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# Comparative analysis of the aroma profile of pineapple beers brewed with juice added at different times

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## Abstract

Pineapple juice was added at three different points in the brewing process, (i) wort, (ii) toward the end of fermentation and (iii) at the beginning of maturation. The physicochemical properties, taste and aroma of the three fruit beers was compared to a control beer without addition of pineapple juice. The beers were analysed using an electronic nose (*E*-nose), head space solid phase micro-extraction gas chromatography-mass spectrometry (HS-SPME-GC-MS) and head space gas chromatography-ion mobility spectrometry (HS-GC-IMS). Of the three fruit beers, esters characteristic of pineapple aroma was found in the beer produced with juice added late during primary fermentation. Further, the abundance of esters-isoamyl acetate, ethyl butyrate, ethyl acetate, ethyl hexanoate and phenethyl acetate-was higher. Sensory analysis showed the beer produced from the addition of juice late in fermentation had a superior aroma and was the most preferred in sensory testing. These results provide technical support for process optimisation and for improving the aroma of fruit beers.

**Keywords:**

Addition time; pineapple juice; fruit beer; volatile organic compounds; aroma profile.

# Introduction

Fruit beer is produced by adding fruits, juice of flavourings to wort for fermentation or adding juice to green beer or during secondary fermentation. Therefore, fruit beers have the characteristics of fruit, such as colour, taste and aroma and tend to have more bioactive compounds (Baigts Allende et al, 2021). In recent years, fruit beers have become more popular due to their sensory quality and nutrition together with the consumer demand and enhanced awareness of nutrition and health. As a result, various fruit beers, such as banana (Carvalho et al, 2009), Saskatoon (Gorzelany et al, 2022), cranberry (Yin et al, 2021) have emerged into the market.

Research on fruit beer focuses on the brewing process, physicochemical characterisation, antioxidant properties and aroma analysis. The antioxidant properties and aroma of fruit beer are associated with the variety (Nardini and Garaguso, 2020) and form at of the fruit (juice, pulp, whole fruit etc) Gasiński et al, 2020) together with the point of addition. da Silva Santos et al (2021) prepared beer with soursop (*Annona muricata* L.) pulp and compared the physicochemical properties and sensory quality of fruit beer with different addition time. Kawa Rygielska et al (2019) reported that beer brewed by adding red cherry juice after fermentation showed the best antioxidant properties. Conversely, Ducruet et al (2017) found that beer with goji berries added at the beginning of fermentation was more popular with consumers and had higher antioxidant activity and more bioactive substances. Our previous work (Gong et al, 2022) with pineapple beer prepared with juice concentrate, reported the physicochemical properties, antioxidant activity and aroma changes during the brewing process.

This paper extends the report of Gong et al, (2022) and explores the point of juice addition on the flavour profile of pineapple beer using sensory evaluation, *E*-nose analysis, gas chromatography-mass spectrometry (GC-MS) and gas-ion mobility spectrometry (GC-IMS).

# Materials and Methods

## Materials and reagents

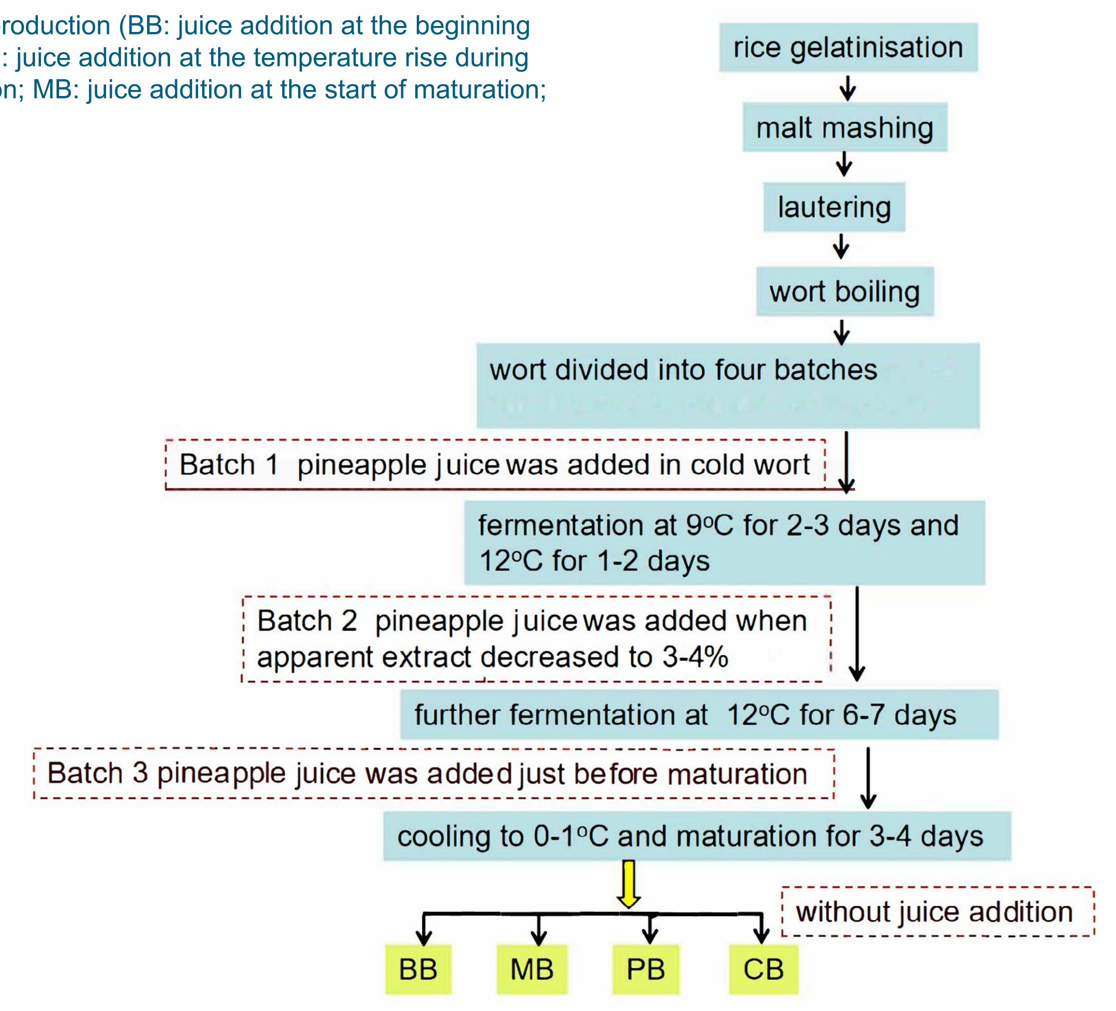
Pilsner malt and rice were from Shun Taimai bud Group (Guangzhou, China) and Sweet Fa Yan Industry (Guangzhou, China). Concentrated pineapple juice with an initial sugar concentration of ~60 Brix was supplied by Guangdong Harvest Canned Foods (Zhanjiang, China). Cascade and Tsingdao flower hops were purchased from YCH (Singapore) and Gansu Tianma Hops (Jiuquan, China). *Saccharomyces cerevisiae* W34/70 (lager yeast) was provided by the Technical University of Munich (Munich, Germany). The n-alkanes ($C_7$-$C_{30}$) were purchased from Sigma Chemical Co., Ltd. and 4-methyl-2-pentanol used as internal standard was purchased from Augsberg, Germany.

