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Improvement in Steam Stripping of Sour Water through an Industrial-Scale Simulation

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Abstract−Sour water containing hydrogen sulfide, carbon dioxide, hydrogen cyanide, and ammonia is mainly purified by steam stripping. Increased concern in recent times over effluent water quality and energy saving has caused sour water stripping to attract considerable attention. However, the development of the operation methodology and structure of this process has not been systemic, leading to inefficiencies in the systems commonly used. In this paper, the characteristics of a sour water stripping column are investigated by using an industrial-scale simulation. From these results, we propose several guidelines (high feed temperature, low composition of CO_2 in the feed, need of rectifying section, low mass flow rate of the second recycle stream, high reflux ratio, and modified structure using a pump-around side cooler) for improving stripper performance through changes in the operating condition and process structure. The proposed structure and guidelines can be applied not only to reduce steam consumption and lower the ammonia concentration in the effluent water but also to operate the system stably.

Key words: Distillation, Process Simulation, Electrolyte System, Sour Water, Steam Stripping

INTRODUCTION

Separation processes are of great importance in the chemical industry. The steam stripping process [Hassan and Timberlke, 1992; Hwang et al., 1992] is widely used to reduce the chemical contaminants in the wastewater generated by refineries and ironworks. Containing hydrogen sulfide, carbon dioxide, hydrogen cyanide, and ammonia, this wastewater is referred to as ‘sour water’.

Nowadays, the increasing emphasis on regulating the quality of effluent water and saving energy has caused sour water stripping to emerge as an important process. However, existing sour water stripping columns show large variations in design and poor separation performance because the design technology and operation methodology were not systematically established. In addition, sour water strippers have several problems such as plugging, excessive use of steam, and difficulty in controlling column pressure. Much research has been focused on the characteristics and operating conditions of the sour water stripping process. Walker [1969] proposed a method to quickly design refluxed and non-refluxed sour water strippers. In addition, he formulated the concept of a stripping efficiency and found the factors that influence the stripping performance. Darton et al. [1978] and Melin et al. [1975] showed simple features of a single sour water stripper by changing operating variables. Hoogendoorn et al. [1988] compared thermodynamic models based on weak volatile electrolyte compounds such as NH_3, H_2S and CO_2. Isla et al. [1989] suggested an improved scheme for a sour water stripper that reduced the heating energy of the reboiler. Stein et al. [1988] concentrated on the improvement of stripping efficiency by controlling the pH. However, most of the studies outlined above concentrated on simple features of a single column that simultaneously separates NH_3 and H_2S.

In comparison to past studies, the present paper considered a wider variety of characteristics of sour water stripping, which were investigated more specifically by an industrial-scale simulation. Based on the steady state simulation results, we proposed several guidelines for improving stripping performance through changes in the operating condition. Furthermore, a modified scheme is considered to control the column pressure effectively. Dynamic simulation is used to examine the controllability and to cope with abnormal situations which may occur in a real plant.

SIMULATION OF THE SOUR WATER STRIPPING PROCESS

1. Process Description and Operating Condition of POSCO

Fig. 1 presents a schematic diagram of the sour water system installed at the Kwangyang Works of POSCO (Pohang Iron & Steel Corporation). The system contains 13 equilibrium stages including

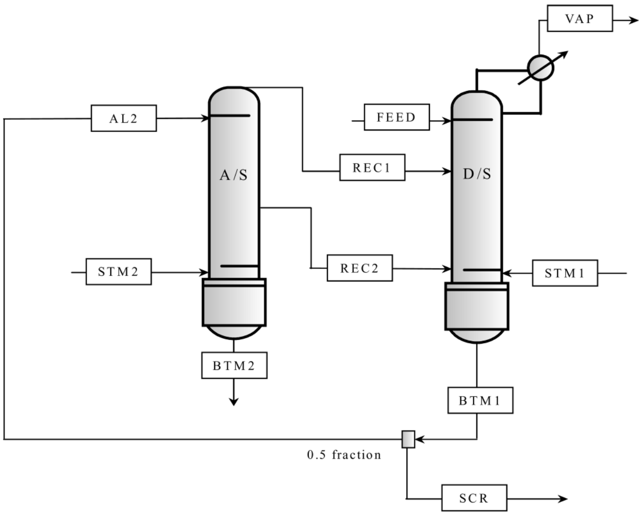

Fig. 1. Sour water stripping system.

