

ABSORPTION OF AMMONIA USING PACKED COLUMN

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Abstract :

A crosscurrent packed column is suitable for gas liquid mass transfer was constructed and operated. The absorption column consisted of a rectangular box constructed of PVC and metal. Deflection baffles are positioned at rectangular interval on the opposite sides of the central packed section to divert the gas phase into the packing affecting a crisscross gas liquid flow pattern. The column was used for absorbing ammonia from air in an ammonia-air-water system. Pressure drop data were collected for the conventional crosscurrent arrangement. The result indicate that crosscurrent operation with $\alpha = 1$ (α is baffle spacing variable has higher pressure drop per meter of packed higher than the countercurrent operation. When α is decreased to 2 and 3 the crosscurrent column has better pressure drop characteristics than the countercurrent column

1.INTRODUCTION :

Gas absorption is the unit operation in which one or more soluble component of a gas mixture are dissolved in a liquid. Objectionable pollutants may be removed from the gas stream or valuable vapor carried by the gas stream may be recovered for reuse. This diffusional transfer of mass from one phase to another has always been important to the chemical industry and is now of primary interest because of growing environmental concerns and increasing solvent costs. One of the most common pieces of absorption equipment is the Packed column. The packed column is a simple device consisting of a vertical shell filled with one of numerous types of inert packing materials. The two phases, usually one gas phase and one liquid phase, flow through the tower and the packing. The pieces of packing cause flow disruptions and are designed to promote contact between the two phases and thus increase the mass transfer. Two types of packed tower schemes are common. The first type is the cocurrent operation in which the gas and liquid phases are introduced into the tower at one end and flow in the same direction through the packing. The second type is the countercurrent operation in which the two phases are introduced at opposite ends of the tower, the liquid into the top and the gas into the bottom, and flow in opposite directions. Ordinarily, countercurrent flow is preferable to cocurrent because a higher concentration driving force and thus better mass transfer exists throughout the tower; this can be easily shown through elementary mass transfer theory. However, there are disadvantages of the countercurrent tower as compared to the cocurrent packed tower. In a countercurrent tower, increasing the flow rate of either phase tends to decrease the flow area available for the other phase. One consequence

of this is that the pressure drop required to move the gas phase through a countercurrent tower can be significantly higher than for a cocurrent tower. In a limiting case, the flows can be such that the liquid phase will stop flowing downward, a condition called flooding. Also, at low liquid rates the liquid tends to collect into small rivulets and flows along localized paths through the packing leaving much of the packing surface dry. This effect is known as channeling and is one of the chief reasons for poor performance in large counter current packed towers. A third less well known type of flow scheme for packed towers do exist. This is called the crosscurrent packed tower. As a single stage unit, the liquid phase enters the top and the gas phase enters at side, causing the gas phase to flow perpendicularly to the liquid flow.

A device that produces a cascade of cross flow units can be constructed. While overall operation is countercurrent, phase flow within individual stages is opposed at 90 degree and inter stage flow patterns are essentially crisscross in nature. The purpose of this project was to construct a pilot-scale multiple stage crosscurrent packed column and conduct overall mass transfer studies in order to effect a critical comparison between crosscurrent and the more conventional countercurrent packed columns. A detailed mass transfer study would include the following variables:

1. Liquid and gas flow rates
2. Vertical baffle spacing
Intercore to outer wall baffle spacing (or open space vs. packed space ratio)
3. Types of packing material
4. Systems with different gas solubilities