## Brewing process

The brewing process was as reported previously Gong et al, 2022). Rice (30%) was gelatinised (mashing temperature 70ºC, raised to 75ºC in 5 min and held for 10 min, and raised to 95ºC at a rate of 1ºC/min to 95ºC and held for 35 min), transferred to the mash vessel and mixed with the malt mash (48ºC). Mashing was performed as follows; the temperature was raised to 69ºC at a rate of 1ºC /min and held for 30 min and raised to 73ºC at the same heating rate and held for 30 min, then raised to 78ºC for lautering without sparging. The wort was diluted to 7.2°P and boiled for 60 min. Tsingdao flower hop (0.14 g/L) and Cascade hops (0.18 g/L) were added after 46 min and 59 min, respectively. After boiling, the wort was clarified and cooled to 10 -15ºC, and 20L wort transferred to 30 L stainless steel tanks. The yeast *Saccharomyces cerevisiae* W34/70 was inoculated at $1.0 \times 10^7$ CFU/ml, (96% viability). Fermentation was initially at 9ºC with a temperature step at the mid point to 12ºC. Maturation was at 0-1ºC. Four beers were prepared (i), beer with pineapple juice added to wort at the beginning of fermentation (BB), (ii) beer with juice addition after the temperature rise (AE = 3-4%) during fermentation (PB), (iii) beer with pineapple juice added at start of maturation (MB) and control beer without any juice addition (CB). Pineapple juice concentrate (100 g/L) was added to beers (i) to (iii) and the flowchart of the beer production process is shown in Figure 1. The wort original extract was 7.6 °P, bitterness 11.5 IBU, pH 5.50, and colour 6.3 EBC.

**Figure 1.**

Flowchart of beer production (BB: juice addition at the beginning of fermentation; PB: juice addition at the temperature rise during primary fermentation; MB: juice addition at the start of maturation; CB: control beer).




## Sensory evaluation

Sensory evaluation was performed by a trained panel of 10 panelists (six women and four men, 30-45 years old). The panelists were experienced (3-8 years)and were qualified to Chinese national beer tasting level 2 and above. Beer samples at 10ºC were coded, and 50 ml beer added to clear plastic cups (capacity of 150 ml) in a random order. The intensity of each sensory attribute was graded from 0 (none) to 5 (very strong) and the average value determined. In the first assessment, odour (malty aroma, hop aroma and pineapple flavour), mouth feel (freshness and fullness), and taste (sweetness, sourness and bitterness) were evaluated. Overall preference was determined in the second evaluation.

## *E*-nose analysis

A PEN3 electronic nose was used in this work. The sensor array consists of 10 metal oxide semiconductors and the characteristics are described in Table 1. Samples were cooled to 4ºC and beer (10 ml) was pipetted into 25ml vials capped with a PTFE silicon stopper and equilibrated at room temperature for 15 min. The *E*-nose analysis was conducted according to Chen et al, (2018) with modifications. The sensor cleaning and automatic zero adjustment time was 180s and 1s, respectively. Pre-sampling lasted 5s. Internal flow rates and the inlet were 300 mL/min. The detection time was 80s, and each sample was measured in triplicate.

**Table 1.**

PEN3 *E* - nose sensor array and main characteristics

| No | name | analyte | detection limit (mg/L) |
|---|---|---|---|
| 1 | W1C | Aromatic compounds | 10 |
| 2 | W5S | Oxynitride | 1 |
| 3 | W3C | Aromatic constituent, mainly ammonia | 10 |
| 4 | W6S | Hydrogen | 0.1 |
| 5 | W5C | Alkanes, aroma-aliphatic compounds | 1 |
| 6 | W1S | Broad methane | 100 |
| 7 | W1W | Sulphides and organic sulphides | 1 |
| 8 | W2S | Broad alcohols | 100 |
| 9 | W2W | Aromatics, organic sulphides | 1 |
| 10 | W3S | Alkanes, especially methane | 10 |

## Volatile analysis

### Headspace solid-phase microextraction, gas chromatography–mass spectrometry (HS-SPME-GC-MS)

A DVB/CAR/PDMS (divinylbenzene/carboxen/polydimethylsiloxane) fibre was used. Samples were cooled to 4°C for volatile analysis and beer (8 mL) transferred to a 20 ml head space vial containing sodium chloride (2g), and (internal standard) 30 µL 4-methyl-2-pentanol (8.23 mg/mL), with the sealed with a PTFE (polytetrafluoroethylene) septum. The vial was equilibrated in thermostatic bath with continuous agitation (500 rpm) at 45°C for 1 hour and the fibre exposed to the head space at 40°C for 30 min. After extraction, the fibre was inserted in the injection port and desorbed at 250°C for 8 min. A VF-WAX (30m × 0.25 mm × 0.25 µm) capillary column was used. The oven temperature programme was: 40°C for 2 min, and raised at a rate of 5°C/min to 140°C, and then heated at a rate of 7°C/min to a final temperature of 220°C and held for 3 min. The carrier gas was helium at a flow rate of 1 ml/min with the splitless mode. The transmission line was heated to 250°C. The MS was set to EI mode with 70 eV and scans 30-500 amu with 6 min solvent delay. Compounds were identified by retention index (RI), matching the recorded mass spectra in the NIST 11 library and by comparison of the linear retention indices (LRI), from running C7-C30 n-alkanes under the same chromatographic conditions. The content of each compound was calculated by comparing the area with that of the internal standard 4-methyl-2-pentanol. Each sample was measured in triplicate.

### Headspace-gas chromatography-ion mobility spectroscopy (HS-GC-IMS)

Beer samples were cooled to 4°C for analysis. SH-GC-IMS analysis was conducted according to the methods described previously (Gong et al, 2022). Quantification using an internal standard with 20 mg/L 4-methyl-2-pentanol (10 µL was added in the sample).

### Statistical analysis

All measurements were performed in triplicate and statistical significance ($p < 0.05$) was analysed using Duncan's test with the software SPSS 18.0 (SPSS Inc., Chicago, USA). The data were presented as the mean ± standard deviation of three replicates.

## Results and discussion

### Sensory evaluation

Sensory attributes determine the quality of beer. Accordingly, the sensory quality of different pineapple beers was evaluated (Figure 2), together with beer composition (Table 2). BB (juice added at start of fermentation) exhibited weak fresh pineapple aroma, prominent sourness and a strong alcohol note (alcohol 5.95% v/v) due to the presence of fruit juice throughout fermentation (real attenuation 74.2%); the pineapple beer with fruit juice added at start of maturation (MB) had pronounced sweetness, relatively weak sourness

**Figure 2.**

Sensory profile of pineapple beers (BB: juice addition at the beginning of fermentation; PB: juice addition at temperature raise during primary fermentation; MB: juice addition at start of maturation).

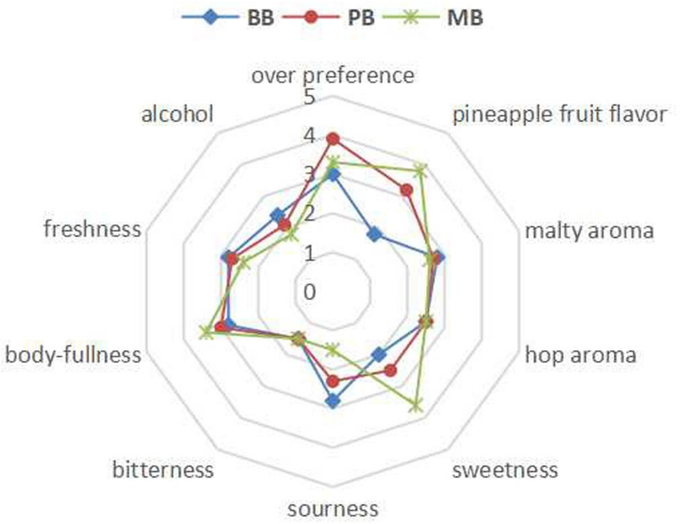



**Figure 3.**

Radar map of *E*-nose's sensors to pineapple beer (PB) and the control beer (CB).

