

Quartz Crystal Microbalance (QCM) Sensor Array with Varying PMMA Coatings for Coffee Roasting Aroma Monitoring

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Article Info

Article history:

Received Jan 20, 2026

Revised Feb 25, 2026

Accepted Mar 18, 2026

Keywords:

Quartz Crystal Microbalance
Aroma Monitoring
PMMA Coating
Qualitative Monitoring
Sensor Array

ABSTRACT

This study investigates how polymethyl methacrylate (PMMA) coating concentration (3–15 wt%) tunes the response of an eight-channel Quartz Crystal Microbalance (QCM) sensor array to real coffee roasting volatiles at 200–240 °C. One channel was left uncoated as a reference, while seven channels were coated with different PMMA concentrations to introduce controlled response diversity. Baseline-corrected frequency shifts were processed into qualitative features describing response magnitude, kinetics, and early recovery, and principal component analysis (PCA) was used to visualize multichannel pattern structure across repeated roasts. Consistent temperature-dependent response patterns were observed, while run-to-run variability increased at higher temperatures. The first two principal components captured ~75% of the total variance (PC1 dominated by integrated response magnitude and PC2 reflecting kinetic variability). Because chamber humidity increased during roasting, a supplementary robustness check was performed using recorded RH; temperature-dependent structure remained after accounting for humidity effects. Overall, discrimination arises from the collective multichannel response, suggesting potential applicability of PMMA-coated QCM arrays for qualitative coffee roasting monitoring, pending further validation with larger datasets and complementary analytical methods..

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1. INTRODUCTION

1.1. Coffee Aroma and Roasting Process

Coffee aroma is a critical quality attribute shaped during the roasting process through a series of chemical reactions, notably the Maillard reaction, caramelization, and pyrolysis [1–3]. These processes lead to the formation of hundreds of volatile compounds, including aldehydes, ketones, esters, phenols, and pyrazines [4–6]. The composition and concentration of these volatiles depend strongly on roasting temperature, time, and bean characteristics [5] [7–9].

For decades, trained sensory panels have been the standard method for evaluating coffee aroma [10] and distinguishing it from other food aromas [11], but growing interest in faster, more objective alternatives has driven research into instrumental and sensor-based techniques. Various sensing technologies have been applied to coffee volatile analysis, from gas chromatography to electronic noses built around metal oxide, electrochemical, or optical sensors [12–15]. Polymer-based sensing materials, however, have received far less attention for coffee aroma detection, especially for monitoring the roasting process in real time. Recent reviews emphasize electronic noses as rapid, non-destructive tools for food quality control and process monitoring, while also noting practical challenges such as sensor drift and environmental cross-sensitivity (e.g., humidity) [16–18].

Quartz Crystal Microbalance (QCM) sensors, which operate based on frequency shifts upon gas molecule adsorption, provide a sensitive and reproducible approach for detecting volatile compounds [19–21]. By incorporating PMMA coatings of different concentrations, these sensors can be tailored to capture a range of coffee aroma components, improving the precision of aroma analysis [22].

1.2. QCM Technology in Aroma Detection

The QCM is a highly sensitive mass sensor that operates by measuring changes in resonant frequency due to mass loading on its surface. The relationship between frequency shift Δf and change of mass Δm is given by the Sauerbrey equation [23] [24]:

$$\Delta f = -\frac{2f_0^2 \Delta m}{A\sqrt{\rho_q \mu_q}} \quad (1)$$

where f_0 is the fundamental frequency, A is the active area, ρ_q is the density of quartz, and μ_q is the shear modulus of quartz. By coating the QCM surface with selective layers, its sensitivity toward specific volatile organic compounds (VOCs) can be tuned [19] [25].

In the present work, the Sauerbrey relation is used to support qualitative interpretation of mass loading under gas-phase conditions. However, polymer films may exhibit viscoelastic effects and environmental sensitivity; therefore, frequency shifts are interpreted as qualitative adsorption–desorption signatures rather than as absolute VOC mass quantification.

1.3. Role of PMMA Coatings

Poly(methyl methacrylate) (PMMA) is an optically transparent, chemically stable, film-forming, non-conducting polymer used in sensor applications [21]. The VOC adsorption characteristics of PMMA are influenced by several parameters including the VOC concentration, film thickness, and surface structure. It has been reported [22] that PMMA effectively adsorbs coffee VOCs, but a systematic evaluation of varying concentrations on QCM arrays remains limited.

From a physicochemical perspective, PMMA contains ester carbonyl groups that can act as hydrogen-bond acceptors and provide dipolar interaction sites, while its hydrocarbon backbone contributes van der Waals interactions. Consequently, PMMA films may interact with multiple VOC classes relevant to coffee roasting (e.g., aldehydes/ketones, acids, furans, phenolics, and nitrogen-containing heterocycles), potentially producing a broad but non-specific adsorption signature suitable for array-based pattern recognition rather than compound-specific identification [28].

A recent study reported by Nalle et al. demonstrated enhanced sensitivity using PMMA and PMMA-rGO composite coatings for coffee aroma under roasting conditions. Their results concluded that PMMA-coated QCM sensors exhibited measurable frequency shifts in response to coffee volatiles, highlighting the capability of PMMA-based coatings to interact effectively with aroma compounds released during roasting. They primarily emphasized enhancing sensitivity by modifying the coating material and analyzing peak response magnitude at the single-sensor level. As a consequence, aspects related to array-level response diversity, qualitative observability, and pattern consistency under repeated roasting conditions have received comparatively limited attention [22].

Unlike previous work that prioritized sensitivity improvement, our focus here is different. We ask whether an array of QCM sensors, each coated with a different PMMA concentration, can produce diverse and repeatable response patterns when exposed to actual coffee roasting fumes. The goal is not to identify specific compounds or measure their concentrations, but to assess whether the array as a whole generates observable, structured signals that vary with roasting conditions.

