

# **Heterogeneous Reburning by Mixed Fuels**

**Wei-Yin Chen**

**Department of Chemical Engineering  
University of Mississippi**

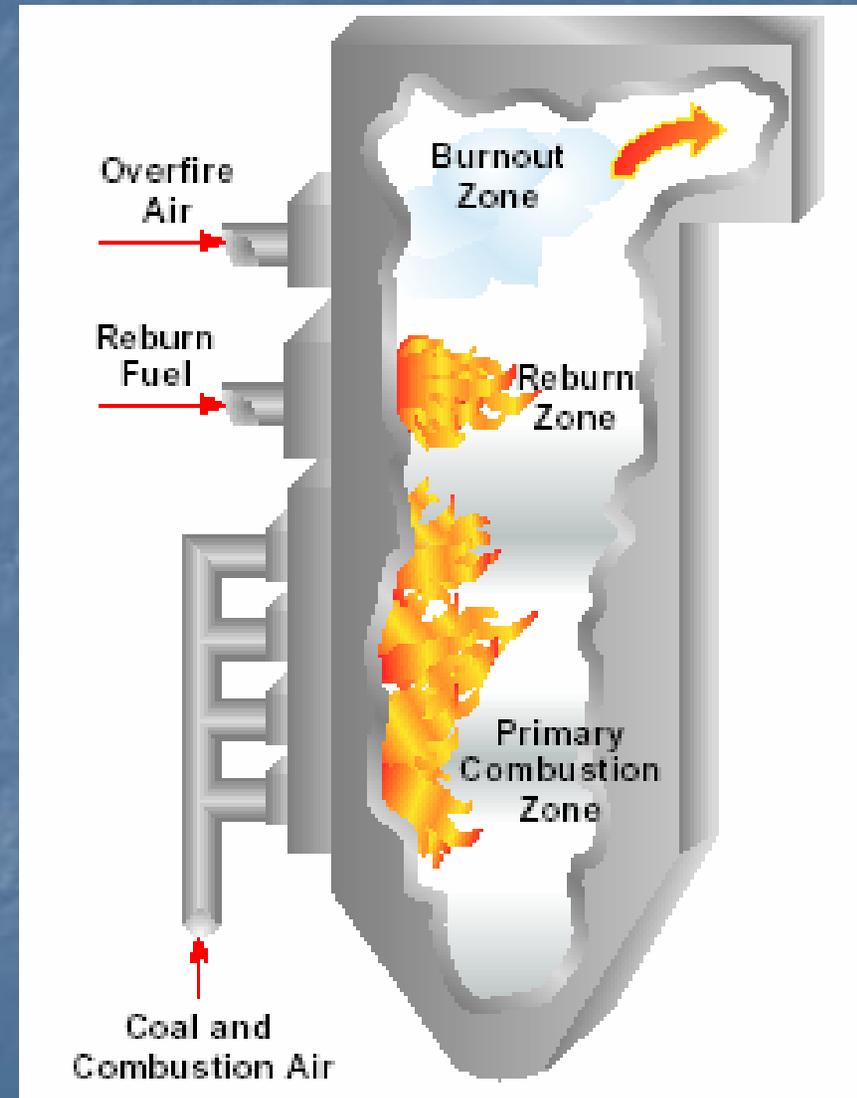
**UCR/HBCU Contractors Review Conference  
Department of Energy  
Pittsburgh, PA  
June 6 and 7, 2006**

# OUTLINE

- **Review of Heterogeneous Reburning Mechanisms**  
*AIChE J.*, 42, 1968-1976 (1996); *AIChE J.*, 47, 2781-2797 (2001)
- **Design of Multi-Function, Mixed Fuel for Efficient Reburning**  
Phase I of the current project: *Fuel*, 85, 1781-1793 (2006)
- **Scale-Up Demonstration at UND EERC**
- **Post-Combustion Reduction of NO from Stationary and Mobile Sources**
- **Char Nitrogen Pathway - Two-Stage (Reburn + Burnout) Experiments**
- **Reactivity of Young Chars in Flame Environments**

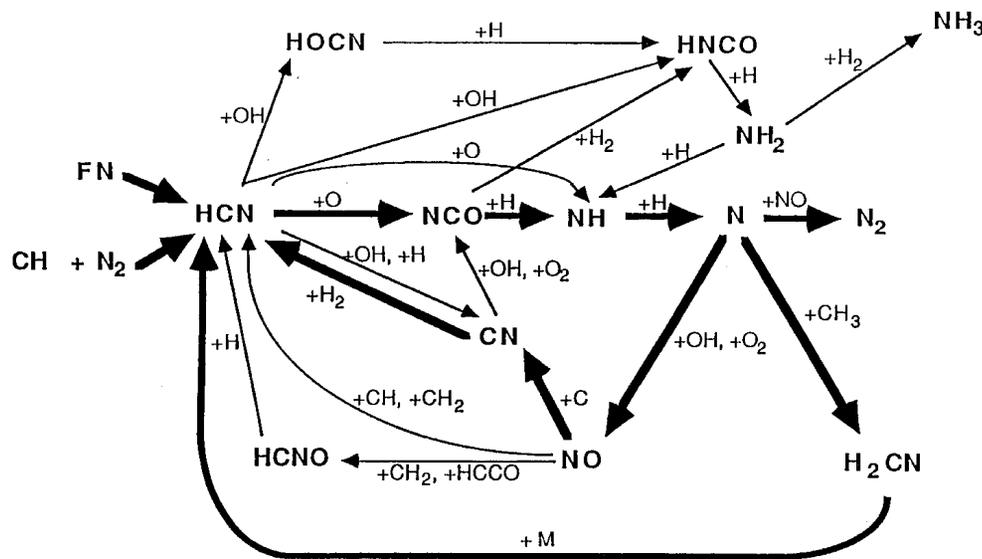
# Background of Reburning

- Able to retrofit old boilers
- Proposed by J. Wendt in 1973
- First tested on a large scale boiler in Japan in 1983 – 50% reduction in NO realized
- 60% reduction floor for over 3 decades



# Natural Gas Reburning

## NITROGEN CHEMISTRY IN RICH FLAMES

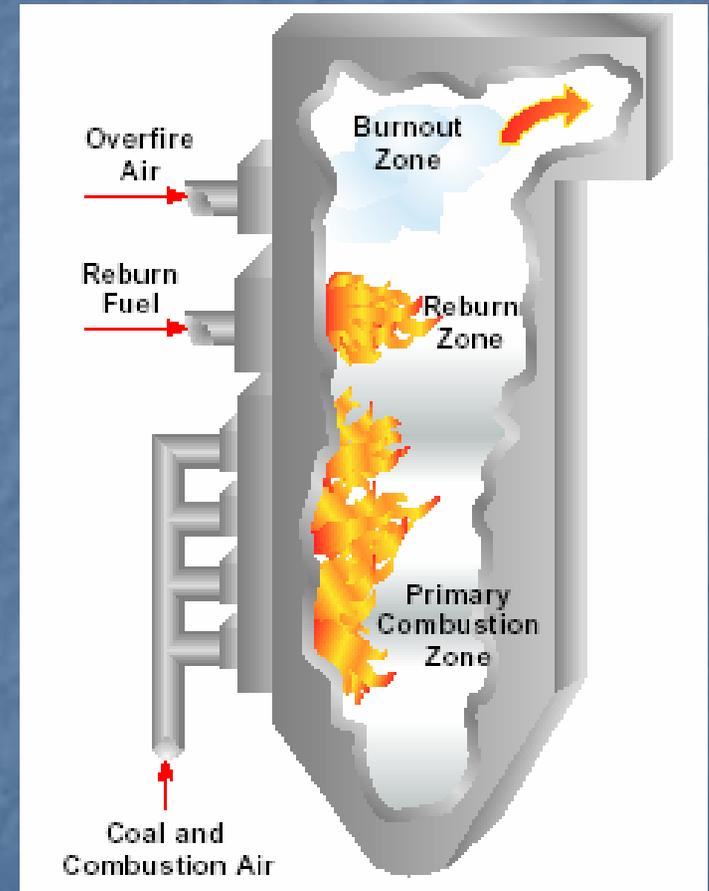


- C-, CH- and CH<sub>2</sub>-, effectively reduce NO to HCN
- HCN is oxidized to NO in the burnout zone
- Thus, there is a 60% NO reduction floor

Miller and Bowman, 1989

# Coal Reburning

- Char is the major reaction intermediate that contains nitrogen
- Char nitrogen is oxidized to NO in the burnout zone
- Thus, there is also a 60% NO reduction floor

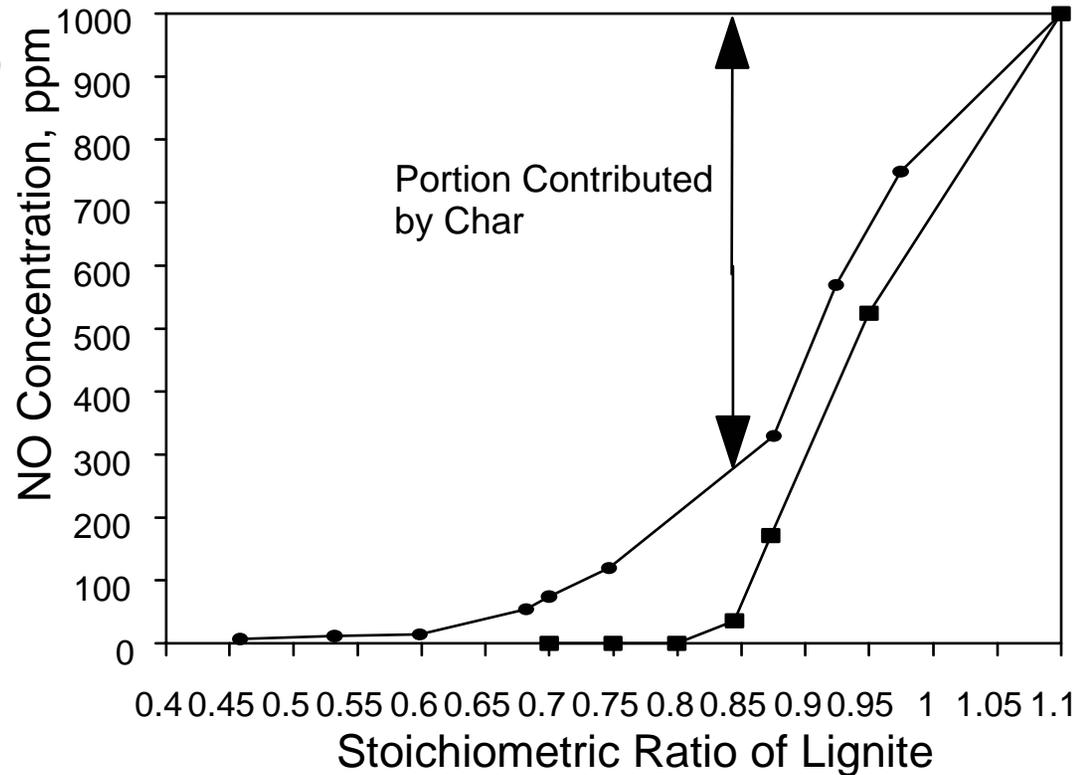


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# Efficient Heterogeneous NO Reduction by Coal-Derived Chars

Primary Zone Stoichiometric Ratio: 1.10  
Feed NO Concentration: 1000 ppm  
Reaction Time: 0.20 Sec  
Temperature: 1100 C



● ND Lignite Char

■ ND Lignite

# Mechanisms of Heterogeneous NO Reduction

- Roles of carbon in the young chars derived from low rank coals
- Roles of CO in the gas phase
- Roles of minerals in the chars

# Roles of Carbon in Young Chars Derived from Low Rank Coals

- NO is chemisorbed on the catalytic minerals
- Oxygen atoms are transferred to the carbon reactive sites by minerals
- Surface oxides, C(O), desorb from the carbon surface, which is usually rate-controlling,  
$$\text{C(O)} \longrightarrow \text{CO}_{(g)}$$

# Roles of Gaseous CO on NO Reduction



- CO scavenges surface oxygen complexes, C(O), and facilitates oxygen turnover
- Gaseous CO regenerates carbon reactive sites, C<sub>f</sub>
- Carbon in char is not consumed, thus, char serves as a catalyst
- Existence of stable surface oxides that desorb only at 1430°C – our work to be discussed later
- Catalytic oxidation of lignite chars produces more CO than oxidation of bituminous-coal chars, e.g., Lizzio *et al.*, 1990; Chen and Tang, 2001