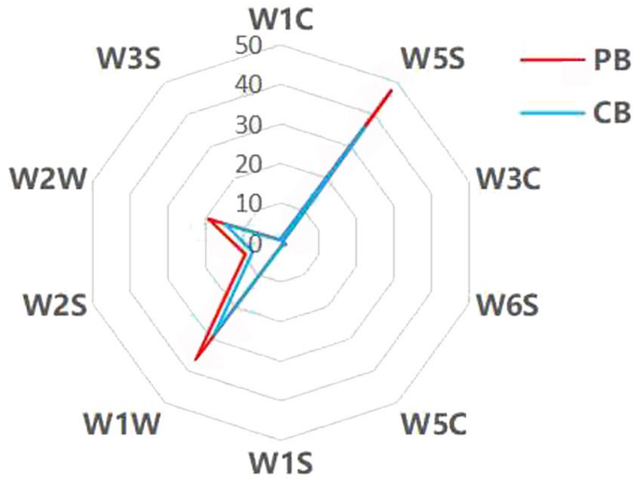



**Table 2.**

Point of juice addition and the physiochemical properties of pineapple beer.

| Beer | Gravity (°P) | pH | colour (EBC) | real attenuation (%) | apparent extract (%) | alcohol % v/v | total acid (mg/100 mL) |
|---|---|---|---|---|---|---|---|
| BB | 12.42 ± 0.09$^{a}$ | 4.26 ± 0.04$^{a}$ | 6.2 ± 0.05 | 74.2 ± 0.1$^{a}$ | 1.22 ± 0.06$^{c}$ | 5.95 ± 0.06$^{a}$ | 4.76 ± 0.15$^{b}$ |
| PB | 12.08 ± 0.08$^{b}$ | 4.35 ± 0.03$^{a}$ | 6.4 ± 0.18 | 60.5 ± 0.1$^{c}$ | 3.24 ± 0.06$^{b}$ | 4.71 ± 0.08$^{b}$ | 4.65 ± 0.08$^{b}$ |
| MB | 12.42 ± 0.12$^{ab}$ | 4.28 ± 0.04$^{a}$ | 6.7 ± 0.22 | 33.6 ± 0.2$^{d}$ | 7.47 ± 0.04$^{a}$ | 2.69 ± 0.06$^{c}$ | 4.76 ± 0.23$^{a}$ |
| CB | 7.39 ± 0.05$^{c}$ | 4.42 ± 0.04$^{a}$ | 5.9 ± 0.14 | 69.6 ± 0.1$^{b}$ | 1.50 ± 0.05$^{c}$ | 3.00 ± 0.05$^{c}$ | 1.80 ± 0.07$^{b}$ |

Means in the same line with different letters are significantly different ($p < 0.05$).

and little flavour due to limited involvement of fruit juice in fermentation (real attenuation 33.6%). PB (juice added after the temperature rise in fermentation) exhibited a fresh pineapple aroma with a balanced sweetness and sourness, and, of the three beers, was most preferred.

## *E*-nose analysis

Figure 3 shows the *E*-nose radar map of the pineapple beer with juice addition at the rise in temperature during primary fermentation (PB) and the control beer (CB). The volatile components of these two beers overlapped, with the signal of PB slightly stronger (particularly the oxynitride sensor) than that of the control beer. This shows that *E*-nose analysis was able to differentiate the two beers and that the pineapple juice contributed to aroma of the beer.

## Volatile analysis of pineapple beers brewed at a different point of juice addition GC-MS analysis

Volatile compounds were analysed using HS-SPME-GC-MS analysis. The identified compounds (matching degree > 85%) and concentration in the four beers are reported in Table 3, with the abundance of the main volatile families shown in Figure 4. In all 44 compounds were detected - 22 esters, 10 alcohols, four ketones, four acids, three terpenes and an aldehyde (Table 3). The content of ethyl hexanoate, ethyl octanoate, ethyl 9-decenoate, ethyl decanoate and isoamyl alcohol was high in all samples, with phenylethanol, isoamyl acetate, ethyl phenylacetate, and octanoic acid also relatively higher.

## Table 3.

Volatile compounds of pineapple beers with juice added at different times detected by GC-MS analysis.

