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Optimisation of Solid-Phase Microextraction/Gas Chromatography-Mass Spectrometry Using Chemometric Approach for the Identification of Volatile Organic Compounds in *Ananas comosus* [L.] Merr

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Every fruit has its aroma and flavour, including *Ananas comosus* [L.] Merr, known as MD2 pineapple, which is greatly cherished for its alluring sweet flavour. Various volatile and non-volatile organic compounds are present in the fruit, which can be utilised to identify and classify different types of pineapples. This study analysed the volatile organic compounds (VOCs) at different maturity stages of MD2 pineapple (immature, half-mature, and mature) using headspace microextraction for the extraction method. Optimising the fibre with 50/30 µm DVB/CAR/PDMS resulted in the extraction of most VOCs during the extraction process. Gas chromatography-mass spectrometry (GC-MS) was used to evaluate the pineapple extracts. A variety of VOCs, including esters, alcohols, ethers, alkanes, alkenes, aldehydes, ketones, thiazole, amine, and carboxylic acid, were found in the examination of the pineapple sample. Following that, principal component analysis (PCA), one of the chemometric methods in multivariate analysis, was used to analyse the VOCs from the pineapple sample. The PCA was used to classify the VOCs at different maturity stages of the pineapple, with PC1 and PC2 accounting for a total variance of 74.2%. This study also aims to observe different storage conditions that affect the shelf life of the pineapple. The pineapple was stored at three different temperatures: 25 °C, 6 °C, and -10 °C. The effect of storage temperature on the changes in colour, firmness, and pH was evaluated during the 7-day storage period. It was found that the quality and shelf life of pineapple were significantly affected by the different storage temperatures. The results showed that storing the sample at a higher temperature extended the shelf life in terms of colour changes in the fruit sample. The results showed that estimating the shelf life is crucial for preserving fresh fruits and vegetables during storage. Therefore, the significance of this study is to compare and identify the number of VOCs in MD2 pineapple at different maturity stages. It is also important to analyse how shelf life and storage temperature can affect the amount of VOCs contained in the MD2 pineapple.

Keywords: HS-SPME; GC-MS; volatile organic compounds; pineapple; shelf life.

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One of the significant tropical fruits is the pineapple (*Ananas comosus*), the sole significant fruit crop for the *Bromeliaceae* family. The significance may be seen in the United Nations Conference on Trade and Development's (UNCTAD) list of commercial tropical fruits for production on a global scale, where pineapple ranks second to bananas. The Malaysian Pineapple Industry Board (MPIB) reports that Malaysia, which was once a significant global producer in the 1970s, has dropped to the 15th position based on statistics from 2014. Nevertheless, among other significant fruit crops, pineapple production remains essential in Malaysia compared to other fruit crops like banana, jackfruit, watermelon, papaya, and mango [1].

There are numerous cultivars with various aromas, shapes, colours, flavours, and odours. Among them, *A. comosus* [L.] Merr, which is known as MD2 pineapple or Gold cultivar pineapple, has distinguished itself in the global markets due to its recognised flavour, juiciness, sensory characteristics, and sweet-to-acidity balance [2]. The pineapple market has expanded significantly due to the fruit's appealing fragrance compounds, nutritional benefits, and high demand at competitive retail rates. Furthermore, due to the moderate climate and even distribution of rainfall, pineapples are primarily grown in tropical and subtropical countries. The crop can produce fruits shortly after flowering, allowing for year-round yield production [3].

Research by [4] stated that MD2 pineapple volatiles have been the subject of diligent research over the past few decades. At the same time, a previous study by [5] used headspace solid-phase microextraction (HS-SPME) and gas chromatography-

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mass spectrometry (GC-MS) to analyse fully ripe pineapples and three post-harvest stages showing distinct changes in their volatiles. Volatile organic compounds (VOCs) were involved during the development of the ageing process of sliced pineapple, as well as exposure to ultraviolet light. The quantities of VOCs in pineapple vary depending on its stage of development and storage conditions. The sample utilised for the analysis is *A. comosus* [L.] Merr, which was obtained from a nearby farm in UiTM Jengka, Pahang. It was at various stages of development (immature, half-mature, and mature), as well as under various storage conditions (room, cold, and freezing temperatures).

