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Physical Absorption of CO_2 and Sulfur Gases from Coal Gasification: Simulation and Experimental Results

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ABSTRACT

High partial pressures of CO_2, H_2S and certain other constituents produced in coal gasification tend to make the use of physical solvents in associated acid gas removal systems more attractive than the use of chemical solvents. In the research program described in this paper operating data obtained on a pilot plant system employing refrigerated methanol as a solvent will be presented. A mathematical model of the packed absorber used in the process was developed.

Predictions of system performance for a feed gas consisting of CO_2 and nitrogen compared favorably to experimental data obtained on the system. In addition, there was very good agreement between predicted and observed distributions of nine of the major components in a feed gas synthesized in a coal gasification reactor. The results show the validity of the modeling procedure and may be used in understanding the general characterists of packed absorbers and strippers.

INTRODUCTION

Acid gas removal systems are used in the petroleum and natural gas industries to remove carbon dioxide and sulfur gases, primarily hydrogen sulfide, from product streams. The general objective of these systems is to remove diluents that reduce the heating value of the product gas and/or compounds that would lead to problems if released to the atmosphere or fed to catalytic conversion processes. However, while many solvents, both physical and chemical, are available to remove acid gases from gas streams, few are considered suited to the new challenges associated with cleaning gases produced by the gasification of coal (1).

At North Carolina State University, a coal gasification-gas cleaning pilot plant is currently operated under a grant from the U. S. Environmental Protection Agency to assess the environmental consequences associated with treatment of gases produced from coal. Details of the facility are discussed by Ferrell et al. (2) and Felder et al. (3). The research program couples a particular coal and acid gas removal solvent for evaluation of system performance. Current operation involves gasification of a New Mexico subbituminous coal and treatment of product gases with refrigerated methanol. Preliminary experimental findings are presented elsewhere (4,5). The purpose of this paper is to discuss modeling the packed absorption column used to treat acid gases in the acid gas removal part of the pilot plant.

SCOPE OF WORK

Of primary interest in the operation of the acid gas removal system is the evolution and fate of compounds of environmental significance. In the course of normal operation, several sulfur, nitrogen, and hydrocarbon species are followed as they enter the system and distribute among the three outlet streams (from an absorber, flash tank, and stripper) or remain in the recirculating solvent. The concentrations of several of these compounds are on the order of a few parts per million by volume (ppmv). In order to make any assessment of the effect of process conditions on the distribution of these compounds, steady operation under carefully controlled conditions is necessary. Overall and individual component mass balances for the major components are used to determine if this requirement is met in any given run. Process modeling has been initiated to provide insight into system performance and to extend experimental findings to process conditions unattainable in the pilot plant.

NON-ISOTHERMAL GAS ABSORPTION IN PACKED COLUMNS

Although a great deal of effort has been expended in the development of calculational techniques for staged absorption columns, much less attention has been focused on packed columns. This is especially true where adiabatic or non-isothermal absorption must be considered. In these cases, the temperature distribution within the column influences the solubilities of the gas phase components, the vapor pressure of the solvent, and values of the transport properties.

Calculational techniques used to describe non-isothermal packed column absorption have shown varying degrees of complexity. Sherwood, Pigford and Wilke (6) describe a generalized design procedure for adiabatic absorption in packed towers in which liquid phase resistance to mass and heat transfer and the effect of temperature on transfer coefficients are ignored. von Stockar and Wilke (7,8) present both rigorous and short-cut techniques for packed column gas absorbers involving large heat effects using a relaxation calculational procedure but describe only the case of a single transferring solute. Holland and co-workers (9,10) use a tray tower analogy to describe non-isothermal gas absorption in packed towers for both simple and multicomponent systems. Only Treybal and Feintuch (11,12) describe an essentially rigorous calculational procedure for the design of a packed absorption column; their procedure includes the influence of thermal effects as well as a capability to handle multicomponent systems. Although they were not able to provide a satisfactory comparison between their model and experimental data for multicomponent systems, it compared favorably for the case of a single transferring solute (13).

In this study, the technique of Feintuch and Treybal (12) was adapted to handle both single solute and multicomponent absorption of acid gases in refrigerated methanol. Adiabatic operation was assumed in the model; this assumption was shown experimentally to correspond to actual system performance. It was also assumed was that there was no axial dispersion of either gas or liquid flows (11). Model predictions and experimental results have compared favorably over a wide range of system operating conditions.

THEORY

In expressing mass and energy balances and equilibrium relationships in an adiabatic packed column, a series of implicit and highly non-linear equations result. If the inlet conditions of both gas and liquid streams are fixed, and the outlet conditions of both streams are to be determined, subject to a specification of the recovery of a key component, a two-point boundary value problem must be solved, where neither boundary condition is completely described. This gives rise to an iterative calculation which is especially difficult to execute for multicomponent systems.

Treybal (11) portrays the situation in a differential section as in Figure 1. This is analogous to the two-resistance theory which explains interphase mass transer in terms of diffusion through liquid and gas phases, and assumes that the interface provides no resistance to transfer. Resistances to both heat and mass transfer within both gas and liquid phases must be included in the model.