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a partial condenser (stage 1) in the Dissociator (D/S), and 16 equilibrium stages in the Ammonia Still (A/S). D/S uses 12 sieve trays, whereas A/S consists of 16 bubble cap trays. In general, H_2S, CO_2 and HCN are stripped in D/S whereas NH_3 is treated in A/S. The primary reason for using 2 columns is the different roles of A/S and D/S. The sour water stripper is a subsystem of the coke oven gas treatment system in the ironworks. The bottom liquid of D/S contains a large amount of NH_3, and a part of this liquid is used to absorb H_2S in other systems such as the H_2S scrubber. In contrast, a part of the bottom liquid of A/S is used to absorb NH_3 in the NH_3 scrubber.

In the process shown in Fig. 1, the feed (FEED) is introduced into the second stage of D/S and stripped by steam (STM1) which is fed into the bottom of D/S. Effluent acid gas (VAP) from the top of D/S is treated to produce sulfur and nitrogen gas in the Catalytic Oven Reactor (COR) and a reflux is returned to the top plate of D/S to enrich the sour gas. AL2, half amount of the condensed ammoniac water (BTM1), is fed into the top of A/S and the rest (SCR) is recycled to the H_2S scrubber, which absorbs H_2S from the Coke Oven Gas (COG). Ammoniac liquor in A/S is stripped until the permitted concentration level (below 100 weight parts/million (ppmw)) is achieved. Finally, this stripped ammoniac liquor (BTM2) is treated in the Biological Effluent Treatment (BET) process to remove fixed ammonia and phenol by using activated sludge before the effluent passes to the outlet. Cyanide compounds should be removed as much as possible before the stripped ammoniac liquor is sent to the BET process because ammoniac liquor containing cyanide compounds has a bad influence on BET and sometimes has a toxic effect on microbes. In addition, ammonia-rich vapor recycle streams (REC1, REC2) from the top and middle (stage 7) stage of A/S are introduced to the middle (stage 9) and the bottom stage of D/S to reduce the effect of pressure variations. The feed conditions and column specifications of A/S and D/S are listed in Table 1.

Table 1. Feed conditions and column specifications of A/S and D/S

D/S feed condition (FEED in Fig. 1)	Diameter (mm)	2300
	Temperature (°C)	64
	Flow rate (kg/hr)	80700
	Pressure (bar)	1.6
	Composition (mg/*l*)	
	NH_3	13000
	H_2S	2500
	CO_2	8000
	HCN	100
D/S column specification	Diameter (mm)	3100
	Condenser type	Partial-Vapor
	Total stages	13
	Condenser stage	1
	Feed stage	2
	Reflux ratio	1.54
	Top pressure (bar)	1.29
	Bottom pressure (bar)	1.35
A/S column specification	Total stages	16
	Feed stage	1
	Top pressure (bar)	1.33
	Bottom pressure (bar)	1.55
D/S steam condition (STM1 in Fig. 1)	Flow rate (kg/hr)	1500
	Temperature (°C)	170
	Pressure (bar)	2
A/S steam condition (STM2 in Fig. 2)	Flow rate (kg/hr)	5900
	Temperature (°C)	170
	Pressure (bar)	2

This objective of the present study was to elevate the stripping efficiency, i.e., to strip much more H_2S and less H_2O in D/S, and to attain an NH_3 composition below 100 ppmw in the effluent water of A/S. Reduction of the mass fraction of H_2O in the effluent vapor of D/S is important because H_2O causes plugging phenomena when combined with other salts and deactivates the catalyst in subsystem COR. In order to study the characteristics of the complex process and analyze the practical problem of a steam stripper with more than two columns, an ASPEN PLUS simulator was used. The RADFRAC module [Aspen Plus® Electrolytes Manual, 1998; Quitain et al., 1999], which is a rigorous model for simulating all types of multistage vapor-liquid fractionation operations, was used to simulate the reactive stripping columns A/S and D/S.

