Performance Evaluation of Ultrasonic Atomization Process for Ethanol Separation and Recovery

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Ultrasonic atomization, a process of generating fine droplets through irradiation of high-frequency ultrasound to a gas–liquid interface from the liquid underneath, is applied to separating ethanol from its aqueous solution. Towards its practical use, the process of **collecting in two cooling stages the ethanol-enriched mist**—generated via an ultrasonic atomizer (ultrasonic transducer operated at 2.4 MHz) with continuous feed of ethanol–water solution—**using two cooling units in a series** has been developed. The effects of operating conditions, especially cooling temperatures and gas flowrate, on ethanol-enrichment and condensation characteristics are examined. It is found that the highly-enriched ethanol recovery could be attained in the 2nd stage by optimizing the 1st- and 2nd-stage cooling temperatures (as moderate as 5°C and up to –10°C, respectively). Regarding the carrier-gas flowrate, ethanol-rich mist consisting of small-size droplets tends to be carried selectively in favor of lower gas flowrate. Nevertheless, the desired recovery of enriched ethanol—i.e., a highest possible value of the recovered quantity of ethanol as well as the recovery concentration itself—is expected to be obtained in the 2nd stage by raising the carrier-gas flowrate under the present operating conditions. While the proposed two-stage cooling process tends to collect rather an appreciable quantity of less-enriched ethanol solution in the 1st stage, it is the 2nd stage that assures the desired quality of enriched-ethanol recovery.

1. Introduction

Ultrasonic atomization (USA) can be induced by applying ultrasound of *high frequency*, i.e., on the order of MHz, to a gas–liquid interface from the liquid **beneath**. It will preferentially occur, due partly to its *high directivity*, in comparison to acoustic cavitation induced favorably under lower-frequency (< MHz, thus technically-easier-to-attain larger amplitude) conditions. When liquid is subjected to high-frequency (≥ 1 MHz) ultrasound, the atomization starts almost instantaneously in association with **liquid fountain (or jet)** formation and breakup as demonstrated in Fig. 1 (Tsuchiya et al., 2007, 2011). It has been claimed in the literature that almost pure ethanol could be obtained directly from ethanol–water solution at 10°C through USA; even at a normal temperature (30°C), appreciable concentration/enrichment of ethanol can be realized (Sato et al., 2001).

Fig. 1 Ultrasonic atomization in association with **liquid fountain** formation and breakup.

Several mechanisms of USA have been proposed, including the cavitation effects as well as the capillary instability induced at the surface of the **liquid fountain**. Sato et al. (2001) claimed that the parametric decay instability of capillary waves, propagating along the **liquid fountain** surface, leads in turn to USA. Kirpalani and Toll (2002), Kirpalani and McQuinn (2006) and Suzuki et al. (2006), on the other hand, suggested that the presence of cavitating microbubbles in the **liquid fountain**, which grow and collapse in regions of acoustic energy accumulated and mist released, should be important. In either case, USA mechanism needs to be further elucidated, especially in regard to the mechanism of ethanol enrichment.

While the mist consisting of USA-generated droplets has been generally recognized to have relatively narrow size distribution, a recent finding by Yano et al. (2007) indicated that USA of an ethanol–water solution could produce a combination of water-rich droplets of micrometer order and ethanol-rich droplets of diameter as small as 1 nm. Droplet-size dependence of ethanol enrichment could be partly explained in terms of surface excess being realized along ethanol-rich monolayer at the surface (Yano, 2005) and/or microscopic phase separation in the bulk of the solution (Wakisaka and Matsuura, 2006). Though the enrichment process is extremely difficult if not impossible to be elucidated or “visualized,” some theories of combination for mist generation and selective ethanol separation have been proposed.

For instance, Sato et al. (2001), as mentioned above, explain that it is the coincidence in location that surface

excess and capillary-wave instability take place simultaneously. Kirpalani and Toll's (2002) conjunction theory signifies the role of cavitating microbubbles—while moving upwards within the **capillary fountain**—in carrying the surface-excess layer surrounding them. Yasuda et al. (2005) modeled an ethanol-enriched droplet originating from the surface-excess layer as a “shell-and-core” sphere in which the former and latter portions, respectively, contain solute and the remaining solution under phase separation. None, however, is conclusive yet at present.