To clarify the positioning of the present study within existing research, Table 1 compares several QCM-based studies on coffee aroma sensing, focusing on differences in coating strategy, number of sensor channels, aroma source, and how the results were evaluated. Most of these studies used only one or two sensors, aimed mainly at improving sensitivity, or tested with simplified aroma samples rather than real roasting conditions. Our work takes a different approach: we use an eight-channel array where each sensor carries a different PMMA concentration, and we expose the entire array to volatile streams from an actual coffee roaster. The question is not which single sensor performs best, but whether the full array produces patterns that are diverse enough and stable enough to distinguish between roasting conditions.

Table 1. Comparison of representative QCM-based coffee aroma sensing studies.

Study	Coating material	Concentration series	Number of channels	Aroma source	Evaluation focus	Multivariate / feature-based analysis
[22]	PMMA, PMMA-rGO	No (single coating)	Single sensor	Real coffee roasting stream	Sensitivity enhancement; single-sensor configuration (no array diversity)	No
[26]	Multiple polymer coatings	No	4-channel QCM array	Generic VOCs (non-coffee)	Classification	Yes (LDA, SVM)
[21]	Polystyrene-based coating	No	Not clearly specified	Coffee aroma	Descriptive response analysis	Not reported
[27]	Unspecified coating	No	QCM array	Coffee aroma (Robusta)	Qualitative sensing	Not reported
This work	PMMA-coated QCM array	Yes (3–15 wt%)	8-channel array	Real coffee roasting stream	Qualitative observability and response diversity	Yes (feature extraction + PCA)

2. MATERIALS AND METHOD

2.1. Sensor Fabrication

Eight AT-cut quartz crystal resonators with a nominal fundamental frequency of 10 MHz were used as sensing elements. Prior to coating, each crystal was cleaned with toluene and dried at room temperature. PMMA solutions were prepared in toluene at seven concentrations: 3, 5, 7, 9, 11, 13, and 15 wt%. Each polymer solution was spin-coated onto the QCM crystal (500 rpm for 5 s, then 3000 rpm for 60 s with 50 μ L dispensed) and thermally annealed at 100 °C for 1 hour. The eighth crystal was left uncoated to serve as a reference channel. The channel assignments and corresponding PMMA concentrations are listed in Table 2.

Film thickness was not directly measured in this study and was estimated from prior in-house characterization of the same PMMA–toluene system, which reported a linear concentration–thickness relationship ($R^2 = 0.98$) with thicknesses of approximately 5–458 nm for 1–11 wt% PMMA [28]. Here, the concentration range was extended to 15 wt%; thicknesses above 11 wt% were not independently verified and are reported as approximate extrapolations.

Table 2. Mapping of QCM channels and PMMA coating concentrations used in the sensor array

Channel	PMMA concentration (wt%)
Ch1	Uncoated
Ch2	3
Ch3	5
Ch4	7
Ch5	9
Ch6	11
Ch7	13
Ch8	15

2.2. Experimental Setup

Coffee beans of 150 g were roasted at predefined target temperatures of 200, 215, 220, and 240 °C. This quantity was kept constant for all measurements to standardize aroma generation conditions. Volatile compounds released during roasting were transferred from the roaster to a sealed sensing chamber housing the eight-channel QCM array.

The experimental setup is shown in Figure 1. A closed lab-scale roaster with computer-controlled heating was connected to the sensing chamber through a valve-controlled transfer line. Two solenoid valves regulated gas flow: one directed roasting volatiles toward the sensor chamber during the exposure phase, and the other allowed purging with ambient air afterward. The sensing chamber was positioned approximately 50 cm from the heat source to minimize direct thermal effects on the QCM sensors.

Each QCM sensor was connected to an oscillator circuit and a frequency counter, enabling continuous, real-time measurement of resonance-frequency shifts. Frequency data from all sensor channels were transmitted to a personal computer via a data acquisition and control unit for logging and visualization. The system was designed to allow simultaneous exposure of all QCM channels under the same experimental configuration and protocol, enabling qualitative comparison of response patterns across different PMMA coating concentrations.

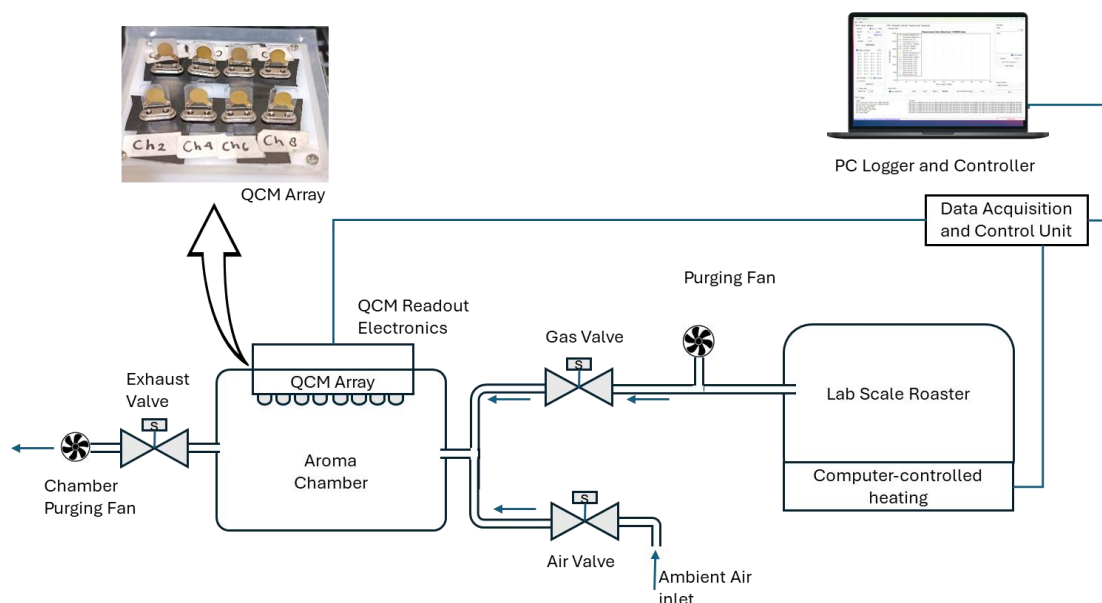


Figure 1. Schematic of the experimental setup for qualitative coffee aroma sensing using a multi-channel QCM array, including the laboratory-scale roaster, valve-controlled aroma transfer line, and separated sensing chamber housing the QCM array.

