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ENGINEERED AG/PAN NANOFIBER SERS SENSORS: A MANUFACTURING SOLUTION FOR ETHYLENE GLYCOL CONTAMINATION IN AGRICULTURAL PRODUCTS

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ABSTRACT: Ethylene glycol (EG) contamination in agricultural products presents a serious food safety hazard due to its high toxicity and difficulty in rapid detection under field conditions. This research addresses the need for a sensitive, rapid, and cost-effective EG detection method. The objective is to develop engineered Surface-Enhanced Raman Scattering (SERS) substrates based on silver nanoparticle-decorated polyacrylonitrile (Ag/PAN) nanofibers. The novelty lies in the in-situ incorporation of silver nanoparticles via a single-step electrospinning process, enabling precise morphological tuning and enhanced plasmonic hotspot formation. Ag/PAN nanofibers were fabricated under optimized electrospinning parameters (15 kV, 15 cm, 0.1 ml/hr) with silver nitrate concentrations ranging from 0 to 15 wt%. Characterization using SEM, TEM, and EDX confirmed uniform AgNP distribution and diameter reduction with increasing silver content. The SERS performance was evaluated using methylene blue as a model analyte and ethylene glycol as the target contaminant. Results showed that 10 wt% Ag/PAN substrates achieved sub-micromolar detection with 3–4 fold enhancement in EG fingerprint modes and nearly 5-fold in CH stretching regions. The study demonstrates a scalable sensing platform with potential for real-time EG monitoring in food safety applications.

KEYWORDS: *SERS; silver nanoparticles; polyacrylonitrile nanofibers; ethylene glycol detection; food safety; electrospinning*

1.0 INTRODUCTION

Surface-Enhanced Raman Scattering (SERS) has emerged as a powerful analytical technique for the trace-level detection of chemical and biological substances, owing to its exceptional sensitivity, molecular specificity, and non-destructive nature [1],[2], [3]. Among the various SERS substrate designs, silver nanoparticle-based nanostructures have consistently demonstrated superior enhancement factors due to their strong localized surface plasmon resonance (LSPR) properties [4], [5]. Recent studies report that optimized silver nanostructures can achieve enhancement factors as high as 10^6 to 10^8, enabling detection in the nanomolar and even picomolar range [6].

Electrospun nanofibers, particularly those based on polyacrylonitrile (PAN), have attracted significant interest as flexible and high-surface-area platforms for SERS applications [3]. Their interconnected porous network provides abundant adsorption sites for analyte molecules, while their tunable morphology allows precise control over plasmonic hotspot distribution. Various approaches have been developed for silver nanoparticle incorporation into PAN nanofibers, including in-situ reduction during electrospinning, post-spinning surface decoration [7], and hybrid methods [8]. Among these, in-situ electrospinning offers a scalable and reproducible pathway for large-scale substrate fabrication, which is essential for real-world sensing applications in manufacturing environments.

However, despite progress in SERS substrate development, current techniques often suffer from critical limitations such as poor reproducibility, insufficient field-deployability, and lack of integration with scalable manufacturing processes [9], [3]. These constraints are particularly problematic in the context of ethylene glycol (EG) contamination, which has become a major concern in food and beverage manufacturing due to its severe toxicity even at low concentrations [10]. Conventional EG detection methods such as GC-MS and HPLC offer high accuracy but are hindered by high cost, complex sample preparation, and the need for laboratory infrastructure — making them impractical for on-site quality control [11].

Recent literature confirms the potential of SERS for EG detection, as EG possesses identifiable vibrational modes (e.g., at 865, 1041, and 1084 cm^{-1}) that are SERS-active when interacting with silver nanostructures [6]. Nevertheless, most existing SERS platforms rely on rigid substrates or require complex fabrication techniques, limiting their utility in rapid manufacturing settings. Furthermore, previous reviews and research focus largely on proof-of-concept demonstrations with insufficient attention to process optimization, substrate uniformity, and reproducibility - aspects that are vital in manufacturing engineering environments [3].