| Compound | Concentration (mg/L) | | | | RI | |
|---|---|---|---|---|---|---|
| | BB | PB | MB | CB | $RI_{cal}$ | $RI_{ref}$ |
| **esters** | | | | | | |
| ethyl butyrate | 0.59 ± 0.04$^{d}$ | 0.58 ± 0.03$^{c}$ | 0.22 ± 0.01$^{d}$ | 0.34 ± 0.02$^{a}$ | / | 1026 |
| isoamyl acetate | 2.80 ± 0.12$^{d}$ | 4.67 ± 0.15$^{a}$ | 2.16 ± 0.08$^{b}$ | 2.33 ± 0.22$^{b}$ | 1119 | 1126 |
| ethyl hexanoate | 20.19 ± 0.55$^{c}$ | 17.46 ± 0.48$^{d}$ | 13.60 ± 0.32$^{b}$ | 3.88 ± 0.24$^{c}$ | 1230 | 1220 |
| hexyl acetate | 0.14 ± 0.02$^{e}$ | 0.29 ± 0.06$^{d}$ | 0.13 ± 0.02$^{d}$ | 0.17 ± 0.04$^{c}$ | 1269 | 1276 |
| ethyl heptanoate | 0.84 ± 0.15$^{a}$ | 0.53 ± 0.12$^{a}$ | 0.92 ± 0.21$^{d}$ | 0.21 ± 0.06$^{a}$ | 1329 | 1332 |
| heptyl acetate | 0.13 ± 0.02$^{c}$ | 0.15 ± 0.02$^{d}$ | 0.06 ± 0.01$^{b}$ | 0.04 ± 0.01$^{b}$ | 1370 | 1377 |
| ethyl octanoate | 66.86 ± 0.72$^{d}$ | 67.53 ± 0.68$^{e}$ | 70.14 ± 0.75$^{d}$ | 45.74 ± 0.62$^{c}$ | 1444 | 1420 |
| ethyl (*Z*)-4-decenoate | 0.13 ± 0.02$^{e}$ | 0.10 ± 0.02$^{b}$ | 0.32 ± 0.12$^{b}$ | 0.11 ± 0.03$^{d}$ | 1584 | / |
| ethyl (*Z*)-4-octenoate | 1.15 ± 0.12$^{d}$ | 0.69 ± 0.08$^{c}$ | 1.14 ± 0.18$^{d}$ | nd | 1475 | / |
| isoamyl hexanoate | 0.50 ± 0.14$^{a}$ | 0.33 ± 0.06$^{a}$ | 1.20 ± 0.04$^{a}$ | nd | 1456 | 1450 |
| propyl octanoate | 0.11 ± 0.02$^{d}$ | 0.18 ± 0.04$^{b}$ | 0.09 ± 0.02$^{e}$ | 0.07 ± 0.01$^{b}$ | 1517 | 1526 |
| ethyl nonanoate | 0.54 ± 0.07$^{c}$ | 0.46 ± 0.06$^{e}$ | 0.54 ± 0.08$^{c}$ | 0.48 ± 0.06$^{d}$ | 1534 | 1528 |
| ethyl 3-methylthiopropionate | 0.16 ± 0.04$^{b}$ | 0.09 ± 0.03$^{b}$ | 0.06 ± 0.03$^{c}$ | nd | 1567 | 1560 |
| ethyl decanoate | 3.58 ± 0.24$^{b}$ | 9.37 ± 0.52$^{d}$ | 9.16 ± 0.44$^{c}$ | 9.39 ± 0.38$^{e}$ | 1641 | 1633 |
| isoamyl octanoate | 1.33 ± 0.14$^{d}$ | 0.91 ± 0.09$^{b}$ | 1.15 ± 0.21$^{a}$ | 0.81 ± 0.10$^{d}$ | 1659 | 1651 |
| ethyl 9-decenoate | 15.30 ± 0.31$^{c}$ | 15.47 ± 0.54$^{d}$ | 16.02 ± 0.40$^{a}$ | 19.55 ± 0.47$^{e}$ | 1698 | 1708 |
| geranyl acetate | nd | 0.06 ± 0.01$^{d}$ | 0.12 ± 0.02$^{d}$ | 0.07 ± 0.01$^{a}$ | 1759 | 1760 |
| phenethyl acetate | 2.00 ± 0.08$^{d}$ | 2.67 ± 0.10$^{c}$ | 1.86 ± 0.14$^{d}$ | 1.83 ± 0.09$^{b}$ | 1820 | 1825 |
| phenethyl butyrate | nd | 0.03 ± 0.01$^{d}$ | 0.06 ± 0.01$^{b}$ | 0.03 ± 0.01$^{c}$ | 1962 | 1958 |
| ethyl laurate | nd | 0.58 ± 0.17$^{a}$ | nd | nd | 1839 | 1849 |
| ethyl myristate | nd | 0.09 ± 0.01$^{a}$ | 0.10 ± 0.02$^{d}$ | 1.29 ± 0.14$^{c}$ | 2043 | 2054 |
| γ-undecalactone | 0.10 ± 0.02$^{a}$ | 0.14 ± 0.03$^{d}$ | 0.09 ± 0.01$^{d}$ | nd | 2033 | / |
| **alcohols** | | | | | | |
| ethanol | 0.50 ± 0.11$^{c}$ | 0.39 ± 0.07$^{a}$ | 0.26 ± 0.04$^{d}$ | 0.33 ± 0.06$^{a}$ | / | 934 |
| hexanol | 0.08 ± 0.02$^{d}$ | nd | nd | 0.11 ± 0.02$^{b}$ | 1358 | 1359 |
| propyl alcohol | 0.87 ± 0.04$^{d}$ | 0.58 ± 0.06$^{b}$ | 0.30 ± 0.02$^{b}$ | 0.35 ± 0.03$^{c}$ | / | 993 |
| isobutyl alcohol | 0.88 ± 0.06$^{a}$ | 0.73 ± 0.10$^{d}$ | 0.47 ± 0.04$^{b}$ | 0.45 ± 0.05$^{d}$ | 1104 | 1108 |
| isoamyl alcohol | 21.17 ± 0.84$^{d}$ | 17.15 ± 0.65$^{c}$ | 11.57 ± 0.33$^{c}$ | 12.58 ± 0.24$^{e}$ | 1223 | 1201 |
| octanol | 0.78 ± 0.04$^{e}$ | 0.57 ± 0.07$^{d}$ | 0.56 ± 0.05$^{d}$ | 0.93 ± 0.14$^{b}$ | 1561 | 1557 |
| methionol | nd | 0.18 ± 0.03$^{a}$ | nd | nd | 1724 | 1722 |
| decanol | 1.00 ± 0.08$^{c}$ | 0.28 ± 0.04$^{a}$ | 0.25 ± 0.02$^{d}$ | 0.96 ± 0.11$^{b}$ | 1768 | 1778 |
| phenethyl alcohol | 12.61 ± 0.24$^{c}$ | 11.85 ± 0.36$^{b}$ | 5.82 ± 0.11$^{c}$ | 7.61 ± 0.17$^{d}$ | 1921 | 1923 |
| 2,3-dimethyl-1-butanol | nd | nd | 0.48 ± 0.09$^{d}$ | nd | / | 1290 |
| **acids** | | | | | | |
| isovaleric acid | 0.16 ± 0.03$^{a}$ | 0.10 ± 0.02$^{c}$ | 0.11 ± 0.01$^{b}$ | 0.06 ± 0.01$^{d}$ | 1672 | 1660 |
| hexanoic acid | 1.56 ± 0.09$^{b}$ | 1.86 ± 0.10$^{a}$ | 1.80 ± 0.11$^{a}$ | 1.04 ± 0.09$^{b}$ | 1847 | 1847 |
| octanoic acid | 2.67 ± 0.34$^{b}$ | 3.61 ± 0.21$^{b}$ | 5.10 ± 0.09$^{c}$ | 2.58 ± 0.11$^{d}$ | 2061 | 2073 |
| acetic acid | 1.21 ± 0.05$^{a}$ | 1.24 ± 0.04$^{a}$ | 0.83 ± 0.02$^{d}$ | 0.61 ± 0.02$^{c}$ | 1459 | 1453 |
| **Terpenes** | | | | | | |
| linalool | 0.48 ± 0.24$^{d}$ | 0.51 ± 0.04$^{d}$ | 0.32 ± 0.03$^{c}$ | 0.37 ± 0.03$^{a}$ | 1547 | 1552 |
| citronellol | 0.46 ± 0.04$^{d}$ | 0.43 ± 0.03$^{a}$ | 0.31 ± 0.03$^{a}$ | 0.54 ± 0.05$^{c}$ | 1772 | 1767 |
| 1,8-Cineole | 0.28 ± 0.02$^{c}$ | 0.01 ± 0.00$^{a}$ | 0.32 ± 0.04$^{c}$ | 0.17 ± 0.02$^{d}$ | 1193 | 1204 |
| **Ketones** | | | | | | |
| cognac heptanone | 1.59 ± 0.04$^{e}$ | 2.06 ± 0.07$^{d}$ | 1.76 ± 0.03$^{c}$ | 1.83 ± 0.05$^{b}$ | 1163 | 1178 |
| 4,6-Dimethyl-2-heptanone | 0.20 ± 0.02$^{e}$ | 0.41 ± 0.05$^{a}$ | 0.32 ± 0.03$^{e}$ | 0.33 ± 0.03$^{b}$ | 1237 | 1262 |
| isobutenyl methyl ketone | 0.09 ± 0.01$^{c}$ | 0.12 ± 0.04$^{d}$ | nd | 0.12 ± 0.02$^{d}$ | 1128 | 1113 |
| strawberry furanone | 0.21 ± 0.02$^{c}$ | 0.25 ± 0.03$^{d}$ | 0.35 ± 0.05$^{a}$ | nd | 2041 | 2034 |
| **aldehydes** | | | | | 2041 | 2034 |
| benzaldehyde | nd | 0.14 ± 0.02$^{b}$ | 0.10 ± 0.03$^{b}$ | nd | 1524 | 1522 |

nd: not detected.

/: cannot be calculated or referenced.

BB: juice addition at the beginning of fermentation; PB: juice addition at temperature rise during primary fermentation; MB: juice addition at start of maturation; CB: the control beer.

$RI_{cal}$ - Retention index calculated by running $C_7$-$C_{30}$ n-alkanes under the same chromatographic conditions.

$RI_{ref}$ - Retention index with the reference data.

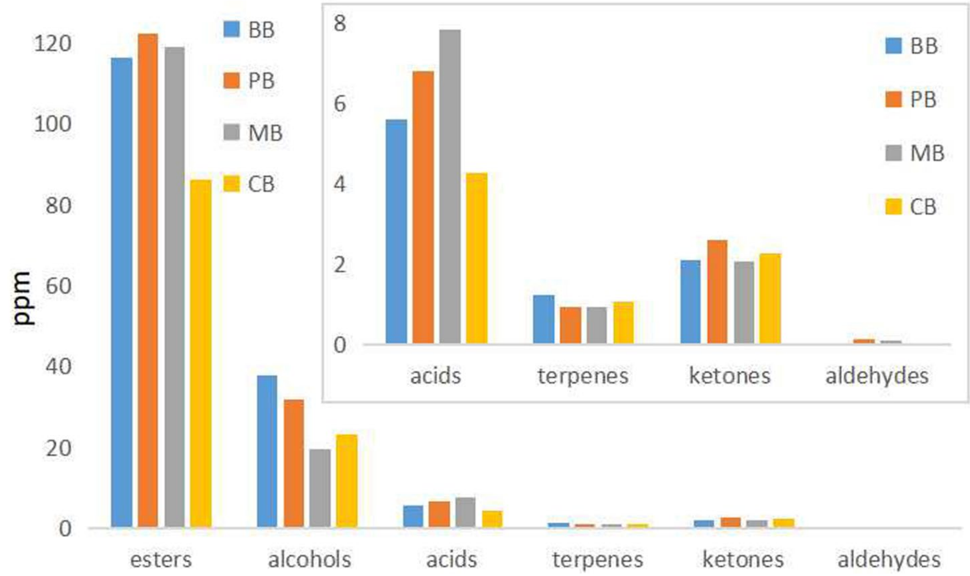



**Figure 4.**

Abundance of the main volatiles families in pineapple beers (BB: juice addition at the beginning of fermentation; PB: juice addition at the temperature rise during primary fermentation; MB: juice addition at start of maturation; CB: control beer).

