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Ohmic-assisted hydrodistillation: A novel method for ethanol distillation

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ABSTRACT

Distillation is one of the most time and energy consuming steps in bioethanol production and some food operations. Ohmic-assisted hydrodistillation (OAHD) is a new proposed extraction method which uses the advantages of ohmic heating and has been used for essential oil separation. In this study, an OAHD device was designed and developed for ethanol distillation using titanium electrodes. Results of the OAHD process were compared to those of traditional hydrodistillation (HD) at constant wattage of 168 W for 3 L of 10% (v/v) ethanol solution containing 0.5% (w/v) sodium chloride as the electrolyte. Results indicated that the required energy for separation of ethanol in OAHD was 33% less than HD. In addition, OAHD completed the distillation process in 75.2 ± 2.2 min while HD required 108.4 ± 5.3 min. The concentrations of ethanol in the final distilled product from both methods were similar (47.3% ± 3.2 for OAHD vs. 50.0% ± 2.6 for HD). Interestingly, in OAHD, process control is faster and distillation can be stopped in seconds and consequently, less unwanted distillate in comparison to HD (0.05% ± 0.01 and 2.28% ± 0.03 of total distilled product, respectively). The findings of this study introduce OAHD as a potentially economical and environmentally friendly method for the ethanol distillation process.

1. Introduction

As the demand for energy continues to increase globally, fossil fuel usage will likewise continue to rise. There is still a plentiful supply of fossil fuels at reasonably low cost, although this is likely to change in the future, but more critically the ever growing use of fossil fuels is unlikely to be sustainable in the longer term principally due to the attributed increase in greenhouse gas emissions from using these fuels and the environmental impacts of these emissions on global warming (Hill et al., 2006). There is therefore significant interest in identifying alternative renewable sources of fuels that are potentially carbon neutral (Hill et al., 2006; Rittmann, 2008; Demirbas, 2009). It was previously reported that ethanol and gasoline mixtures can be used as fuels for reducing environmental contamination. In addition, blending gasoline with anhydrous ethanol has been reported to improve octane index (Meirelles et al., 1992).

Ethanol is usually produced by the fermentation process although at a relatively low concentration. Its concentration in production media can vary from less than 3 to more than 12% depending on raw materials and fermentation conditions. To obtain a fuel-grade product, levels of ethanol should be increased to more than 99% (ASTM Standard D4806, 2010). Distillation is the most common separation operation for miscible liquid mixtures in the chemical industry, including facilities producing ethanol (Zacchi and Axelsson, 1989). Recovery of ethanol from the fermentation broth starts by distillation

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of the dilute aqueous alcohol to reach a concentration of about 96% ethanol by repeating distillation several times and followed by a hydrophilic molecular-sieve-based drying system. The energy used for distillation can reach up to 40% of the total energy demand in bioethanol production (Huang et al., 2008). Thus, using the traditional distillation method (i.e., hydrodistillation, HD) to concentrate ethanol solutions has disadvantages of being time and energy intensive, therefore alternative methods have received much recent research interest.