Adopting the nomenclature used by Feintuch and Treybal (11,12), the molar flux for each component, N_j, in a differential section can be expressed as:

$$N_j adz = R_j F_{Gj} \ \ln\{(R_j - y_{ji})/(R_j - y_j)\} \ adz = -dG_j \qquad (1)$$

$$= R_j F_{Lj} \ \ln\{(R_j - x_j)/(R_j - x_{ji})\} \ adz \qquad (2)$$

where $R_j = N_j / \sum N_j$

and F_{Gj} and F_{Lj} are the Drew-Colburn gas and liquid mass transfer coefficients.

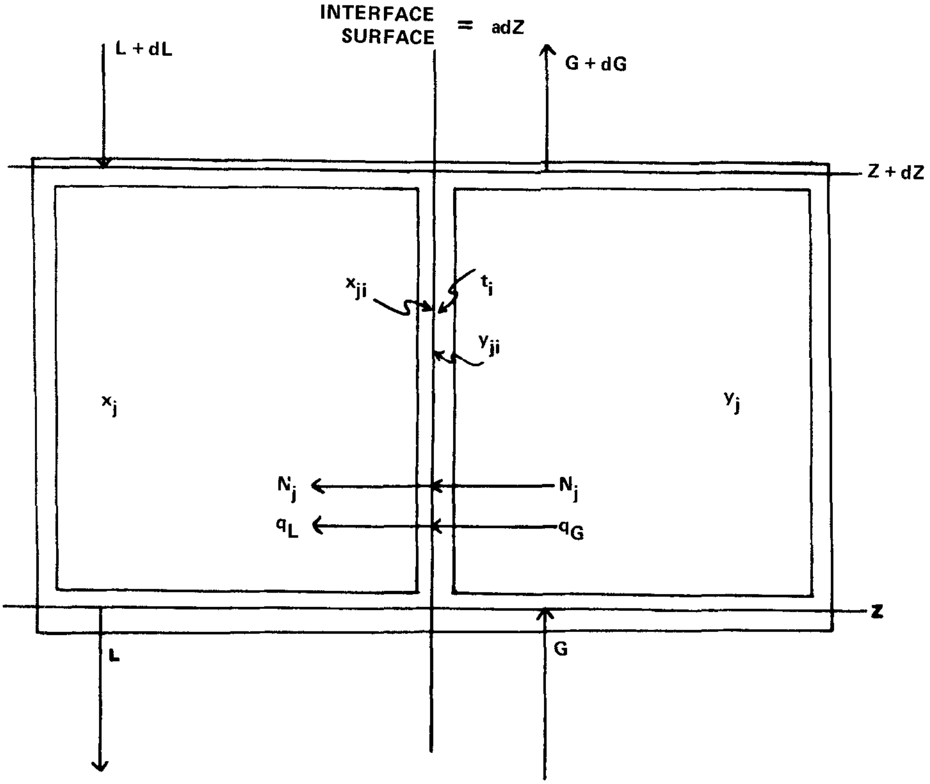

Figure 1. Differential Section

The concentration gradient in each differential section can then be expressed as:

$$dG_j/dz = R_j F_{Gj} a \ \ln \{(R_j - y_{ji})/(R_j - y_j)\} \tag{3}$$

$$dG_j/dz = R_j F_{Lj} a \ \ln \{(R_j - x_j)/(R_j - x_{ji})\} \tag{4}$$

Using a similar approach, the temperature gradient in a differential section can be described for the gas phase as:

$$dt_G/dz = -h_G a\ (t_G - t_i) / \sum G_j C_j \qquad (5)$$

where h is the gas phase heat transfer coefficient.

In each differential section the gas phase mole fraction at the interface can be expressed by either the equilibium relationship or by equating the right hand sides of Equations (1) and (2) and solving for y_{ij}:

$$y_{ji} = f\ (x_{ji},\ t_i,\ P) \qquad (6)$$

$$y_{ji} = R_j - \{R_j - y_j\} \{(R_j - x_j)/(R_j - x_{ji})\}^{F_{Lj}/F_{Gj}} \qquad (7)$$

Finally, the interfacial temperature can be calculated using the following expression:

$$t_i = t_L + (1/h_L a) \sum (H_{ji} - (C_j(t_G - t_o) + \lambda_j)\ \ dG_j/dz$$
$$+ (1/h_L a)\ \ dt_G/dz \sum\ G_j C_j \qquad (8)$$

These equations, the necessary mass and energy balances, physical and transport property information, and phase equilibrium relationships are used to describe adiabatic packed column performance.

CALCULATIONAL PROCEDURE

The computer program implemented receives as input the temperature, composition, and flow rate of the inlet gas and liquid streams. Also included in the program input are the column pressure, the removal efficiency of the key component (usually CO_2 in the systems studied), the number of sections that the column is to be divided into, and convergence tolerances for certain iterative calculations.

