

ORIGINAL ARTICLE

DOI 10.58430/jib.v129i2.19



Enhancement of dimethyl sulphide separation during wort boiling by a single spinning cone evaporator

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Abstract

Wort boiling is the most energy intensive stage in the brewing process. Novel wort boiling systems have been explored to reduce primary energy consumption and improve wort quality and beer flavor stability. Low thermal stress boiling is proposed for wort boiling, but the content of dimethyl sulphide (DMS) in the wort may exceed the required product threshold. A single spinning cone evaporator (SCE) is proposed to enhance the separation of DMS and minimise the energy consumption for wort boiling. The performance of a SCE was evaluated by measurement of fluid flow, ratio of DMS removal, wort self-evaporation ratio based on sensible heat, and thermal efficiency. The results show that use of a spinning cone evaporator, reduced DMS by up to 90% with 2.1% wort self-evaporation ratio with less primary heat consumption. The SCE operation exceeds the evaporation of a gravity film cone. Under reduced pressure, the spinning cone evaporator was less effective in DMS removal.

Keywords:

wort boiling, energy saving, spinning cone, dimethyl sulphide (DMS), flash evaporation

Introduction

Wort boiling is an energy intensive process (Dillenburger et al, 2017; Scheuren et al, 2014a; Scheuren et al, 2016) that removes unwanted flavour compounds, especially dimethyl sulphide (DMS) (Scheuren et al, 2016). In practice, several methods can be applied to recover the heat from the exhaust steam, including mechanical or steam jet compressor (Miedaner, 1986; Willaert and Baron, 2004) and tube heat exchangers as vapour condenser (Willaert and Baron, 2004). These approaches may effectively reduce the primary energy consumption, but under the same total boiling evaporation ratio the thermal stress exerted on the wort is not changed. Thermal stress is positively correlated with the content of carbonyl compounds related to aging in wort (Ogane et al, 2006). Therefore, various wort boiling techniques have been developed to reduce thermal stress by changing the total boiling evaporation ratio, such as high temperature boiling with very short boiling times, low pressure boiling and dynamic low pressure boiling (Willaert and Baron, 2004). In current commercial boiling systems, the minimum total boiling evaporation ratio is approximately 4.5% (Willaert and Baron, 2004). Further reduction in the total evaporation ratio is unfavorable for the separation of DMS from wort. Film evaporation (Willaert and Baron, 2004; Scheuren et al, 2015a), vacuum stripping (Willaert and Baron, 2004), and steam stripping (rectification column) (Dillenburger et al, 2017; Willaert and Baron, 2004) were introduced to reduce the amount of DMS in wort at a lower total boiling evaporation ratio. Feilner et al (2011; 2013a; 2013b; 2015) developed a novel wort stripping system to evaporate DMS in turbulent trickle-film using the sensible heat in the hot wort. In addition, wort self evaporation before or after the whirlpool rest contributes an energy saving benefit in cooling the wort before fermentation by pitching yeast (Dillenburger et al, 2017).

It is known that a spinning cone column (SCC), a variation of vacuum distillation equipment, could enhance heat and mass transfer of gas (vapour)-liquid film. Catarino and Mendes (2011) used SCC distillation as a new process to produce non-alcoholic beer. Belisario-Sanchez et al (2009; 2011)

showed that SCC distillation as a dealcoholisation technique is minimally destructive to wine phenolic compounds. Makarytchev et al (2002) reported analysis of computational fluid dynamics (CFD) which showed that unstable gas flow occurs with pressure pulsations in a dry column at a hydraulic Reynolds number above 100. A recent simulation by Bae et al (2020) showed distinct internal flows and the average velocity was increased about 2.5 times, while the mass transfer area was estimated to increase between 20 and 40% due to the generated liquid droplets.

A spinning cone column enables the evaporation of volatile components in the liquid phase and removal of DMS from hot wort (Catarino and Mendes, 2011; Belisario-Sánchez et al, 2009; Belisario-Sánchez et al, 2011). Here, a single spinning cone evaporator (SCE) was evaluated in forming a liquid film of wort for DMS removal using the sensible heat of hot wort, so as to minimise the primary energy consumption and thermal stress for wort production. The performance of the SCE was evaluated in terms of DMS concentration and the self-evaporation ratio of the wort. It was found that the wort in a centrifugal turbulent film could use sensible heat to evaporate the DMS. The energy saving potential of the SCE system is discussed based on the reported experimental results. The combination of low thermal stress boiling with SCE provides a boiling system with low energy cost, reduced thermal stress and sufficient stripping of unwanted volatiles.

Materials and methods

Spinning cone evaporator rig

The key component of the SCE is a spinning cone pan with an opening of 0.14 metre outer diameter and cone angle of 90°, inserted in a column of 0.15 metre inner diameter (Figure 1(a)). For comparison, a static gravity cone with the approximate evaporation area of the spinning cone was used as film evaporator (Figure 1(b)), generating a wort film by gravity from the apex of the cone.

Figure 1.

Cone geometry of the spinning cone pan (a) and static gravity cone (b)

