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Integrated electronic nose and FTIR spectroscopy combined with PCA for freshness classification of *Clarias Gariepinus* during storage

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Abstract Rapid and objective assessment of fish freshness is essential for ensuring food safety and maintaining product quality throughout the seafood supply chain. Conventional methods for evaluating fish freshness, such as sensory inspection, microbiological analysis, and chemical indicators, are often time-consuming, destructive, and unsuitable for rapid monitoring. Therefore, the development of rapid and non-destructive analytical approaches is increasingly important for food quality control. This study aimed to evaluate the potential of an integrated E-nose and Fourier Transform Infrared (FTIR) spectroscopic system combined with PCA for classifying freshness degradation in freshwater *Clarias gariepinus* during storage. A total of 40 homogenized fish samples were prepared and divided into four storage groups (0, 12, 24, and 36 hours). Volatile compounds released from the samples were measured using an E-nose equipped with five metal oxide semiconductor sensors (MQ-3, MQ-4, MQ-136, MQ-137, and TGS-822). Each sample measurement was repeated five times, and the averaged sensor responses were used for subsequent analysis. Chemical structural changes in the samples were further investigated using FTIR spectroscopy within the wavenumber range of 4000–400 cm^{-1}. PCA was applied to both E-nose and FTIR datasets to reduce dimensionality and visualize clustering patterns among storage conditions. The PCA results of E-nose data showed that PC1 and PC2 explained 98.5% and 0.12% of the total variance, respectively, with the MQ-3 sensor exhibiting the highest sensitivity to volatile changes during storage. The PCA score plot revealed clear separation among samples stored at different time intervals. FTIR analysis also demonstrated progressive spectral variations associated with protein degradation and lipid oxidation, particularly in O–H, Amide I–II, C–H, and C=O functional groups. PCA of FTIR spectra explained 63.1% and 26.2% of the variance for PC1 and PC2, respectively, indicating consistent classification patterns. These results suggest that the integration of E-nose sensing and FTIR spectroscopy combined with PCA provides complementary information for monitoring fish freshness changes during storage.

Keywords: chemometrics, food freshness, gas sensors, infrared spectroscopy, seafood quality, volatile compounds

1. Introduction

Fish freshness is a critical quality parameter in the fisheries and seafood industries because it directly affects food safety, consumer acceptance, and the economic value of fish products (Madhubhashini et al., 2023; Wang, 2025). During storage, fish undergoes biochemical, microbiological, and sensory changes that gradually reduce its quality. These changes include protein degradation, lipid oxidation, and the production of volatile organic compounds that alter the odor profile of the fish (Fengou et al., 2019; García et al., 2022). Consequently, the rapid and reliable detection of fish freshness is essential to ensure product safety and maintain quality throughout the supply chain.

Catfish (Clarias gariepinus), locally known as “lele” in Indonesia, is one of the most widely consumed freshwater fish due to its affordability, high protein content, and availability in local markets. However, similar to other fish species, catfish is highly

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perishable and undergoes rapid quality deterioration during storage. Biochemical reactions and microbial activity lead to structural and chemical changes in fish tissues, resulting in the formation of compounds associated with spoilage and off-odor. Therefore, developing reliable methods to evaluate catfish freshness is important for supporting quality control in fish distribution and marketing systems (Power & Cozzolino, 2020; Ramkumar et al., 2025).

Conventional techniques used to evaluate fish freshness include sensory evaluation, microbiological testing, and chemical indicators such as total volatile basic nitrogen (TVB-N). Although these methods are widely applied, they have several limitations. Many of them are destructive, time-consuming, and require laboratory analysis, making them less suitable for rapid monitoring in real supply chain conditions. In addition, sensory evaluation relies heavily on human perception, which introduces subjectivity and variability in freshness assessment (Gilman et al., 2019; Sun et al., 2025). These limitations highlight the need for rapid, objective, and non-destructive analytical approaches for fish freshness monitoring.