Compared with the control beer (CB), the concentration of ethyl hexanoate, ethyl heptanoate, ethyl octanoate, acetic acid and hexanoic acid were enhanced in all the pineapple beers. Ethyl hexanoate and ethyl heptanoate contribute a pineapple fruit flavour, and a pineapple wine/ liquor-brandy note (Surburg and Panten, 2006). Further, ethyl hexanoate was reported to be the characteristic aroma compound of fresh pineapple pulp and core (Wei et al, 2011). Volatile fatty acids such as acetic acid and hexanoic acid are the main sources of organic acids in fruit juice itself and are also produced by fermentation. These two acids contribute to the sourness of fruit beer. In addition, the content of ethyl butyrate, isoamyl acetate, ethyl (Z)-4-octenoate, isoamyl hexanoate, isoamyl alcohol, isobutanol and phenylethanol in BB and PB were also significantly higher compared with MB, suggesting that these compounds were enriched when the juice was involved in fermentation. Isoamyl alcohol and isobutanol are responsible for the malty and burned flavour (Christoph and Bauer-Christoph, 2007); phenylethanol presents a rose flavour and contributes to the flowery flavour of beer. Ethyl butyrate has a typically fruity flavour, somewhat like that of pineapple (Zhu and Yu, 2020); and isoamyl acetate presents a typical banana aroma.

Ethyl (Z)-4-octenoate, isoamyl hexanoate, ethyl 3-methylthiopropionate, γ-undecalactone and 2,5-dimethyl-4-hydroxydihydrofuran-3-ketone (strawberry furanone) were detected in the pineapple beers but not in the control beer. Ethyl 3-methylthiopropionate, also known as pineapple ethyl ester (pineapple-like and citrus-like), is the characteristic odour compound of pineapple. A fruity, peach-like aroma are the odour characteristics of γ-undecalactone, which is often selected to enrich the fruity note of pineapple (Zhu and Yu, 2020). The pineapple/caramel flavour characteristic of 2,5-dimethyl-4-hydroxydihydrofuran-3-ketone has been reported in pineapple juice by GC-MS (Holt et al, 2019), and suggested to contribute to the aroma of fresh pineapple (Takeoka et al, 1989). Wei et al (2011) detected ethyl (Z)-4-octenoate in both pineapple pulp and core by GC-MS, and isoamyl hexanoate has a fruity flavour of banana and pineapple (Antalick et al, 2010).

The beer brewed with juice added at start of fermentation (BB) had a relatively higher content of alcohols, such as ethanol, n-propanol, isobutanol, isoamyl alcohol, 1-octanol, 1-decanol, which reduce drinkability and contribute negatively to consumer acceptance (Alves et al, 2020). These alcohols are the most abundant in beer BB (Figure 4), which agrees with the sensory evaluation of a strong alcohol note. Beer PB had the highest content of esters, including isoamyl acetate, hexyl acetate, propyl octanoate, ethyl phenylacetate, ethyl laurate. These esters present a fruity aroma and have a positive contribution to the flavour of fruit beer. However, beer MB had a lower content of ethyl butyrate, isoamyl acetate, ethyl hexanoate and ethyl phenylacetate compared with both BB and PB, which confirmed that these esters were mainly produced during fermentation when the juice was utilised by yeasts.

Pineapple juice contains a considerable amount of sugar which was metabolised by yeast during fermentation to produce alcohol and other by-products. Further, some volatile components contributing positively to pineapple aroma will have been converted to other products or lost by gas washing. Accordingly, the more juice involved in fermentation, the higher the alcohol concentration and the less apparent the pineapple aroma in the final beer. Therefore, when fruit juice was present throughout fermentation (beer BB) this led to the biggest loss or transformation of aroma volatiles, with the beer having the highest real attenuation, but the weakest pineapple aroma. When the juice was added at start of maturation, beer MB was more of a fruity juice drink due to the limited fermentation (real attenuation 33.6%) and the abundance of the juice (apparent extract 7.47%). Reduced exposure during fermentation (beer PB) was fermented to a moderate extent (real attenuation 60.5%) with more esters and sugar (apparent extract 3.24%) left in the beer. As a result, PB exhibited the most obvious pineapple aroma and a balanced sweetness and sourness.

In conclusion, GC-MS analyses showed that beer PB had the most abundant esters with the characteristic odour of fresh pineapple fruit, and least prominent content of alcohols. Accordingly, the aroma of PB was coordinated and balanced, which is consistent with the overall preference by sensory evaluation.

## HS-GC-IMS analysis

Headspace-gas chromatography-ion mobility spectroscopy is an effective tool to discriminate fruit beers and to assess food authenticity which has been widely used to determine volatile profiles combined with GC-MS and other techniques (Yang et al, 2022; Zhang and Abdulla, 2022; Segura-Borrego et al, 2022). Figure 5 shows the 3D visualisation and top view plots of the GC-IMS spectrum of pineapple beers brewed with juice addition at different times. Compared with the control beer (CB), the GC-IMS spectrum (Table 4) of pineapple beers was more complex. Both the number of volatiles and the abundance of most compounds increased significantly, indicating that the involvement of pineapple juice in fermentation can enrich flavour volatiles and contribute to aroma. This was consistent with the results from the *E*-nose and GC-MS analyses.

Figure 6 shows the non-targeted fingerprints by selecting signal peaks on the specific spectrum. The abundance of isoamyl acetate, ethyl acetate, acetic acid, isoamyl alcohol, isobutanol, ethanol in all samples was high and is consistent with their contribution to beer flavour and aroma in beer (Holt et al, 2019). Isoamyl acetate (banana, ester, fruit flavour) and ethyl acetate (solvent-like, fruity, sweet) contribute fruity flavours (Holt et al, 2019) whereas higher alcohols (isoamyl alcohol and isobutanol) influence the malty and burnt notes (Christoph and Bauer-Christoph, 2007). Acetic acid (vinegar note) provides sourness, enhances the fruity flavour (Zhu and Yu, 2020) and balances the sweet note of pineapple.

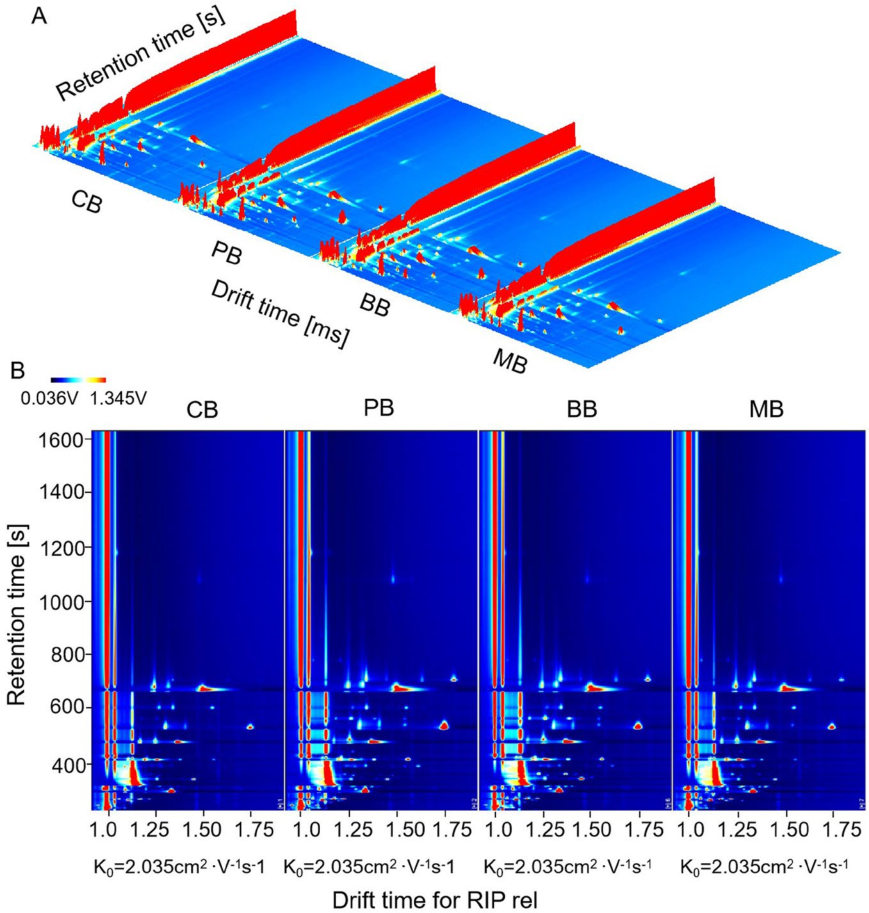
Flavour compounds in pineapple beers increased significantly compared with the control beer. The greatest number of volatiles were in the beer brewed with the juice added late in primary fermentation (PB), followed by juice added at the beginning of fermentation (BB), with the least when added during maturation (MB). The concentration of isoamyl acetate, ethyl butyrate, ethyl acetate, ethyl hexanoate, isobutyl acetate, methyl 3-methylbutyrate and nonal exhibited the most obvious increase in beer PB. Of these, isoamyl acetate (banana, fruity note), ethyl butyrate (fruity) and ethyl acetate (ester, solvent flavour) all contribute fruity flavour and enrich the aroma of fresh pineapple (Zhu and Yu, 2020). Further, isobutyl acetate - found in pineapples, raspberries and pears – contributes a fruity character to beer (Park et al, 2009). Other characteristic volatiles in PB included propyl acetate, isobutyl acetate, isobutyl propionate, 2, 5-dimethylpyrazine and unidentified compounds (4, 6 - Table 4).