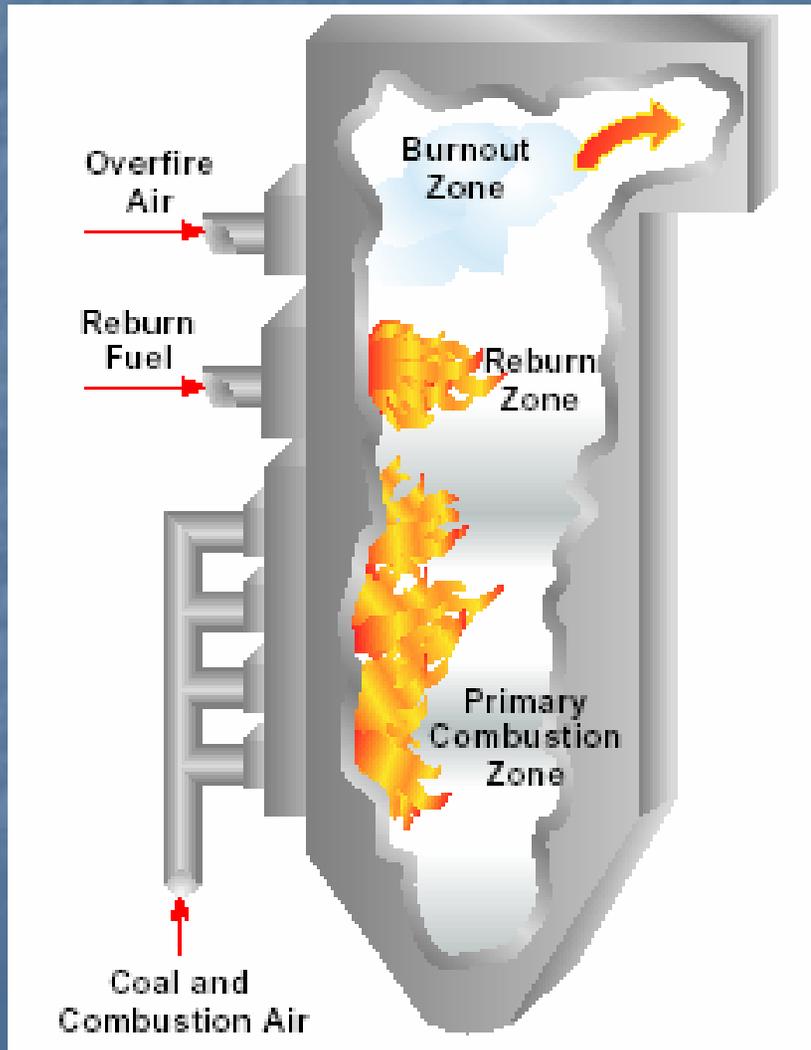
# Roles of Minerals in Lignite

- They adsorb NO, and also serve as oxygen shuttling agents
- During reburn, they catalytically reduce HCN, thus reduce HCN oxidation to NO in the burnout stage

# OUTLINE

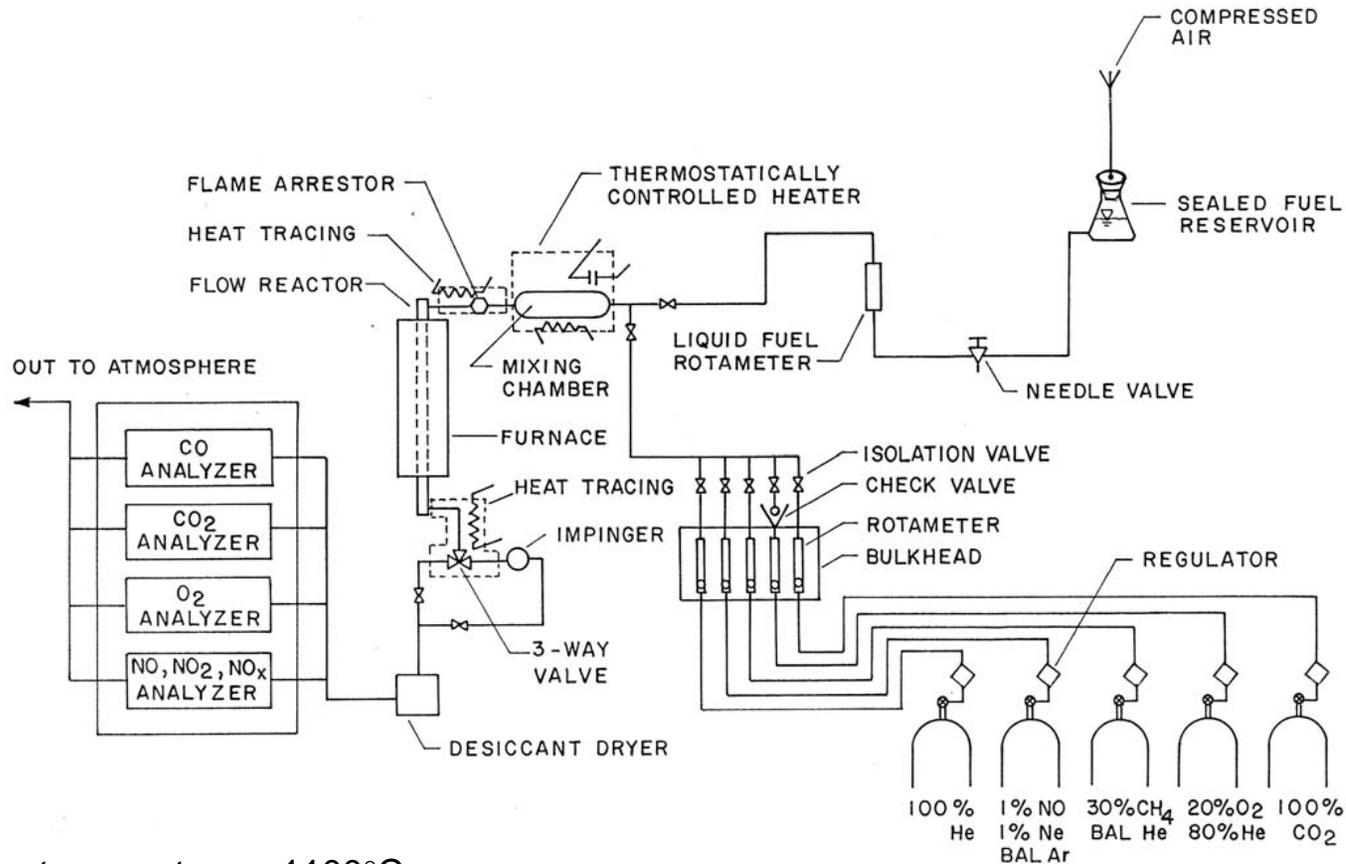
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# Design of Multi-Function, Mixed Fuel for Efficient Reburning



- It reduces NO, catalytically or non-catalytically
  - e.g., natural gas or biomass fly ash
- It catalytically reduces HCN, thus minimize HCN oxidation to NO in the burnout stage
  - e.g., lignite ash or biomass flyash