In recent years, sensor-based technologies such as the electronic nose (E-nose) and spectroscopic techniques like Fourier Transform Infrared (FTIR) spectroscopy have gained increasing attention in food quality analysis (Govari et al., 2022; Wijaya et al., 2023). The E-nose is designed to detect volatile compounds released from food products using sensor arrays that respond to specific gases and volatile organic compounds. This technology has been widely applied to analyze aroma profiles and detect spoilage-related volatiles in food products including meat, dairy, and seafood (Wu et al., 2022). By capturing patterns of volatile compounds, the E-nose can provide rapid and objective information regarding freshness and quality changes during food storage.

Meanwhile, FTIR spectroscopy provides complementary information by identifying chemical composition and molecular structures through infrared absorption patterns (Lv et al., 2018). This technique allows the detection of characteristic functional groups associated with proteins, lipids, carbohydrates, and water content in food matrices. FTIR spectroscopy has been widely used in food analysis to monitor chemical transformations such as protein denaturation, lipid oxidation, and moisture changes occurring during storage and processing (Zhang et al., 2015). Because of its rapid and non-destructive nature, FTIR has become an important analytical tool for investigating chemical changes in food materials.

To interpret complex datasets obtained from sensor systems and spectroscopic measurements, chemometric methods are commonly applied. One of the most widely used multivariate techniques is Principal Component Analysis (PCA), which enables dimensionality reduction and visualization of patterns within multivariate datasets. PCA transforms correlated variables into a set of principal components that capture the majority of data variance, allowing the identification of clustering patterns and relationships among samples (Tian et al., 2011). In food quality studies, PCA has frequently been used to classify samples according to freshness levels, storage conditions, or processing treatments based on sensor or spectral data (Saputra et al., 2018).

Recent advances in sensor-based technologies have demonstrated the potential of electronic nose systems for detecting volatile compounds associated with food spoilage, particularly in fish and seafood products (Wang & Chen, 2024). Similarly, FTIR spectroscopy has been widely applied to investigate chemical composition and structural changes in food materials through the analysis of characteristic functional groups (Saraiva et al., 2017). Several studies have reported the successful use of E-nose systems combined with multivariate analysis to classify food freshness based on volatile compound profiles (Putri et al., 2024). Likewise, FTIR spectroscopy combined with chemometric techniques has been applied to detect chemical transformations such as protein denaturation and lipid oxidation occurring in stored fish products (Natalia et al., 2020).

However, most previous studies apply E-nose and FTIR techniques separately or focus on limited stages of storage degradation, which restricts the ability to capture both volatile and molecular changes occurring simultaneously during fish spoilage (Çebi et al., 2023). Furthermore, integrated analytical approaches that combine volatile sensing and spectroscopic chemical characterization within a unified chemometric framework remain relatively limited. Such integration may provide a more comprehensive understanding of fish spoilage processes by linking volatile compound evolution with molecular structural changes in fish tissue.

Based on this background, the present study aims to develop a fish freshness classification approach by integrating E-nose sensing and FTIR spectroscopy combined with PCA. Freshwater catfish in Indonesia, was selected as the model species due to its economic importance and high susceptibility to quality deterioration during storage. The analysis was conducted at four storage time points (0, 12, 24, and 36 hours) to evaluate both volatile compound changes and chemical structural transformations associated with fish spoilage. The novelty of this study lies in the integration of E-nose volatile sensing and FTIR spectroscopic analysis combined with PCA to simultaneously characterize volatile and molecular changes occurring during fish spoilage across multiple storage stages.

2. Materials and Methods

2.1. Materials

The main material used in this study was freshwater catfish (Clarias gariepinus), locally known in Indonesia as “Iele”, obtained from a local market on the same day as the experiment. This species was selected because it is one of the most widely consumed freshwater fish in Indonesia and is highly susceptible to quality deterioration during storage. Aquades (distilled

water) was used to assist in the homogenization process prior to analysis. The instrumentation included an E-nose system equipped with five metal oxide semiconductor (MOS) gas sensors: MQ-3, MQ-4, MQ-136, MQ-137, and TGS-822, which were used to detect volatile compounds emitted from fish samples. In addition, FTIR was employed to analyze chemical functional groups present in the samples. Supporting equipment included a blender for sample homogenization, beaker glass and a hot plate for sample heating, an electric pump and tubing system to transfer vapor into the E-nose chamber, and a freeze dryer for removing moisture from the samples prior to FTIR analysis. A computer system was used for data acquisition and statistical analysis.

