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Development of Disposal Sorbents for Chloride Removal from High-**Temperature Coal-Derived Gases**

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CONTRACT INFORMATION

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Period of Performance

September 30, 1993 to September 29, 1995

Schedule and Milestones

FY95 Program Schedule

Task	s	0	N	D	J	F	М	Α	М	J	J	Α	s
Sorbent Preparation and Characterization	23333	 											
Provision of a Bench-Scale Unit	200000			********									
Bench Scale Testing			********	**********		00000000	200000000	00000000					
Parametric Testing											0000000		
Data Analysis	200000	**********	**********	**********		000000000	000000000	00000000	00000000	000000000	0000000		
Final Report												30000000	00000000

OBJECTIVE

The objective of this program is to develop alkali-based disposable sorbents capable of

reducing HCl vapor concentrations to less than 1 ppmv in coal gas streams at temperatures in the range 400° to 750°C and pressures in the range 1 to 20 atm. The primary areas of focus of this

program are investigation of different processes for fabricating the sorbents, testing their suitability for different reactor configurations, obtaining kinetic data for commercial reactor design, and updating the economics of the process.

BACKGROUND INFORMATION

The integrated gasification combined cycle (IGCC) process produces electricity from coal with high efficiency by converting the fuel to coal gas that drives a combustion gas turbine or feeds molten carbonate fuel cells. Both generating methods require relatively contaminant-free gas. Hence, methods must be incorporated between the gasifier and the electricity generator to remove impurities that are formed during gasification from the sulfur, chlorine, nitrogen, and heavy metal constituents indigenous to the coal. One highly undesirable impurity is hydrogen chloride (HCl), a reactive, corrosive, and toxic gas.

The concentration of HCl in coal gas has not been documented extensively, but estimates suggest that it varies widely in the range 1 to 500 ppmv [TRW, 1981]. Bakker and Perkins [1991] noted that concentrations of HCl in coal gas are likely to be about five times greater than those in a coal-fired boiler flue gas stream because of the low specific volumetric flow rate of the coal gas stream. The actual concentration of HCl vapor in a coal gas stream will depend on the chlorine content of the coal, the gasification temperature, and the type of gasifier. Recently, the concentration of HCl vapor was found to be about 300 ppmv in the gas stream from a fixed-bed gasifier using a coal containing 0.24 wt% Cl [Gal et al., 1994].

The effect of impurity HCl in coal gas that fuels a gas turbine is not well defined, and currently no concentration limit standards exist. Nevertheless, the removal of HCl vapor from the gas can only be beneficial in any generating plant, because of the great corrosion potential of the vapor in contact with metal components. Perkins et al. [1990] report that chloride deposits on syngas coolers accelerated the rate of corrosion of the heat exchanger material. HCl reacts with the deposited slags forming low-melting iron chlorides, and thereby accelerates the corrosion rate.

Although high-temperature molten carbonate fuel cells (MCFC) for IGCC power generation

plants are still in the developmental stage, they require a feedgas free of contaminants such as particulate matter, sulfur, and chloride species. During sustained operation, chlorides are deleterious to MCFCs because they can lead to severe corrosion of hardware [Kinoshita et al., 1988]. HCl also can react with the molten carbonate electrolyte to form halides such as LiCl and KCl. The high vapor pressures of these compounds enhance electrolyte loss. An increase in the cell resistance and a corresponding decrease in the cell voltage were observed in feed gas containing 1 ppmv HCl vapor [Pigeaud and Wilemski, 1992] Hence, the allowable HCl concentration in a MCFC feedgas is estimated to be less than 0.1 ppmv.

Available processes for removing HCl vapor from industrial and incinerator waste gases scavenge HCl by adsorption on activated carbon or alumina, or by reaction with alkali or alkaline earth carbonates or oxides. Typically, commercial sorbents called chloride guards are used for HCl vapor removal from chemical plant process feed stocks. These sorbents reduce chloride contaminant levels to less than I ppmv, but they must operate at temperatures less than 450°C. They are also relatively expensive, and cannot be economically regenerated. Hence, they are not suitable for chloride removal from hot coal-derived gas streams. Inexpensive, efficient, and disposable sorbents are needed for these applications.

