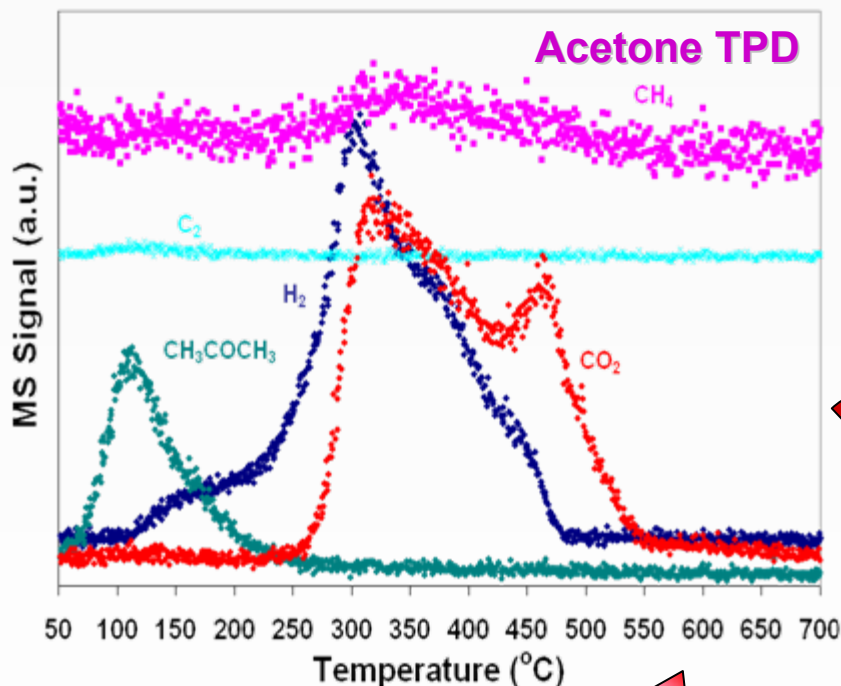
1745cm⁻¹

● C-C-C group stretching:

1232cm⁻¹

- Acetone molecules are adsorbed onto the surface at room temp.
- Evidence of C-C-C and C=O
- The cracking of acetone at lower temperatures
- Acetate species form at higher temperatures compared to ethanol TPD

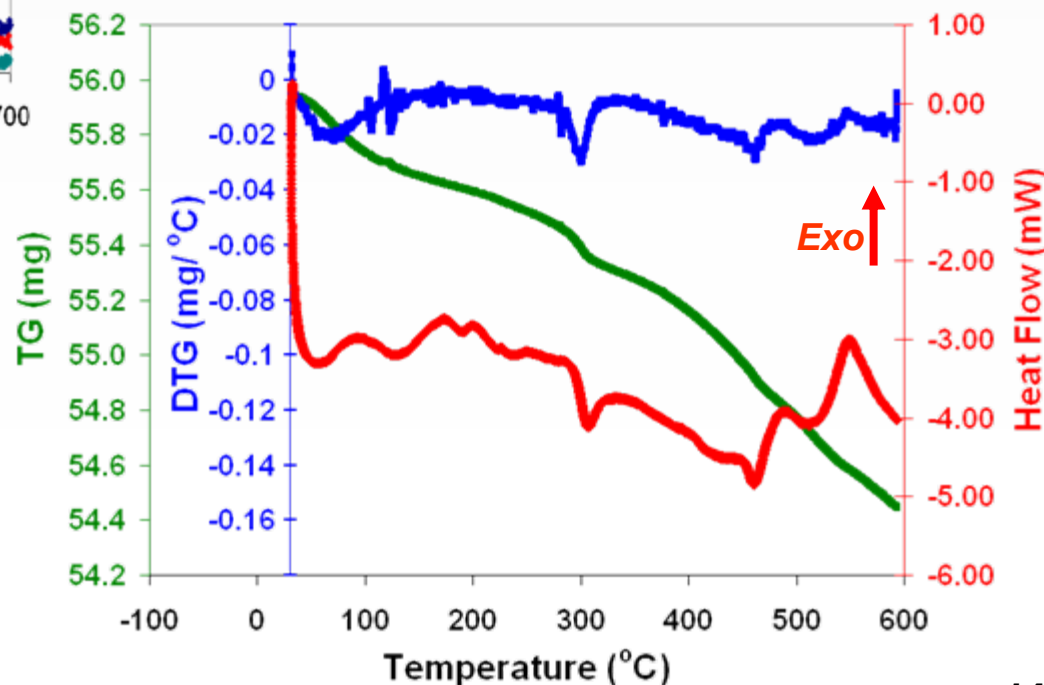
Reaction Network and Intermediates: Acetone TPD (MS-TGA-DSC)



10 wt% Co/ZrO₂

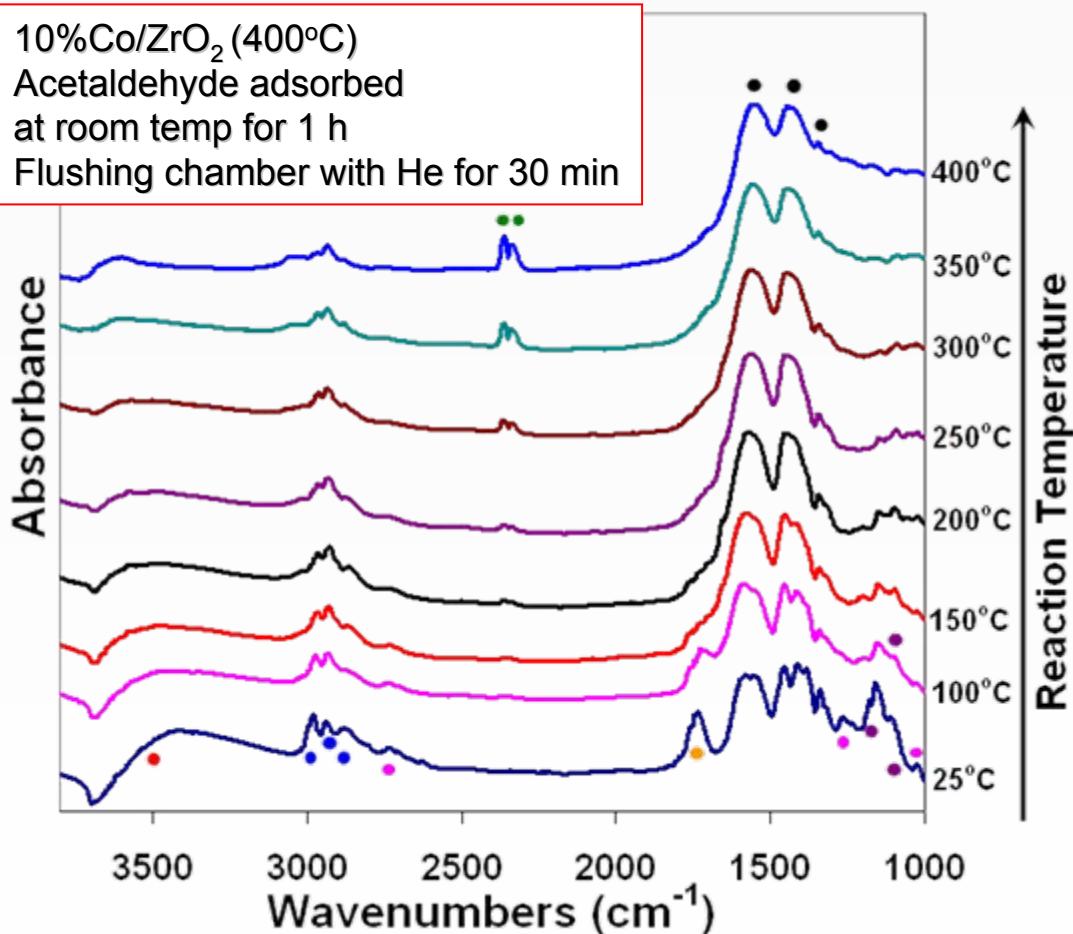


- ❖ **Sample:**
10%Co/ZrO₂
- ❖ **Pretreatment:**
400°C for 30min.;
Reduced at 350°C for 2h under 5%H₂/He;
400°C for 1h under He to remove moisture
- ❖ **Adsorption:**
Acetone vapor was generated by flowing He at room temperature;
- ❖ **Desorption:**
Under He at ramping rate of 5°C/min



Reaction Network and Intermediates: In-Situ DRIFTS-Acetaldehyde TPD

10%Co/ZrO₂ (400°C)
Acetaldehyde adsorbed
at room temp for 1 h
Flushing chamber with He for 30 min



