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Probing the Impact of a Pesticide on the Dielectric Properties of Vegetable Peel Extracts in the Low-Frequency Regime

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The frequency-dependent dielectric properties of pristine and a pesticide treated bottle gourd and pumpkin peel extracts have been measured in the frequency range 100Hz-2MHz. Dielectric parameters such as dielectric permittivity (ε′), dielectric loss (ε″), and electrical conductivity (σ) of pure and fipronil mixed peel extracts are compared to assess the effect of fipronil on the dielectric properties of the vegetable peel extracts. In the low-frequency regime, the values of ε′ and ε″ of peel extracts are observed significantly higher due to the dominance of water-triggered surface charge polarization and dipolar polarizations. Remarkable changes in the values of ε′, ε″, and σ for bottle gourd and pumpkin peel extracts were observed on increasing the fipronil concentration. Almost monotonic increase in ε′, ε″, and σ values is observed with the mixing concentration of fipronil as the concentration of fipronil increases. This concentration (of fipronil) dependent behavior of dielectric parameters could offer the way to assess the presence of the pesticide residues in vegetable extracts. The observed dielectric behavior of samples was further analyzed by principal component analysis that offered the discrimination of pure and fipronil treated samples. The presented observations would serve as a tool for the dielectric spectroscopy-based discrimination of pesticide residues in horticultural extracts.

Keywords: Dielectric spectroscopy, Vegetable extracts, Pesticides, Electrical conductivity, Principal component analysis

1 Introduction

Green vegetables are enriched with natural sources of vitamins and minerals and are being consumed greatly all over the world[1, 2]. Attempts are being made to increase the crop yield of these natural green foods to meet the rising demand of the increasing population of the world[1-4]. Significant losses of crops are occurring due to insects and pests. To prevent them, farmers are forced to use insecticides/pesticides multiple times over the period of the vegetable crop. The excess use of pesticides on vegetable crops has raised a great concern of health risk due to the consumption of green vegetables containing traces of pesticides/ insecticides[4-6]. There are many chemicals being used to prepare the pesticides that are banned in many of the countries, including India[7]. However, many insecticides/pesticides are still being used by the farmers in our country[7]. Malathion, fipronil and dimethoate etc., are some of the pesticides that are currently being used to protect the vegetable crops from pests/insects[8, 9]. Fipronil is a broad-spectrum phenyl pyrazole insecticide that kills insects via contact or ingestion by blocking their central nervous system receptors, causing paralysis and death of insects. The fipronil is used to control pests like termites, borers, thrips, and beetles. Besides use of fipronil on growing crops, it is also being used in soil application, seed treatment, and its exposure could also occur to humans through skin/eye contact, accidental ingestion, causing potential for acute and long-term effects[10, 11]. It is worth to mention that the effect of the pesticides degrades with time after their use, and many such reports have appeared accounting for the active period of the pesticides[12, 13]. Such studies could provide the knowledge about the period after which the pesticide-treated vegetables may be consumed without any significant health risk[14]. However, it is important to assess the presence of pesticides in the vegetables being consumed. There are various methods being exercised to detect the presence of pesticides/insecticides in vegetables[15, 16].

In a study, fluorescent coumarin dye-based approach was developed for the selective and sensitive detection of dimethoate in green tea extracts. The study demonstrated a high degree of sensitivity, achieving a limit of detection (LOD) of 3.2μg/L. The