2.3 Measurement Protocol

For each target roasting temperature condition, repeated experiments were performed (overall $n = 4$ – 6 per temperature). For multivariate analyses that require consistent feature and humidity records, a balanced subset of runs was used ($n = 16$ total; $n = 4$ per temperature at 200, 215, 220, and 240 °C). Relative humidity (RH) and the chamber temperature were recorded but not actively controlled; therefore, the results are interpreted as a qualitative feasibility assessment under practical roasting variability rather than under strictly controlled environmental conditions.

Each experiment followed a consistent three-phase protocol: baseline, gas-on, and recovery. Prior to roasting, the heater was off and the exhaust pathway was activated to remove residual volatiles from previous runs. The bean sample (150 g) was loaded into the roaster, QCM frequency acquisition was initiated, and sensor signals were allowed to stabilize under ambient conditions. The roaster temperature was then increased gradually to 140 °C while aroma vapors were continuously vented away from the sensing chamber. After reaching 140 °C, venting was maintained for an additional 10 minutes to remove early-stage volatiles formed at low temperatures.

Following this period, the aroma transfer pathway was opened to initiate the gas-on phase, allowing coffee aroma vapors to enter the QCM sensing chamber. The chamber was exposed to the aroma stream for an initial 5 min, after which the roaster temperature was adjusted to the target roasting temperature ($X = 200, 215, 220, \text{ or } 240$ °C) and maintained for an additional 20 min while continuous sensing was performed (total gas-on duration = 25 min). The heating process was then stopped to terminate the gas-on phase.

The system was allowed to cool naturally at room temperature, defining the recovery phase, during which sensor frequencies were recorded until partial baseline recovery was observed. Based on this protocol, the measurement phases were defined as follows:

- Baseline phase: from the start of data acquisition until initiation of aroma transfer
- Gas-on phase: from the opening of the aroma transfer pathway through the roasting period at the target temperature
- Recovery phase: from termination of heating until cooling at room temperature

This three-phase measurement protocol was applied consistently across all roasting temperatures and experimental runs to ensure compatibility with the signal-processing and feature-extraction procedures described in Section 2.4. For clarity and reproducibility, the key experimental conditions and measurement parameters applied consistently across all roasting runs are summarized in Table 3.

Table 3. Summary of experimental conditions and measurement protocol

Parameter	Value / Description
Coffee origin	Malang, Indonesia
Coffee species	Robusta
Coffee mass	150 g
Roasting temperatures	200, 215, 220, 240 °C
Number of runs	n = 4–6 per temperature (200 °C: 5; 215 °C: 6; 220 °C: 4; 240 °C: 4)
Sensor type	AT-cut QCM, 10 MHz
Coating material	PMMA (3–15 wt%)
Number of channels	8 (1 uncoated + 7 coated)
Measurement phases	Baseline – Gas-on – Recovery
Gas-on duration	25 min
Recovery condition	Natural cooling at room temperature
Chamber mixing	Internal fan (fixed speed)
Environmental control	Not actively controlled

2.4 Data Processing and Exploratory Analysis

2.4.1 Signal definition and baseline correction

The QCM sensor output is recorded as a time series of resonance frequency for each channel. To ensure consistency across channels and roasting conditions, all responses are expressed as frequency shifts relative to a pre-exposure baseline. For the i -th QCM channel, the frequency shift is defined as

$$\Delta f_i(t) = f_i(t) - \bar{f}_{i,0}, \quad (2)$$

where $f_i(t)$ is the measured resonance frequency at time t , and $\bar{f}_{i,0}$ is the mean baseline frequency calculated over a stable pre-roasting interval. The baseline mean and baseline noise level are given by

$$\bar{f}_{i,0} = \frac{1}{N_0} \sum_{t \in \mathcal{B}} f_i(t), \quad (3)$$

$$\sigma_{i,0} = \sqrt{\frac{1}{N_0 - 1} \sum_{t \in \mathcal{B}} (f_i(t) - \bar{f}_{i,0})^2}, \quad (4)$$

where $\mathcal{B}$ denotes the set of time indices corresponding to the baseline phase and N_0 is the number of baseline samples.

2.4.2 Feature extraction

Each frequency response curve is segmented into three sequential phases: (i) baseline, prior to aroma exposure; (ii) gas-on phase, during which coffee roasting aroma is introduced and frequency decreases due to mass loading; and (iii) recovery phase, when aroma exposure ceases and the frequency gradually returns toward baseline. Feature extraction is performed separately on these phases to characterize response magnitude, temporal dynamics, and recovery behavior.

Several qualitative features are extracted from the baseline-corrected frequency responses to describe sensor behavior:

1) Maximum frequency shift. The response magnitude of the i -th channel is quantified by the maximum frequency shift during the gas-on phase:

$$\Delta f_{\max,i} = \min_{t \in \mathcal{G}} \Delta f_i(t) \quad (5)$$

where $\mathcal{G}$ denotes the gas-on interval. While mass loading produced negative frequency shifts, the magnitude $|\Delta f_{\max,i}|$ is used for comparative visualization.

2) Area under the curve (AUC). The overall response intensity and duration are characterized by the area under the frequency shift curve during the gas-on phase:

$$\text{AUC}_i = \sum_{t \in \mathcal{G}} |\Delta f_i(t)| \Delta t \quad (6)$$

where Δt is the sampling interval.

