Development of a New Type of Centrifugal Vacuum Distillation System

Takashi Hanamoto*

1. Introduction

We have developed and launched a new type of centrifugal vacuum distillation system (model, CEH-400B II). Improved from conventional distillation systems in terms of compactness and operability, this new system can be operated even by those who are unskilled in distillation process operations.

Recently, packaging technologies and semiconductor devices have made progress and positive environmental measures have been introduced in the fields of electronic parts, FPD and other electronics materials. This situation has spurred demand for new functions and more advanced performance than ever before in material fields in recent years. There is also demand for more diversified and higher function products in conventional fine chemical industries, including medical and cosmetic products. At the same time, there is demand for further cost reduction and development of high value added products due to a higher crude oil price than ever before.

In these situations, fine chemical industries are required to stabilize the physical properties of materials in products, remove particular impurities and improve degrees of purity. They are forced to shift production from cost-oriented mass production to production of a wide variety of products in small quantities focusing on added value, showing a tendency toward made-to-order production.

Target components can be separated and refined from chemically synthesized materials and natural materials by using a membrane separation process or a separation

Table 1 Applications to Industrial Products

Material	Purpose (1)	Purpose (2)
Fluorine oil	To remove low boiling point components (Lubrication oil in vacuum equipments)	To adjust vapor pressures
Vacuum pump oil	To remove low boiling point components	To adjust vapor pressures
Epoxy resins	To remove chloride components (Electronic part materials)	To remove undesired substances
Photoresist	To remove metallic components (Electronic part materials)	To remove undesired substances
Resin hardener	To remove components colored by thermal denaturation (Electronic part materials)	To remove colors

Table 2 Applications to Food, Health Food and Medical Products

Material	Useful substances	Purpose
Fish oil (Sardine oil)	EPA ester Effective for preventing blood clots and reducing LDL cholesterol	For higher purity
Deep-sea shark liver oil	Squalane	For higher purity
Palm oil	Natural carotene	For concentration
Deodorized waste oil of soybean oil and other cooking oils	Vitamin E (Tocopherol; α, β, γ and δ-tocopherol is present in nature, classified by differences in molecular structures.) Effective against arteriosclerosis and vein thrombosis	For recovery For higher purity
Vegetable oil (Canola oil, soybean oil, palm oil, etc.)	Distillation of monoglyceride Emulsifiers for food and detergents	For higher purity
Refinement of materials of medical products	Removal of unreacted substances from synthesized products	For higher purity

* Industrial Equipment Division, ULVAC, Inc.

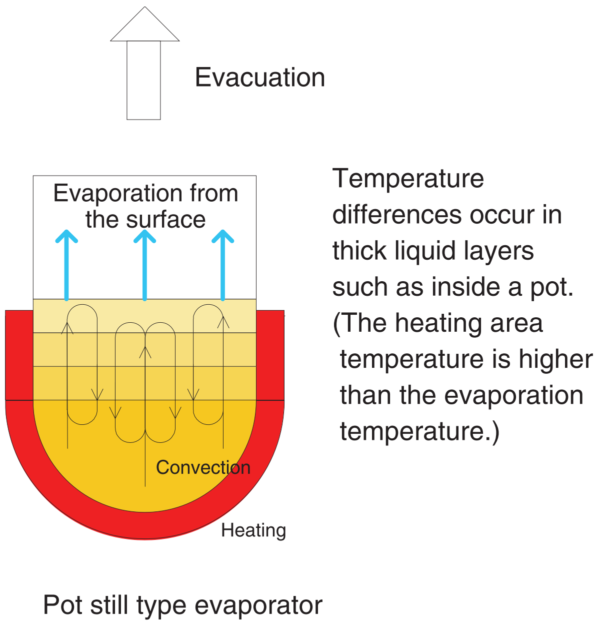


Figure 1　Schematic Diagram of Vacuum Evaporator

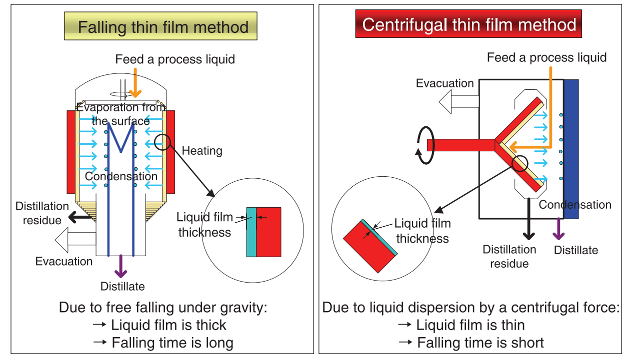


Figure 2　Falling Thin Film Method and Centrifugal Thin Film Method

process using adsorptive substances. A distillation process separates and refines particular components based on differences in their vapor pressures (boiling points) with easier industrial operations than the processes mentioned above. This method is adopted due to the following advantages:

1) Less product loss
2) Easy operation
3) Reduced maintenance cost

Because of high distillation temperatures at atmospheric pressure, vacuum distillation is used for products containing substances that oxidize, polymerize, decompose or change their nature in other ways. (Tables 1 and 2)

2.　Utilization of Vacuum Distillation Systems

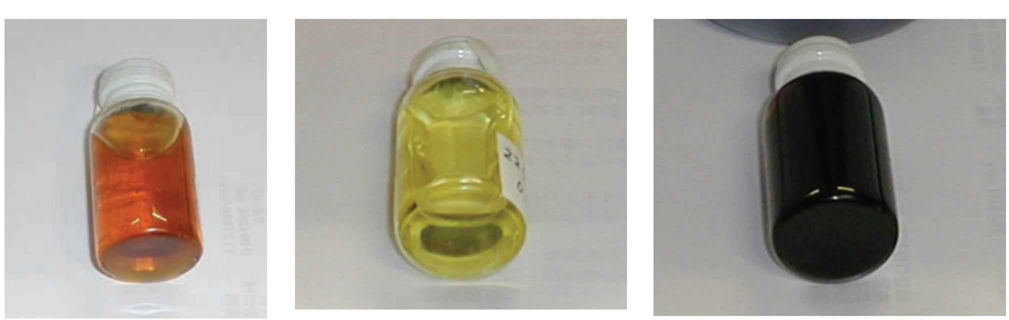
In one example, water evaporates vigorously at 100℃ in normal atmospheric pressure. When the pressure is decreased to 611 Pa in vacuum, water evaporates vigorously even at 0℃. Just like water, every liquid has a characteristic vapor pressure, and the evaporation temperature lowers as the pressure lowers.

At atmospheric pressure, evaporation temperatures are so high that heat-sensitive substances decompose or polymerize during operations at high temperatures and become impossible to refine by distillation. By reducing the operation pressure to vacuum, the evaporation temperatures decrease, enabling distillation operations that cannot be achieved at atmospheric pressure.

Heat-sensitive substances, such as vitamins, can be distilled at relatively low temperatures. This process has the advantages of reducing thermal modification and preventing the substances from decomposing or polymerizing due to oxidation, since a distillation system contains little oxygen.

On the other hand, in a vacuum liquids evaporate from the surfaces in contact with the vacuum. In a thick liquid layer, liquid heated to a high temperature rises to the surface (in contact with a vacuum) in convection currents before it evaporates. This is inefficient. (Figure 1)

Vacuum distillation processes use falling thin film evaporators and centrifugal thin film evaporators to reduce the



Material to distill　　Distillate　　Residue

Photo 1　Distillation Samples

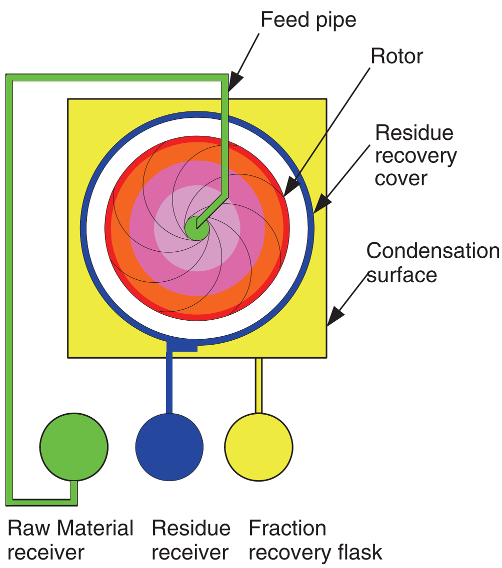


Figure 3　Schematic Diagram of Centrifugal Thin Film Method

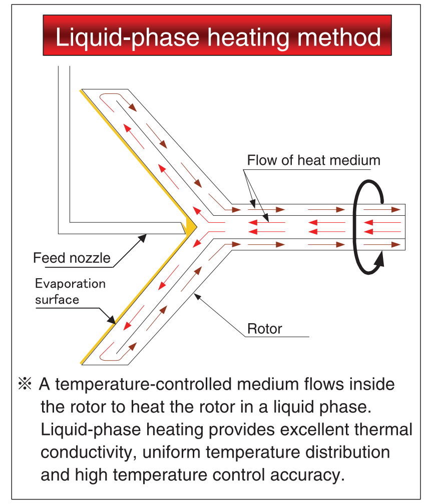


Figure 4　Conceptual Diagram of Liquid-phase Heating Method

depth of the processing liquid and increase the surface area in contact with the vacuum to improve evaporation efficiency as well as to reduce the liquid film thickness in order to decrease the difference between the heating temperature and the evaporation temperature. (Figure 2)

The falling thin film method feeds a processing liquid to a rotating dispersing plate to disperse the liquid uniformly to the upper part of a vertical cylindrical evaporator. The processing liquid evaporates as it flows along the inner wall of the evaporator. This method requires a liquid film thickness of 0.1 to 1 mm and a falling time of two to several tens of seconds.