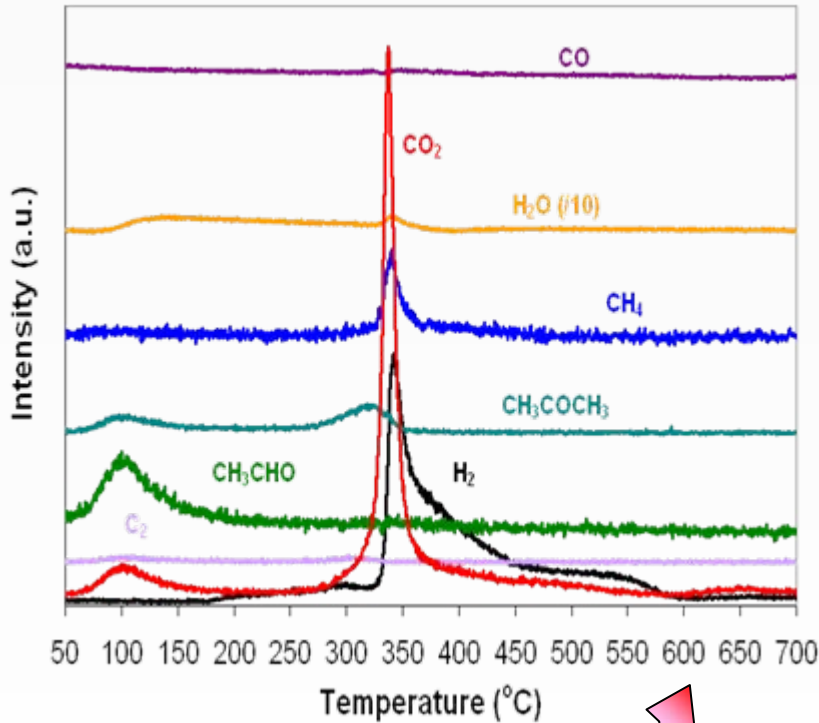
- **M-OH**
3650~3150cm⁻¹: O-H stretching
- **CH₃- or CH₃CH₂-**
2980, 2940, 2889cm⁻¹:
C-H stretching
- **Adsorbed CO₂**
2366, 2355cm⁻¹:
O=C=O stretching
- **Acetates**
1556, 1442, 1360cm⁻¹
- **Adsorbed acetaldehyde** [1,2]
2736, 1267, 1025cm⁻¹
- **Monodentate acetate**
1180, 1161, 1100cm⁻¹
CCO stretching
- **Molecularly adsorbed acetaldehyde**
1745cm⁻¹: O=C- stretching

- Acetaldehyde molecules are adsorbed onto the surface room temp.;
- Part of the adsorbed acetaldehyde is reduced to ethoxide
- Part of the acetaldehyde is oxidized to acetate at room temperature;
- Acetaldehyde is the surface intermediate during ethanol TPD

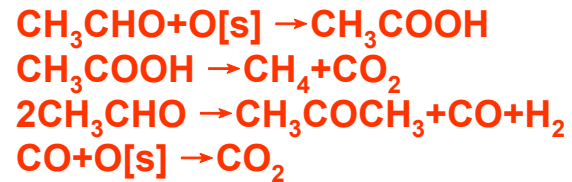
[1] Carlo Resini, et al. React. Kinet. Catal. Lett., 90 (2007) 117-126

[2] J. Llorca, et al. J. Catal., 227(2004) 556-560

Reaction Network and Intermediates: Acetaldehyde TPD (MS-TGA-DSC)



10 wt% Co/ZrO₂



❖ Pretreatment:

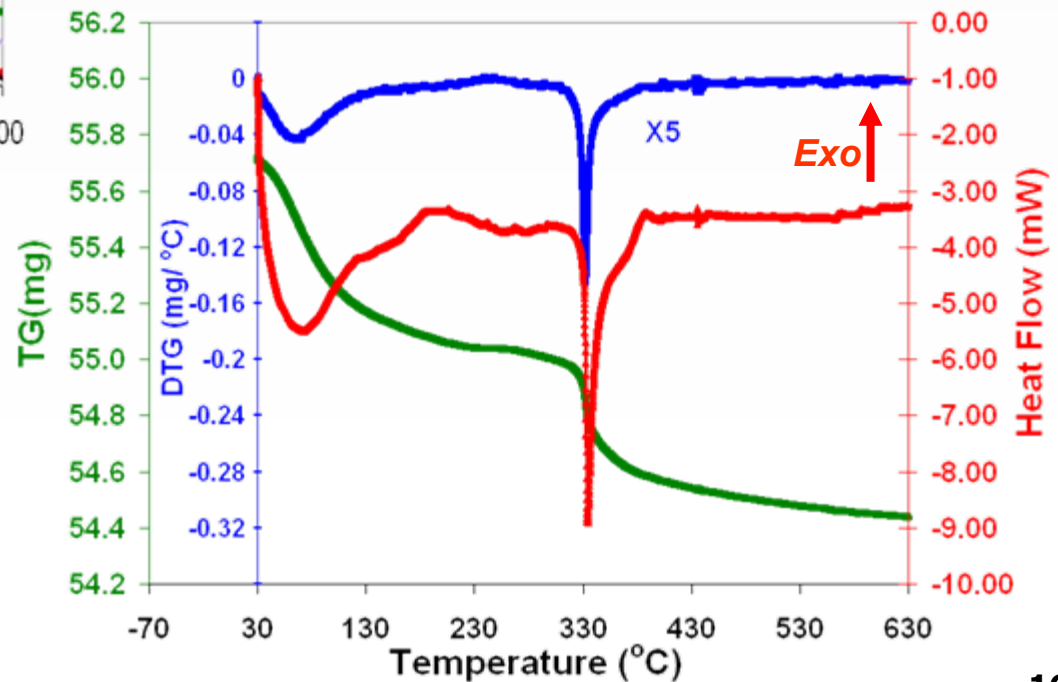
400°C for 3h;
Reduced at 350°C for 2h
Under 5% H₂/He;

❖ Adsorption:

Use He as the carrier gas to generate
CH₃CHO vapor at room temperature for 1h;
Flow He to purge the line;

❖ Desorption:

Under He (10°C/min.)

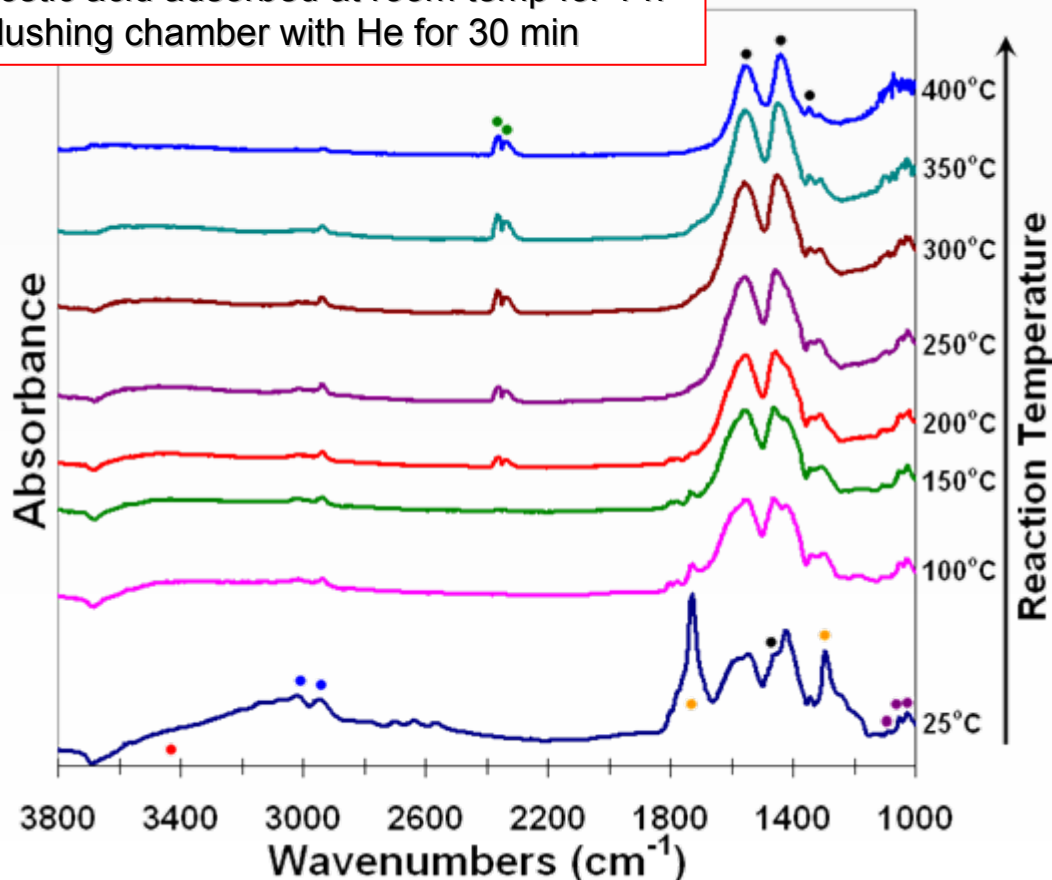


Reaction Network and Intermediates: In-Situ DRIFTS-Acetic Acid TPD

10%Co/ZrO₂ (400°C)

Acetic acid adsorbed at room temp for 1 h

Flushing chamber with He for 30 min



The assignment of surface acetate species is confirmed

- **M-OH**
3650~3150cm⁻¹, O-H stretching
- **CH₃-, -CH₂-**
3050~2800cm⁻¹:C-H stretching
- **Adsorbed CO₂**
2366, 2345cm⁻¹
O=C=O stretching
- **Surface Acetates**
1558, 1465, 1446, 1350cm⁻¹
- **Monodentate and bidentate ethoxide**
1091, 1053, 1026cm⁻¹
CCO stretching
- **Molecularly adsorbed acetic acid**
1728cm⁻¹: O=C- stretching
1297cm⁻¹: OH bending