2.2. Sample Preparation

Fresh catfish were cleaned by removing internal organs, washed thoroughly with water, and dried using tissue paper. The fish flesh was then homogenized using a blender with the addition of 10 mL of aquades to facilitate the homogenization process. A total of 2000 g of homogenized fish sample was prepared and divided into four groups according to storage time: 0 hours (0 h), 12 hours (12 h), 24 hours (24 h), and 36 hours (36 h). Each storage group consisted of ten independent samples, resulting in a total of 40 samples analyzed in this study. All samples were stored at room temperature (27–29 °C) according to their assigned storage duration. After the storage period, samples intended for FTIR analysis were frozen and subsequently freeze-dried to remove moisture while preserving their chemical composition.

2.3. Experimental Setup

For E-nose measurements, approximately 50 g of homogenized fish sample was placed in a beaker and heated on a hot plate until the temperature reached 60 °C to promote the release of volatile compounds. The generated vapor was transferred into the E-nose sensor chamber using an electric pump. Before the measurement process, the E-nose sensors were allowed to stabilize in ambient air for approximately 15 minutes to establish a stable baseline signal. This stabilization step ensured consistent sensor responses before exposure to the sample volatiles. Each E-nose measurement lasted 5 minutes, and sensor readings were recorded every 5 seconds. To improve measurement reliability, each sample was measured five times under identical conditions (technical replicates). The sensor responses obtained from these five repeated measurements were averaged to reduce instrumental noise and improve signal stability. The averaged values were then used as the final dataset for subsequent statistical analysis. After each measurement, the sensor chamber was flushed with nitrogen gas for approximately 10 minutes to remove residual volatile compounds and allow the sensors to return to their baseline condition before analyzing the next sample. This procedure minimized sensor drift and cross-sample contamination. For FTIR analysis, the freeze-dried samples were analyzed using an FTIR spectrometer within a wavenumber range of 4000–400 cm^{-1} with a spectral resolution of 4 cm^{-1}. The resulting spectra were used to identify changes in chemical functional groups associated with protein degradation, lipid oxidation, and other chemical transformations occurring during storage.

2.4. Statistical Analysis

Multivariate statistical analysis was performed using PCA with Origin software to evaluate patterns in both E-nose sensor responses and FTIR spectral data. PCA was applied to reduce the dimensionality of the multivariate dataset and to visualize clustering patterns among samples stored for different durations. The PCA results were interpreted using eigenvalues, scree plots, and two-dimensional score plots, which enabled the identification of the most significant principal components explaining the variance in the data. The clustering patterns obtained from E-nose PCA were compared with those obtained from FTIR PCA to evaluate the consistency between volatile compound detection and chemical spectral changes during fish degradation. The averaged sensor signals obtained from repeated measurements were used as the input data for PCA to ensure measurement stability and minimize experimental noise.

3. Results

3.1. E-nose Analysis

3.1.1. Radar Plot

The response patterns of the five MOS sensors in the E-nose system for freshwater fish samples stored at different times (0, 12, 24, and 36 h) are presented in Figure 1. The radar plot shows that each sensor exhibited distinct response patterns across the storage intervals. Among the sensors, the MQ-3 sensor displayed the most pronounced variation in signal intensity across storage times. The difference between the readings at 0 h and 36 h was particularly evident for the MQ-3 sensor, indicating a significant change in the detected volatile compounds during storage. Other sensors, including TGS-822, MQ-4, MQ-137, and MQ-136, also showed variations in response, although the magnitude of change was smaller compared with MQ-3. These differences in sensor responses suggest that the E-nose system was able to detect variations in volatile compound profiles associated with fish storage duration. The radar plot visualization provides a comparative overview of sensor responses, highlighting the variation of volatile compound signals detected by each sensor across the four storage conditions.