# Bench-Scale Apparatus

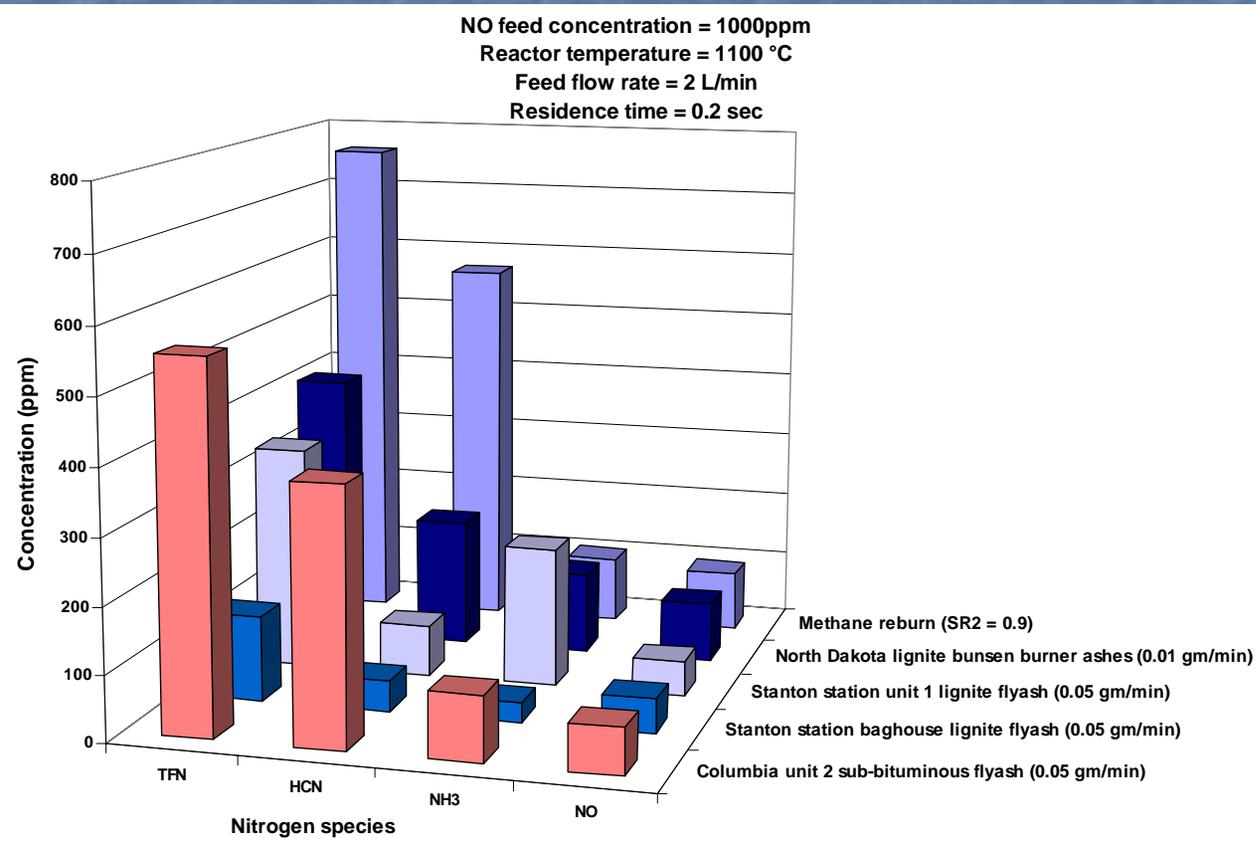


- Gas temperature = 1100°C
- Residence time = 0.2 s

# Experimental Apparatus

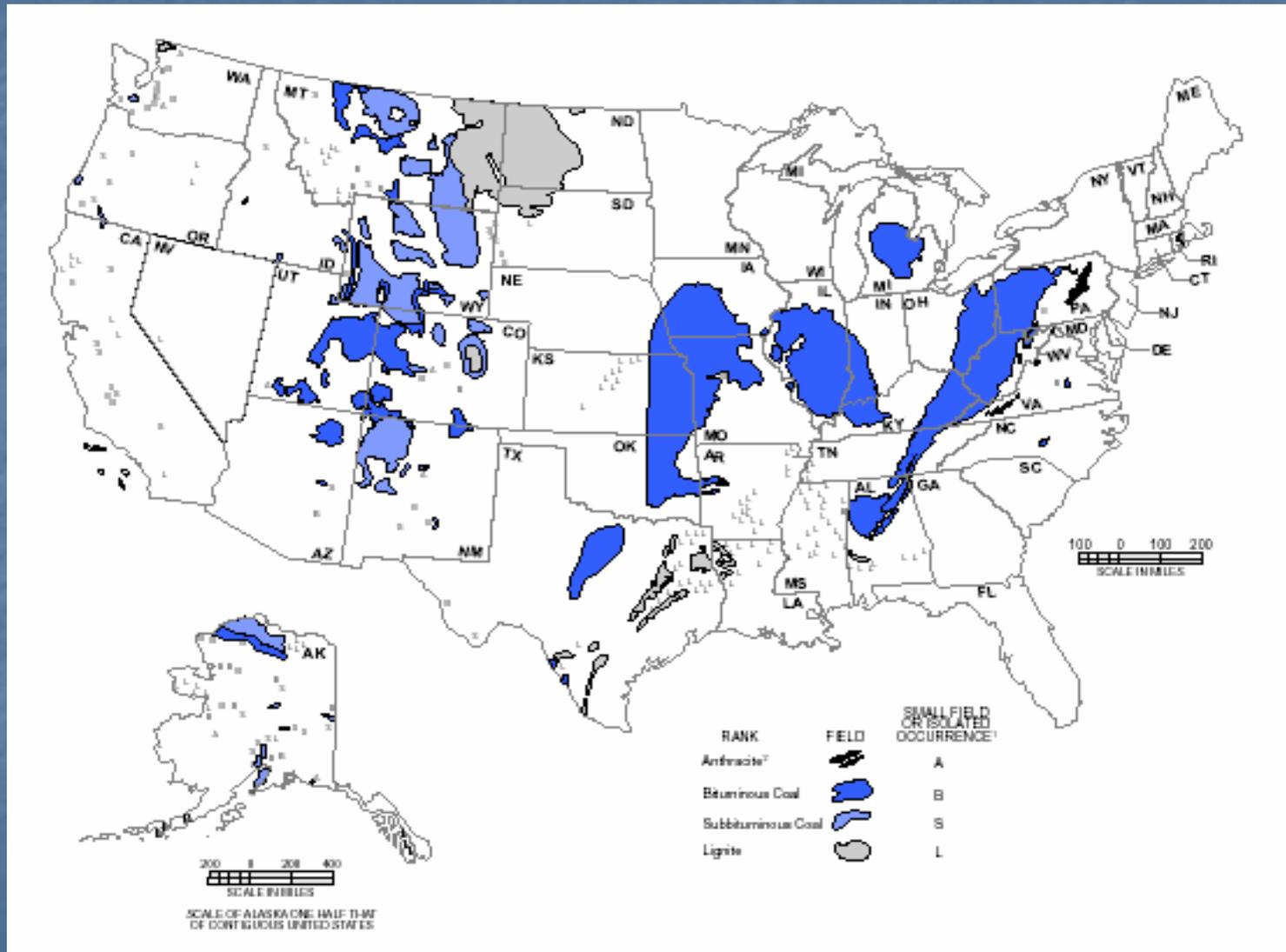


# Results for Ash Feed Rate of 0.05 gm/min



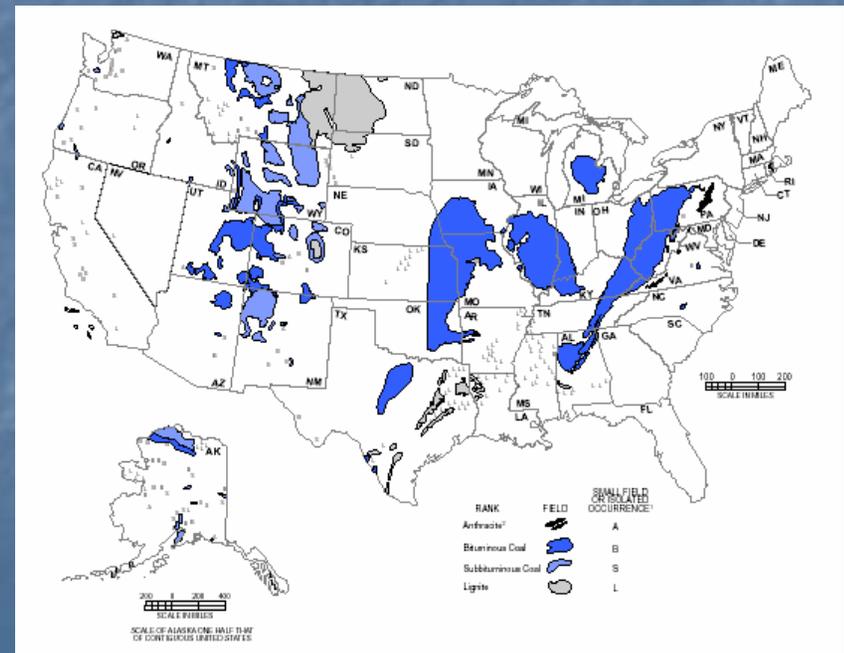
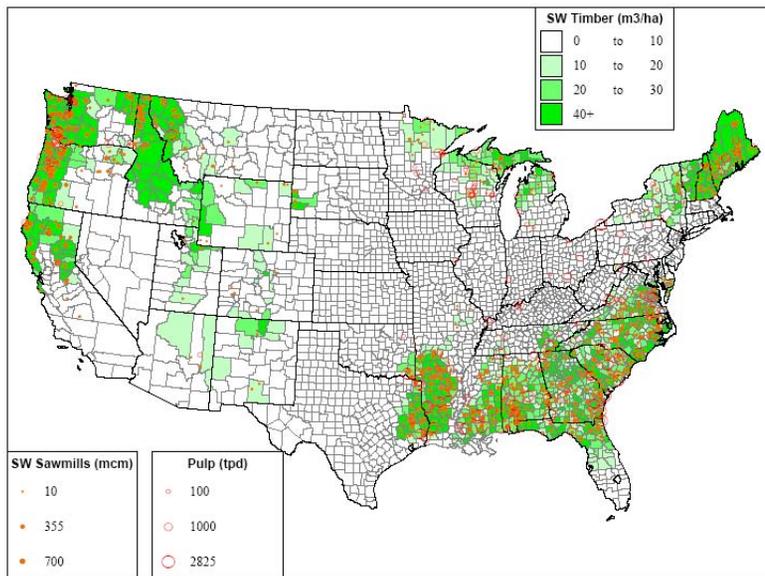
- Bag-house ashes are more effective in HCN reduction than those from an electrostatic precipitator, they are also effective at  $\text{NH}_3$  reduction
- HCN reduction increases with higher feeding rate of ash
- Methane and ash from lignite-fired power plant achieved over 90% conversion of NO

# Distribution of Lignite in the US



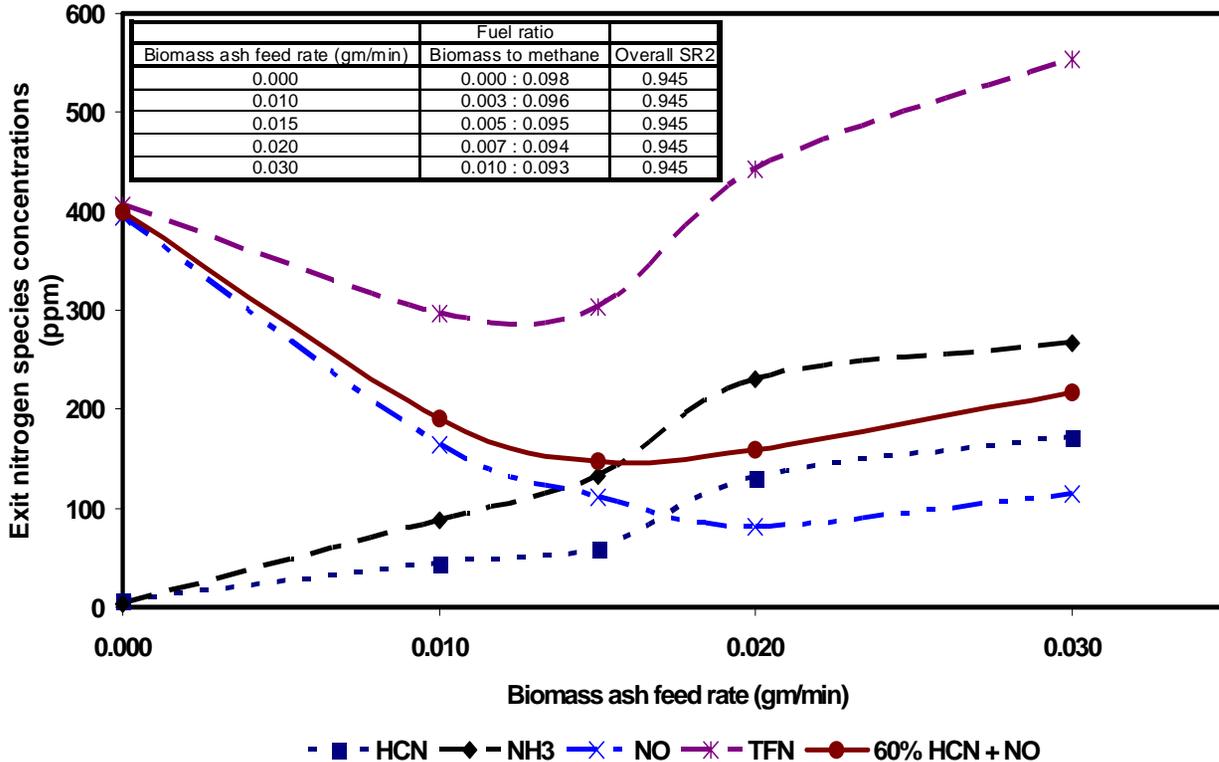
# Advantages of Alternative Selection

- Biomass fly ash is widely available
- It contains carbon that efficiently reduces NO
- It catalytically reduces HCN
- It has low nitrogen content
- Its cost is low



# Biomass Ash at SR2 = 0.945

NO feed concentration = 1000ppm  
 Reactor temperature = 1100 °C  
 Feed flow rate = 2 L/min  
 Residence time = 0.2 sec  
 SR2 = 0.945



- Biomass ash effectively reduces HCN, and appears to provide better NO reduction than methane for a fixed SR2
- Assuming 60% of HCN is converted back to NO in burnout zone, the TFN yields at optimal feeding rate imply a conversion of over 80% of NO

# Economic Analysis

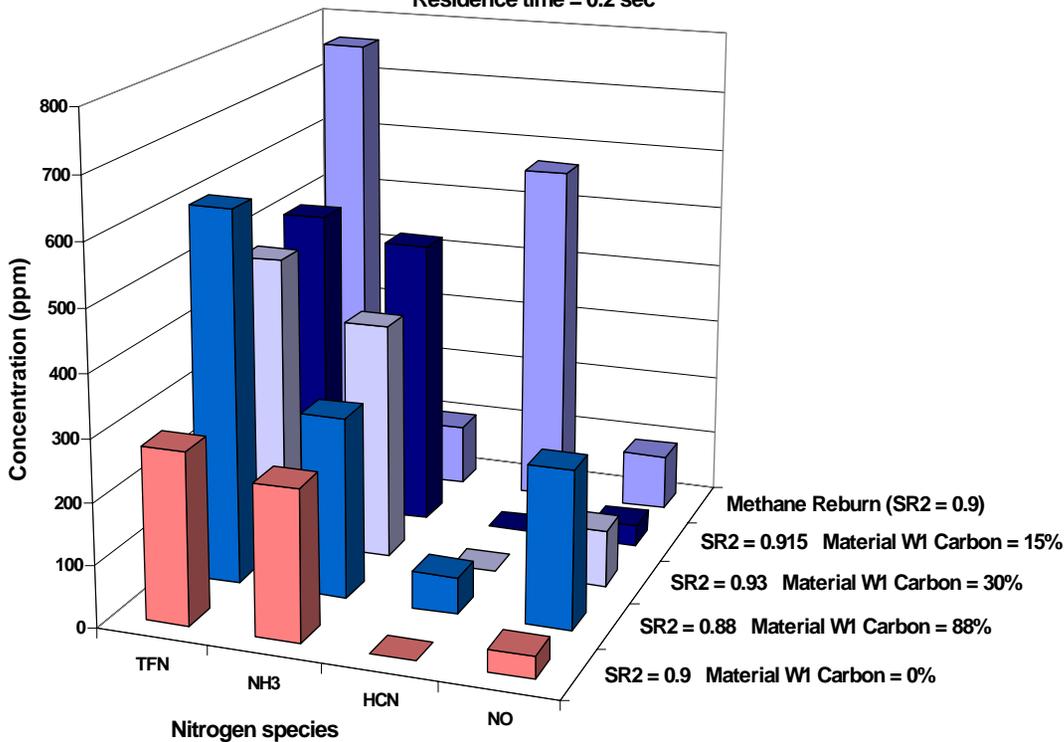
- **Raw-material cost benefits:**
  - It's more cost-effective in terms of per ton of NOx removed than traditional gas reburning.
  - It's competitive with selective catalytic reduction (\$2,000 to \$5,000 per ton of NOx) which can achieve similar efficiencies.

