Kocaeli University

Department of Chemical Engineering

General Rules of Chemical Engineering Laboratories

1. Students take a written quiz about the experiment to be performed in the beginning of the laboratory. Any student whose quiz grade is smaller than 50% will take zero from the performance and report. In addition student is considered ‘absent’ in that week. But if student wants to participate in the experiment just for learning and practicing, he/she can join the laboratory and also help the preparation of experiment report.

2. The student who does not attend the quiz or laboratory is considered ‘absent’. The performance and report grades for this student are zero.

3. All students have to obey the laboratory rules to be announced. The student who opposes these rules will be dismissed without any warning. The performance and report grades for this student are zero.

4. The damaged/broken glassware belonging to experimental set-up must be obtained and submitted to the laboratory coordinator. All equipments used during experiments are under the responsibility of the experimenting group.

5. The experiment report must be prepared according to the report format using computer or with hand-writing and submitted as group-report (not individual reports).

6. It is obligatory to submit reports in the next experiment day (normally next week) till 17:00. In case of one day of delay in submission, report is accepted but will be evaluated over 80%. The delays of more than one day are not acceptable! In this case report grade will be zero. During the submission of the reports, all group members should be ready for oral quiz (discussion) conducted to measure the level of participation of the members in “report preparation”.

7. The evaluation of experiments (experiment grades) will be conducted as follows:
   - Written quiz (held before the experiment): 30%
   - Experiment performance: 20% (Given by the assistant of the experiment with respect to the participation of the students to the experiments, the level of satisfaction of the answers to the questions of assistants during the experiments, and general behavior of students between the borderlines of the laboratory rules)
   - Report: 50% (including the oral quiz/discussion)

8. 80% attendance (9/11 experiments) is a must to attend the final exam.

9. The general evaluation of Chemical Engineering Laboratory will be conducted as follows:
   - Average of experiment grades: 60%
   - Final Exam: 40%
EXPERIMENT 1. GAS ABSORPTION

OBJECTIVES

The objective of the experiment is to determine absorption of carbon dioxide (CO₂) to water and dependence of the pressure drop on the flow rates in a packed column. Effect of chemical reaction on absorption mechanism will be investigated.

1. Introduction

In gas absorption process, a component carrier by a gas stream is absorbed in a liquid phase. As in literature noted, instead of a tray (plate) column, a packed column can be used for various unit operations such as continuous or batch distillation or gas absorption. With a tray column, the vapours leaving an ideal plate will be richer in the more volatile component than the vapour entering the plate by one equilibrium "step". When packings (or packing materials) are used instead of trays, the same enrichment of the vapour will occur over a certain height of packings, and this height is termed the height equivalent to a theoretical plate (HETP). As all sections of the packings are physically the same, it is assumed that one equilibrium (theoretical) plate is represented by a given height of packings. Thus the required height of packings for any desired separation is given by (HETP x Number of ideal trays required). HETP values are complex functions of temperature, pressure, composition, density, viscosity, diffusivity, pressure drop, vapour and/or liquid flowrates, packing characteristics, etc. Empirical correlations, though available to calculate the values of HETP, are restricted to limited applications. The main difficulty lies in the failure to account for the fundamentally different action of tray and packed columns.

Packed columns are used in chemical industry to absorb a gas from a mixture of gases or strip a volatile substance from a liquid. The columns are, usually, countercurrent gas-liquid contactors in which gas flows upward and liquid downward. To provide large interfacial area for mass transfer between gas and liquid, the columns are filled with packing. Design of the column involves estimation of diameter of the column and height of the packing required for specified separation. The diameter is determined from flooding characteristics of the column and, the height is found from mass transfer characteristics of the packing and the gas-liquid system. Flooding depends on pressure drop across the column, it being higher at the bottom and lower at the top to allow the gas to flow upward. The gas flow is usually turbulent and in a dry column, the pressure drop rises with gas flow with an exponent of 1.8 to 2.0 - a typical feature of turbulent flow. The pressure drop rises with an increasing flow of liquid because liquid fills up the column and the space for gas flow is reduced. Up to the loading point, the pressure drop follows the same relation as in dry run. Beyond the loading point, the pressure drop rises rapidly with gas flow and the liquid hold up in the column also rises. Eventually, at the flooding point, the pressure drop rises drastically and the liquid may splash back from
the column. The gas velocity corresponding to the flooding point is called flooding velocity and the column is operated at some fraction of this velocity. Tower height is determined by packing characteristics, namely, interfacial area, mass transfer coefficient of the gas-liquid system and extent of separation \[1,2\].

The gas and water streams enter the column from the bottom and the top, respectively. A typical gas absorption column is given in figure 1. The flow rates of these streams are controlled using rotameters. The CO\(_2\) composition of the inlet gas stream is controlled by valves for CO\(_2\) and air. The pressure drop in the gas phase is measured using the manometer. The CO\(_2\)-air mixture flows up a packed column where it comes in continuous contact with water travelling downward. Mass transfer takes place over the packing in the column whereby some of the CO\(_2\) is absorbed into water. The rate of mass transfer is governed by stream flow rates, interfacial contact area, component, diffusivities, temperature, pressure, and concentration. The concentration of CO\(_2\) in the liquid streams are determined using titration method by NaOH.