The beer brewed with juice added to wort at the start of fermentation (BB) showed enhanced abundance in 1-propanol, 1-butanol, ethyl propionate and 2-pentanone, and the characteristic aroma compounds were 1-butanol, ethyl 2-methylbutyrate, ethyl propionate and 2-pentanone.

Compared with beers BB and PB, volatile compounds in the beer with juice added at the start of maturation (MB) are fewer, and the concentration of isoamyl acetate, ethyl acetate, ethyl hexanoate, ethyl butyrate and acetic acid was lower due to the limited fermentation at 0-1°C when the juice was added at start of maturation. This agrees with the

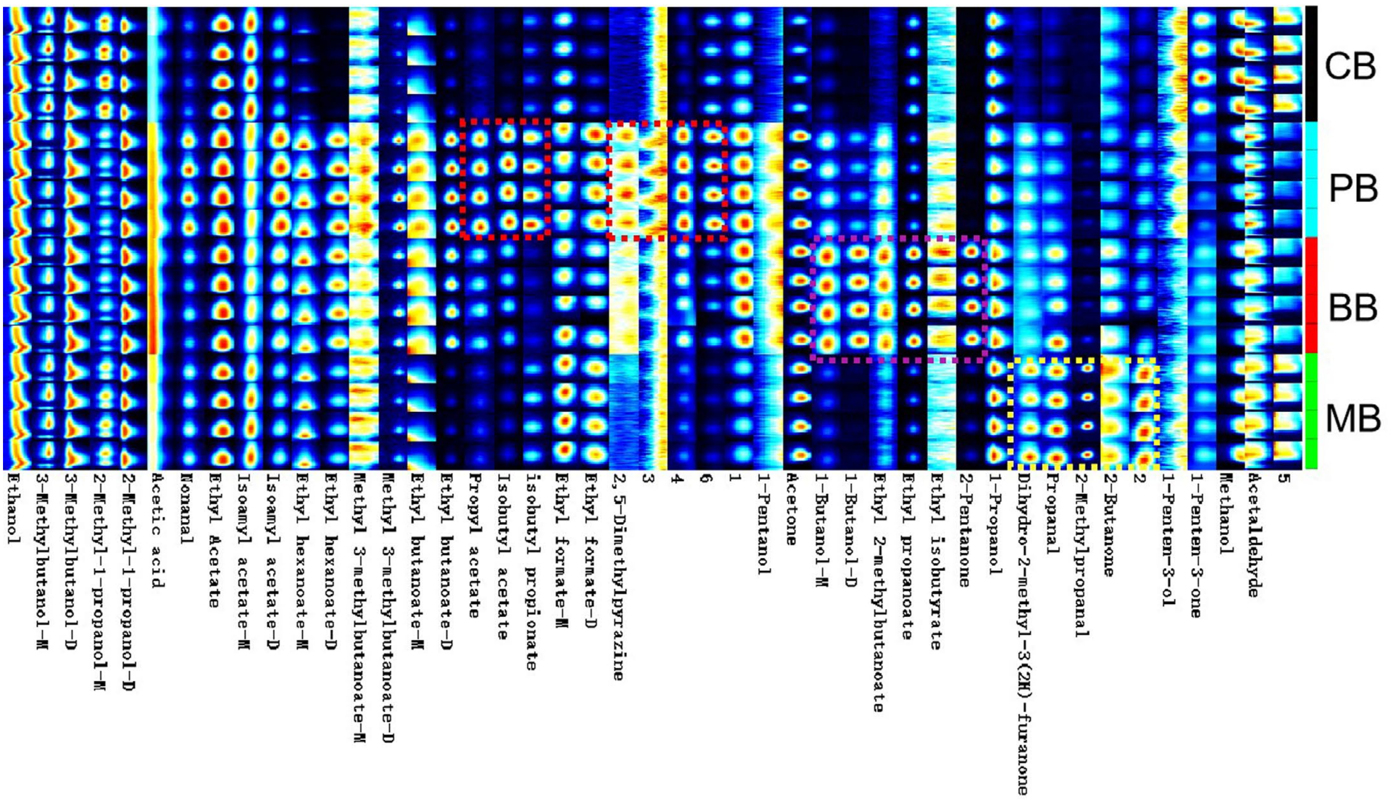
## Figure 5.

GC-IMS spectrum of pineapple beer samples. A - 3D plot, B - top view plot (CB: control beer; BB: juice addition at the beginning of fermentation; PB: juice addition at temperature rise during primary fermentation; MB: juice addition at start of maturation). RIPrel - relative ion peak, K0 - ion mobility.




## Figure 6.

GC-IMS fingerprints of pineapple beers with juice added at different times (CB: control beer; BB: juice addition at the beginning of fermentation; PB: juice addition at the temperature rise during primary fermentation; MB: juice addition at start of maturation).



## Table 4.

GC-IMS analysis of pineapple beers with juice added at different times.