The final result would be to develop a conceptual model and correlations that incorporate these variables into a general design Procedure for crossflow devices. The scope of this investigation is limited to studying the effects of the first two. Pall rings were chosen for the packing material for all runs because previous studies reported favorable pressure drop characteristics when used in crossflow applications. The ammonia-air-water system was chosen because it is of high commercial importance, is relatively safe and easy to work with when proper precautions are taken, and necessary thermodynamic and physical data are readily available. Although published data for absorption of ammonia using a countercurrent packed tower are available, similar data were also taken using the same pilot-scale tower. Comparison with published data can be misleading since small packed towers often behave unlike large ones. Superficially, it would appear that crosscurrent flow devices are less desirable than countercurrent ones because the crosscurrent flow has inherently less efficient mass transfer characteristics than the countercurrent mode. However, the vigorous contacting patterns produced in a cross flow cascade may compensate for

this loss inefficiency. Also, other favorable characteristics such as low pressure drops, constant redistribution of the liquid phase, and wide operating ranges may make the multiple stage crosscurrent packed column a viable alternative mass transfer device. This report will first examine the literature on crosscurrent columns. Then the equipment used for this study and all relevant calculations and operating procedures will be described. Finally, the results of this experimental study will be presented and discussed.

EXPERIMENTAL PROCEDURE :

Firstly, The water flow rate was set at 1L/min, and the water was allowed to flow for 10 minutes to allow the packing to be fully wetted. Then bottle one and two were filled up with 5ml and 20ml of sulphuric acid respectively before being diluted with distilled water up to an equal mark on both bottles. 6 drops of Phenolphthalein indicator was then added to the two solutions. Air flow was set to 14L/min and the air was allowed to bubble through the Ammonia supply bottle. The air then went up through the column into the safety bottle of sulphuric acid. The stop watch was initiated as the three-way valve was turned on allowing the air to go through the absorption train (Bottle one & two). When the contents of bottle one changed colour to pink, the test was stopped by closing the three way valve and recording the time taken using the stop watch. Then the water containing the NH₃ was collected over the same period. The two bottles of acid were mixed together and were then used in back titration using 0.01M solution of sodium Hydroxide (NaOH). After that, 25ml sample of the water collected was also titrated using 0.01M solution of Sulphuric acid H₂ SO₄. The experiment was repeated for different air flow typically (14, 10, 8, 4 L/min). The results were tabulated in the tables shown in results & calculations section. The body of the paper consists of numbered sections that present the main findings. These sections should be organized to best present the material.

OBSERVATIONS :

The Column with the packed Raschig rings was showing vapour. The indicator showed the purple colour whenever the parameter changed. As soon as the three way valve is opened the ammonia/air mixture bubbles through the acid bottles. The Raschig rings are randomly positioned to give maximum contact area. The air rotameter was fluctuating at higher flow rates. As the Air flow rate increased errors started occurring and less reliable results were obtained. The volume of NaOH used for the back titration in the first test was unusually very high and the test was rerun and found to be normal. The air rotameter and water flow meter were not steady throughout the experiment. This could lead to possibilities of minor errors in the readings obtained. The Three-way valve was controlled manually. There are possibilities of discrepancies in the time recorded to shut the valve.

PACKING :

The packing is the most important component of the system. The packing provides sufficient area for intimate contact between phases. The efficiency of the packing with respect to both HTU and flow capacity determines to a significant extent the overall size of the tower. The economics of the installation is therefore tied up with packing choice. The packings are divided into those types which are dumped at random into the tower and those which must be stacked by hand. Dumped packing consists of units 1/4 to 2 inches in major dimension and is used mostly in the smaller columns. The units in stacked packing are 2 to about 8 inches in size; they are used only in the larger towers.

The Principal Requirement of a Tower packing are:

- 1) It must be chemically inert to the fluids in the tower.
- 2) It must be strong without excessive weight.
- 3) It must contain adequate passages for both streams without excessive liquid hold up or pressure drop.
- 4) It must provide good contact between liquid and gas.
- 5) It must be reasonable in cost.

Thus most packing is made of cheap, inert, fairly light materials such as clay, porcelain, or graphite. Thin-walled metal rings of steel or aluminum are some types used.

Common Packings are:

- a) Berl Saddle.
- b) Intalox Saddle.
- c) Raschig rings.
- d) Lessing rings.
- e) Cross-partition rings.
- f) Single spiral ring.
- g) Double - Spiral ring.
- h) Triple - Spiral ring.