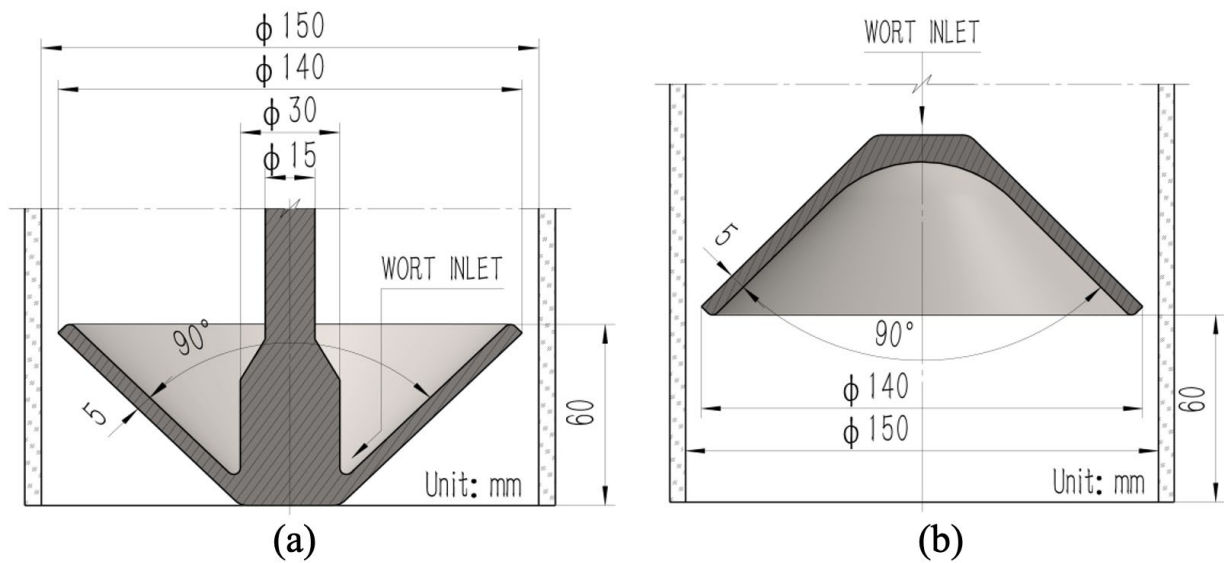


Figure 2 (a).

The spinning cone evaporator rig hardware

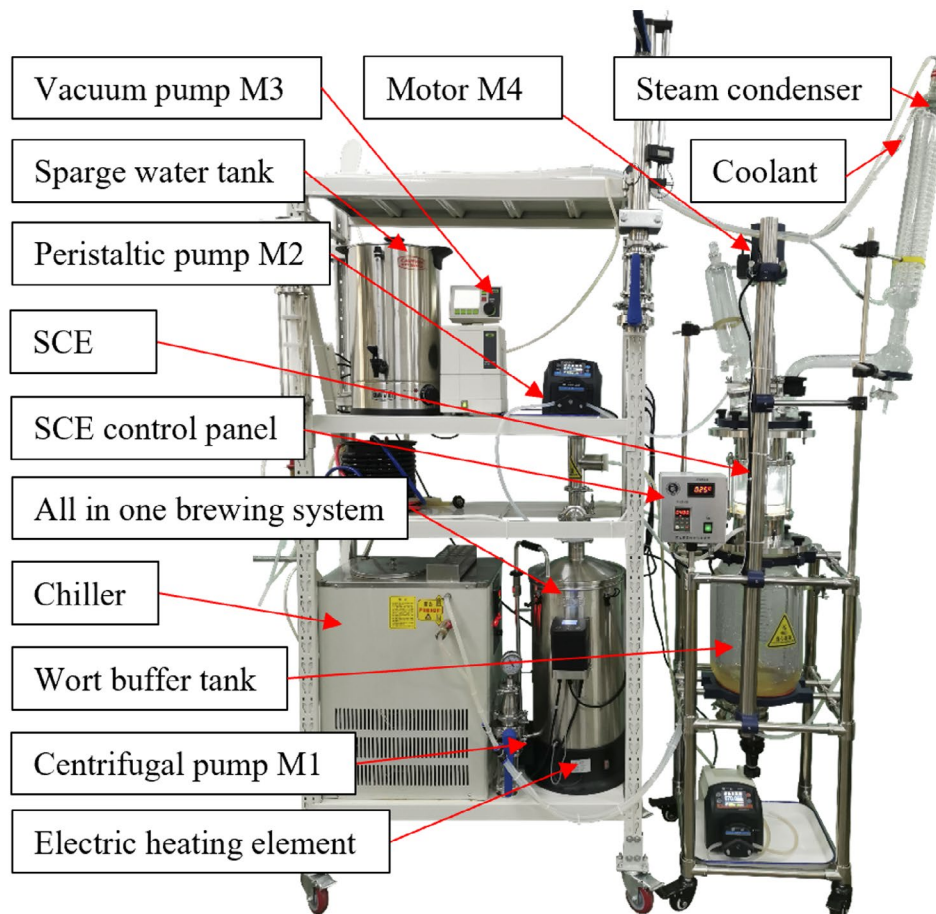
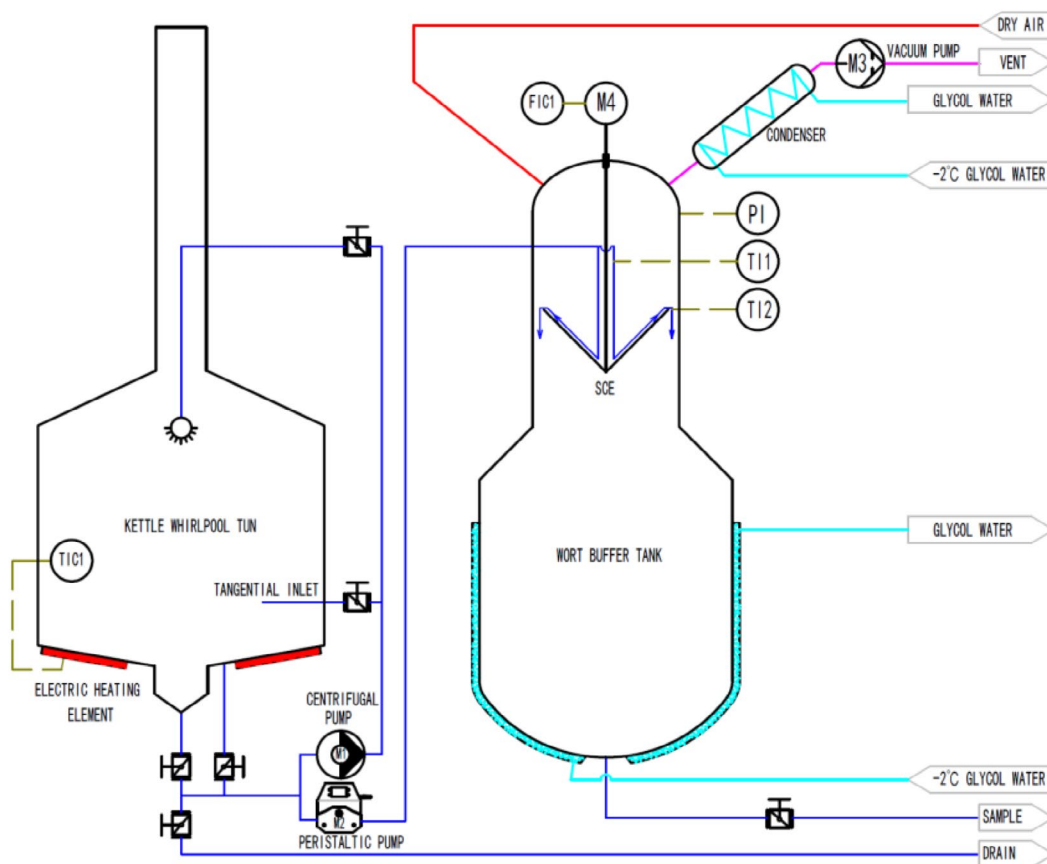