Ohmic heating is defined as a process wherein an alternating electrical current is passed through materials and can be used to generate heat within the product (Knirsch et al., 2010). The heating occurs in the form of internal energy transformation (from electric to thermal which is due to Joule effect) within the material (Sastry and Barach, 2000). Ohmic processing enables materials to be heated at extremely rapid rates from a few seconds to a few minutes (Sastry, 2005). This alternative heating method addresses some shortcomings of traditional heating systems by removing hot surfaces from the heating of the fluids (Sakr and Liu, 2014). Ohmic heating only works in electrically conductive fluid systems due to the necessity of ionic mobility. Therefore, materials with low electrical conductivity (e.g. ethanol–water mixtures or fermented alcoholic mixtures) may require the addition of extra electrolytes to improve their conductivity (Goullieux and Pain, 2005). Consequently, salted water is used as the liquid phase for ohmic treatments (Goullieux and Pain, 2005). Electrical conductivity is the main parameter in heating rate in ohmic heating treatments. The electrical conductivity of materials changes by temperature and the current density would vary accordingly if a constant voltage applied (Goullieux and Pain, 2005; Gavahian et al., 2012). Ohmic heating can be considered as an energy saving process in comparison to conventional heating (Goullieux and Pain, 2005; Gavahian et al., 2012). Environmental impact can be reduced by reducing electrical energy consumption provided that this energy generated from fossil fuels combustion. To obtain one kWh electricity from coal or fuel, about 800 g of CO_2 will be emitted to the atmosphere during combustion of fossil fuels (Ferhat et al., 2006). Ohmic heating as an alternative extraction technique of medicinal plants was reported by Sensoy and Sastry (2001). This heating system in combination with a Clevenger type apparatus was recently utilized for the extraction of essential oils from some medicinal plants and named "ohmic assisted hydrodistillation" (OAHD). Previous studies have shown that this new method consumes less energy and has shorter extraction times in comparison to traditional HD systems (Gavahian et al., 2011, 2012, 2013). Therefore, ohmic heating (OAHD) is thought to be advantageous in the ethanol distillation process.

The main objectives of this study were to evaluate the efficacy of using OAHD as an alternative method to traditional HD for ethanol separation and concentration, and compare the time and energy demands of these two methods.

2. Materials and methods

2.1. Solutions preparation

Three liters of 10% (v/v) of ethanol (300 mL ethanol 200 proof DeconLabs, USA and 2700 mL distilled water) containing 0.5% (15 g) NaCl (as the electrolyte) were prepared. This salt concentration was selected to imitate the conductance of an available sample of fermented corn broth (electrical conductance of 0.4 S/m). These samples were placed in the heating vessel for the distillation process (using either traditional HD or OAHD). Prior to the distillation process, the temperatures of all samples were adjusted to $31 \pm 1\,^{\circ}C$ by preheating the heating chamber.

2.2. OAHD

OAHD was performed using an ohmic distillation device equipped with stainless electrodes (coated by titanium), designed and developed in the Department of Food, Agricultural and Biological Engineering, The Ohio State University (Fig. 1). The electrodes were in rectangular shape and with dimension of 37 mm by 55 mm. The treatment vessel was a normal round-bottom laboratory glass flask with capacity of 5 L which was connected to a condenser (with the length of 920 mm). Processing parameters (i.e. processing time, temperature, applied voltage, current density and power consumption), were precisely monitored using a software developed and connected to the computer via a data logger similar to that described by Ramaswamy et al. (2014). Electrical conductivity is the main parameter in heating rate in ohmic heating treatments (Goullieux and Pain, 2005). As the electric conductivity of materials changes by temperature (Goullieux and Pain, 2005), the current density would vary accordingly. Therefore voltage was adjusted to keep the OAHD device running in constant wattage of 168 ± 5 W. Input voltages were controlled from the power supply of the ohmic device using a variable autotransformer (The Superior Electric Company, Bristol, CT, USA).

In the OAHD procedure, 3 L of salted water–ethanol mixture (containing 10%, v/v ethanol) were heated in the apparatus flask from an initial temperature of $31 \pm 1\,^{\circ}C$ (similar to initial temperature of the material used in HD method). The heating process continued until the temperature reached 98 °C, so the amount of the ethanol in feed was reduced to a constant amount in all the experiments (according to the ethanol–water diagram) (Rieder and Thompson, 1950). As the distillation started, the amount of collected ethanol was recorded every 10 s using a digital scale (1000C-3000D Precisa, Switzerland). The distilled product was then equilibrated to 15 °C in a preset water bath (Fisher Scientific, Isotemp 1016S, USA), and the concentration of ethanol in that product was measured by an alcoholmeter (BSG HandCraft, USA).