3) Initial response slope. The initial response rate is estimated from the early portion of the gas-on phase over a time window of length τ :

$$S_i = \frac{\Delta f_i(t_{\text{on}} + \tau) - \Delta f_i(t_{\text{on}})}{\tau} \quad (7)$$

where t_{on} denotes the onset time of aroma exposure.

4) Time to peak response. The time required for the sensor to reach its maximum response is defined as

$$\begin{aligned} T_{\text{peak},i} &= t_{\text{peak},i} - t_{\text{on}}, \\ t_{\text{peak},i} &= \arg \min_{t \in \mathcal{G}} \Delta f_i(t). \end{aligned} \quad (8)$$

5) Recovery progress within the first 50 s (R50). The recovery progress of the i -th sensor channel within the first 50 samples after termination of aroma exposure (corresponding to 50 s at a 1 Hz sampling rate) is defined as

$$R_{50,i} = \frac{\Delta f_i(t_{\text{off}} + 50\Delta t) - \Delta f_{\text{max},i}}{0 - \Delta f_{\text{max},i}}, \quad (9)$$

where t_{off} denotes the end of the gas-on phase and Δt is the sampling interval. The fixed-window recovery metric provides an operational descriptor of early post-exposure relaxation: positive values indicate partial recovery toward baseline within the first 50 s, while negative values may occur when the response continues to evolve (e.g., delayed transport or continued adsorption) immediately after gas-off. This fixed-window approach avoids ambiguity associated with incomplete or irregular long-term recovery curves and is suitable for qualitative feasibility assessment under realistic roasting conditions.

6) Signal-to-noise ratio. To assess signal discernibility relative to baseline fluctuations, the signal-to-noise ratio is defined as

$$\text{SNR}_i = \frac{|\Delta f_{\text{max},i}|}{\sigma_{i,0}} \quad (10)$$

SNR was computed to verify that all channels produced responses well above baseline noise levels, but was not included in the multivariate feature vector to avoid redundancy, since it is directly derived from $|\Delta f_{\text{max}}|$ and baseline noise, both of which are already captured by the primary features.

2.4.3 Exploratory multivariate analysis

The extracted qualitative features are treated as descriptive representations of sensor behavior rather than variables for formal hypothesis testing. For multivariate analysis, the channel-wise features obtained from each roasting test are concatenated into a single array-level feature vector. Using the channel index $i = 1, \dots, 8$, the feature vector for a given test is defined as:

$$x_{\text{run}} = [|\Delta f_{\text{max},1}|, \dots, |\Delta f_{\text{max},8}|, \text{AUC}_1, \dots, \text{AUC}_8, S_1, \dots, S_8, T_{\text{peak},1}, \dots, T_{\text{peak},8}, R_{50,1}, \dots, R_{50,8}]$$

This array-level representation preserves both the heterogeneous response characteristics of individual sensor channels and their collective behavior under identical roasting conditions.

Prior to multivariate analysis, all feature dimensions are standardized using global z-score normalization:

$$z_{n,k} = \frac{x_{n,k} - \mu_k}{\sigma_k}, \quad (11)$$

where $x_{n,k}$ denotes the value of feature k for sample n , and μ_k and σ_k are the mean and standard deviation of feature k computed across all runs, respectively. This standardization ensured comparable scaling across features with different physical units and dynamic ranges.

Principal component analysis (PCA) is then applied to the standardized feature matrix to visualize qualitative variations in aroma response patterns under different roasting conditions. PCA is employed here as an exploratory visualization tool rather than for predictive classification or statistical inference.

This section defines the preprocessing, feature construction, and normalization pipeline used in the multivariate analyses. Beyond exploratory PCA, Section 3.5 reports PERMANOVA/PERMDISP and a LOOCV LDA classifier as an illustrative robustness check; given the limited sample size and practical variability, LDA results are interpreted descriptively. For LOOCV LDA, z-score normalization was computed within each training fold and applied to the held-out run to avoid information leakage.

2.4.4 Assessment of humidity influence and compensation

Coffee roasting releases considerable water vapor, and PMMA is known to exhibit cross-sensitivity to humidity. Therefore, a supplementary analysis was performed to assess whether the observed temperature-

dependent patterns could be explained by humidity fluctuations alone. For the balanced subset of runs ($n = 16$; $n = 4$ per temperature) with available humidity sensor records, the mean RH during baseline and gas-on phases was computed, and the RH change (ΔRH) was used as an environmental covariate. Feature residualization was performed by regressing each feature dimension against ΔRH and using the residuals (followed by z-score normalization) for subsequent multivariate analyses. In addition, reference-channel normalization using the uncoated QCM channel was considered to reduce common-mode environmental drift. These steps were used to evaluate robustness of the multivariate pattern structure, not for compound-specific VOC identification.

3. RESULTS AND DISCUSSION

3.1. Real-time response behavior of the multi-channel QCM array

Figure 2 shows representative time-resolved frequency shift responses of the eight-channel QCM array during coffee aroma exposure at a roasting temperature of 240 °C. Prior to aroma exposure, all sensor channels exhibit stable baseline behavior. Upon opening of the aroma valve ($t = 0$), a rapid decrease in resonance frequency was observed across all channels, indicating adsorption of coffee roasting volatiles. The response magnitude and temporal evolution differed markedly among channels, reflecting response diversity associated with different PMMA coating concentrations. Following termination of aroma exposure, partial recovery toward the baseline was observed, with recovery rates varying between channels.

Although all channels responded to aroma exposure, striking differences were observed in response magnitude and temporal evolution. The uncoated reference channel and the PMMA-coated channel exhibited distinct response trajectories, including variations in initial response rate, signal evolution during roasting, and initial recovery behavior after cessation of aroma exposure. These differences indicated that varying the PMMA coating concentration could introduce response variability across the sensor array. Such variability was advantageous for sensor selection in array-based sensing, as it could yield multichannel response patterns rather than a simple peak signal magnitude of each sensor.