Figure 2 (b).

The spinning cone evaporator rig piping and instrumentation.



The SCE experimental rig is outlined in Figure 2. The wort was produced by an all-in-one brewing system (G30, Grainfather, New Zealand) with the electric heating element and temperature controller. The hot wort was pumped by peristaltic pump M2 (BT601L, Baoding Lead Fluid Technology Co., Ltd., China) at a preset inlet flow rate and evaporated in the SCE chamber under various spinning speeds. A vacuum pump (V-700, BUCHI, Switzerland) was used to adjust the evaporation pressure in the SCE, which was monitored by a diaphragm pressure gauge, PI. The exhaust steam with evaporated DMS was collected by cooling with glycol at -2°C (chiller, DFY-5L/20, Gongyi Yuhua Instrument Co., Ltd., China), and the flow rate of resultant condensate was measured. The outflow wort was stored into the buffer tank and cooled with glycol to -2°C . The wort temperature at the inlet and outlet of the SCE (T_i and T_o) were measured with K-type thermocouples, T11 and T12.

Materials

The malt for wort production was from the Weyermann Specialty Malting Company. Other materials used for analysis of the DMS content in wort and condensed steam are described in Table 1.

Milled Pearson malt (5 kg) was added to 20 L of hot water at 72°C , and the mash temperature was raised to 64°C and held for 90 minutes. The spent grain was separated by lautering and sparging with hot water at 80°C . Sweet wort (25 L) was collected with a density of 1.040 g/mL . The wort was heated at 98°C for 60 minutes with an evaporation ratio of less than 1%, and was transferred to the SCE. The experiments were carried out under atmospheric pressure, with some under reduced pressure (800 mbar , absolute pressure).

Table 1.

Materials used in this work

Material	Purity/Type	Supplier	Application
Malt	Pearson	Weyermann	Wort
DMS standard	≥ 99%	Sigma-Aldrich Chemie GmbH	DMS analysis
Methyl ethyl sulfide	≥ 98%	J&K Scientific	DMS analysis
Glycol	99.5%	Shanghai Macklin Biochemical Co., Ltd.	DMS analysis
Water	Ultrapure water	Sichuan Youpu Ultrapure Technology Co., Ltd.	DMS analysis

Performance of the spinning cone evaporator

The fluid flow in the SCE was observed by tracking the diffusion of red ink with water using video footage captured at a speed of 60 frames per second (iPhone 12, 4K pixels). A red ink droplet was added to the bottom of the spinning cone via a 500 µL gas chromatography injection needle. The diffusion of the red ink was recorded and its residence time in the SCE calculated by analysing frame images.

The DMS concentration in the inlet and outlet wort as well as in the steam condensate was measured using ASBC analysis methods (American Society of Brewing Chemists, 2008) using a Gas Chromatograph (Nexis GC-2030, Shimadzu Co., Ltd). Helium gas was used as a carrier at a flow rate of 14 mL/min with a diversion ratio of 10. The column (HP-INNOWAX size at 30 m × 0.250 mm × 0.25 µm, Agilent Technologies, Inc.) was heated to 40°C, held for 5 minutes, raised to 120°C at a rate of 5°C/min and then to 200°C at 10°C/min with holding 1 minute.

The wort self-evaporation ratio (E), DMS removal ratio (E_{DMS}), and DMS loss rate (φ) are defined as follows:

$$E = \frac{V_s}{V_i} \cdot 100\% \quad (\text{Eqn.1})$$

$$E_{DMS} = \left(1 - \frac{(V_i - V_s) \cdot C_o}{V_i \cdot C_i}\right) \cdot 100\% \quad (\text{Eqn.2})$$

$$\varphi = \left(1 - \frac{V_s \cdot C_s}{V_i \cdot C_i - (V_i - V_s) \cdot C_o}\right) \cdot 100\% \quad (\text{Eqn.3})$$

Where V_s and V_i are the volume flow rates of steam condensate and feed wort; C_o , C_i , and C_s are the DMS concentrations of outflow wort, feed wort, and steam condensate.

The steam evaporation required latent heat (E_L), the wort released sensible heat (E_s) and the evaporation thermal efficiency (γ) are calculated as follows:

$$E_L = V_s \cdot \rho_s \cdot H \quad (\text{Eqn.4})$$

$$E_s = (V_i - V_s) \cdot \rho_i \cdot C_p \cdot (T_i - T_o) \quad (\text{Eqn.5})$$

$$\gamma = \frac{E_L}{E_s} \cdot 100\% \quad (\text{Eqn.6})$$

Where H is the latent heat of secondary (exhaust) steam, C_p is the specific heat of wort, ρ_i is the density of wort, ρ_s is the density of condensing steam, T_i and T_o are the temperatures of inlet and outlet wort. In order to simplify the calculation, the steam latent heat and density were taken as 2258 kJ/kg and 1000 g/L. The wort density is assumed a constant value of 1040 g/L, and its sensible heat is 4.1 kJ/(kg·°C).

