Technology

Cryogenic Air Separation Unit (ASU) Pilot Plant

KIHARA Hitoshi*

1. Introduction

Our company designs packed columns and condensers, which are the primary components of cryogenic air separation units (ASUs), using proprietary software developed through fundamental research. These packed columns and condensers are designed by theoretically modeling mass and heat transfer phenomena based on transport kinetics, and can be applied regardless of operating conditions such as composition or pressure[1) 2)]. For verification of these design tools, we have primarily used data obtained from a bench-scale experimental facility at our Plant Engineering Center in Kawasaki [3)].

Unlike general chemical plants, ASUs do not require consideration of fluctuations in feedstock composition. Therefore, process optimization through improved design accuracy of distillation columns and condensers directly leads to reductions in both capital investment and operating costs. To further strengthen the development of high-performance packing and condensers in recent years, we constructed a pilot-scale ASU - a new cryogenic demonstration test facility - at our Tsukuba Laboratory in 2021, replacing the former bench-scale experimental facility (Fig. 1).

2. Overview of the Facility

2.1 Equipment configuration

The main specifications of this facility are shown in Table 1, and the equipment configuration is illustrated in Fig. 2. The facility consists of a vacuum-insulated cold box that houses the packed column test units and condenser test unit, and an perlite-insulated cold box containing vaporizers and condensers for supplying and recovering gas and liquid to and from the test units. The test units to be evaluated are replaceable. Instead of compressing air, the facility is pre-charged with N_2, Ar, and O_2 of any desired composition according to the experimental conditions. These are circulated by means of an electric heater and liquid nitrogen supplied from a storage tank (not shown). This configuration makes it possible to reproduce arbitrary composition, pressure, and gas-liquid load conditions.

2.2 Operation Modes

Depending on the objective, this facility is capable of performing the following three types of experiments:

(1) Packed distillation column experiments

Measurement of the distillation performance and pressure drop of packing, assuming the lower, upper, and argon columns of a double-column-rectification ASU, as well as single- column- rectification nitrogen generators.

Fig. 1 ASU Pilot Plant at Tsukuba Laboratory

* Cryogenic Separation Development Dept., Tsukuba Laboratory, R&D Unit

(2) Condenser experiments

Measurement of the heat transfer and operating performance, assuming the main and argon condensers of a double-column-rectification ASU[4].

Table 1 Main specifications of the facility

Gas processing capacity		867 kNm^3/day
Perlite-insulated cold box		W3.4 × D2.9 × H22 m
Vacuum-insulated cold box		ϕ 2.8 × H18 m
Vaporizer heater capacity		200 kW
Packed column test units	Column diameter	ϕ 0.4 m
	Packing height	1.5 m, 6 m
	Operating pressure	Up to 0.8 MPa (G)
Condenser test unit	Shell diameter × height	ϕ 0.9 × 6 m

(3) Mixing column experiments

Verification of the packing performance and operating characteristics of mixing column processes, which can reduce power consumption for low-purity oxygen (<97%) ASUs[5]. The auxiliary distillation column housed in the perlite-insulated cold box is used to produce liquid oxygen (LO_2) , which is supplied to the top of the packed column test unit 1.

3. Packed Distillation Column Experiments

As an example, Ar-O_2 system distillation experiments using packed column test unit 2 are described below, based on Fig. 2 (blue line).

3.1 Experimental Procedure

Liquified Ar-O_2 mixture pre-charged into the vaporizer is heated with an electric heater, and the generated vapor is introduced into the bottom of packed column test unit 2. The vapor rises through the column while contacting the reflux liquid, increasing the concentration of Ar, which is the more volatile component. The vapor discharged from the top of the column is completely liquefied in the condenser and returned to the top of packed column test unit 2 as reflux, while a portion is returned directly to the vaporizer (piping not shown). A portion of the condensed liquid is not used as reflux in order to reproduce partial-reflux operation asumed for the argon column of an ASU. Experiments were conducted at 150 kPa (abs) with bottom vapor O_2 concentrations of 8 vol% and 0.2 vol%. By adjusting the heater output, the O_2 concentrations in the gas and liquid at