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probe exhibited a linear response across a concentration range of 7.8 to 292 µg/L[4]. However, optical detection methods such as fluorescence spectroscopy and surface-enhanced Raman scattering, while offering rapid and cost-effective analysis, often exhibit reduced accuracy compared to laboratory procedures due to smaller sample sizes and inherent errors in their quick response[5]. The chromatography-mass spectrometry is the gold standard for laboratory-based precision, especially given the complexity and diversity of matrices in biological materials[6]. In a study by Srivastava et al., GC-MS (Gas Chromatography-Mass Spectroscopy) was employed for the non-target analysis of 31 pesticide residues in diverse agricultural matrices, including complex samples like cauliflower, brinjal, and green pea[2]. Their rigorous analytical validation demonstrated high accuracy (% recovery 79-104 %) and exceptional sensitivity, achieving LOD as low as 1.49 mg kg^{-1}. Despite the high precision of these techniques, there is a growing need for more versatile and efficient screening tools that offer rapid analysis. The efficacy of UV-Visible spectrophotometry and Fourier transform infrared spectroscopy (FTIR) for the detection of Flonicamid insecticide in vegetables was demonstrated, achieving a significantly lower LOD of 0.007 µg/mL[16]. The UV-Visible spectroscopy technique could also be utilized to differentiate between the pure, diluted, and adulterated fruit extracts[17]. The characteristic peaks (absorption) of fruit extracts were found to show significant change in their intensity and peak wavelengths on increasing their dilution and adulteration[17]. Spectroscopic techniques enable non-destructive and multi-component analysis and are particularly suited for rapid screening applications[9, 10]. Dielectric spectroscopy (DS) deals with the molecular dynamics of the constituent molecules of the samples and could provide a safe, non-invasive, and portable means of monitoring in quality processes via molecular dynamics. The fundamental principle of DS relies on how does a material interacts with electric field, defined by its complex permittivity ($\varepsilon^* = \varepsilon' - j\varepsilon''$). The DS of a sample could depict the response of the constituent species to the applied ac electric field of varying frequency. The response is measured in terms of the real part of dielectric permittivity (ε') and the imaginary part termed as dielectric loss (ε'')[18]. The dielectric behavior of biological samples like fruits and vegetable extracts is found to be strongly dependent on the frequency of the applied field[19]. That is why the high frequency domain (~GHz range) has been greatly explored due to comparatively stable dielectric response. The low-frequency dielectric response of fruits/vegetable extracts is known to be dominated by electrode polarization (EP), dipolar contributions and is rarely explored. The DS technique has been explored for moisture sensing and pest control in grains and its application was expanded to include the sensing of maturity and quality in fresh fruits and vegetables[20]. The open-ended coaxial probe was utilized to measure the dielectric behavior of botanical samples across a wide frequency range (200 MHz to 8.5 GHz), confirming that temperature and frequency do significantly influence the dielectric constant and loss factor[21]. Recent studies are specifically being focused on the molecular dynamics within the biological tissues. A double-shell model has been used to simulate dielectric relaxation in vegetable tissues, and it was found that β-relaxation was activated from plasma membrane polarization[22]. Additionally, the dielectric properties of specific vegetable derivatives, such as beetroot juice, have been characterized to understand how energy storage and dissipation vary with frequency, providing a baseline for microwave-assisted processing[23]. In this method, the structure and composition of materials are determined by measuring the dielectric properties that are sensitive to ionic components and density variations[11]. These properties are fundamentally determined by the molecular structure and distribution of electric charges within the material, suggesting that dielectric measurements can uniquely identify specific chemical compositions and physical states[11, 12].

The response of pristine and pesticide-treated vegetable extracts could display detectable changes in dielectric parameters, such as ε', ε'', and σ of such samples. However, investigations based on DS-assisted assessment of pesticides in vegetables, especially in the low-frequency regime, are rarely reported[24]. This creates a scope to assess the presence of pesticides in the vegetable extracts by comparing their dielectric properties. The interaction of electromagnetic fields with the molecular moieties of these samples could offer the discrimination of pesticide residues based on their dielectric relaxation behaviors. This detection can be further enhanced through the integration of chemo metric and machine learning algorithms to improve accuracy[13, 14]. Recent studies have demonstrated that combining dielectric measurements with artificial intelligence can significantly enhance the sensitivity and

selectivity of detection systems, addressing common limitations such as matrix effects. In this paper, we have compared the dielectric properties of pristine and fipronil-treated vegetable (bottle gourd and pumpkin) peel extracts in the frequency range from 100Hz to 2MHz. The modification in the values of ε′ and σ of vegetable peel extracts with the mixing concentration of fipronil is analyzed systematically. The principal component analysis (PCA) of dielectric permittivity and electrical conductivity has revealed observable distinction between pure and fipronil treated peel extracts.