2. Vapor-Liquid Equilibrium and Weak Electrolyte Reaction

The solutes to be removed are weak electrolytes that are partially dissociated into ions in solution. Therefore, the phase equilibrium of each weak electrolyte in solution plays an important role in the removal of pollutants from sour water through steam stripping. The main feed components in this system are H_2O, NH_3, H_2S, CO_2 and HCN. Henry's law is applied to these components in the simulation because they are light gases. In ASPEN PLUS, the value of Henry's constant for each component is assigned as an input variable. The following reactions are of interest for this study.

$$2H_2O \leftrightarrow H_3O^+ + OH^- \tag{R.1}$$

$$H_2S + H_2O \leftrightarrow H_3O^+ + HS^- \tag{R.2}$$

$$HS^- + H_2O \leftrightarrow H_3O^+ + S^{2-} \tag{R.3}$$

$$CO_2 + 2H_2O \leftrightarrow H_3O^+ + HCO_3^- \tag{R.4}$$

$$HCO_3^- + H_2O \leftrightarrow H_3O^+ + CO_3^{2-} \tag{R.5}$$

$$NH_3 + H_2O \leftrightarrow NH_4^+ + OH^- \tag{R.6}$$

$$NH_3 + HCO_3^- \leftrightarrow NH_2COO^- + H_2O \tag{R.7}$$

$$HCN + H_2O \leftrightarrow H_3O^+ + CN^- \tag{R.8}$$

Reactions are assumed to reach equilibrium and equilibrium constants are correlated by the following equation.

$$\ln(K_{eq}) = A + B/T + C\ln(T) + DT \tag{1}$$

where K_{eq} is the equilibrium constant, T is temperature in Kelvin, and A, B, C, and D are the equilibrium coefficients. The coefficients of Eq. (1) are given in Table 2.

Many equations of state have been formulated to predict the thermodynamic properties of vapor and liquid mixtures. However, sour water is a weak electrolyte mixture and consequently needs a more complex model such as the electrolyte-NRTL activity coefficient model (ELECNRTL) [Aspen Plus® Electrolytes Manual, 1998]. ELECNRTL is therefore the recommended option for the simulation of the sour water electrolyte mixture. In general, ELECNRTL is used to calculate liquid phase properties, and vapor phase properties are calculated from the Redlich-Kwong equation of state. ELECNRTL can represent aqueous and aqueous/organic electrolyte sys-

Table 2. Equilibrium constant data

Reaction	A	B	C	D	K_{eq} (363 K)
$2H_2O \leftrightarrow H_3O^+ + OH^-$	132.8989	−13445.9	−22.4773	0	$1.232*10^{-16}$
$H_2S + H_2O \leftrightarrow H_3O^+ + HS^-$	214.5824	−12995.4	−33.5471	0	$5*10^{-9}$
$HS^- + H_2O \leftrightarrow H_3O^+ + S^{2-}$	−9.74196	−8585.47	0	0	$3.14*10^{-15}$
$CO_2 + 2H_2O \leftrightarrow H_3O^+ + HCO_3^-$	231.4654	−12092.1	−36.7816	0	$7*10^{-9}$
$HCO_3^- + H_2O \leftrightarrow H_3O^+ + CO_3^{2-}$	216.0504	−12431.7	−35.4819	0	$1.336*10^{-12}$
$NH_3 + H_2O \leftrightarrow NH_4^+ + OH^-$	−1.25656	−3335.7	1.4971	−0.037057	$2.84*10^{-7}$
$NH_3 + HCO_3^- \leftrightarrow NH_2COO^- + H_2O$	−4.58343	2900	0	0	**30.1306**
$HCN + H_2O \leftrightarrow H_3O^+ + CN^-$	22.90254	−9945.53	0	−0.049579	$1.7*10^{-10}$

$\ln(K_{eq}) = A + B/T + C\ln(T) + DT$ T in Kelvin.

tems over the entire range of electrolyte concentrations with a single set of binary interaction parameters.

