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ANALYSIS OF OXALATE LEVELS IN BROCCOLI (*Brassica oleracea, L.*) AND CAULIFLOWER (*Brassica oleracea. L vr botrytis sub var. Cauliflora DC)* USING THE UV SPECTROPHOTOMETRY METHOD

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ABSTRACT

Vegetables are nutritious food containing vitamins and minerals needed by the body and cheap prices. Brukoli and cabbage flowers are many vegetables containing vitamins A, B, C, minerals, and calcium. In addition to vegetables that contain lots of nutrients, vegetables also contain anti-nutrition, one of which is oxalic acid. Oxalic acid can interfere with the intestinal lining if consumed and can be fatal if it is in high doses so it needs to be limited to the safe range of 0.60-1.25 g per day. The purpose of this study is to determine the levels of oxalate in broccoli and cabbage. flower. The method used is UV spectrophotometry with a wavelength of 351 nm. This research begins with the validity of the method which includes linearity, detection limits, quantization limits, precision and accuracy using addition methods. The results of the validation of the calibration curve analysis method have a regression line equation y = 0.0224 x + 0.2407 with a correlation coefficient of 0.993. The detection limit and the quantization limit can be calculated statistically respectively 1.0045 and 3.4860. Variance coefficient of 1.7570 and percent recovery, respectively 87, 89 and 92%. The results of this study showed that the levels of oxalate in broccoli were 1.3797 mg / 100 g and cauliflower was 0.1707 mg / 100 g. Oxalate levels in broccoli and cauliflower are at safe limits.

Keywords: Broccoli, cauliflower, oxalate, UV spectrophotometry

INTRODUCTION

Vegetables are one food that is nutritious, easily obtained, and the price is cheap and can be reached by all levels of society. Vegetables are foods that contain vitamins and minerals needed by the human body, although in relatively small amounts. Vegetables are part of the Indonesian people's diet that can be processed into various dishes **[1]**.

Vegetables contain lots of vitamins A, B, C, minerals and calcium, and contain lots of calories, protein, fat, and carbohydrate. Every content contained in vegetables is needed by our body. But vegetables also contain several non-essential nutrients found in their tissues.

Broccoli (*Brassica oleracea*, L.) and Flower Cabbage (*Brassica oleracea*. L vr botrytis sub var. Cauliflora DC) belong to the cabbage family which is a vegetable plant family of Brassicaceae Broccoli contain a variety of important minerals such as calcium, potassium, iron, and selenium. Flavonoids and fiber also enrich the nutritional content of broccoli. The content of Vitamin C in Broccoli is 93.2 mg / 100 g **[2]**. Broccoli which is an authentic Italian vegetable can be consumed or eaten raw, boiled, or for soup. Broccoli is high in Vitamin B, Vitamin C, Folic Acid and Beta Carotene. Besides, broccoli also contains several minerals, such as calcium, iron, phosphorus, potassium, and sulfur **[3]**.

Flower cabbage is a vegetable that has many benefits, one of which is to meet the nutritional content of the community because cauliflower contains protein, fat, calories, carbohydrates, fiber, calcium, potassium, ash, phosphorus, iron, sodium, niacin, vitamin A, vitamin B1, vitamins B2, vitamin C and water. **[4]**.

In addition to vegetables that contain lots of nutrients, vegetables also contain anti-nutrition, one of which is oxalic acid. Oxalic acid can irritate the intestinal lining if consumed and can be fatal if it is in high doses. Oxalic acid can combine with metals such as calcium in the body to form oxalate crystals that can irritate the intestine and kidneys.

Oxalic acid found in broccoli will form neutral salts with alkali metals such as Sodium and Potassium which have high solubility in the water while Calcium

Oxalates are practically insoluble in water. Oxalic acid in addition to having many uses can also cause toxic to the body if consumed in large amounts of high levels. According to **[5]**, high oxalate content in urine or blood components can cause kidney disease, vitamin deficiencies, intestinal diseases, and hyperoxaluria. The formation of oxalate crystals in plants or vegetables will cause kidney stone disease

if the vegetables are consumed by humans in excess.

The oxalic acid content of broccoli (*Brassica oleracea*, L.) and cauliflower (*Brassica oleracea*. L vr botrytis sub var. Cauliflora DC) using UV-Visible spectrophotometric method with a wavelength of 352 nm. Oxalate can activate the catalytic oxidation reaction of iodide by bromate with iron (II) as a catalyst by producing I3 which is formed in proportion to the oxalate in the sample **[6, 7]**. In addition to the spectrophotometric method analysis of oxalic acid can use High-Performance Liquid Chromatography and qualitative analysis using the FTIR method.