2. Experimental Details

2.1 Sample Preparation

For the present investigations, two vegetables, bottle gourd (*Lagenaria siceraria*) and pumpkin (*Cucurbita moschata*), were procured from the local vendors. In view of the fact that the pesticide residues primarily accumulate in the surface (outer layers) of these vegetables, the extracts from the peel of the vegetables were used. The peels were processed manually in the laboratory using mechanical extraction to ensure that the natural composition of the extracts remained intact. After extraction, the vegetable peel extracts were filtered using Whatman filter paper (pore size 11 μm) to remove large particulate matter to obtain clear liquid matrix for subsequent testing. The insecticide fipronil (FIP) was purchased from the local pesticide/seeds shop. The purchased fipronil powder contains fipronil 80 % W/W. The experimental samples were prepared in three distinct categories;

i Fipronil dissolved water samples: At first the fipronil powder in desired amounts (0.016g, 0.033g, 0.066g and 0.132g) were dissolved in the 200 ml of tap water (TW) were dissolved in the 200 ml of tap water (TW) to prepare four different concentrations in weight/volume (w/v) denoted as M1, M2, M3, and M4 respectively. The mixtures were sonicated in ultrasonic bath for 10 minutes at room temperature (RT).

ii Bottle gourd/fipronil solutions: the aqueous solutions (of different concentrations) of fipronil were mixed with the BG peel extracts. To prepare such solutions, 1:1 v/v ratio of aqueous fipronil (M1-M4) and the freshly prepared BG peel extracts (10ml each) were mixed properly to obtain the resultant concentrations of fipronil in BG extracts as 0.004 % (w/v), 0.008 % (w/v), 0.016 % (w/v), and 0.032 % (w/v) respectively. These samples are designated as BG+M1, BG +M2, BG +M3, and BG+M4 respectively.

iii Pumpkin /fipronil solutions: similarly, the aqueous solutions (of different concentrations) of fipronil were mixed with the pumpkin peel extracts to obtain the resultant concentrations of fipronil in BG extracts as 0.004 % (w/v), 0.008 % (w/v), 0.016 % (w/v), and 0.032 % (w/v) respectively. These samples are designated as P+M1, P +M2, P +M3, and P+M4 respectively.

For the dielectric characterization of the samples, the sample cell was used in the form of a parallel plate capacitor. The indium tin oxide (ITO) coated glass substrates (conducting area 100 mm^2) were used to form the capacitor, and the thickness of the cell was maintained using mylar spacers (~20 μm). Figure 1 schematically represents the fabrication process of the parallel-plate capacitor sample cell used to investigate the dielectric properties of pure and fipronil-mixed vegetable peel extracts. The pure and mixed samples (liquid) were placed at the opening to creep inside the capacitor. An LCR meter (E4980A, Keysight, USA,

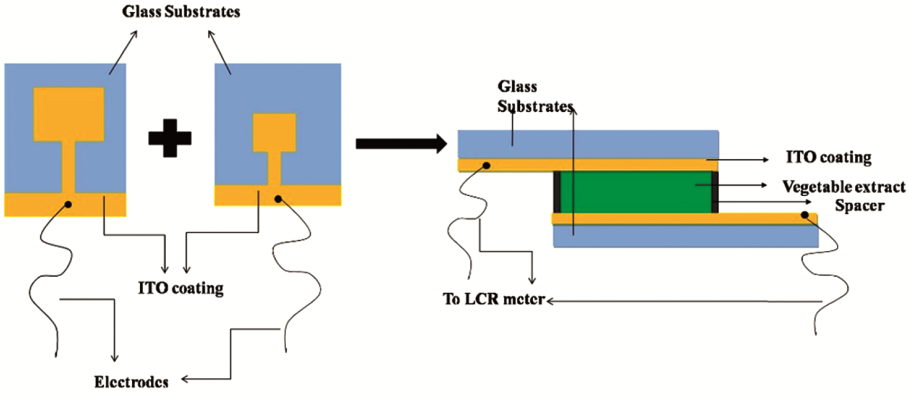

Fig. 1 — Schematic illustration of the fabrication of the parallel-plate capacitor sample cell used for measuring the dielectric properties of pure and fipronil-mixed extracts of vegetable peels