| No | Compound | RI | Rt [sec] | Dt [RIPrel] | Concentration （μg/kg） BB | PB | MB | CB |
|---|---|---|---|---|---|---|---|---|
| 1 | Acetic acid | 1445.6 | 1181.79 | 1.05337 | 758.0 ± 25.93[d] | 761.72 ± 70.55[c] | 610.83 ± 34.60[a] | 504.69 ± 36.26[c] |
| 2 | Nonanal | 1402.5 | 1080.97 | 1.48045 | 151.61 ± 18.61[c] | 261.20 ± 7.76[a] | 182.63 ± 7.95[c] | 100.11 ± 6.98[d] |
| 3 | Dihydro-2-methyl-3(2H)-furanone | 1270.1 | 785.94 | 1.06896 | 40.22 ± 3.88[d] | 48.19 ± 2.37[b] | 69.69 ± 5.09[d] | 24.92 ± 3.47[c] |
| 4 | Ethyl hexanoate-M | 1227.9 | 711.52 | 1.3417 | 596.88 ± 23.95[d] | 631.87 ± 9.86[b] | 505.11 ± 12.92[c] | 149.08 ± 8.73[d] |
| 5 | Ethyl hexanoate-D | 1227.4 | 710.69 | 1.79874 | 447.33 ± 32.14[d] | 481.80 ± 16.92[a] | 216.19 ± 4.95[b] | 22.57 ± 2.57[a] |
| 6 | 3-Methylbutanol-M | 1208.6 | 677.40 | 1.2422 | 762.26 ± 4.85[b] | 772.34 ± 19.88[b] | 900.32 ± 30.52[d] | 952.82 ± 17.02[d] |
| 7 | 3-Methylbutanol-D | 1208.6 | 677.40 | 1.48843 | 3622.21 ± 45.18[d] | 3509.01 ± 29.53[c] | 3142.38 ± 21.16[d] | 2656.70 ± 64.87[e] |
| 8 | 1-Butanol-M | 1148.1 | 568.35 | 1.1798 | 121.78 ± 1.93[d] | 73.36 ± 2.50[c] | 33.64 ± 1.12[e] | 23.76 ± 1.79[c] |
| 9 | Isoamyl acetate-M | 1128.9 | 533.39 | 1.29954 | 507.36 ± 10.80[a] | 530.17 ± 14.42[d] | 536.83 ± 15.22[d] | 538.34 ± 8.55[a] |
| 10 | Isoamyl acetate-D | 1128.4 | 532.56 | 1.73971 | 1507.56 ± 36.77[a] | 2349.66 ± 21.10[d] | 940.87 ± 15.01[d] | 692.89 ± 42.22[d] |
| 11 | 1-Penten-3-ol | 1165.1 | 599.15 | 0.93864 | 10.76 ± 0.28[d] | 13.80 ± 0.43[c] | 12.79 ± 0.81[c] | 15.42 ± 0.66[b] |
| 12 | 2-Methyl-1-propanol-M | 1099.6 | 480.12 | 1.17137 | 308.70 ± 10.35[c] | 303.95 ± 9.08[d] | 386.14 ± 8.11[c] | 403.96 ± 6.14[b] |
| 13 | 2-Methyl-1-propanol-D | 1100.5 | 481.78 | 1.36869 | 1808.50 ± 22.53[c] | 1707.90 ± 10.07[b] | 1419.20 ± 13.80[d] | 1035.22 ± 33.92[b] |
| 14 | Ethyl 2-methylbutanoate | 1061.1 | 436.00 | 1.24558 | 23.99 ± 2.22[a] | 19.83 ± 0.69[a] | 12.64 ± 1.88[d] | 7.47 ± 0.53[e] |
| 15 | Ethyl butanoate-M | 1043.9 | 417.25 | 1.20708 | 122.48 ± 3.60[b] | 125.86 ± 4.20[b] | 64.28 ± 0.82[e] | 67.32 ± 2.41[d] |
| 16 | Ethyl butanoate-D | 1043.9 | 417.25 | 1.56241 | 312.44 ± 9.66[c] | 398.92 ± 11.22[c] | 71.11 ± 1.81[a] | 81.47 ± 4.06[b] |
| 17 | Methyl 3-methylbutanoate-M | 1018.2 | 389.07 | 1.19593 | 51.44 ± 3.21[b] | 65.84 ± 1.66[b] | 59.53 ± 1.88[b] | 53.59 ± 2.42[d] |
| 18 | Methyl 3-methylbutanoate-D | 1018.2 | 389.07 | 1.53036 | 52.39 ± 1.31[a] | 67.82 ± 2.43[c] | 43.16 ± 2.86[b] | 35.00 ± 3.04[c] |
| 19 | Propyl acetate | 984.4 | 356.79 | 1.47602 | 19.29 ± 0.99[b] | 28.24 ± 0.45[d] | 9.84 ± 1.12[b] | 7.42 ± 0.49[e] |
| 20 | Ethyl isobutyrate | 973.3 | 349.16 | 1.56102 | 4.59 ± 0.60[e] | 2.19 ± 0.22[d] | 1.80 ± 0.15[c] | 1.80 ± 0.14[a] |
| 21 | Ethyl propanoate | 963.8 | 342.70 | 1.44675 | 495.42 ± 8.48[b] | 145.98 ± 4.61[d] | 129.16 ± 4.15[d] | 202.78 ± 11.61[c] |
| 22 | Ethanol | 936.5 | 323.92 | 1.13323 | 23140.29 ± 41.02[a] | 23127.08 ± 38.96[a] | 23191.01 ± 50.38[b] | 23204.55 ±38.31[b] |
| 23 | Ethyl Acetate | 896.9 | 296.78 | 1.33416 | 2171.72 ± 3.15[c] | 2407.33 ± 9.65[b] | 1834.70 ± 4.29[c] | 1802.14 ± 30.81[d] |
| 24 | 2-Methylpropanal | 848.9 | 263.88 | 1.27887 | 12.72 ± 1.13[a] | 13.72 ± 0.66[e] | 31.52 ± 1.77[d] | 7.58 ± 1.47[a] |
| 25 | Acetone | 850.2 | 264.80 | 1.10657 | 680.73 ± 10.09[b] | 643.06 ± 11.69[b] | 639.70 ± 3.22[c] | 323.88 ± 20.92[d] |
| 26 | Propanal | 838.9 | 257.03 | 1.06285 | 52.91 ± 10.90[b] | 33.48 ± 2.84[d] | 70.26 ± 2.13[d] | 15.97 ± 0.48[c] |
| 27 | Acetaldehyde | 811.5 | 238.29 | 1.01913 | 416.42 ± 35.53[d] | 345.05 ± 21.52[e] | 466.72 ± 8.48[a] | 304.33 ± 7.40[d] |
| 28 | Methanol | 914.2 | 308.66 | 0.98313 | 228.66 ± 22.05[d] | 210.22 ± 19.37[a] | 279.86 ± 19.04[a] | 370.01 ± 14.69[b] |
| 29 | 2-Butanone | 911.5 | 306.84 | 1.06033 | 21.21 ± 1.36[c] | 16.93 ± 1.25[b] | 29.38 ± 0.59[e] | 17.12 ± 1.12[b] |
| 30 | 1-Propanol | 1042 | 415.11 | 1.25959 | 709.77 ± 1.66[c] | 545.85 ± 2.94[c] | 589.09 ± 8.05[b] | 462.31 ± 14.89[d] |
| 31 | 2,5-Dimethylpyrazine | 1313 | 872.13 | 1.10546 | 14.70 ± 0.73[a] | 23.02 ± 3.61[a] | 12.36 ± 1.09[d] | 10.56 ± 1.83[b] |
| 32 | 1-Pentanol | 1262.3 | 772.28 | 1.23267 | 11.99 ± 0.55[c] | 12.50 ± 1.35[b] | 9.89 ± 0.45[c] | 7.00 ± 0.59[b] |
| 33 | 1-Butanol-D | 1147.7 | 567.57 | 1.38419 | 161.27 ± 1.27[d] | 64.45 ± 1.55[d] | 14.75 ± 1.44[a] | 11.61 ± 1.81[e] |
| 34 | Isobutyl acetate | 1020.2 | 391.28 | 1.6169 | 42.92 ± 1.82[b] | 93.70 ± 3.48[d] | 18.12 ± 1.30[b] | 10.66 ± 1.26[a] |
| 35 | isobutyl propionate | 1069.4 | 445.15 | 1.27185 | 10.80 ± 0.28[d] | 38.26 ± 3.75[c] | 22.02 ± 2.13[b] | 14.39 ± 0.79[c] |
| 36 | Ethyl formate-M | 855 | 268.08 | 1.06427 | 99.07 ± 15.70[b] | 101.90 ± 5.56[b] | 133.21 ± 3.84[e] | 70.47 ± 5.35[d] |
| 37 | Ethyl formate-D | 856.7 | 269.22 | 1.21937 | 99.65 ± 12.12[a] | 177.32 ± 6.53[c] | 146.34 ± 2.99[d] | 30.78 ± 2.37[c] |
| 38 | 1-Penten-3-one | 1017.6 | 388.41 | 1.09082 | 43.36 ± 2.13[c] | 41.59 ± 1.77[a] | 40.38 ± 2.19[d] | 62.85 ± 9.42[a] |
| 39 | 2-Pentanone | 991.5 | 361.68 | 1.36903 | 150.00 ± 2.70[c] | 9.79 ± 0.98[e] | 23.95 ± 0.82[d] | 7.28 ± 0.72[a] |
| | Unidentified | | | | | | | |
| 40 | 1 | 1041.8 | 414.90 | 1.37708 | 150.85 ± 3.56[c] | 147.93 ± 2.25[c] | 70.15 ± 1.13[d] | 71.31 ± 3.60[a] |
| 41 | 2 | 848.9 | 263.88 | 1.19658 | 13.52 ± 0.5[b] | 15.13 ± 0.37[c] | 31.31 ± 0.42[d] | 5.85 ± 0.51[d] |
| 42 | 3 | 1020.7 | 391.825 | 1.56947 | 13.41 ± 0.99[b] | 16.35 ± 1.13[d] | 13.59 ± 1.33[b] | 13.96 ± 0.76[b] |
| 43 | 4 | 821.6 | 245.15 | 1.0867 | 73.55 ± 6.57[e] | 110.84 ± 4.75[c] | 34.05 ± 0.64[d] | 24.67 ± 3.34[c] |
| 44 | 5 | 904.2 | 301.808 | 1.0217 | 19.05 ± 2.20[c] | 15.45 ± 0.48[d] | 32.43 ± 1.05[d] | 30.50 ± 0.51[b] |
| 45 | 6 | 823.5 | 246.476 | 0.952 | 68.45 ± 5.61[d] | 168.75 ± 3.72[a] | 31.98 ± 0.99[c] | 79.46 ± 2.75[b] |