MATERIAL AND METHODOLOGY

Sample Preparation

Samples were taken from several supermarkets in Bandung. Beginning by cutting the sample into small pieces and weighing as much as 1 gram, put into a beaker and add 10 ml of hot aqua dest and filtered using filter paper

Determination of the Calibration Curve

For the determination of the calibration curve, a series of standard solutions with series of concentrations of 2, 4, 6, 8, 10, and 12 ppm were made, and then a pipette of 1 ml each was put into a 10 mL flask. To each flask add 2 mL acetate buffer solution (pH 5), 1 ml Fe (II) ammonium sulfate, 1 ml KI 0.12 mol / L, 1 ml (KbrO3) 0.1 mol / L and dilute with aqua dest to the limit mark. Measure the absorbance at a wavelength of 351 nm.

The graph is obtained by plotting the relationship between absorbance and weight in 10 mL of a standard solution. As a parameter for the existence of a linear relationship the coefficient of determination (r2) and the coefficient of regression function variance are used in the linear regression analysis Y = bx + a.

Based on the calibration curves obtained, calculations, linearity, detection limits, and quantization limits are performed

Determination of Equality

Equivalence is measured by measuring 8 ppm oxalate concentration, a standard of concentration of 100 ppm pipette as much as 0.8 ml put in a 10 ml flask and then extracted as much as 3.0 ml sample add reagents carried out repetition 6 times measured at a wavelength of 351 nm

Determination of Accuracy

The accuracy parameter is shown by calculating the percent recovery through the addition method by measuring 3 different concentrations of 4, 8 and 12 µg / ml. then treated as the preparation of the test solutions with each of the three replications and measured at a wavelength of 315 nm. Determination of accuracy is done by calculating the recovery,

Sample evaluation

The sample extract was pipetted as much as 1ml added with 2ml of acetate (pH 5). 1 ml

Fe (II) ammonium sulfate, 1 ml KI 0.12 M and 1 ml KBrO3 0.1 M and measured at 315 nm wave stage

RESULTS AND DISCUSSION

According to **[6]** on the determination of Oxalate can be determined by UV Vis spectrophotometry, Oxalate acts as an activator of the Fe (II) catalyst so that a reaction between BrO3 and KI can result in triiodides that are measured in spectrophotometry with a wavelength of 352 nm.

For the determination of oxalate wavelengths using standard oxalate solution with a concentration of 10 µg / ml measured in the range of wavelengths 200 - 400 nm and the maximum absorption obtained at 315 nm wavelength can be seen in **Figure 1**.

[8] explained that the ability of analytical methods to provide proportional responses to the concentration of analytes is expressed in linearity. Linearity can be obtained from the oxalate calibration curve. This oxalate calibration curve can be made from oxalate mother liquor made in 6 series of multilevel concentrations, ie concentrations of 2 - 12 µg / ml **(Figure 2)**.

Method Validation

Based on the measurement results of the calibration curve, the linearity region was obtained at a concentration of 2, 12 µg / ml. Calibration curves can be seen in Figure 2 and linearity parameter data can be seen in **Table 1.**

Based on the data from the calibration curve calculated statistically obtained by equation Y = 0.0224X +0.2404 with a correlation coefficient r of 0.993. This r value meets the requirements of the correlation coefficient for contamination that is r2 ≥ 0.98. These results indicate that in the range of 1-12 µg / ml the linearity test is fulfilled The coefficient of regression variance obtained is 1.75% lower than the requirement of ≤ 5%. The detection limits and quantization limits were 1,004 and 3,468 µg / ml, respectively

In this research, the accuracy test is done by adding severalndard methods (standard addition method) with a certain concentration on the sample to be examined and then analyzed with the method that has been determined. The reason the method is used is that the sample is in the form of natural materials or in the form of secondary metabolites where the matrix level is not known with certainty. In this method, three different concentrations are used, namely 4, 8, and 12 **(Table 2)**. From the research data obtained the value of percent recovery (amounting to 87-92%), the results obtained are said to be sufficient good because it meets the percent requirement of recovery is 80-110% **[8]**.

The precision test conducted in this study was conducted with repeatability and

reproducibility parameters. This test is carried out by measuring the oxalate solution that has been added by several samples made 6 repetitions measured for 3 days using a UV spectrophotometry at a wavelength of 351 nm. Equality criteria are given if the method gives a 2% or less relative standard deviation (KV). Examination (precision) with the parameter repeatability (reparability) is carried out the same analit under the same conditions and at short time intervals (in days) **(Table 3)**. This repeatability method aims to determine the consistency of the analyte, the level of difficulty of the method and the suitability of the method.

Based on the results obtained **(Table 4)** from the precision test with repeatability parameters, the% KV value of each treatment is less than 2%, where the KV values are 0.3468%, 0.2655%, and 0.1936%, the results show that the test method used in the determination of oxalate levels using UV spectrophotometry has good accuracy. Judging from the results obtained from the RSD data shows that from the results of precision carried out three times on different days get 0.3468%, 0.2655%, and 0.1936%. So it can be concluded that the precision test conducted is valid because the results of% RSD show less than 2%.