**Table 6: Raw-Material Cost Benefits of Mixed Fuel Reburn**

Power Plant	Size (MWe)	Gas Reburn Heat Input (%)	Baseline NOx (lb/MMBtu)	Gas Reburn (lb/MMBtu)	% Reduction	Mixed Fuel Reburn (lb/MMBtu)	% Reduction	Gas Reburn Cost (dollars/tonNOx)	Mixed Fuel Reburn Cost (dollars/tonNOx)
Hennepin Unit 1	80	18	0.73	0.24	67%	0.11	85%	7360	5043
Lakeside Unit 7	40	23	0.73	0.29	60%	0.11	85%	10520	5107
Kodak Park	62	18	0.73	0.32	56%	0.11	85%	8806	5106
Niles Station	108	8 to 18	0.73	0.37	50%	0.11	85%	9863	5134

# Materials W1 and W2 at Varying SR2 - Natural Gas Replaced

NO feed concentration = 1000ppm  
Reactor temperature = 1100 °C  
Feed flow rate = 2 L/min  
Residence time = 0.2 sec



- A combination of materials W1 and W2 would avoid fluctuations in natural gas price
- HCN yields are insignificant and there is no longer a concern of HCN conversion to NO in the burnout zone

# Conclusions on Mixed Fuel Reburning

- By careful design of a mixed fuel, reburning technology is likely to meet the Environmental Protection Agency's regulation of removing 85%, or 0.15 lb/million BTU, of NO<sub>x</sub> in a three-stage reburning process
- Cost is comparable to SCR
- Alternatives to natural gas appears be feasible and even more effective
- Pilot-scale tests at even larger units is anticipated
- A patent application is pending

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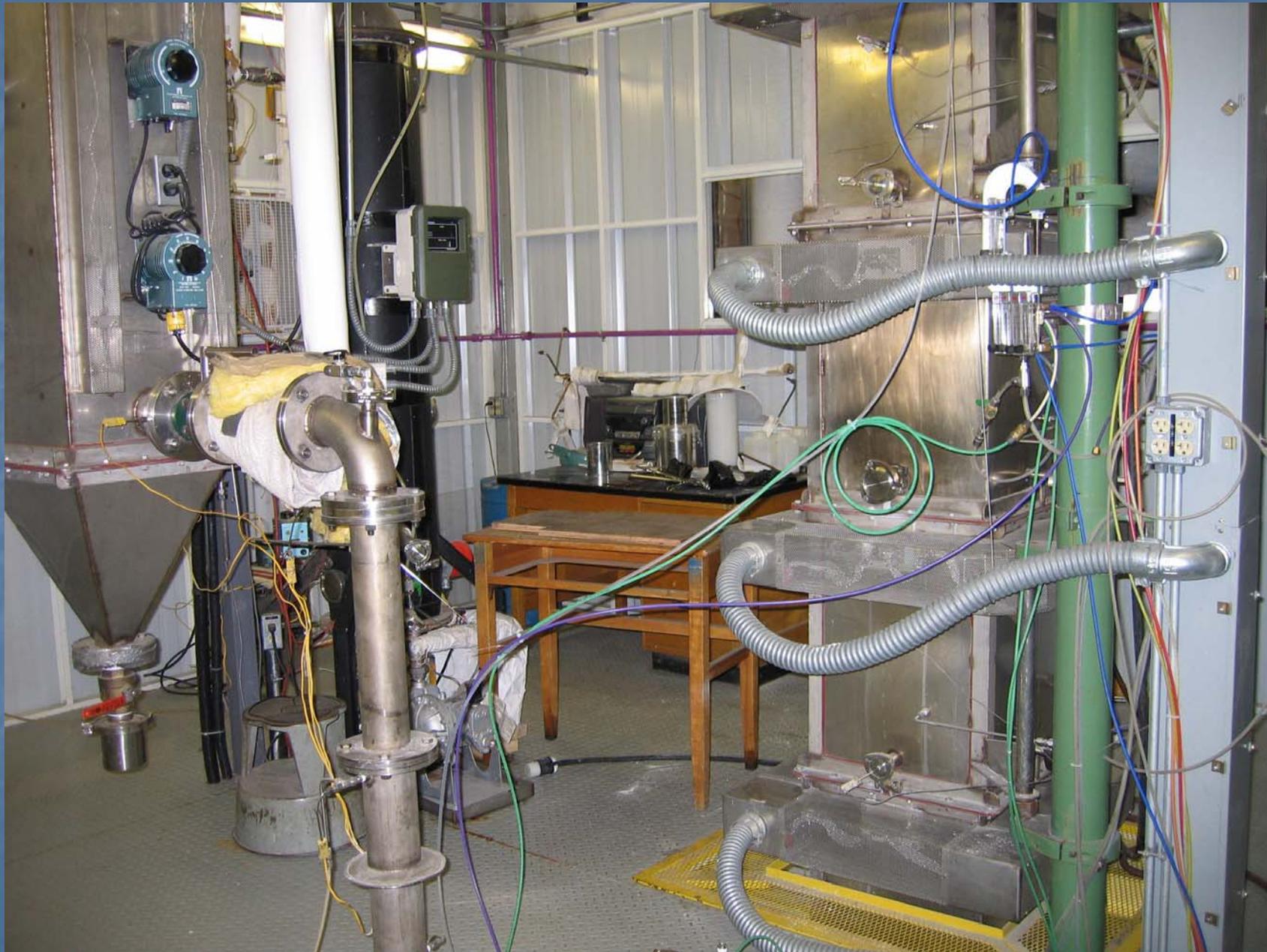
# Scale-Up

- The major results observed at the University of Mississippi have been demonstrated in a 7-kW pilot scale combustion facility at the University of North Dakota's Energy and Environmental Research Center (UND EERC)
  - Single-staged, simulated reburning – completed
  - Two-staged, simulated (reburn + burnout)
    - Reburn with lignite char
    - Reburn with biomass ash

Third floor



## Second floor



First floor



# First floor



# Results of Scale-Up of Mixed Fuel Reburning using Natural Gas and Lignite Flyash

Reburn Fuel	at 1100°C NO, ppm	at 1450°C NO, ppm
Gas	86	26
Gas + Ash	74	73
Gas + Ash + 1600 ppm SO <sub>2</sub>	77	65

- Gas reburn data at both temperatures are consistent with the bench-scale data collected at UM
- HCN could not be reliably measured (due to condensation on downstream metals), but the trend is consistent with those taken at the University of Mississippi
- Effects of SO<sub>2</sub> is nominal

# Results of Scale-Up of Mixed Fuel Reburning using Natural Gas and Biomass Flyash

Reburn Fuel	at 1100°C NO, ppm	at 1450°C NO, ppm
Gas + Ash	62	144
Gas + Ash + 1600 ppm SO <sub>2</sub>	75	150

- Reburn by (gas and ash) at 1100°C is consistent with the bench-scale data collected at UM
- HCN could not be reliably measured (due to condensation on downstream metals), but the trend is consistent with those taken at the University of Mississippi
- Effects of SO<sub>2</sub> is nominal

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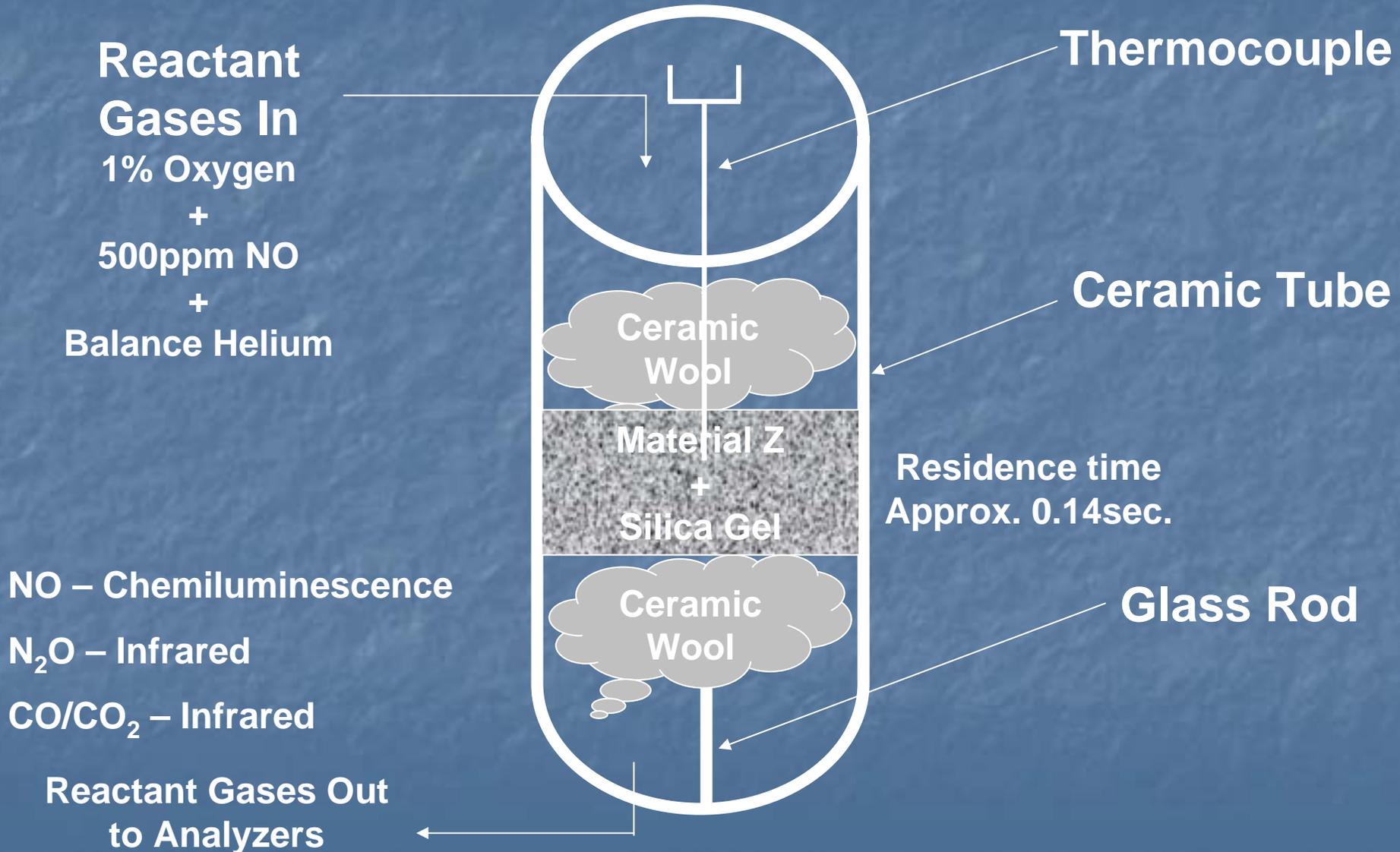
# Post-Combustion Reduction of Nitrogen Oxide

- Mechanistic information from recent studies (Chen and Tang, 2001) have provided basis for the design of a novel reburning fuel
- These principles have been adopted by Fan and Gupta (2001) in designing post-combustion NO reduction technology using lignite and bituminous chars
- We would like to investigate if the mechanistic information can be applied to post combustion zone where temperature is low and O<sub>2</sub> is in excess

# Fixed-Bed Experimental Setup

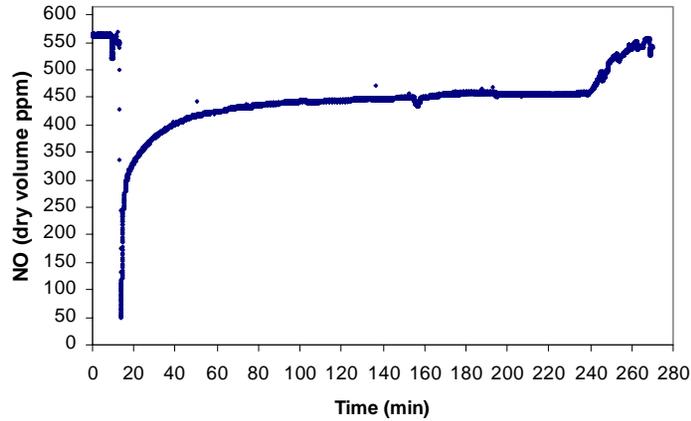
- Alumina tube with a fixed bed of 3 gm of material Z.
- Simulated flue gas containing 1% O<sub>2</sub>, 500ppm NO and the balance helium.
- Material Z is diluted using silica gel particles at 1-to-2 weight ratio to ease the pressure and temperature fluctuations.