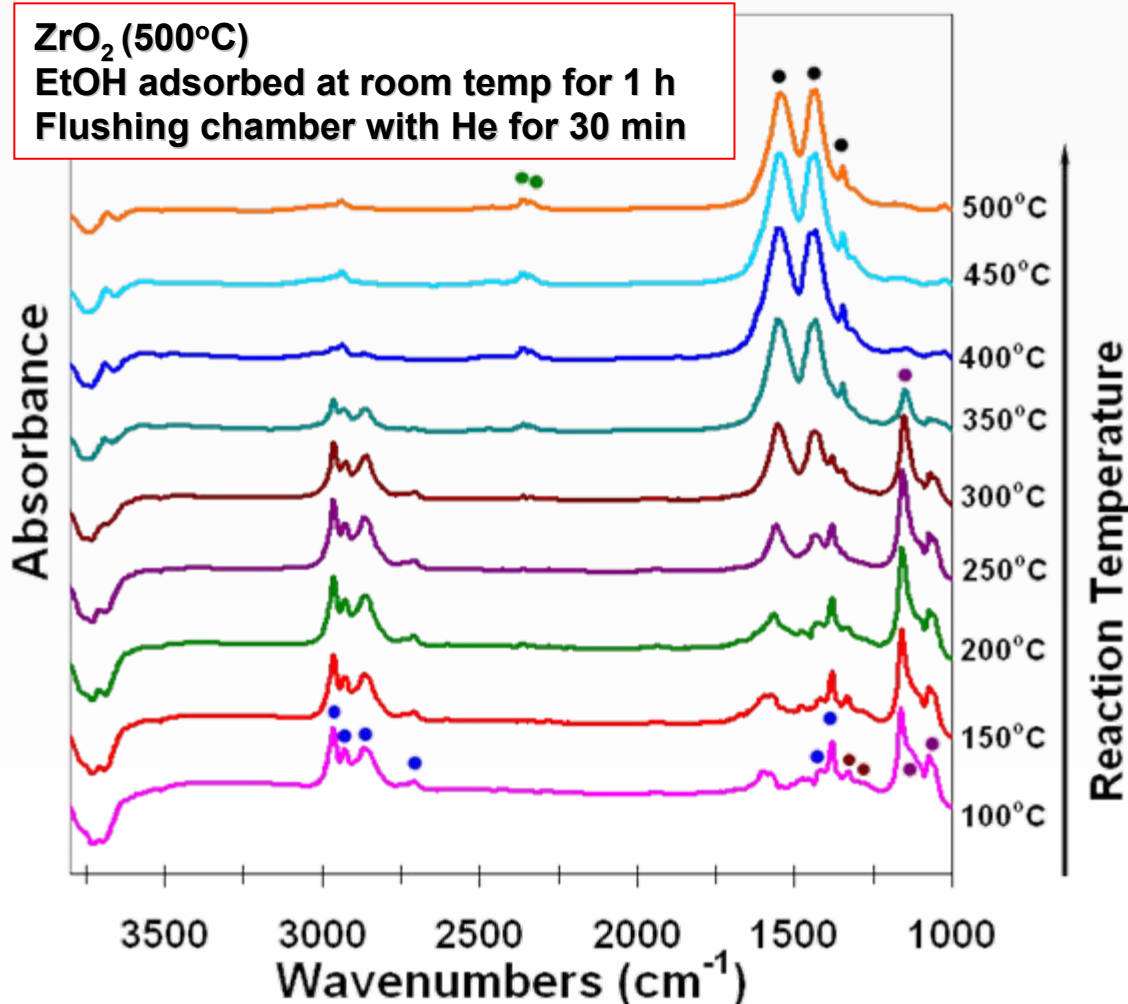
- Acetic acid molecules are adsorbed onto the surface at room temp.
- Surface acetates are observed even at room temp.
- Molecularly adsorbed acetic acid disappears along with increase of temp.
- Linearly adsorbed CO₂ present without experiencing carbonate intermediate.

In-Situ DRIFTS-Ethanol TPD on Bare Support

ZrO₂ (500°C)

EtOH adsorbed at room temp for 1 h

Flushing chamber with He for 30 min



● CH₃- , -CH₂-

3050~2800cm⁻¹: C-H stretching
1429, 1383cm⁻¹: C-H bending

● Adsorbed CO₂

2368, 2352cm⁻¹
O=C=O stretching

● Surface Acetates

1552, 1446, 1348cm⁻¹

● Ethoxide

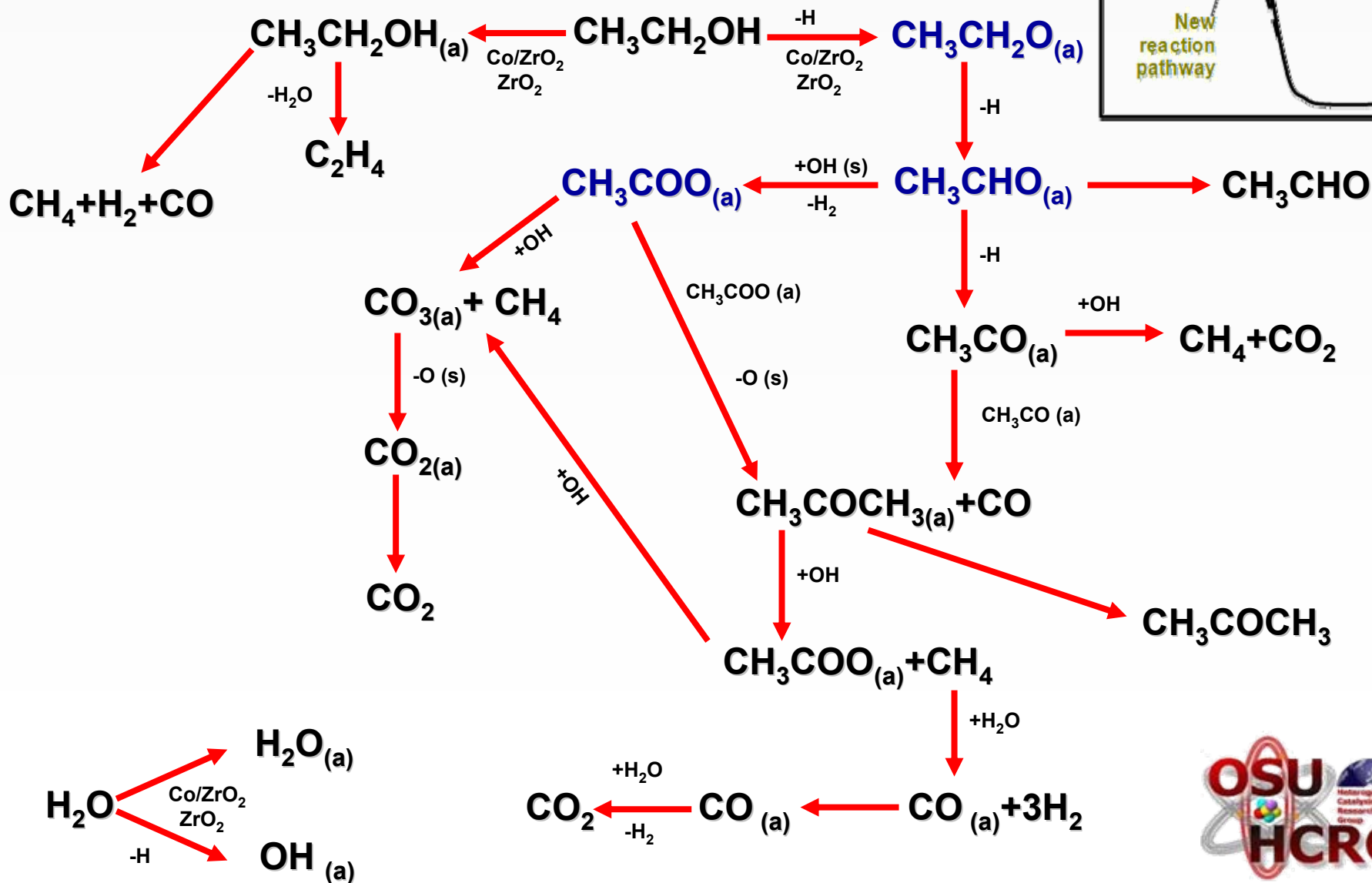
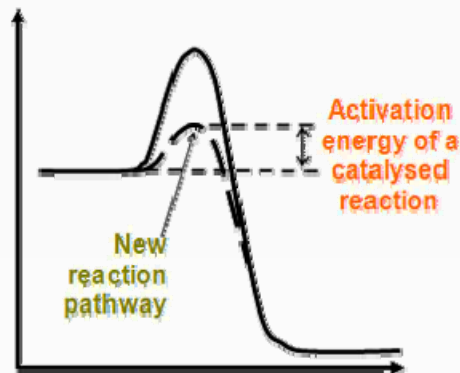
1126, 1074cm⁻¹: C-O stretching
1182cm⁻¹: C-C stretching