To address these challenges, this research proposes a novel, in-situ fabricated silver-decorated PAN nanofiber substrate via electrospinning, specifically optimized for sensitive and scalable SERS-based EG detection [12]. Unlike traditional approaches, our method allows precise control over nanoparticle loading and nanofiber morphology, enabling the formation of uniform plasmonic hotspots. This study systematically investigates the impact of silver nitrate concentration on nanofiber structure and SERS performance using methylene blue (MB) as a model analyte and ethylene glycol as the target contaminant.

The objective of this work is to develop a reproducible, field-deployable SERS substrate with sub - micromolar EG detection capability, suitable for food and chemical safety monitoring in manufacturing contexts. The novelty lies in the integration of material design, fabrication scalability, and analytical performance - providing a comprehensive solution aligned with the needs of modern manufacturing industries.

2.0 METHOD AND CHARACTERIZATION

This research introduces a novel and scalable fabrication strategy for developing high-performance SERS substrates using silver nanoparticle-decorated polyacrylonitrile (Ag/PAN) nanofibers. This approach allows real-time control over nanoparticle distribution and fiber morphology, resulting in enhanced plasmonic hotspot generation critical for Raman signal amplification [13]. Unlike conventional multi-step methods, the proposed single-step electrospinning protocol supports reproducibility and large-scale manufacturability - addressing a crucial limitation in the translation of SERS technology to industrial applications. The methodological framework of this study follows a four-stage process:

(i) Preparation of Ag/PAN precursor solution,
(ii) Electrospinning under optimized process parameters,
(iii) Comprehensive physicochemical characterization, and
(iv) Performance validation via SERS analysis using methylene blue and ethylene glycol analytes.

2.1 Materials and Chemicals

This research investigates the fabrication and optimization of silver-decorated polyacrylonitrile (PAN) nanofibers via electrospinning for Surface-Enhanced Raman Scattering (SERS) substrate applications. The study comprehensively characterizes the nanofibers' morphological and structural properties using scanning electron microscopy (SEM), transmission electron microscopy (TEM), and fiber diameter analysis, with varying silver nitrate concentrations (0-15 wt%). Dual-analyte detection capabilities are evaluated using methylene blue (MB) as a standard reference molecule and ethylene glycol (EG) as the target contaminant in agricultural products.

Table 1: List of Equipment

Equipment	Specification
Digital Balance	(OHAUS, model PR224) with internal calibration capability
Magnetic Stirrer with Hot Plate	C-MAG-HS7 Hotplate Magnetic Stirrer (IKA, Germany)
Electrospinning Unit	
Stainless Needle	NIPRO 21Gx1"
Plastic syringe	Plastic Disposable Syringe (Nipro) 10 ml.
SEM	TESCAN, Model: MIRA
TEM	Thermo Scientific, Model: Talos F200X G2
Raman spectroscopy	HORIBA, Model: XploRA plus

Table 2: List of Chemicals

	Materials	Specification
1	Polyacrylonitrile, PAN	Mw = 150,000 g/mol, Sigma-Aldrich
2	Dimethylformamide, DMF	anhydrous, 99.8% from Sigma Aldrich
3	Polyvinylpyrrolidone, PVP	Mw ~40,000 g/mol, ≥99.9% purity, Sigma-Aldrich
4	Silver nitrate, $AgNO_3$	$AgNO_3$, ≥99.0%, Sigma-Aldrich
5	Methylene blue, MB	$C_{16}H_{18}ClN_3S{\cdot}xH_2O$, analytical grade, Sigma-Aldrich
6	Ethylene glycol, (EG)	EG ≥99.5%, Sigma-Aldrich