# Fixed Bed Setup

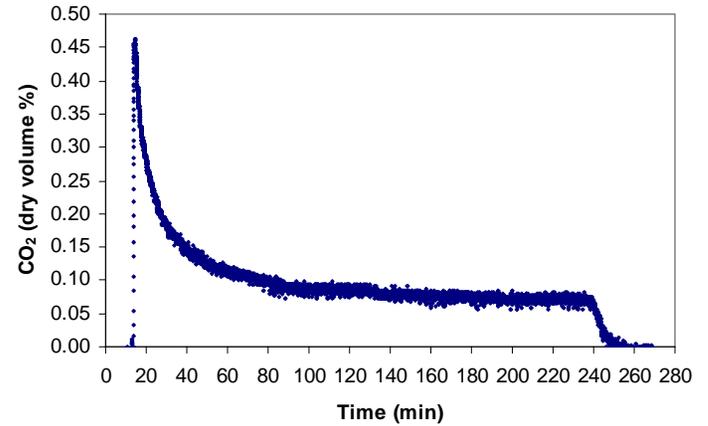


# Sample Raw Data

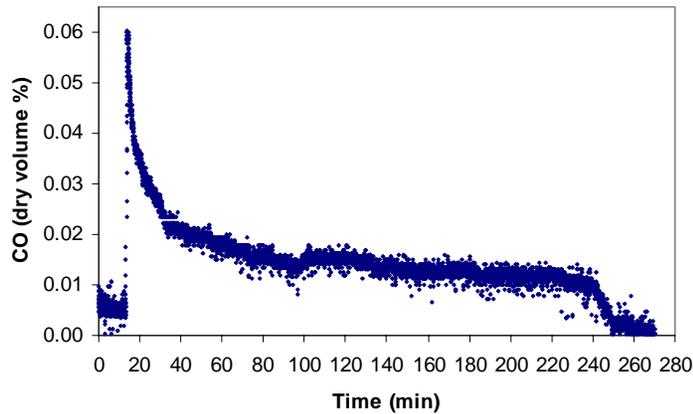
NO vs. Time



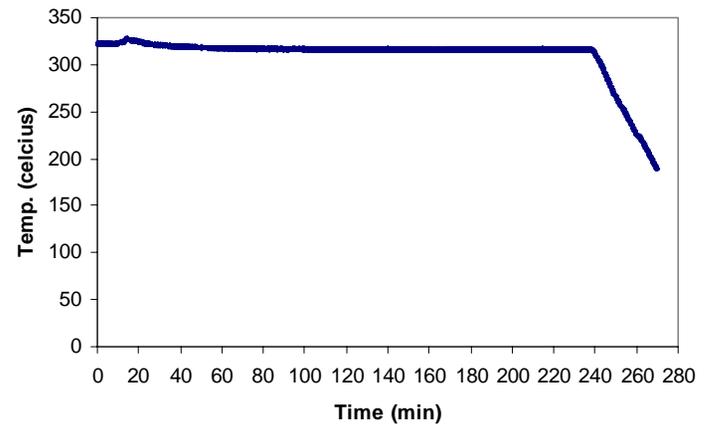
CO<sub>2</sub> vs. Time



CO vs. Time



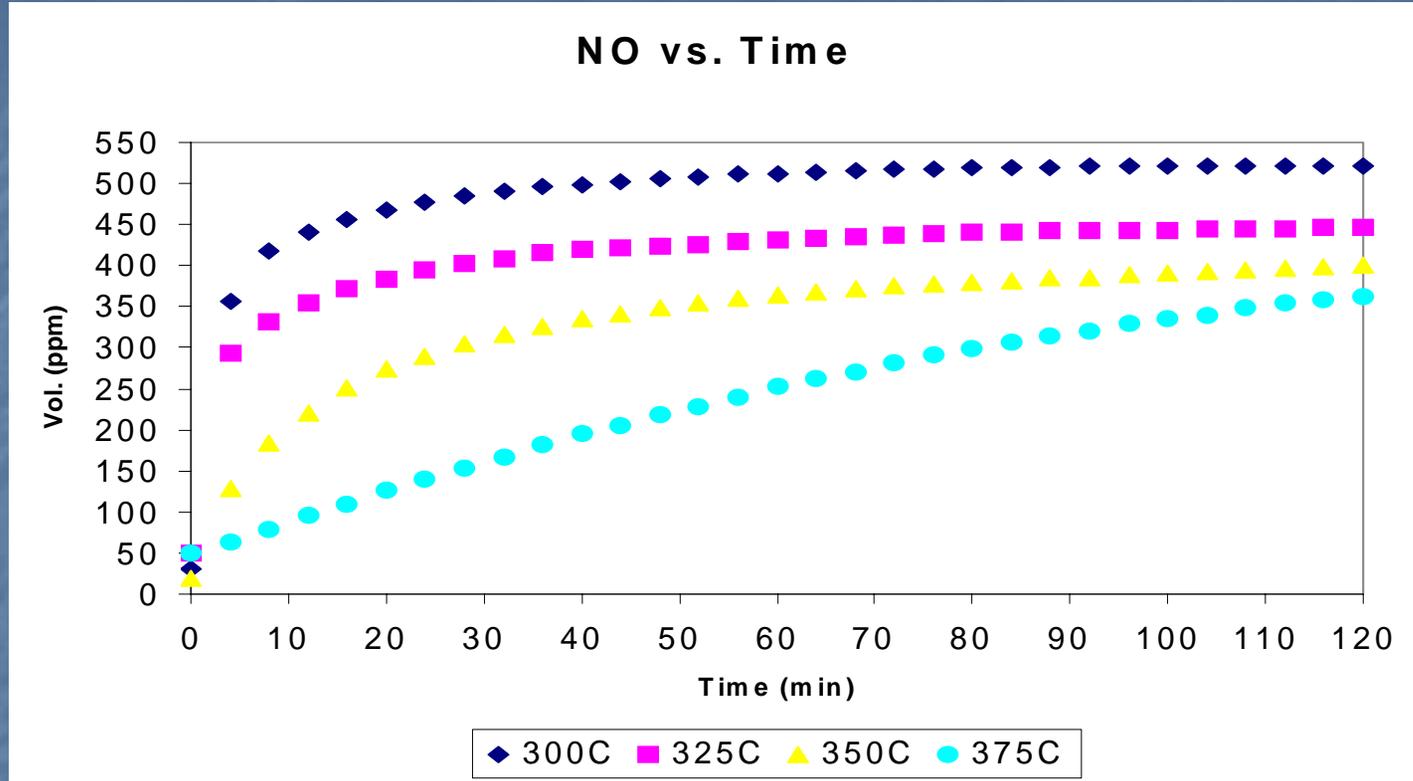
Bed Temp. vs. Time



# Kinetic Analysis

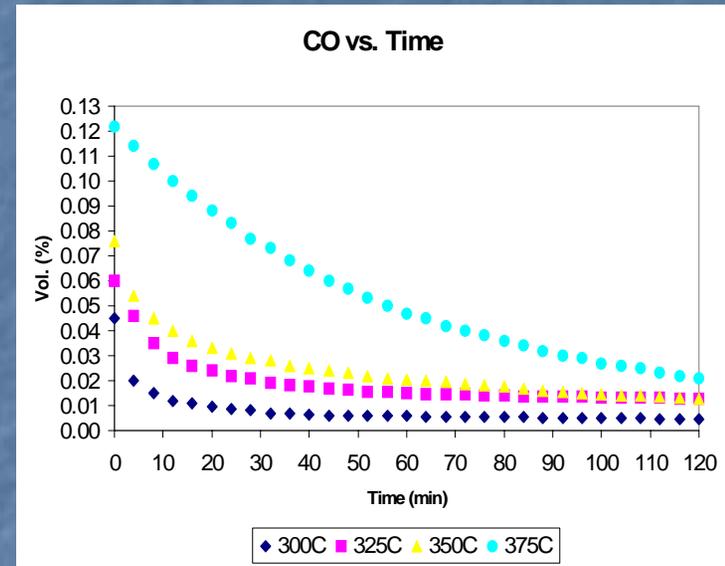
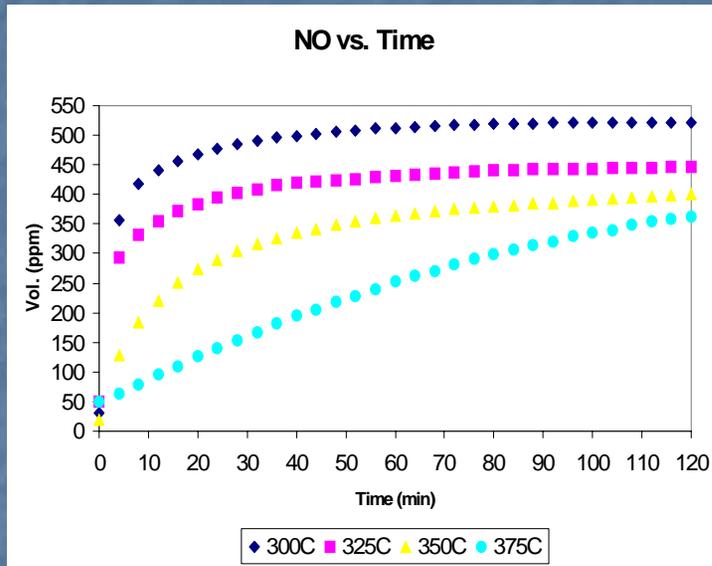
- Reaction rate constants are determined using power-law kinetics (with char deactivation) for carbon oxidation – not discussed
- Basic power-law kinetics with reaction order = 1 for NO reduction

# NO Reduction Results



- Remarkable efficiency of material Z seems promising for the development of a post-combustion technology for NO abatement.
- Structures of the NO dynamics suggest they may follow different physical or chemical mechanisms at 375°C and above.

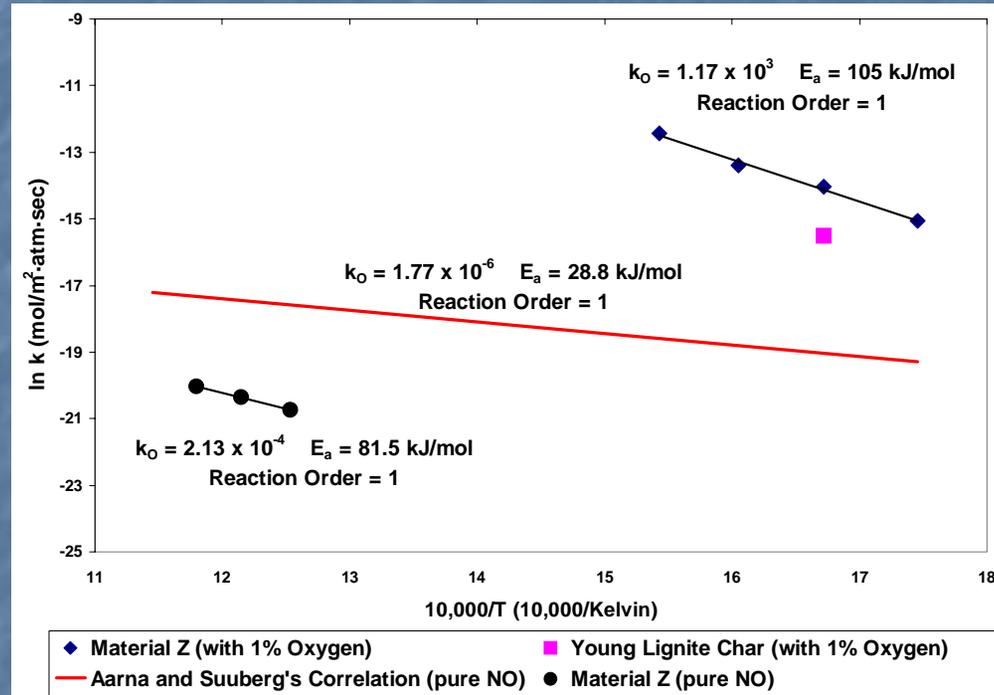
# NO Reduction Results



- These NO structures appear to be closely related to CO structures, and therefore, the rate of carbon oxidation

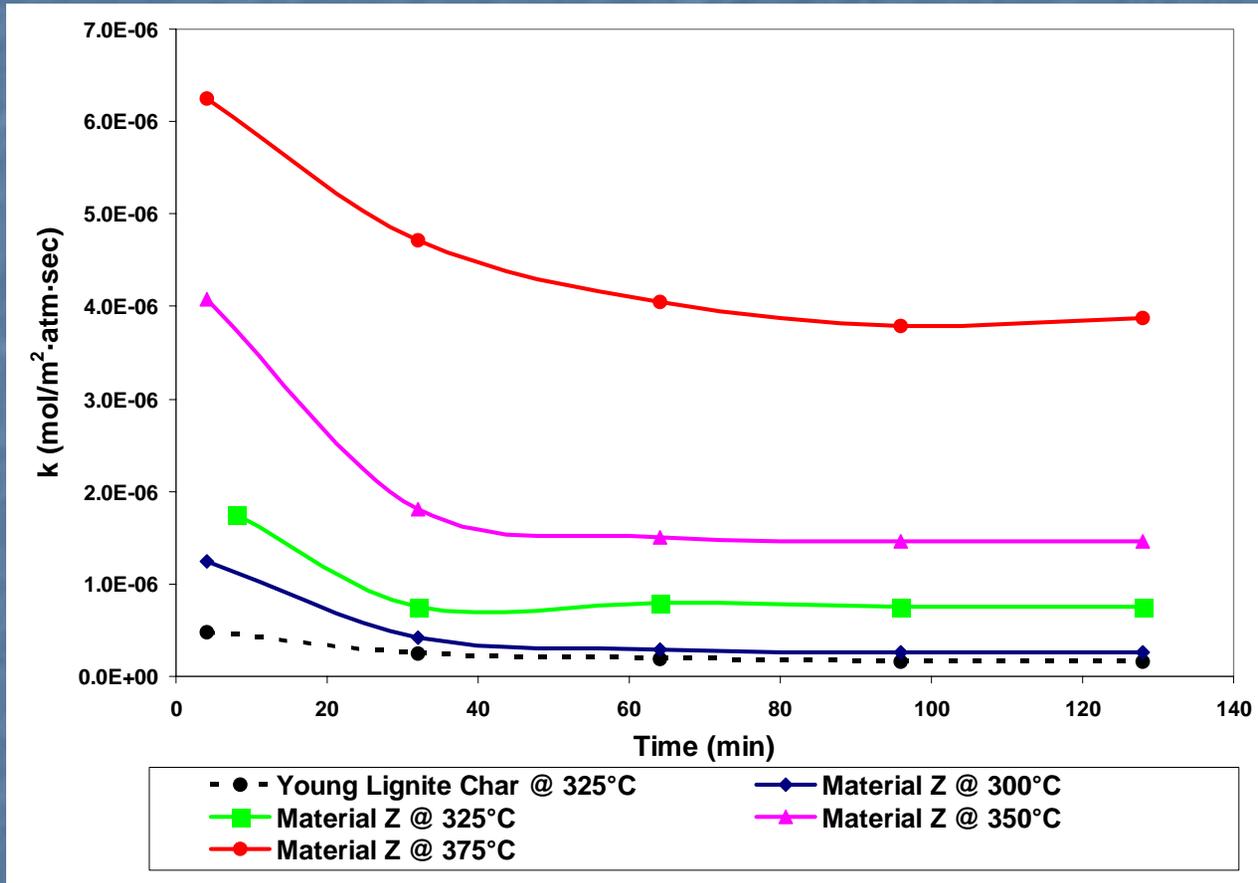
# Kinetic Analysis of NO Reduction

Rates are based on data after 60 min



- The presence of a small amount of oxygen enhances NO reduction
- This increase may be due to formation of CO which reacts with the NO

# Kinetic Analysis of NO Reduction



- Material Z deactivates faster than young lignite char.