Equilibrium thermodynamic calculations showed that only Na- and K-based sorbents are capable of reducing HCl levels to less than 1 ppmv at 500°C. Because NaCl has a lower vapor pressure than KCl and sodium minerals are more abundant than potassium minerals, Na-based sorbents were selected as candidate materials [Krishnan et al., 1986].

In a previous program, laboratory- and benchscale experiments were performed by SRI International [Krishnan et al., 1986] to evaluate three natural carbonate minerals—nahcolite (NaHCO₃), shortite (Na₂CO₃·2CaCO₃), and dawsonite (NaAl(OH)₂CO₃)—as HCl scavengers for simulated coal gas. All the tested sorbents reacted rapidly with HCl vapor and reduced the HCl vapor concentration from about 300 ppmv to about 1 ppmv. The performance of nahcolite was superior in its adsorption capacity; the spent sorbent contained up to 54 wt% chloride. Furthermore, the presence of H_2S and trace metal impurities in the coal gas did not significantly affect the performance of the bed for HCl sorption. An economic evaluation for HCl cleanup costs in a 100 MWe plant indicated that the use of nahcolite to remove HCl vapor would add only about \$0.002/kWh (2 mills/kWh) to the cost of the generated electric power.

Thus, alkali-based naturally occurring minerals have been shown in bench-scale experiments to be capable of reducing HCl vapor levels to about 1 ppmv in simulated coal gas streams.

PROJECT DESCRIPTION

The current program aims to develop alkalibased disposable sorbents capable of reducing HCl vapor in high-temperature coal gas streams to less than 1 ppmv in the temperature range 400° to 750°C and the pressure range 1 to 20 atm. Investigating different processes for fabricating the sorbents, testing their suitability for different reactor configurations, and updating the economics of the process are the primary areas of focus of this program. This project is a collaborative effort between SRI, the prime contractor, and subcontractors Research Triangle Institute (RTI) and General Electric Corporate Research and Development (GE-CRD). RTI is developing and testing sorbents for fluidized-bed reactors, while GE-CRD is preparing sorbents for moving-bed reactors. SRI is preparing and testing sorbents for fixed-bed reactors.

The program is divided into the following six tasks: (1) information required for National Environmental Policy Act (NEPA), (2) sorbent preparation and characterization, (3) provision of bench-scale test unit, (4) bench-scale testing, (5) parametric testing, and (6) data analysis.

In the first-year of the project, several sorbent formulations were prepared for both fixed- and fluidized-bed applications [Krishnan et al., 1994]. Based on results of earlier studies, nahcolite, an abundantly available natural mineral, was selected as the primary choice for the sorbent material. About 600 kg of nahcolite powder, obtained by solution mining from deposits near Rifle,

Colorado, has been provided free of charge by NaTec Resources, Inc., Houston, Texas. Sorbent samples were prepared by pelletizing or granulating the powder with binders and texturizing agents, using pilot-scale equipment. Nahcolite sorbent pellets for use in fixed-bed reactors were extruded in sizes varying from 1.5 to 5 mm diameter and they contained either bentonite or sodium silicate as a binder. The physical properties such as surface area and pore volume of the pellets were not significantly affected by the type of binder used. Differential reactor experiments showed that the sorbent pellets and granules are capable of absorbing large quantities of HCl vapor. The spent sorbents contained as high 55 wt% chloride. These results were confirmed with integral, fixedand fluidized-bed reactor experiments using simulated coal gas streams.