Built into the program are physical and transport property subroutines for mixture properties based on techniques suggested by Reid et al. (14) and Perry (15). The mass transfer coefficients are determined from the correlations found in Treybal (16). Heat transfer coefficients are then determined using the j-factor analogy. It is assumed that Henry's law can be used to express the phase equilibrium relationships for all components. Henry's law coefficients were taken from Prausnitz (17) or calculated from information found in the literature (18). Heats of solution at infinite dilution are calculated using the Henry's law coefficients. Since no experimental data are available to determine the effective interfacial area in adiabatic absorption with 1/4" Intalox saddles, correlations developed for Berl saddles by Shulman and presented by Treybal (16) were used to estimate the effects of all variables, except packing size, on this quantity. The effect of packing size was incorporated by fitting an adjustable parameter to experimental results. A discussion of the use of several types of correlations to express effective interfacial areas in packed columns will be presented later (19).

The calculation begins by assuming values for the outlet gas flow rate of each component, including the solvent, and the outlet gas temperature. The flow rate of each component in the outlet liquid and the temperature of this stream are calculated from material and energy balances around the column. A stepwise calculation is then begun at the bottom of the column; the calculation moves up the column in a number of increments specified in the program input.

To specify the conditions in each section of packing, values of the interfacial temperature, the interfacial liquid mole fraction, x_{ij}, and normalized molar fluxes, R_j, are assumed. The correct combination of R_j and x_{ij} have been chosen when the difference between y_{ij} calculated from Equations (6) and (7) is essentially zero for each component. At this point, the sum of the x_{ij} values is checked to see if it is within a specified tolerance of 1.0. If this check is satisfied, the gas phase composition and temperature gradients are then calculated by Equations (3) and (5) and used in Equation (8) to determine the interfacial temperature. This calculated value of the interfacial temperature is compared to the value assumed at the beginning of the iteration. If they are within a specified tolerance, the gradients are used to determine the conditions at the next level of packing. If not, a new value of the interfacial temperature is assumed and the iteration continues.

The calculation continues moving up the column until the specified concentration of the key component is reached at the next packing level. At this point, the assumed values of all outlet gas flow rates and the outlet gas temperature is compared to the calculated values. If these values agree within a certain tolerance, the calculation is complete. Otherwise, new values of all outlet gas flow rates and the outlet gas temperature are chosen and the iteration continues.

The algorithm used in this study is similar to that used by Feintuch (12) and is shown in Figures 2 and 3. A detailed description of this algorithm and more information on its convergence characteristics will be included in a future report (19).

EQUIPMENT

The packed absorption column used in this study contains approximately 21 feet of ceramic 1/4" Intalox saddles and is divided into 3 sections. Mass transfer studies can, therefore, utilize 7, 14, or 21 feet of packing. The column is 5 inches in diameter and is insulated to approach adiabatic operation. Care has been taken to minimize end effects by segregating liquid and gas flows at the bottom of the column. Inlet and outlet gas flow rates are measured with orifice meters; temperature sensors are located on the column wall to measure the temperature profile in the lower part of the column. Column pressure and differential pressure are also monitored. The data generated during an experimental run is logged every five seconds by a PDP 11/34 minicomputer with real time display of process variables on a ISC color graphics terminal. The column inlet liquid and gas flow

