Application of MOS gas sensors for detecting mechanical damage of tea plants

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Abstract

Mechanical damage of tea plant is a serious problem in tea production. This work employed metal oxide semiconductor (MOS) gas sensors and gas chromatography-mass spectrometer (GC-MS), as an auxiliary technique, to detect tea plants with different types of mechanical damage in different severities. Various algorithms were applied. The results showed the uniformity of the results of gas sensors and GC-MS. While, it was hard for gas sensors to discriminate among tea plants with different types of mechanical damage. However, the feasibility of gas sensors for predicting the damage severity in different damaged types based on gas sensors was proven, which was more meaningful. Finally, multi-layer perceptron neural networks (MLPNN) were employed and the results showed that the correct discrimination accuracy rate for damage severity was 99.07% for the training set and 95.83% for the testing set, which indicated that MLPNN was an excellent algorithm for damage severity determination. This study provided a new technique for mechanical damage of tea plant detection and was very meaningful for tea plant protection.

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Introduction

Tea (*Camellia sinensis*) with great flavor and a high content of beneficial substances (Yang *et al*., 2023), is the most widely consumed beverage aside from water (Tang *et al*., 2024). Furthermore, the tea plant is grown around the world and is an important crop in many countries. Morocco, Japan, and China have consumed green tea for centuries (Abiri *et al*., 2023). Especially for China, who is the homeland of tea that boasts a long-standing history of tea planting, tea is one of the most significant economic crops (Jiang *et al*., 2023). Tea leaves and buds are raw materials of tea, and their health is therefore a crucial factor for producing high-yield and high-quality tea.

However, tea plant easily suffers mechanical damage, which is a type of stress that occurs mostly because of environmental factors and pests' feeding, causing several negative impacts on the plant and leading to severe consequences for marketing and consumption. Firstly, mechanical damage will cause morphological and physiological changes in plants and decrease the nutritive value of fruit (Miller, 1992). Secondly, mechanical damage influences the physiological processes of plants, especially photosynthesis. The decrease in leave area because of mechanical damage lowers the quantity of biomass produced through photosynthesis and some defense materials are induced influencing photosynthesis (Nykänen *et al*., 2004). Moreover, the photosynthetic rate decreases when the foliage of this plant is damaged (Zangerl, 2002), even for the photosynthetic rate of undamaged leaves (Lautner *et al*., 2005). In addition, mechanical damage makes the infestation of disease easier (Chacón-Fuentes *et al*., 2023; Lee *et al*., 2006), which leads to a decrease in quantity and quality of tea.

However, there is not an appropriate method to detect it. Machine vision (Ghooshkhaneh and Mollazade, 2023) is a widely applied technology for damage detection in agriculture. But, as to the situation in this study, the mechanical damage usually hides in leaves, which makes it difficult to detect based on machine vision. On the other hand, a lot of research about the reaction of plants to mechanical damage have been reported and the results show that volatile organic compounds (VOCs) emitted by plants changed after the plant suffered damage (Bezerra *et al*., 2021; Holopainen *et al*., 2010). The reason is that the VOCs, which mainly contain green leaf volatiles, are stored in plant cells and emitted immediately after mechanical damage.

The type and severity are two main parts of mechanical damage, and they should be combined to study. For different types of mechanical damage, the evaluation indexes of their severities are quite different. For example, it is very hard to compare the severity of mechanical damage between the types of cut and scratch. And, the previous studies about it are not found. Hence, in this study, tea plants with different types of mechanical damage in different severities were detected by gas sensors and gas chromatography-mass spectrometer (GC-MS) for giving a more comprehensive result. Gas sensors (Ma *et al*., 2023), which are a nondestructive technique that mimics the human olfactory system detecting volatiles emitted by samples, therefore have the potential to detect plants with damage. The working principle of this technique is that a large number of different compounds contribute to define a measured smell, the sensor array then provides a pattern output that represents a combination of all the components, also known as "fingerprints" of data (Piłat-Rożek *et al*., 2023). The metal oxide semiconductor (MOS) gas sensors are widely used for their lower power consumption and low susceptibility to humidity and temperature (Sibi *et al*., 2024). Furthermore, it is an easy operation and quick detection technique, which make it potential for actual application. On the other hand, GC-MS is a precise and stable detection technique, and able to determine the constituents of volatiles and their amounts. Furthermore, constituents presenting in low-concentration and complex matrices are also available (Aghoutane *et al*., 2023). In this study, the mechanical damage characteristics of tea plants were presented as VOCs and explored in detail. Mechanical damage type and severity were studied together. Furthermore, the damage severity of different damaged types was predicted in one model for giving more practical results. The objectives were: i) to prove the difference of VOCs emitted by tea plants with different types of mechanical damage in different severities using GC-MS; ii) to evaluate the ability of gas sensors in discriminating among tea plants with different types of mechanical damage; iii) to evaluate the ability of gas sensors in predicting damage severity of tea plant.