Each value is calculated using 10 µl 4-mthyl-2-pentanol (20 mg/L) as an internal standard, and the result was expressed as mean ± SD (n = 4). Means in the same line with different letters are significantly different ($p < 0.05$).

RI - retention index calculated using n-ketones $C_4$-$C_9$ as external standard on Mxt-WAX column, Rt - retention time in the capillary GC column, Dt - migration time in the drift tube, $RIP_{rel}$ - relative reactive ion peak.

GC-MS analysis. The characteristic volatiles of MB were dihydro-2-methyl-3 (2H)-furanone, propanal, 2-methylpropanal, which were also detected in pineapple juice by GC-IMS. As the concentration was low in beers BB and PB, it is inferred that these three compounds came from pineapple juice.

To recap, the beer brewed with juice addition during primary fermentation (PB) had the most characteristic volatiles with pineapple flavour especially fruity aroma. The characteristic compounds were propyl acetate, isobutyl acetate, isobutyl propionate, 2, 5-dimethylpyrazine. The specific aroma components in beer BB were 1-butanol, ethyl 2-methylbutyrate, ethyl propionate and 2-pentanone, and in beer MB dihydro2-methyl-3 (2H)-furanone, propanal and 2-methylpropanal.

## PCA analysis

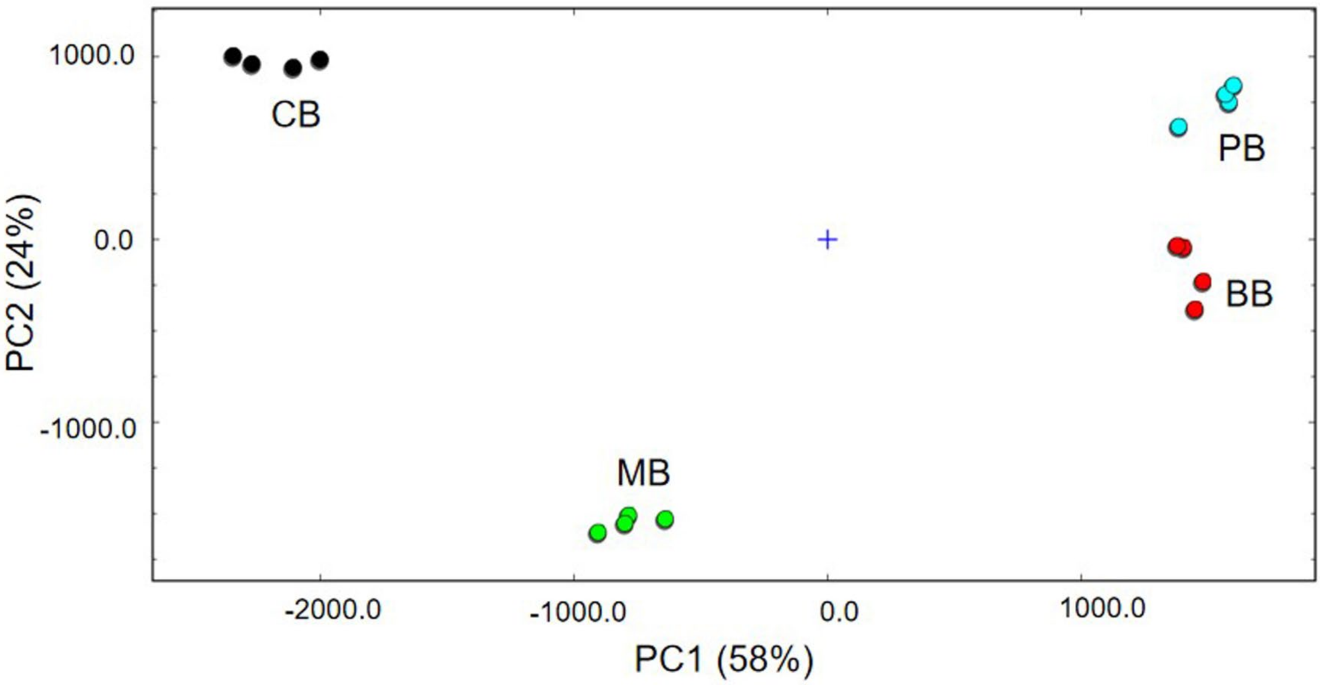
Principal component analysis was performed to extract and highlight the profile of chemical differences between pineapple beers brewed with different juice addition time (Figure 7). The cumulative contribution rate of the first two principal components of PCA was 82%, indicating that the different pineapple beers and control beer could be differentiated.

Beers BB and PB showed high similarity, and both were located at the positive side of PC1, while the control and beer MB at PC1 negative axis. Therfore, BB and PB were dispersed from the control beer CB. This supported the view that the juice added at different times of pineapple juice during fermentation contributed to the aroma of the fruit beer. The control (CB) and MB were distinguished according to the score value of PC2 where they were in the positive and negative side of PC2, respectively. This is consistent with the GC-IMS results where beer MB had the characteristic flavour volatiles dihydro-2-methyl-3 (2H) -furan, propanal, 2-Methylpropanal, which differed from the control beer CB.

In conclusion, identification of fruit beers with juice added at different pints in the process can be achieved by a combination of GC-IMS fingerprinting and principal component analysis. This supports and verifies our previous research (Yang et al, 2022).

### Figure 7.

PCA score plot of pineapple beers brewed with juice added at different times (BB: juice addition at the beginning of fermentation; PB: juice addition at the temperature rise during primary fermentation; MB: juice addition at start of maturation; CB: control beer).



## Results and discussion

The effect of the addition time of pineapple juice on aroma and sensory evaluation of pineapple beers were compared in this study. The results showed that the pineapple beer brewed with juice added late in primary fermentation exhibited the freshest pineapple aroma and had the highest overall preference. The volatiles of the control beer were important aroma components in pineapple beers, and pineapple juice contributed to the aroma of the fruit beer as assessed by electronic nose, GC-MS and GC-IMS analyses. The content of alcohols increased significantly (GC-MS and GC-IMS) in the pineapple beer where juice was added at the start of fermentation and had an obvious alcohol note. The pineapple beer with juice added during primary fermentation had esters with the greatest abundance. However, the concentration of some esters with characteristic pineapple aroma decreased when juice added at start of maturation.

This work supports the process optimisation and the aroma regulation of pineapple beers and will contribute to the enhanced quality of fruit beer. However, this study was not been able to identify the key characteristic aroma volatiles of pineapple beer. Accordingly, further investigations are required to identify the key aroma components of pineapple beers brewed with juice added at different points by combining with odour active value (OAV), dilution and recombination tests.

## Author contributions

**Qing Yang:** investigation, methodology, data analysis, writing (original draft, review and editing).
**Xiao Gong:** methodology, investigation, design and supervision.
**Jingxia Tu:** project administration, funding acquisition and supervision.
**Ming Chen:** resources, technical support and project administration.
**Yuan Yuan:** GC-MS testing and analysis.
**Xiuyan Zheng:** E-nose testing and analysis.

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## Conflict of interest

The authors declare no conflict of interest.

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