Concentration of Gengnian'an Extract with a Vapor–Liquid–Solid Evaporator

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A multiple-tube evaporator with a vapor–liquid–solid flow of external natural circulating was established to investigate the characteristics of heat-transfer enhancement and prevention of fouling in the concentration of Gengnian'an extract, which is a kind of Chinese patent medicine. The evaporator is composed of five stainless steel heating tubes. The cylindrical solid particles are made of physiologically inert material. Steam was used as the heating medium. Heat-transfer enhancement was obtained with the three-phase evaporator. For the concentration of an alcohol extract of Gengnian'an, the overall average heat-transfer coefficients and the evaporation intensity with three-phase flow are all about 1.6-fold higher than those of the vapor–liquid two-phase evaporator under similar operating conditions. For the evaporation of a water extract of Gengnian'an, the evaporation intensity for the three-phase evaporator is about 1.3-fold higher than that of the vapor–liquid two-phase evaporator. Prevention of on-line fouling was also observed. After running more than 300 h, fouling was not seen on the surface of the heating tubes of the three-phase evaporator, although it does form in the evaporator with vapor–liquid flow when the operation time exceeds about 100 h under the same conditions. The concentration process can be simplified from the traditional two-step concentration to one-step concentration for such high viscous or easily fouled solutions. The quality of thick liquor was very stable according to the content analysis of the active ingredients, thus meeting the requirement for product quality standards. *AIChE J,* 51: 759–765, 2005

Keywords: concentration, evaporator, heat-transfer enhancement, vapor–liquid–solid boiling flow, fouling prevention, Chinese patent medicine (TPM), Gengnian'an extract

Introduction

In the production of Chinese patent medicine (TPM), the effective ingredients in the crude drugs are initially extracted with solvents such as water and potable alcohol, after which the extract is often concentrated by means of an evaporation process.[1,2] However, scale deposits often form on the surface of the heating tubes in the conventional evaporator as the concentration increases, thus generating some problems such as reduction in the production capacity and fluctuations in the quality of the thick liquor. Evaporator cleaning is unavoidable after operating for some hours. For certain varieties of TPM with high viscosity or high concentration, the concentration process has to be stopped, even though the relative density of the concentrated liquid has not yet met the established quality standard of concentration technology.

Much excellent work, especially the work conducted by European scientists, previously showed that solid particles can prevent fouling formation in addition to enhancing the process of boiling heat transfer.[3-11] Thus, adding some solid particles composed of physiologically inert material into a conventional TPM concentrator to form a vapor–liquid–solid (V-L-S) flow may be an effective preventive measure against fouling formation.[12] A preliminary experiment on evaporation of

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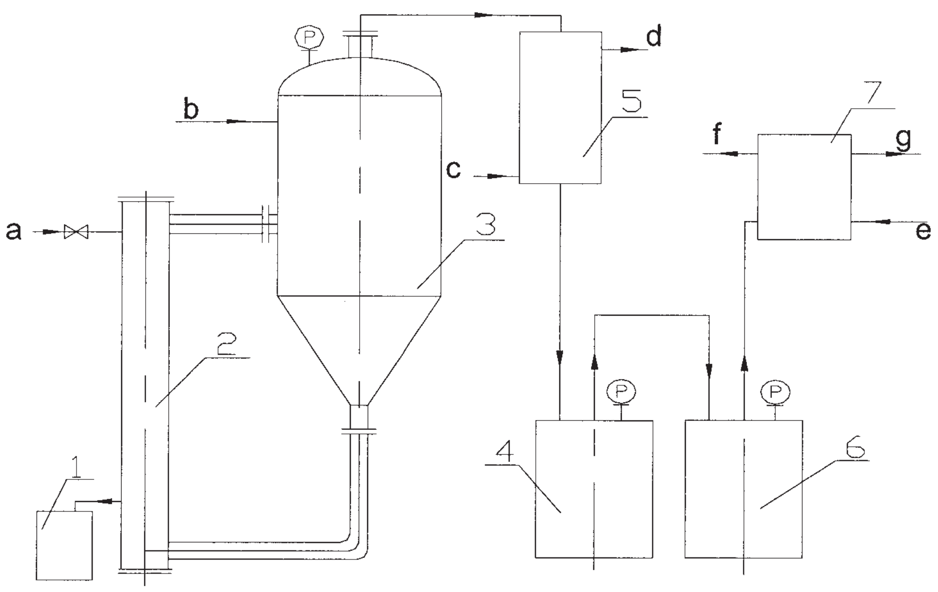