Materials and Methods

Samples preparation

Tea plant cultivar "clone Longjing43" (25~30 cm high and 8~10 leaves for each plant) was employed in this study. They were provided by the Tea Research Institute, Chinese Academy of Agricultural Sciences. These tea plants were cultivated for experiments specifically and taken care of carefully, making sure that each tea plant was healthy. Moreover, each tea plant was transplanted to the laboratory one week before the experiment and kept under controlled conditions (24±2 C, 75-85% of relative humidity). Ten treatments (tea plants with no damage, with cut damage in three severities: one cut, two cuts and three cuts, with scratched damage in three severities: one scratch, two scratches and three scratches, and with punctured damage in three severities: five punctures, ten punctures and fifteen punctures) were carried out in this experiment and labeled as group Undamaged, 1Cut, 2Cut, 3Cut, 1Scratched, 2Scratched, 3Scratched, 5Punctured, 10Punctured, 15Punctured, respectively. For GC-MS measurement, 3 tea plant samples were prepared for each group, while for gas sensors measurement, 20 tea plant samples were prepared for each group.

For the damaged type of cut, the length of it was 20 mm for each leaf. Groups 1Cut, 2Cut and 3Cut had one, two and three leaves damaged, respectively. For the damaged type of scratch, the area of it was 1 mm^2 for each leaf. Groups 1Scratched, 2Scratched and 3Scratched had one, two and three leaves damaged, respectively. For the damage type of puncture, groups 5Punctured, 10Punctured and 15Punctured had 5 needle pricks, 10 needle pricks and 15 needle pricks, respectively. The treatment can be seen in Figure 1.

GC-MS measurement

Purge-and-trap coupled with GC-MS was employed to determine VOCs in different samples (Andrews *et al*., 2015). In this experiment, 8 h was used for collecting VOCs. Then, the collected VOCs were eluted with 900 μL of dichloromethane. Ethyl caprate was taken as the internal standard, and 3 μL of Ethyl caprate dichloromethane (50 μL/L) was injected into the eluted solution by a syringe with a range of 10 μL. The solution made above was analyzed by GC-MS.

In this study, VOCs were analyzed in an HP 6890 series gas chromatograph equipped with a flame ionization detector and coupled to an HP 5973 mass spectrometer selective detector (Agilent Technologies, Palo Alto, CA, USA). An HP-5 methyl siloxane chromatographic column (30 m, 0.25 mm internal diameter, and 0.25 μm film thickness; Alltech, Deerfield, IL, USA) was used for separation. Helium (24 mL/min) was used as the carrier gas.

A splitless injection was carried out (injection temperature 250°C and remained for 3 min for desorption; injected volume 2 μL). Following injection, the column temperature was programmed from 45°C (2 min) to 140°C at 3°C/min, then increased to 200°C at 6°C/min, and finally reached 260°C at 20°C/min. The ion source temperature was set to 230 °C. The electron impact of mass spectra was recorded at 70 eV ionisation energy within a mass range of 40~350 amu. Compounds were identified by comparing the recorded mass spectra with the National Institute of Standards and Technology (NIST 11.0) mass-spectral library. In addition, the retention indices (RI) were calculated using a homologous series of n-alkanes (C8-C20) (Sigma-Aldrich Shanghai Trading Co., Ltd., Shanghai, China).

Gas sensor measurement

In this study, an E-nose system (PEN2, Airsense Analytics,GmBH, Schwerin, Germany) equipped with ten different gas sensors is applied. Their characteristics of them are given in Table 1. Prior to the measurement, each sample was put into a Ziploc bag, and the bag was sealed with clips for 30 minutes, ensuring that headspace volatiles were stable enough for detection. During the measurement process, the tea plant headspace volatiles were pumped into a chamber where gas sensors were in it. The flush time (the time of zero gas passing through the sensor array) was set to 50 seconds with a rate of 600 mL/min, while the measurement time (the time sample gas passing through the sensor array) was 80 seconds with a rate of 200 mL/min. The data was collected and recorded by computer once per second.