20 Hz to 2 MHz) was used to analyse the frequency-dependent dielectric parameters such as dielectric permittivity (ε′), dielectric loss (ε″), and electrical conductivity (σ) of the prepared samples at room temperature.

3 Results and Discussion

The air capacitance (C_0) of the sample cell was measured with frequency. As can be seen from the figure, the value of C_0 is very small and observed to be almost independent of the frequency in the mid frequencies. The value of C_0 at 10KHz frequency (stable) was used to determine the cell gap using the formula;

$$C_0 = \varepsilon_0 \frac{A}{d}$$

The thickness of the sample cell was calculated using the formula $d = \varepsilon_0 \frac{A}{C_0}$, where, ε_0= 8.85×10^{-12}C^2/Nm^2; A=100 mm^2. It is worth to mention here that the air capacitance of these ITO based sample cells remain almost frequency independent in the frequency range used in the present study. However, the ITO's conductivity starts contributing to the capacitance at higher frequencies (MHz and beyond).

As the pesticides are generally diluted with water before being sprayed over the crop, the cell was filled with TW and its dielectric parameters were measured in the frequency range of 100 Hz to 2MHz. Then, four samples of fipronil in tap water (TW+FIP) with varying concentrations of fipronil were filled in the sample cells, and their dielectric parameters were measured. The ε′ values of TW were observed significantly high in the low frequencies (< 10^3 Hz), and it decreased remarkably at higher frequencies. The higher value of ε′ at low frequencies is attributable to the phenomenon of EP[25]. It is caused by the accumulation of charges forming the electrode double layer, and is more pronounced in water and aqueous solutions. This large value of ε′ in the low-frequency regime greatly screens the dielectric parameters of aqueous biological samples[26]. At low frequencies, the two phenomena *vis.* alignment of dipoles in the direction of the applied field and the drift of ion-pairs towards the electrodes (Maxwell-Wagner-Sillars effect) could occur simultaneously[27, 28]. The presence of additional ionic impurities could greatly augment the ε′ values to give increased permittivity on mixing the fipronil in water. The loss spectra of TW and fipronil mixed TW exhibited a distinct relaxation peak around 10^3 Hz. The magnitude of ε″ was appeared significantly higher for fipronil-contaminated samples compared to that of TW, indicating increased energy dissipation due to the presence of ionic impurities or the polar nature of the fipronil molecule. The conductivity remained relatively constant at low frequencies, increased at higher frequencies and fipronil-mixed samples showed significantly higher conductivity than that of TW. However, the experimental data of TW exhibited significant deviations in the ε′ and σ values when compared with the established properties of highly pure distilled water. These differences are primarily attributable to the presence of dissolved ionic species (salts and minerals) in TW, which are largely absent in distilled water[29]. Moreover, the dielectric measurements were repeated many times for consistency and the data is observed to replicate itself. However, the slight variations in the dielectric data were noticed in the low frequency regime (below 300 Hz) and beyond this frequency, the data were observed consistent.