SIMULATION RESULTS

When evaluating the performance of steam stripping plants, it is important to investigate whether the units are operated below the limits of government regulation. The operating conditions should also be examined for the purpose of maintaining the process close to the economically optimum operation in the steady state. However, tests are usually impossible or dangerous in commercial plants. Hence, a process simulator is normally used to examine the effect on the process of variations in a range of variables [Park and Kang, 1995; Oh and Moon, 1998]. In the present work, simulations were carried out to analyze the influence of various operating conditions on the stripping efficiency. The stripping efficiency of each component is the measure of the ability to strip the contaminant, and can be defined as follows [Walker, 1969].

$$\text{stripping efficiency} = \frac{\text{mass flow rate of stripped vapor in D/S}}{\text{mass flow rate of feed in D/S}} \times 100 \qquad (2)$$

Using this equation, the stripping efficiency of each component was calculated by a simulation through changes in the operating condition. The value was low in all cases, and differed for each component. The variables studied were the feed temperature, feed location and feed composition. Other variables that can affect the stripping efficiency such as the steam flow rate, reflux ratio, and recycle stream rate were also investigated. Though the multivariate analysis considering all operating variables is needed, it is very time-consuming to simulate all cases. In our paper, the stripping efficiency is evaluated through univariate analysis, respectively. To analyze the tendency of stripping efficiency according to an operating variable may also be helpful to find optimal operating condition since the other related variables are assumed to be controlled perfectly.

The results of the steady state simulation were compared with the actual plant data to verify the reliability of the simulation. It is shown that the behavior of the actual plant could be predicted quite well by the simulation model (see Table 3). It should be noted that in the steady state simulations all stages are treated as equilibrium reactive stages, and the control of column pressure is perfect.

1. Effect of Changing the Feed Temperature

As the feed temperature increases (60-74 °C), the interior temperature of A/S and D/S increases uniformly. This phenomenon

Table 3. Comparison of data between actual system and simulation results

			Actual data	Simulation results
D/S	Effluent vapor flow rate (kg/h)		No data	830
	Top temperature (°C)		91-94	93.5
	Bottom temperature (°C)		99-101	100.3
A/S	Top temperature (°C)		98-101	99.9
	Bottom temperature (°C)		111-113	112.3
D/S effluent liquid (BTM1)	Average flow rate (kg/h)		41540	43849
	Composition (mg/*l*)	NH_3	17000-23000	21166
		H_2S	1200-2200	2075
		CO_2	2000-6000	2980
		HCN	No data	131
A/S effluent liquid (BTM2)	Average flow rate (kg/h)		86600	86842
	Composition (mg/*l*)	NH_3	Below 100	93
		H_2S	Below 15	1
		CO_2	Below 150	<1
		HCN	No data	10

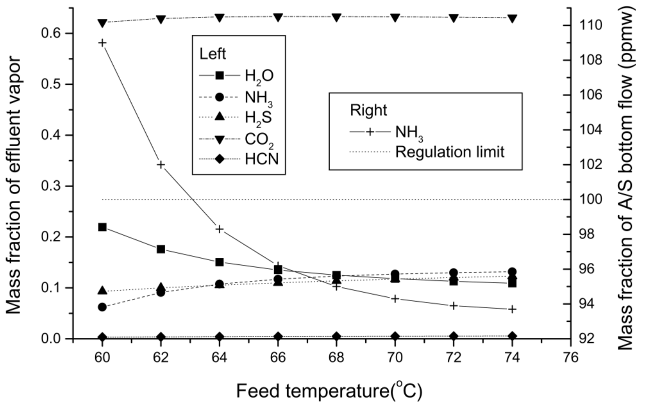


Fig. 2. Effect of changing feed temperature on the mass fraction of each component in VAP and that of NH_3 in BTM2.

affects the rates of vapor and liquid mass flow in D/S (vapor rates increase, and liquid rates decrease). In particular, the stripping efficiency of all components can be improved by increase of feed temperature in D/S. This is clear from Fig. 2, which shows that the mass fractions of NH_3 and H_2S increase with increasing temperature while that of H_2O decreases. This means that less steam is required to remove H_2S and NH_3 if the feed is preheated to higher temperature. However, the feed need not be preheated above 70 °C because the mass fractions of H_2S and NH_3 in the effluent vapor (VAP) barely increase above this temperature.