Figure 1. Experimental setup of multiple-tube evaporator for Gn extract concentration with a vapor–liquid–solid flow.

Legend: 1. condensate gauge bank of steam; 2. multiple-tube heat exchanger; 3. seperator; 4. condensate gauge bank of vapor; 5. condensator of vapor; 6. buffer tank; 7. vacuum pump. a, inlet of steam; b, inlet of Gn extract; c, inlet of cooling water; d, outlet of cooling water; e, inlet of cooling water of vacuum pump; f, outlet of cooling water of vacuum pump; g, vent.

Gengnian'an (Gn) extract in a single-tube evaporator with a V-L-S flow was carried out in a previous investigation,[2] results of which indicate that the Gn concentration heat-transfer process was enhanced and fouling was not observed in the evaporator with the addition of solid particles.

To accumulate the necessary data for industrial design and scale-up and to further optimize the operating conditions for the concentration of Gn extract, a multiple-tube evaporator with a V-L-S flow was established and the characteristics of heat-transfer enhancement and fouling preventing on-line were investigated and are reported herein.

Many structural styles or operation modes for a fluidized bed evaporator have been reported in the literature.[4,8,10] The multiple-tube evaporator developed in this work was an external naturally circulating mode. This operation mode is distinguished primarily by such features as low power consumption, high operation steadiness, and simple structure. A survey of the open literature indicates that no investigation has been reported with respect to Gn extract concentration by a multiple-tube external naturally circulating evaporator with a V-L-S flow, even though similar work on the evaporation of liquids of white water, black liquor, and wastewater has been done.[4,5]

Experimental

Installation flow chart and parameter measurement

The multiple-tube evaporating installation system was set up as shown in Figure 1.

The main section of the system is an external naturally circulating evaporator. In the evaporator, there are five heating tubes made of 316 stainless steel (0.033 m ID, 0.0025 m thickness, and 3 m in length).

The steam was supplied by the steam boiler. The variation ranges of the values of operation parameters during evaporating experiments are shown in Table 1.

The evaporator operated in a fluidization mode of external circulation with a V-L-S flow. The state of three-phase boiling flow was primarily in the nucleate boiling regime and the circulating velocity of liquor was about 0.6 m/s, according to visual observation, using the CCD measuring technique, carried out in a transparent heating glass tube under similar operating conditions.[2,13,14]

The mass flow rate and temperature of the steam condensate were measured using a gauge bank and thermometer, respectively. The pressure and liquid level in the separator were measured by use of a diaphragm manometer and a level indicator, respectively. The temperature of concentration liquor in the separator was obtained with a thermometer. The steam pressure was measured by a manometer. The vapor was condensed in a condenser, after which it was collected into a condensate gauge bank. In addition, the relative density, viscosity of the concentration liquor, and the volume fraction of the mixture of alcohol condensate were obtained by a relative density meter, a capillary viscometer, and a volume fraction meter of alcohol, respectively. Concentrations of solid substances in the liquor were also measured.

Table 1. Variation Ranges of the Values of Operation Parameters in the Experiments

Operation Parameter	Variation Range of Values
Absolute pressure of steam, kPa	150–250
Absolute pressure in separator or vapor space, kPa	20–120
Temperature of liquor in separator, °C	60–105
Temperature drop of heat transfer or LMTD, °C	20–60
Fluctuation range of liquid level in separator, m	0–0.2

Table 2. Typical Physical Properties of Liquid Phase

	Temperature (°C)	Relative Density (Dimensionless)	Concentration of Solid (wt %)	Viscosity (mPa · s)
Alcohol extract	20	0.94	11.39	3.15
	79	1.35	89.01	18.53
Water extract	20	1.03	4.43	1.69
	88	1.35	94.07	20.68

Operation parameter data in the separator were also obtained by an automatic measuring system developed specifically for this investigation.