● Molecularly adsorbed ethanol

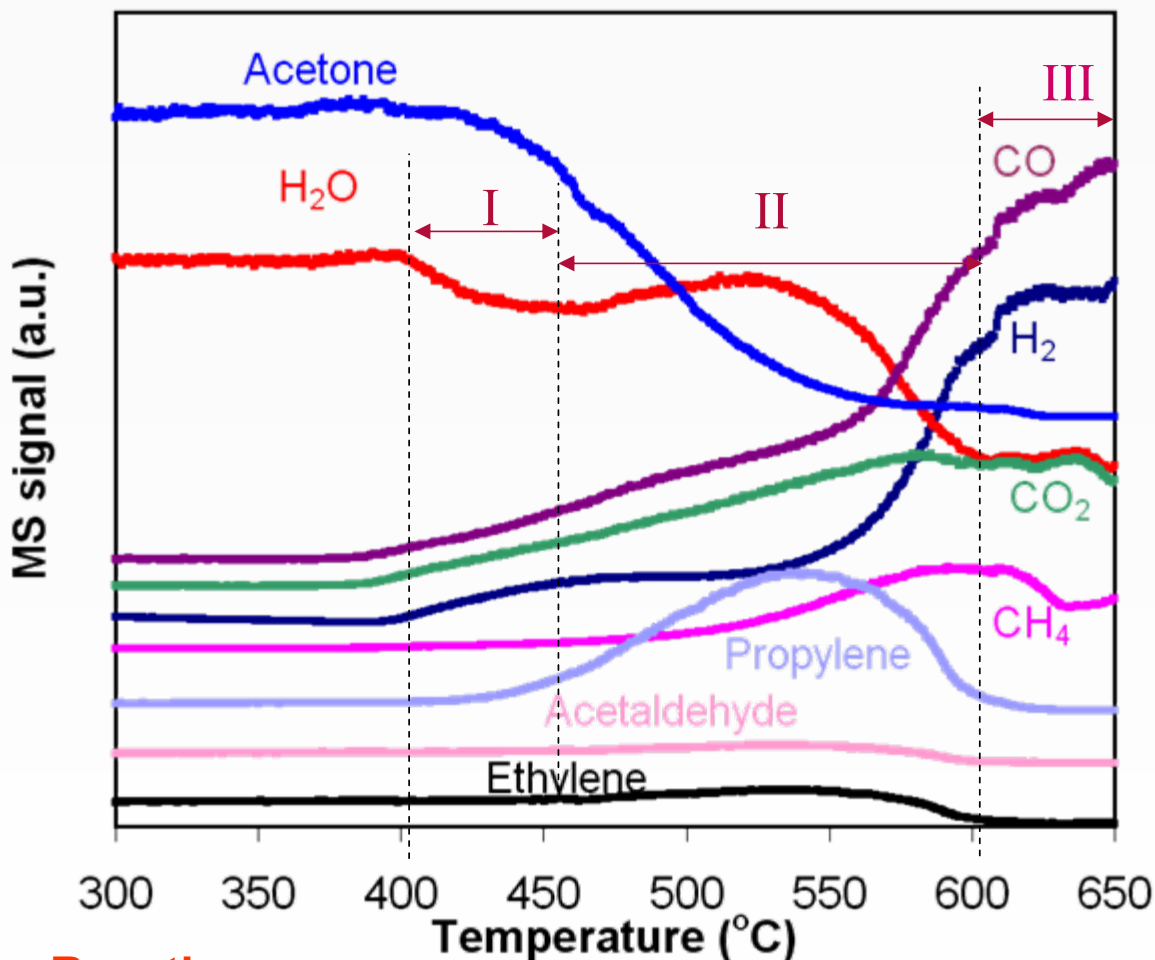
1330cm⁻¹: C-H bending
1292cm⁻¹: OH bending

- C-H bonding is observed at higher temp., compared with Co impregnated sample.
- C-C bonding is seen at higher temp., compared with Co impregnated sample.
- Ethoxide species disappear at higher temp., compared with Co impregnated sample.

Possible Pathways of BESR over Co/ZrO₂



Effect of T on Reaction Network: TPRxn - Acetone +H₂O



Reactions:

- I. $\text{CH}_3\text{COCH}_3 + 3\text{H}_2\text{O} \rightarrow 2\text{CO} + \text{CO}_2 + 6\text{H}_2$
- II. $\text{CH}_3\text{COCH}_3 + \text{H}_2 \rightarrow \text{C}_3\text{H}_6 + \text{H}_2\text{O}$; $\text{CH}_3\text{COCH}_3 + \text{H}_2\text{O} \rightarrow \text{CO}_2 + 2\text{CH}_4$
 $\text{CH}_3\text{COCH}_3 + \text{H}_2\text{O} \rightarrow \text{CO}_2 + \text{C}_2\text{H}_4 + 2\text{H}_2$
- III. $\text{CO}_2 + \text{H}_2 \rightarrow \text{CO} + \text{H}_2\text{O}$; $\text{CH}_4 + 2\text{H}_2\text{O} \rightarrow \text{CO}_2 + 3\text{H}_2$

❖ Sample:

10%Co/ZrO₂

❖ Pretreatment:

400°C for 30 min;

Reduced at 350°C for 2h

under 5%H₂/He;

Degassing at 400°C for 1h
under He

❖ Reaction:

CH₃COCH₃:H₂O=1:10
(molar ratio)

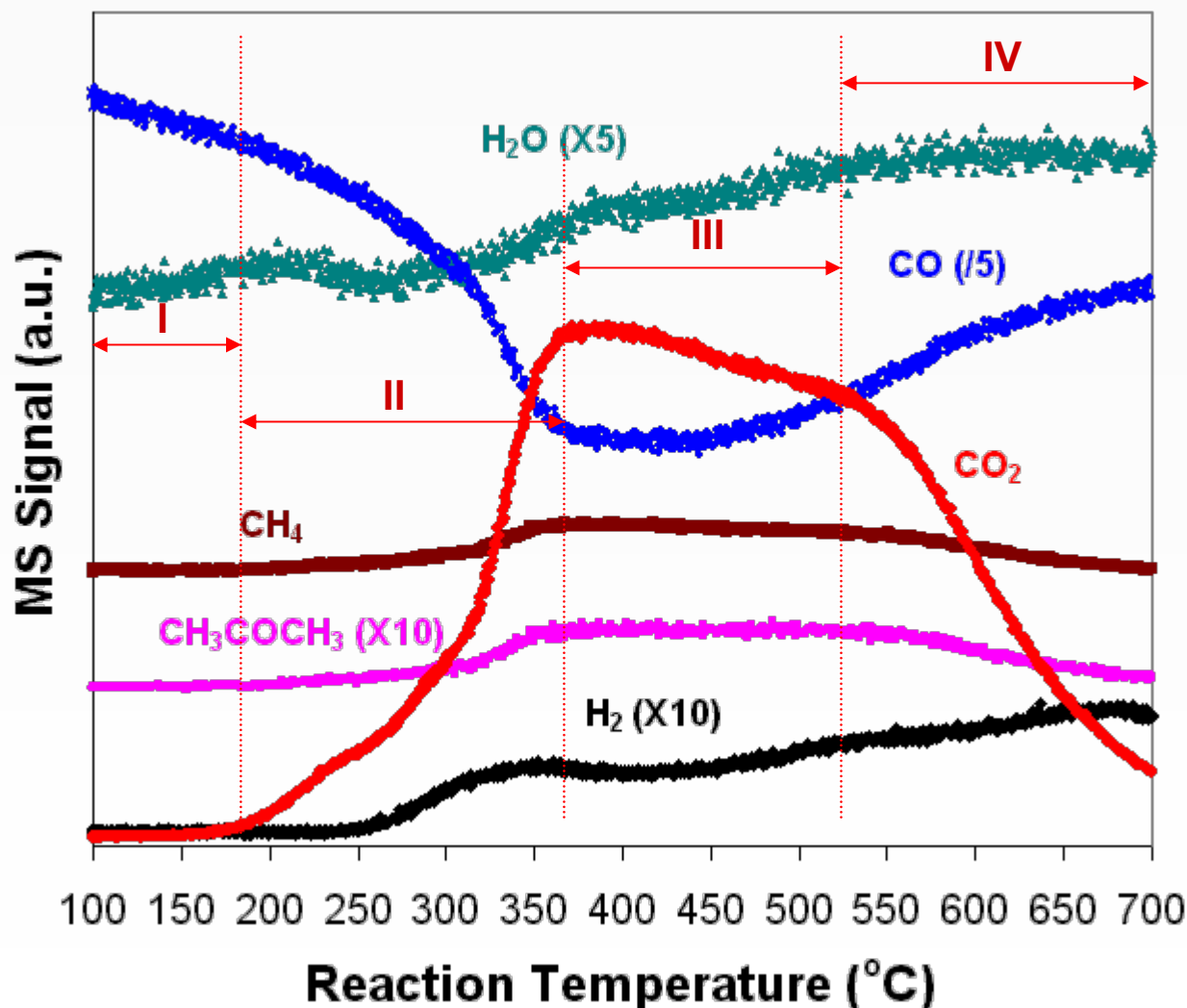
Total flow=55ml/min.

Ramp rate:10°C/min.