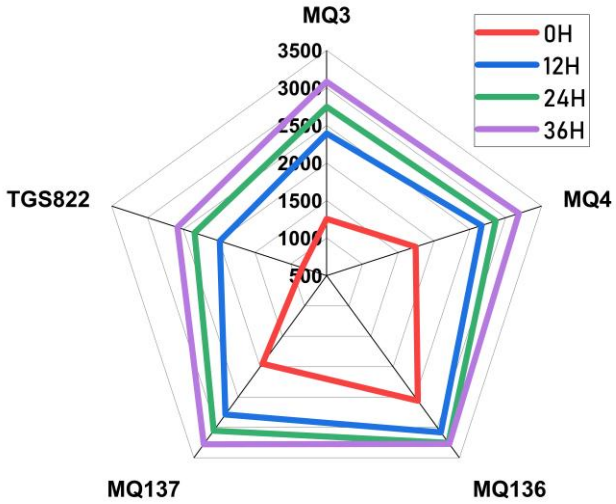

Figure 1 Radar plot of the E-nose with freshwater fish samples at different storage time variations (0, 12, 24, and 36 h).

3.1.2. Scree Plot

The PCA scree plot obtained from the E-nose sensor data is shown in Figure 2. The scree plot illustrates the relationship between eigenvalues and principal component numbers. The first principal component (PC1) showed a very high eigenvalue, close to 5, while the eigenvalues of PC2 to PC5 decreased sharply. This pattern indicates that PC1 accounts for the majority of the variance in the sensor dataset. The variance explained by PC1 was 98.5%, while PC2 contributed 0.12%, resulting in a cumulative variance of approximately 99.7% for the first two principal components. The sharp decline between PC1 and PC2 suggests that most of the variability in the E-nose sensor signals can be represented using the first principal component.

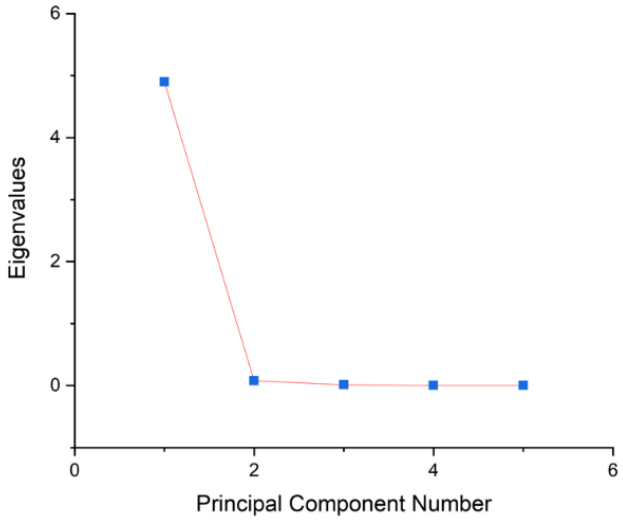

Figure 2 Scree plot of the E-nose with freshwater fish samples at different storage time variations (0, 12, 24, and 36 h)

3.1.3. PCA Biplot

The two-dimensional PCA biplot derived from the E-nose sensor data is presented in Figure 3. The plot shows the distribution of samples according to the first two principal components. PC1 explained 69.79% of the variance, while PC2 explained 30.02%, resulting in a cumulative variance of 99.8%. The PCA biplot reveals clustering patterns corresponding to the storage duration of the fish samples. Samples measured at 0 h are positioned on the negative side of PC1, while samples stored for 12 h, 24 h, and 36 h show gradual shifts along the PC axes. Some data points from the 12 h group appear slightly separated from the main cluster, approaching the position of the 24 h group. Similarly, several points from the 24 h group display a wider distribution, indicating variation within that storage interval. The loading vectors displayed in the biplot represent the contribution of each sensor to the principal components. The sensors MQ-3 and MQ-136 appear closer to the region associated with the intermediate storage samples, while MQ-4, MQ-137, and TGS-822 are oriented toward the region corresponding to the longer storage duration samples.