Humans rely on the physical appearance of fruits to determine their maturity by considering different factors, such as size, colour, texture, and shape. The process of fruit identification in the fruit-picking sector is relatively significant. However, it becomes challenging for farmers to identify the maturity stages of crops during harvesting [6]. In the fruit industry, fruits are harvested through visual inspection to determine their maturity [7]. However, manual observation is a lengthy, biased, and time-consuming process, which is prone to human error. Therefore, identifying VOCs in fruits can help predict the time range of growth and fruit maturation, thereby preventing waste due to spoilage.

Solid-phase microextraction is an essential development in sample preparation and has proven to be an effective extraction method [8]. Despite that, SPME is a complex process that requires optimisation before further analysis. This technique can provide chromatographic analysis on dilute solutions with complex matrices, whether in gaseous or liquid form. Several factors that can affect the extraction efficiency must be taken into account to obtain an optimal extraction, including the type of fibre used. The selection of fibres for SPME is crucial as the technique only uses a small amount of extraction sample and is highly selective.

Handling post-harvest fruit is essential to maintain fruit quality, especially for export purposes. Post-harvest produce can undergo transpiration and respiration processes until they cease [9]. Due to this, proper planning and procedures for storage are necessary to ensure the quality of the fruit, prevent spoilage, and ensure safety for consumption. Physiological changes are significantly influenced by environmental temperature and humidity. The physiological changes can also affect the fruit's degradation rate, shortening its shelf life. Pre-cooling is necessary to minimise microbial food contamination and prevent damage and spoilage [10]. A study by [11] stated that based on a report by United Nations, 14% of food produced is wasted annually in the retail and harvest sectors. Therefore, precise shelf-life evaluation is crucial to enhance marketability and economic sustainability.

EXPERIMENTAL

Sample Preparation

The materials used for this study were MD2 pineapples of grade C without any wounds with different maturity stages, obtained from a farm at UiTM Pahang Campus Jengka, as well as 1 g of sodium chloride (NaCl) for 15 mL of the sample, 3% hydrogen peroxide (H_2O_2), and distilled water. First, the pineapple fruit was washed and then submerged in 3% H_2O_2 for 5 min to disinfect it, and peeling was done carefully. Pineapple slices were blended in a juicer and strained through a cloth filter. The juices were immediately frozen using a refrigerator until further analysis. The other halves of the pineapple and peels were used as samples under different storage conditions. The frozen juice sample was thawed in a water bath at 35 ℃ for 30 min and then stirred using a magnetic stirrer for 5 min. A 15 mL aliquot of the samples and 1 g of NaCl were added into the sample and stirred again for 5 min. A 7 mL aliquot of the juice sample was added to a 15 mL glass vial. To study the effect of different storage conditions, the pineapple slices were packed in plastic bags and kept in three different storage conditions (room, cold, and freezing temperatures) at 25 ℃, 6 ℃, and -10 ℃, respectively, for 0 to 7 days. The pH of the fruit was recorded based on three different time intervals.

SPME and GC-MS Analysis

A manual SPME holder was used to perform HS-SPME. The crosslinked phase, 50/30 μm DVB/CAR/PDMS, 85 μm CAR/PDMS, and 85 μm PA supplied by Supelco, Bellefonte, USA, was installed in the SPME fibre assembly. The fibre was thermally conditioned as per the manufacturer's recommendation. Extraction vials (15 mL) with silicon septa were acquired from Supelco (Bellefonte, USA). The SPME syringe was conditioned for 7 min prior to extraction. After conditioning, the syringe was manually inserted into the headspace of the vial and exposed to fibre coating for 29 min at 30 °C. An ionisation energy of 70 eV was used for GC-MS detection in the mass range of 50–550 a.m.u. The temperature programme started at 40 °C with a 2-min hold duration. The temperature was then raised to 150 °C at a rate of 5 °C/min with a 1-min hold duration. Subsequently, the temperature was increased to 250 °C at a rate of 10 °C/min and held for 5 min, with a total run time of 35 min per analysis. The interface temperature was 280 °C, whereas the ion source was at 230 °C. The compounds were identified by comparing the mass spectra of the volatile compounds to those in commercial spectra databases (NIST14 Mass Spectral Library, version 2.0; National Institute of Standards and Technology, Gaithersburg, MA).