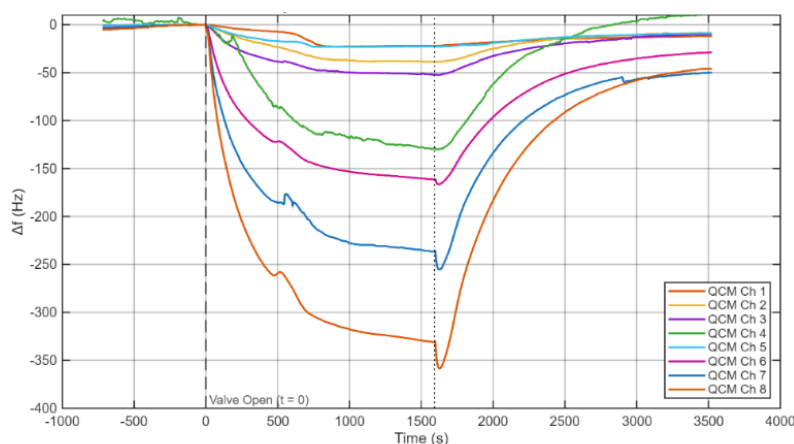


Figure 2. Representative time-resolved frequency shift responses (Δf) of the eight-channel QCM array during coffee aroma exposure at a roasting temperature of 240 °C.

During the recovery phase, partial signal relaxation toward baseline was observed with different rates across channels. Under roasting conditions, complete recovery was not always achieved due to residual volatiles, chamber adsorption effects, and environmental fluctuations. Consequently, early-window recovery descriptors, as defined in Section 2.4, were relevant for characterizing post-exposure response behavior. These descriptors enabled rapid assessment of recovery tendencies without requiring full equilibrium restoration.

The real-time response profiles in Figure 2 demonstrate that the PMMA-coated multi-channel QCM array generated stable and distinguishable response patterns during coffee roasting. This confirmed the feasibility of capturing dynamic aroma information in real time and provided the experimental foundation for temperature-dependent analysis (Section 3.2), qualitative feature comparison across channels (Section 3.3), and multivariate pattern visualization (Section 3.4).

3.2. Influence of roasting temperature on sensor response patterns

Figure 3 compares the frequency-shift responses of the QCM sensor array recorded at different roasting temperatures, showing that temperature affected both response magnitude and temporal characteristics. At lower temperatures, the sensor responses were relatively moderate and exhibited smoother temporal evolution across channels. As the roasting temperature increased, the frequency shifts became more pronounced, with steeper response slopes and increased divergence among channels. This behavior reflected

the intensified generation of volatile compounds at elevated temperatures, driven by enhanced thermal degradation and Maillard reactions. These processes led to higher release rates and concentrations of volatiles, captured by the QCM array as increased mass loading and faster response kinetics. The growing separation among PMMA-coated channels further indicated that coating concentration influenced not only sensitivity but also the temporal response behavior under intensified aroma exposure.

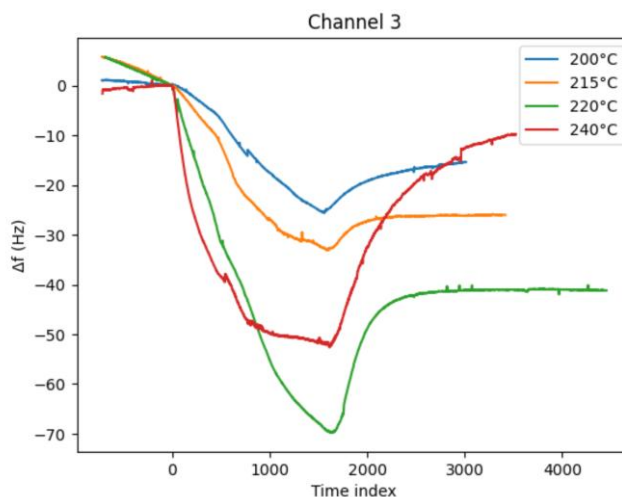


Figure 3. Temporal frequency shift (Δf) response of QCM channel 3 under different coffee roasting temperatures (200, 215, 220, and 240 °C), illustrating temperature-dependent variations in response magnitude and temporal behavior.

The recovery behavior following termination of aroma exposure was likewise temperature dependent. At higher roasting temperatures, recovery profiles tend to exhibit slower initial relaxation and greater variability across channels, likely due to stronger adsorption interactions, elevated residual vapor levels, and prolonged desorption within the measurement chamber. Under such conditions, complete recovery within a short observation window was not always achieved. This reinforced the relevance of early-window recovery descriptors for comparative analysis, as they enabled characterization of response behavior without requiring full baseline restoration. From a pattern-recognition perspective, these temperature-modulated response and recovery characteristics enhanced the discriminative richness of the sensor array. Rather than acting solely as a disturbance factor, temperature contributed structured variations that could be exploited to characterize roasting conditions through reproducible multi-channel response patterns.

Overall, the results in Figure 3 demonstrate that roasting temperature played a critical role in shaping both the amplitude and dynamics of the QCM sensor responses. These findings supported the use of multi-channel, polymer-coated QCM arrays for capturing temperature-dependent aroma patterns, which were further examined through qualitative feature comparison and multivariate visualization.

3.3. Qualitative feature comparison across PMMA-coated channels

To further examine response diversity within the sensor array, qualitative features were extracted from the frequency response profiles and summarized in Table 4. The selected features characterized key aspects of the sensor behavior, including response magnitude, temporal evolution during aroma exposure, and early recovery behavior after termination of the gas flow. Rather than focusing on absolute sensitivity alone, these features were selected to capture inter-channel variability that reflected differences in PMMA coating concentration and adsorption-desorption dynamics.