2.2 Methods

2.2.1 Production of Silver Nanoparticle-Decorated PAN Nanofibers via Electrospinning Process

The preparation of Ag/PAN solution starts with dissolving PAN in DMF to obtain a 0, 5, 10, and 15 wt.% concentration. The solution was continuously stirred using a magnetic stirrer at 60°C for 24 hours, followed by cooling to room temperature. Subsequently, silver nitrate ($AgNO_3$) was incorporated into the prepared 10 wt.% of PAN solution. Polyvinylpyrrolidone (PVP) was then added at a 1:1 ratio relative to $AgNO_3$. The resulting mixture underwent an additional 4-hour stirring period to ensure homogeneous distribution of the components.

Following the solution preparation, the fabrication of Ag/PAN nanofibers was carried out using the electrospinning process. The prepared PAN solution (10 ml) was loaded into a plastic syringe equipped with a metallic needle. Subsequently, the syringe assembly was mounted onto the electrospinning apparatus for the production of silver-decorated PAN nanofibers (Ag/PAN), as illustrated in Figure 1. The electrospinning parameters were optimized under the following conditions: The process was conducted with an Ag/PAN solution flow rate of 0.1 ml/hr through the spinneret. A DC power supply was utilized to generate an applied voltage of 15 kV, and the aluminum foil collector was positioned at a working distance of 15 cm from the needle tip to facilitate optimal fiber formation.

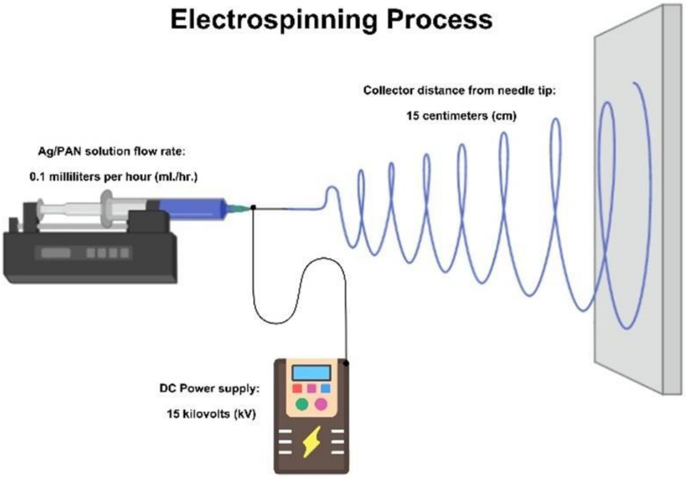


Figure 1: Electrospinning process setup for Ag/PAN nanofibers.

2.2.2 Characterization of Ag/PAN Nanofibers

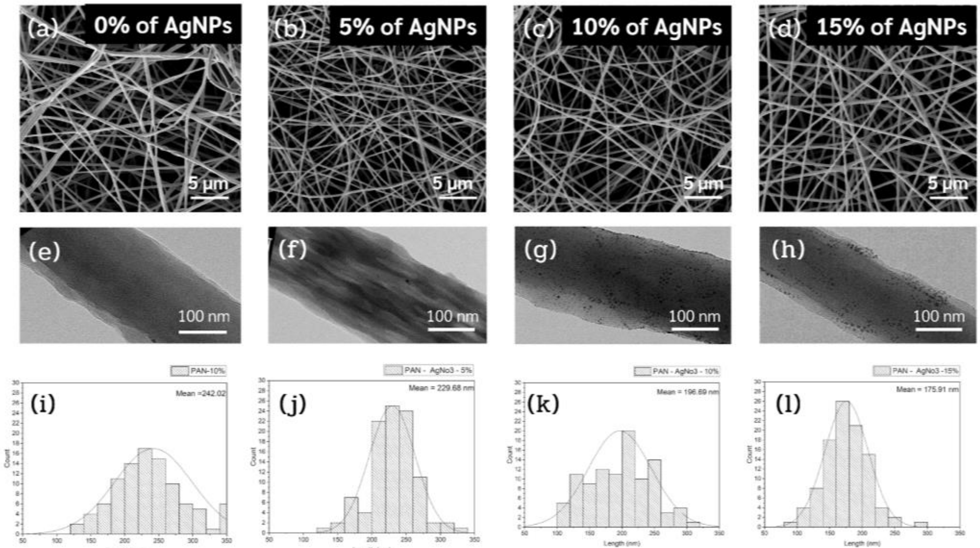
The provided figure illustrates the morphological transformations of PAN nanofibers with increasing silver nitrate ($AgNO_3$) concentrations (0-15 wt.%), as characterized by SEM and TEM and fiber diameter distribution histograms.