Results and discussion

Fluid flow characteristics

The fluid flow in the spinning cone evaporator (SCE) was characterised by the behaviour of a single drop of red ink at the bottom of inverted cone (pan) with water flow, which was dispersed upwards along the cone inner surface and then left the cone. **Figure 3** and **Figure 4** show some images of red ink trajectory at two water flow rates, 40 L/h and 80 L/h, respectively. Ink residues were observed in the bottom of SCE at 200 rpm for the two water feed rates. But the volume of ink residue decreased with increasing rotation rate; when the rotation rate was above 400 rpm, the ink residue at the bottom of the SCE almost disappeared, and the water flow patterns became complex.

Figure 3.

Dispersion of red ink at 40 L/h between 200 and 1200 rpm at 60 frames/sec

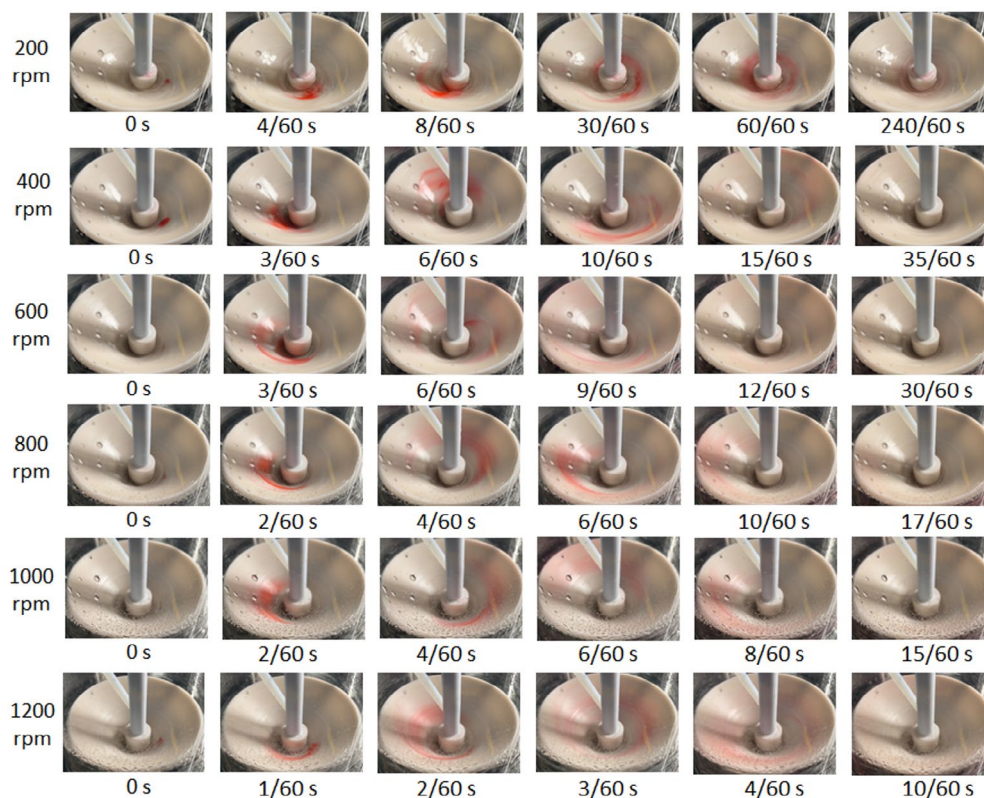
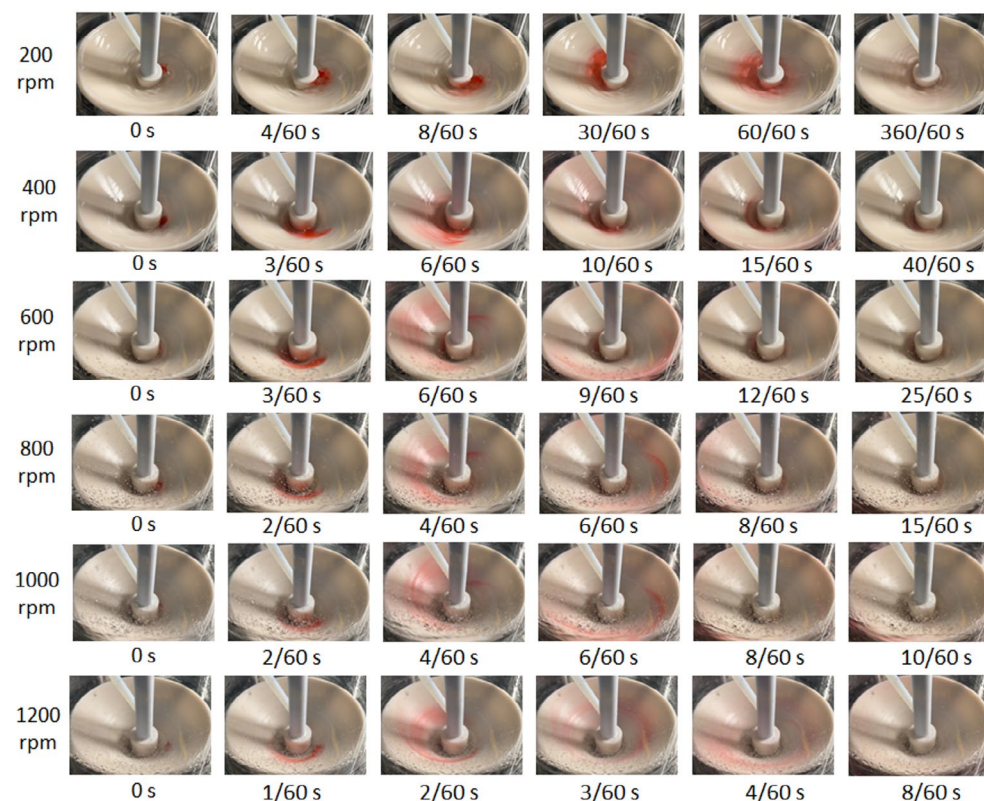


Figure 4.

Dispersion of red ink at 80 L/h between 200 and 1200 rpm at 60 frames/sec



Langrish et al (2003) reported that the water film in an SCC as a wavy layer on top of a laminar sub-layer attached to the disc surface.