❖ Experiment:

Monitoring product stream
with MS

Effect of T on Reaction Network: TPRxn - Water Gas Shift



❖ **Sample:**
10%Co/ZrO₂

❖ **Pretreatment:**
400°C for 3h;
Reduced at 350°C for 2h
under 5%H₂/He;
400°C for 1h under He to
remove moisture

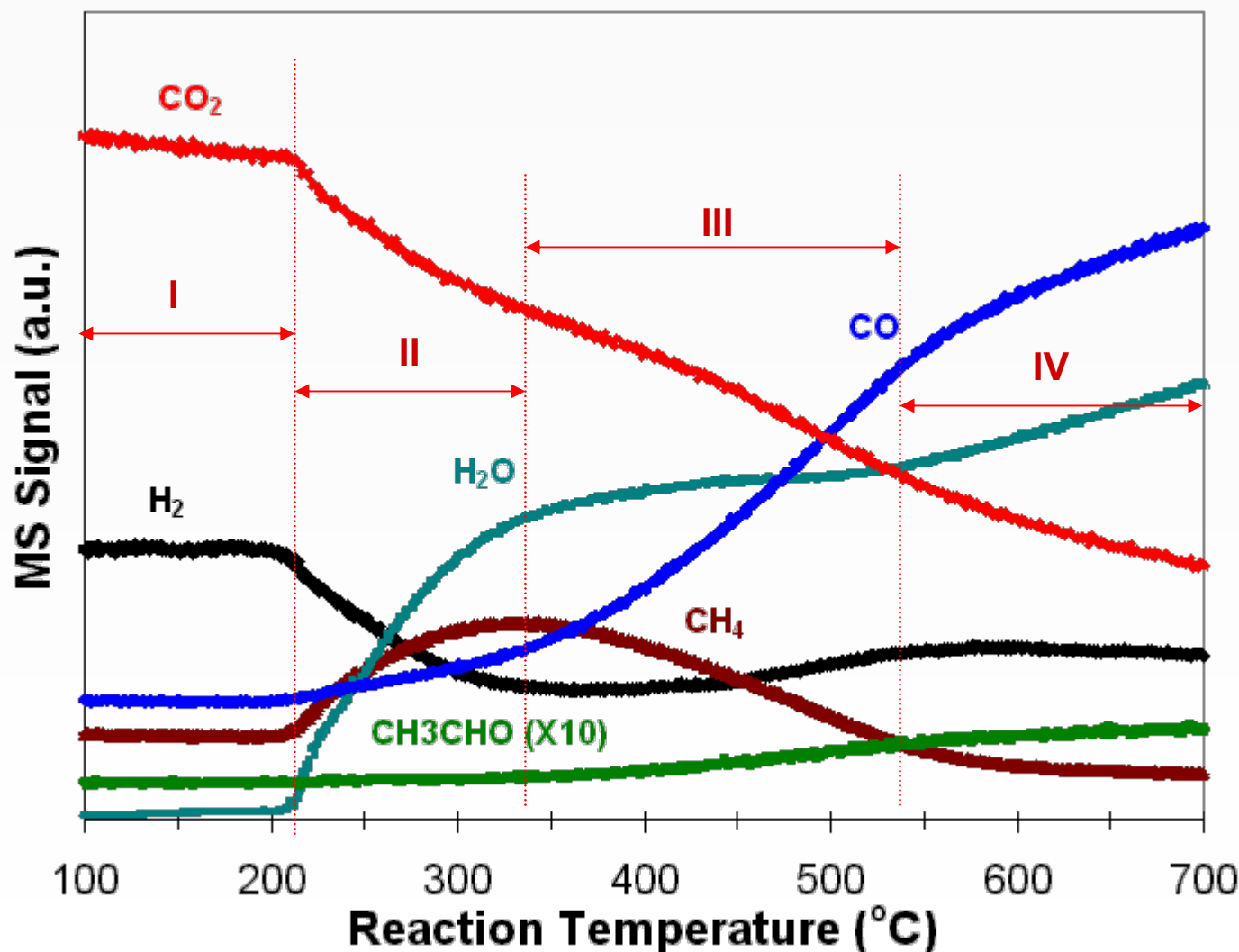
❖ **Reaction:**
CO:H₂O=1:8 (molar ratio)
Total flow=51ml/min.
Ramp rate:10°C/min.

❖ **Experiment:** Monitoring
product stream with MS

I: no reaction; **II:** $\text{CO} + 3\text{H}_2 \rightleftharpoons \text{CH}_4 + \text{H}_2\text{O}$; $\text{CO} + \text{H}_2\text{O} \rightleftharpoons \text{CO}_2 + \text{H}_2$

III: same reactions as step II; **IV:** $\text{CH}_4 + \text{H}_2\text{O} \rightleftharpoons \text{CO} + 3\text{H}_2$; $\text{CO}_2 + \text{H}_2 \rightleftharpoons \text{CO} + \text{H}_2\text{O}$

Effect of T on Reaction Network: TPRxn - Reverse Water Gas Shift



❖ **Sample:**
10%Co/ZrO₂

❖ **Pretreatment:**
400°C for 3h;
Reduced at 350°C for 2h
under 5%H₂/He;
400°C for 1h under He to
remove moisture

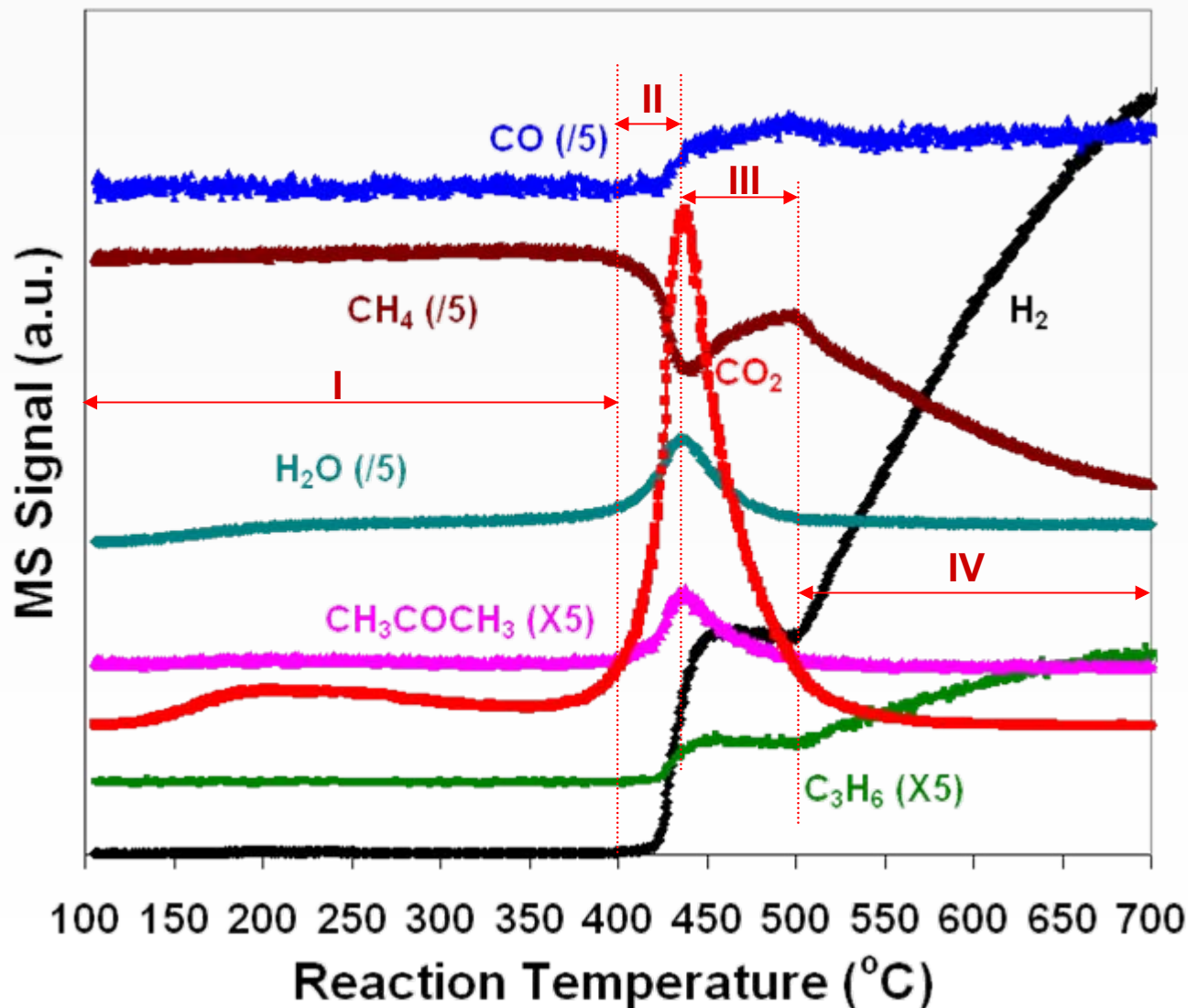
❖ **Reaction:**
CO₂:H₂=1:1 (molar ratio)
Total flow=60ml/min.
Ramp rate:
10°C/min.

