OPTIMASI DAN VALIDASI METODE SPEKTROFOTOMETRI UV-VIS DERIVATIF PADA PENETAPAN KADAR KALSIUM DAN MAGNESIUM DALAM IKAN TERI (*Stolephorus spp*.)

ABSTRAK

Kalsium dan magnesium memiliki peran penting dalam tubuh, sehingga pemenuhan kebutuhan asupan keduanya harus dikontrol. Asupan kalsium dan magnesium dapat diperoleh dari ikan teri (*Stolephorus spp*.). Dibutuhkan metode analisis yang mampu menetapkan kadar kalsium dan magnesium pada ikan teriagar kebutuhan keduanya seimbang. Beberapa metode analisis yang digunakanadalah SSA dan kompleksometri, tetapi pelaksanaannya masih memiliki kelemahan yaitu preparasi sampel yang panjang, mahal, kurang spesifik dan sensitif. Metode spektrofotometri derivatif dapat digunakan untuk mengatasi hal tersebut. Selain lebih sederhana, metode ini lebih murah, cepat, serta dapat meningkatkan sensitivitas. Tujuan penelitian ini adalah mendapatkan metode penetapan kadar kalsium dan magnesium dalam ikan teri yang selektif, peka, teliti, dan akurat.

Jenis Penelitian *analitical observasional*. Penelitian dilakukan dengan optimasi metode meliputi optimasi pelarut dan reagen, penetapan spektra UV-Vis, λ analitik, *operating time*, dan orde derivatif. Preparasi sampel dilakukan dengan metode destruksi basah. Terakhir dilakukan validasi metode meliputi selektivitas, linearitas, akurasi, presisi, LOD, LOQ dengan menggunakan spektrofotometer UV-Vis *dual beam*.

Hasil penelitian menunjukkan bahwa kondisi optimum kalsium dan magnesium pada lamda 555 nm orde 2 dan 466 nm orde 4. Hasil validasi kalsium dan magnesium mempunyai nilai koefisien korelasi (r) 0,9650 dan 0,9892. Nilai recovery kalsium dan magnesium adalah 87,17-100,55% dan 89,74-109,29%, nilai RSD 1,5-4,8% dan 4,35-5,5%, nilai LOD 5,7166 ppm dan 66,6942 ppm, nilai LOQ19,0555 ppm dan 222,31 ppm, dan keduanya selektif pada lamda 555 nm dan 466 nm.

Kesimpulan yang diambil bahwa penetapan kadar kalsium dan magnesium dalam ikan teri (*Stolephorus spp*.) secara spektrofotometri derivatif memenuhi persyaratan selektif, peka, teliti, akurat, dan linear.

Kata Kunci: Optimasi metode, validasi metode, spektrofotometri derivatif, kalsium, magnesium, ikan teri (*Stolephorus spp*.).

OPTIMIZATION AND VALIDATION OF DERIVATIVE UV-VIS SPECTROPHOTOMETRIC METHODS FOR THE DETERMINATION OF CALCIUM AND MAGNESIUM IN ANCHOVY (*Stolephorus* spp.)

ABSTRACT

Calcium and magnesium have an important role for good health. Thus, the dietary intake of calcium and magnesium should be controlled. They can be obtained from Stolephorus spp. is one of the sources of calcium and magnesium. Hence, the method for analysis the level calcium and magnesium level of Stolephorus spp. is required. It was aimed to make both intake balanced. There were some methods of analysis which were used. They were SSA and complexometry but in the implementation, the disadvantages still found. They were a long sample preparation, expensive, less specific and less sensitive. Hence, derivative spectrophotometric method can be used to overcome these weaknesses. This method was simpler, cheaper, fast, and increase the sensitivity. This research was aimed to optimize and validate the derivative UV-Visible spectrophotometric methods for analysis of calcium and magnesium in Stolephorus spp.

This was an observational analytical study. In phase I, the optimization methods included the optimization of reagent addition, the determination of wavelength and derivatives order used for the analysis, as well as the determination of operating time. The samples were prepared by wet destruction method. In phase II, the milk sample were validated according to ICH guideline including selectivity, linearity, accuracy-precision, LOD and LOQ using dual beam UV-Vis spectrophotometer.

The result showed that the optimum condition of selective calcium determination in Stolephorus spp. was found in 2nd order to derivatives wavelength 555 nm with linearity (r=0.9650) and the optimum conditions of selective magnesium determination levels were found in 4th order to derivatives wavelength 466 nm with linearity (r=0.9892) the calcium and magnesium values of recovery, %RSD, LOD, and LOQ were found in the 87.17-100.55% and 89.74-109.29%, 1.5-4.8% and 4.35-5.5%, 5.7166 and 66.6942 ppm, 19,0555 and 222.31 ppm respectively.

In conclusion, the determination of calcium and magnesium can be performed simultaneously with derivative uv-vis spectrophotometric method and the method meet the validation requirements.

Keywords: Optimization methods, validation methods, derivative UV-Vis spectrophotometric method, calcium, magnesium, (Stolephorus spp.)