The maximum frequency shift provided a coarse measure of overall mass loading, and its values differed consistently across channels. While higher PMMA concentrations generally led to larger response magnitudes, the relationship was not strictly monotonic, indicating that factors beyond simple coating thickness, such as film morphology and diffusion effects, also influenced the observed responses. Similarly, features describing response duration revealed variations in how long each channel maintained elevated signal levels during roasting, further highlighting heterogeneous temporal behavior across the array.

The recovery descriptor requires particular clarification. Rather than measuring the time required for full return to baseline, which was rarely achieved under real roasting conditions due to residual volatiles and slow desorption from polymer films, the recovery feature quantifies the degree of signal relaxation within a fixed 50-sample window immediately after termination of aroma exposure. This early-window approach

provided a consistent and comparable metric across all channels and runs, without being influenced by the irregular profiles that longer recovery traces often exhibited.

The qualitative features in Table 4 reveal structured yet non-uniform response characteristics among PMMA-coated channels. The combination of differences in response magnitude, temporal persistence, and early recovery behavior confirmed that the array encoded aroma information through multi-dimensional patterns rather than through a single dominant metric. These results provided the basis for subsequent multivariate analysis, where the collective feature space is examined to assess pattern separability across roasting conditions.

Table 4. Summary statistics of qualitative response features extracted from individual QCM channels at 240 °C across independent roasting runs ($n = 4$). Mean values and standard deviations are reported to assess run-to-run repeatability and channel-to-channel heterogeneity.

Channel	$ \Delta f_{max} $ (Hz)	AUC (Hz·s)	Initial slope (Hz/s)	Tpeak (s)	R50
Ch1	56.6 ± 37.0	51232 ± 27908	0.049 ± 0.039	1477 ± 365	0.022 ± 0.026
Ch2	72.9 ± 31.1	66599 ± 18886	0.088 ± 0.047	1622 ± 74	0.026 ± 0.027
Ch3	71.8 ± 21.2	86335 ± 31020	0.177 ± 0.101	1636 ± 42	0.023 ± 0.022
Ch4	172.8 ± 93.7	205831 ± 130332	0.206 ± 0.222	1619 ± 43	0.017 ± 0.030
Ch5	40.3 ± 28.0	49038 ± 32434	0.137 ± 0.100	1490 ± 311	0.034 ± 0.027
Ch6	169.2 ± 75.2	217422 ± 118699	0.696 ± 0.530	1639 ± 34	-0.007 ± 0.043
Ch7	238.3 ± 97.5	316287 ± 142631	1.130 ± 0.668	1643 ± 35	-0.059 ± 0.042
Ch8	349.0 ± 161.3	463084 ± 225454	1.572 ± 1.010	1642 ± 36	-0.058 ± 0.043

3.4. Multivariate visualization of aroma response patterns

To examine the collective behavior of the extracted qualitative features, principal component analysis (PCA) was applied to the balanced 16-run dataset ($n = 4$ per temperature) used for the humidity-robustness analysis. Prior to PCA, z-score feature normalization was applied to ensure that differences in scale did not dominate the analysis. PCA is used here as an exploratory visualization tool rather than as a classifier, with the aim of assessing pattern structure and relative separability across conditions. Figure 4 presents the resulting score plots, providing a low-dimensional visualization of the multi-channel response patterns obtained at different roasting temperatures.

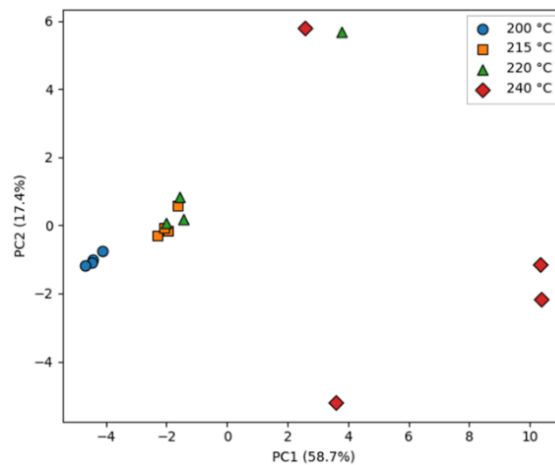


Figure 4. Principal component analysis (PCA) of qualitative aroma response features extracted from multi-channel QCM measurements under different roasting temperatures.

Score plots indicated temperature-dependent clustering of runs in the principal component space. Lower roasting temperatures exhibited tighter within-temperature grouping, whereas higher temperatures showed increased within-group spread and clearer separation along the dominant components. As the roasting temperature increased, the spread of the data became more pronounced, with clearer separation along the dominant principal components. This expansion reflected the increasing diversity in response magnitude, temporal characteristics, and recovery behavior captured by the qualitative features as aroma generation intensified.

Specifically, the response associated with the highest roasting temperature exhibited the greatest dispersion in the PCA space. Rather than indicating instability, this behavior is consistent with previous observations in Sections 3.2 and 3.3, where higher temperatures led to stronger adsorption effects, slower recovery tendencies, and greater inter-channel variability. Under such conditions, the sensor array is exposed

to a more complex and dynamic volatile mixture, and the resulting response patterns naturally occupied a wider feature space. The PCA visualization thus captured this structured variation without requiring explicit compound-level recognition.

Overall, the multivariate visualization in Figure 4 demonstrates that the PMMA-coated QCM array produces reproducible, temperature-dependent response patterns that can be distinguished within a smaller feature space. It supported the feasibility of using physically interpretable qualitative features for pattern-based monitoring of the coffee roasting process, providing an initial indication that further development toward machine learning-based classification may be warranted with expanded datasets.

Table 5. Dominant PCA loadings for PC1, highlighting features associated with overall response magnitude and integrated adsorption intensity.

Rank	Feature	Loading (PC1)
1	AUC_3	+0.203
2	AUC_2	+0.201
3	$ \Delta f_{max,3} $	+0.199
4	$ \Delta f_{max,6} $	+0.196
5	$ \Delta f_{max,8} $	+0.194
6	$ \Delta f_{max,2} $	+0.194
7	AUC_8	+0.194
8	AUC_7	+0.193
9	$ \Delta f_{max,7} $	+0.193
10	$R50_7$	-0.193

Table 6. Dominant PCA loadings for PC2, highlighting channel-specific variations in response kinetics and run-to-run variability.