Size of the Packing :

Now we will find the maximum size of Intalox saddle which would be used for this particular diameter of the column.

Packing size = 0.0666 m = 66 mm

Although the efficiency is higher for small packing, it is generally accepted that it is economical to use these small sizes in an attempt to improve the performance of a column. It is preferable to use the largest recommended size of a particular type of packing and to increase the packed height to compensate for small loss of efficiency.

Fundamentals of Material & Energy Balance :

Material balances are the basis of process design

Material balance is also a useful tool for the following:

1. The study of the plant operation & troubleshooting.
2. Check performance against design.
3. Check the instrument calibration

Conservation of Mass:

The general mass balance equation:

Input – output + generation – consumption = Accumulation

For steady-state non reactive system: Input = Output

[Number of equations = Number of components]

For steady-state reactive system: Input – output + generation – consumption = 0.0

If air consists of 20% by weight of nitrogen and 80% by weight of Ammonia

(a) the mean molecular weight of air

(b) the mole fraction of ammonia

(c) the concentration of ammonia in mole/m³ and kg/m³ if the total pressure is 1.5 atmospheres and the temperature is 25 °C

(a) Taking the basis of 100 kg of air: it contains 20/28 moles of N₂ and 80/17 moles of NH₃

Total number of moles = 0.71 + 4.70 = 5.41 moles.

So mean molecular weight of air = 100 / 5.41 = 18.48

Mean molecular weight of air = 18.48

b) The mole fraction of NH₃ = 4.7 / (0.71 + 4.7) = 4.7 / 5.41 = 0.86

Mole fraction of NH₃ = 0.86

(c) In the gas equation, where n is the number of moles present: the value of R is 0.08206 m³

atm/mole K and at a temperature of 25 °C = 25 + 273 = 298 K, and where V = 1 m³

pV = nRT and so, 1.5 x 1 = n x 0.08206 x 298

n = 0.061 mole/m³ weight of air = n x mean molecular weight = 0.061 x 18.48 = 1.12 kg / m³

and of this 23% is NH₃, so weight of NH₃ = 0.8 x 1.12 = 0.896 kg in 1 m³

Concentration of NH₃ = 0.896 kg/m³

When a gas is dissolved in a liquid, the mole fraction of the gas in the liquid can be determined by first calculating the number of moles of gas using the gas laws, treating the volume as the liquid, and then calculating the number of moles of liquid directly.

Energy Balance :

Energy takes many forms, such as heat, kinetic energy, chemical energy, potential energy but because of interconversions it is not always easy to isolate separate constituents of energy balances. However, under some circumstances certain aspects predominate. In many heat balances in which other forms of energy are insignificant; in some chemical situations mechanical energy is insignificant and in some mechanical energy situations, as in the flow of fluids in pipes, the frictional losses appear as heat but the details of the heating need not be considered. We are seldom concerned with internal Energies.

Therefore practical applications of energy balances tend to focus on particular dominant aspects and so a heat balance, for example, can be a useful description of important cost and quality aspects of process situation. When unfamiliar with the relative magnitudes of the various forms of energy entering into a particular processing situation, it is wise to put them all down. Then after some preliminary calculations, the important

ones emerge and other minor ones can be lumped together or even ignored without introducing substantial errors. With experience, the obviously minor ones can perhaps be left out completely though this always raises the possibility of error. Energy balances can be calculated on the basis of external energy used per kilogram of product, or raw material processed, or on dry solids or some key component. The energy consumed in food production includes direct energy which is fuel and electricity used on the farm, and in transport and in factories, and in storage, selling, etc.; and indirect energy which is used to actually build the machines, to make the packaging, to produce the electricity and the oil and so on. Food itself is a major energy source, and energy balances can be determined for animal or human feeding; food energy input can be balanced against outputs in heat and mechanical energy and chemical synthesis.

In the SI system there is only one energy unit, the joule. However, kilocalories are still used by some nutritionists and British thermal units (Btu) in some heat-balance work. The two applications used in this chapter are heat balances, which are the basis for heat transfer, and the energy balances used in analysing fluid flow.

