Release of electrode materials during the processing of liquid foods using pulse electric field treatment

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Abstract— Application of pulsed electric fields (PEFs) is an alternative treatment to thermal pasteurization of liquid foods, which inactivates microorganisms without degrading flavor, texture, and nutrients compared to other conventional technologies. This paper scrutinizes the applicability of PEF processing of carbonated beer using a titanium electrode based sealed processing chamber. The release of metal ions during PEF processing was evaluated by using Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES). As metal ions directly affect the organoleptic properties of beer, a sensory evaluation is conducted with the panelists comprised of brewing experts. It has been found that the released amounts of metal ions are lower than the detection levels of ICP-AES, and much lower than what is accepted in consumable foods. Supporting the metal analysis data, the sensory panel also reported non-off flavors in the PEF-processed beer. In addition, analytical indicators like the deterioration of trans iso-α-acid confirm no chemical changes in beer before and after PEF treatment under the treatment conditions studied.

I. Introduction

The application of pulsed electric fields (PEFs) in food processing industry has gained interest as a valuable non-thermal pasteurization method. The practical features of PEF application with optimal control of metallic ion release are its ability to retain the original chemical composition and other organoleptic characteristics of food while reducing the microbial load. Since consumers prefer fresh products, PEF processed foods can be a promising choice for the growing demand of minimally processed fresh like foods [1, 2].

A wide range of foods can be successfully treated with the PEF process including juices of apple, cranberry, orange, tomato, as well as milk, beer, and yogurt drink [3-10]. Beer is one of the oldest and most consumed alcoholic beverages worldwide. The application of thermal pasteurization is a prevailing method in beer industry [7]. However, thermal pasteurization causes adverse effects on sensory characteristics and nutritional attributes of food. Since PEF treatment minimizes the adverse thermal effect on food while increasing the shelf life, an evaluation of PEF applicability in the beer industry is considered worthy.

Normally, during PEF processing, electrochemical reactions can occur at the electrode-liquid interface, which releases metal ions into food. The presence of metal ions may have consequences on the flavor, safety, and quality of the food. In fact, a metallic mouth feeling has been reported during sensory analysis of PEF treated beer [11]. In addition, the metal ions can produce reactive oxygen species, which oxidize organic compounds in beer, altering the organoleptic properties and nutritional quality. In this regard, the present work uses a treatment chamber built with high-grade titanium and smooth electrode contours to eliminate the release of metal ions.

The sensory evaluation of beer considering release of metal ions is an important approach in monitoring the quality control of the PEF treated foods. Due to the subjective nature of sensory analysis, analytical indicators like the deterioration of trans iso-α-acid to evaluate the chemical changes [12, 13] and concentration of metal ions like iron and titanium have been measured using HPLC and ICP AES respectively. The study shows a strong correlation between the sensory test evaluations from panelists comprised of brewing experts and the analytical tests data; both endorsing the quality of PEF treated beers using the above mentioned titanium treatment chamber.

II. Materials and Method

A. Materials

The trans iso-α- acid (DCHA-Iso, ICS-14) was purchased from American Society of Brewing Chemists (ASBC, MA, USA). All beer samples were supplied by a local craft beer company located in Waterloo, Ontario, Canada.

B. PEF Processing Set up

The main components of PEF set up are a pulse power generator, which is responsible for generating high field short-duration pulses, and a processing chamber. Details on the PEF treatment facility can be found in our earlier publication [6]. In brief, a thyratron-based RC discharge circuit was used, which produces the exponentially decaying waveforms of voltage and current. The capacitors were charged by using a 100 mA current source to a dc voltage of 4 kV. The type of liquid in the chamber, and the treatment temperature, affect the equivalent resistance; hence, the width of the pulses generated during processing.

C. PEF treatment of beer

The liquid food is treated by using high field short duration pulses. The electric field is generated in 1 mm annular gap between inner and outer electrodes. The PEF set up is sealed to avoid exposure to oxygen and cross contamination during processing. In order to maintain the low processing temperature of the beer, the inlet and outlet tanks are immersed in ice and a cooling jacket encloses the processing chamber. A continuous flow is maintained inside the chamber during processing.