To put USA into practical use, the process of collecting the ethanol-enriched mist in high efficiency should be developed. Typical collection methods involve thermodynamic—condensation via cooling and pressurization—as well as electrostatic processes. The latter process, or electrostatic precipitation/precipitator (ESP), is effective for collecting droplets of diameters ranging 0.1–3 μm (cf. PM2.5) provided the droplets are sufficiently charged—through the collision with corona-ionized gas molecules (Bologa et al., 2004; Tepper and Kessick, 2008). In a theoretical attempt to determine such “inherent limitations,” Tapuchi et al. (2010) proposed an analytical criterion for the “minimum diameter for droplets to be charged”; for water droplets in air at ambient temperature and pressure, it can be estimated from $d_{\min} \cong 140/\sqrt{E}$ with $d_{\min}$ in $[\mu\text{m}]$ and E , the electric field intensity, in $[\text{V/m}]$.

While practical ESPs apply the operating range of E as high as 6 MV/m (Tapuchi et al., 2010), which can lead to $d_{\min}$ as small as 30 nm, lab-scale ESPs usually employ $E \approx \{(5-10)[\text{kV}]\}/\{(20-50)[\text{mm}]\}$, which would result in $d_{\min} \approx (0.20-0.45)[\mu\text{m}]$, roughly corresponding to the primary particle size of cigarette smoke. Tsuchiya et al. (2003), in their preliminary experiments examining the capability of ESP to collect USA-generated mist, found that 50% of the (water) mist was collected for E values ranging $(0-4)[\text{kV}]/(50[\text{mm}])$, without corona discharge observed; once the electrostatic voltage exceeded (a transition range of) 4–6 kV—at which corona discharge was assumed to be triggered—the recovery efficiency jumped to as high as 80% and was maintained at that level for voltages up to 15 kV. In this regard, Matsuura et al. (2003), using a bench-scale USA–ESP-recovering prototype system, **pointed out** the necessity of **maintaining** the size of droplets in the mist—above $d_{\min}$ —so as to **realize** a large fraction of **the** mist chargeable—thus recoverable; in practice, ESP should then be performed **under cooling** to enhance the growth or even re-condensation of the mist droplets.

In utilizing the thermodynamic principle/process, Nii et al. (2010) explored the effectiveness of pressurization for mist recovery. They recommend the stepwise operation with proper pressure control, in aid of specific findings that production of fine ethanol-rich droplets was facilitated at reduced pressures while the recovery was enhanced at elevated pressures; both the recovery efficiency and ethanol concentration of collected mist were indeed increased. It is presumed, however, that the pressure-operating scheme as well as the energy associated

needs to be further explored.

Suzuki et al. (2006) investigated the characteristics of mist recovery through condensation following USA. Although the product (ethanol) concentration exceeded that under vapor–liquid equilibrium (VLE) for feed concentrations greater than 40 mol%, the ethanol concentration of collected mist by cooling was lower than that reported by Sato et al. (2001), who estimated the ethanol concentration of mist based on material balance. The reliability of material balance had been confirmed by Matsuura (1997), utilizing activated carbon for almost complete adsorption (followed by desorption) of mist. For continuous processing, Matsuura et al. (2007) pursued an efficient mist recovery and dehydration by combining condensation and pressure swing adsorption (PSA) with molecular sieves (rather than activated carbon). Having such a combination towards dehydrated ethanol production, they were successful in obtaining the final recovery as almost pure ethanol. **It is desired at an initial stage of the process development, however, to make it rather simple by not employing additional mechanism** such as adsorption; simple—yet still promising—is condensation via cooling, which is worth examining as a basic means to attain the purpose.

In this study, the process of effectively collecting *in two stages* the ethanol-enriched mist—generated via a USA unit with continuous feed of ethanol–water solution—*by cooling* has been developed. The two-stage cooling process is expected and partly proved to have an advantage of assuring the desired quality of enriched-ethanol recovery in the second stage, though with a smaller quantity (Tanaka et al., 2008). To optimize the operating conditions, the effects of the temperature(s) of the cooling unit(s) and the gas flowrate for carrying the mist on the characteristics of ethanol enrichment and condensation are further investigated.

