cbecimat26
26° Congresso Brasileiro de Engenharia e Ciência dos Materiais
22 a 26 de novembro de 2026
Costão do Santinho
Florianópolis-SC

Study And Implementation Of Methodologies For The Identification And Quantification Of Erucamide Molecules Released By EU/MMT-NPZnO Nanocomposites Using High-performance Liquid Chromatography (HPLC)

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Introduction

Erucamide is an unsaturated fatty acid amide derived from erucic acid and has been widely used as a slip additive in polymeric materials, particularly in the production of plastic packaging. The identification and quantification of erucamide are critical steps for both quality control and understanding material behavior over time. In this context, High-Performance Liquid Chromatography with Diode Array Detection (HPLC-DAD) offers promising potential for this purpose.

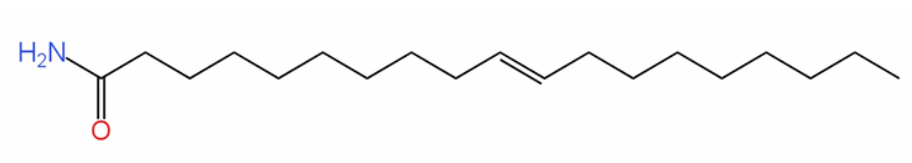


Figure 1. Structure of the polymeric slip additive erucamide.

Objectives

- Conduct solubility tests of erucamide using acetonitrile and methanol as solvents, evaluating their efficiency and compatibility with the analytical method.
- Assess whether the solvent affects the sample analysis, considering that solvents may also absorb UV radiation at wavelengths near 200 nm.
- Calculate analytical validation parameters, including the limit of detection (LOD), limit of quantification (LOQ), and linearity, to ensure the robustness and reliability of the method.

Methodology

- The erucamide (EU) used in this study was supplied by the Tec-Pol (Polymer Technology) group and manufactured by PMC Biogenics (South Korea).
- The stock solutions were prepared at an erucamide concentration of 1000 ppm. A mass of 25 mg of erucamide was weighed and dissolved in 25 mL of an acetonitrile–methanol mixture (50:50, Solution A) and 25 mL of methanol (Solution B).
- For HPLC analysis, an Agilent Microsorb-MV 100 C18 column (250 × 4.6 mm, 5 µm particle size) will be used, with a mobile phase consisting of water and methanol (5:95, v/v). The injection volume will be 10 µL, the flow rate will be 1 mL/min, the column temperature will be 50 °C, and the run time will be 15 minutes.
- LOD and LOQ will be calculated using the equation presented in Figure 2.

$$LD = 3{,}30 * h \qquad LQ = 10{,}0 * h$$

Figure 2. Equations for determining the estimated peak heights corresponding to the limit of detection (LD) and limit of quantification (LQ)

Results & Discussion

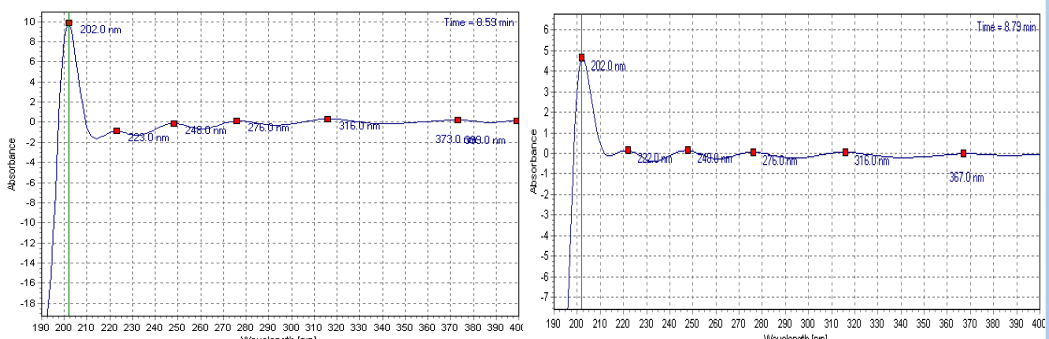


Figure 3. UV spectra of the 125 ppm erucamide samples from Solutions A (a) and B (b).