The liquid residence time in the SCE pan, taken as the time interval between red ink dropping and disappearing, varies inversely with the rotation rate, and the effect of doubling the water feed rate is not obvious, as shown in Figure 5. At a rotation rate of 200 rpm, the red ink was not seen after about 4 seconds and 6 seconds for 40 L/h and 80 L/h, despite some water remaining in the bottom. This indicates that liquid renewal is good in SCE.

The DMS content in the wort outflow and steam condensate are reported in Table 2. Based on (Eqn.2), it was calculated that 46.5 and 55.2% of the DMS was removed, with wort evaporation ratios at 1 and 0.6% at the wort feed rates of 40 and 80 L/h. Alternatively, the ratio of DMS removal may be obtained from the DMS concentration in steam condensate (i.e., $\frac{V_s \cdot C_s}{V_r \cdot C_i}$). By comparison with the result from (Eqn.2), some of the DMS was not included in the steam condensate, possibly due to the steam not being completely condensed or through sampling errors. According to (Eqn.3), the DMS loss rates were between 6.6 and 8.9%.

Figure 5.

Residence time and SCE rotation rate

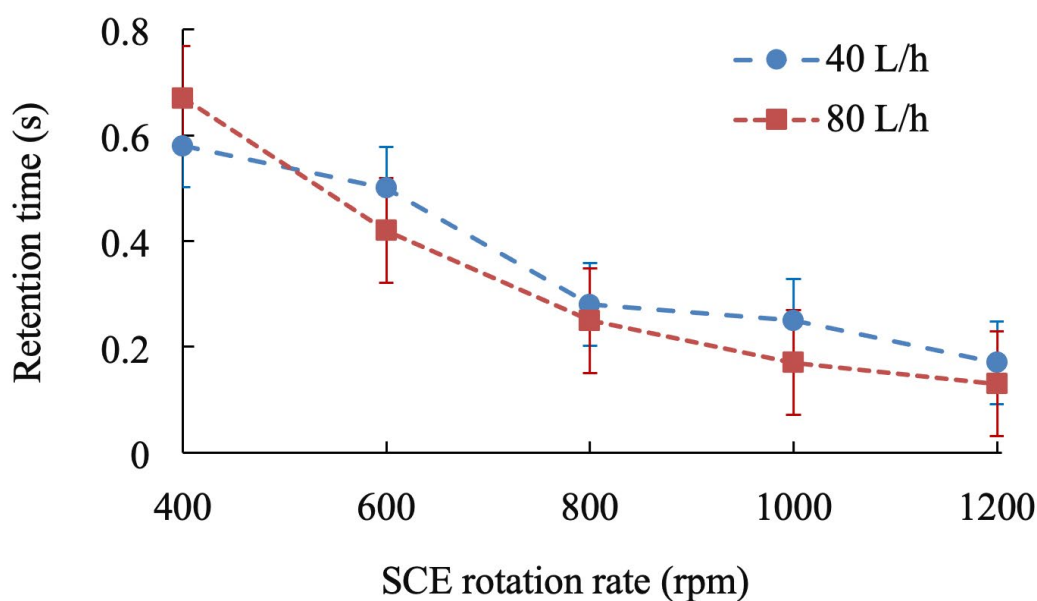


Table 2.

DMS content in wort outflow and steam condensate

Flow rate (L/h)	Outflow 1 ($\mu\text{g/L}$)	Outflow 2 ($\mu\text{g/L}$)	Outflow 3 ($\mu\text{g/L}$)	Average ($\mu\text{g/L}$)	Steam condensate ($\mu\text{g/L}$)
40	300.4	288.9	296.0	295.1	30081.2
80	353.0	370.5	329.0	350.8	38252.8

DMS removal

DMS removal by gravity film cone evaporator

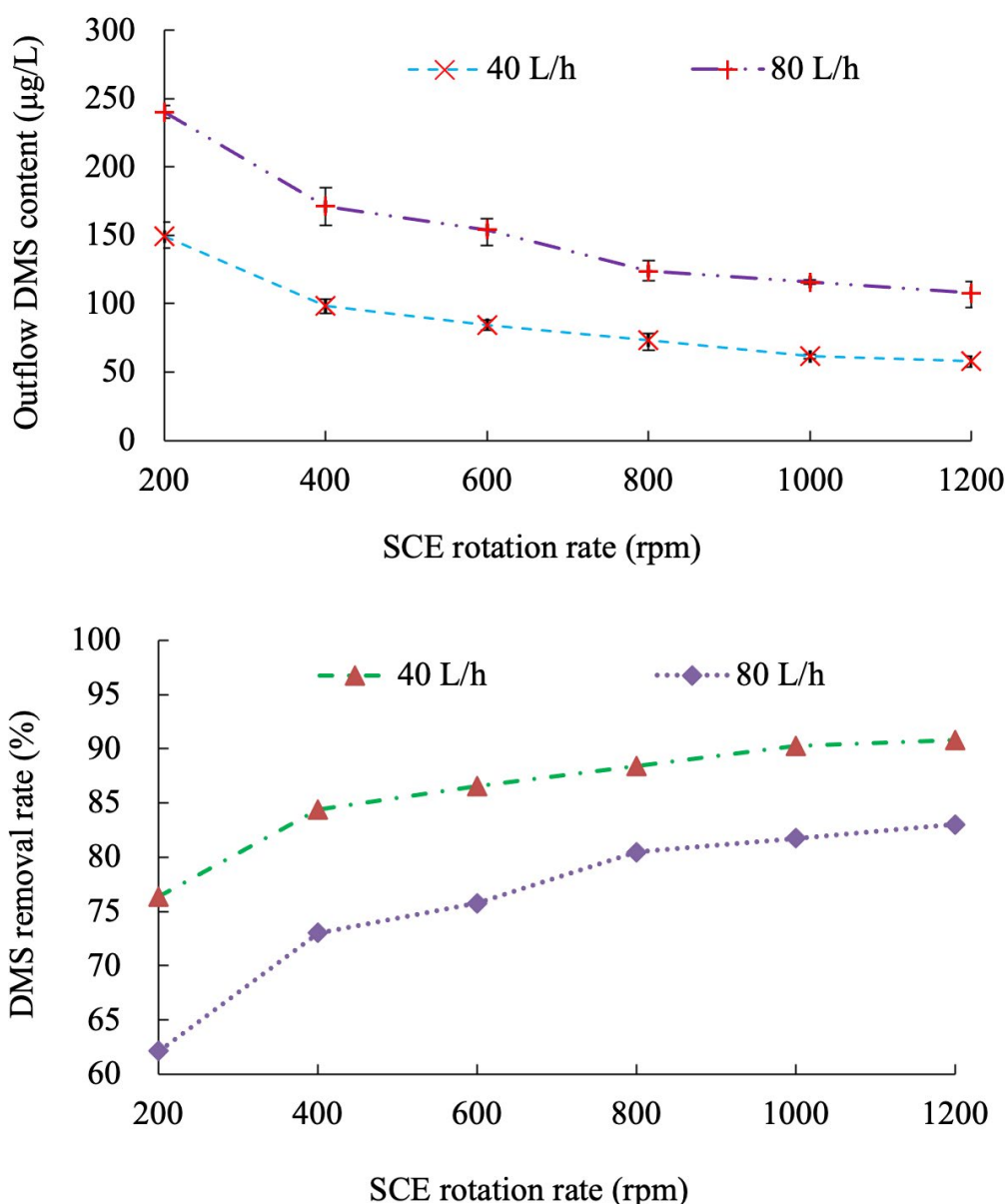
Gravity film evaporation is one of the most effective technologies to remove DMS after boiling or the whirlpool rest (Willaert and Baron, 2004; Scheuren et al, 2015a; Feilner et al, 2013a; Scheuren et al, 2015b). A gravity film cone evaporator (Figure 1 (b)) was used with a wort feed rate of 40 or 80 L/h. The initial DMS concentration of wort was 635 µg/L. The measured steam condensate flow rates were 0.4 and 0.48 L/h at the above feed rates.