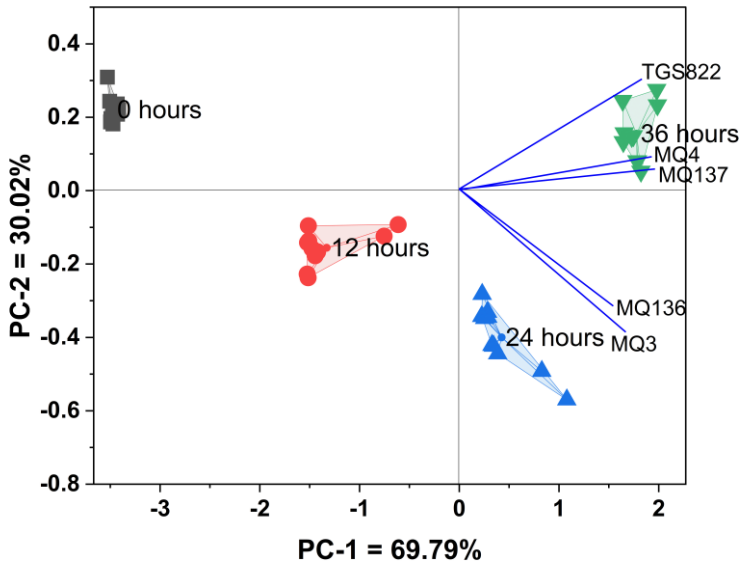

Figure 3 Biplot of the E-nose with freshwater fish samples at different storage time variations (0, 12, 24, and 36 h).

3.2. FTIR Analysis

3.2.1. Scree Plot

The scree plot derived from PCA analysis of the FTIR spectral data is shown in Figure 4. In contrast to the E-nose data, the FTIR scree plot exhibits a more gradual decline in eigenvalues from PC1 to PC3. PC1 shows the highest eigenvalue, followed by PC2 and PC3, while PC4 approaches zero. The proportion of variance explained by PC1 is 63.1%, while PC2 accounts for 26.2%. Together, the first two principal components explain approximately 89.3% of the total variance in the FTIR dataset. This cumulative variance indicates that most of the spectral variability can be represented within the first two principal components.

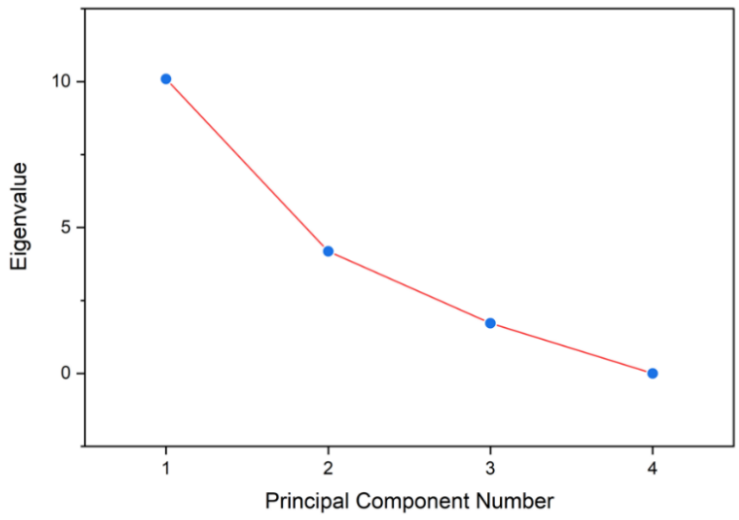

Figure 4 Scree plot of the E-nose with freshwater fish samples at different storage time variations (0, 12, 24, and 36 h).

