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## **Optimization and Validation of Gas-Chromatography Mass spectroscopy (GC-MS) and Head-Space Solid Phase Microextraction (HS-SPME) for the Analysis of the Extraction of Volatile Organic Compounds (VOCs) in *Nigella Sativa* Seed Oils.**

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### **Structured Abstract**

**Background:** *Nigella Sativa*. *L* also called black seed is a type of herbs are the members of the Ranunculaceae (Buttercup family). Moreover, *Nigella Sativa*. has a strong aroma and slightly bitter nutty-peppery taste used as a spice and traditional medicine as it has a religious and historical background. The samples are used to study the efficiency of the solid-phase micro-extraction (SPME) fiber and the multivariate analysis using the Response Surface Method (RSM) combined with Central Composite Design (CCD) to determined and verification of the authenticity of the sold *Nigella sativa* seed oils in the market.

**Methods:** The optimization process unfolded in two key stages. Firstly, four types of SPME fibers were examined to determine the ideal extraction time and temperature for each. Subsequently, gas chromatography (GC) conditions, including temperature programming and column flow, were meticulously fine-tuned. Following optimization, the refined method underwent comprehensive validation, encompassing assessments of accuracy, precision, linearity, and the establishment of Limit of Detection (LOD) and Limit of Quantification (LOQ).

**Results:** The optimized temperature settings for the GC oven showed minimal baseline errors, starting at 40°C, reaching 180°C, and then 240°C. The most effective SPME fiber was 50/30µm PDMS/DVB/CAR, identifying 12 compounds with a total peak area of 6.3797 x 10<sup>8</sup> mAU. Using RSM-CCD analysis, the best conditions for extracting VOCs were found to be at 41°C for 31 minutes, achieving a desirability score of 1. The validated method had a linear response ( $R^2 = 0.9931$ ) for concentrations ranging from 0.5 to 2.0 mg/mL. The LOD and LOQ values for Olive House were 0.608 mg/mL and 2.027 mg/mL, for Shifaa, 0.195 mg/mL and 0.65 mg/mL, and for Zahra, 0.211 mg/mL and 0.703 mg/mL. Intra-day precision (%RSD) was determined as 22.7% for Olive House, 15.1% for Shifaa, and 13.04% for Zahra by analyzing three repeated samples on the same day. Showed a percentage recovery of 110% for Olive House, 69.6% for Shifaa, and 90% for Zahra, demonstrating the method's accuracy.

**Conclusion:** Overall, the optimized and validated method enhances the accuracy and reliability of analysing *Nigella sativa* seed oils, ensuring the authenticity of the product.

**Keywords:** GC-MS, HS-SPME, *Nigella sativa.L*, RSM-CCD, VOCs

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