2. Effect of Changing the D/S Feed Location

The effect of the injected feed stage of D/S on the mass fraction of D/S product streams is shown in Fig. 3. When the rectifying sections are used, the reflux has a very high concentration of NH_3 and H_2S and can affect the concentration profile in the column. Fig. 3 shows that the mass fraction of H_2O is minimized and that of H_2S is maximized when the feed is injected into the fourth or fifth stage. These results prove that the rectifying section is necessary for the efficient removal of H_2S and NH_3.

3. Effect of Changing the Feed Composition

Simulations in which the feed composition is varied over the range of the actual data listed in Table 3 show that changing the NH_3 composition affects the column temperature profile more than changing any other component. Fig. 4(a) shows that the temperature of each stage in D/S decreases as the feed composition of NH_3 increases, except for the first and second stages. The temperature inversion at the first and second stages can be explained as follows. The inversion is caused by the heat of vaporization that is generated when each liquid component converts into vapor. From the simulation results, the vapor fraction of NH_3 and H_2O in the top of D/S increases as NH_3 composition in the feed increases. The components that generate the most heat on condensation to liquid are NH_3 and H_2O; hence, tray temperature inversion occurs. Changing the composition of H_2S, CO_2 and HCN in the feed has almost no influence on the temperature of A/S and D/S. It can be concluded from these simulation results that the change of NH_3 composition in the feed is the principal cause of the change in column temperature.

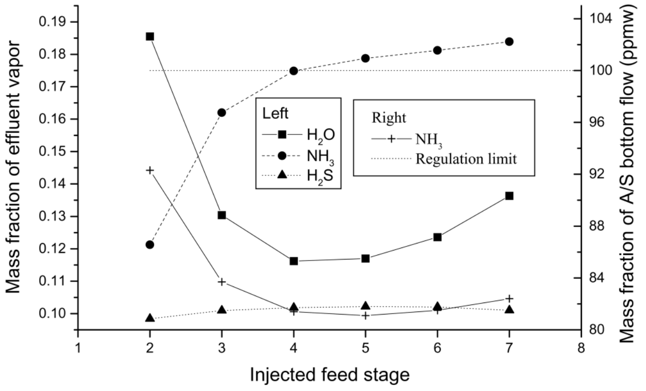
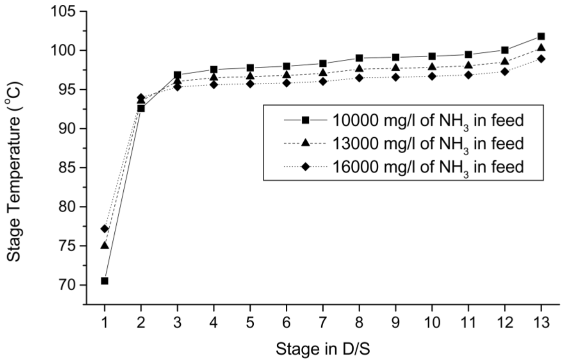
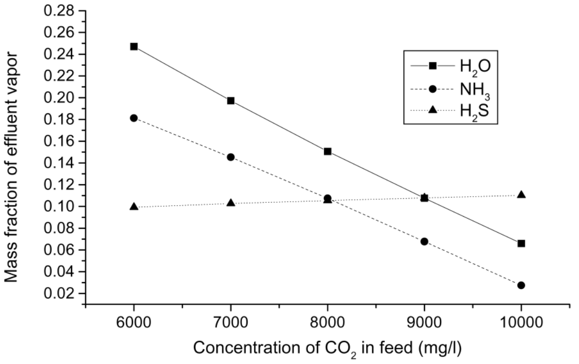


Fig. 3. Effect of changing the injected feed stage of D/S on the mass fraction of each component in VAP and that of NH_3 in BTM2 (HCN result is not shown due to its relatively low value).



(a) Effect of feed composition change of NH_3 on temperature profile in D/S



(b) Effect of feed composition change of CO_2 on mass fraction of effluent vapor (HCN is not be shown owing to relative low value)

Fig. 4. Effect of changing the feed composition of (a) NH_3 on the temperature profile in D/S and (b) CO_2 on the mass fraction in VAP.

