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## Optimization of Volatile Organic Compound (VOC) In Stingless Bee Honey Using the Solid Phase Microextraction (SPME) Method with Gas Chromatography- Mass Spectrometry (GC-MS)

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### Structured Abstract

**Background:** Honey, a natural sweet substance produced by honeybees, has been widely recognized for its medicinal properties and nutritional benefits. Stingless bees, known as kelulut bees in Malaysia, produce honey with enhanced therapeutic benefits, showcasing antioxidant, anti-inflammatory, and antimicrobial properties. This unique honey has gained attention for its potential in the pharmaceutical industry, particularly for wound healing applications. Unlike traditional honey, stingless bee honey possesses distinct characteristics, including a horizontal hive structure and specific production processes. The purpose of this study is to optimize the volatile organic compound (VOCs) using (SPME) and Response Surface Methodology Central Design (RSM-CCD).

**Methods:** The stingless bee honey was extracted using Solid Phase Microextraction (SPME) method. 4 g of stingless bee honey sample was mixed with 4 mL of saturated salt solution (NaCl) and transferred to a 15 mL clear glass screw cap vial (PTFE/silica septa). The fiber was exposed to the sample in the headspace (HS) - SPME mode. For 30 minutes, the vial was incubated at 60°C with steady magnetic stirring. The study of the volatile components in the headspace sample was then completed using (GC-MS).

**Results:** The method was verified in terms of precision, accuracy, sensitivity, coefficient of determination, repeatability, and recovery. Inter and intra-day replicate analyses were used to measure accuracy and precision. Three times, 2000 ppm B-caryophyllene was injected into the GC-MS for analysis. Precision was expressed as a percentage of the relative standard deviation (%RSD), and accuracy was expressed as a percentage of the predicted concentration. The method's sensitivity was established by the limits of quantification (LOQ) and detection (LOD). The coefficient of determination ( $r^2$ ) was assessed using a five-point calibration curve over the range of 0.5 ng/ml–5000 ng/ml.

**Conclusion:** In conclusion, our study successfully developed a quick and reliable method for analyzing volatile organic compounds (VOCs) in stingless bee honey using GC-MS. The validation of this method, which employed solid-phase microextraction (SPME) fibers and gas chromatography-mass spectrometry (GC-MS), demonstrated its effectiveness. The validation process, covering precision, accuracy, limit of detection (LOD), and limit of quantification (LOQ), showed moderate variability in replicate concentrations. This indicates commendable precision and consistency in our analytical measurements.

**Keywords:** Stingless bee honey, VOCs, SPME, RSM-CCD, GC-MS

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