2.3. Hydrodistillation

HD is the traditional method of distillation in ethanol separation (Huang et al., 2008). HD was carried out in a similar way as OAHD but by using a 5 L electromantle heater (12035-25 Hemisphere Mantle Glas-Col, USA) as the heating source (instead of ohmic heating). The device was run at a constant voltage (100 ± 5 V), current (1.6 ± 0.1) and wattage (168 ± 5 W). Otherwise, the flask and condenser used in HD were exactly the same as that used for the OAHD process. In addition, processing parameters (i.e. processing time, temperature, applied voltage, current density and power consumption) were monitored using the designed software and a data logger (Ramaswamy et al., 2014). Similar to OAHD, 3 L of 10% (v/v) ethanol (containing 0.5% NaCl) was heated in the apparatus flask from an initial temperature of $31 \pm 1\,^{\circ}C$. As with OAHD, the distillation process continued until reaching the

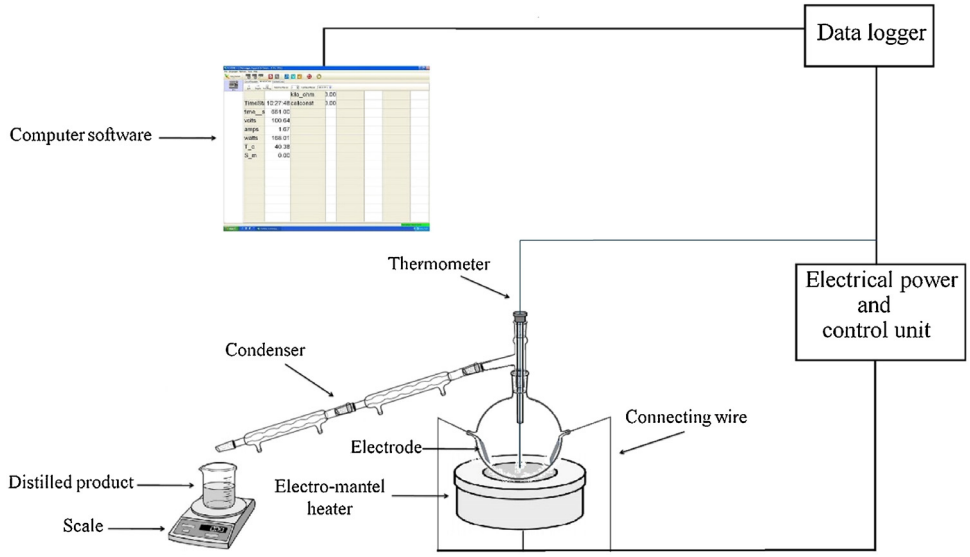

Fig. 1 – Schematic representation of distillation equipment.

temperature of 98 °C. As the distillation started, the weight of collected ethanol was recorded at 10-s intervals using a digital scale (1000C-3000D Precisa, Switzerland). The concentration of ethanol in the distilled product was measured by an alcoholmeter (BSG HandCraft, USA) after equilibrating to 15 °C in a water bath (Fisher Scientific, Isotemp 1016S, USA).

2.4. *Evaluation of process control*

Process control is one of the important aspects which should be considered before industrialization of an emerging technology. For this purpose, after disconnecting the electrical current (disconnecting the input energy to the system), we measured, both for OAHD and HD, the length of the time that distillation process continued and also the volume of distilled product which was collected during this period.

2.5. *Statistical analysis*

All experiments in this research were performed in triplicate. Analysis of variance (ANOVA) was performed to determine significant differences between the means and Duncan multiple range test was used to compare among the means using SPSS (version 19.0.0; IBM Institute Inc., USA).

3. Results and discussion

3.1. *Comparison of temperature profiles of the ethanol–water mixture during distillation*

The temperature profile during OAHD and HD is shown in Fig. 2. To reach the boiling point (about 93 °C) and collect the first ethanol droplet, it was necessary to heat the feed mixture for 35.6 ± 0.1 min in OAHD while it was more than 50 min for HD (53.3 ± 0.6 min). The rate of temperature rise was obtained from the slope of the temperature profile (Fig. 2). Table 1 shows that this parameter in OAHD was almost 1.5 times greater than that of HD. This finding is in line with previous reports on OAHD (Gavahian et al., 2011, 2012). It was previously reported that the rate of temperature increase in OAHD of the 1% (v/w) salted water containing 30 g dried Shirazi thyme was six times greater than the traditional system at a constant voltage of 100 V (Gavahian et al., 2011). A similar result was reported on