The liquid flow is maintained under a pressurized environment (maximum 40 psi), which helps to reduce the probability of arching, since the breakdown strength of bubbles increases with the pressure. The pressure difference of inlet and outlet tanks are maintained around 1 psi, which facilitates the smooth flow of the liquid during processing. The outlet and inlet tanks are initially pressurized by beverage grade CO_2.

Before treating beer, the system is flushed with 1% powdered brewery wash (PBW) cleaners, and then followed by distilled water and 70% ethanol solution. The preliminary treated beer is flushed through the system and collected in the waste tank. The subsequently treated beer is collected in the inlet tank using a three-way switch to control the flow directions.

D. Detection of Metal Ions

In this study, titanium and iron (Ti and Fe) ions are analyzed by using ICP-AES. Before ICP-AES analysis, the beer samples are subjected to wet digestion as described in [9]. The ICP-AES has an average detection limit of 8 parts per billion (ppb) for Fe and 2 ppb for Ti ions.

E. Sensory Analysis

A sensory panel comprising of experienced panelists tastes the untreated and PEF-treated beer samples. The sensory test allows for comparison of aroma, appearance, flavor, sensation in the mouth, and overall impression, between the PEF treated and untreated samples. Panelists rank the samples and also include any special comments. Four different samples were used in sensory analysis, of which two are untreated; one is PEF-treated without high voltage application, and one is PEF-treated with high voltage applications. Samples are arranged randomly and then given to the sensory panel for tasting.

F. Processing Conditions

Three different processing conditions are applied to the beer before the sensory tests. The first sensory test is performed with the PEF treated beer at 4 kV, 30 Hz, and 50 ml/min flow rate with 0.3 µF capacitor. The second sensory test is conducted with the beer treated at 3 kV, 21 Hz, and 100 ml/min with 0.6 µF capacitor, and lastly, the beer used for the third sensory test is treated at 3.2 kV, 21 Hz, and 30 ml/min flow rate with 0.6 µF capacitor.

G. HPLC Analysis

HPLC analysis is conducted by using a Waters Alliance System (2695 separation model) with PDA 2996 Diode Array system. Chromatographic separation of trans iso-α-acid was accomplished with a Agilent Zorbax Eclipse XDB-C18 column (4.6 × 250 mm, 5 µm, Agilent Co.). The data analysis is carried out with Waters Empower TM software. The mobile phase comprised of 34% solvent A, made using 960 ml of ultrapure water, 37 ml H3PO4 , 3 ml of 0.1M Na2EDTA and 66% of solvent B (methanol, Sigma Aldrich.). The injection volume is 100 µl with a flow rate of 1 ml/min. The column temperature is maintained at 40˚C. The calibration is performed with five standard solutions (20, 40, 60, and 100 ppm) of DCHA-ISO, ICS-14 prior to each experiment. The statistical analysis is performed to obtain a mean comparison of trans iso-α-acid content by using a one-way ANOVA with Tukey test. Minitab 18 statistical software is used for the analysis.

III. RESULTS AND DISCUSSION

During the PEF treatment, the applied electric field strengths should be sufficient to inactivate microorganisms. As reported in the literature, the range of 20 to 50 kV/cm field strength with 1-10 µS pulse duration is the ideal range [11]. Additionally, the energy density should be maintained in the range of 50 - 1000 kJ/kg to obtain successful inactivation [11, 14]. In this study, the applied energy density varies from 95 J/ml to 315 J/ml. Fig.1 shows the typical current and voltage waveforms generated during PEF processing of beer with the calculated power curve. The calculated total energy by using Fig.1 is 304 J/ml with 0.3 µF, which exhibits a relatively shorter pulse width compared to the PEF treatment conducted with 0.6 µF.

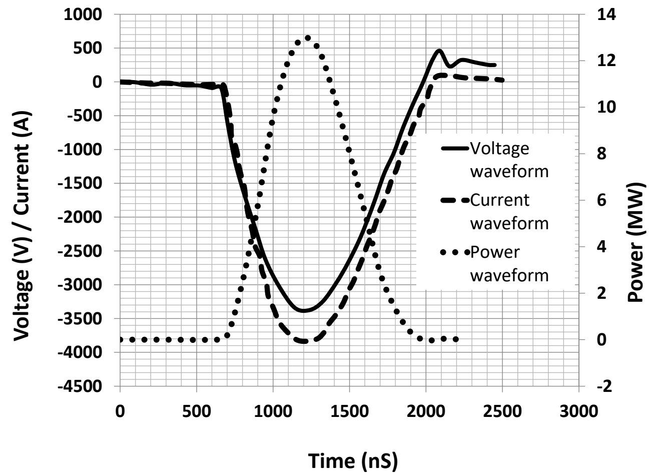

Fig. 1. Typical voltage and current waveforms generated during PEF treatment of beer