Two bench-scale stainless steel pressure vessels capable of operating up to 650°C and 20 atm were constructed. The system at SRI was used for fixed-bed reactor studies whereas the system at RTI was used fluidized-bed experiments. The sorbents were contained in 5-cm ID quartz tubes inside the stainless steel vessels and noncorrosive components of simulated coal gas mixtures were passed through the annulus between the quartz tubes and the stainless steel vessel to minimize corrosion. HCl and H₂S were added to this gas mixture inside the quartz tube, upstream of the sorbent bed. The pressure inside the reactor was controlled by a servo-controlled valve made of Hastelloy C metal. The gases leaving the reactor were cooled in a water-cooled heat exchanger made of glass. During cooling, the residual HCl vapor dissolved in the steam condensate and the chloride level in the condensate was determined using ion chromaography. The residual HCl vapor level was calculated from the condensate analyis and the procedure is capable of measuring sub-ppm levels of HCl vapor.

RESULTS

Fixed-Bed Reactor Studies

Fixed-bed reactor experiments showed that the pelletized nahcolite sorbents are capable of reducing HCl vapor level in simulated coal gas streams to less than 1 ppmv at 400°C (Figure 1). At a space velocity of 3000 h⁻¹, the HCl vapor

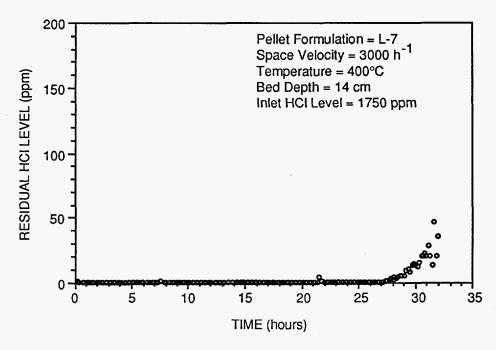


Figure 1. The HCl Breakthrough Curve of Nahcolite Sorbent Pellets in a Fixed-Bed Reactor

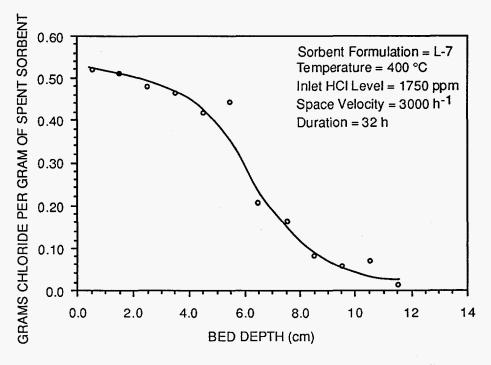


Figure 2. Distribution of Chloride in the Nahcolite Sorbent Bed

level was reduced from 1750 ppmv to less than 1 ppmv for nearly 25 h. The experiment was terminated when the residual HCl level reached 50 ppmv. At the end of the experiment, the chloride level in the sorbent pellets was determined as a function of the bed depth. The results showed that the upstream end of the bed was nearly saturated with chloride whereas chloride absorption was beginning to occur at the downstream end of the bed (Figure 2). Similar results were observed at 500° and 600°C.

The kinetics of HCl absorption by a fixed bed of sorbent can be ascertained from the distribution of chloride along the axis of the bed after a period of exposure, or by the rise in outlet HCl concentration with time as breakthrough is reached. A gradual HCl breakthrough and gently sloping profiles of absorbed Cl imply slow absorption kinetics, while a rapid HCl breakthrough and sharp absorbed Cl profiles imply rapid absorption rates. To determine rate constants from integral reactor data, it is necessary to define the rate laws with respect to local HCl concentration and local extent of sorbent conversion. For the present analysis, it is assumed that the rate is proportional to the partial pressure of HCl (P_{HCl}) and the local unconverted fraction of sorbent (1-X), where $X = S/S_0$, the fractional conversion of sorbent. S and So are the local amount of chloride sorbed and the maximum capacity of the sorbent, respectively, in mol Cl per g sorbent. Thus, the local rate of HCl absorption and NaCl formation can be approximated by equation (1):

Rate (z) =
$$\left(\frac{\text{Uo}}{\text{RT}_{o}}\right) \frac{dP_{\text{HCl}}}{dz} = M_{\text{Cl}}^{-1} \rho_{S} S_{o} \frac{dX}{dt} = k P_{\text{HCl}} (1 - X) \rho_{S}$$
Equation (1)