OPERATING PROCEDURE :

1 .Preliminary Operating Procedures: The column and all support equipment were constructed in the manner and dimensions previously described. A routine maintenance check on the blower was done and repeated after every one hundred hours of operation. The column's upper access port and top retainer screen were removed and the central section was filled with the structured rings. This packing material was dumped into the column in a random Manner. During this operation, the column contained no liquid and was not shaken. Once placed in the tower, the packing material was not disturbed for the

remainder of the study. During this investigation the column was operated as both a crossflow and countercurrent device. The necessary internal arrangements were accomplished by removing the 127 mm by 2.44 m PVC sides. The baffles or wood blocks were arranged properly; the PVC sides were repositioned; all but joints were sealed with clear Silicone and, the sides were secured by tightening the metal screws. The Silicone Seal was allowed to dry overnight before continuing. Solutions of sodium hydroxide and sulfuric acid of approximately twotenth normal were prepared and titrated against standards to determine exact concentrations.

2. Procedure for data run: After arranging the internals of the column, the following procedure was followed to generate the required data.

Start-up: The following steps were performed:

1. Open the liquid outlet valve.
2. Open manometer system valves 1, 4, and 5.
3. Install the appropriate orifice for the desired flow range (beveled edge on the downstream side).

4. Turn on the air blower and open air control valve mechanism about one-third. Allow about twenty minutes before data collection for inlet air temperature to stabilize. 5. Open water control valve for small rotameter until float registers about ten percent of scale.

6. Open main valve on the ammonia tank and slowly open the regulator until 70 to 100 kPa (10 to 15 psi) registers on the outlet pressure meter. Keep the rotameter control valve closed and check ammonia system for leaks (ammonia produces a white cloud in the presence of concentrated hydrochloric acid solution).

3. Shut down :After the desired data were taken, the following procedure was followed to shut-down the column.

1. Close the valve on the ammonia tank and then close the regulator valve and the ammonia Rotameter control valve.

2. Turn off the water supply and drain all water lines.

3. Turn off the air blower and record operating time in the maintenance log.

4. Close all valves in the manometer system to prevent evaporation of manometer fluid.

ANALYSIS OF ERRORS :

The significance of conclusions based upon the numerical results of this study can be properly determined only after careful consideration of the reliability of the data. Several types of errors can contribute to the inaccuracy of the results; efforts were taken to minimize the errors associated with poor equipment performance and those that arise as a result of approximations and assumptions made in the theoretical development of equations. However, as in any investigation, the results obtained were a compromise between desired accuracy and minimization of time and

expense required to obtain that accuracy. All measurements may be classified as either direct or indirect. Direct measurement is made whenever the magnitude of the measured quantity is determined by direct observation from the measuring instrument. Estimates of errors associated with these measurements are based on the reported errors given in operating manuals or on the scale increments of the instrument; estimated error is ordinarily one-half the scale increment that is easily read. The magnitude of an indirect measurement is determined by calculation from the magnitudes of other quantities directly measured using some functional relationship that exists among the quantities. The estimate of error is more difficult to determine for an indirect measurement than for a direct measurement since the errors in the direct measurements may either augment or offset each other's effect on the error of the calculated result, depending upon their signs and the form of the functional relationship.

CONCLUSIONS

The results of this study show that the multiple stage crosscurrent packed column is comparable to and at some conditions more efficient than the conventional countercurrent packed column. The crosscurrent column can process significantly higher flow rates with the same pressure drop and absorption efficiency and with the same packed volume as the countercurrent packed column. However, this study only considered the effect of two variables, flow rates and vertical baffle spacing, and conclusions cannot be generalized without further research. The favorable results with the ammonia-air-water system, structured ring, and the open area to packed area ratio of this experimental column definitely justify further research. The effects of all possible variables need to be studied before the performance of the multiple stage crosscurrent packed column can be completely understood and the design utilized.

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