DMS removal in the spinning cone evaporator

The SCE performance was investigated at atmospheric pressure with the same wort feed rates (40 L/h and 80 L/h) but increasing the rotation rate from 200 to 1200 rpm with an interval of 200 rpm. The wort inlet temperatures (measured by a K-type thermal couple at the end of the feed tube) at 40 and 80 L/h were 95.9 and 97°C, and the initial DMS contents were 630 and 635 µg/L as the wort was prepared at different times. It is assumed that this was insignificant in the analysis and evaluation of experimental results.

Figure 6.

(a) DMS content in wort outflow and (b) removal rate with SCE rotation rate



With increasing the cone rotation rate, the condensate flow rate also increased from 0.592 to 0.852 L/h for a wort feed rate of 40 L/h and from 0.804 to 1.292 L/h for 80 L/h. The residual amount of DMS in the wort outflow was consequently reduced (Figure 6a). At 40 L/h and a rotation rate over 400 rpm, the DMS content was less than 100 µg/L, meeting the residual DMS requirement of DIN standard 8777. At 80 L/h, the residual values were higher, along with a lower ratio of DMS removal (Figure 6b). Film thickening, due to increased wort feed rate, is not favorable to DMS evaporation. Increasing the rotation rate was insufficient to make the wort film attached to the cone surface thinner at the higher feed rate. In addition, the wort residence time in the SCE was shorter at the higher feed rate (Figures 3 & 4), such that the wort passed through the SCE with sufficient evaporation. In comparison, DMS removal with a gravity film evaporator was 61-69% for the thin film stripping (Feilner, 2011), 83.3% for the Schulz boiling system with subsequent vacuum evaporation, and 87.6% for the Nerb boiling system with subsequent vacuum evaporation (Willaert and Baron, 2004).

The SCE demonstrated better performance at a feed rate of 40 L/h, the DMS removal ratio achieving

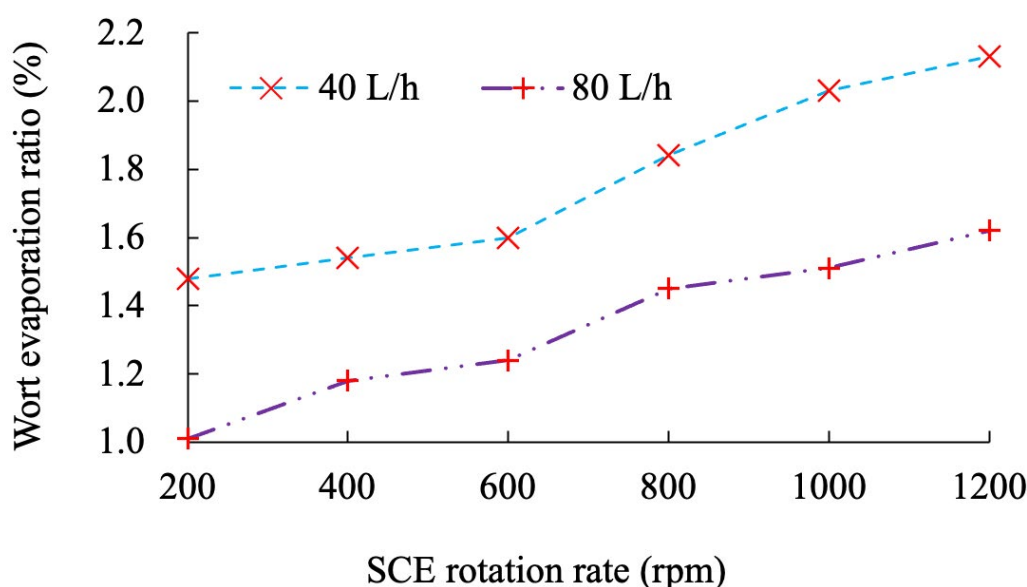
more than 84.4% when the rotation rate was not less than 400 rpm and up to 90.0% with increased rotation rate (e.g., 1000 rpm). In the literature, computer fluid dynamics simulation of SCC indicated that the coefficient of mass transfer from the liquid to the vapour phase through the surface of a liquid film flowing over the spinning cone, increased with the liquid-gas slip velocities (Makarytchev et al, 2002; Makarytchev and Langrish, 2005), which depends upon the cone rotation rate.