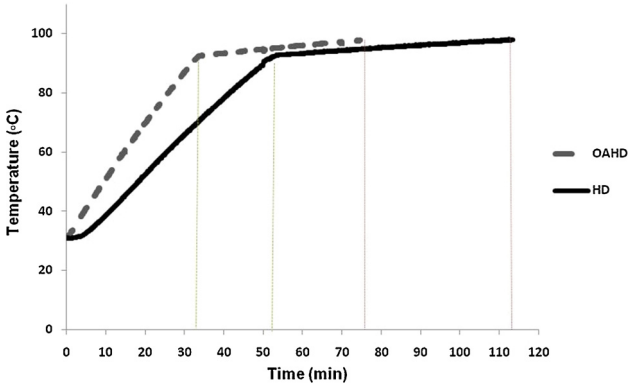

Fig. 2 – Temperature-time profile of feed ethanol–water mixture as a function of time during distillation for OAHD and HD.

Table 1 – Effect of distillation method on rate of temperature increase, come up time, distillation duration, total process time and the rate of ethanol distillation in OAHD and HD.

	OAHD	HD
Rate of temperature increase (°C/min)	$1.9^{a} \pm 0.0$	$1.3^{b} \pm 0.0$
Come up time (beginning of distillation) (min)	$35.6^{b} \pm 0.1$	$53.3^{a} \pm 0.6$
Distillation duration (min)	$40.6^{b} \pm 2.3$	$55.2^{a} \pm 5.6$
Total process time (min)	$75.2^{b} \pm 2.2$	$108.4^{a} \pm 5.3$
Rate of ethanol distillation (g/min)	$11.2^{a} \pm 0.2$	$7.2^{b} \pm 0.1$

[a,b] The same letters in each row indicates that the means are not significantly different ($p < 0.05$).

OAHD of common thyme at the same condition (constant voltage of 100 V, electrolyte concentration 1% (v/w) and 30 g dried plant material) in comparison to traditional HD (Gavahian et al., 2012). Similarly, the rate of temperature increase in this study was higher than traditional HD. Electrical conductivity

can influence the rate of ohmic heating and it will vary with electrolyte concentration (Ramaswamy et al., 2014). Moreover, the input energy determines the heating rate of ohmic process (Goullieux and Pain, 2005). The difference between OAHD and HD in terms of the rate of temperature increase in this study was less than previous reports on OAHD (1.5 times vs. 6 times, respectively) due to less electrolyte and lower input voltage.

The most commonly used heating techniques in industry rely on heat transfer from a hot surface. This heat can be generated directly via an electrical heating element (such as the heating system in an electromantle heater used in this study) or indirectly from a hot medium (e.g. steam) via a heat exchanger (e.g. plate). As one of the traditional heating methods, HD requires a temperature gradient to transfer heat to the process liquid. Some limitations in traditional heating systems are fouling (due to high temperature in the surface and small heat transfer rate) which can be a possible issue in distillation of real fermented liquids, and also relatively low heat transfer rates (as the heat transfer will occur mainly due to conduction and convection). Ohmic heaters (OAHD) address the aforementioned problems by removing hot surfaces from the heating of the fluids and generate heat inside the material. As a result, higher heating rates can be an advantage of ohmic heating systems (Goullieux and Pain, 2005; Sakr and Liu, 2014).

3.2. *Comparison of distillation kinetics*

The kinetics of ethanol distillation using OAHD was compared with that of HD (Fig. 3). Distillation with OAHD started much earlier than HD methods: about half an hour for OAHD and almost 1 h for HD. This is due to the more efficient heating in the ohmic system. Unlike the classical conductive heating methods, ohmic heating can heat the entire sample almost simultaneously and at a higher rate, therefore it is able to generate heat inside products rapidly (Goullieux and Pain, 2005; Sastry, 2005).