2. Experimental

Figure 2 shows a schematic diagram of the experimental system for USA followed by mist recovery. The

Fig. 2 Schematic diagram of the experimental system for ultrasonic atomization and recovery.

system consists of a cylindrical USA unit (with an acrylic draft tube of 50-mm ID and 0.34-m height, placed coaxi-

ally within the outer column of 120-mm ID and 0.28-m height), a separable flask, two cooling units (Dimroth condensers of 35-mm OD and 0.30-m length) and two cooling devices. The 50-mm ID (or lateral distance square) is judged by visual observations to assure minimum interference of the tube wall on the liquid-fountain dynamics. Cooling units 1 and 2 are tilted by 50° and 35° from the vertical direction, respectively—both essentially 45°—for smooth connection to the recovery flasks as well as the connecting lines. An ultrasonic transducer (Honda Electronics HM-2412) is set on the bottom of the outer column; the transducer element (oscillating disk) itself is of 20.0 mm in diameter and 15 mm above the bottom. 1 L of ethanol–water solution, with 39 wt% (20 mol%) ethanol concentration, is circulated between the atomizer and the separable flask, which is placed in a 25°C constant-temperature bath. The liquid level is kept constant 45 mm above the element.

When the liquid is subjected to the ultrasound (2.4 MHz; 16-W input power), USA starts immediately after liquid fountain formation and breakup. N₂ is used as the carrier gas, introduced at a flowrate of either 3 or 6 L/min, to transport the generated mist into the cooling units. The carrier-gas flowrate, Q , employed in this study is selected to be in a minimum range for the mist transportation. In the first and second cooling units, mist and/or vapor are to be recovered. The gas temperature is monitored with four thermocouples at the inlet and outlet of both the cooling units. The temperatures of Cooling device 1 (Yamato Neo-cool Circulator CF700), T_{C1} , and Cooling device 2 (EYELA Low-Temperature Bath NCB-2400), T_{C2} , are both changed. First, T_{C1} is changed from 25 to −10°C at a 5°C interval, while T_{C2} is maintained sufficiently low at −20°C. Once T_{C1} is optimized, T_{C2} is set at −20, −15, −10 or 0°C for further optimization (laying emphasis on energy saving). The duration of atomization for each run is 90 min—long enough to assure the measurement reliability (especially that based on mass balance), yet short enough to maintain the feed/initial concentration; during the duration, C_0 varies by 4 wt%, that is, within 37 ± 2 wt% at most. The liquid-phase ethanol concentration is measured with a gas chromatograph (Shimadzu GC-12A).

3. Results and Discussion

3.1 Effects of First Cooling Temperature and Carrier-Gas Flowrate on Mist Recovery

Figure 3 shows, for a pair of given gas flowrates, the effects of the 1st-stage cooling temperature T_{C1} on (a) the ethanol concentration, C_R , and (b) the solution weight, W_R , recovered in each stage. As expected, both C_R and W_R in the 1st stage tend to increase and then level off with decreasing T_{C1} ; it is noted, however, that the 1st-recovery concentration (C_{R1}) under the given experimental conditions never reaches the corresponding concentration under vapor–liquid equilibrium for ethanol–water solution at 101 kPa. The 2nd-recovery concentration (C_{R2}), on the other hand, is almost always above the equilibrium value (provided $T_{C1} \leq 15^\circ\text{C}$) and greatly exceeds C_{R1} . In addition, with decreasing T_{C1} , C_{R2} levels off—keeping relatively

high ethanol enrichment [Fig. 3(a)], while W_{R2} tends to decrease [Fig. 3(b)]—resulting in a less extent of recovery in the 2nd stage than in the 1st stage ($W_{R2} < W_{R1}$) as C_{R2} reaches the highest value. It is to be noted, however, if the two stages were combined to be a longer single condenser (cooling unit), the expected results would be different from the two-stage cooling process: the recovered solution would be the same as the mixture of the 1st and 2nd recoveries; thus, the solution weight would be $W_{R1} + W_{R2}$, but the ethanol concentration would not reach the desired level (indeed found to be about the same as VLE level).