The wort evaporation ratio, which contributed to the removal of DMS, increased with rotation rate (Figure 7). At 1200 rpm, wort evaporation was at a maximum of 2.1 and 1.6% for 40 L/h and 80 L/h with a DMS removal rate greater than 90 and 83%. A lower wort feed rate had a higher wort evaporation ratio, together with the lower wort outlet temperature (83.9°C at 40 L/h, 88.3°C at 80 L/h) at 800 rpm.

With other parameters being the same, the volume flow rate of steam condensate for 80 L/h could be 1.5 times higher than for 40 L/h, but the DMS content in the steam condensate remained higher (Figure 6a). Based on the temperature-pressure dependency of free DMS to water, the separation of DMS at low temperature is possible due to the high

Figure 7.

Rate of DMS loss against rotation rate



volatility of undesirable aroma combinations, but the higher transience at low temperature is subject to the mass transport in the boundary layers of the desorption process (Feilner et al, 2013a).

Figure 8 shows that the calculated DMS loss rate by (Eqn.3) was in the range of 8.7-10.3% for 40 L/h and 2.8-9.5% for 80 L/h. In general, the experimental error based on the DMS loss rate is less than 10% for the SCE and gravity film cone evaporation.

Figure 9 shows the DMS content and evaporation rate against SCE rotation rate at 800 mbar (absolute pressure). It was shown that reduced pressure

promoted DMS evaporation and reduction in DMS content in wort at lower cone rotation rate (200-600 rpm), especially for high feed rates (80 L/h). At 400 rpm, the vacuum auxiliary effect is more obvious. Interestingly, at 800 rpm, DMS removal under reduced and atmospheric pressure was almost the same, and the vacuum effect was negligible. However, under high rotation rate (1000 and 1200 rpm), the vacuum reduced DMS removal, compared with atmospheric pressure. Although some reports on SCC suggest that vacuum operation can enhance performance, a fuller analysis should be made considering the specific wort film formed in operation (Feilner et al, 2013a; Catarino and Mendes, 2011; Belisario-Sánchez et al, 2011).

Figure 8.

Rate of DMS loss against rotation rate

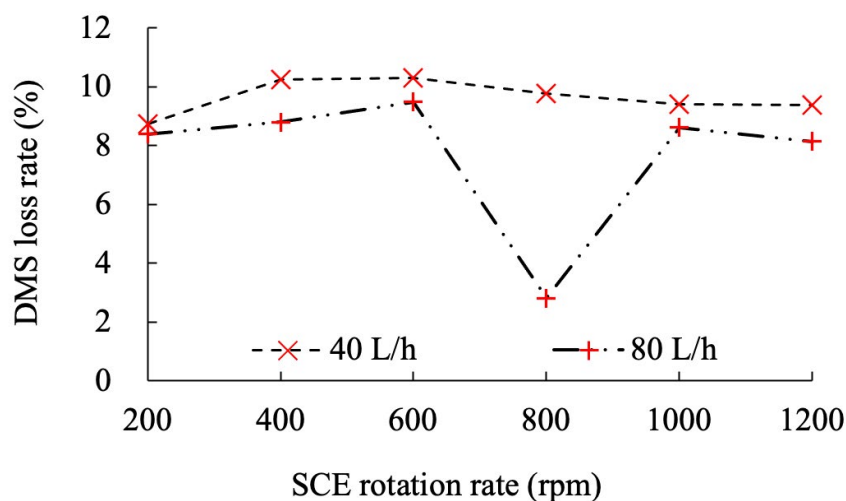


Figure 9(a).

Vacuum operation of SCE (800 mbar, absolute pressure) - DMS content in the wort outflow

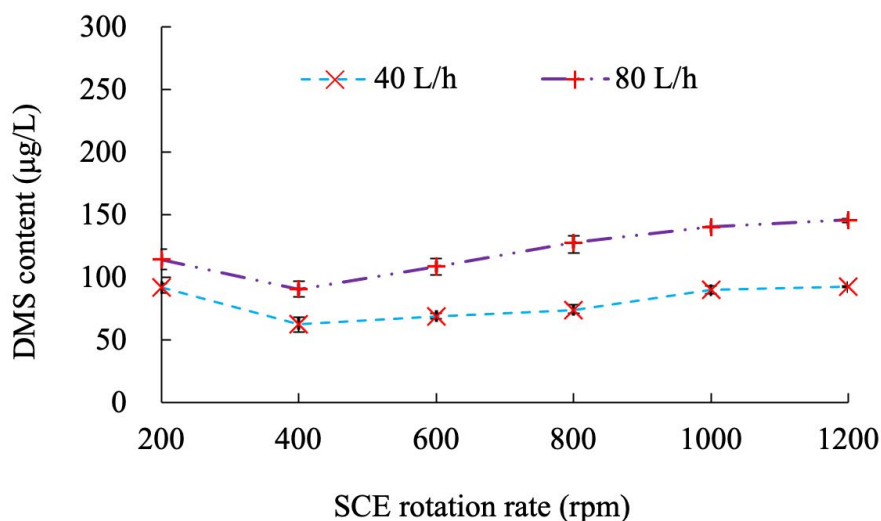


Figure 9(b).