Rank	Feature	Loading (PC2)
1	$ \Delta f_{max,5} $	+0.347
2	AUC_5	+0.345
3	$ \Delta f_{max,1} $	+0.324
4	AUC_1	+0.308
5	S_1	+0.281
6	S_5	+0.266
7	$T_{peak,1}$	+0.211
8	$T_{peak,5}$	+0.207
9	$T_{peak,4}$	+0.185
10	$T_{peak,2}$	+0.177

The contributions of dominant features to the two principal components of the PCA are summarized separately in Table 5 (PC1) and Table 6 (PC2). For the 16-run dataset, the first principal component (PC1) explains 57.1% of the total variance and is dominated by integrated response magnitude features (AUC) and peak response features ($|\Delta f|_{max}$) across several QCM channels. The second principal component (PC2) explains 17.2% of the variance (PC1+PC2 cumulative 74.3%), while inclusion of PC3 increases the cumulative explained variance to 82.2%. PC2 is determined by a combination of response magnitude and kinetics features, including AUC, initial slope (df/dt), and recovery-related descriptors, indicating that it captures variations in adsorption dynamics and inter-run differences, particularly at higher temperatures. Although certain channels exhibit stronger contributions to specific components, no single channel dominates the discrimination of responses across roasting temperatures. These results indicate that discrimination arises from the collective multichannel response, supporting the use of a QCM sensor array for aroma characterization during roasting.

3.5. Humidity influence and robustness of temperature-dependent patterns

Humidity in the sensing chamber increased markedly during roasting, from $\sim 65.7 \pm 10.0\%$ at baseline to $95.1 \pm 3.7\%$ during the gas-on phase. To assess potential confounding, we analyzed the balanced 16-run subset (4 runs per temperature) and removed the linear contribution of ΔRH (gas-on minus baseline) from the feature space prior to multivariate testing. Temperature remained a significant factor both before and after adjustment (PERMANOVA: pseudo- $F(3,12)=4.88$, $p=0.0001$, $R^2=0.550$; after ΔRH residualization: pseudo- $F(3,12)=4.57$, $p=0.0001$, $R^2=0.533$; 9999 permutations). However, within-temperature dispersion differed significantly in both cases (PERMDISP: $p=0.0175$ before and $p=0.0017$ after adjustment), indicating greater multivariate spread at higher roasting temperatures and warranting caution when interpreting centroid separation alone.

To provide an initial supervised benchmark, a leave-one-out LDA (Fisher's classical LDA using SVD decomposition, with per-fold z-score normalization) achieved 43.8% classification accuracy on the raw features and 62.5% after Δ RH residualization. Given the small sample size ($n = 16$) and the high feature-to-sample ratio, these values are reported as illustrative rather than as a validated classification benchmark. Nevertheless, the improvement after removing the linear humidity contribution, together with the significant PERMANOVA result, indicates that the temperature-associated patterns are not solely attributable to rising chamber humidity. The larger within-group scatter at high temperatures is consistent with the increased inter-run variability observed in Sections 3.2 and 3.3.

3.6. Practical considerations: recovery time, fouling, and positioning versus GC–MS

For practical deployment in roasting environments, sensor cycle time is constrained by recovery. In addition to the early-window recovery descriptor R50, an operational recovery time metric T50 was evaluated as the time required for a channel to recover 50% of its gas-off response magnitude. Table 7 summarizes array-level T50 values (mean across coated channels Ch2–Ch8), computed across all available runs per temperature ($n = 4$ –6). Recovery times were on the order of several minutes, while full return to baseline was not achieved within the recorded window in most runs. The T50 values also did not follow a monotonic trend across temperatures and exhibited substantial run-to-run variability. These characteristics illustrate why T50 was not adopted as a multivariate feature: its dependence on achieving a fixed recovery threshold makes it unreliable when recovery is incomplete or irregular. By contrast, the fixed-window descriptor R50 (Section 2.4.2) provides a consistent and always-computable metric regardless of whether full or partial recovery is achieved, making it more suitable for array-level pattern analysis under realistic roasting conditions.

Table 7. Array-level operational recovery time (T50) across roasting temperatures, computed as the mean across coated channels (Ch2–Ch8)

Roasting temperature (°C)	n	T50, array mean (s)
200	5	739 ± 58
215	6	391 ± 171
220	4	477 ± 133
240	4	595 ± 29

Coffee roasting also emits oils and particulates that may contaminate polymer coatings and cause long-term drift (sensor poisoning). The present work focuses on repeatability across multiple runs per temperature but does not constitute a lifetime study. Accordingly, fouling resistance and cleaning/conditioning protocols are identified as important future work; practical mitigation includes particulate filtration, condensate traps upstream of the sensor chamber, and periodic purge/thermal conditioning with re-calibration using a reference channel.

From an application perspective, these observations position the proposed QCM array as a rapid, process-oriented sensing tool rather than as a replacement for compound-specific analytical techniques such as GC-MS. Whereas GC-MS provides detailed VOC identification but is generally offline and time-consuming, the PMMA-QCM array provides a real-time qualitative response pattern that is potentially useful for roasting-process monitoring. Within the scope of the present study, the proposed system can be regarded as a complementary tool for qualitative, pattern-based aroma monitoring under realistic roasting conditions.

3.7. Comparative Discussion and System-Level Implications

The observed frequency shifts across the PMMA-coated channels confirm effective interaction between the selected coating material and coffee roasting VOCs, consistent with previous reports that polymer–VOC affinity is influenced by solubility-driven interactions [20]. Since coffee roasting generates a chemically complex mixture containing many polar compounds, the consistent adsorption-induced responses observed in the present study support the suitability of PMMA as a sensing layer for qualitative aroma monitoring under real roasting conditions. A comparison with representative coffee aroma sensing studies is summarized in Table 8.