Figures 2(a–d) present SEM images of PAN nanofibers with different AgNP loadings (0–15%). The pristine fibers (a) exhibit a smooth and uniform morphology. With increasing AgNP content (b–d), the fibers remain continuous and well-distributed, although slight surface roughness becomes more apparent. Although individual AgNPs are not clearly observed, the progressive surface irregularities indicate their incorporation and dispersion within the fibrous network without compromising the overall structural integrity. To further examine the physical characteristics of the silver nanoparticles, TEM analysis will be employed in subsequent investigations.

Figures 2(e–h) present TEM images of PAN nanofibers with increasing AgNP loadings (0–15%). The pristine fiber (e) shows a smooth morphology without nanoparticles. At 5% AgNPs (f), a small number of silver nanoparticles begin to appear on the fiber surface. With 10% loading (g), numerous dark spots corresponding to AgNPs are observed and uniformly distributed along the surface. At 15% (h), the surface shows a denser coverage of nanoparticles with partial aggregation. This progressive surface decoration of AgNPs increases the surface roughness and enhances the density of active sites, which is essential for improving the SERS performance of the nanofiber substrates.

3.0 RESULTS AND DISCUSSION

Figures 2(i–l) show the fiber diameter distributions of PAN nanofibers with different AgNP loadings (0–15%). The pristine sample (i) exhibits a mean diameter of ~242 nm with a broad distribution. At 5% AgNPs (j), the mean diameter decreases to ~230 nm with a narrower distribution. With 10% AgNPs (k), the diameter further reduces to ~197 nm and becomes more uniform. At 15% AgNPs (l), the mean diameter reaches ~176 nm, the lowest observed, though partial aggregation may occur at this level. Overall, the results demonstrate that increasing AgNP concentration progressively reduces fiber diameter and improves uniformity, highlighting the role of AgNPs in enhancing solution conductivity and electrospinning stability.



Figures 2: (a–d) present SEM images of PAN nanofibers with different AgNP loadings (0–15%); (e–h) present TEM images of PAN nanofibers with increasing AgNP loadings (0–15%); (i–l) show the fiber diameter distributions of PAN nanofibers with different AgNP loadings.

Figures (a–d) present the elemental mapping of PAN/Ag nanofibers obtained by EDS analysis. The carbon signal (a) is uniformly distributed throughout the fibers, confirming PAN as the primary structural component. Nitrogen (b) also shows a homogeneous distribution, which is consistent with the nitrile groups of the PAN polymer, validating the stable chemical composition of the matrix. Oxygen (c) appears in a dispersed manner along the fibers, which may arise from partial surface oxidation or residual solvent, but this does not significantly alter the overall fiber structure. In contrast, silver (d) is clearly observed and uniformly distributed on the fiber surfaces, indicating the successful decoration and effective dispersion of Ag nanoparticles. The presence of silver on the surface is particularly significant, as it enhances surface roughness and increases the density of active sites, which play a crucial role in improving the SERS performance of the nanofiber substrates.