❖ **Experiment:**
Monitoring product
stream with MS

I: no reaction; **II:** CO₂ + 4H₂ ⇌ CH₄ + 2H₂O; CO₂ + H₂ ⇌ CO + H₂O

III: CH₄ + 2H₂O ⇌ CO₂ + 4H₂ **IV:** CH₄ + H₂O ⇌ CO + 3H₂; CO₂ + H₂ ⇌ CO + H₂O

Effect of T on Reaction Network: TPRxn-Methane Steam Reforming



❖ **Sample:**
10%Co/ZrO₂

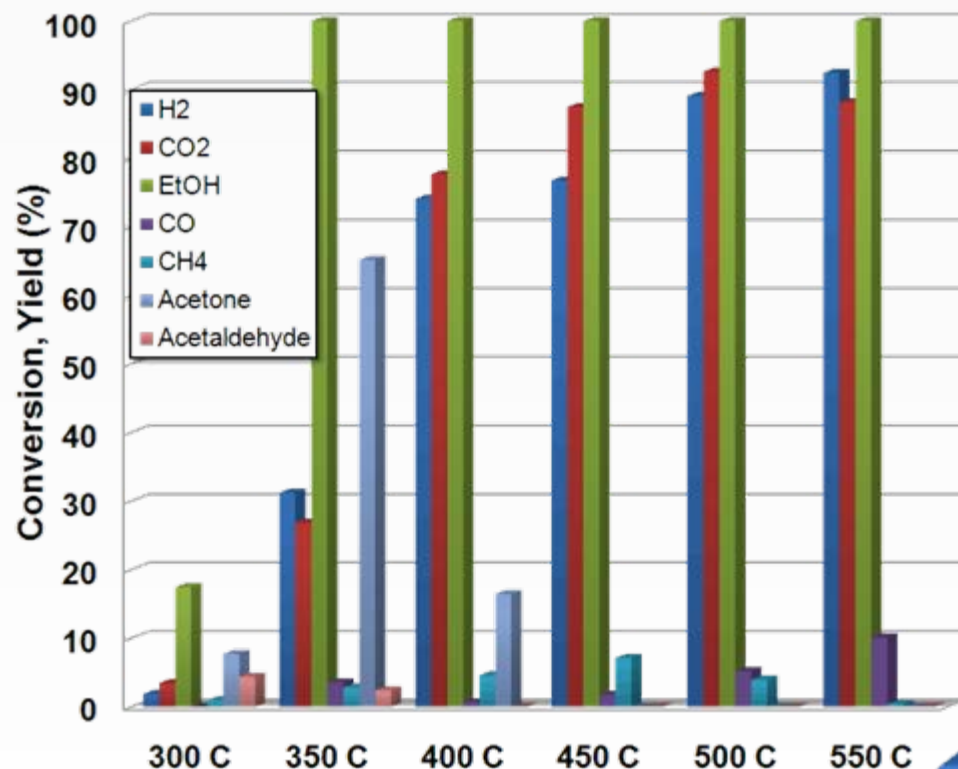
❖ **Pretreatment:**
400°C for 3h;
Reduced at 350°C for 2h
under 5%H₂/He;
400°C for 1h under He to
remove moisture

❖ **Reaction:**
CH₄:H₂O=1:8 (molar
ratio)
Total flow=51ml/min.
Ramping rate:10°C/min.

❖ **Experiment:**
Monitoring product
stream with MS

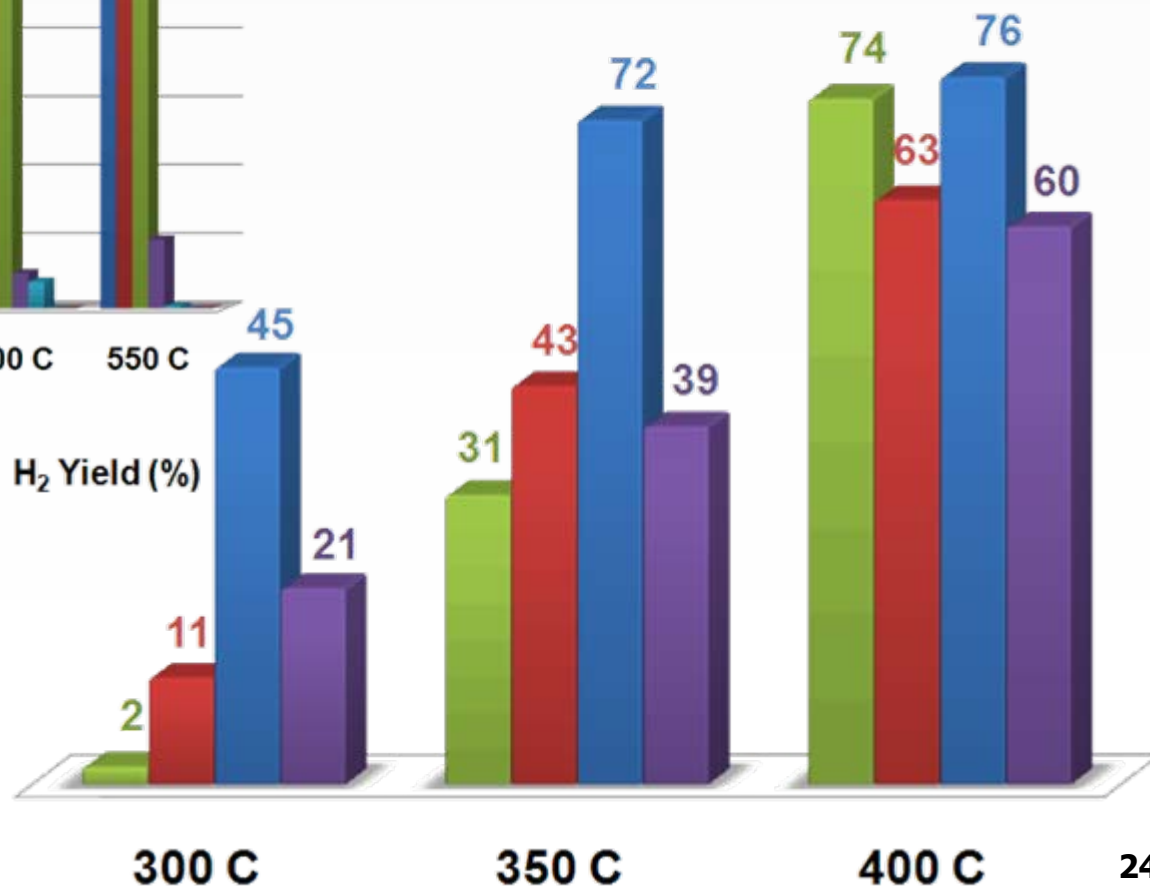
I: No Reaction II: CH₄+2H₂O ⇌ CO₂+4H₂ ; CH₄+H₂O ⇌ 3H₂+CO; 2CH₄+CO₂ ⇌ CH₃COCH₃+H₂O
III: CO₂+2H₂ ⇌ CH₄+2H₂O IV: CH₄ ⇌ C+2H₂; CH₃COCH₃+H₂ ⇌ C₃H₆+H₂O

Modification of the Initial Catalyst Formulation



With modified formulations, higher hydrogen yield can be achieved at lower temperatures

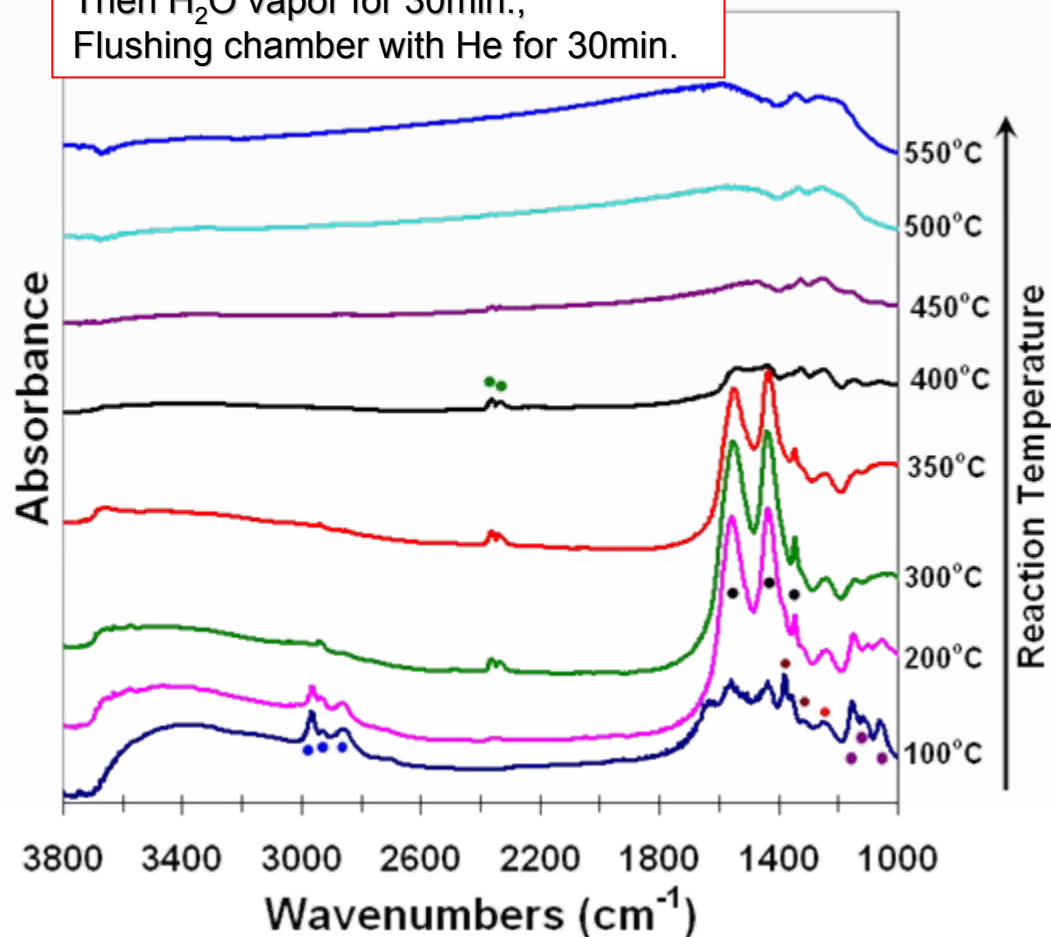
← 10%Co/ZrO₂ 10%Co/10CeO₂-ZrO₂
1%Re-10%Co/10%CeO₂-ZrO₂ Equilibrium Calculation



EtOH: H₂O: dilution=1:10:75
GHSV=5,000h⁻¹
C_{EtOH}=1.2%

Modification of the Initial Catalyst Formulation: In-Situ DRIFTS - EtOH+H₂O Reaction

10%Co/10%CeO₂-ZrO₂ (400°C)
Introducing EtOH vapor for 1h;
Then H₂O vapor for 30min.;
Flushing chamber with He for 30min.