Fig. 4(b) shows that CO_2 influences the stripping efficiency of other components. NH_3 is stripped less easily in the presence of CO_2, but H_2S and HCN are stripped more easily. This phenomenon can be explained by the following equations.

$$CO_2+2\,H_2O\leftrightarrow H_3O^{+}+HCO_3^{-} \tag{R.2}$$

$$NH_3+HCO_3^{-}\leftrightarrow NH_2COO^{-}+H_2O \tag{R.3}$$

CO_2 combines with H_2O to give HCO_3^-. The reaction of HCO_3^- with NH_3 causes NH_3 to quickly become NH_2COO^- in water (refer to K_{eq} of this reaction in Table 2). Because the ionic form (NH_2COO^-) cannot be stripped by steam, the composition of NH_3 in the effluent vapor (VAP) decreases in D/S as the composition of CO_2 in the feed

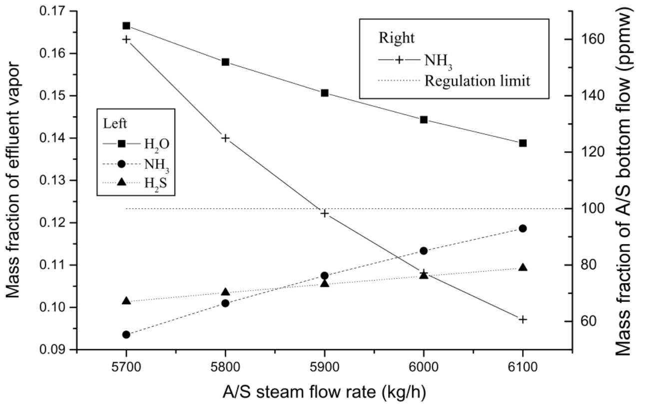


Fig. 5. Effect of changing the steam rate (STM2) on the mass fraction of each component in VAP and that of NH_3 in BTM2 (HCN result is not shown due to its relatively low value).

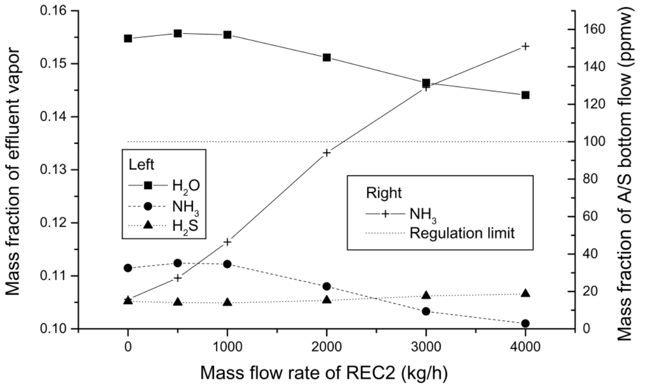


Fig. 6. Effect of changing the mass flow rate of the second side stream (REC2) on mass fraction of each component in VAP and that of NH_3 in BTM2 (HCN result is not shown due to its relatively low value).

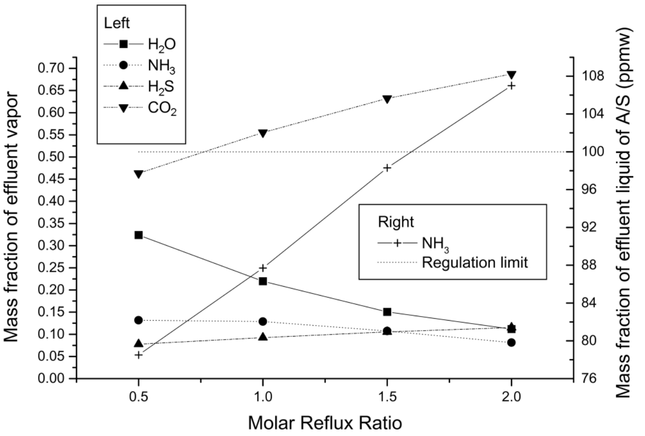


Fig. 7. Effect of changing the reflux ratio on the mass fraction of each component in VAP and that of NH_3 in BTM2 (HCN result is not shown due to its relatively low value).