Figure 2

MCOMP FLOWCHART

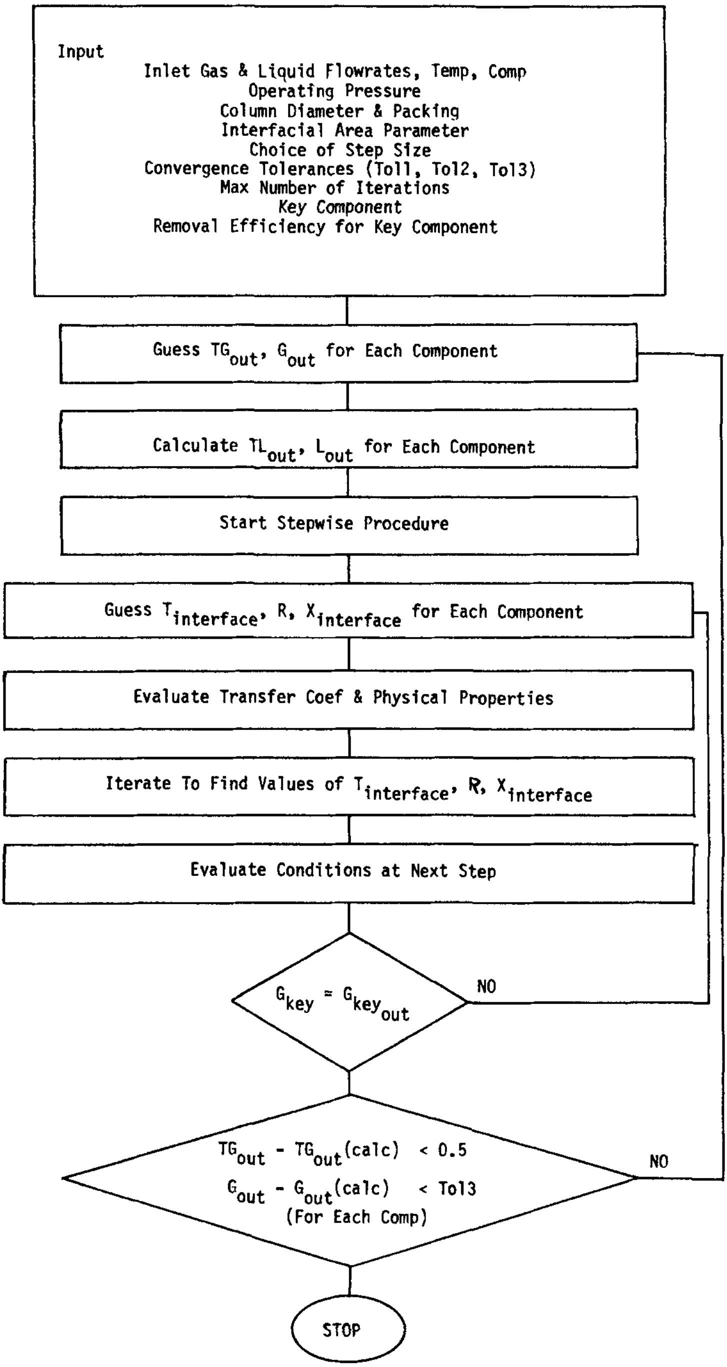

Figure 2. MCOMP Flowchart

Figure 3

ITERATIVE PROCEDURE IN EACH DIFFERENTIAL SECTION

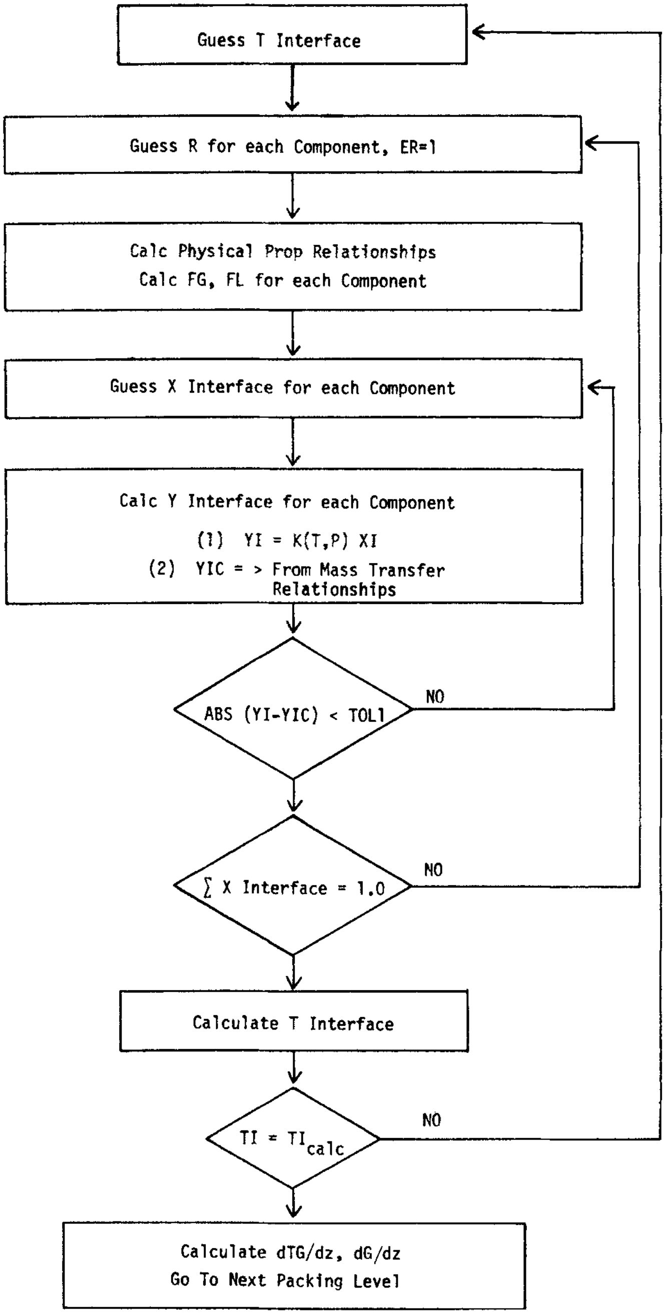

Figure 3. Iterative Procedure in each Differential Section

rates, temperatures and column pressure are controlled by means of a Honeywell TDC 2000 process control computer.

Gas composition analysis is done with the following equipment:

Permanent gases	Tracor 550 Gas Chromatograph	Thermal Conductivity Detector
Sulfur gases	Varian 3700 Gas Chromatograph	Flame Photometric Detector
Hydrocarbons	Tracor 550 Gas Chromatograph	Flame Ionization Detector

Because inert gas is used to calibrate flow meters, a correction is made to the flow rates measured by the orifice meters after the run.

As mentioned before, the entire facility is discussed in detail elsewhere (2,3).

EXPERIMENTAL APPROACH

The objective of most pilot plant runs is to determine the fate of selected compounds present in the gasifier product gas as they enter and distribute in the acid gas removal system. Because several of these compounds are present in very small quantities, it would be difficult to draw any conclusions from any run if the system is not operated at carefully controlled conditions. An assessment of the quality of the experimental data is made by checking several pressures, flow rates, temperatures and differential pressures in the acid gas removal system to see that only relatively small changes occur during the steady state period.