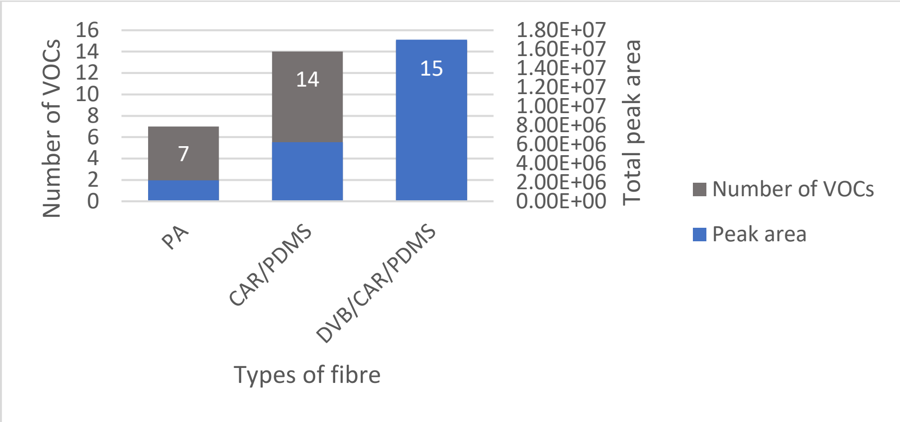

Figure 1. GC-MS analysis of different fibres used in the study.

RESULTS AND DISCUSSION

Selection of Fibre

Various fibres with different thicknesses and fibre coatings can be used according to their analyte. Therefore, choosing the right fibre and technique is essential to achieve the best possible effectiveness of VOC extraction using SPME [12]. Optimising fibre is necessary to achieve better separation in the chromatogram and the maximum efficiency of SPME using HS-SPME-GC-MS throughout the analysis of pineapple samples. Three commercially available SPME fibres with different solvent phases and thickness coatings were evaluated: DVB/CAR/PDMS, CAR/PDMS, and PA. The total area of GC-MS peaks was examined to determine the overall effectiveness of the selected SPME fibre. In addition to examining the total area, the number of VOCs found in each fibre was also evaluated.

From Figure 1, it can be seen that DVB/CAR/PDMS was the most effective in extracting VOCs from the pineapple, as it exhibited the highest total peak area and the highest number of VOC peaks (i.e., 15 peaks). Based on the polarity of the fibres, DVBCAR/PDMS and CAR/PDMS fibres are suitable for extracting bipolar compounds compared to PA fibre, which is suited to extract polar analytes; thus, the PA fibre extracts the least amount of VOCs in this study. Even though DVB/CAR/PDMS and CAR/PDMS are suitable for extracting bipolar analytes, the results show that DVB/CAR/PDMS extracts the most VOCs compared to CAR/PDMS. This is attributed to the more mixed-phase coating fibre in DVB/CAR/PDMS, enabling the fibre to extract most of the volatile compounds. Research by [13] showed that DVB/CAR/PDMS fibre was selected for extracting fruit brandy because it extracted more peaks compared to CAR/DVB. This study shows that DVB/CAR/PDMS extracted many VOCs with the highest total peak area compared to CAR/DVB and PA. In a chromatogram from GC-MS analysis, one can interpret the aroma profile contained in the fruit by analysing the intensity of the peak. Optimising the fibre before further analysis is necessary to determine which fibre extracts the most VOCs during the extraction prior to GC-MS analysis.

The chromatogram in Figure 2 illustrates that the DVB/CAR/PDMS fibre extracted the most VOCs with high peak intensity and better separation between peaks compared to the CAR/PDMS fibre in Figure 3 and the PA fibre in Figure 4. According to [14], using PA fibre for the extraction was inefficient as it resulted in low recoveries for the targeted compounds compared to using mixed coating fibre. The PA fibre extracted low VOCs, and many of the peaks broadened, thus reducing the resolution of the separation and intensity of the peak. Figure 3 shows the chromatogram of the CAR/PDMS fibre, where the peak resolution and intensity were better than the PA fibre. Although the number of VOCs extracted was almost the same as the DVB/CAR/PDMS fibre with 14 VOCs (Figure 1), the intensity and resolution of CAR/PDMS fibre were not desirable compared to the chromatogram of DVB/CAR/PDMS fibre. Furthermore, the DVB/CAR/PDMS fibre extracted the most VOCs with high peak intensity and good resolution between the peaks, making it the most efficient fibre in this research. Mixed-phase fibre coating can absorb a wide range of analytes with different chemical properties compared to single-phase fibre coating [12].