A. Migration of Electrode Materials

During PEF treatment, the Ti electrode is in direct contact with the beer. At the interface of the liquid and electrode, the charged ions are transferred from food to electrode or electrode to food. The transfer rate of metal ions depends on the chamber design, pulse width, magnitude of current, shape of the pulse, material of electrode, peak voltage, and the composition of food [4-6].

The observed initial Fe ion concentration is different in different untreated samples, which can be attributed to the type of beer, maturity level of the beer, the raw materials used in the beer processing, and the duration of beer stored inside the stainless steel canister. The conductivity and pH values of the beer treated are 2.4 ± 0.05 mS/cm and 4.4 ± 0.1 respectively.

It is apparent from the results that there is no release of Fe ions into the beer during processing. This phenomenon can be attributed to the replacement of Fe based electrode with Ti electrode in the treatment chamber, eliminating the source of Fe for the possible oxidation reactions that can occur at the interface. Some of the possible electrochemical reactions that may take place at the titanium electrode on the interface can be shown as follows [15-20]:

$$Ti_{(s)} \rightarrow Ti^{3+}_{(aq)} + 3e^- \quad (1)$$

$$Ti^{3+}_{(aq)} \rightarrow Ti^{4+}_{(aq)} + e^- \quad (2)$$

The concentration of Ti ions are lower than the detection level, which may be due to the absence of reactions at Ti electrode at the interface or may be due to the significantly lower reaction rate, which produces significantly lower amount of metal ions compared to stainless steel electrodes. This result is in accordance with the earlier research conducted to predict the corrosion rate of Ti electrode and the study conducted on performance of different electrode materials during PEF processing [21, 22].

B. Sensory Characteristics of beer

The sensory characteristics of the beer before and after treatment are evaluated. The ranking done by panelists indicates that the PEF treated sample at 304 J/ml is the best sample out of the four samples tasted. The sensory evaluation of the beer treated at 315 J/ml with 0.6 µF received lower ranking compared to the beer treated at 94 J/ml with 0.6 µF and 304 J/ml with 0.3 µF. In addition, an improvement with the appearance of the beer was identified by the panelists, which may be attributed to the removal of yeast cells from the beer during PEF treatments. These results are well correlated with the metal ion release data obtained for the treated and untreated samples. Furthermore, the panelist did not find any metallic flavor in the treated samples.

C. Analysis of HPLC data

Due to the subjective nature of the sensory data, the use of analytical approaches to evaluate the possible chemical changes in PEF treated food is important. HPLC analysis was conducted to evaluate the trans iso-α-acid content in beer. One of the main contributing factors for flavor instability of beer is the degradation of trans iso-α-acids during storage, which limits the shelf-life of the beer [12, 23, 24].

The concentration of trans iso-α-acids in beer can vary between 15 and 100 mg/l [12, 25]. Besides contributing to bitterness, trans iso-α-acids are imperative for beer foam stability and shelf-life, since it can act as a preservative due to distinct antibacterial attributes [15-17]. Fig. 2 shows the HPLC chromatograms derived for beer, it is apparent from Fig. 2, the peaks appeared for trans- isocohumulone, trans-isohumulone, and trans-isoadhumulone between 9 to 13 min retention time period. The quantification of iso-α-acids was done for each substance separately by using a calibration curve derived prior to each experiment. Fig. 3 shows the comparison of iso-α-acids for treated and untreated beer samples at 304 J/ml and 0.3 µF. It is apparent from Fig. 3, the quantity of trans- isocohumulone, trans-isohumulone, and trans-isoadhumulone has not changed significantly ($P > 0.05$) during PEF treatment. This confirms the sensory data obtained for the beer treated at 304 J/ml and 0.3 µF, since two-third of the panelists have selected this sample as the best sample.