3.2.2. PCA Score Plot

The PCA score plot derived from FTIR spectral data is presented in Figure 5. The distribution of samples along the PC1 and PC2 axes reveals separation patterns according to storage duration. Samples corresponding to 0 h are located on the negative side of PC1, forming a cluster distinct from the other storage groups. The 12 h and 24 h samples shift toward the positive side of PC1, with the 24 h samples showing higher PC2 values compared with the 12 h samples. The 36 h samples occupy a region on the positive side of PC1 but with relatively lower PC2 values, forming a distinct cluster from the other storage intervals. This distribution indicates that the PCA of FTIR spectra captures systematic variations in the spectral dataset across different storage times.

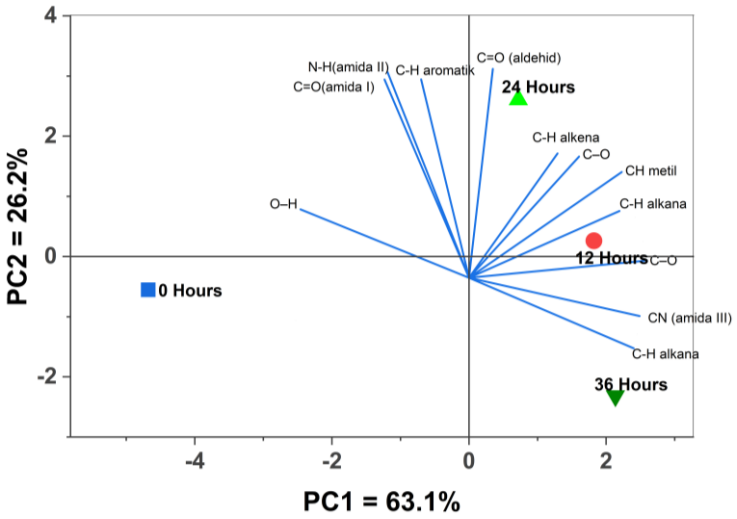

Figure 5 Score plot of the FTIR with freshwater fish samples at different storage time variations (0, 12, 24, and 36 h).

3.2.3. FTIR Spectrum

The FTIR spectra of freshwater fish samples stored at different time intervals are presented in Figure 6, covering the wavenumber range of 4000–500 cm^{-1}. Several prominent absorption bands corresponding to key functional groups can be observed. A broad absorption band appears around 3300–3400 cm^{-1}, corresponding to O–H stretching vibrations associated with water and hydroxyl-containing compounds. In the region of 3000–2850 cm^{-1}, absorption bands corresponding to C–H stretching vibrations of aliphatic groups are visible. A distinct peak near 1740 cm^{-1} is observed, corresponding to C=O stretching vibrations commonly associated with carbonyl compounds. In addition, strong absorption bands are present in the region of 1650–1540 cm^{-1}, representing the Amide I and Amide II bands related to protein structures. The region between 1200–1000 cm^{-1} shows absorption bands corresponding to C–O stretching vibrations. Variations in the intensity and shape of these absorption bands can be observed across the different storage times presented in Figure 6, indicating changes in the chemical composition of the fish samples during storage.

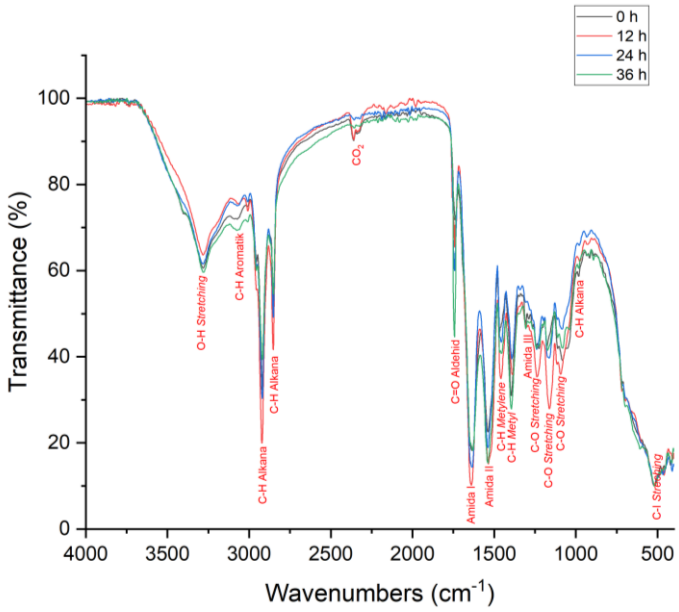

Figure 6 Functional group of the FTIR with freshwater fish samples at different storage time variations (0, 12, 24, and 36 h).