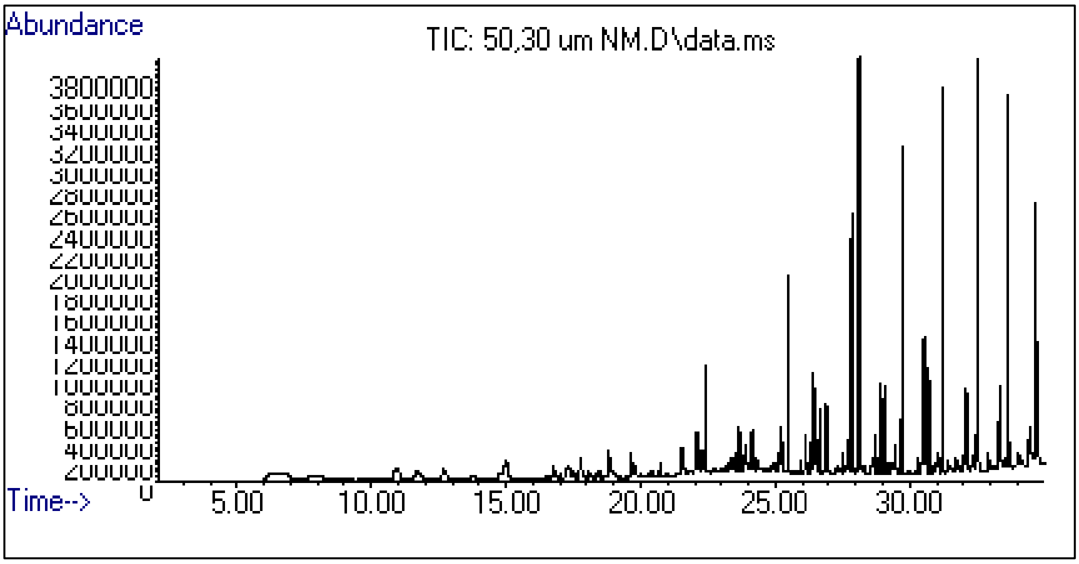

Figure 2. Chromatogram of DVB/CAR/PDMS fibre.

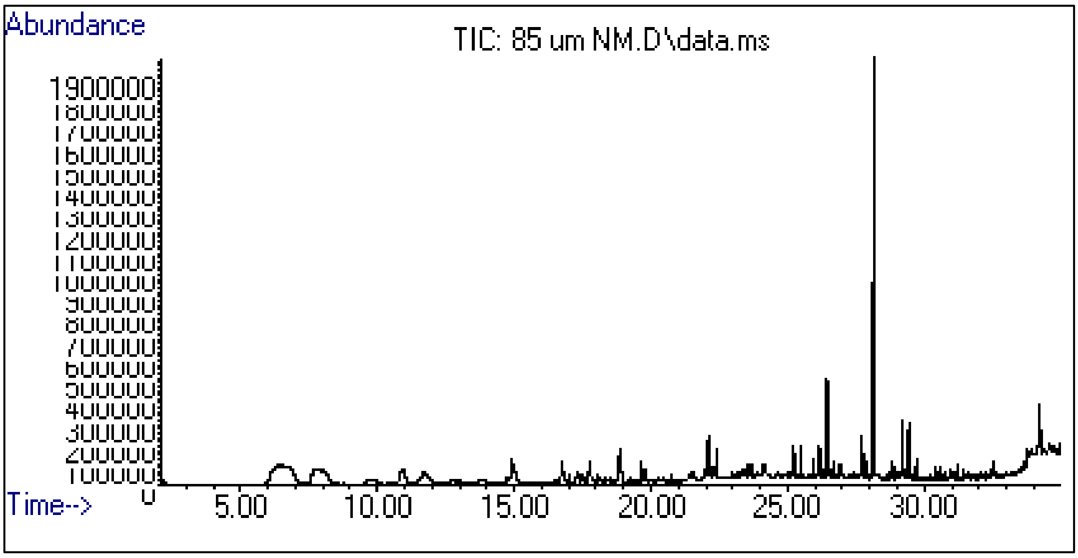

Figure 3. Chromatogram of CAR/PDMS fibre.