 $U_o = P_o = T_o = z = z$ superficial gas velocity **Total Pressure**

Temperature depth into the bed

bed density

 $\rho_{S} = M_{Cl} = P_{HCl} = X =$ molecular weight of chlorine partial pressure of HCl at z

fractional conversion of sorbent at z

First order rate constant

If Y_{HCl} is defined as P_{HCl}/P_O and equation 1 is solved, equation (2) is obtained:

$$Y_{HCI}(z,t) = Y_o \left[\frac{exp\left(\frac{kP_oY_ot}{S_oM_{CI}^{-1}}\right)}{exp\left(\frac{kP_oY_ot}{S_oM_{CI}^{-1}}\right) + exp\left(\frac{k\rho_sRT_oz}{U_o}\right) - 1} \right]$$

Equation (2)

Similarly, the amount of chloride absorbed by the solid at a distance z from the upstream end of the bed and time t is given by equation (3):

$$S(z,t) = \frac{S_o \exp(kP_o Y_o t / S_o M_{Cl}^{-1})}{\exp(kP_o Y_o t / S_o M_{Cl}^{-1} + \exp(k\rho_s R T_o z / U_o) - 1)}$$

Equation (3)

where Y_0 is the inlet concentration of HCl.

By integrating the expression for S over discrete intervals of bed depth, i.e., $\{\ell_i, \Delta \ell_i\}$, equation (4) is developed:

$$S_i(t) = S_o - \frac{S_o}{\lambda \Delta_i} \ln \left[\frac{\exp(\lambda \ell_i) + \exp(\alpha t) - 1}{\exp(\lambda \ell_{i-1}) + \exp(\alpha t) - 1} \right]$$

Equation (4)

 $\lambda = k\rho_s RT_0/U_0 (cm^{-1})$

 $\alpha = kP_0Y_0/S_0 M_{CI}^{-1} (h^{-1})$

 Δ_{\bullet} = bed depth interval (cm)

Equation (4) for $S_i(t)$ is very similar to equation (3) for $S(z_i,t)$ with the substitution, $z_i = (\ell_i + \ell_{i-1})$ 1)/2. Non-linear regression analysis can be used to fit measured values of $Y_{HCI}(L,t)$ and $\{S_i(t_f)\}$ where L is the total bed depth and tf is the total elapsed time of the integral fixed-bed isothermal absorption reactor experiments. A two parameter fit to the HCl breakthrough data for $Y_{HCl}\left(L_{t}\right)$ gives α and (λL) while a three parameter fit to the measured chloride content of bed segments gives λ , (αt_f), and S_0 .

The rate constants measured for the nahcolite pellets show little variation with temperature over the range from 400° to 600°C (Table 1). The effectiveness factor for HCl absorption can be estimated as essentially unity, given the measured pore volume distribution before and after chloride

absorption (discussed below). The low apparent activation energy for absorption suggests that gas phase diffusion of HCl, not the chemical reaction or solid-state diffusion of Cl through a NaCl film, governs the absorption rate.

Table 1. HCl Absorption Rate Constants For Nahcolite Sorbents

Data Set for Curve-Fit	T (°C)	α (h ⁻¹)	λ(cm ⁻¹)	S _o (g _{Cl} /g _s)	k (mol _{HCl} · atm ⁻¹ ·g ⁻¹ ·h ⁻¹)
Si	400	0.21	0.61	0.58	2.6
Y _{HCI}	400	0.55	1.48	_	5.2
Si	500	0.33	1.13	0.58	4.1
Si	600	0.13	0.60	0.51	2.2
Y _{HCI}	600	0.39	1.08	_	3.7

Conditions: $Y_o = 1750$ ppmv; $U_o = 13.3$ cm/s; $T_o = 298$ K; $\rho_s = 0.54$ g_{Cl}/g_s ; $t_f = 30.0$ h (400°C), 13.0 h (500°C), 30.6 h (600°C); $z_L = 14.0$ cm; $P_o = 1.0$ atm.