Also, mass balances are made for nine compounds present in significant amounts in the feed gas. If reasonably good component and total mass balances (within 10% of complete closure) are obtained for all nine components, steady state operation and proper sampling and analytical methods are confirmed. Tables 1A and 1B, along with Figure 4, illustrate the results from a particular run. Since the modeling efforts have focused on the absorber, only the results describing the absorber performance will be discussed here.

Because the absorber column normally operates at elevated pressures (15-30 atmospheres) and low temperatures (0 to -35^{o}F), taking liquid or gas samples from locations in the packing is extremely difficult. Alternatively, the rate of mass transfer of any highly soluble gas in methanol is reflected by the magnitude of the temperature rise associated with the heat of absorption. By monitoring the temperature profile in the column, it is possible to follow the mass transfer of the more concentrated acid gas components such as H_2S and CO_2. This, in fact, has been the approach of other investigations using other solvent-solute systems (13).

An additional problem arose from the fact that maintaining the integrity of the column pressure rating prohibited additional machining of the column wall to install temperature sensors. Instead, platinum temperature sensors were attached to the exterior of the column wall to measure the temperature profile. A portable sensor has been used to check for differences in temperature around the column circumference at the same packing level but no

TABLE 1A

Stream Compositions for AM-43 (volume %)

Comp.	Sour Gas	Sweet Gas	Flash Gas	Strip Gas	Acid Gas	Absorb* Bottom	Flash* Bottom	Stripper* Bottom
CO2	20.050	0.000	50.580	0.0	68.370	5.331	4.837	0.000
H2S	0.295	0.022	0.254	0.0	0.890	0.074	0.072	0.002
COS	0.012	0.001	0.015	0.0	0.032	0.003	0.003	0.000
MEOH	0.000	0.000	0.000	0.0	1.100	93.754	94.671	99.838
H2	32.420	43.400	9.390	0.0	0.000	0.000	0.000	0.000
CO	21.230	25.790	14.810	0.0	0.080	0.283	0.125	0.126
N2	18.700	23.010	9.080	100.0	28.210	0.188	0.092	0.000
CH4	6.590	7.530	10.640	0.0	0.520	0.187	0.073	0.034
C2H4	0.308	0.062	2.054	0.0	0.621	0.069	0.048	0.000
C2H6	0.508	0.112	3.083	0.0	1.020	0.112	0.079	0.000

* calculated values

TABLE 1B

Mass Balances on Run AM-43 (amounts in lb-moles/hr)

Comp.	Sour Gas	Strip Gas	Sweet Gas	Flash Gas	Acid Gas	Total Input	Total Output	% Closure
CO2	0.506	0.000	0.000	0.051	0.506	0.506	0.558	110.2
H2S	0.007	0.000	0.000	0.000	0.007	0.007	0.007	97.8
COS	0.000	0.000	0.000	0.000	0.000	0.000	0.000	91.2
MEOH	0.000	0.000	0.000	0.000	0.008	0.000	0.000	0.0
H2	0.818	0.000	0.856	0.010	0.000	0.818	0.866	105.8
CO	0.536	0.000	0.509	0.015	0.001	0.536	0.525	97.9
N2	0.472	0.182	0.454	0.009	0.209	0.654	0.672	102.8
CH4	0.166	0.000	0.149	0.011	0.004	0.166	0.163	98.2
C2H4	0.008	0.000	0.001	0.002	0.005	0.008	0.008	101.6
C2H6	0.013	0.000	0.002	0.003	0.008	0.013	0.013	100.6
TOTAL	2.2523	0.182	1.973	0.102	0.740	2.708	2.812	103.8

significant gradients were observed. In addition, tests were conducted using a solvent flow held at a fixed temperature to see if wall temperatures accurately reflected the liquid temperature. Excellent agreement between the inlet temperature and the wall temperatures were observed. It is thought, therefore, that the