The effective coolant temperature was measured to be 7–8°C for the cooling-device set-point temperature of 5°C ($= T_{C1}$), which resulted in the gas-phase (vapor) temperature of ca. 17–18°C. The sustained attainment of constant value of C_{R2} at a maximum level for T_{C1} up to 5°C is followed by a gradual decline with temperature. This shift in trend, however, cannot be explained at the moment, at least from a viewpoint of equilibrium argument alone.

Fig. 3 Effects of first-stage cooling temperature on (a) ethanol concentration and (b) solution weight recovered.

Regarding the effect of carrier-gas flowrate Q , the following general trends, though some preliminary, prevail: At a gas flowrate of 3 L/min, C_R (in either the 1st or the 2nd) signifies higher enrichment, while W_R is as low as half the value at 6 L/min. This trend could concur with the general finding in the literature that ethanol-rich mist consists of small-size droplets (Yano et al., 2007), which tend to be carried selectively (in comparison to larger droplets) in favor of lower gas flowrate. As discussed above, both C_{R1} and W_{R1} tend to decrease with increasing T_{C1} . Even if the difference between room temperature and cooling temperature becomes negligible, which means that vapor condensation hardly occurs, C_{R1} still keeps decreasing even

below the initial concentration at the higher gas flowrate. Such extremity (i.e., $C_{R1} < C_0$) could be evidence of having the USA-generated mist consisting of droplets which span a broad range of ethanol concentration—from higher values attained with smaller droplets to values as low as the initial concentration of the feed solution or even less. This latter situation could result from the carriage, into the 1st stage, of very large droplets attainable via higher gas flowrate. Note that at higher (say, $> 20^\circ\text{C}$) temperatures, only larger droplets could settle in the 1st recovery.

Since the enrichment—particularly in the 2nd stage—should be less sensitive to Q , it appears to be favorable in the present two-stage cooling recovery process—to have a higher gas flowrate. It is probably most important to note that the ethanol concentration of the 2nd recovery takes the value as well as the trend almost identical at both 3- and 6-L/min gas flowrates. The desired recovery of enriched ethanol—i.e., highest value of $W_R C_R$ as well as C_R —is expected, as shown in Fig. 4 [as well as inferred from Fig. 3(a)], to be attained in the 2nd stage with an optimum T_{C1} at as moderate as 5 or even 10°C under the present operating conditions. In determining an optimum gas flowrate, the net weight of ethanol recovered, i.e., $W_R C_R$ or that per unit volume of carrier gas $W_R C_R / Q$, in the 2nd stage can be one of the key process parameters. A higher value of $(W_R C_R / Q)_2$ at 6 L/min (provided $T_{C1} \leq 15^\circ\text{C}$) than that at 3 L/min should signify an effective use of the carrier gas (Fig. 4).

Fig. 4 Effects of first-stage cooling temperature on ethanol weight recovered per unit volume of carrier gas.

Fig. 5 Effects of first-stage cooling temperature on recovery efficiency.

To further confirm reasonable attainment in the recovery efficiency of the mist generated, the total recovery efficiency (or rate/ratio of the recovered to fed solution) defined as:

$$\text{Total recovery efficiency} = \frac{W_{R1} + W_{R2}}{W_{F0} - W_F} \quad (1)$$

averaged over the total duration of atomization, as well as the 1st- and 2nd-recovery efficiencies is provided in Fig. 5. While as much as 70–80% of the total mist has been recovered over the whole range of the 1st cooling temperature, at least a total of 70%—out of which as high as 40% of ethanol solution enriched to desired quality (i.e., 2nd-stage quality)—can be attained in an optimum 1st-stage temperature range (i.e., $5\text{--}10^\circ\text{C}$).

3.2 Link between Mist Recovery and Gas-Phase Temperature

Since the extent of ethanol recovery—by cooling—out of the mist generated through USA depends on the carrier-gas flowrate as well as the cooling temperature(s), as just discussed, the performance/efficiency of the cooling system, or its heat-transfer characteristics, will be affected by the temperature and flowrate of both the refrigerant and gas phases. The mist in this study is carried by dry N_2 gas out of the atomizer into the recovering process. To evaluate the cooling efficiency of the present recovering process with *partial insulation*, the gas-phase temperature—measured at four points (i.e., the inlets and outlets of Cooling units 1 and 2, T1–T4, shown in Fig. 2) along the carriage path—is plotted in Fig. 6 against the space time along the recovering path. The space time is defined as the cumulative value of effective volume of each unit/section along the cooling path divided by Q at room temperature. While not all the details are explainable in this plot, some characteristics are worth mentioning.