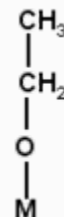
● Monodentate and bidentate ethoxide

2968, 2935, 2871cm⁻¹: C-H stretching

1155cm⁻¹: C-C stretching;

1139cm⁻¹: C-O stretching (mono)

1061cm⁻¹: C-O stretching (bi)

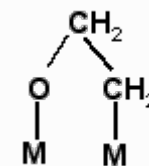


● Acetates

CH₃COO

1562, 1440cm⁻¹: COO stretching

1348cm⁻¹: CH₃ bending



● Molecularly adsorbed ethanol

1380, 1340cm⁻¹

● Adsorbed CO₂

2370, 2347cm⁻¹

● -OH group: 1267cm⁻¹ bending

- The addition of CeO₂ facilitates the conversion of ethanol, leading to the appearance and disappearance of surface reaction intermediate at much lower reaction temperature

Investigation on alternative methods for Co-based catalyst synthesis

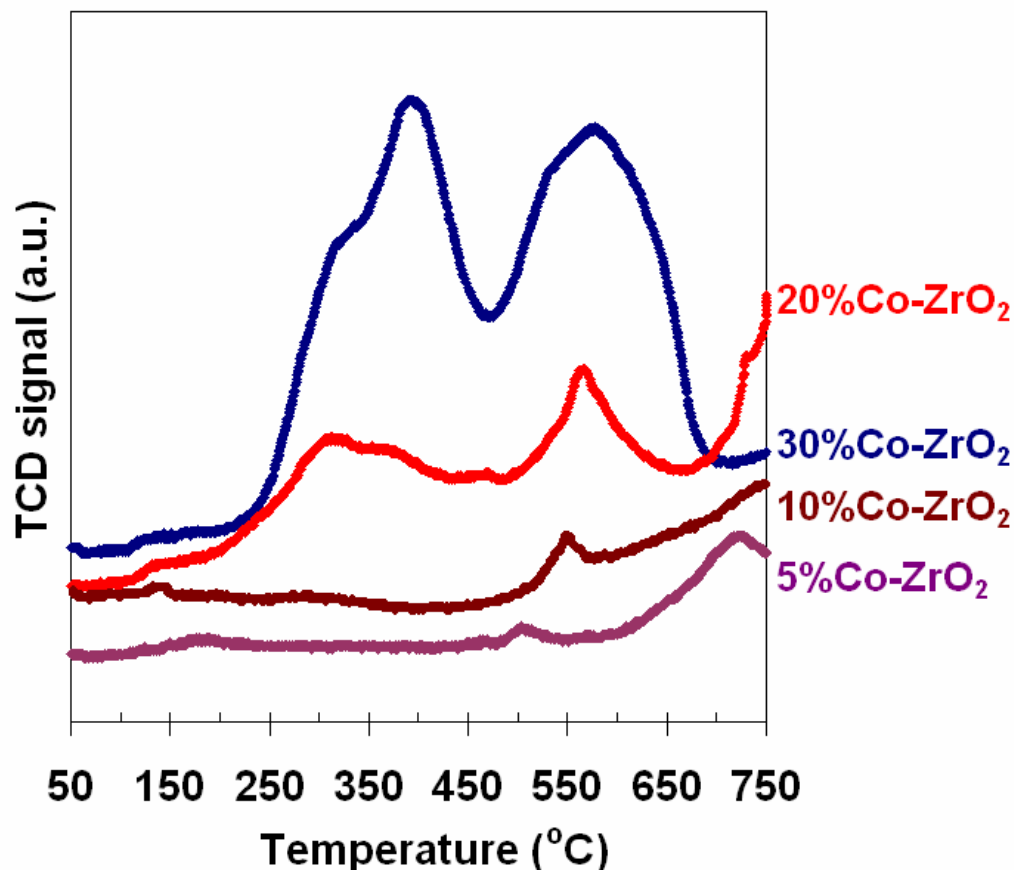
Motivation of research on sol-gel synthesis of Co-ZrO₂ catalysts

- ❖ For IWI preparation, all active metals are dispersed on the surface, which may cause aggregation of metal particles or sintering at high temperatures--phenomena closely related to catalyst stability and selectivity,
- ❖ Sol-gel technique provides a way to uniformly distribute active metal particles in the sample. Particle size is easy to control by varying synthesis parameters

So-gel CoZrO₂ catalyst preparation

- ❖ Zirconium propoxide and cobalt nitrate as precursor
- ❖ Mixing of zirconium propoxide and cobalt nitrate aqueous solution at 65°C and stirring for 1hr
- ❖ Oven dry at 110°C overnight
- ❖ Calcination in air before use

Temperature programmed reduction for Co-ZrO₂ sol-gel catalysts with different Co loadings

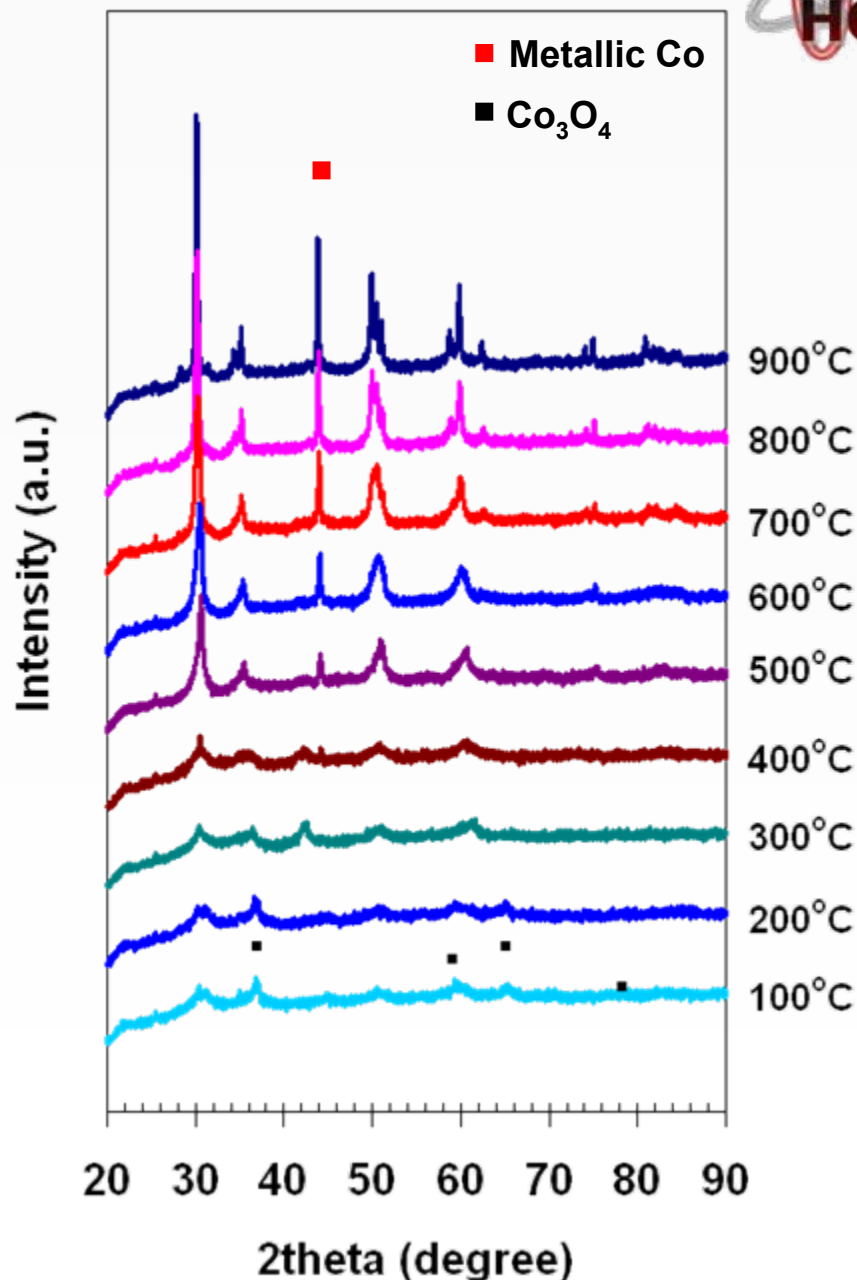


- ❖ With sol-gel preparation, there is better interaction between cobalt and the zirconia support. When Co wt% is less than 10%, there is hardly any reduction features till 500°C.
- ❖ With increasing Co loadings, there is more cobalt on the surface and is easier to reduce

