

## **Optimizing the Microwave-Assisted Digestion Parameters using Box-Behnken Design for Determination of Antimony in Polyethylene Terephthalate (PET) by Inductively Coupled Plasma - Optical Emission Spectrometry**

Nur Faten Hazira binti Mohd Ghazali<sup>a</sup>, Nor Suhaila Mohamad Hanapi<sup>ab\*</sup>

### **Structured Abstract**

**Background:** Antimony, a toxic heavy metal, is widely used as a catalyst in the production of polyethylene terephthalate (PET) due to its high catalytic efficiency and cost-effectiveness. However, its potential to leach into consumables raises significant health and environmental concerns, highlighting the need for accurate quantification methods. This study aimed to optimize microwave-assisted digestion parameters for the determination of antimony in PET using inductively coupled plasma-optical emission spectrometry (ICP-OES). The optimization process employed the Box-Behnken Design (BBD) to systematically evaluate and refine three critical factors: digestion temperature, digestion time, and sample weight.

**Methods:** The procedure involved sample collection, preparation using a blender, digestion using microwave-assisted acid digestion, and analysis by ICP-OES. Data were evaluated using the Box-Behnken Design (BBD) to systematically optimize three critical parameters: digestion temperature, digestion time, and sample weight.

**Results:** A total of 15 experimental runs were conducted, including three replicates at the center point. Statistical analysis confirmed the model's significance, with an R<sup>2</sup> value of 0.9945, indicating that 99.45% of the variability in the response could be explained by the model. The calibration curve, constructed over the range of 0–10 ppm, showed excellent linearity, with an R<sup>2</sup> value of 0.9885, confirming the reliability of the analytical method. The optimal conditions were determined as 200°C digestion temperature, 15 minutes digestion time, and 0.1 g sample weight. Under these optimized conditions, the method exhibited excellent analytical performance, achieving a limit of detection (LOD) of 1.079 mgL<sup>-1</sup> and a limit of quantification (LOQ) of 3.270 mgL<sup>-1</sup>. A spike recovery rate of 106% and 0.81% RSD further validated the method's accuracy and precision, demonstrating its robustness in analyzing complex PET matrices.

**Conclusion:** In conclusion, compared to conventional methods, the optimized approach significantly reduced digestion time, minimized reagent use, and maintained comparable sensitivity, making it both efficient and sustainable. These findings validate the reliability of the developed method for routine antimony analysis in PET matrices. Additionally, this framework can be extended to quantify other heavy metals in PET or similar materials, contributing to enhanced safety compliance, and environmental sustainability.

**Keywords:** Antimony Quantification, Polyethylene Terephthalate, Box-Behnken Design, Microwave-Assisted Digestion

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\*Correspondence: [norsuhaila979@uitm.edu.my](mailto:norsuhaila979@uitm.edu.my)

<sup>a</sup> School of Chemistry & Environment, Faculty of Applied Sciences, Universiti Teknologi MARA, Shah Alam, Malaysia

<sup>b</sup> Trace Analysis Research Group, Universiti Teknologi MARA, Shah Alam, Malaysia