Fig. 6 Change in temperature of gas phase along cooling path.

The temperature rise from T2 (outlet of Cooling unit 1) to T3 (inlet of Cooling unit 2) occurs due to partial insulation—“thorough insulation” was extremely difficult to set up in the present study. Table 1 compares the difference between partial insulation and “extensive” insulation. The former insulation signifies the distinctive two-stage cooling; the latter can avoid the temperature rise from the 2nd to the 3rd point, essentially equivalent to a single-

stage configuration (twice-longer condenser). Despite this measure, there are no significant differences in ethanol concentration and weight of both the 1st and 2nd recoveries, indicating that the partial insulation process has rather an advantage of saving energy for insulation.

Table 1 Effect of extent of insulation on recovery

Insulation	Partial		Extensive	
	R1	R2	R1	R2
EtOH concentration (wt%)	59.3	83.7	60.6	84.0
EtOH weight (10^{-3} kg)	10.6	10.8	10.4	9.7

The influence of flowrate, or space (more properly, residence) time, of the gas phase with *extra* heat content can be realized in controlling the temperature drop in each cooling unit. As inferred above, the shorter residence time (at 6 L/min) and the optimum T_{C1} ($= 5$ – 10°C)—having resulted in a moderate temperature drop in the 1st-stage cooling and relatively sharp temperature drop in the 2nd-stage cooling—work favorably in obtaining the desired quality of enriched-ethanol recovery. Rough estimates of the Reynolds number based on an effective cross-sectional (annular) area-equivalent dimension (i.e., hydraulic diameter) and the gas properties at room temperature are 650 and 1300 (both laminar), respectively, for $Q = 3$ and 6 L/min. While not turbulent, the latter flow situation in the presence of internal helical coil may have resulted in enhancement of heat transfer due to some turbulent contribution—in addition to less exposure time to the ambient at higher Q .

3.3 Effect of Second Cooling Temperature on Mist Recovery

In a series of strategic improvements—in obtaining the desired product in the 2nd stage with the gas residence time and the 1st-stage cooling temperature T_{C1} optimized, as has been done above—the 2nd-stage cooling temperature T_{C2} needs to be optimized as well. Figure 7 shows the effects of T_{C2} on (a) the ethanol concentration and (b) the solution weight recovered in each stage at 6 L/min for an optimum T_{C1} ($= 5^{\circ}\text{C}$). The 2nd-recovery concentration C_{R2} tends to maintain a constant value for $T_{C2} \leq -10^{\circ}\text{C}$ [Fig. 7(a)]; T_{C2} is much less influential on recovery than T_{C1} , while W_{R2} tends to decrease with increasing T_{C2} [Fig. 7(b)]. As expected, on the other hand, both C_R and W_R in the 1st stage are not affected by T_{C2} . From these trends, “the desired recovery of enriched ethanol can be attained in the 2nd stage at an optimum T_{C2} going up to -10°C , with T_{C1} optimized at 5°C , and the higher gas flowrate (6 L/min) under the present operating conditions.”

3.4 Characteristics of Two-Stage Cooling Recovery

Combining the trends, presented so far, in C_{R1} and/or C_{R2} variations with T_{C1} (Fig. 3) and T_{C2} (Fig. 7), one may postulate the following change in state or “history” of the mist (originally in a state of bulk liquid) accompanying the carrier gas, as it passes through the 1st and 2nd cooling stages.

Fig. 7 Effects of second-stage cooling temperature on (a) ethanol concentration and (b) solution weight recovered.

Mist which consists of droplets with a broad range of ethanol concentration may not be collected all at once in the 1st stage, even if T_{C1} be reduced to as low as -20°C . This is because, first of all, the dry carrier gas will provide driving forces for the droplets to evaporate; droplets containing multi-components (or more specifically, binary components such as in the present ethanol–water system) would preferentially “release” more volatile component(s)—in the present case, ethanol—into the (initially) dry carrier gas while they are transported. Prior to entering the 1st stage, the carrier gas with room temperature inevitably induces this *preferential ethanol evaporation*.