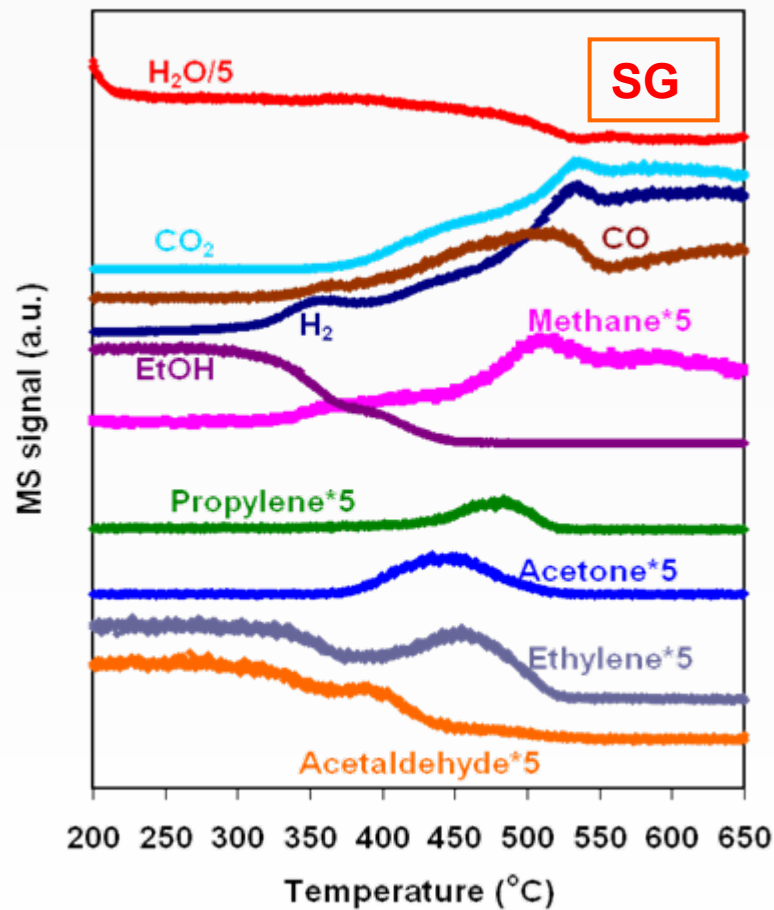
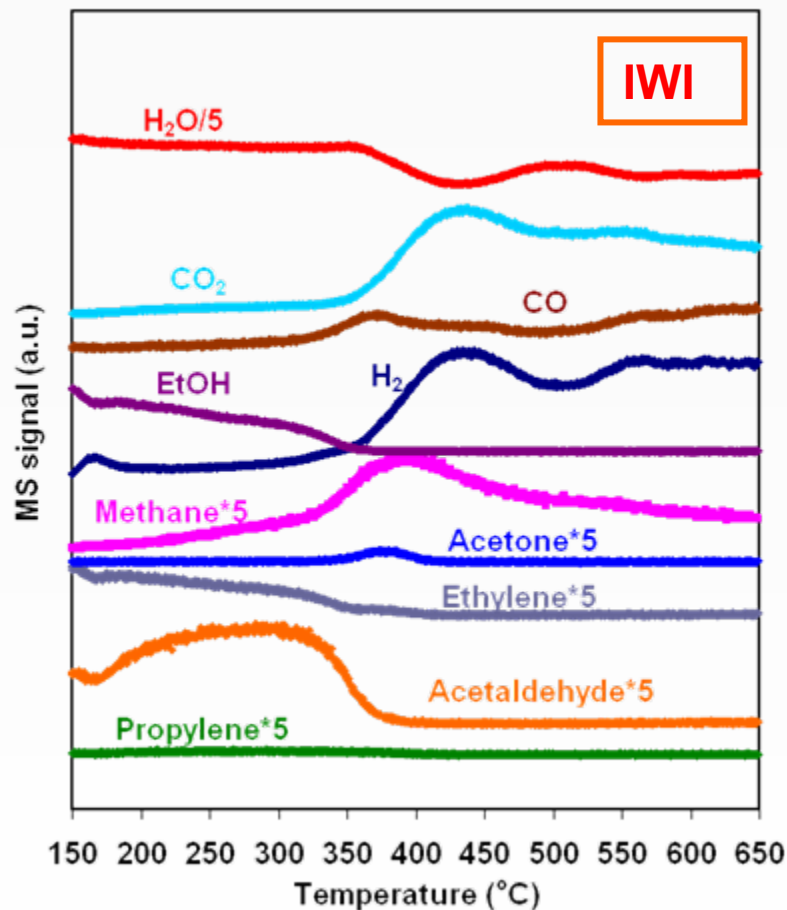
In-situ XRD during Reduction

Reduction: 5% H_2/N_2
SG-30% CoZrO_2
From 100°C to 900°C with 100°C/step

- ❖ Cool to 100C Monoclinic ZrO_2 with Co
- ❖ 900C Tetragonal ZrO_2 with a small amount of Monoclinic ZrO_2 and Metallic Co
- ❖ 800C Tetragonal ZrO_2 with a small amount of Monoclinic ZrO_2 and Metallic Co
- ❖ 700C Tetragonal ZrO_2 with a small amount of Monoclinic ZrO_2 and Metallic Co
- ❖ 600C Cubic ZrO_2 with Co phase
- ❖ 500C Cubic ZrO_2 , CoO and Co coexist
- ❖ 400C CoO and Co coexists together with Cubic ZrO_2
- ❖ 300C Cubic ZrO_2 with CoO
- ❖ 200 C Co_3O_4 phase with cubic ZrO_2
- ❖ 100C Co_3O_4 phase with cubic ZrO_2



10%CoZrO₂ catalysts prepared by two different methods result in different product distributions



- (1) Pretreat: 400°C He for 30 min (2) Reduction: 600°C (350°C for IWI) 5% H₂/He 2 hrs
(3) Purge 400°C He for 1 hr (4) TPRxn with EtOH:H₂O=1:10 using Cirrus MS

It may be possible to affect the product distribution and stability by using new synthesis techniques based on organometallic chemistry.

Publications and Presentations

- ❖ Song, H. Zhang, L. Watson, R.B., Braden, D., Ozkan, U.S., “Investigation of Bio-ethanol Steam Reforming over Cobalt-based Catalysts” *Catalysis Today* (in press);
- ❖ Song, H., Zhang, L., Ozkan, U.S., “Effect of Synthesis Parameters on the Catalytic Activity of Co/ZrO₂ for Bio-ethanol Steam Reforming” *Journal of Green Chemistry* (in press);
- ❖ Song, H. and Ozkan, U.S., “Fuel Cell Grade Hydrogen Production from the Bio-Ethanol Steam Reforming over Co-based Catalysts: An Investigation of Reaction Networks and Active Sites” *Ohio Fuel Cell Symposium*, Canton, Ohio, May 2006;
- ❖ Song H., Zhang, L., Ozkan, U.S., “Investigation of bio-ethanol system reforming over cobalt-based catalysts”, 232nd ACS National meeting & exposition, San Francisco, CA, September 2006;
- ❖ Song H., Zhang, L., Ozkan, U.S., “ Investigation of bio-ethanol steam reforming over cobalt-based catalysts”, U.S. Department of Energy Bio-derived Liquids to Hydrogen Distributed Reforming Working Group Kick-Off Meeting, Baltimore, MD, October 2006;
- ❖ Song, H. Zhang, L., and Ozkan, U.S., “Catalytic Hydrogen Production from Renewable Sources” *OSU-Honda Research Forum*, Columbus, Ohio, February 2007;
- ❖ Song, H., Zhang, L., Ozkan, U.S., “Investigation of Reaction Networks and Active Sites in Steam Reforming of Bio-ethanol over Cobalt based Catalysts” 233rd ACS National Meeting, Chicago, IL, March 2007.

Future Work



- ❖ **Kinetic and mechanistic investigations coupled with *in-situ* characterization**
- ❖ **Performing economic analysis based on updated catalyst system knowledge database**
- ❖ **Performance optimization**
- ❖ **Investigation of catalyst deactivation and regeneration characteristics**
- ❖ **Catalyst scale-up through industrial partnerships**

Project Summary

- ❖ **Target:** development of a catalytic system that does not rely on precious metals and that can be active and selective in the 350°C-550°C temperature range.
- ❖ **Relevance:** help to develop small-scale distributed hydrogen production technologies from renewable liquid energy sources.
- ❖ **Approach:** develop a systematic optimization strategy for evaluating the catalytic performance of different catalyst systems.
- ❖ **Accomplishments:**
 - Understanding the competing reaction networks
 - Identifying active sites during reaction
 - Optimizing catalyst system based on modification to the IWI sample and SG prepared catalyst
- ❖ **Future Work:** Mechanistic investigations coupled with in-situ characterization; economic analysis; deactivation/regeneration studies.



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