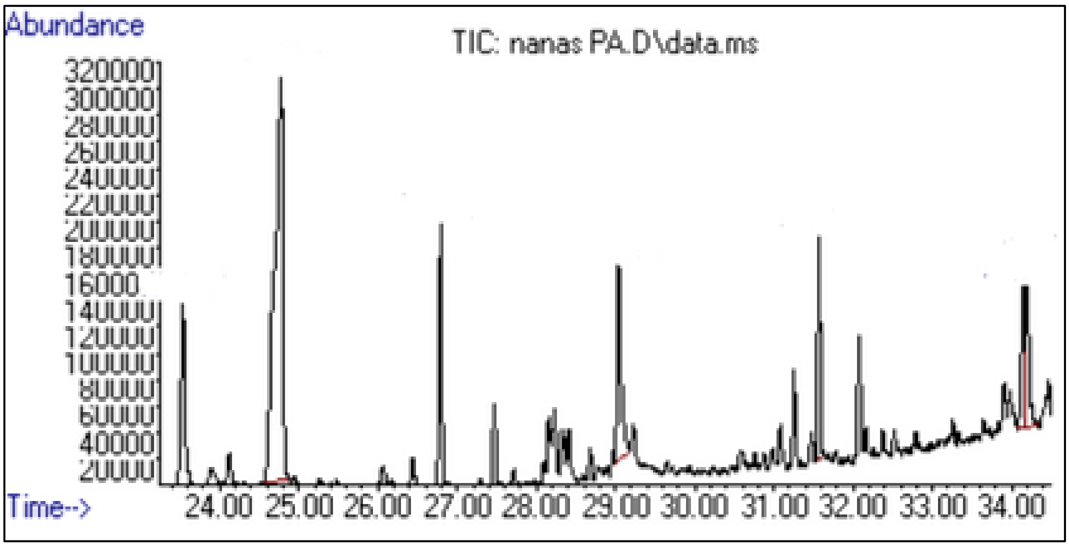

Figure 4. Chromatogram of PA fibre.

Volatile Organic Compounds of MD2 Pineapple

A total of 44 VOCs were identified from the analysis of MD2 pineapple, including fourteen esters; ten alcohols; five ethers; four alkanes, alkenes, and aldehydes, respectively; two ketones; and one thiazole, amine, and carboxylic acid, respectively. Twenty-two VOCs were detected at various maturity stages during this study. Ester was the dominant aroma-active compound found, followed by alcohol. The characteristic aroma of MD2 pineapple is mainly derived from esters, which are the major compounds in the fruit. During postharvest storage, their concentration increases because they are present in the fruit's flesh [15].

Different maturity stages of MD2 pineapple develop various VOCs within the fruit. The VOCs in the fruit play a significant role in developing the aroma and flavour of MD2 pineapple, which can influence its colour changes and taste. Data analysis was conducted based on the chemometric approach known as principal component analysis (PCA), which is one of the multivariate analyses to identify clusters, trends, and outliers in the first two principal components. In this study, no significant differences (p >0.05) were observed in the variation of VOC composition during different maturation stages of pineapple. The PCA was determined based on the peak area of the VOCs detected in the GC-MS analysis.

Figures 5 and 6 show the score plot and loading plot of different maturity stages in MD2 pineapple, respectively. Based on the results in Table 1, the chemometric model explained 100% of the total variance among the samples, with contributions of 46.4% from PC1, 27.9% from PC2, and 25.7% from PC3. Based on the score plot in Figure 5, the MD2 pineapple sample was not classified into clusters based on three different maturity stages. This indicates that the data from this study cannot be used to identify the differentiation in VOCs of the MD2 pineapple based on maturity stages.

Table 1. Eigenanalysis of the correlation matrix using PCA.