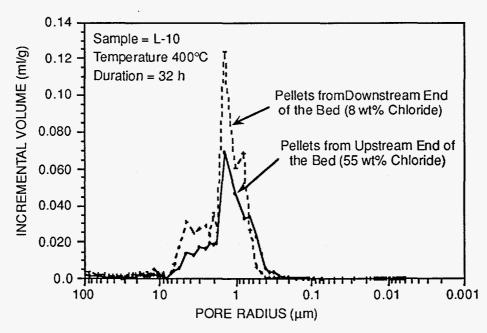


Figure 3. Pore Size Distribution of Nahcolite Sorbent Pellets

Nahcolite (NaHCO₃) decomposes at temperatures above 200°C to form Na₂CO₃ and CO₂:

$$2NaHCO3(s) = Na2CO3(s) + CO2(g)$$

In the presence of HCl vapor, the Na₂CO₃ is converted to NaCl:

$$Na_2CO_3(s) + 2HCl(g) = 2NaCl(s) + H_2O(g) + CO_2(g)$$
 analyzed for their chloride and sulfide contents

The reaction rate will be hindered if the product NaCl obstructs the pores inside the sorbent, reducing the passage of gas molecules. However, mercury porosimetry showed that the pore size distribution is not altered significantly during chlorination of the sorbent (Figure 3). Experiments were also carried out to determine the sorption of H₂S by the nahcolite sorbent according to the following reaction:

$$Na_2CO_3(s) + H_2S(g) = Na_2S(s) + H_2O(g) + CO_2(g)$$

A simulated coal gas containing 1700 ppmv HCl and 3000 ppmv H₂S was passed through the nahcolite sorbent pellets at 400° and 600°C. At both temperatures the residual HCl level was less than 1 ppmv initially and no significant change in the 1-ppmv breakthrough time was observed. At the end of the runs, the sorbent pellets were analyzed for their chloride and sulfide contents (Table 2). At 400°C, the absorption of H₂S by the sorbent was negligibly small. The amount of sulfide retained on the sorbent was higher at 600°C than at 400°C, but even at 600°C less than 1% of the total H₂S in the gas phase was absorbed on the pellets. The above results indicate that presence of H₂S does not have a significant impact on the HCl removal performance of nahcolite pellets.

Table 2.	Ol. 1 1 - 1 -	A1	C16	O	α	C 4	NT-1124-	D-11-4-
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Temperature (°C)	Location	Chloride Level (wt%)	Sulfide Level (wt%)
400	Upstream end	58.0	0.08
400	Downstream end	3.20	0.01
600	Upstream end	50.5	0.57
600	Downstream end	0.57	0.15

Fixed-bed reactor experiments indicated no significant differences in the HCl reactivity between pellets made with sodium silicate and bentonite binders. The maximum chloride capacity of the pellets with sodium silicate binder was 10% higher than that of the pellets with bentonite binder, but the rate of initial reaction did not depend strongly on the nature of the binder. Pellets containing sodium silicate binder agglomerated when the reaction temperature was higher than 500°C. No such agglomeration was observed when bentonite was used as the binder. The crush

strength of the pellets did not change significantly with the extent of chlorination.

Fluidized-Bed Reactor Experiments

Nahcolite granules (NS-01) suitable for fluidizedbed reactor applications were prepared by slurrying the powder in water with 10 wt% kaolinite and 5 wt% bentonite as binders and spray-drying the slurry to obtain granules in the size range 40 to 120 μ m. The fluidization characteristics of this sorbent was found to be excellent as shown in Figure 4.

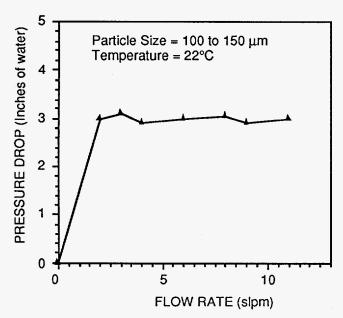


Figure 4. Fluidization Behavior of NS-01 Sorbent

In this cold-flow experiment, the pressure drop increased linearly with increasing gas velocity initially and then remained nearly constant over a wide range of gas velocities. This behavior indicated that the spray-dried sorbent could be fluidized easily.