It was observed that the ε′ value of pure BG extract appeared greater (two-fold) than that for TW, attributable primarily to EP and the Maxwell-Wagner-Sillars effect, where free charge carriers accumulate at the interface between the extract and the electrodes[30]. A sharp decrease in ε' was observed as frequency increases due to the fact that the permanent dipoles within the BG extract and the fipronil molecules cannot orient themselves rapidly enough to follow the high-frequency alternating electric field, leading to a dipolar lag[31]. The prominent dielectric relaxation peak observed in the frequency range of 10^2 to 10^3 Hz indicative of dipolar relaxation. The peak occurs when the frequency of the external field matches the characteristic relaxation time of the molecular dipoles[32]. The magnitude of the loss peak varies with fipronil concentration. As fipronil is added, the change in peak height and position suggests a modification in the viscous environment and the intermolecular interactions within the mixture, altering the energy required for dipole reorientation[33]. The modifications in the values of ε′ and ε″ of the BG extract due to fipronil could be due to the ions causing the increase of ε′ and ε″. The peel extract of BG contains mostly water (dipole moment 1.83D), and the addition of fipronil (dipole moment 5.83D) could increase the dipolar contribution. At lower frequencies, the conductivity remains relatively low and stable, dominated by DC conduction due to hopping charge carriers. However, in

the high-frequency region (>10^3 Hz), conductivity increases significantly. This trend follows Jonscher's Power Law, where the increase in conductivity is due to the enhanced mobility of ions and polar molecules responding to the rapidly oscillating field[34]. The impact of fipronil on the dielectric behaviour of pumpkin extract was also monitored. The value of ε′ and ε″ of fipronil mixed with pumpkin extract were observed slightly lower than that of pure pumpkin extract. A sharp dielectric relaxation peak near 500 Hz in the pumpkin and fipronil treated pumpkin mixtures was observed attributable to α-relaxation (dipolar relaxation), originating from the rotational movement of molecular dipoles within the pumpkin's aqueous environment[13]. The conductivity of fipronil mixed pumpkin peel extracts displayed a very small change with the concentration of fipronil. A sharp upward increase in σ at higher frequency could be associated with the fipronil threshold concentration[35, 36].

At lower frequencies (<10 kHz) the dielectric parameters often exhibit overlapping values and fluctuations that made it difficult to clearly correlate the data with fipronil concentration. In the lower frequencies, the actual dielectric properties of BG/P extract are significantly screened due to dominant aqueous contribution and to extract a meaningful effect of fipronil on the BG/P extract, we have analyzed the dielectric behavior of ε′, ε″, and σ at higher frequencies. Figure 2 shows the variation of ε′, and σ of TW with different concentrations of fipronil at certain frequencies. As can be seen from figure, the value of ε′ is observed mostly greater for fipronil mixed TW, and it increased on increasing the concentration of fipronil (Fig. 2 (a)). The presence of fipronil caused a significant enhancement (~2 fold) in the conductivity of TW (Fig. 2 (b)). The transition from TW to the first concentration level (M1) resulted a sharp increase in conductivity value from 200 to 400 μS/m. This indicates that fipronil significantly enhances the ionic conduction of the solution, likely through the introduction of additional mobile charges or the disruption of the hydrogen-bonded water network[37].

Figure 3 depicts the variation of ε′ and σ of pure and fipronil mixed BG peel extract. The value of ε′ of BG/FIP mixtures is found to increase almost

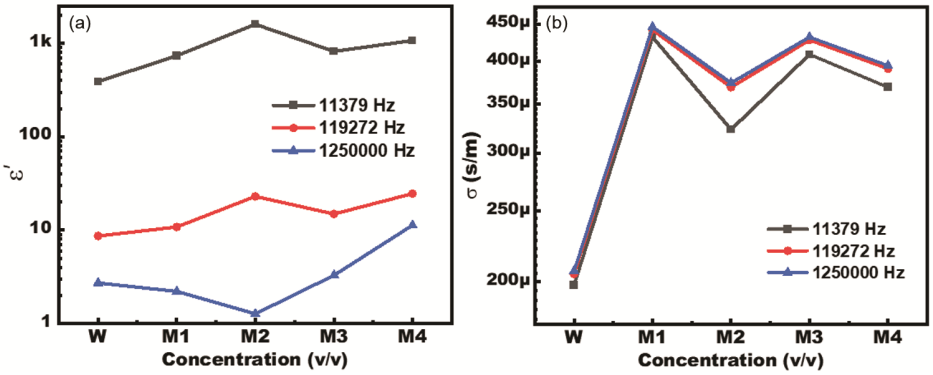

Fig. 2 — Variation of (a) real part of permittivity (ε'); and (b) AC conductivity (σ) of TW and fipronil mixed TW with some selected frequencies