Equality test with parameters of reproducibility is a method of equality carried out by the same analyte, different times (between days). The research was carried out by taking at least 6 mixed solutions of sample and oxalate and measured by UV spectrophotometry. The results obtained are seen in table VI.5

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Determination of levels

Oxalate levels were determined from fresh vegetables including broccoli and cauliflower. First, the fresh sample is mashed and then weighed as much as 1 gram after it is brewed with hot water, soaking the sample is done for about 10 minutes, the goal is that the oxalate contained in vegetables can be pulled completely **(Table 5)**. After soaking, the filtered sample is put into a 25 ml volumetric flask, then the aqua dest solvent is added to the boundary mark. Then the absorption of each solution that has been made is measured using UV-visible spectrophotometric Shimadzu 1800 brand, analysis using spectrophotometry is one of the most widely used methods for the determination of content, because this method is relatively inexpensive and easy.

The determination of content is measured at a wavelength of 351 nm. Oxalate acts as an activator of the Fe (II) catalyst so that a reaction between BrO3 and KI can result in triiodides being measured in spectrophotometry with a wavelength of 351 nm **[6]**.

Figure 1: Maximum wavelength of oxalate

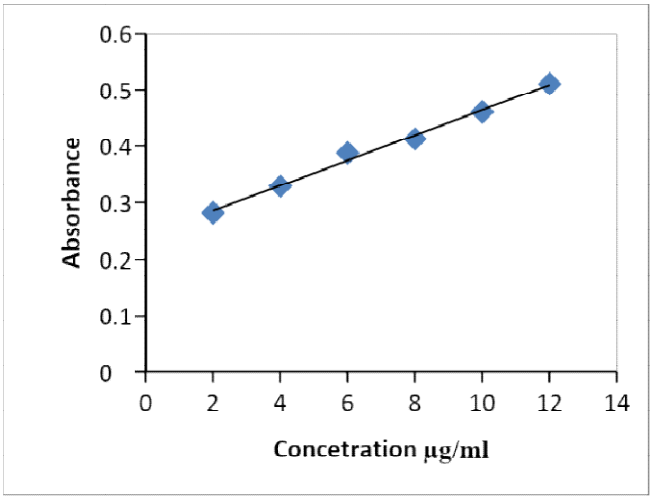

Figure 2: oxalate calibration curve

Table 1: Linearity Parameters

Name	Nilai	Syarat
Linear regression equation	Y=0,0224x+0,2407	
Slop((b)	0,0224	
Intercept(a)	0,2407	
Sy/x	0,007809	
Koefisien Korelasion(r)	0,993	>1
OD	1,0045801	
LOQ	3,486004	
Vxo%	1,757049	≤ 5,0%

Table 2: Oxalate Accuracy Results

S. No	Concentration (µg/ml)	measurable levels	Recovery (%)	Average (%)	SD
1	4	8,8527	89	87	0,1568
		8,9866	85		
		9,1652	85		
2	8	12,6027	91	89	0,0893
		12,6920	89		
		12,7813	88		
3	12	16,5313	93	92	0,6753
		16,6205	92		
		16,6354	91		

Table 3: Oksalat precision results data (in days)

No	Day 1 concentration	Day2 concentration	Day3 concentration
1	12,6027	12,6027	12,6027
2	12,6920	12,6473	12,6473
3	12,6473	12,6473	12,6473
4	12,6920	12,6920	12,6473
5	12,6920	12,6920	12,6027
6	12,6027	12,6473	12,6027
Average	12,6548	12,6548	12,6250
SD	0,0439	0,0336	0,0244
%KV	0,3468	0,2655	0,936

Table 4: Oxalate precision data (between days)

Days to	Concentration (µg/ml)
1	12,6548
2	12,6548
3	12,6250
Average	12,6448
SD	0,0341
%KV	0,2689

Table 5: Results Sample Content

Sample	Oxalic levels (mg/100g)	SD	Average
Brocoli	1,3313		
	1,3871	0,1804	1,3797
	1,4206		
Kembang kol	0,1038		
	0,1819	0,2486	0,1707
	0,2265		

From the measurement results using UV spectrophotometry, the absorption data of each vegetable sample can be used to measure the levels of each sample by converting available absorbance data into the form of concentration (µg/ml) via the linear regression equation y = 0, 0224x + 0.2407. Based on the results obtained (**Table 5)** for the highest levels of oxalate in broccoli in the amount of 1.3797 mg / 100 g and cauliflower at 0.1707 mg / 100 g.

CONCLUSION

The results showed oxalate levels in broccoli and cauliflower samples using UV spectrophotometry methods respectively 1.3797 mg / 100g broccoli and 0.1707 mg / 100g cauliflower Oxalate levels in broccoli and cauliflower are at safe limits.

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