

Optimasi metode analisis katekin dan epikatekin dalam biji kakao serta produk olahannya secara kroma tografi cair spektrometri massa = Optimization on Analytical Methods of Catechin and Epicatechin in Cacao Bean and Cacao Products Using Liquid Chromatography Mass Spectrometry

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Abstrak

Cacao bean Processing into its products may affect catechin and epicatechin contents in the final products. Temperature treatments during cacao processing can induce epimerization reaction of (-)-epicatechin to be (-)-catechin. The aims of this study were : (1) to obtain the valid analytical method which can be used to analyze catechin and epicatechin in cacao bean and cacao products by liquid chromatography mass spectrometry, and (2) to know the influence of temperature during Processing of cacao beans, especially concentration of catechin and epicatechin.

Experiment was conducted in three steps, i.e.: optimization of analytical method, validation of selected method, and studying the influence of temperature to catechin and epicatechin concentration during cacao bean processing. Optimization of analytical method was carried out by varying solvents (acetonitrile and methanol) composition using gradient elution. Mobile phase flow rate was set at 0.5, 0.6, 0.8, and 1.0 ml/min. Catechin and epicatechin in cacao samples was detected by mass spectrometry. Condition of mass spectrometer was run by varying ESI voltage, nebulizer pressure, desolvation temperature, and desolvation gas. Validations test included some parameters such as specificity/ selectivity, linearity of calibration curve, limit of detection and limit of quantitation, precision and recovery test. Samples taken during the process were cacao nib, cacao mass, cacao powder and cacao butter.

The results of this study showed that analytical conditions for catechin and epicatechin were using mobile phase A (0.1 % formic acid in deionized water) and mobile phase B (acetonitril-methanol - 50:50) at flow rate of 0.5 ml/min. Gradient elution were set at 0 minutes (10% B), 15 minutes (35% B), 20 minutes (40% B), 30 minutes (50% B), 35 minutes (60% B), and 35.1 minute (10% B). Mass spectrometer was set at ESI voltage (-) 3500 volt, desolvation temperature 300 °C, nebulizer pressure 50 psi, desolvation gas 10 L/min, and fragmentor voltage (-) 160 volt. Limit of detection and limit of quantitation of catechin were 0.28 and 0.93 ppm, respectively, while epicatechin were 7.15 and 23.84 ppm, respectively. Based on concentrations of catechin and epicatechin, heat treatment during cacao mass Processing showed a decrease tendency of catechin and epicatechin ratios.