Eigenvalue	1.3914	0.8354	0.7732
Proportion	0.464	0.278	0.258
Cumulative	0.464	0.743	1
Variable	**PC1**	**PC2**	**PC3**
Immature (NM)	-0.548	-0.817	-0.183
Half-mature (HM)	0.601	-0.232	-0.765
Mature (M)	0.582	-0.529	0.618

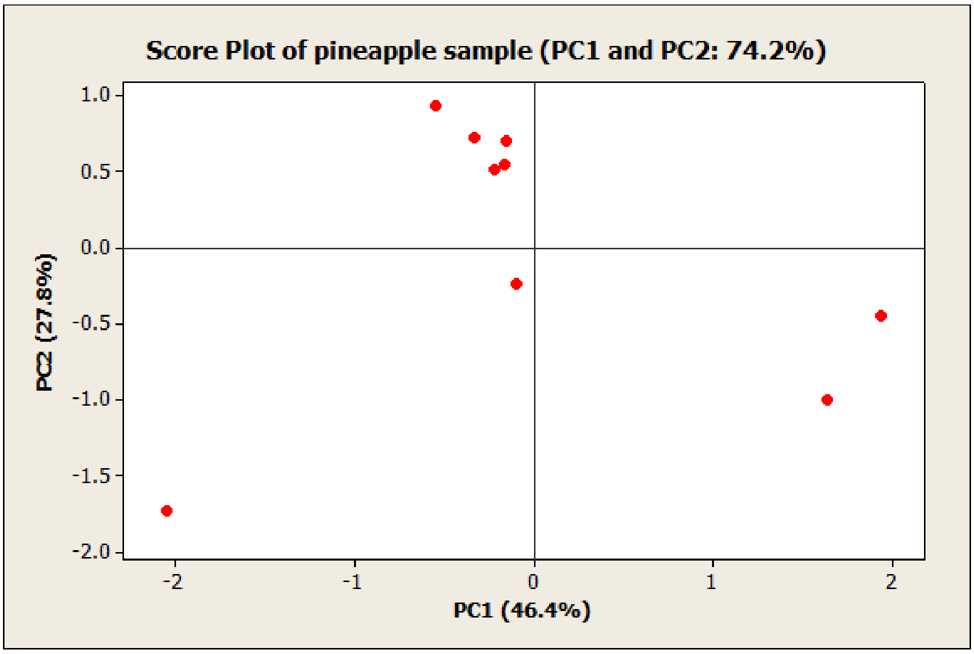

Figure 5. Score plot of different maturity stages in MD2 pineapple.

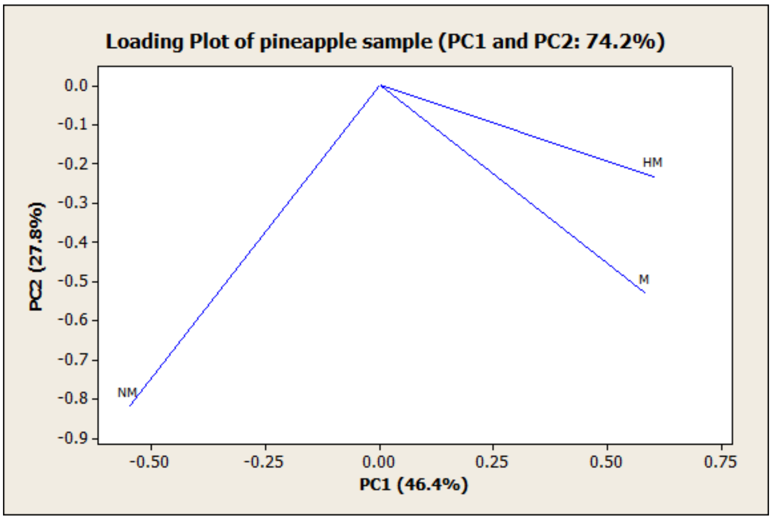

Figure 6. Loading plot of different maturity stages in MD2 pineapple.

Figure 6 displays the loading plot used to assess the relationship between the MD2 pineapple sample and the pineapple's maturity. Based on Figure 6, immature pineapple was located in the negative region on PC1 and PC2, indicating that immature pineapple has a negative correlation with half-mature and mature pineapple samples. As for half-mature and mature pineapples, Figure 6 illustrates that both are positive on PC1 and negative on PC2. However, half-mature pineapple is slightly higher than mature pineapple, with a moderate correlation between the VOCs of half-mature and mature pineapple samples. The outcome of this study differs from earlier research by [16], where the pineapple samples were classified based on different pineapple varieties, with the VOCs of MD2 pineapple were found on the positive side of PC1. This may be due to environmental factors, the chemical composition of the pineapple, sample preparation, and the extraction method.