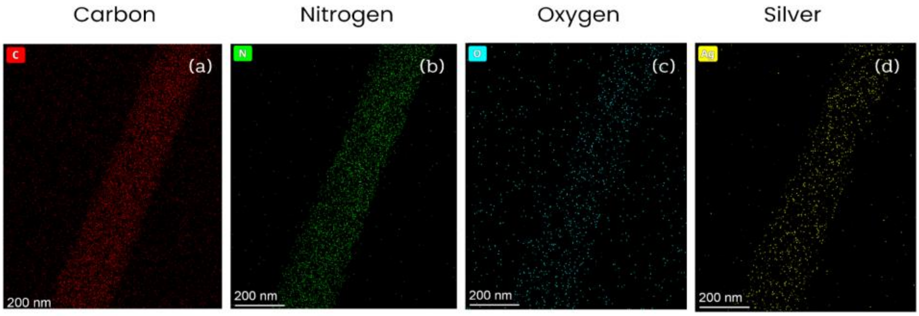


Figure 3: EDS elemental mapping of PAN/Ag nanofibers: (a) Carbon, (b) Nitrogen, (c) Oxygen, and (d) Silver well-dispersed on fiber surfaces.

Table 3 summarizes the elemental quantification of Ag/PAN nanofibers. Carbon (C) and nitrogen (N) represent the major constituents of the PAN matrix, with mass fractions of 77.5% and 13.7%, respectively, confirming PAN as the primary structural component. Oxygen (O) is detected at 3.27%, which may originate from partial oxidation or residual solvent. Silver (Ag) is quantified at 5.50%, validating the successful incorporation of Ag nanoparticles into the nanofiber system. The associated mass error values remain relatively low (≤2.39% for C and N, and ≤0.643% for O and Ag), indicating good accuracy and reliability of the elemental analysis results.

Table 3: Elemental quantification of Ag/PAN nanofibers

Element	Mass Fraction (%)	Mass Error (%)
C	7.75×10^1	2.27

N	1.37 x 10^{1}	2.39
O	3.27	6.43 x 10^{-1}
Ag	5.50	6.02 x 10^{-1}

The SERS performance of Ag/PAN nanofibers was systematically evaluated using Methylene Blue (MB) as a model analyte. The experimental protocol involved preparing MB solutions at varying concentrations (10^{-1}, 10^{-6}, and 10^{-7} M), drop-casting onto Ag/PAN nanofiber substrates, and allowing them to dry at ambient temperature. For Raman analysis, the system was calibrated using a silicon wafer, and measurements were performed in triplicate at multiple sampling points following parameters in Table 4.

Following MB validation, ethylene glycol (EG) detection was performed using the same protocol with environmentally relevant concentrations (0.084 Molar). The optimized 10 wt% Ag/PAN substrates were used to analyze EG's characteristic vibrational modes.

Table 4: Operating Parameters for Raman Spectroscopic Analysis

Parameter	Value/Setting
Exposure time (s)	15
Hole (um)	500
Filter	0.1%
Slit (um)	200
Objective	x100_VIS
Laser	532nm_Edge
Grating	600 (750nm)

This study demonstrates the analytical performance of silver nanoparticle-polyacrylonitrile (AgNP/PAN) nanofiber composites as SERS substrates for methylene blue detection. The spectroscopic analysis in figure 4 reveals two characteristic Raman peaks at 1396 cm^{-1} and 1624 cm^{-1}, corresponding to C-N symmetric stretching and aromatic C-C stretching vibrations of the phenothiazine ring system, respectively.