4. Discussion

4.1. E-nose Response to Fish Degradation

The E-nose analysis indicates that the volatile compound profile of catfish samples changes progressively with storage time. As shown in the radar plot (Figure 1), the responses of the MOS sensors vary across storage durations, with the MQ-3 sensor exhibiting the largest signal variation. This observation suggests that compounds associated with alcohol and related

volatile organic compounds increase as the fish undergoes spoilage. During fish storage, biochemical and microbial degradation processes lead to the production of various volatile compounds such as alcohols, aldehydes, hydrocarbons, and ammonia. These compounds are commonly associated with fish spoilage and contribute to the odor characteristics detected by gas sensor arrays. MOS-based sensors are particularly sensitive to such volatile organic compounds, which explains the increasing signal patterns observed in the E-nose measurements. Similar sensor response patterns have been reported in previous studies investigating the use of electronic noses for detecting spoilage-related volatiles in seafood products (Karunathilaka et al., 2021; Yadav et al., 2025).

The PCA analysis further demonstrates the ability of the E-nose system to capture these variations in volatile profiles. The scree plot (Figure 2) indicates that the first principal component accounts for the majority of variance in the sensor dataset. This suggests that the dominant variation among samples is associated with the increase of spoilage-related volatiles during storage. Similar PCA behavior has been reported in previous studies where E-nose sensor arrays successfully distinguished food freshness levels based on volatile compound patterns (Franceschelli et al., 2021). The clustering pattern observed in the PCA biplot (Figure 3) also shows that fish samples stored for different durations occupy distinct regions in the principal component space. This separation indicates that the volatile signals detected by the E-nose sensors are sufficiently sensitive to capture changes associated with storage-induced degradation. Comparable clustering patterns have been observed in previous studies where PCA was applied to classify freshness levels of fish and meat products using electronic nose data (Ramkumar et al., 2025).

Some dispersion observed in the clusters, particularly for the 12 h and 24 h groups, may reflect natural variability in the rate of biochemical degradation during storage. Factors such as microbial growth dynamics, enzyme activity, and slight differences in sample conditions may influence the concentration of volatile compounds detected by the sensors. Similar variability in sensor-based freshness detection has been reported in previous studies involving food spoilage monitoring (Gilman et al., 2019).

4.2. Chemical Changes Detected by FTIR

FTIR spectroscopy provides complementary information by detecting structural changes in the biochemical composition of fish tissue during storage. The PCA results derived from FTIR spectral data (Figure 4 and Figure 5) indicate that the first two principal components capture the majority of spectral variability associated with storage time. The PCA score plot (Figure 5) shows a clear separation of samples according to storage duration, indicating that chemical changes in the fish matrix occur progressively during storage. Fresh samples (0 h) appear as a distinct cluster, while samples stored for longer durations gradually shift in PCA space. Similar PCA-based separation of food samples based on FTIR spectra has been reported in studies analyzing chemical changes during food storage and degradation (Lv et al., 2018).

The FTIR spectra presented in Figure 6 reveal several important absorption bands associated with major biochemical components of fish tissue. The broad absorption band around 3300–3400 cm^{-1} corresponds to O–H stretching vibrations related to water molecules and hydrogen bonding interactions. Changes in this region may indicate alterations in water content and protein hydration structures during storage. Similar observations have been reported in previous FTIR studies examining structural changes in protein-rich food materials (Zhang et al., 2022). The Amide I and Amide II bands located in the region of 1650–1540 cm^{-1} are characteristic of protein structures. Variations in these bands are often associated with protein denaturation, peptide bond cleavage, and enzymatic degradation processes. Protein degradation is a well-known phenomenon during fish spoilage and contributes significantly to quality deterioration during storage. Previous studies have reported similar FTIR spectral changes in protein structures during storage of fish and other seafood products (Govari et al., 2022).