Concentration fluid and material

In the experiment, alcohol and water extracts of Gn were taken as the liquids to be concentrated. The typical physical properties of the liquid phase are listed in Table 2.

Based on the physical properties of Gn extract, the technical requirements of three-phase flow, and the experimental results of the single-tube evaporator,[2,13,14] the optimized parameters of solid particles were chosen and determined.[2] The cylindrical solid particles, 0.003 m in both length and diameter, having a density of 2200 kg/m^3, are composed of physiologically inert material.

The mass of alcohol thin liquor of Gn is 1308 kg and the evaporating time is about 10 h for each batch. The mass of water extract of Gn to be concentrated is 10,800 kg and the concentration time is about 96 h per batch. Alcohol and water extracts of Gn were prepared by use of the multifunction extract apparatus. To obtain data with respect to the time of fouling prevention, the evaporator was not cleaned, even though it should have been cleaned between different batches according to the demands of Good Manufacturing Practice (GMP). The Gn extract was concentrated consecutively for three batches, and the total concentration time was about 300 h.

Method of data treatment

The overall heat-transfer coefficient U of the evaporator is calculated from the following equation

$$U = \frac{q}{A\Delta t_m} = \frac{q}{A(T_s - t_b)} \tag{1}$$

The temperature of the extract at boiling point t_b is calculated by adding the temperature of the extract measured in the separator and the boiling point elevation resulting from the effect of liquid head. The rate of heat transfer q is calculated by two different methods with negligible heat dilution. One method is based on the mass rate of steam condensate, as shown in the following equation

$$q = m_s\lambda_s - q_l \tag{2}$$

Equation 2 includes the latent heat of steam condensation and the heat loss to ambient atmosphere from the outside surface of the evaporator.

The second method is based on the mass rate of vapor condensate. In this method, q is expressed by

$$q = m_v\lambda_v + m_f C_{pf}(t_b - t_f) \tag{3}$$

In Eq. 3, the latent heat of vaporization from Gn extract at the boiling point and the sensible heat required to heat the liquor from inlet temperature to its boiling point are considered. The specific heat of extract C_{pf} is evaluated approximately by the specific heat of water C_p and λ_v is estimated by the addition principle for alcohol extract of Gn (see Eq. 5).

The two methods mentioned above are both sufficiently accurate for water extract. For the alcohol extract, the first method can still be used with satisfactory accuracy; however, the second method provides only an approximation for alcohol extract because in the calculation, it is assumed that only water and alcohol are in the mixture of vapor condensate. The addition principle is used to estimate its characteristic properties such as the density and latent heat of vapor condensate. In fact, there are a several ingredients in the vapor condensate mixture of alcohol extract.

According to the addition principle, mass friction x_a, instead of volume friction w_a, is needed in the calculation of the properties of vapor condensate of alcohol extract. Thus, the transfer formula is given as

$$x_a = \frac{w_a\rho_a}{w_a\rho_a + (1 - w_a)\rho} \tag{4}$$

The corresponding property parameters can be estimated from the addition principle. Here, the latent heat of vaporization from the alcohol extract is taken as an example, described by the following

$$\lambda_v = \lambda_a x_a + \lambda(1 - x_a) \tag{5}$$

The ratio of evaporator capacity to area of heat transfer surface is called evaporation intensity.

Results and Discussion

Overall heat-transfer coefficient U

Besides investigating the overall average value of U, which is an important parameter for industrial design and scale-up, the variations of U for water extract and alcohol extract with a running time t are also analyzed, as shown in Figures 2 and 3.