A fluidized-bed sorbent is likely to be fed into the reactor from a hopper using a screw feeder or other feeding device. The angle of repose gives an indication of the flowability of the powder. The flowability is considered to be excellent if the angle of repose is less than 30° and is poor if the angle is more than 45°. The angle of repose measured for NS-01 in the size range 90 to 150 μ m was 34° indicating that the powder will flow smoothly.

The attrition resistance of this material was also tested using a standard 3-hole air-jet attrition tester. The NS-01 formulation exhibited a higher attrition resistance than the raw nahcolite powder and a commercial FCC catalyst (Table 3).

The reactivity of the spray-dried sorbent was initially determined in a thermogravimetric apparatus. The HCl absorption of NS-01 sorbent was superior to that of the raw nahcolite powder and commercially available grade #2 baking soda (Figure 5).

Table 3. Attrition Resistance of Nahcolite Sorbents

Sorbent	5-h Loss (wt%)	20-h Loss (wt%)
Raw Powder	12.8	37.6
Grade #2 Baking Soda	4.4	13.2
NS-01	5.4	5.8
FCC Catalyst	16.0	14.2

The rate constant and chloride capacity of the spray-dried sorbent (45 to 90 µm in size) were determined as a function of temperature using a gas stream containing 2.1% HCl flowing at less than the minimum fluidization velocity. The rate constants at 450°, 550°, and 650°C were found to be 0.2, 3.0, and 3.0 mole gas-atm⁻¹·g⁻¹·h⁻¹ respectively. These values are comparable to those found with 3-mm sorbent pellets. However, the rate constant calculated for the raw nahcolite powder under similar conditions was about 5 times lower than that for the spray-dried sorbent. The chloride capacities of the NS-01 sorbent at 1-ppmv breakthrough time were 32.4, 51.2, and 52.7 wt% respectively at 450°, 550°, and 650°C. The capacities at 550° and 650°C represent near complete utilization of the sorbent.

The high reactivity of the spray-dried sorbent was also confirmed in tests conducted in a 2.5-cm ID fluidized-bed reactor. A series of experiments were conducted with three different nahcolite formulations at 550°C with simulated Texaco coal gas containing 12.5% v/v steam and 1750 ppmv of HCl vapor. The superficial gas velocity was 3.2 times that of the minimum fluidization velocity. As shown in Figure 6, the spray-dried (NS-02) sorbent had the best overall breakthrough performance among these three formulations. The N-01 formulation is a raw powder without any further processing while N-23 was prepared using a granulation technique. The chloride capture capacity of these sorbents were 26.8 (NS-02), 21.0 (N-23) and 9.8 (N-01) wt.% at 1-ppm breakthrough time.

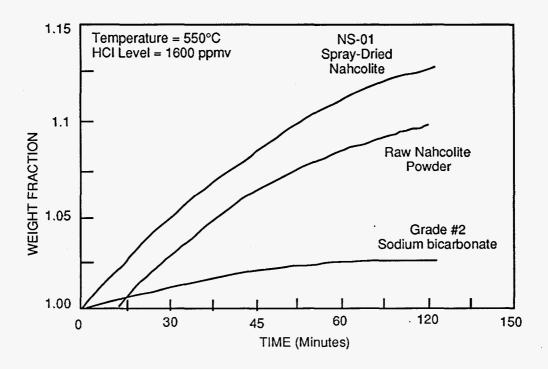


Figure 5. The HCl Reactivity of Various Fluidized-Bed Sorbents

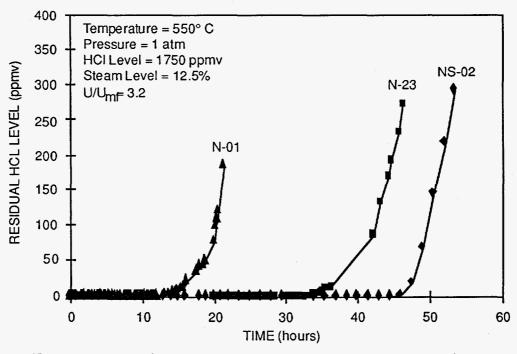


Figure 6. The HCl-Breakthrough Curves for Various Nahcolite Sorbents in a Bubbling Fluidized Reactor