The substrate exhibits exceptional concentration-dependent response across three orders of magnitude, from 0.1 M to 0.1 µM. Peak intensities demonstrate a clear linear relationship with concentration, achieving maximum enhancement of approximately 27,000 a.u. at 0.1 M and maintaining detectable signals down to 0.1 µM (~3,000 a.u.), establishing sub-micromolar detection limits. The detection limit obtained in this study (0.1 µM for MB) is less sensitive than those reported in recent works. For instance, [14] demonstrated an ultralow detection limit of 0.1 nM using porous silicon photonic crystals decorated with AgNPs, while [15] achieved 1 nM with AgNP-modified ZnO nanoplates. These results clearly indicate superior sensitivity compared to the present AgNP/PAN system. However, the substrates developed in this work offer several notable advantages. First, they provide a broad linear detection range across three orders of magnitude (0.1 M to 0.1 µM), ensuring accurate quantification across diverse concentration levels. Second, the PAN nanofiber matrix offers excellent structural stability and reproducibility, minimizing batch-to-batch variations and ensuring consistent SERS responses. Finally, the fabrication process of AgNP/PAN nanofibers is relatively simple, scalable, and cost-effective compared to more complex nanostructured systems. These advantages highlight the potential of AgNP/PAN nanofiber substrates as robust and practical SERS platforms for real-world sensing applications, despite their comparatively higher detection limit.

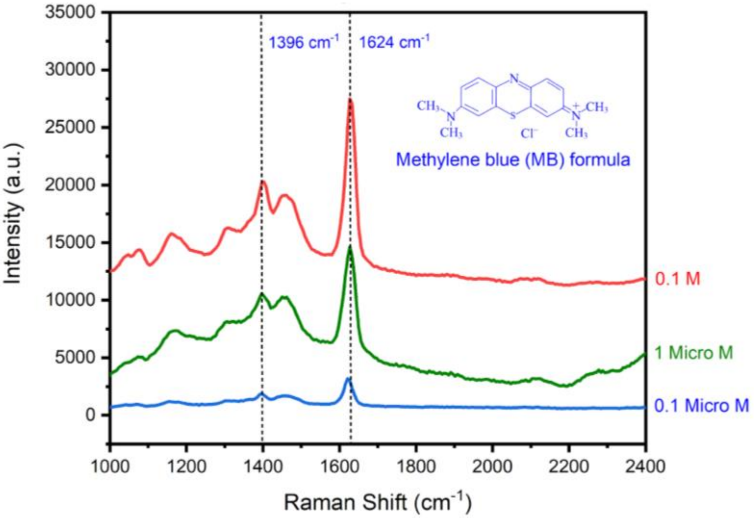


Figure 4: SERS Analysis of 0.1 micromolar to 0.1 molar of MB Detection using Ag/PAN Nanofibers.

Figure 5 presents the Surface-Enhanced Raman Scattering (SERS) analysis of ethylene glycol (EG) detection using the Ag/PAN nanofiber substrate. The plot specifically compares the Raman spectrum of EG obtained with SERS enhancement (black line) against a non-SERS reference (red line), clearly showcasing the dramatic signal amplification achieved by the developed material. For ethylene glycol detection, the SERS enhancement produces a significant 3-4 fold amplification in fingerprint region modes, observed between 865-1462 cm^{-1}. Furthermore, a remarkable nearly 5-fold amplification is seen in the CH stretching vibrations, specifically between 2844-2939 cm^{-1}. This dramatic signal improvement, evidenced by the primary CH stretch signal increasing from approximately 4,500 to 22,000 a.u., establishes the platform's broad molecular applicability. From a materials science perspective, this demonstrates the effectiveness of the optimized Ag/PAN nanofibers in creating numerous plasmonic hot spots that significantly enhance the weak Raman signals of small polar molecules like EG, proving the material's high efficiency as a SERS substrate for critical detection applications.

Previous study [16] demonstrated the capability of SERS to identify ethylene glycol as part of the organic shell surrounding Ag nanoparticles, confirming that this small polar molecule can be effectively probed by plasmonic substrates. While their work focused on characterizing the molecular coating of nanoparticles rather than direct analyte detection, it provides important evidence supporting the feasibility of SERS-based ethylene glycol analysis. In contrast, the present study directly investigates ethylene glycol as the target analyte using Ag/PAN nanofibers, achieving a clear signal amplification across both fingerprint and CH stretching regions. This distinction highlights that our substrate not only validates earlier findings on the SERS activity of ethylene glycol but also extends the application toward practical detection with enhanced sensitivity and reproducibility.