The centrifugal thin film method feeds a processing liquid to the center of a conical evaporation surface and disperses the liquid by a centrifugal force generated by the rotating cone. This method can reduce the liquid film thickness to 0.01 to 0.1 mm and the time the liquid must stay on the heating surface to 1 second or shorter. (Figure 3)

For these reasons, the centrifugal thin film method is more suitable for distillation of heat-sensitive substances in high vacuum.

3.　Outline of the System

A centrifugal thin film evaporator has a mortar-shaped rotating disc (rotor) mounted sideways inside a vacuum tank to prevent the processing liquid from stagnating.

A high temperature liquid heat medium circulates inside the rotating disc to uniformly heat the evaporation surface. (Figure 4) A processing liquid is fed to the center

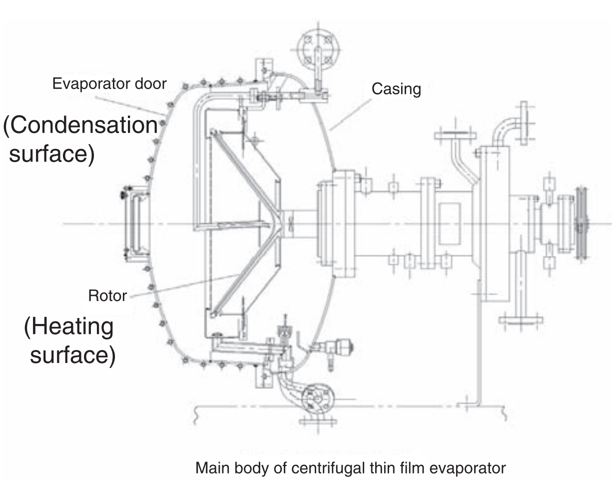


Figure 5　Schematic Diagram of Centrifugal Thin Film Evaporator

Figure 6　Automatic Operation Screen

Photo 2　Rotor

Photo 3　Overview of CEH-400B II

of the rotor, and forced to flow to the periphery of the evaporation surface in the shape of a thin film by a centrifugal force. The process substance evaporates in a short time when it flows over the rotor surface. The evaporated substance moves to the condensation surface (the evaporator door) positioned opposite the heating surface, and it is cooled and liquefied on the condensation surface. The structure of the heating surface and the condensation surface closely facing each other causes almost no pressure loss, being well suited to high vacuum distillation and molecular distillation. (Figure 5)

Any liquid that has not evaporated is collected by the rotor cover and returned to the receiver. (Photo 2)

① A small-sized plant unit designed for production of a wide variety of product in small quantities.

An all-in-one unit that contains all devices required for a centrifugal vacuum distillation process inside a cabinet with dimensions of 2100W × 1150L × 1900H. The unit has been made easier to carry in, with a total height reduced from 2350 mm to 1900 mm. In addi-