# Simulation of NO Reduction

## ■ Power-Law Kinetics

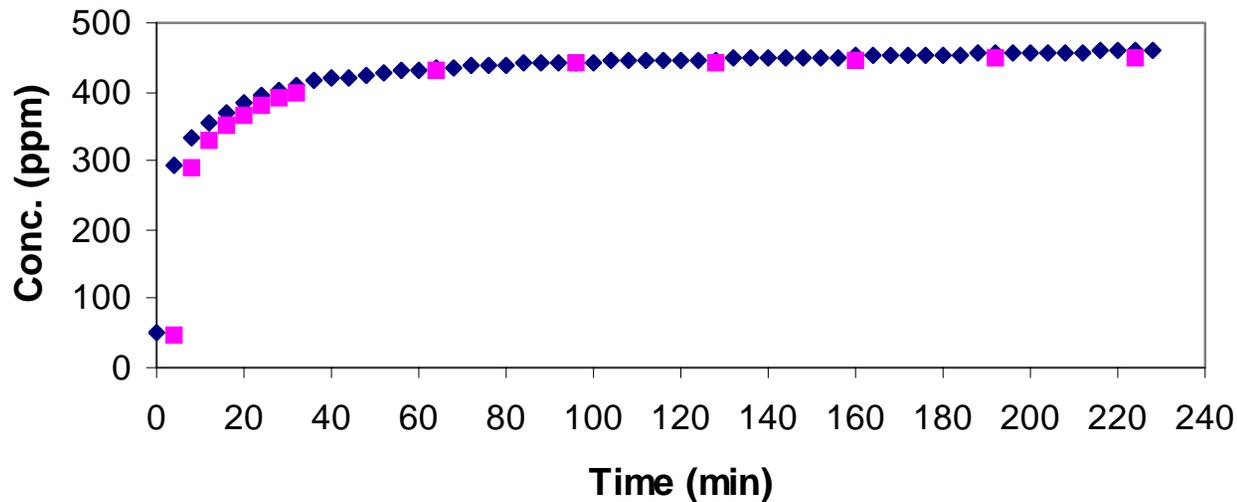
$$r = k \cdot P_{\text{NO}}^m \cdot A$$

$$m = 1$$

### NO Concentration at Reactor Outlet

using Carbon Simulation results

Temp. = 325 °C, Oxygen = 1%, NO = 560ppm



◆ Experimental ■ Simulation

# Conclusions on Post-Combustion NO Control

- Remarkable NO conversion with a contact time of only about 0.14 s
- High NO reduction is accompanied with high CO concentration, signifying the importance of previously revealed mechanisms (Chen and Tang, 2001)
- Study of catalysis of CO oxidation continues
- An invention disclosure has been filed

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- Char Nitrogen Pathway - Two-Stage (Reburn + Burnout) Experiments

# Char Nitrogen Pathway – Two-Stage (Reburning and Burnout) Experiments

## ■ Objectives

- to seek potential NO control methods through better understandings of the char nitrogen pathways
  - to find the temperature and residence time effects on the total fixed nitrogen speciation in flame environments; i.e., temperatures between 1150 and 1700°C
  - To find the extent of HCN and NH<sub>3</sub> recycling to NO in the burnout zone
- 
- Use of high-temperature furnace (up to 1700°C) for reburning stage
  - Use of lower-temperature furnace (up to 1150°C) for burnout stage

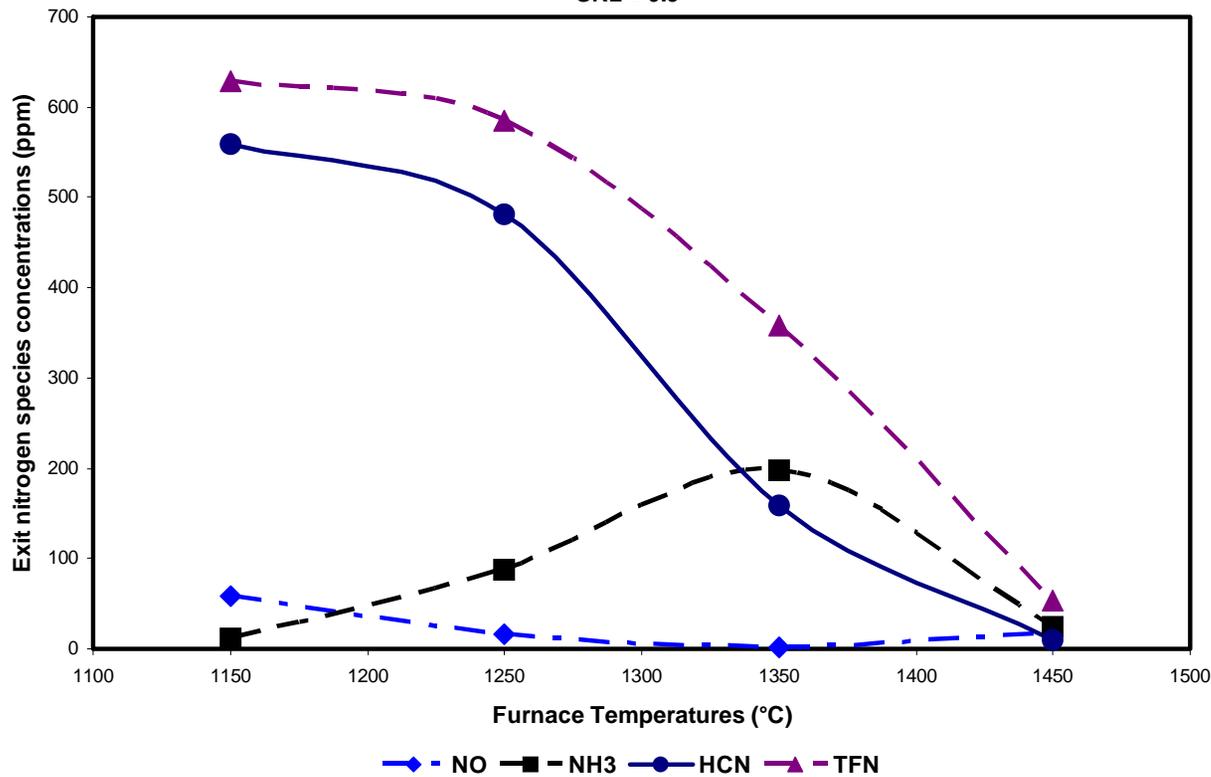


# Initial Two-Stage Results

- Experiment at (reburning temp. =  $1150^{\circ}\text{C}$  and burnout temp. =  $1150^{\circ}\text{C}$ ) indicate there is HCN recycling to NO in the burn-out stage
  - confirms our previous assumption of 60% recycling
  - will this assumption valid in the flame region,  $1400^{\circ}\text{C}$ ?
- Two-stage experiment at (reburning temp. =  $1450^{\circ}\text{C}$  and burnout temp. =  $1150^{\circ}\text{C}$ ) indicate no HCN recycling to NO in the burn-out stage
  - reburning temperature,  $1450^{\circ}\text{C}$ , too high?
  - residence times, 0.9 s, too long?
  - wall-catalyzed reactions?

# Partitioning of Nitrogen Species during Natural-Gas Reburn at Temperatures above 1150°C

NO feed concentration = 1000ppm  
Furnace Temperatures = 1150 °C to 1450 °C  
Feed flow rate = 2 L/min  
Residence time = ? sec  
SR2 = 0.9



- Reburn stage HCN yields at 1400°C are lower than expected
- Current residence time, 0.9 s, in reburn zone is longer than that in the actual flames
- Roles of reactor wall?
- Role of mixing in actual furnace?

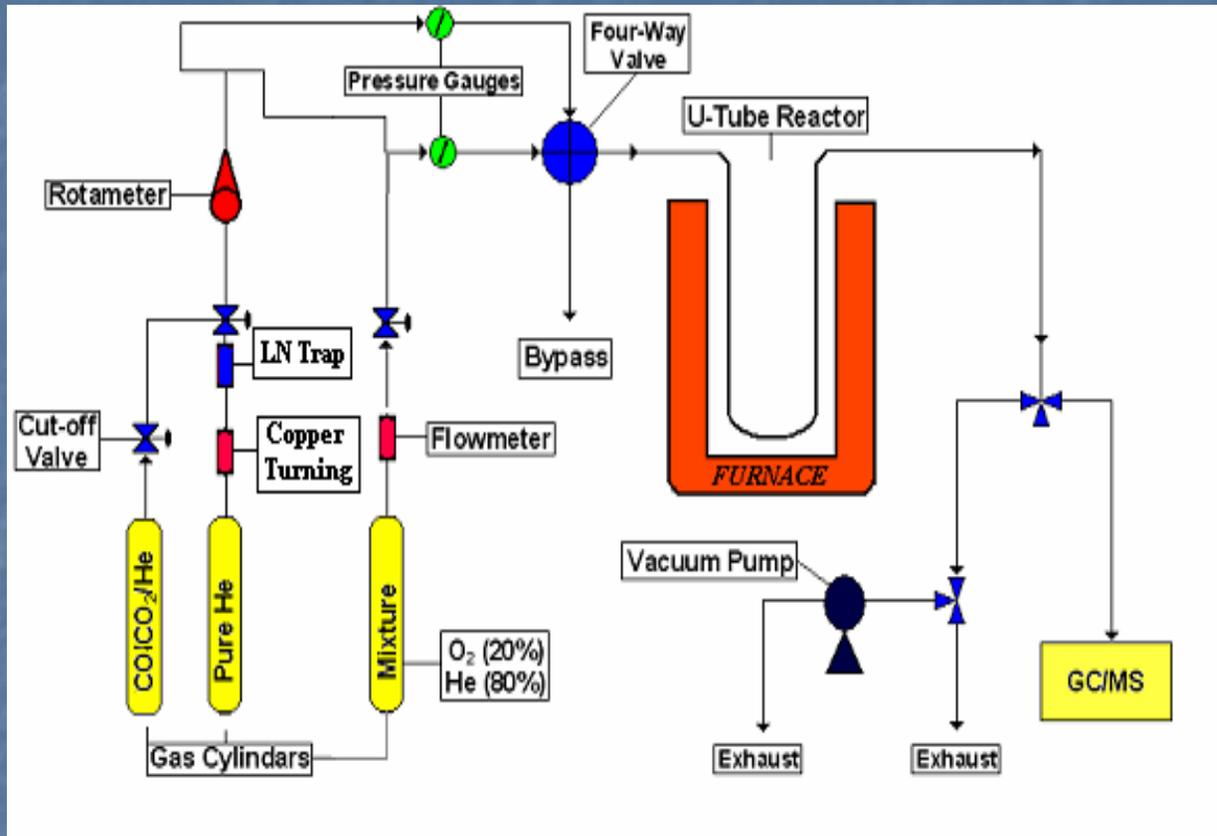
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# Measuring Char Reactivity

- Traditional method
  - coal devolatilization and char oxidation with separate procedures in laboratories
  - thermally treat the chars in an inert gas at about 1000 °C for about *1 to 3 hr*
- Nevertheless,
  - devolatilization and burning in typical flames usually take place *within seconds*
  - *char deactivation* is a known phenomenon
- Reactivity of “young” chars and char deactivation in flames consist a *missing link*
- Our objectives are to
  - investigate the reactivity of char
    - produced from pyrolysis and combustion at flame temperatures, 1100 to 1700°C
    - residence times in an order of seconds
  - quantify char deactivation in-situ by measuring the number distribution and strength of surface oxides

# Experimental Apparatus



- U-shaped, semi-flow ceramic reactor
- desorption of surface oxides is conducted in-situ where the char is produced
- char sample does not have contact with any reactive agents before the TPD experiments
- analyzed by an online GC/MS system



# Particle Injection Methods

## ■ Original Design

- mechanically pushed by a stainless steel piston
- susceptible to small leakage of air