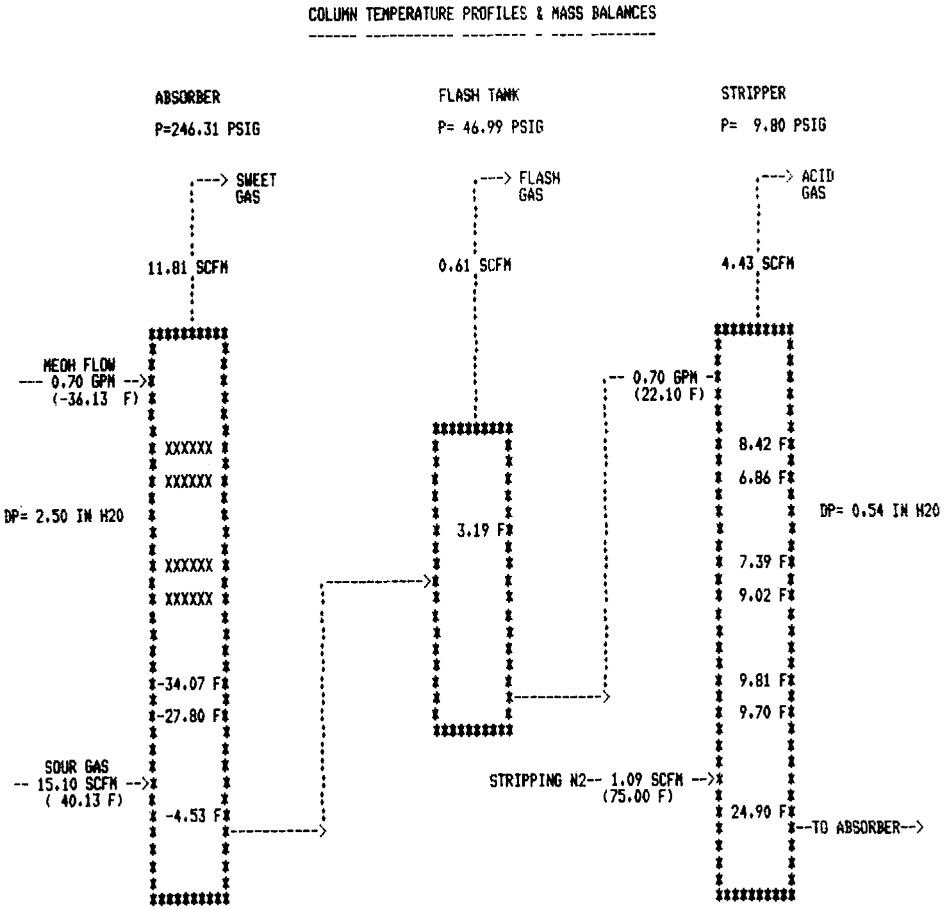

Figure 4. Run Number A-M-43. Integrated Run. Date 11/9/80

installed temperature sensors are a good indication of liquid temperature.

COMPARISON OF MODEL PREDICTION WITH EXPERIMENTAL RESULTS

To check model predictions against experimental results, calculated liquid temperature profiles are compared to experimental data. In addition, the outlet gas composition and flow rates are compared to measured quantities.

There are two formulations of the model that have been implemented. The first formulation deals with the situation where only one solute is transferred from the gas to the liquid phase. Evaporation and/or condensation of the solvent is also allowed. This situation is much easier to model than the multicomponent case because the liquid phase consists of only one solute and the solvent; the mole fraction of one component is not independent of that of the other. Also, since the sum of the normalized molar fluxes, R_j, must be one, the normalized molar flux of the solute is one minus the flux of the solvent; the inert carrier gas does not transfer.

This formulation of the model was tested by comparisons with experimental runs made with the three component system carbon dioxide-methanol-nitrogen. In addition to testing model performance, information concerning the effective interfacial area associated with the column packing was obtained for the mass transfer coefficient correlation used in the model.

As an example of model performance, the operating conditions and comparison to model prediction are shown for run AM-32 in Table 2 and Figure 5. The actual and predicted liquid temperature profiles show excellent agreement in this run where a synthetic gas stream was fed to the absorber from gas bottles.

The other formulation of the model is for mulicomponent cases and has been described in the earlier sections of this paper. This formulation was tested by comparisons with experimental data taken during runs in which the absorber feed gas was produced in the gasifier. Although there are a wide variety of compounds present in the product gas, the model currently deals with only the nine compounds present in the greatest concentrations. As more data becomes available additional compounds will be included in the model.

TABLE 2

Process Conditions for Synthetic Gas Run AM-32

Liquid Flow Rate	61.05 lb moles/hr/ft^2
T_L in	-36.1^oF
Gas Flow Rate	17.31 lb moles/hr/ft^2
T_G in	57.4^oF
Pressure	28.0 atm absolute
Inlet Gas Composition	33.73 mole percent CO_2 66.27 mole percent N_2
Outlet Gas Composition	0.92 mole percent CO_2 99.08 mole percent N_2
CO_2 Removal Efficiency	98.10%

An example illustrating the comparison between model prediction and experimental data is shown in Tables 3 and 4 and Figure 6.