Data analysis methods

The stable value of signal response is a widely applied feature in gas sensor results analysis (Jiang *et al*., 2017). In this study, the 80th of the signal response of each sensor was extracted as stable values and applied for the next analysis. The principal components analysis (PCA) is a method seeking to use a linear combination of the original variables to derive an index measure of multilateral data while capturing their maximum variance (Lu *et al*., 2020; Tonin *et al*., 2024) and is able to handle high-dimensional and highly correlated data by projecting the data onto a lower-dimensional subspace that constrains most of the variance of the raw data. Finally, several principal components (PCs) are selected for

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their higher contribution rates. By contrast, locally linear embedding (LLE) is a nonlinear method that computes low-dimensional, neighbourhood-preserving embeddings of high-dimensional inputs (He *et al*., 2023). This algorithm attempts to discover nonlinear structures in high dimensional data by exploiting the local symmetries of linear reconstructions and is able to learn the global structure of nonlinear manifolds (Wang, 2012). Linear discriminant analysis (LDA) is a supervised method and allows easy visualization of almost all the information contained in the dataset. Furthermore, it is able to extract useful information from the data to explore the data structure, including correlations between variables and relationships between subjects (Lin *et al*., 2021).

K-means cluster analysis is a kind of "hard" partitioning methods, in which each point is assigned to only one particular cluster. K-means cluster analysis starts with k cluster centers that are chosen at random or according to some heuristic procedure. In each iteration, each instance is assigned to its nearest cluster center, resulting in a re-calculation of the cluster center. This process is repeated until a convergence criterion is met. The k-means is popular for its ease of interpretation, speed of convergence and adaptability (Sheikhhosseini *et al*., 2021).

Multi-layer perceptron neural network (MLPNN) is one of the most popular neural networks in use today (Seo and Min, 2023). An MLPNN consists of three layers, including one input layer, one output layer, and one or more hidden layers. The number of neurons in the hidden layers is determined by a trial-and-error procedure. Neurons between two layers are connected through communication links-associated weights, which are determined by a learning (training) algorithm (Zhu *et al*., 2021).

The data processing methods (LDA, k-means cluster analysis and MLPNN) were analyzed by SPSS version 22 (SPSS Inc., Chicago, IL, USA). PCA and LLE were performed by MATLAB 2020a software (MathWorks, Natick, MA, USA).

Results

Results of GC-MS

The main VOCs emitted by tea plants with ten different groups were identified by GC-MS and their amounts are presented in Figure 2. In Figure 2a the results of group Undamaged are illustrated, while Figure 2 b,c,d represent the results of groups Cut, Scratched and Punctured in three different damage severities, respectively. The x-axis is the value of RI and the y-axis is the amount of each VOC. One RI corresponds to one certain VOC. The values of RI were compared with those in other literatures, which made the identified VOCs more reliable. Finally, six main VOCs (tetrachloroethylene, α-pinene, 3-carene, limonene, nonanal and naphthalene), whose RI are 801, 930, 1006, 1026, 1104 and 1178, respectively, are presented. In Figure 2, VOCs emitted by group Undamaged are quite different from those emitted by groups with mechanical damage. For group Undamaged, only one of six VOCs is detected. While, for groups with mechanical damage, six VOCs are all detected. As shown in Figure 2, for the groups with the same type of mechanical damage in different severities, the amounts of VOCs are different and their amounts increase as the raise of severity of mechanical damage generally. For the groups with mechanical damage, the amounts of VOCs for groups with cut and punctured damage are similar, which range from 30 to 400 ng/8hours. While, for the groups with scratched damage, the amounts of VOCs are relatively low. However, their amounts would increase and be similar to those of the other two groups if the severity of scratched damage becomes more serious. Furthermore, the proportions of VOCs among groups with different types of mechanical damage are similar, where the amounts of α-pinene, 3-carene and limonene are relatively high and those of the other three VOCs are low.

According to the description above, VOCs emitted by group Undamaged are quite different from those emitted by groups with

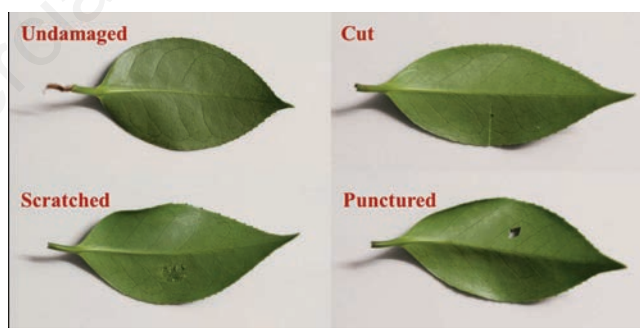

Figure 1. Four treatments for the tea plant.