Table 1 shows the effect of distillation method on starting time of distillation (i.e. come up time), ethanol distillation duration, total process time and speed of ethanol distillation. As the data shows, by the time the ethanol distillation with HD started (i.e. about 53 min), almost half of the distilled product had been collected with OAHD. For the same amount of distilled product recovery (80%), OAHD needed 75 min while HD required more than 108 min.

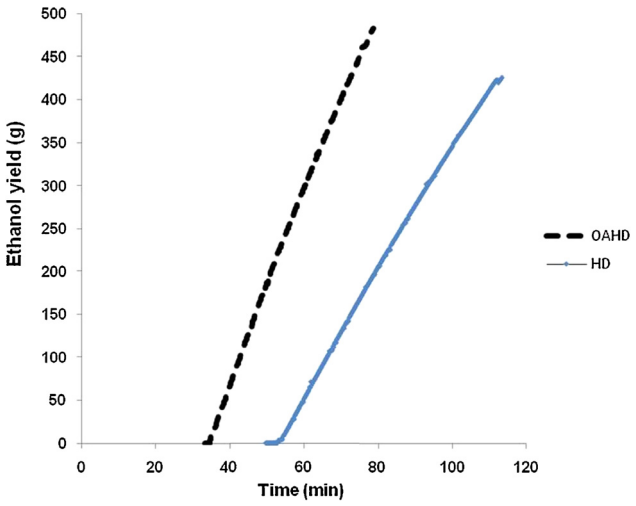

Fig. 3 – Kinetics of ethanol distillation for 3 L of feed mixture in OAHD and HD.

Table 2 – The properties of distilled ethanol obtained by OAHD and HD.

	OAHD	HD
Ethanol recovery (%, w/w)	$79.0^{a} \pm 4.3$	$82.1^{a} \pm 3.7$
Distilled ethanol concentration	$47.3^{a} \pm 3.2$	$50.0^{a} \pm 2.6$
Distilled ethanol appearance	Clear	Clear

[a,b] The same letters in each row indicate that the means are not significantly different ($p < 0.05$).

Table 3 – Waiting time and the amount of extra distilled ethanol after disconnecting the electrical current (stopping) in OAHD and HD.

	OAHD	HD
Required time to stopping the process (s)	$29.7^{a} \pm 0.2$	$276.7^{a} \pm 9.7$
Amount of extra distilled ethanol in stoppage period (g)	$0.2^{a} \pm 0.0$	$96.1^{a} \pm 0.1$

[a,b] The same letters in each row indicate that the means are not significantly different ($p < 0.05$).

The rates of ethanol distillation of OAHD and HD are shown in Table 1. This parameter was obtained from the slope of each graph in Fig. 3 by dividing the amount of distilled ethanol (g) to its corresponding time (min), which is equal to the mean rate of ethanol distillation (g/min). The results show that OAHD isolates ethanol almost 55% faster than the conventional HD method. This data illustrates that the rate of distillation in OAHD was greater. In addition, the OAHD method has a shorter distillation duration time (i.e. the difference between total process time and starting time of distillation). This is due to higher distillation rate in OAHD. As can be seen in Fig. 3, the extraction graph of OAHD is steeper than for HD. The feed mixture containing ethanol, water and salt will be heated rapidly due to the Joule effect (Goullieux and Pain, 2005).

3.3. *Distilled product quantity and quality*

As the data presented in Table 2 shows, the concentration and visual color of distilled ethanol by OAHD and HD were almost similar. After recovering the same amount of the feed ethanol (about 80% of available ethanol of the feed), OAHD yielded a distilled ethanol with almost the same concentration of that of HD (79.0% ± 4.3 and 82.1% ± 3.7, respectively). In addition, the concentrations of distilled ethanol by OAHD and by HD had no significant difference (47.3% ± 3.2 and 50.0% ± 2.6, respectively). Moreover, the visual appearances (visual color) of these distilled products were similar.