At low fluidization velocities (u/u_{mf} =3), especially at elevated pressures, some amount of caking of the NS-01 sorbent was observed. But the cake was not mechanically strong and it was broken easily. The caking problem may be less severe at high gas velocities.

The effect of steam level in the coal gas was also evaluated in the fluidized-bed reactor experiments. The initial residual HCl level was

< 1 ppmv in experiments conducted at 480°C with a simulated coal gas containing 13 or 26% v/v steam. The 1-ppmv breakthrough occurred at the same time in both cases (Figure 7). But the breakthrough profile was sharp at the high steam level compared with the case for low steam level. In the gas containing 13% v/v steam, the residual HCl level remained at about 3 ppmv for an additional 300 minutes, after which it increased sharply.

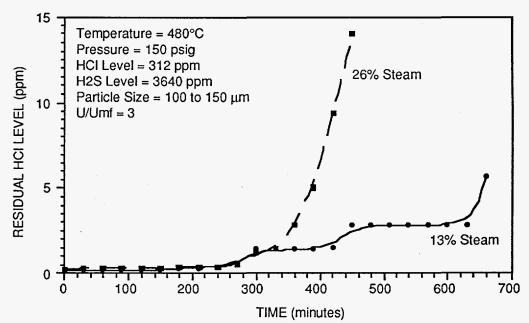


Figure 7. The HCI-Breakthrough Curve in a Fluidized-Bed Reactor in simulated coal gases containing two different steam levels

A limited number of fluidized-bed reactor tests were also conducted with a Grade #2 baking soda in the size range 90 to 150 μm. After calcination at 550°C, the surface area of this powder was less than 0.1 m²/g. In a fluidized bed reactor test at 550°C and 1 atm pressure, severe caking of the powder was encountered which prevented an accurate determination of the rate of HCl uptake.

Circulating Fluidization Bed Reactor Test

The HCl vapor removal capability of the spraydried sorbent was determined at a fixed-bed gasifier facility located at the General Electric Corporate Research and Development site. During this 100 h test, conducted at about 500°C and 280 psig, the HCl level in the hot coal gas was estimated to be about 580 ppmv. During the first 95 h of testing, a grade #2 baking soda was fed into a circulating fluidized-bed (CFB) reactor. Although the residual HCl level was in the range 50 to 150 ppmv initially, it decreased to less than 15 ppmv as the inventory of the sorbent accumulated. During the last 5 h, the spray-dried nahcolite sorbent (NS-02) was fed into the reactor and the residual HCl level remained at a level less than

15 ppmv. The NS-02 sorbent was fed into the reactor using a screw feeder with out any problem. The material withdrawn from the CFB cyclone contained mainly the spent sorbent and a small amount of fly ash (1.5 wt%).

The chemical analysis of the spent NS-02 sorbent showed that its sodium and chloride contents are about 33.5 and 37.6 wt% respectively. Material balance calculations indicated that the sorbent utilization was about 71%. This pilot-scale test demonstrates that nahcolite sorbent is efficient in reducing the HCl vapor level to very low values in actual coal gasifier conditions.

FUTURE WORK

Future activities in the program include parametric testing of nahcolite sorbents by varying space velocity (2,000 to 5,000 h⁻¹), temperature (400° to 650°C), and pressure (1 to 20 atm). The experimental data will be analyzed to determine the effectiveness of the various sorbent formulations for removal of hydrogen chloride vapor using various types of reactors. A preliminary economic assessment is planned to determine the suitability of the sorbents for removing HCl vapor from hot coalderived gas streams on an industrial scale.

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