Table 1. Sensors used and their main applications.

Number in array	Sensor name	General description	Reference
S1	W1C	Aromatic compounds	Toluene, 10 ppm
S2	W5S	Very sensitive, broad range sensitivity, react on nitrogen oxides, very sensitive with negative signal	NO_2, 1 ppm
S3	W3C	Ammonia, used as sensor for aromatic compounds	Propane, 1 ppm
S4	W6S	Mainly hydrogen, selectively (breath gases)	H_2, 100 ppb
S5	W5C	Alkanes, aromatic compounds, less polar compounds	Propane, 1 ppm
S6	W1S	Sensitive to methane (environment) ca. 10 ppm. Broad range, similar to no. 8	CH3, 100 ppm
S7	W1W	Reacts on sulphur compounds, H2S 0.1 ppm. Otherwise sensitive to many terpenes and sulphur organic compounds, which are important for smell, limonene, pyrazine	H_2S, 1 ppm
S8	W2S	Detects alcohol's, partially aromatic compounds, broad range	CO, 100 ppm
S9	W2W	Aromatics compounds, sulphur organic compounds	H_2S, 1 ppm
S10	W3S	Reacts on high concentrations >100 ppm, sometimes very selective (methane)	CH_3, 10 CH_3, 100 ppm

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mechanical damage. On the other hand, for the groups with the same type of mechanical damage in different severities, their amounts of VOCs follow a certain rule and are easily identified. However, for the groups with different types of mechanical damage, the amount range and proportion of VOCs have similarities, which make it hard for discrimination. The possible reason is that the VOCs, which are stored in plant cells and emit after mechanical damage, are the same for different types of mechanical damage. In addition, that is the main factor for the change of VOCs after mechanical damage and influences the VOCs most.

Results of gas sensors

In this study, three types of mechanical damage (cut, scratch and puncture) were employed and undamaged tea plants were included as the controlled group. PCA, LLE and LDA were applied for visualizing the groups' distribution and their results are shown in Figure 3 and Figure 4. Figure 3 is the results of PCA. Figure 3 a,b represents the score plot and the loading plot, respectively. In Figure 3a, the red, yellow, cyan and blue points are the groups Undamaged, Cut, Scratched and Punctured, respectively. The two PCs explain 97.13% of the total contribution variance, which is sufficient enough to represent the whole information of gas sensor results. Moreover, four clusters can be seen, which indicates the possibility of discrimination. From Figure 3b, the relationships of sensors and PCs can be seen. The point of each sensor is far away from the zero-point, which indicates a close relationship. Furthermore, S1, S3 and S5 have similar performances. S6 and S8 are similar. Combining with Figure 3 a,b, the correlation between gas sensors and each sample could be seen, which indicates the feasibility of those gas sensors. For example, Group Undamaged is relevant to S6 and S8. Group Cut is correlated with S1, S3 and S5. All in all, four groups could be discriminated correctly and gas sensors are feasible for tea plant detection.

Then, LLE and LDA were employed, and the results are shown in Figure 4. Better results are obtained. Especially for Group Undamaged, it is far away from other groups. On the other hand, the results of GC-MS also show a big difference between group Undamaged and other groups, which indicates the consistency. Next, more in-depth research was carried out. PCA, LLE and LDA were employed again to deal with the results of gas sensors in detecting tea plants with different types of mechanical damage in different severities. Figure 5 and Figure 6 represent the results of PCA, LLE and LDA, respectively. Nine groups (three types of mechanical damage in three severities) are included. Points of groups 1Cut, 2Cut, 3Cut, 1Scratched, 2Scratched, 3Scratched, 5Punctured, 10Punctured and 15Punctured are black square, red circle, orang upper triangle, yellow lower triangle, green diamond, green left triangle, light blue right triangle, blue hexagon and dark blue five-pointed star, respectively.