3.4. *Evaluation of process control*

The ability of OAHD in process control compared with that of HD is reported in Table 3. As the data shows, better process control is one of the significant advantages of OAHD. After disconnecting the electrical current, OAHD continued the distillation process only for half a minute while this time for HD was about 4.5 min. In other words, after stopping the electrical current in the distillation systems, HD will continue for 4.3% ± 1.7 of its total process time while OAHD will continue only for 0.7% ± 0.1 of its total process time. Regarding

Table 4 – Effect of distillation method on energy consumption and emitted carbon dioxide.

	OAHD	HD
Consumed energy (Wh/g ethanol)	$0.49^{b} \pm 0.01$	$0.71^{a} \pm 0.04$
CO_2 rejected (g/g of ethanol)	$0.393^{b} \pm 0.011$	$0.569^{a} \pm 0.028$

[a,b] The same letters in each row indicate that the means are not significantly different ($p < 0.05$).

the amount of distilled product after stoppage of electrical current, OAHD produced only 0.2 g of distilled product while HD yielded 96.1 g of distilled product. Stopping the process at the right time can play a crucial part in producing the product with the exact determined concentration and quality.

3.5. *Cost, cleanliness and scale-up*

In terms of time and energy consumption, the reduced cost of distillation is clearly an advantage for the proposed OAHD method (Table 4). As shown in Fig. 3, the traditional method (HD) requires an initial time of 53.3 min for heating 3 L of ethanol–water mixture before the distillation process begins and also 55.2 min for the distillation process, while OAHD required only a total of 75.2 min to complete the heating and distillation process, resulting in a significant reduction in the distillation time. The energy requirement to perform the distillation process, based on the electrical power consumptions for 1 g of distilled ethanol, was 0.71 Wh for HD while this value was 0.49 Wh for OAHD. This indicates a substantial saving in the distillation cost by OAHD compared to the conventional HD technique in the case of using electricity as the source of energy.

Regarding environmental impacts, the calculated quantity of carbon dioxide (the primary greenhouse gas) emitted to the atmosphere is higher in the case of HD (0.569 g CO_2/g of ethanol) than for OAHD (0.393 g CO_2/g of ethanol). That is, using OAHD will reduce by about 31 percent of the amount of emitted carbon dioxide in comparison to HD for distillation of an equal amount of ethanol.

In some countries the main energy source is electricity generated from nuclear sources (e.g. 75% in France, 50% in Russia, and 30% in Japan) and this trend is expected to grow in the future (World Nuclear Association, 2015). Nuclear power may be touted as an environmentally friendly source of energy. However, this source of energy has potentially negative environmental effects including the risk of radiation exposure for communities adjacent to nuclear plants. Nuclear plants also release radiological material and waste into the surrounding area that may threaten the ecology and nearby communities. The history of the nuclear industry suggests that more nuclear disasters are possible (Foss, 2011). So if even nuclear power becomes a major way to generate electricity, reducing the energy consumption will be considered as an environmentally friendly method.

Based on the findings of this research and the previously reported data on ohmic heating, OAHD therefore can be suggested as an environmentally friendly distillation method (from the energy consumption viewpoint). OAHD can also be proposed for the production of larger quantities and higher concentrations of ethanol after more investigation and by scaling up of the equipment to be used in biofuel factories, instead of the conventional HD process.

4. Conclusion

Our results show that OAHD is a promising time- and energy-saving distillation method. OAHD resulted in a reduced distillation time and a substantial energy saving compared to the conventional HD technique. After 75 min of OAHD process, it was possible to collect almost 80% of feed ethanol, whereas HD provided the first drops of distilled ethanol after 53 min and collected almost 80% of feed ethanol after 108 min. Distilled ethanol obtained by HD and OAHD were almost similar in their concentrations and visual color. Compared to traditional HD techniques, OAHD can be considered as a green technology since it consumes less energy to operate. Such advantages could make OAHD an alternative distillation method for alcohol distillation. The findings of this research were in agreement with the previous studies on the application of OAHD. Further investigations are required on large scale distillation of ethanol and commercialization of OAHD.

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