## ■ Improved Design

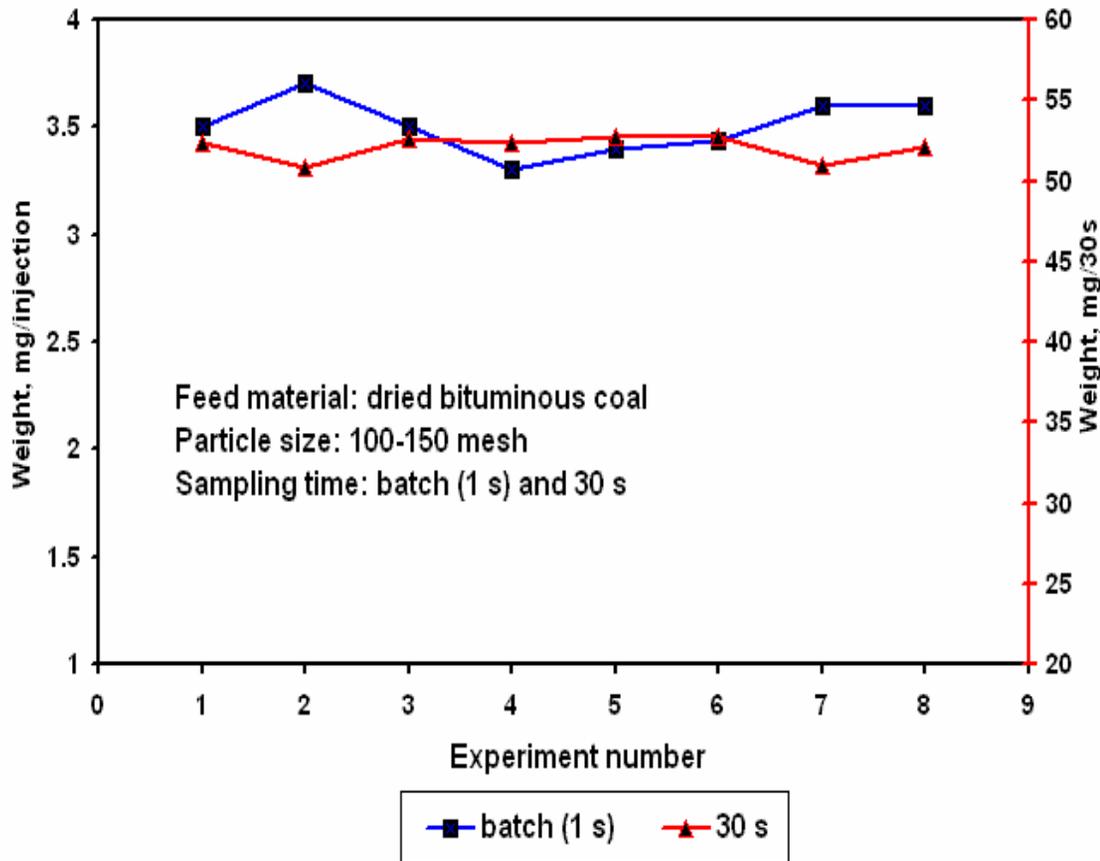
- sample blown into the reactor by pressurized He
- pressure fluctuations lasts a few seconds – not desirable for the study of young chars

## ■ New Design

- no moving parts at periphery and leakages are prevented
- no pressure disturbances
- feeding at batch or practically continuous mode
- over a wide range of feed rate
- low cost
- applications in both combustion and pharmaceutical research labs; an invention disclosure is planned

# New Particle Feeder

Short-term and Long-term stabilities



The fluctuations during batch (1 injection per s) and continuous feedings are both within 5%

The variance for batch feeding is  $0.0014 \text{ mg}^2$ , and that for continuous feeding is  $0.608 \text{ mg}^2$

The average feed rate at continuous mode (30 s or 30 injections) is 52.1 mg; only 0.9% different from that calculated based on batch mode, 52.5 mg

# Reactor Tubes and Supporting Materials

- **Reactor Tubes**
  - U-shaped alumina tube (consist of 99.7% of  $\text{Al}_2\text{O}_3$ )
  - Straight alumina tube with 99.8% of  $\text{Al}_2\text{O}_3$
  - Hexoloy SA/SP SiC tube (98%SiC)
- **Supporting materials**
  - Ceramic wool, chips, alumina beads
  - SiC powder, SiC foam and SiC rod

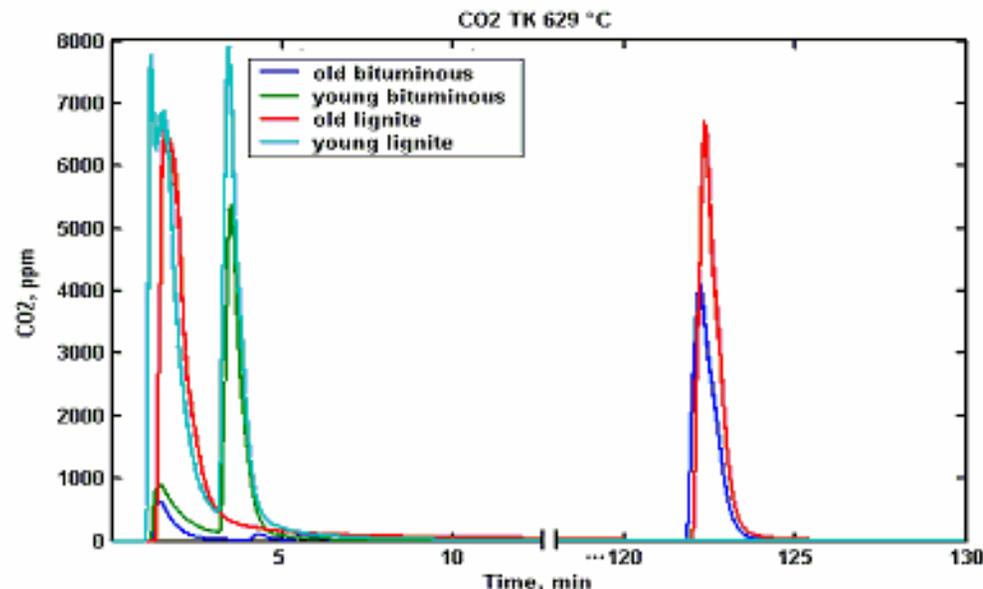
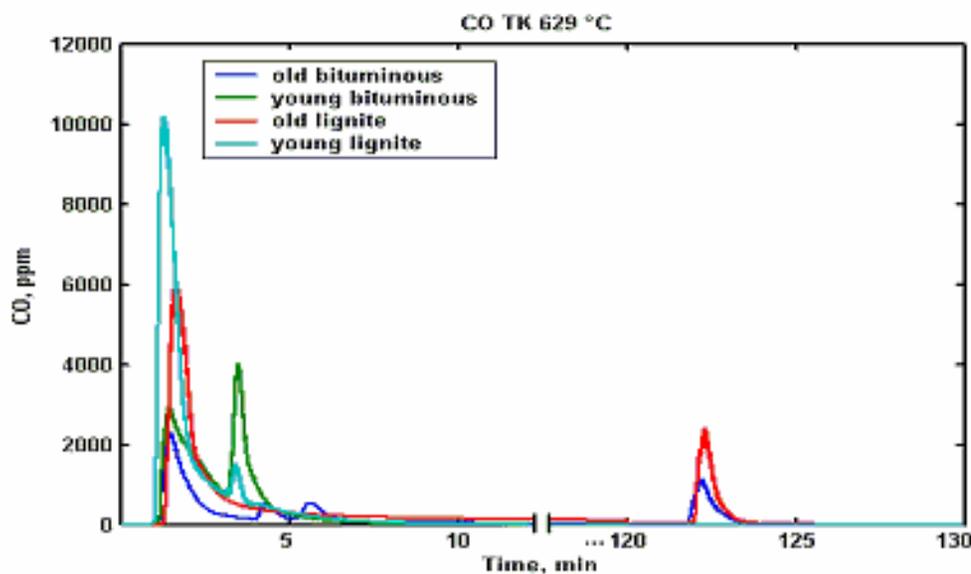
# Pyrolysis, Oxidation, and Transient Kinetics (TK)

- Injecting 1gm sample into the preheated reactor at 629°C or 1400°C with He flow
- After 2 min or 2 h pyrolysis time, young or old char, switching the gas to a mixture of 20% O<sub>2</sub> balanced with He at 0.8 L/min for 15 s

## Temperature-Programmed Desorption (TPD)

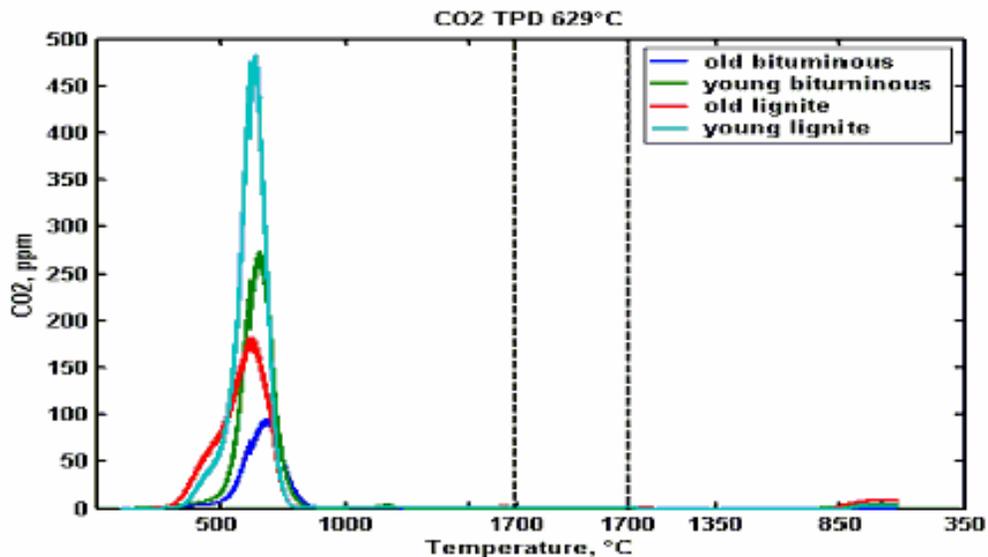
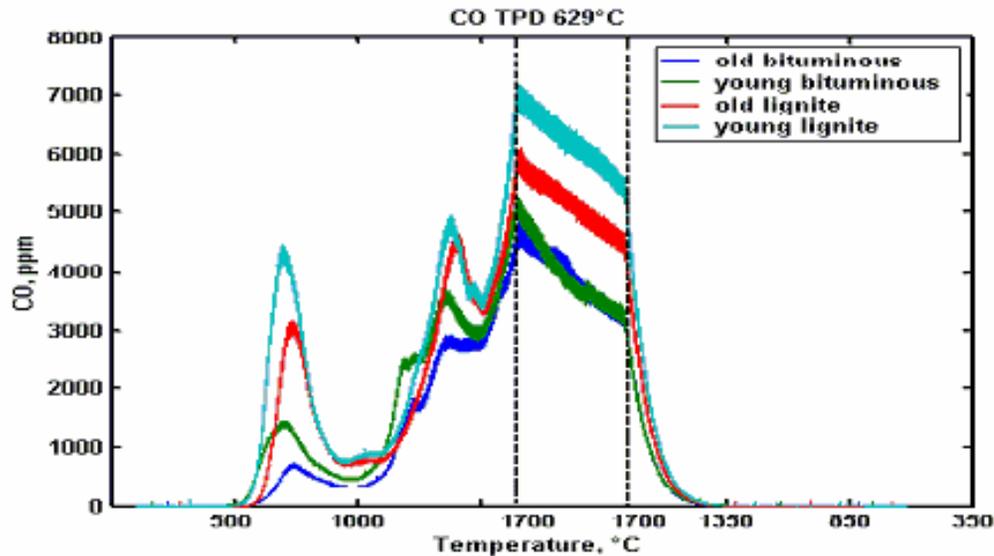
- Cooling naturally to 100°C, TPD was initiated immediately with 5°C/min heating rate up to 1700°C and with 80 ml/min He
- In the study of reactions involving reactor wall and sample support materials, the same heating and cooling profiles as above are adopted

# Oxidation and Transient Kinetics (TK) at 629°C



- Young chars produces more CO and CO<sub>2</sub> than old chars during TK. Young lignite char produces more CO<sub>2</sub> but less CO than bituminous coal chars.
- The faster decay of oxides from young chars (of both lignite and bituminous coal) suggests higher turnover rates of oxygen on young chars.