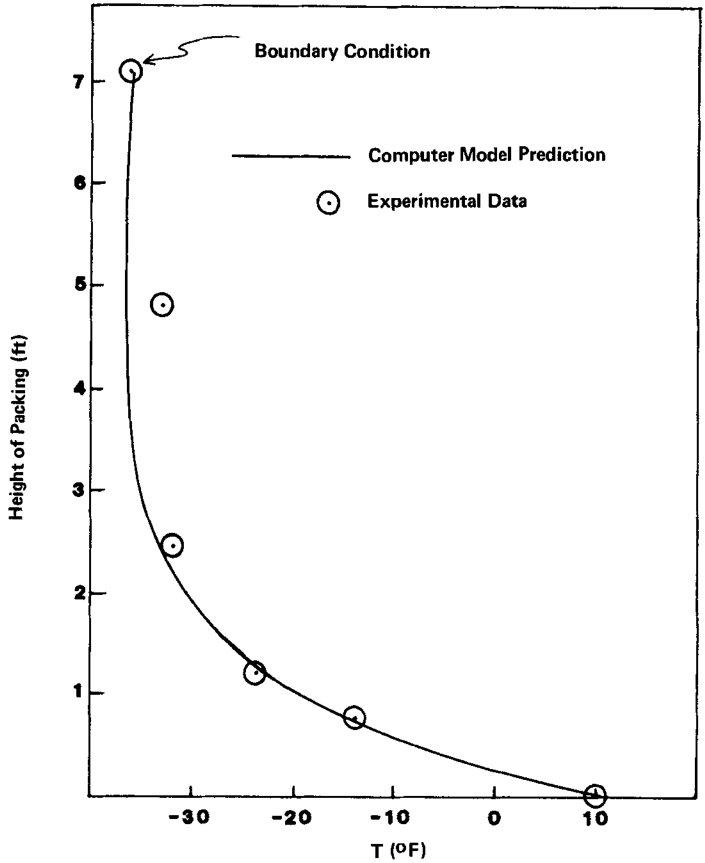

Figure 5. Packed Absorption Column Liquid Temperature Profile for Syngas Run AM-32

TABLE 3

Process Conditions for Integrated Run AMI-43

Liquid Flow Rate	65.10 lb moles/hr/ft^2
T_L in	-36.13^{o}F
Gas Flow Rate	18.10 lb moles/hr/ft^2
T_G in	40.13^{o}F
Pressure	17.760 atm absolute

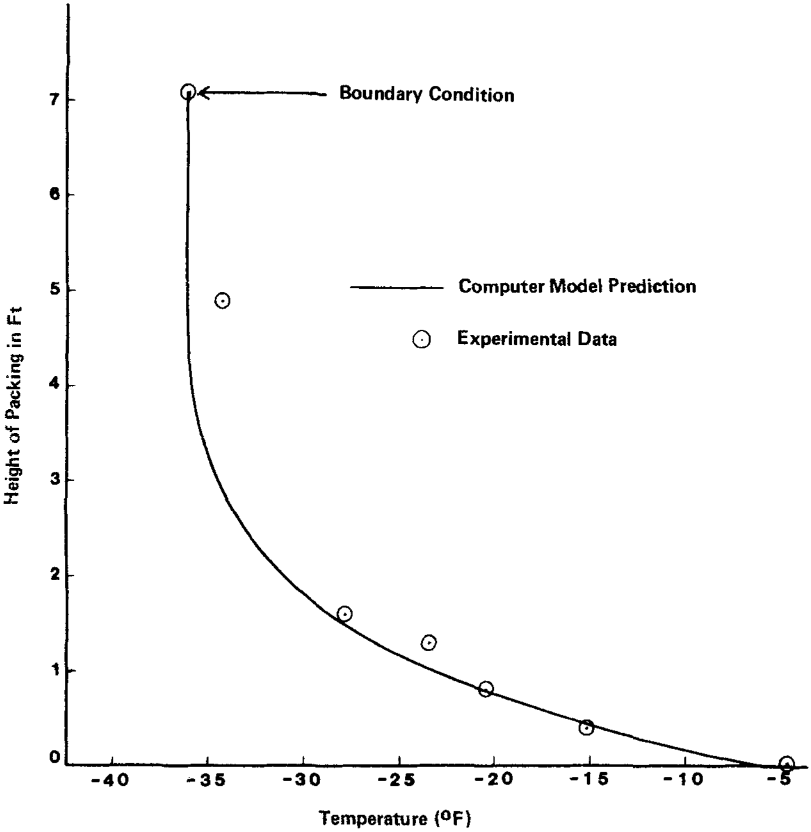

Figure 6. AMI-43. Integrated Run. 11/19/80

TABLE 4

Predicted vs. Experimental Results for AMI-43

Component	Sour Gas (experimental)	Sweet Gas (experimental)	Sweet Gas (predicted)
CO2	20.05	trace	0.0128
H2S	0.295	0.022	<0.0001
COS	0.012	0.001	<0.0001
MEOH	-	trace	<0.0001
H2	32.42	43.40	41.40
CO	21.23	25.79	26.38
N2	18.70	23.01	23.50
CH4	6.59	7.53	8.01
C2H4	0.308	0.062	0.218
C2H6	0.508	0.112	0.358
Flow rate(SCFM)	15.07	11.81	11.74

Good agreement is seen between predicted and actual liquid temperature profiles. The difference between the measured temperatures at 4.90 feet have been attributed to electronic problems that were discovered after the run.

While there is very good agreement between the predicted and experimentally determined concentrations for most compounds, as seen in Table 4, data and model predictions for H_2S, C_2H_4, and C_2H_6 do not compare as well as those for other compounds. The difference between experimental and predicted values of H_2S were later shown to be related to the fact that the entering methanol was not adequately stripped and contained some H_2S. The input data to the model assumed that clean methanol was fed to the column. The difference between the values for C_2H_4 and C_2H_6 may be related to the fact that Henry's law is not an accurate assumption for these cases. This point is currently being examined using results from several other runs. Presented in Figure 7 is the computed concentration profiles for all components except COS and methanol which were present in very small concentrations and do appear on the scale shown. The concentrations of H_2S, C_2H_4, and C_2H_6 are all scaled by a factor of ten.