Figure 5 a,b are the score plot and the loading plot, respective-

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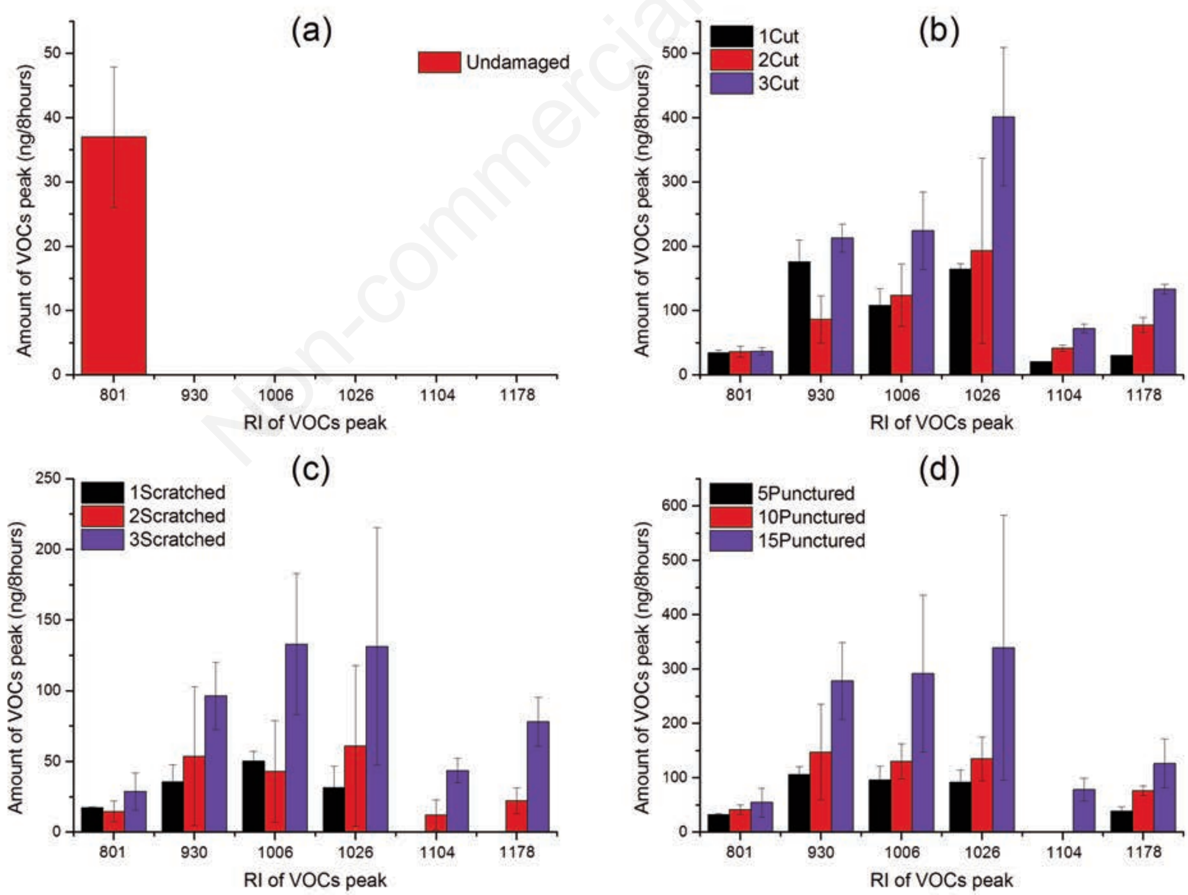

Figure 2. Six main VOCs emitted by tea plants with ten different treatments were identified by GC-MS. **a**) Undamaged tea plants; **b**) tea plants with cut damage; **c**) tea plants with scratched damage; **d**) tea plants with punctured damage.

ly. As shown in Figure 5a, two PCs explain 97.13% of the total contribution variance, which is sufficient enough to represent the whole information of gas sensor results. The results of Figure 5b are similar to those of Figure 3b, which indicates the reliability of PCA. However, groups are clustered and hard to be discriminated. Hence, LLE and LDA were employed again. The results of LLE (results in Figure 6a are similar to those of PCA in Figure 5). Two main parts can be divided. The first part includes groups 2Cut, 3Cut, 10Punctured and 15Punctured, while the second part includes groups 1Scratched, 2Scratched, 3Scratched and 5Punctured. Group 1Cut stayed between these two parts. Moreover, it could be easily found that the first part is the tea plants with higher severity of mechanical damage, while the second part is the ones with lower severity. On the other hand, three types of mechanical damage (cut, puncture and scratch) are clustered in one part, which indicates the unfeasibility of gas sensors in discriminating among tea plants with different types of mechanical damage. In Figure 6b, the group's distribution becomes clearer, and the group's distribution from left to right is groups 3Cut, 2Cut, 15Punctured, 10Punctured, 1Cut, 5Punctured, 3Scratched, 2Scratched and 1Scratched respectively, if all groups are projected onto the x-axis. Combined with Figure 2 (the amount of VOCs), it also could be considered as the decrease in severity of mechanical damage. Besides, groups 2Scratched and 3Scratched overlap, the reason of which might be that their damage severities are too similar for detection and the severity of scratched damage is hard to be distinguished because of low damage severity.