Table 4. Comparison of system before and after changing condition

	Component	Before	After
D/S effluent vapor (mass fraction)	H_2O	0.151	0.125
	NH_3	0.107	0.179
	H_2S	0.105	0.102
	CO_2	0.632	0.590
	HCN	0.004	0.004
A/S bottom liquid (mass fraction)	NH_3	98 ppmw	22 ppmw
	HCN	10 ppmw	5 ppmw

Before: mass flow rate of the second recycle stream: 2,100 kg/h and feed stage is 2.

After: mass flow rate of the second recycle stream: 500 kg/h and feed stage is 4.

(FEED) increases.

4. Effect of Changing the Steam Rate of A/S and D/S

Fig. 5 shows the stripping efficiency versus the injected steam in A/S. As the steam rate of A/S increases, the mass fraction of the effluent vapor (VAP) increases for NH_3 and H_2S, but decreases for H_2O. The same effect occurs in D/S when STM1 is increased. Fig. 5 shows that NH_3 removal has a relatively strong dependence on the steam rate (STM2).

5. Effect of Changing the Recycle Stream Rate

Assume that the amount of the second recycle stream (REC2 in Fig. 1) can be manipulated. Fig. 6 demonstrates that as the second recycle stream (REC2) increases above 1,000 kg/h the stripping efficiency of H_2S in D/S increases, whereas the stripping efficiency of NH_3 and H_2O decreases. And the NH_3 composition of the bottom effluent (BTM2) of A/S violates environmental regulations (100 ppmw) when the second recycle stream (REC2) is above 2,100 kg/h. As shown in Fig. 6, the NH_3 bottom concentration of the bottom effluent (BTM2) of A/S can be reduced effectively when the second recycle stream (REC2) is maintained at a minimum value.

6. Effect of Changing the Molar Reflux Ratio

The reflux ratio is an important factor in the operating column. In general, products of higher purity are acquired with increasing reflux ratio. However, a compromise must be found in an industrial environment between product purity and the increased cost of the additional equipment and energy that are required to achieve a high reflux ratio. Fig. 7 shows that several effects occur as a result of increasing the reflux ratio: the mass fractions of CO_2 and H_2S in the effluent vapor (VAP) increase, while the mass fractions of H_2O and NH_3 decrease.

7. Improvement of Operating Efficiency

The simulation results presented above show that the column efficiency is improved when the CO_2 composition of the feed is at a minimum, the feed stage is 4 or 5, the mass flow rate of the second recycle stream is at a minimum, the reflux ratio is high, and the feed temperature is high. However, increasing the feed temperature or reflux ratio is very costly. Moreover, it is difficult to reduce the CO_2 concentration in the feed (reducing the CO_2 concentration in advance is another issue). Therefore, it is recommended to manipulate the remaining conditions. Table 4 compares the simulation results before and after changing the operating conditions (the mass flow rate of the second recycle stream is decreased from 2,100 to 500 kg/h and the feed stage is changed from 2 to 5. The separation performance is improved after these changes in the operating condition. Under the new conditions, the steam quantity can be reduced from 5,900 kg/h to 5,560 kg/h when the NH_3 composition of A/S bottom flow (BTM2) is fixed at the value of 100 ppmw.

ALTERNATIVE SCHEME FOR EFFECTIVE PRESSURE CONTROL

Most of the simulations described above were performed with a view to improving the sour water stripping efficiency and reducing the operating costs under given situations. Generally, a sour water stripper requires a large amount of steam to reduce the NH_3 contents of the final effluent. Furthermore, when the NH_3 composition of the feed increases, the column pressure rises rapidly, causing the operation to become unstable in the real system [Lee et al., 2002]. Controlling the column (D/S) pressure by manipulation of the effluent vapor rate is problematic in a real system; hence, the column pressure is controlled by adjusting the feed flow rate or steam rate. However, this causes other problems such as entrainment and maldistribution. Manipulating the feed temperature may provide an alternative method for controlling the column pressure. However, adjusting the feed temperature involves problems such as increased energy consumption and a long time delay to activate control action because of the large feed quantity (about 80,000 kg/hr). Lee et al. [2002] proposed an alternative scheme for effective pressure control of sour water stripping. In this paper, we introduce the key idea of the alternative scheme since it gives a general guideline for stably operating the sour water system. Under the proposed scheme shown in Fig. 8, a pump-around side cooler is used to reduce the heat duty of the overhead condenser and the vapor loading in the steam distillation. When the NH_3 composition of the feed increases, heat generation takes place, causing an increase in the vapor load in the column. This increase in vapor load causes a rapid rise in the column pressure. Use of a pump-around side cooler allows us to control the column pressure by reducing the vapor load. The results of dynamic simulations of the proposed control scheme, presented in Fig. 9, show that the column pressure is properly controlled (liquid of 16,000 kg/hr is pulled from the second tray (third equilibrium stage) of D/S and reenters the first tray (second equilibrium stage)). This structure gives a fast control action for abrupt increase of the top pressure in D/S. The slight drop in stripping efficiency associated with the proposed method can be compensated for by changing the operating condition in the manner mentioned in simulation results section.