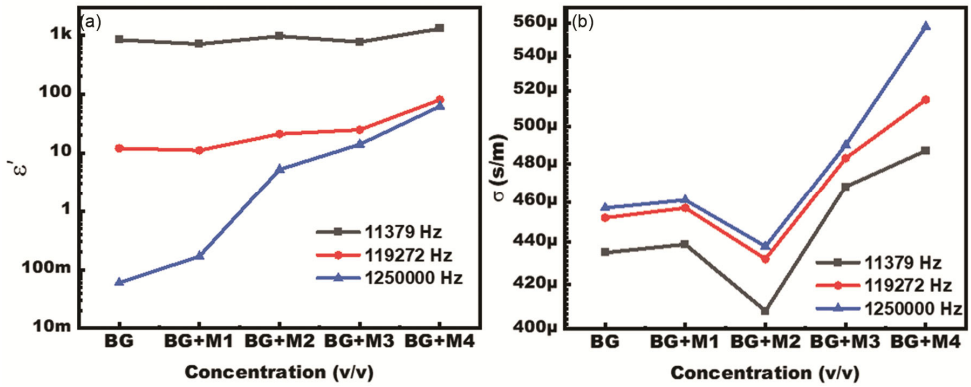

Fig. 3 — Variation of (a) real part of permittivity (ε'); and (b) AC conductivity of bottle gourd (BG) and fipronil mixed BG extract with some selected frequencies

monotonically with the mixing concentration of fipronil for all three frequencies, with the greatest slope at ~1.25 MHz.

Also, the magnitude of ε′ is observed to be greater in comparison to that of TW/FIP mixtures. This higher magnitude of ε′ could be due to the additional contributions from the constituent dipolar molecules of BG peel extract. Moreover, the monotonic increase in ε′ values is the manifestation of increasing concentration of fipronil. It is worth to mention that the behavior of σ of BG/FIP mixtures showed the opposite trend in comparison to that of TW/FIP mixtures. In case of BG/FIP samples, the magnitude of σ is monotonically increased with the concentration of FIP for all the frequency points (Fig. 3 (b)). Moreover, it is noticeable that an abnormality in σ values is observed for the second (M2) concentration of fipronil for all the samples (e.g., TW + FIP, BG + FIP, and P + FIP). This concentration M2 is the recommended dose as prescribed by the manufacturer of the pesticide for its use over crops. Also, the values exhibited a remarkable dip in σ for TW + M2 and BG + M2, indicating some specific interaction for the recommended dose. This anomaly could possibly due to a temporary molecular binding or complexation between the pesticide molecules and the organic solutes (proteins or saccharides) naturally present in the bottle gourd, which temporarily hinders charge mobility before increasing again at higher concentrations[26]. However, more investigation is needed by involving different pesticides/extracts to predict its generality.

The pumpkin extract, too, displayed a consistent rise in ε' at 1.25 MHz as the pesticide concentration increased (Fig. 4 (a)). This reinforces the utility of the MHz frequency range for bypassing complex organic interference. The value of σ showed almost an upward trend (however, with a dip at P +M1) reaching its peak at P+M4, confirming that the dielectric signature of fipronil remains detectable even within complex organic vegetable matrices at higher frequencies away from the ionic/EP (Fig. 4 (b))[27]. It is worth to mention here that the ITO-based electrodes (transparent and conducting) used in the present study could offer the monitoring of optical changes in the orientation of surface (interaction) sensitive liquid crystal materials[37]. This has motivated us to employ ITO-based electrodes for dielectric measurements and it provides scope for detection of pesticides by analysing both, dielectric as well as optical changes caused by pesticides.