According to the description above, the mechanical damage type is hard to discriminate if combined with damage severity. However, there is a relationship between damage severity and gas sensor output. Clearer and more accurate results of the relationship are necessary. Hence, K-means cluster analysis and MLPNN were employed for next analysis. For K-means cluster analysis, nine classes were set because there were nine groups for discrimination. The results are shown in Table 2. Each row means each tea plant group, and each column means the class of K-means cluster analysis. Moreover, the class that highest number of tea plants belonging to is considered as the target one. For example, there are 10 samples of group 1Cut belonging to class 1, which is the highest. Hence, group 1Cut is considered as in class 1. Then, six main parts could be finally clustered. The first part contains group 1Cut, the second part contains groups 2Cut and 3Cut, the third part contains groups 1Scratched, 2Scratched and 2Scratched, the fourth, fifth and sixth parts are groups 5Punctured, 10Punctured and 15Punctured, respectively. Furthermore, the groups in the same part are the neighbour according to Figure 6b, which indicates its correctness. Moreover, the severity of each class also can be seen combined with Figure 6, whose severities from slight to serious are the second part, sixth part, fifth part, first part, fourth part and third part, respectively. On the other hand, the groups' distribution gives

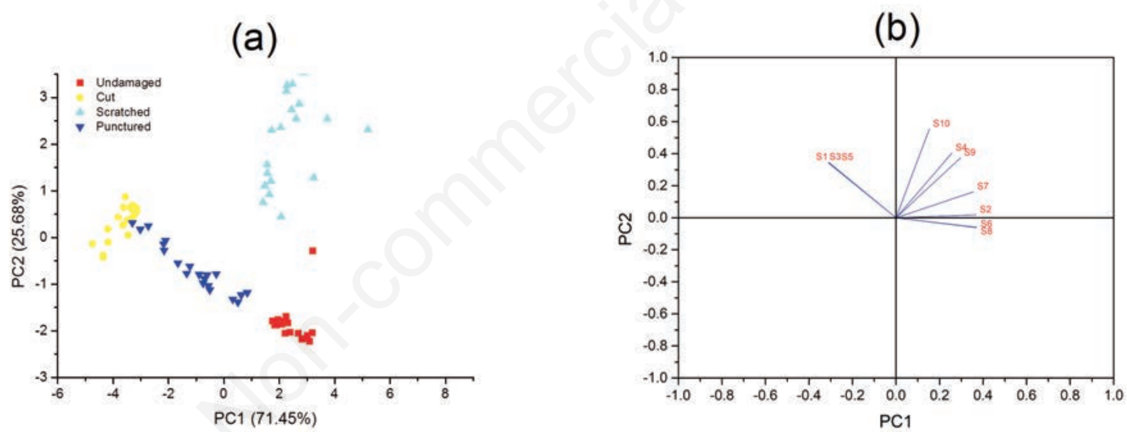

Figure 3. PCA results of tea plants with different types of mechanical damage. **a**) Score plot; **b**) loading plot.

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Table 2. Rresults of K-means cluster analysis.

Class	1	2	3	4	5	6	7	8	9
1Cut	10			1	1	1	1	3	3
2Cut		15							5
3Cut		20							
1Scratched	1		2		1	14		2	
2Scratched	1		3			16			
3Scratched	2		1			17			
5Punctured	2		1			1		16	
10Punctured							1		19
15Punctured		2					17		1

a guide for MLPNN analysis next. For MLPNN, a model with three layers was employed. The activation function was set as the sigmoid transfer function and the learning rate was set as 0.3. 180 samples (20 samples for each group) were divided randomly into training and testing subsets, 108 samples (12 samples of each group) for the training set and 72 samples (8 samples of each group) for the testing set. The groups that were hard to be discriminated were considered as in the same part for the similarity of damage severity. The number of parts was the output dimension of MLPNN, which was determined by the results of the K-means