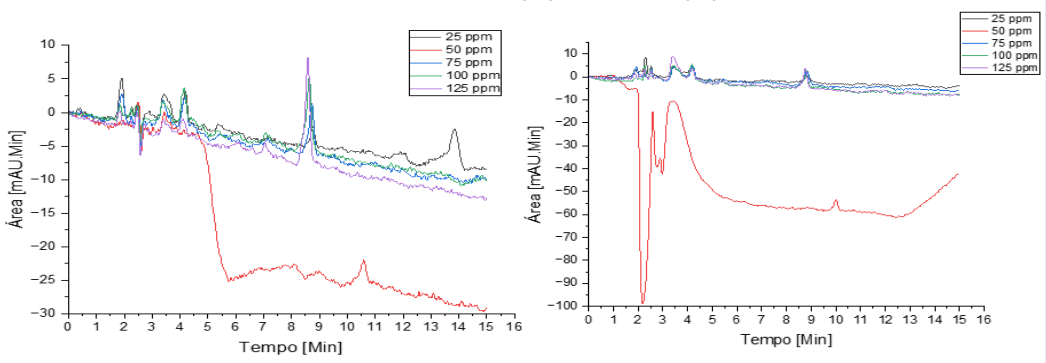


Figure 4. Chromatograms of the 25, 50, 75, 100, and 125 ppm concentration ranges for Solutions A (a) and B (b).

Concentration of EU (ppm)	Acetonitrile–Methanol (50:50) Area (mAU·min)	Methanol Area (mAU·min)
25	0,7	0,7
50	1,0	1,1
75	1,8	1,5
100	2,7	2,1
125	3,5	1,9

Table 1. Area values obtained from the chromatograms of the calibration curves for Solutions A and B.

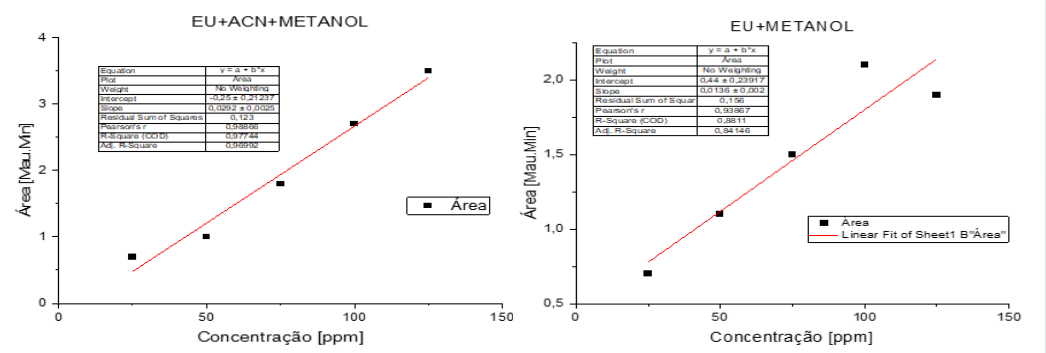


Figure 5. Calibration curves for Solutions A and B.

- The LOD and LOQ were calculated according to Equation 1, using the lowest concentration point of the calibration curve (25 ppm) as a reference. The obtained LOD and LOQ values were approximately 4.45 ppm and 15.15 ppm, respectively.

Conclusions

- Based on the preliminary results, the presence of erucamide (EU) could be identified through a characteristic signal in the chromatograms, with a consistent retention time across most concentrations and an absorption spectrum compatible with that of the reference standard (λmax = 202 nm).
- The linearity of the calibration curves was satisfactory for at least one of the solutions, suggesting the feasibility of the method for quantitative purposes.

References & Acknowledgements

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