4 Principal Component Analysis of Samples

PCA was conducted using the web-based tool Agri Analyze to explore the differences in dielectric properties (ε′ and σ) of both pure and fipronil-treated TW, BG, and pumpkin samples over a frequency range of 1 kHz to 2 MHz (Fig. 5)[39]. The values of ε′ and σ at frequencies 1 kHz, 5 kHz, 10 kHz, 20 kHz, 40 kHz, 60 kHz, 80 kHz, 1 MHz, 1.25 MHz, 1.5 MHz and 2 MHz were first extracted (in tabular form) for all the TW, TW+FIP, FIP + BG, and FIP +P samples and the PCA was performed separately for ε′ and σ data. The PCA analysis corresponding to ε′ data using singular value decomposition on nine variables, produced nine principal components (PCs). Only the first two PCs had eigen values greater than one, meeting Kaiser's criterion (Fig. 5 (a)). Together, these two components explained 99.26 % of the total variance, with PC1 making up 77.54 % and PC2

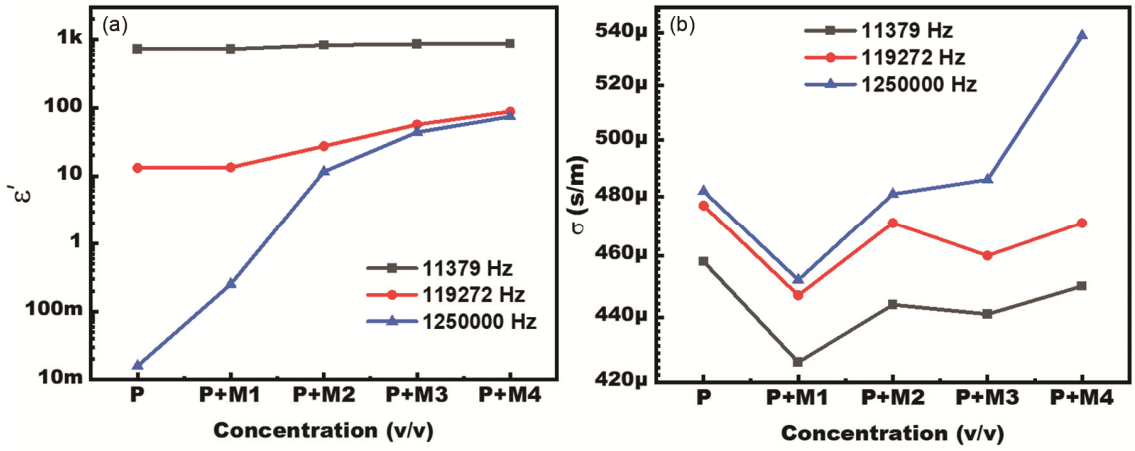

Fig. 4 — Variation of (a) real part of permittivity (ε′); and (b) AC conductivity of Pumpkin (P) and fipronil mixed P extract at some selected frequencies