The curves of U plotted against operation time t for the water extract are shown in Figure 2. The vertical line shows the time point of charge and discharge of concentration liquid. In Figure 2, there is a vertical dot–dash line, which expresses the initial time of changing operation pressure in the separator. The time interval between the vertical dot–dash line and the first vertical thick line counted from the left side is the time length of changing the operation pressure in the separator. In this period of time, the substantial fluctuation and clear difference between the two calculation methods of U can be seen.

Figure 2 shows that the values of U obtained by the two methods are very close for most of the time, and the difference between the estimated values by the two methods is obvious only when the operation pressure was adjusted. The experimental goal for the first batch of water extract is to seek the optimum operating condition, especially to find a suitable op-

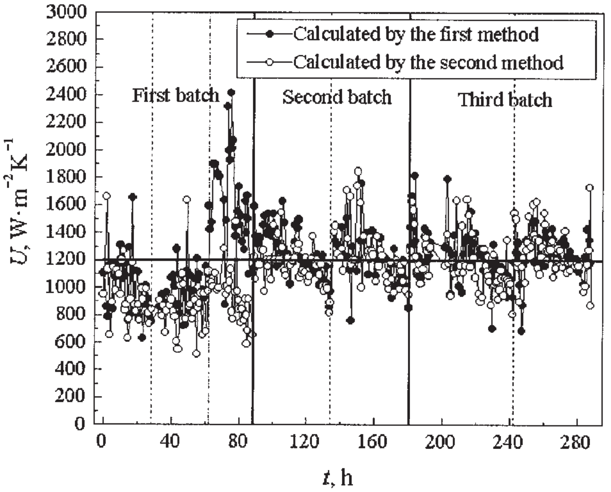


Figure 2. Overall heat-transfer coefficient vs. operating time for concentration of Gn extract of water.

eration pressure in vapor space of the evaporator. Thus, the fluctuation of U value is severe. With respect to the optimum particle parameters, such as the size, shape, material, and volume friction in units of solid particles, they were basically determined according to the single-tube evaporation experiment as mentioned. The primary cause of fluctuation of U in the whole process is considered to be the fluctuation of boiler steam pressure. The fluctuation range of the absolute pressure of steam is 150–250 kPa. The average, maximum, and minimum values of U are listed in Table 3. It can be seen that as the absolute pressure increases or the degree of vacuum decreases in the separator, U increases. The effect of operation pressure in the separator on the process of three-phase concentration will be analyzed in detail later.

From the vertical lines in Figure 2, it can be seen that when the relative density of concentrated liquid reaches the required value, the value of U is gradually reduced, attaining the lowest value at the time of discharge of thick liquor. This is easily understood because at this time both the relative density and the viscosity of concentration liquid are all higher, and U decreases, thus diminishing the evaporation capacity. However, unlike the traditional vapor–liquid evaporation process, where fouling occurs and the process has to be stopped, the operation of a three-phase evaporator or concentrator is still operating normally.

The variation patterns of U for the evaporation process of alcohol extract are illustrated in Figure 3. The curves obtained under optimum operating conditions are analogous to those of the water extract concentration process. Similar to the result of the water extract concentration process, the value of U for alcohol concentration in three batches also fluctuates around an average value. The reason for the fluctuation is considered to be similar to that of the water extract concentration process. The average, maximum, and minimum values of U for three batches are listed in Table 4.

It can be seen from Figure 3 that, unlike the values of U for the water extract concentration process, U values calculated by the two methods are much different. The main reason is related to the approximation of the second calculating method, which was discussed in the previous section. In this method, the assumptions of mixture components and the addition rule of physical properties may not be consistent with the actual situation.

The U value (about 1200 W m^{-2} K^{-1}) of the three-phase evaporator may be the same for the concentration of alcohol extract and water extract of Gn. For the objective of industrial application, the valve of U obtained by the second method is recommended for safety reasons.

Obviously, the measurement of physical parameters of TPM extract to establish the data bank is one of the necessary foundational directions for future research. It is worth noting that, to maintain the stable operation of an evaporation process, one of the important steps is to stabilize the steam pressure.