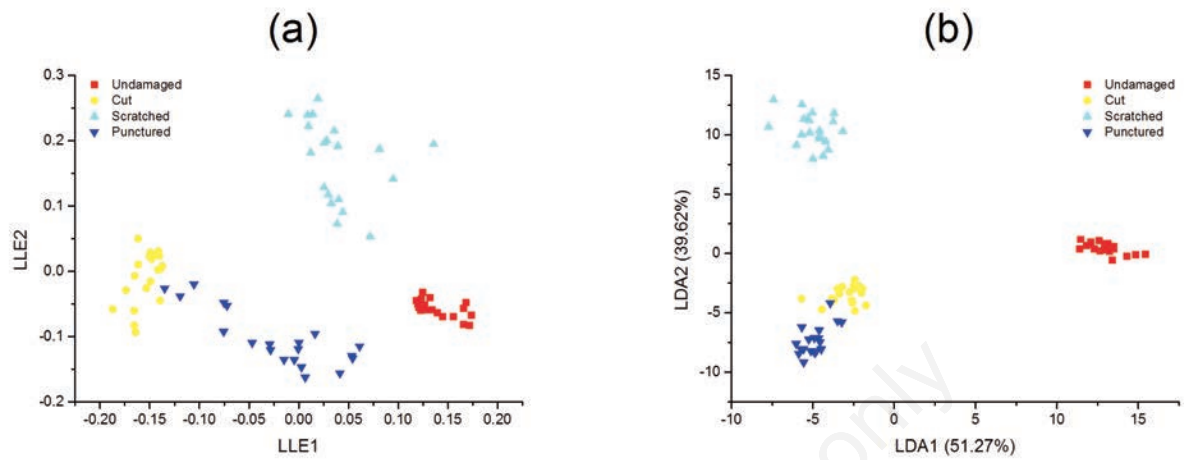

Figure 4. Visualization analysis of tea plants with different types of mechanical damage. **a**) Results of LLE; **b)** results of LDA.

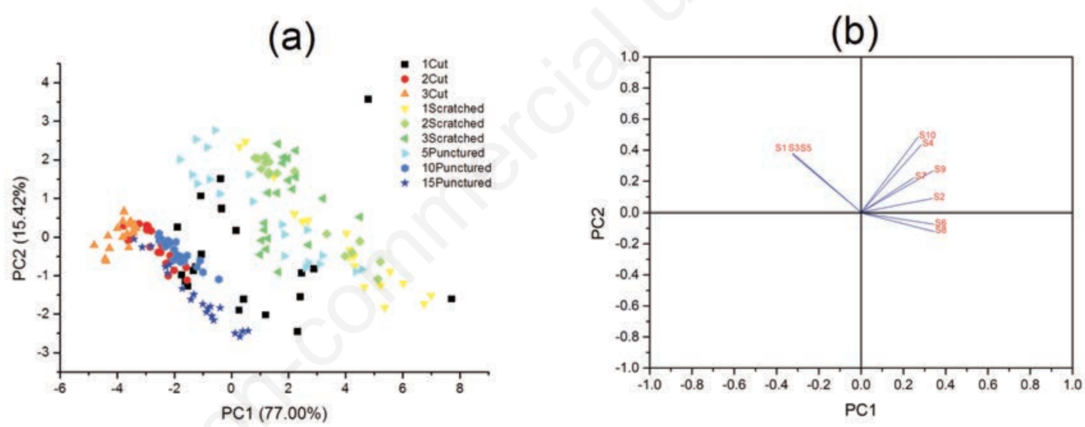

Figure 5. PCA results of tea plants with different types of mechanical damage in different severities. **a**) Score plot; **b**) loading plot.

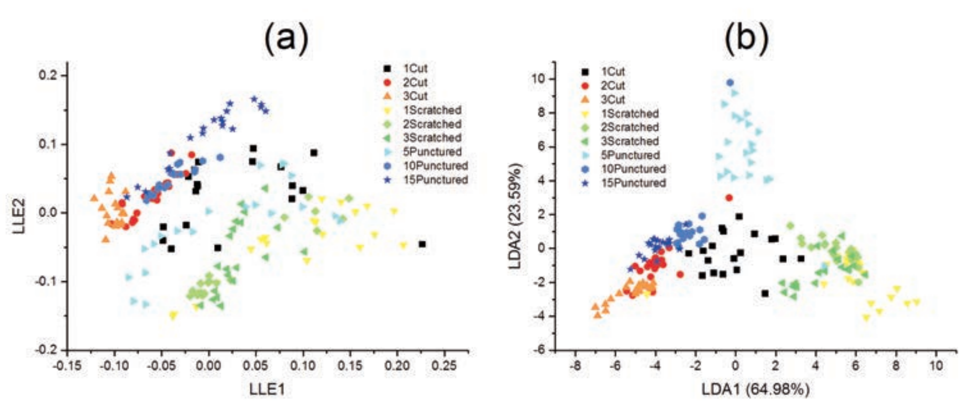

Figure 6. Visualization analysis of tea plants with different types of mechanical damage in different severities. **a)** Results of LLE; **b**) results of LDA.