![Figure 1. Packed tower flow and characteristics for absorption](image)

2. EXPERIMENTAL

2.1. Procedure

1. Fill the feed tank with water.
2. First open air rotameter valve slowly to reading of 10 L/min and wait until steady state condition is reached. Then record pressure drop on manometer. Repeat this step by changing air flow (from 20 to 80) to measure pressure drops.
3. For wet column open water rotameter valve to reading of 10 L/min and read pressure drop while air flow rate is 10 L/min. Proceed this procedure while water and air flow rates are changed.

4. At the final step, set the CO₂ 20 L/min, air 20 L/min and water 10 L/min. And take 20 mL sample from the tank and bottom of the column after 15 minutes for titration. Repeat the same procedure in 30 and 45th minutes.

5. Titrate each samples with 0.5 M NaOH using 2-3 drops of phenolphthalein as indicator. Record the volume of NaOH used at neutralization point.

Figure 2. Gas absorption column

2.2. Useful Data
D (inner diameter of column): 75 mm
Hₘ (length of column): 1400 mm

References
EXPERIMENT 2: SOLID - LIQUID EXTRACTION BY SOXHLET APPARATUS

OBJECTIVES

The object of this experiment is to study the basic principles of solid-liquid extraction by using soxhlet apparatus.

1. INTRODUCTION

The transfer of a solute from one phase to another is a very common technique in organic chemistry. It is called extraction, and it is also a common technique in everyday life. When you steep a tea bag in boiling water, add a bay leaf to a pot of soup, or wash a load of laundry, you are performing a solid/liquid extraction. In addition, in many areas of industry we can see the solid-liquid extraction process.

2. SOLID-LIQUID EXTRACTION

Solid–liquid (or liquid–solid) extraction, involves the removal of a soluble fraction (the solute or leachant) of a solid material by a liquid solvent. The solute diffuses from inside the solid into the surrounding solvent. Either the extracted solid fraction or the insoluble solids, or both, may be valuable products. Solid–liquid extraction (Leaching) is widely used in the metallurgical, natural product, and food industries. In metallurgy, leaching may involve oxidation or reduction reactions of the solid with the solvent.

Solid–liquid extraction process can be considered in three parts:

1. Diffusion of the solvent through the pores of the solid
2. The diffused solvent dissolves the solutes (i.e. transfer the solute to the liquid phase).
3. Transfer of the solution from porous solid to the main bulk of the solution.

Figure 1. Schematic extraction – before extraction (left) and after extraction (right): 1 solvent, 2 extraction material (solid carrier phase with transition component), 3 transition component, 4 depleted solid carrier phase, 5 solvent with dissolved transition component.

Industrial applications of leaching include: (1) removal of copper from ore using sulfuric acid, (2) recovery of gold from ore using sodium-cyanide solution, (2) extraction of tannin from tree bark using water, (3) recovery of proteins and other natural products from bacterial cells. (4) Soybean oil, the main cooking oil in North America, is obtained by leaching
soybeans with hexane. (5) Sugar is leached out of beets or sugar cane with water. (6) Caffeine is removed from coffee beans with methylene chloride, ethyl acetate, or supercritical carbon dioxide. (7) Less benignly, cocaine is recovered from cocoa leaves with acetone.

2.1. Soxhlet Apparatus

Soxhlet, which has been used for a long time, is a standard technique and the main reference for evaluating the performance of other solid–liquid extraction (or leaching) methods. Soxhlet extraction is a general and well-established technique, which surpasses in performance other conventional extraction techniques except for, in limited field of applications, the extraction of thermolabile compounds. In a conventional Soxhlet system as shown in Fig. 1, plant material is placed in a thimble-holder, and filled with condensed fresh solvent from a distillation flask. When the liquid reaches the overflow level, a siphon aspirates the solution of the thimble-holder and unloads it back into the distillation flask, carrying extracted solutes into the bulk liquid. In the solvent flask, solute is separated from the solvent using distillation. Solute is left in the flask and fresh solvent passes back into the plant solid bed. The operation is repeated until complete extraction is achieved.

![Experimental Soxhlet extraction apparatus.](image)

**Figure 2.** Experimental Soxhlet extraction apparatus.

2.1.1. Practical issues for Soxhlet extraction

- Solvent choice
- Operating conditions
- Matrix characteristics
2.1.2. Advantages and disadvantages of Soxhlet extraction

Advantages;

1. the displacement of transfer equilibrium by repeatedly bringing fresh solvent into contact with the solid matrix,
2. maintaining a relatively high extraction temperature with heat from the distillation flask, and
3. no filtration requirement after leaching. Also, the Soxhlet method is very simple and cheap.

Disadvantage;

1. the extraction time is long,
2. a large amount of solvent is used,
3. agitation can not be provided in the Soxhlet device to accelerate the process,
4. the large amount of solvent used requires an evaporation/concentration procedure and
5. the possibility of thermal decomposition of the target compounds can not be ignored as the extraction usually occurs at the boiling point of the solvent for a long time.

3. EXPERIMENTAL PROCEDURE

1. Weigh a quantity of nuts or oil containing ground seeds.
2. Crush and dry them in an oven for 30 minutes @ 105°C and determine the moisture content.
3. Place approximately 360 mL of the extraction solvent into a 500-mL round bottom flask, assemble the Soxhlet apparatus and start leaching and continue the process up to the end of the fourth siphon.
4. When the extraction time is complete, remove the filter paper & sample and carefully transfer it into an evaporating dish. Weigh the sample. Dry it in an oven. Weigh the sample the next day again.
5. Measure the volume of the hexane+oil mixture into the round bottom flask.
6. Measure the refractive index of the hexane+oil mixture into the round bottom flask.

REFERENCES