Influence of operation pressure in separator

The variation trends of U, mass flow rate of steam condensate m_s, and mass flow rate of vapor condensate m_v with the increase of absolute pressure in the separator for concentration of the water extract are shown in Figure 4.

Figure 4a indicates that U generally tends to decrease with the decrease of absolute pressure or the increase of vacuum degree in the separator. This is easily understood by analyzing Eq. 1 when considering the increase of heat-transfer temperature, which diminishes as a result of the decrease of the boiling point of the water extract of Gn arising from the increase of vacuum degree in the separator. Although more effective ingredients in the concentrated liquid will be protected and the quality of concentrated liquid will be retained as a result of the low temperature, when the absolute pressure is too low, viscosity of the solution will increase. Thus, the three-phase flow circulation may be impeded. Meanwhile, when the temperature decrease of heat transfer is too high, the V-L-S flow may change from that of a bubble nucleate boiling regime—characterized by stable operation and high heat-transfer coefficient—to that of a transition boiling regime or film boiling regime, which can easily promote an unstable operation and a decrease of U. Moreover, the energy consumption will increase because of the increase of vacuum degree. Therefore, the value of absolute pressure must be maintained at sufficiently high levels. However, the operation

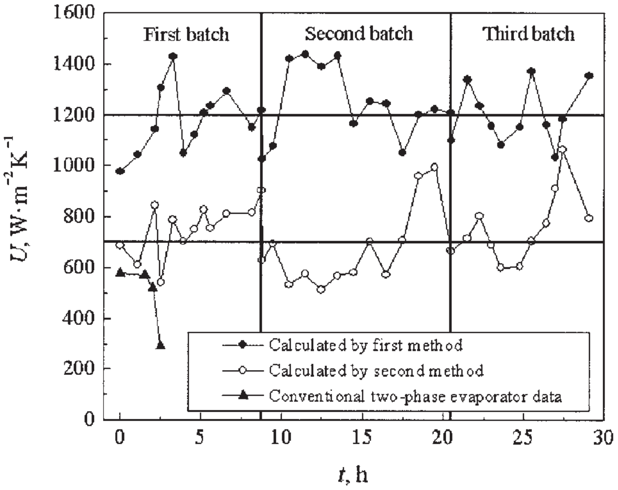


Figure 3. Overall heat-transfer coefficient vs. operating time for concentration of alcohol extract of Gn.

Table 3. Average Value, Maximum and Minimum of *U* for Concentration of Water Extract of Gn

Batch Number	Absolute Pressure in Separator (kPa)	U (W m^{-2} K^{-1}) Calculated by First Method Average	Max.	Min.	Calculated by Second Method Average	Max.	Min.
1	30	993	2411	626	905	1661	513
	70	1625	2411	874	956	1478	588
2	70	1256	1619	759	1204	1758	816
3	70	1234	1813	682	1214	1735	806

pressure cannot be kept too high; otherwise, a condition of positive pressure may be formed in the separator. Although U increases to some extent, the vapor cannot be immediately removed from the separator, which will influence the evaporation rate and the production capacity.

As shown in Figure 4b, with the increment of operation pressure, either m_s or m_h decreases after a platform variation. So, considering the various influence factors, an optimum value of operation pressure must be chosen in the three-phase evaporation operation of the concentration of TPM extract. In this work, the optimum range of absolute pressure is 40–70 kPa.

Variation trends of m_s and m_v along with time

The typical variation curves of m_s and m_v for water extract with time for a single batch are given in Figure 5. At an earlier stage of the concentration process, m_s and m_v fluctuate along with time in a certain range, but at a later stage, m_s and m_v obviously decrease and achieve a minimum as thick liquor is discharged, arising from the increases of relative density and viscosity of extract, as shown by the dotted line in Figure 5a. The result corresponds with the variation rule of U, as shown in Figure 2. At this moment, a decrease of capacity is observed. Compared against the curves in Figure 5a, the decreases of m_s and m_v with the increase of t for the alcohol extract concentration process are not clear.