SUMMARY AND FUTURE WORK

Based on the results obtained, it appears that the algorithm proposed by Feintuch and Treybal (12) can be used successfully to predict packed absorption column performance. Problems encountered thus far may be traced to inaccurate phase equilibrium information

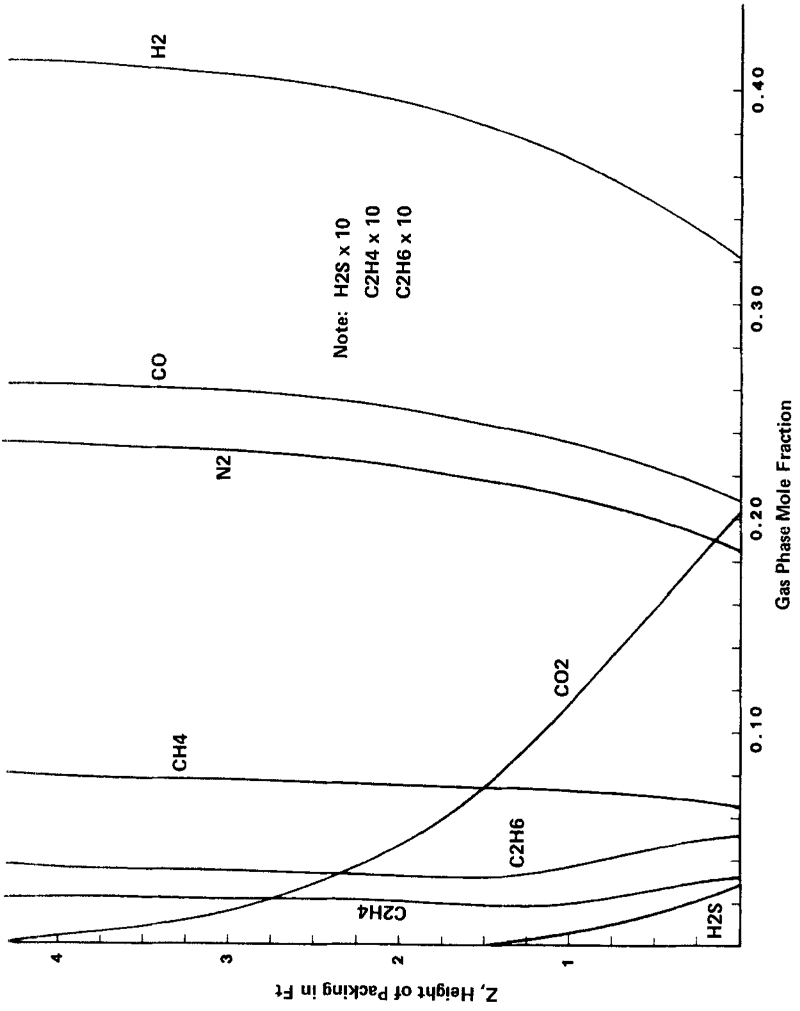

Figure 7. AMI-43. Integrated Run. 11/19/80

or failure to specify accurately the composition of the inlet liquid stream.

Current efforts are focused on several areas, including improvements in the calculational procedures and accumulation of better experimental data. The effect of specified convergence tolerances for several internal iterative calculations on computation time is being examined along with the effect of step size on model prediction accuracy.

Because of the lack of information in the literature on the effective interfacial area of this packing in adiabatic absorption, several different correlations are being tested. At present, if the mass transfer coefficient correlations suggested by Shulman and presented in Treybal (16) are correct for this system, the effective interfacial area for mass transfer is less than 5% of the total packing surface area.

In the near future, several more compounds, such as propane, propylene and butane, will be added to the model. In addition, the techniques used to describe absorber operation will be applied to the stripper and compared to experimental results. The overall objective of this work is to develop the capability to simulate acid gas removal system performance.

ACKNOWLEDGMENT

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NOMENCLATURE

a	specific interfacial surface area, sq ft/cu ft
C_j	heat capacity of component j as a gas, Btu/lb mol-^oF
F_{Gj}	gas phase mass transfer coefficient for j, lb mol/hr-ft^2
F_{Lj}	liquid phase mass transfer coefficient for j, lb mol/hr-ft^2
G_j	gas mass velocity for component j, lb mol/hr-ft^2
H_{ji}	partial molar enthalpy of j in solution at concentration x_i, and temperature t_i, Btu/lb mol
h_G	gas phase heat transfer coefficient, Btu/hr-ft^2-^oF
h_L	liquid phase heat transfer coefficient, btu/hr-ft^2-^oF
N_j	molar flux of j, lb mol/hr-ft^2 of interfacial surface
P	total pressure in atmospheres
q_G	energy flux in gas phase, Btu/hr-ft^2 of interfacial surface
q_L	energy flux in liquid phase, Btu/hr-ft^2 of interfacial surface
R_j	normalized molar flux
t_o	reference temperature, ^oF
t_i	interfacial temperature, ^oF
t_G	gas temperature, ^oF
t_L	liquid temperature, ^oF
x_j	liquid mole fraction of component j
x_{ji}	interfacial liquid mole fraction of component j
y_j	gas mole fraction of component j
y_{ji}	interfacial gas mole fraction of component j
z	packing height measured from bottom of column, ft
λ	latent heat of vaporization, Btu/lb mol at t_o

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