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cluster analysis. Hence, six parts, which also can be considered as six damage severity degrees, were divided. The correct discrimination accuracy rate was obtained. Finally, its results show that the correct discrimination accuracy rate for damage severity is 99.07% for the training set and 95.83% for the testing set, respectively, which is good enough for damage severity prediction.

Discussion

The results of GC-MS and gas sensors have been displayed above. Furthermore, the relationship about damage severity among different types of mechanical damage became clearer and clearer from the visualization analysis (PCA, LLE and LDA) to K-means cluster analysis and MLPNN.

According to the results of GC-MS (Figure 2), there is only one main VOC (Tetrachloroethylene) detected by GC-MS for undamaged tea plants. Hence, the other five VOCs can be considered as emission because of mechanical damage. On the other hand, the amount range and proportion of VOCs are both similar, which makes it hard for discrimination based on VOCs. It also means that it is hard for gas sensors to discriminate among tea plants with different types of mechanical damage, which is inconsistent with the results of Figure 3 and Figure 4. The possible reason is that the damage severity of tea plants with different types of mechanical damage detected by gas sensors is different and causes the good discrimination results of Figure 3 and Figure 4. It is very hard to obtain the same severity of mechanical damage with different types. Hence, the true item that discriminated by gas sensors is the severity of mechanical damage. Furthermore, according to the results of GC-MS, the VOCs emitted by tea plants with the same type of mechanical damage in different severities are also different, which indicates that it is highly possible to discriminate among them based on VOCs. However, the discrimination performances of damaged and undamaged tea plants are all great according to either GC-MS or Figures 3 and 4.

Hence, the discrimination performance of damaged tea plants needs to be discussed next. According to the results of Figure 5 and Figure 6, it is hard to discriminate among tea plants with different types of mechanical damage, which corresponds to the results of GC-MS. In more detail, the amount of VOCs emitted by group 3Cut is the highest and those of VOCs emitted by scratched groups are relatively low, which is consistent with the results of LDA. By contrast, the damage severity is able to be evaluated from serious to slight generally, which is a more important parameter for evaluating damage loss.

For K-means cluster analysis, nine groups are divided into six parts based on the damage severity scale, which is similar to the results of LDA. However, according to Figure 6b and Table 2, there are still some samples that overlap with other groups. Hence, MLPNN was employed for more accurate and detailed results. Finally, its results show that the correct discrimination accuracy rate for damage severity is 99.07% for the training set and 95.83% for the testing set, respectively. The results indicate the good performance of MLPNN and the feasibility of damage severity prediction, which is more meaningful for pant protection than damage type prediction. This study provides a new method for damage severity prediction of tea plant.

Conclusions

This study employed E-nose with gas sensors to detect tea plants with different types of mechanical damage in different severities. GC-MS was applied as an auxiliary technique. The discrimination ability of different types of mechanical damage and their damage severities were studied, respectively. Conclusions were reached here:

i) The results of GC-MS showed that the amount's range and proportion of VOCs were all similar for the groups with different types of mechanical damage, which made it hard for discrimination based on VOCs. However, for the groups with different mechanical damage severities, their amounts of VOCs were different and increased as the increase of severity of mechanical damage generally.

ii) In combination with the results of GC-MS, it was unfeasible to discriminate among tea plants with different types of mechanical damage based on VOCs. However, it was assured that the group Undamaged could be identified from the damaged groups.

iii) Three algorithms (PCA, LLE and LDA) were employed to visualize the discrimination results of tea plants with three types of mechanical damage in three severities. LDA had the best performance and the results showed the groups' distribution as the increase of damage severity and indicated that it was potential for gas sensors in predicting the damage severity of tea plants.

iv) K-means cluster analysis indicated six damage severity scales, which corresponded to the results LDA and was set as a parameter for MLPNN. The prediction results of MLPNN were good enough, whose correct discrimination accuracy rate for damage severity was 99.07% for the training set and 95.83% for the testing set. All in all, the results proved that the severity of different mechanical damage types was able to be identified by gas sensors, which was more meaningful for pant protection. Furthermore, gas sensors have the characteristics of simple operation and quick detection, making them potential for practical applications.

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