Comparison of evaporation capacity

Although the V-L-S plant is different in structure to some extent from that of the vapor–liquid apparatus being used in a pharmaceutical factory and the direct comparison of the characteristic parameters between the two installations is not strict, the overall performances of the two units can basically be judged by a rough comparison and thus the comparison is carried out.

Generally, the three-phase evaporator is used in TPM manufacturing to achieve the concentration of water extraction of Gn. Now the data of evaporation of water extract of Gn measured on-site from a three-phase concentrator with vapor–liquid flow is compared with those obtained from the evaporator with a V-L-S flow. The operating conditions are very similar. The total area of heat-transfer surface for the two-phase equipment is about 42 m^2, and the heating area of each phase is about 14 m^2. The evaporation capacity of the installation is designated by the manufacturer as 0.55556 kg · s^{-1}. Thus, the evaporation intensity is 0.01322 kg m^{-2} s^{-1}for each unit of the evaporator. For the three-phase installation of a single unit, on the other hand, the evaporation intensity calculated from the experimental data is 0.01775 kg m^{-2} s^{-1}. Therefore, under similar operating conditions the evaporation intensity of the three-phase flow concentrator for water extract of Gn is 1.3-fold higher than that of the two-phase flow evaporator.

The field data measured from a two-phase evaporator of a single factory unit was used to compare the operation performance of the two units when the concentration liquid is alcohol extract of Gn under similar operating conditions. The heat-transfer area of the vapor–liquid flow concentrator is about 7 m^2. If the operation time for each batch is 4 h (in fact, it is much longer than 4 h), then the evaporation intensity is 0.01306 kg m^{-2} s^{-1}. Nevertheless, it is 0.02128 kg m^{-2} s^{-1} for the three-phase flow evaporator. Therefore, under similar operating conditions, the evaporation intensity of the three-phase evaporator is 1.6-fold higher than that of the factory unit.

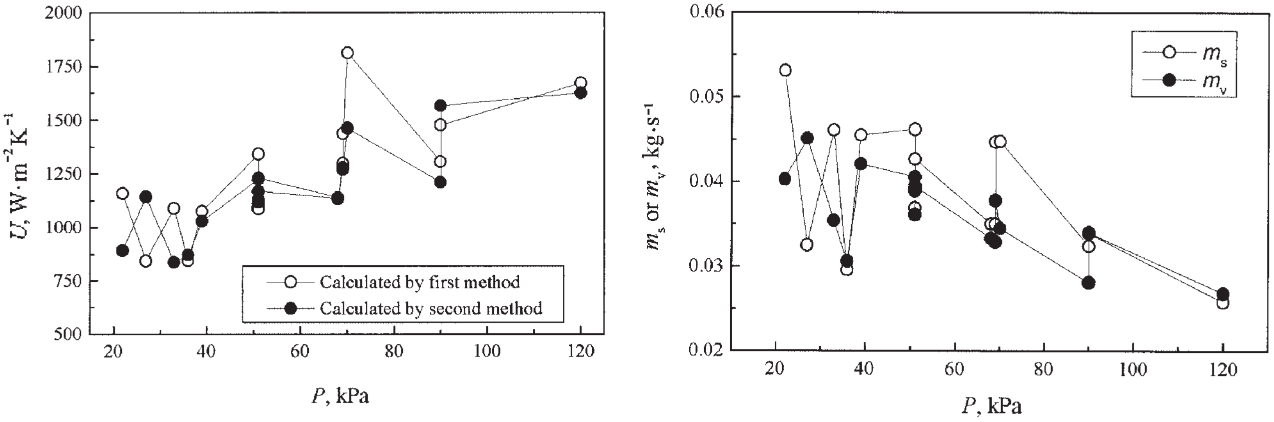
The factory data of U are also measured on site and calculated from a single-unit concentrator with conventional vapor–liquid two-phase flow for the concentration of alcohol extract. The value of U decreases from 576 to 291 W m^{-2} K^{-1} (calculated by the second method) with increasing operation time and the average value is 444 W m^{-2} K^{-1}, which is shown in Figure 3. However, the average value of U for the three-phase flow concentrator is 726 W m^{-2} K^{-1}. Consequently, under similar operating conditions, the average value of U for the three-phase flow concentrator is 1.6-fold higher than that of the factory concentrator with two-phase flow for concentration of alcohol extract.