In terms of response magnitude, the present results are quantitatively comparable with previous QCM-based coffee aroma sensing studies. Nalle et al. [22] reported a maximum response magnitude of 66.05 ± 28.77 Hz for a PMMA-coated QCM during Robusta roasting at 220 °C, whereas Sakti et al. [21] reported approximately 73.4 Hz for a polystyrene-coated QCM under static headspace conditions. The response of Ch3 in the present work (71.8 ± 21.2 Hz) is in close agreement with these values, indicating that the baseline sensitivity of polymer-coated QCM sensors remains broadly consistent across different coating chemistries and exposure configurations.

Beyond this baseline agreement, the primary contribution of the present work lies in the expansion of the sensing response space through a multi-channel PMMA concentration series. Unlike previous single-sensor

approaches, the proposed array spans a substantially wider dynamic range (71.8–349.0 Hz) and captures distinct inter-channel response kinetics and early recovery behavior. This structured response diversity enables the construction of a multi-dimensional feature space based on $|\Delta f|_{\max}$, AUC, slope, T_{peak} , and R_{50} , allowing roasting conditions to be represented as array-level fingerprints rather than as isolated peak-response values.

From a classification perspective, the extracted feature space exhibits meaningful structure, as reflected by the PCA organization and the significant PERMANOVA results. At the same time, the supervised analysis presented here should be interpreted cautiously. The moderate LDA accuracy obtained on the balanced subset indicates preliminary feasibility, but does not yet constitute a validated classification benchmark because of the limited sample size and the high feature-to-sample ratio. In this respect, the present results support the existence of structured separability in the data rather than the establishment of a finalized high-performance classifier.

This distinction is important when comparing the present study with Julian et al. [26], who reported LDA and SVM accuracies of 98% and 99%, respectively, for a four-channel polymer-coated QCM array exposed to seven pure VOCs under controlled laboratory conditions. That classification task is substantially simpler than real coffee roasting, where the volatile composition evolves continuously with temperature and is accompanied by humidity and process-related variability. The lower supervised performance observed here should therefore be interpreted primarily as a consequence of analyte complexity and dataset limitations, rather than as a limitation of the sensing platform itself.

A broader comparison can also be made with MOS-based coffee aroma studies such as Zulfikar et al. [28]. Although MOS arrays operate through a different sensing mechanism and are therefore not directly comparable to QCM in terms of frequency-shift sensitivity, they support the same general conclusion: real coffee aroma contains exploitable multivariate structure that can be accessed through array-level pattern recognition. For this reason, it is included here as a cross-technology reference for classification feasibility rather than as a direct benchmark for QCM performance.

Overall, the proposed 8-channel PMMA-QCM array achieves sensitivity comparable to earlier QCM-based coffee aroma studies while providing a substantially richer description of response magnitude, kinetics, and early recovery under realistic roasting conditions. These results support its potential as a structured qualitative monitoring platform and as a basis for future supervised-learning development. The remaining gap relative to controlled-condition studies highlights the need for larger labelled datasets, stronger humidity and drift compensation, and more advanced learning models in subsequent work.

Table 8. Performance-oriented comparison of representative coffee aroma sensing studies.

Study	Sensor type	Measurement condition	Representative response/performance	Analysis focus	Main relevance to this work
Sakti et al. [21]	QCM, polystyrene-coated	Static headspace	~73.4 Hz	Descriptive response	Comparable single-sensor magnitude
Nalle et al. [22]	QCM, PMMA-coated	Real roasting	66.05 ± 28.77 Hz	Peak response magnitude	Comparable PMMA-QCM sensitivity
Julian et al. [26]	4-channel polymer-coated QCM array	Pure VOCs, controlled lab	LDA 98%; SVM 99%	Classification	Benchmark for controlled-condition ML performance
Zulfikar et al. [28]	MOS array	Real coffee aroma	RSD < 5%	PCA + neural network	Cross-technology reference for pattern recognition
This work	8-channel PMMA-QCM array	Real roasting	71.8–349.0 Hz	Feature-based qualitative monitoring	Multi-channel response diversity under realistic roasting

4. CONCLUSION

This work demonstrates the feasibility of using an eight-channel Quartz Crystal Microbalance (QCM) sensor array with polymethyl methacrylate (PMMA) coatings of varying concentrations for qualitative monitoring of coffee roasting aroma. Across repeated roasting runs, the array produced consistent, temperature-dependent response patterns, indicating that multichannel QCM sensing can capture structured variations associated with roasting conditions in real time.

By systematically varying PMMA concentration, the array encoded aroma information through heterogeneous response magnitude, kinetics, and recovery behavior rather than a single sensitivity metric. Feature-based multivariate analysis confirmed that PC1 and PC2 captured 74–76% of total variance ($PC1+PC2 = 74.3\%$ for the 16-run dataset; the first three PCs exceeded 80%), supporting pattern-based discrimination across roasting temperatures. Because chamber humidity increased strongly during roasting, a supplementary robustness check using recorded RH (ΔRH residualization) confirmed that the temperature effect remains significant (PERMANOVA $R^2 \approx 0.53\text{--}0.55$, $p < 0.001$), while dispersion increased markedly at higher temperatures (PERMDISP $p < 0.05$), which may reflect more dynamic volatile release and increased process

variability. From a practical standpoint, operational recovery times (T50) were on the order of minutes under the present protocol, highlighting the need for purge and chamber design optimization. Future work will address long-term fouling resistance and calibration stability, and will focus on FPGA-based embedded classification, multi-polymer coating strategies, and broader aroma applications to advance the present proof-of-concept toward a portable electronic nose system.

ACKNOWLEDGMENTS

The authors would like to acknowledge the Sensor Laboratory, Department of Physics, for providing experimental facilities and technical assistance during sensor preparation until data acquisition.

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