Similar results are observed for the beer treated with 0.6 µF, as seen from Fig.4, the PEF treated samples at 94 J/ml and 315 J/ml are not significantly different ($P > 0.05$) compared with untreated samples. This further confirms the stability of trans iso-α-acids during PEF treatment at the energy level applied.

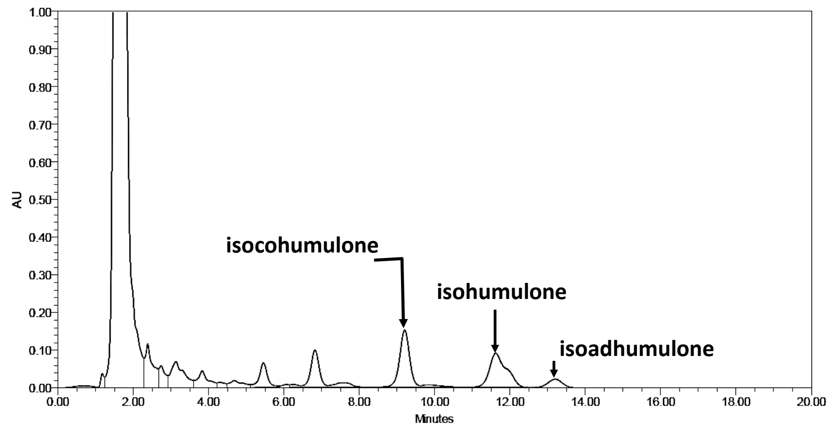

Fig. 2. HPLC analysis of iso-α-acids: chromatogram derived from beer (Lager type) for trans-isocohumulone, trans-isohumulone, trans-isoadhumulone.

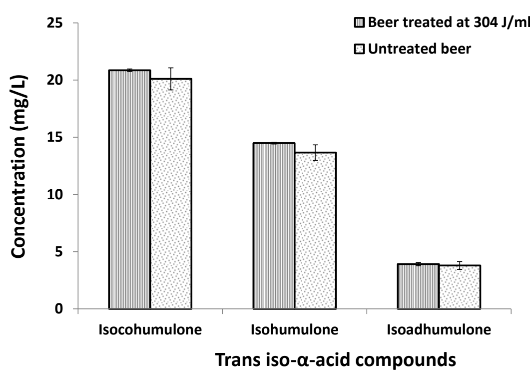

Fig. 3. Comparison of trans iso-α-acids (isocohumulone, isohumulone, and isoadhumulone) in treated and untreated beer with 0.3 µF.

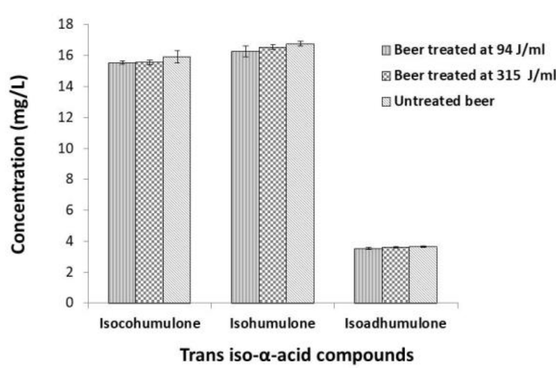

Fig. 4. The derived concentration of trans iso-α-acids (isocohumulone, isohumulone, and isoadhumulone) from treated and untreated beer with 0.6 µF.

IV. CONCLUSION

The PEF processing of craft beer is promising with the system and experimental conditions used in this study. The Fe ion concentration found after the PEF treatment of beer does not exceed the concentration found in untreated beer, confirming that there is no release of Fe ion into the beer during PEF processing. The sensory panelists did not report any metallic mouth feeling in the processed beer, validating the analytical results of Fe ion concentration. The release of electrode material, Ti ions, in treated beer was lower than the detectable level of ICP for all the treatments considered, which further confirmed the safety of PEF treated beer for the consumers with the experimental conditions proposed. The analysis of trans iso-α-acids does not show a significant change between the treated and untreated beer. Supporting the analytical test data, sensory panelists have also reported that PEF treated beer at 304 J/ml energy level is better than the untreated beer; and further indicated that PEF treatment improved the appearance of the beer with reduced haze level.

V. ACKNOWLEDGEMENT

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