[16] reported that hexanoic acid methyl ester and butanoic acid 2-methyl- methyl ester were the common aroma components found in pineapple. These compounds were identified as the primary odour-active compounds present in the fruit. Hexanoic acid methyl ester gives a fruity, waxy, sweet, and green fragrance to the pineapple, while butanoic acid 2-methyl-methyl ester produces an aroma profile of green, sweet, and sharp odour. In this study, ether was the most common compound found in the green-ripe pineapple. Ether (ethylene) is responsible for the ripening of fruit. Unripe fruit contains very little ethylene, but as the fruit matures, it produces a high amount of ethylene to accelerate the ripening process, also known as the “climacteric” ripening stage [17]. The GC-MS results from Table 2 show that the immature pineapple also consists of benzaldehyde, which gives the green/grassy smell and bitter taste of the fruit; additionally, 1-hexanol is responsible for the fresh green odour [18].

There are no significant differences in the volatile profile found in half-mature and ripe pineapples. This is because only four and three VOCs were detected in the GC-MS analysis, respectively, as shown in Table 2. Alcohol profiles were detected in half-mature and mature pineapples, referring to their fruity and earthy aroma. This shows that the trend of VOCs in different maturity stages of pineapple decreased as the fruit ripened. Based on a study of wild blackberries, the content of VOCs decreased significantly as the size of the fruit increased [19]. From the GC-MS analysis, it can be concluded that as pineapple ripens, the profile of VOCs changes, and different pineapple types can have varying VOC profiles, which can affect their smell and taste.

Storage Conditions for MD2 Pineapple

The evaluation of the shelf life of MD2 pineapple in this study focused on storage conditions, as they are directly related to the shelf life and quality of pineapple. Temperature is crucial in preserving the quality of fruit, as it can affect the physical and chemical attributes of pineapples, including firmness, skin colour changes, SSC, and titratable acidity, TA. The test examined the physical appearance of the fruit at three different temperatures and time intervals. From Table 3, the pH values of the fruit at different temperatures during storage ranged from 3.02 to 3.20, indicating that the fruit is in acidic conditions. The pH values of MD2 pineapple in the study were higher than those reported by [20], ranging from 2.40 to 3.30 at different storage temperatures. In addition, these results were also significantly lower than the study reported by [21], which found that the pH of pineapples at different maturity stages ranged from 3.70 to 4.30. The difference might be attributed to microbial activity during storage. Variations in pH may be influenced by the growth environment. The acidity of the fruit can be influenced by soil composition, climate, and agricultural practices. For instance, soil nutrients and pH can affect the acidity of pineapples [22].

Table 2. Selected VOCs in three different maturity stages identified from the GC-MS analysis.

Immature	Half-Mature	Mature
Cyclopentasiloxane, decamethyl	Silanediol, dimethyl-	Silanediol, dimethyl-
Cyclohexasiloxane, dodecamethyl-	3-(Methylthio)propanoic acid methyl ester	Naphthalene
tridecane	3(2H)-Furanone, 4-methoxy-2,5-dimethyl	2,2,4-Trimethyl-1,3-pentanediol diisobutyrate
Cycloheptasiloxane, tetradecamethyl	Oxime-, methoxy-phenyl-	
2-Ethylhexanol		
Silanediol, dimethyl		
Benzaldehyde		
Oxime-, methoxy-phenyl		
Acetophenone		
Cyclononasiloxane, octadecamethyl		
Benzaldehyde, 4-ethyl-		

Table 3. pH values at three different temperatures and time intervals.