Operation feature and fouling prevention state

Besides the increase in evaporation intensity or heat-transfer coefficient, the three-phase flow concentrator can still run when the density is much higher. The experiments carried out in the three-phase evaporator show that it can run steadily even though the relative density of concentrated liquid has reached

Table 4. Average Value, Maximum and Minimum of *U* for Concentration of Alcohol Extract of Gn

Batch Number	Absolute Pressure in Separator (kPa)	U (W m^{-2} K^{-1}) Calculated by First Method Average	Max.	Min.	Calculated by Second Method Average	Max.	Min.
1	70	1181	1426	978	752	902	539
2	70	1240	1438	1026	669	993	513
3	70	1197	1372	1033	757	1064	600



(a) **(b)**

Figure 4. Effect of absolute pressure in separator on the process of concentration of water extract of Gn.

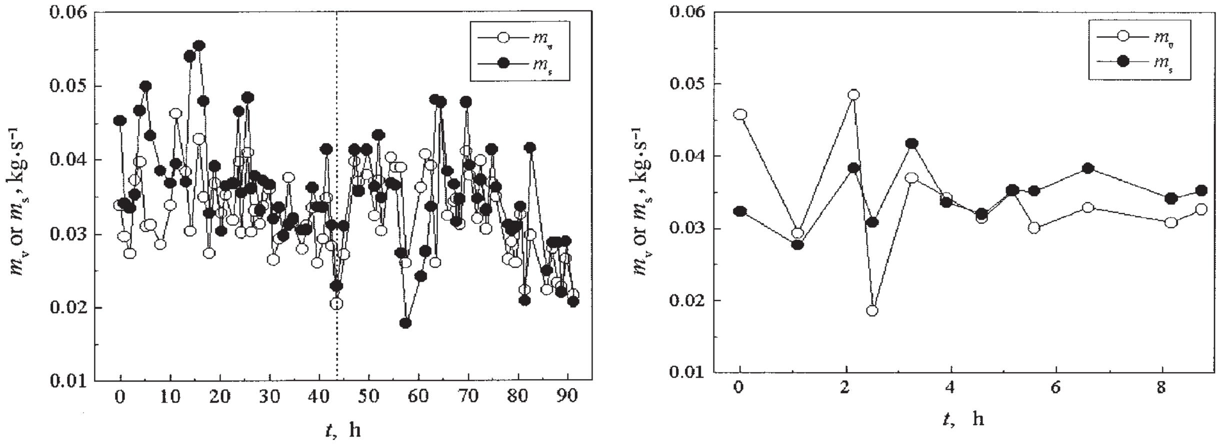
(a) Coefficients of heat transfer; (b) mass flow rates of condensate.

the technological standard of the Gn concentrating process (the value should exceed 1.3 at the temperature of 80°C). In vapor–liquid flow concentration, the process has to be stopped, even though the relative density of the concentrated liquid has not met the standard because of the occurrence of fouling. In actuality, the process has to be divided into two steps and, accordingly, two types of concentrators are used: vapor–liquid evaporator (for concentration of low-density extract) and spherical concentrator (for concentration of high-density extract). The reason that the spherical concentrator is often used as the unit for the second concentration step is that it is easily cleaned if fouling occurs during the concentration of the higher-density liquid. If a three-phase flow concentrator is applied, the concentration process of Gn can be simplified from a two-step concentration to a one-step concentration. That is to say, the application of the spherical concentrator is unnecessary.

The experiments of the three-phase flow evaporation verified that, after running stably about 300 h, the operation was still normal and fouling did not appear on the heating tube walls in the concentrator (the unit was opened after the experiment, and no fouling could be seen on the wall). However, fouling occurred on the wall of the tube in the conventional two-phase unit at about 100 h.