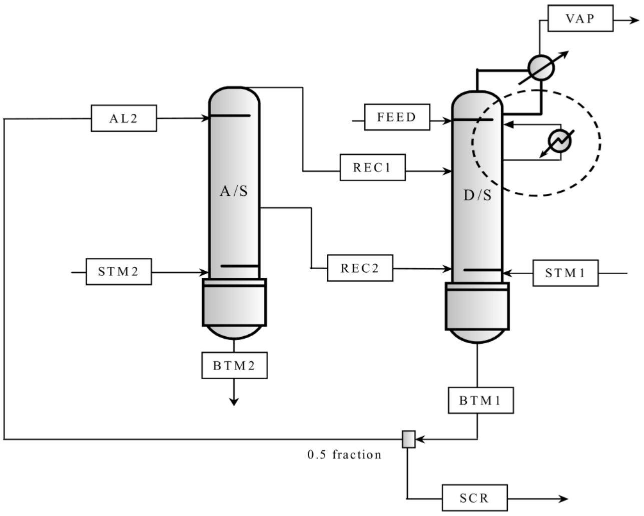


Fig. 8. Modified scheme to properly control the column pressure.

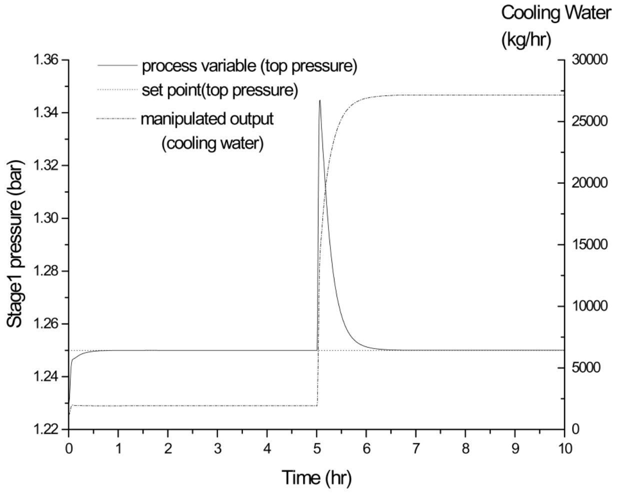


Fig. 9. Control performance of the new control scheme when NH_3 concentration in FEED increases from 13 g/*l* to 14 g/*l* at 5 hr.

CONCLUSION

The operation of, and problems associated with, sour water stripping were studied. Various steady state simulations were performed using the ELECNRTL property method to evaluate the effects associated with changing the following process parameters: location of feed injection, feed temperature, feed composition, steam rate, recycle stream rate, and reflux ratio. The simulation results reveal that CO_2 substantially influences the stripping efficiency of other components. Moreover, the simulation results show that the stripping efficiency is improved when the feed temperature is high, the feed composition of CO_2 is at a minimum, the feed stage is 4 or 5 (demonstrating the necessity of the rectifying section), the mass flow rate of REC2 is at a minimum, and the reflux ratio is high. In addition, we introduce a modified scheme to effectively control the column pressure. This modified structure shows acceptable control performance under sudden pressure changes. Consequently, the proposed structure and guidelines can be applied to reduce steam consumption and to operate the system stably.

ACKNOWLEDGMENT

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NOMENCLATURE

A, B, C, D : equilibrium coefficients
K_{eq} : equilibrium constant
T : temperature [K]

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