In addition, the carbonyl absorption band around 1740 cm^{-1} corresponds to C=O stretching vibrations commonly associated with lipid oxidation products such as aldehydes and ketones. Lipid oxidation is another important pathway contributing to fish spoilage and the formation of off-flavor compounds. Similar FTIR spectral features related to lipid oxidation have been reported in studies investigating oxidative degradation in stored seafood products (X. Wu et al., 2022).

4.3. Relationship Between E-nose Signals and FTIR Spectral Changes

The integration of E-nose and FTIR techniques provides a more comprehensive understanding of fish spoilage processes. While the E-nose detects volatile compounds released into the headspace of the sample, FTIR spectroscopy reveals molecular structural changes occurring within the fish tissue. These two techniques therefore capture complementary aspects of the degradation process. During fish storage, biochemical reactions such as protein degradation and lipid oxidation generate both structural changes in the fish tissue and volatile compounds that contribute to odor formation. Protein degradation can produce ammonia and amine compounds, whereas lipid oxidation generates aldehydes, ketones, and other volatile molecules. These compounds contribute to the volatile profile detected by the E-nose sensors while simultaneously altering the chemical functional groups observed in FTIR spectra.

The consistency between the clustering patterns observed in the PCA analysis of E-nose data (Figure 3) and FTIR spectra (Figure 5) suggests that both sensing techniques respond to the same underlying biochemical degradation processes. Similar

relationships between volatile detection systems and spectroscopic analysis have been reported in previous multi-sensor studies of food freshness evaluation (Fengou et al., 2019; Wang, 2025). The combination of volatilomic detection through E-nose and chemical structural analysis through FTIR therefore enhances the reliability of fish freshness classification. Multi-sensor approaches that integrate complementary analytical techniques have been increasingly explored in food quality monitoring because they provide more robust information about complex spoilage processes.

5. Conclusions

This study demonstrates that the integration of an E-nose system and FTIR combined with PCA can be used to evaluate the freshness degradation of freshwater catfish (Clarias gariepinus) during storage. The E-nose sensor array was able to detect variations in volatile compound profiles associated with different storage durations, with the MQ-3 sensor showing the most prominent response among the sensors used. PCA analysis of the E-nose data successfully revealed clustering patterns corresponding to the different storage intervals. The FTIR analysis further supported these observations by revealing changes in key functional groups associated with fish spoilage processes. Variations in O–H, Amide I–II, C–H, and C=O absorption bands indicated chemical transformations related to protein degradation, lipid oxidation, and changes in water content during storage. The PCA analysis of FTIR spectra also showed distinct separation among samples stored for different durations, confirming the presence of progressive chemical changes in the fish tissue. The combined analysis of E-nose and FTIR data suggests that these techniques provide complementary information regarding the volatile and chemical characteristics of fish degradation. The consistency between the clustering patterns obtained from both analytical approaches indicates that multi-sensor methods can enhance the reliability of freshness classification during storage. However, this study was limited to a single fish species and controlled laboratory storage conditions. Further studies involving different fish species, larger datasets, and additional chemometric or machine learning approaches are required to evaluate the broader applicability of the proposed method. Future work may also explore the development of portable sensing systems for real-time fish freshness monitoring in practical supply chain environments.

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6. Declarations

6.1. Ethical considerations

Not applicable.

6.2. Use of artificial intelligence (AI)

The authors declare that the generative artificial intelligence (AI) tool ChatGPT was used exclusively for language editing and/or grammatical improvement. The use of AI did not influence the scientific content, study design, data analysis, data interpretation, results, or conclusions of the manuscript. Full responsibility for the content remains with the authors.

6.3. Conflict of Interest

The authors declare no conflicts of interest.

6.4. Funding

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