# TPD Spectra of Char Oxidized at 629°C



- Young chars produce more CO and CO<sub>2</sub> during TPD, lignite char more than bituminous coal char
- Peaks at about 725°C are surface oxides well documented
- Peaks at about 1430 and 1700°C are not well documented, which became the focal points of our study

# CO Production at 1430°C during TPD

- Pan and Yang (1992) reported surface oxides on the *basal planes* of graphite that fully desorbed at 1500°C for 3 h
- Senneca et al. (2004) presented HRTEM images illustrating the development of structural anisotropy during char oxidation, from which they postulate the possibility of formation of oxides on the *basal planes* of coal-derived chars.
- Our data seem to suggest the formation of stable surface oxides form on the basal planes of the chars
- The activation energy of char oxidation is expected to be higher than 300 kJ/mol

# Surface Oxides and Activation Energy

		Surface Oxide, mL/gm coal			Oxygen Balance <sup>(a)</sup>	Rate Constants <sup>(b)</sup>		Rate Constants <sup>(c)</sup>	
		TK, 629°C	TPD, <1100°C	TPD, 1430°C	Fraction	K0, Min <sup>-1</sup>	E, kJ/mol	K0, Min <sup>-1</sup>	E, kJ/mol
Young Lignite	CO	1.33	14.36	4.00	0.37	2.57*10 <sup>5</sup>	130	7.95*10 <sup>12</sup>	443
	CO <sub>2</sub>	3.91	0.91						
Old Lignite	CO	1.05	10.03	3.68	0.29	1.2*10 <sup>7</sup>	170	1.40*10 <sup>12</sup>	423
	CO <sub>2</sub>	3.52	0.56						
Young Bituminous	CO	2.21	6.56	3.84	0.23	1.22*10 <sup>4</sup>	107	1.12*10 <sup>9</sup>	317
	CO <sub>2</sub>	2.40	0.57						
Old Bituminous	CO	0.58	3.22	2.07	0.14	6.98*10 <sup>4</sup>	126	6.25*10 <sup>8</sup>	309
	CO <sub>2</sub>	2.42	0.25						

(a) Oxygen balance is computed as  $(0.5\text{CO} + \text{CO}_2) / (\text{O}_2)_{\text{input}}$

(b) If K0 is set at 10<sup>10</sup> min<sup>-1</sup>, E = 219.3 kJ/mol, 228.6 kJ/mol, 227.1 kJ/mol, 228.5 kJ/mol

(c) If K0 is set at 10<sup>10</sup> min<sup>-1</sup>, E = 352.1 kJ/mol, 355.8 kJ/mol, 346.8 kJ/mol, 343.8 kJ/mol

# CO Production at 1700°C during TPD

## - In Search of Oxygen Source for CO Production

### ■ Oxidative impurity in He? NO

- He was passed through a copper-turning bed maintained at 520°C, followed by a column of MS-13X molecular sieve immersed in liquid N<sub>2</sub> at 77 K
- TPD of pyrolyzed char (without oxidation) also demonstrates the same amount of CO yields
- The He purification procedure was adopted in routine experiments

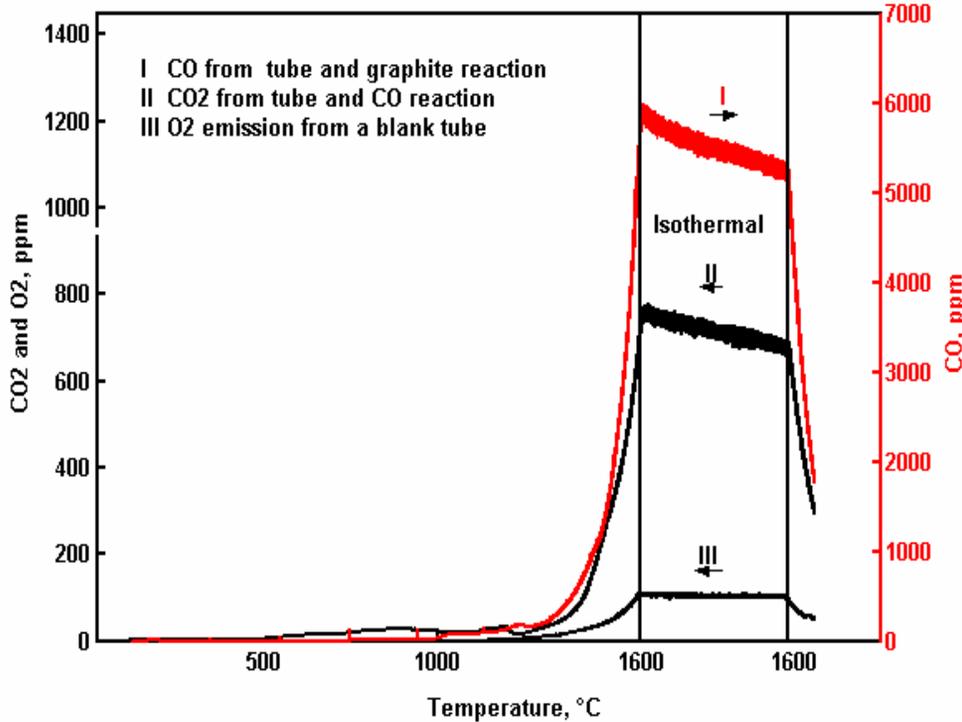
### ■ Inorganic compounds in coal? NO

- TPD of graphite also demonstrates the same amount of CO yields

### ■ Wall reaction in alumina tube?

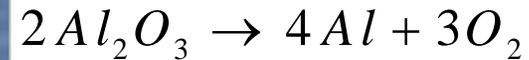
- our focus is then shifted to the roles of reactor tube and its support materials

# Reactions of Alumina Tube



- gas flowrate = 80 mL/min
- heating rate = 5 °C/min

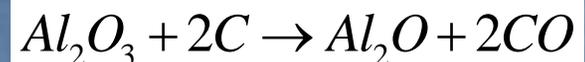
Alumina decomposes to Al<sub>2</sub>O



O<sub>2</sub> partial pressure is  $4.642 \cdot 10^{-26}$  bar at 1600 K (JANAF Table)

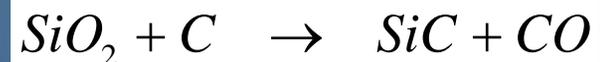


Al<sub>2</sub>O partial pressure  $1.695 \cdot 10^{-15}$  bar at 1600 K (JANAF Table)

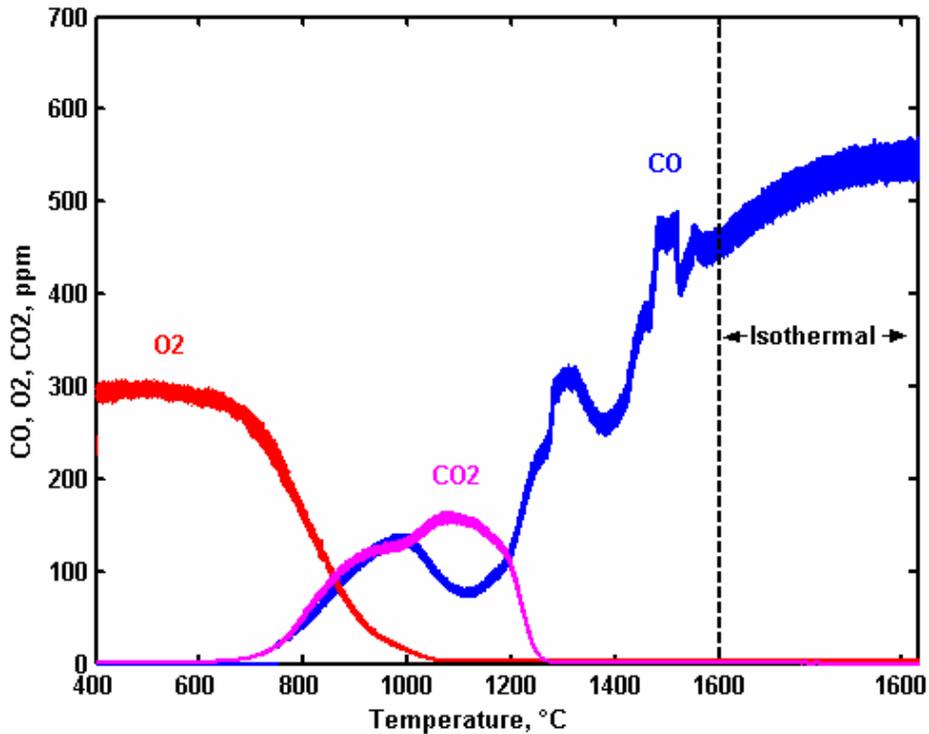


CO partial pressure  $6.382 \cdot 10^{-3}$  bar at 1900 K - consistent with data on the right

The alumina U-tube has small amount of SiO<sub>2</sub>

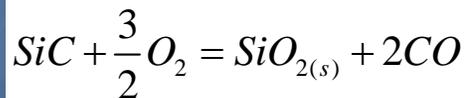


# SiC Tube Oxidized by O<sub>2</sub>

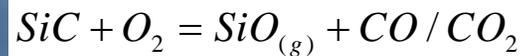


- This experiment is heated up to 1600°C at 5°C /min with a gas flow rate of 80 ml/min
- The straight SiC tube is heated with O<sub>2</sub> gas. The oxidation began at about 650°C. CO<sub>2</sub> and CO was formed in the beginning, and then only CO was produced after about 1250°C

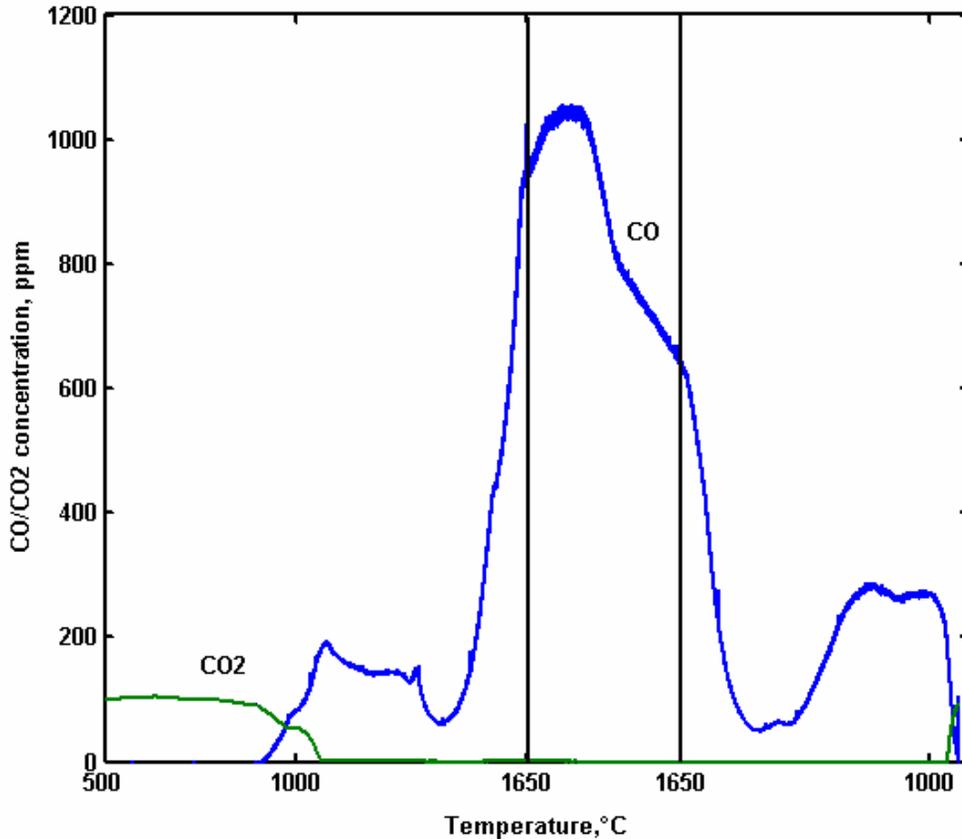
Passive oxidation



Active oxidation



# Straight SiC Tube Oxidized by CO<sub>2</sub>

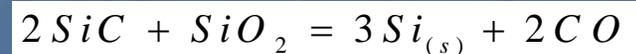
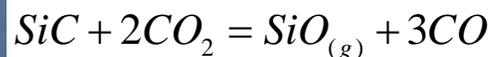


- The straight SiC tube is heated with a gas containing 100 ppm of CO<sub>2</sub> gas with a flow rate at 80 mL/min and a heating rate of 5°C/min
- Production of high CO is from both active and passive oxidation of SiC
- High production of CO is from the reduction of previously formed SiO<sub>2(s)</sub> to SiO<sub>(g)</sub>, which disappear after repeated heating; fresh SiC surface emerges after Si<sub>(g)</sub> is swept away

Passive oxidation



Active oxidation



# Strategy for the Study of TK and TPD of Young Chars at High Temperatures

- Alumina is not suitable for reducing environments
- SiC is not suitable for oxidative environments
- Two-staged experiments
  - alumina tube for oxidation
    - residence time, controlled by gas flowrate, is reasonably close to flame conditions, 0.2 to 0.5 s
  - SiC tube for TPD and for supporting the sample
    - although SiC can be oxidized by desorption product,  $\text{CO}_2$ , its production is known to be low above  $900^\circ\text{C}$
- New particle injection port without moving part at system periphery



# Conclusions

- Young char contains more abundant surface oxides than those on old char over a wide range of temperature. Oxygen turnover rate on young char is much higher than that on old char.
- The TPD peaks appearing around 725°C for different chars show an activation energy varied in the range of 107~170 kJ/mol; the peaks at 1430°C, corresponding to the activation energy over 300 kJ/mol, signifying the possible roles of strongly bound oxides on the basal planes.
- The alumina tube releases O<sub>2</sub> and reacts with carbon to form CO and Al<sub>2</sub>O at high temperature.
- SiC tube reacts with oxidants like O<sub>2</sub>, CO<sub>2</sub> and H<sub>2</sub>O.
- SiC foam, rods and powders after heat-treatment can be used to support the sample in the SiC tube.

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