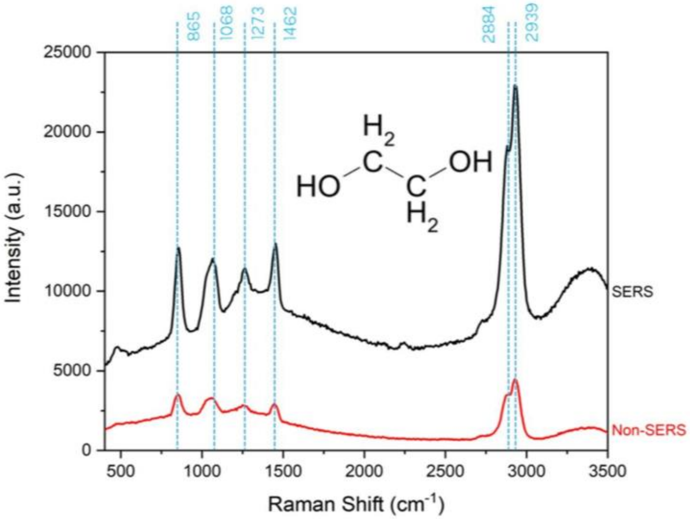


Figure 5: SERS Analysis of Ethylene glycol Detection using Ag/PAN Nanofibers.

4.0 CONCLUSION

This research successfully demonstrates the practical utility of engineered silver-decorated polyacrylonitrile (Ag/PAN) nanofibers as highly effective Surface-Enhanced Raman Scattering (SERS) substrates for the rapid and sensitive detection of ethylene glycol (EG) in agricultural products. Our key contribution lies in the systematic optimization of electrospinning parameters and the precisely controlled in-situ incorporation of silver nanoparticles, which collectively lead to tailored nanofiber morphology and enhanced plasmonic hotspot formation [1],[4]. This innovative engineered fabrication approach distinguishes itself from conventional methods by enabling direct and fine-tuned control over both silver nanoparticle distribution and nanofiber diameter reduction, factors critical for maximizing SERS efficiency and ensuring a field-deployable solution [9],[17][18].

The comprehensive characterization using SEM, TEM, and EDX confirmed the successful and uniform incorporation of silver nanoparticles (10-30 nm) and a systematic reduction in fiber diameter (from 242.02 nm to 175.91 nm) with increasing silver content, directly correlating with enhanced SERS performance. The optimized 10 wt% Ag/PAN substrates exhibited exceptional sensitivity for methylene blue

(MB) detection down to sub-micromolar concentrations. More critically, for EG detection, these substrates achieved remarkable SERS enhancement, with 3-4 fold amplification in fingerprint region modes and nearly 5-fold amplification in CH stretching vibrations. This robust signal amplification enables sub-micromolar EG detection sensitivity, positioning this technology as a valuable addition to current analytical methodologies for ensuring food product safety and protecting public health [10],[11].

This work significantly advances the field of SERS-based analytical chemistry by providing a scalable, reproducible, and field-deployable solution for critical food safety monitoring. The established methodological framework for engineered Ag/PAN nanofibers serves as a promising model for detecting other harmful contaminants in complex matrices, potentially revolutionizing on-site food safety assessment protocols through its combination of high sensitivity, practical applicability, and cost-effectiveness [3].

AUTHOR CONTRIBUTIONS

K. Juntaracena and P. Wanla: Conceptualization, Methodology, Writing- Original Draft Preparation, Data Collection and Data Analysis, and Writing-Reviewing and Editing.

CONFLICTS OF INTEREST

The manuscript has not been published elsewhere and is not under consideration by other journals. All authors have approved the review, agree with its submission, and declare no conflict of interest on the manuscript.

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