The apparatus features of on-line fouling prevention, steady operation, heat-transfer enhancement, and decrease in the wall temperature are all favorable for the quality of concentrated products of TPM. Analysis of the composition content of frangula emodin (6-methyl-1,3,8-trihydroxy-anthraquinone) in the thick liquor of Gn by high-performance liquid chromatography shows that the content of the thick liquor by the three-phase unit is stable and achieved the required standard of Gn product quality.

With respect to the erosion problems of solid particles and the heating tube, the experimental results indicate that erosion of the heating tube is negligible. The attrition of solid particles, although inevitable, is not noticeable. The reason is that solid particles in the heating tube, which move in a vapor–liquid flow with a low velocity, consist of a kind of antiabrasive plastic material. How-



(a) **(b)**

Figure 5. Mass flow rates of condensates of steam and vapor vs. operation time.

(a) Water extract of Gn; (b) alcohol extract of Gn.

ever, measures to further reduce the degree of erosion of solid particles is presently under investigation.

Conclusions

The investigation results of the multiple-tube evaporator with a V-L-S flow for the concentration of Gn extract generated the following conclusions:

(1) The multiple-tube evaporator with a V-L-S flow can enhance the heat-transfer process because the heat-transfer coefficient and the evaporation intensity in this system are all about 1.6-fold higher than those of the vapor–liquid flow evaporation system under similar operating conditions for the concentration of the alcohol extract of Gn. With respect to the concentration of the water extract of Gn, the evaporation intensity in the V-L-S flow system is about 1.3-fold higher than that of the vapor–liquid flow evaporation system under similar operating conditions.

(2) Prevention of on-line fouling is also observed because, after an operating run of more than 300 h, scale is not observed on the surface of heating tubes; conversely, fouling will be formed in the vapor–liquid evaporator when the operation time exceeds about 100 h under high relative density and viscosity.

(3) The three-phase evaporator is still in a normal operation state, even though the relative density and viscosity are very high. Thus, use of the spherical concentrator to further increase the relative density of the succeeding concentration becomes unnecessary.

These conclusions, including the experimental data and the optimum operating conditions obtained from the three-phase evaporator, may be used as the basis for design, operation, and control of an industrial three-phase flow concentration unit for TPM extracts and other similar liquids.

Acknowledgments

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Notation

A = area of heat-transfer surface, m^2
C_{pf} = specific heat of extract at constant pressure, J kg^{-1} K^{-1}
C_p = specific heat of water at constant pressure, J kg^{-1} K^{-1}
m_f = mass flow rate of feed of extract, kg · s^{-1}
m_s = mass flow rate of steam condensate, kg · s^{-1}
m_v = mass flow rate of vapor condensate, kg · s^{-1}
p = pressure of steam, kPa
q = rate of heat transfer, W
q_l = rate of heat loss, W
T_s = temperature at saturation pressure of steam, K
t = time, s
t_f = inlet temperature of feed of extract, K
t_b = temperature of the extract at boiling point, K
U = overall heat-transfer coefficient of multiple-tube evaporator, W m^{-2} K^{-1}
w_a = volume fraction of alcohol in vapor condensate at the condensing temperature of vapor, dimensionless
x_a = mass fraction of alcohol in vapor condensate, dimensionless

Greek letters

ε_p = friction of solid particle, dimensionless
λ = latent heat of vaporization from water at boiling point, J · kg^{-1}
λ_a = latent heat of vaporization from alcohol at boiling point, J · kg^{-1}
λ_v = latent heat of vaporization from Gn extract at boiling point, J · kg^{-1}
λ_s = latent heat of condensation of steam, J · kg^{-1}
μ = absolute viscosity, Pa·s
ρ = density of water at the condensing temperature of vapor, kg · m^{-3}
ρ_a = density of alcohol at the condensing temperature of vapor, kg · m^{-3}
ρ_r = relative density, dimensionless
Δt_m = average temperature drop of heat transfer, K

Subscripts

a = alcohol
b = boiling point
f = feed of extract
l = heat loss
m = logarithm average value
p = pressure
s = steam
v = vapor

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