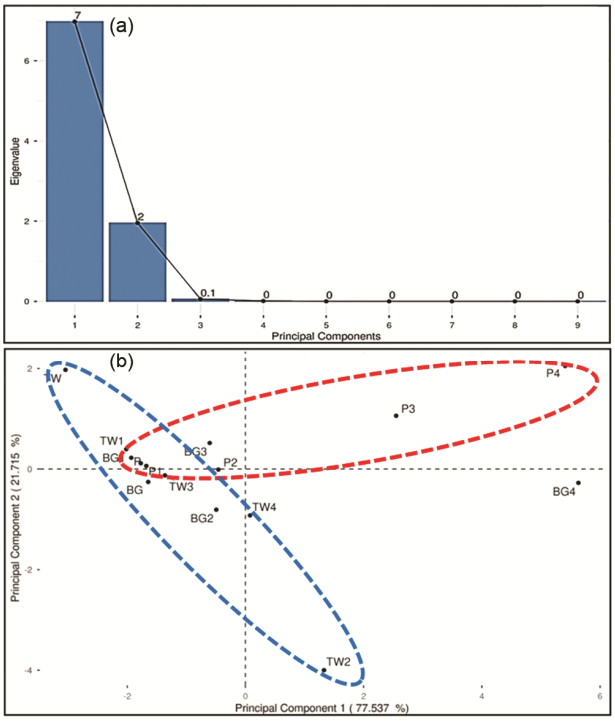

Fig. 5 — (a) The scree plot indicating the eigen values for 9 PCs considered; and (b) biplot between PC1 and PC2 for pure and fipronil-treated water, bottle gourd, and pumpkin extracts

contributing 21.72 %. This shows that most of the information in the dataset is captured in a two-dimensional space. The loading plot showed that all frequency variables exhibited a strong positive link to PC1. This means that PC1 mainly represented the overall dielectric response of the samples across the full frequency range. In contrast, PC2 separated frequency domains: higher frequencies (≥1 MHz) contributed positively, while lower frequencies (10–40 kHz) contributed negatively. Intermediate frequencies (60–80 kHz) had lower contributions to PC2. PC2 highlighted the differences between low and high-frequency dielectric behavior, showing the frequency-dependent dispersion characteristics of the samples.

The PCA score plot, when viewed with the loading vectors, offered important insights into how the samples differed. Tap water samples (TW, TW1, TW3) were gathered closely, showing little variation in their dielectric properties under lower treatment levels (Fig. 5 (b)). In contrast, TW4 displayed a slight deviation, indicating the influence of higher fipronil concentration. TW2, however, was clearly separated along the negative PC2 axis. This aligns with low-frequency variables, suggesting a strong contribution from low-frequency dielectric responses. Moderate dispersion was observed in the samples of BG which were placed near or slightly along the PC1 and indicated intermediate behaviour of dielectric with no class dominating in a particular region of frequency. The pumpkin samples (P–P4) appeared further separated along both PC1 and PC2, and demonstrated alignment toward high-frequency loading vectors. It indicates that the samples of pumpkin have higher sensitivity to variability of high-frequency dielectric properties. In addition, the fact that points change progressively from P to P4 along the direction of loading vectors suggests a concentration-dependent response towards fipronil treatment and illustrates how PCA adequately represents subtler changes in biological systems induced by pesticide exposure.

Collectively, the tandem consideration of score and loading plots reveals that dielectric relaxations in both low and high-frequency regions are relevant features for separating the treated samples from untreated ones. Both high-frequency variables are strong contributors for differentiating between the two pumpkin samples, suggesting careful analysis of multi-frequency comparison might be valuable, and low-frequency results show a very sensitive response to only specific treatments (such as TW2). The results imply that PCA-aided DS has great potential as a versatile tool to discriminate between pesticide-treated vegetable samples. Nevertheless, additional investigations based on larger quantities of samples and other pesticide classes would be needed to confirm and expand the use of this method for the quantification and detection of pesticide residues in agricultural products.

5 Conclusion

In the present report, the dielectric behavior of bottle gourd and pumpkin peel extracts is measured in the low-frequency regime and compared with that of tap water. Importantly, the effect of the pesticide, fipronil, on the dielectric parameters is analyzed by varying its concentration. It has been found that the low frequency spectrum of biological mixture is significantly dominated by the phenomenon of electrode polarization and dipolar contributions due to their dominant aqueous environment. The fipronil, one of the common insecticides, possess large dipole moment (5.83 D) and its dipolar contribution was resulted in terms of increased values of ε', ε'' and σ of biological mixtures. The transition from pristine to fipronil-mixed extracts produced measurable shifts in

electrical properties, establishing a potential baseline for pesticide discrimination method based on dielectric spectroscopy. The dielectric spectroscopy-based investigations on other pesticides/vegetable extracts and consideration of the effect of bias voltage/ temperature may further explore the potential of this technique in assessing the discrimination of pesticide residues (preferably assisted by PCA or machine learning).

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