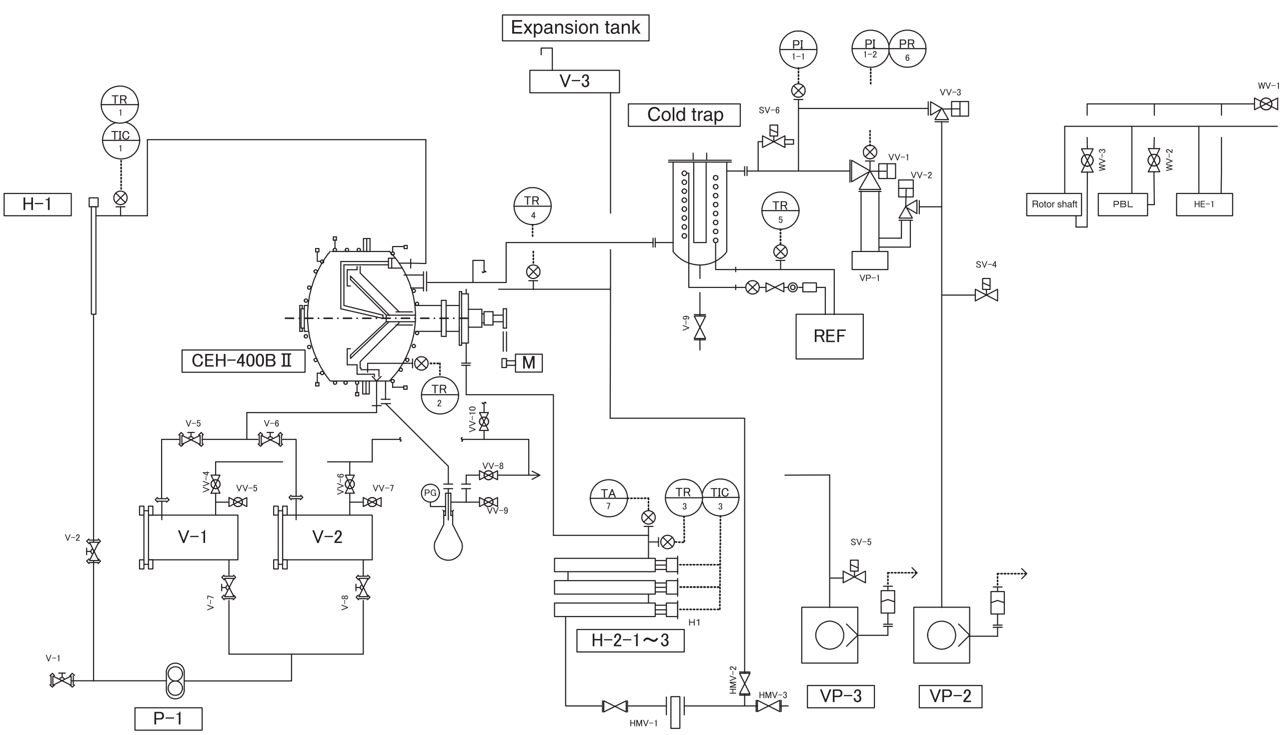


Figure 7　Systematic Diagram of Centrifugal Thin Film Evaporation System

tion, the evaporator with a reduced height facilitates internal observation during distillation, cleaning, maintenance, and inspection of shaft seal parts. (Photo 3)

Process wetted parts and pipes have structures easy to disassemble and wash at the time of change in the type of product. The touch panel and PLC control allow operators to set process conditions suitable for the type of product, and enable automatic batch distillation operations. (Figure 6)

② The unit enables continuous distillation operations with continuous feeding, and also supports large-volume lot operations with an optional large receiver.

③ The unit is equipped with a pressure regulating function as standard equipment and the world's only liquid-phase heating system to achieve a high-efficiency and high-accuracy distillation process.

④ The cabinet contains all required devices, enabling immediate start of production just by connecting the utilities, and also facilitating reconstruction and relocation of production lines.

⑤ In consideration of the environment, the unit uses R-404A, a refrigerant with an ozone depletion coefficient of zero, as the refrigerant in the freezer used for cold trap cooling.

4. Configuration

The cabinet contains an evaporator main body, a receiver, a liquid feed pump, an evacuation system, a heat medium heating system, a cold trap cooling system and a control system. The system can start operation just by feeding electric power, cooling water and compressed air (0.5 to 0.9 MPaG) after installation. (Figure 7)

5. Specifications for CEH-400B II

Materials of main parts	Evaporator main body	SUS304
	Rotor heating surface	SUS316L
	Receiver main body	SUS304
	Piping for liquid	SUS316
	Piping for vacuum (Cold trap to vacuum pump)	SGP
Specifications of main parts	Rotor diameter	ϕ 400 mm
	Heat transfer area	0.15 m^2
	Number of rotations of rotor	1000 rpm
	Capacity of heat medium heater	10 kW
	Weight of material supplied	～ 50 kg ／ Hr
	Condenser cooling method	Air cooling or water cooling
	Ultimate pressure (degree of vacuum) With vacuum regulating mechanism	0.5 Pa
	Heating temperature of heat medium Heat medium temperature control accuracy	Room temperature ～ max 260℃ ± 1.0 deg
	Cold trap cooling method	Freezer cooling or Refrigerant input
	Outer dimensions of system	Width 2100 mm Depth 1150 mm Height 1900 mm
	Weight of system	Approx. 1200 kg
Utilities	Cooling water Power supply Compressed air	32℃ Approx. 900 L/Hr 200 V × 50 Hz × 3 ϕ Approx. 25 kVA 0.5 ～ 0.9 MPaG

6. Conclusion

Most substances to be refined by vacuum distillation are heat sensitive. Thermal influences on the substances can be minimized by using thin film distillation under a pressure kept as low as possible.

Valve switching, temperature setting and other troublesome operations have been automated to create a user-friendly system that allows anybody to perform the operations of distillation processes.

We hope applications of this system will spread to laboratories, workplaces of low-volume production, and the fields of electronic parts, FPD and other new materials.