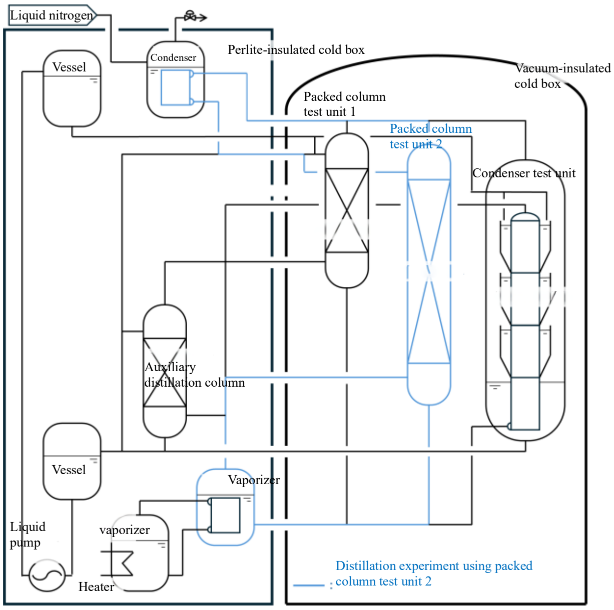

Fig. 2 Equipment configuration

the top and bottom of the column, as well as the pressure drop, were measured under different gas-liquid load conditions.

3.2 Experimental Results

The obtained distillation performance and pressure drop are shown in Fig. 3. The horizontal axis represents the vapor load expressed as the ratio to the design reference point. The vertical axes of the upper and lower graphs represent the H.E.T.P. (Height Equivalent to a Theoretical Plate) and pressure drop, respectively, also expressed as the ratio to the design reference point.

As shown in the figure, the distillation performance gradually improves as the vapor load exceeds the reference point, reaching its peak (minimum H.E.T.P.) near the loading point where the pressure drop increases sharply, and then deteriorates rapidly. On the other hand, no decrease in distillation performance was observed even when the vapor load (and thus the liquid load) was reduced. By incorporating these results into the design program, the parameters for the packing used in the experiments were determined, enabling optimal design of packed columns even under different operating conditions, such as composition and pressure.

4. Conclusion

Since the completion of this facility in 2021, our company has conducted performance evaluations of various packing structures and verification of cascade-type condensers. We are currently working on the development of proprietary condensers. In recent years, there has been an increasing demand for product configurations that differ from conventional ones, such as for the electronics industry, and ASU processes have become increasingly diversified[6]. To address these needs, we will continue to make use of this facility to reduce ASU equipment costs and operating power consumption.

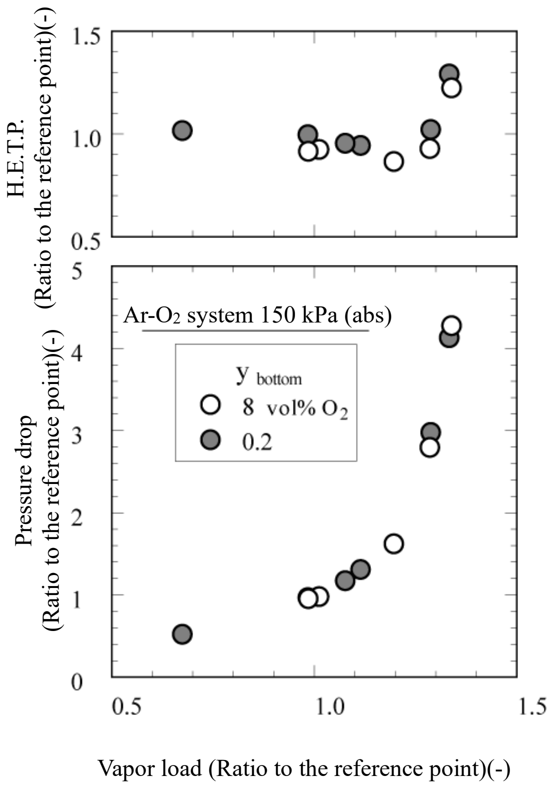

Fig. 3 Distillation test results of the packed column

References:

1) N. Egoshi, et al., Heat and Mass Transfer Model Approach to Prediction of Separation Performance of Cryogenic Air Separation Plant by Packed Columns with Structured Packing, J. Chem. Eng. Japan, 2001, 34(1), pp. 22-29.
2) S. Sakaue, Boiling heat transfer of Nitrogen in a thermosyphon condenser-reboiler, AIChE J., 1997, 43(2), pp. 339-344.
3) K. Ishizaki, Design of Packed Distillation Columns Using a Liquid Flow Simulator, Taiyo Nippon Sanso Technical Report No. 38, 2019.
4) S. Takayanagi, Concentration and Accumulation of Nitrous Oxide in a Cascade Condenser-Reboiler, Taiyo Nippon Sanso Technical Report No. 41, 2022.
5) N. Egoshi, Simulation of Mixing Column for Cryogenic Air Separation Unit, Taiyo Nippon Sanso Technical Report No. 40, 2021.
6) H. Tachibana, Nitrogen Generation Process for Co-production of Small Amounts of Ultra High Purity Oxygen and Argon, Taiyo Nippon Sanso Technical Report No. 43, 2024.