Secondly, noting the general finding in the literature that ethanol-rich mist consists of small-size droplets (Yasuda et al., 2005; Yano et al., 2007), one can easily presume that such preferential evaporation should occur more extensively for smaller (i.e., higher-specific-surface-area) droplets. Since the 1st stage cannot attain immediate cooling, thus condensation, especially in the entrance region, *smaller (thus, richer-in-ethanol) droplets are encouraged to evaporate or shrink to a greater extent* than larger droplets with less ethanol content. If the corresponding vapor–liquid equilibrium be achieved in the 1st stage—more specifically, the carrier-gas phase being saturated with water vapor and ethanol vapor—and if the larger droplets be recovered there, C_{R1} will hardly reach the equilibrium value. As demonstrated in Fig. 3, this trend would prevail at $T_{C1} \leq -10^{\circ}\text{C}$ or even down to -20°C .

Thirdly, the remaining mist comprising the smaller droplets that have not been trapped in the 1st stage could be recovered, along with vapor-phase ethanol (and water),

altogether in the 2nd stage if it can indeed attain effective cooling. Since the latter, less volatile component (i.e., water) may have condensed to a greater extent in the 1st stage, the remaining—i.e., highly-enriched ethanol—recovery would be expected in the 2nd stage.

Finally, the behavior of gas-borne droplets, with their sizes ranging below submicrometers—or of even 1-nm order—is in general greatly influenced by diffusional effects. In the present flow situation, however, the convective contribution of the carrier gas flowing in the condensers assumes the dominance; diffusion of smaller (i.e., larger-diffusivity) droplets is thus presumed to be not enhanced via both Fickian and thermophoretic mechanisms, the latter due to the temperature gradient near the cooling surface, which may not be sufficient to induce the thermal effects (Walsh et al., 2006).

Two separate recoveries, in the 1st and 2nd stages, with lower and higher ethanol contents, respectively, can be realized by optimizing—from an energy-saving perspective—cooling temperatures and carrier-gas flowrate. The two-stage cooling process has thus an advantage of obtaining desired product by virtue of *collectivity* and *transportability*, respectively.

4. Concluding Remarks

The proposed two-stage cooling process for recovering the ultrasonic-atomized mist has a *promise* of assuring the desired quality of enriched-ethanol recovery in the 2nd stage by optimizing the cooling as well as gas-flow conditions. By setting the 1st cooling temperature below 15°C, the ethanol concentration in the 2nd recovery is always maintained above the corresponding vapor–liquid equilibrium value and greatly exceeds the 1st-recovery concentration which never reaches the equilibrium value. While the fraction of ethanol-rich mist, which may consist primarily of small-size droplets, would remain to be higher for lower gas flowrate, the desired recovery of enriched ethanol—i.e., highest value of the net weight of ethanol recovered in addition to the recovery concentration—in the 2nd stage is realized at the higher gas flowrate under the present operating conditions. Such optimum recovery can be achieved with the 1st cooling temperature at as moderate as 5 or even 10°C and with the 2nd cooling temperature up to –10°C.

The performance of the two-stage recovery thus depends strongly on the cooling temperatures and carrier-gas flowrate, or the mist collectivity and transportability, respectively. For its practical use, however, further investigation and a series of strategic improvements should be attempted, including recycling the effluent (low-quality product) from the 1st stage.

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Nomenclature

C_R	=	ethanol concentration of recovery	[wt%]
C_0	=	ethanol concentration of initial/original solution	[wt%]
$d_{\min}$	=	minimum diameter for droplets to be charged	[m]
E	=	electric field intensity	[V/m]
Q	=	gas flowrate	[L/min]
T_C	=	cooling temperature	[°C]
T_0	=	temperature of feed liquid	[°C]
W_F	=	solution weight of feed	[kg]
W_R	=	solution weight of recovery	[kg]

<Subscript>

0	=	initial
1	=	1st stage
2	=	2nd stage

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