Temperature	Day	pH Value
	0	3.20
25 ℃	4	3.12
	7	3.06
	0	3.12
6 ℃	4	3.05
	7	3.00
	0	3.15
-10 ℃	4	3.07
	7	3.02

The pH values decreased at three different temperatures for different time intervals, as can be seen in Table 3. It can be observed that the pH value was the highest at 0 days at three different temperatures. The decrease in pH value is associated with fruit maturity, which complied with the previous outcomes [20, 23], ranging between 3.00 and 3.20, falling within the acidic range. The pH values at 0 days were different at 25 ℃, 6 ℃, and -10 ℃, possibly due to soil properties, climate temperature, and water content, which can influence the variation of pH values within the same cultivar. The high pH value of soil can affect photosynthesis and the antioxidant system, which may disrupt the growth of fruits [24]. The relationship between pH and specific VOCs can vary and impact the concentration of volatiles in MD2 pineapple. Many factors, such as the cultivar, level of ripeness, storage conditions, and processing techniques, can significantly impact the content of VOCs in pineapple [25].

Table 4. Colour changes of MD2 pineapple samples after one week of storage.

Storage Temperature	Day 0	After 1 Week
-10 ℃ (Freezing)		

6 °C (Cold)		
25 °C (Room Temperature)		

Table 5. VOCs obtained according to different storage temperatures.

Storage Temperature (°C)	**Volatile Organic Compounds**
Room	• Phenylethyl alcohol
Cold	• Octanoic acid, ethyl ester • 3(2H)-Furanone,4-methoxy-2,5 dimethyl • E)-Hexadec-2-enal • Phenylethyl alcohol • Phenol, 4-ethyl-2-methoxy- • Phenol, 4-ethyl-
Freezing	• Phenylethyl alcohol

Storage temperature is also associated with the physicochemical changes in pineapple, leading to colour changes during storage. According to Table 4, the MD2 pineapple sample changed colour after one week of storage, leading to alterations in its physicochemical properties. Although no analysis of physicochemical properties was conducted in this study, the physicochemical and pH of the pineapple are significantly related. According to [21], the texture, colour, and total soluble solids of the fruit were significantly impacted by the pineapple's declining pH during storage.

After one week of storage at room and cold temperatures, browning was observed on the sample compared to freezing temperature. MD2 pineapples stored at 20 °C exhibited more severe browning compared to those stored at 5 °C [26]. However, internal browning can be triggered by chilling injury, which can occur during storage at low temperatures [27]. The internal browning was caused by physicochemical changes influenced by calcium and gibberellic acid dipping [28]. There was no internal browning for the pineapple sample at freezing temperature. Studies have shown that an effective way to preserve the quality of pineapple and prevent internal browning is by rapidly freezing it using liquid nitrogen [26].

Table 5 shows the VOCs obtained at different storage temperatures. The cold temperature resulted in the highest volatile components in the pineapple sample compared to room and freezing temperatures. However, a previous study by [29] determined that high concentrations of aroma compounds were found to be correlated with high storage temperature in 'phulae' pineapple. Table 5 also indicates that phenyl ethyl alcohol was detected in the pineapple sample at different storage temperatures. No research has mentioned about phenyl ethyl alcohol. Therefore, it is unclear how phenyl ethyl alcohol affects storage, but ethanol has been discovered in fresh-cut pineapple after a particular storage period [30]. [29] also reported that cold storage temperatures have no significant effect on the volatile compounds but have significantly changed the quality of the fruit. Depending on pH, the enzymes in pineapple that control the production and degradation of VOCs may vary. Esterases, lipases, and lipoxygenases are a few enzymes that assist pineapples in producing and breaking down VOCs. The functionality of these enzymes may be pHdependent, suggesting that pH variations may impact the concentration of VOCs [29].

CONCLUSION

The SPME and GC-MS analysis can identify the VOCs of MD2 pineapple. Optimising the SPME fibre is necessary for determining the best extraction method, where DVB/CAR/PDMS has shown to be capable of separating VOCs from MD2 pineapple, resulting in the most intense peaks in the chromatogram. In addition, the differentiation of VOCs for different maturity stages based on PCA results was unsuccessful in classifying the VOCs of each maturity stage. This could be attributed to the limited number of peaks that appeared in the chromatograms, incorrect sample preparation, and extraction method. The evaluation of storage temperature is significant in predicting the fruit's quality and shelf life. The storage temperature is related to physicochemical changes, including colour changes and pH. Decreasing pH significantly affected the physicochemical properties of MD2 pineapple, leading to colour changes that resulted in internal browning. The storage at 25 °C showed severe internal browning, followed by storage at 6 °C, while the pH value decreased significantly during the storage of MD2 pineapple at different temperatures.

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