Vacuum operation of SCE (800 mbar, absolute pressure) - DMS evaporation rate

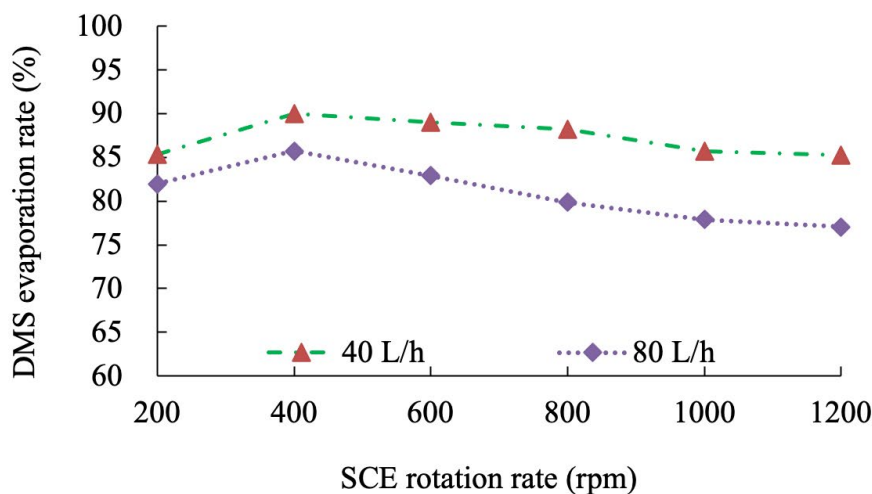
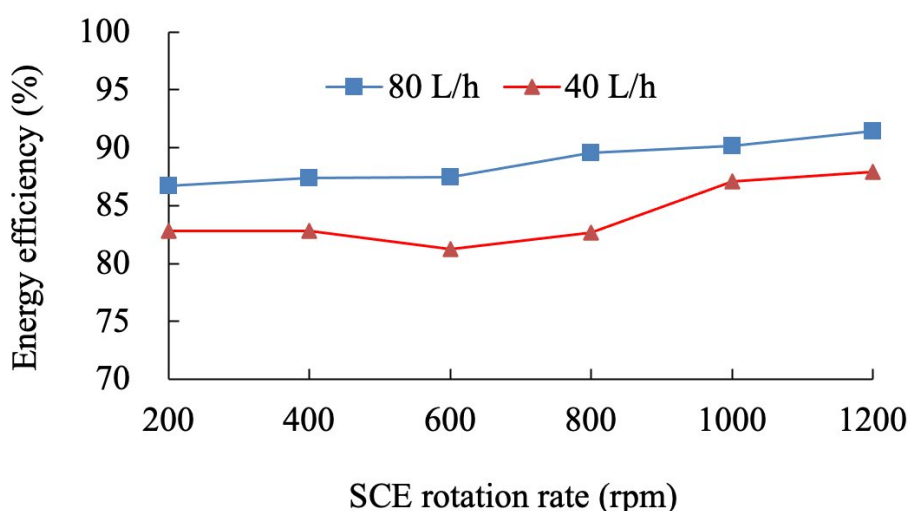


Figure 10.

Thermal efficiency with the SCE rotation rate



In the SCE operation, the latent heat (E_L) required to remove DMS in the wort by evaporation is derived its sensible heat (E_S), corresponding to the decrease of wort outflow temperature. Due to the heat loss through the device wall, the thermal efficiency could reach more than 80% for 40 L/L and 85% for 80 L/h (Figure 10).

In the literature, the total boiling evaporation ratio for DMS removal is 2.5-8% at a heating temperature over 100 °C for more than 30 minutes (Willaert and Baron, 2004). Therefore, most of the operations need extra thermal energy or vacuum.

The thermal energy matching the evaporation requirement (E_w) should be where V_w is 1 litre of wort.

$$E_w = E \cdot V_w \cdot \rho_i \cdot H \quad (\text{Eqn.7})$$

While the SCE operation in this study consumed only the wort sensible heat, the wort outflow temperature was reduced to some extent and reduced the cooling load of the next process. Table 3 compares the evaporation heat required for DMS removal using the current commercial boiling systems with SCE. Here, at a feed rate of 40 L/h

Table 3.

Evaporation heat of boiling with different systems

Boiling system	T (°C)	Boiling time (min)	Total evaporation ratio (%)	Evaporation heat (kJ/L)	Remarks
Atmospheric boiling (Willaert and Baron, 2004; Scheuren et al, 2014b)	100	60-80	8	187.9	Classical
High temperature (Willaert and Baron, 2004)	130-140	2.5-3	6-8	140.9-187.9	
Low pressure (Willaert and Baron, 2004)	103-104	55-65	6-7	140.9-164.4	Classical
Low pressure (Willaert and Baron, 2004; Scheuren et al, 2014c)	103-104	45-50	4.5	105.7	Dynamic
Meura system with packing column (Willaert and Baron, 2004)	100	40-45	2	47.0	0.5~2% primary steam
Tray column (Dillenburger et al, 2017)	100	90	2.7-4.5	63.4-105.7	Secondary steam
Ziemann system (Willaert and Baron, 2004)	100	40-50	4	93.9	Vacuum
Nerb system (Willaert and Baron, 2004)	103/99	50-60	4.7-5.4	110.4-126.8	Vacuum
Schulz system (Willaert and Baron, 2004)	97.5	60	1	23.5	7% vacuum evaporation
Merlin system (Willaert and Baron, 2004)	100	35	5-6	117.4-140.9	Film stripping
Trickle-film (Feilner, 2011)	96	60-90	4	93.9	Gas + self-evaporation
Spinning cone evaporator	98	60	1	23.5	self-evaporation

and the rotation rate of 400 rpm, the wort total boiling evaporation ratio and self evaporation ratio of the wort was 1.0 and 1.5%. Compared with classical atmospheric boiling, it is suggested that use of the SCE may reduce 164.4 kJ/L of heating energy for DMS removal and 42.0 kJ/L of cooling energy for the next operation.

Results and discussion

The performance of the single spinning cone evaporator shows the following:

- The residual DMS in wort outflow was reduced to <100 µg/L at a wort feed rate of 40 L/h and a rotation rate of ≥400 rpm.
- The wort evaporation ratio was increased with the rotation rate. The maximum evaporation rate was 2.1 and 1.6% with a DMS removal of 90 and 83% for the wort feed rate of 40 and 80 L/h.
- For the SCE operation under reduced pressure (800 mbar), the residual DMS content in the wort fell to a minimum at 400 rpm and then increased. The residual DMS content was lower than at atmospheric pressure within a rotation rate of 200-600 rpm.

- A potential energy saving using a spinning cone evaporator was achieved in comparison with other boiling systems.

Author contributions

Xiaoyong Dai: Ideas, data collection and analysis, literature search, experimental design, writing (original draft).

Pengyu Wang: Data collection.

Qing Xu: Writing (review and editing), data analysis and project administration.

Long Wu: Literature search.

Zhanyong Li: Supervision and funding acquisition, writing (review and editing).

Acknowledgments

This work was supported by Key-Area Research and Development Program of Guangdong Province, China (No.2020B0202010004).

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