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REVIEW ANALYSIS OF ORGANOCHLORINE PESTICIDE RESIDUES IN PLANTS WITH GAS CHROMATOGRAPHY METHODS

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ABSTRACT

The use of pesticides in the agricultural sector can overcome pest attacks and diseases on crops. Pesticide residue levels that exceed the maximum residue limit (BMR) will be a bad effect on health and the environment. Pesticides that cause the most damage to the environment and human health are the organochlorine group. Levels of organochlorine pesticide residues can be analyzed through gas chromatography (GC) methods. This study aims to describe the levels of organolcorin pesticide residues in a wide variety of plants using gas chromatography (GC) methods. The results of analysis from several literature studies show that the use of gas chromatography (GC) methods can analyze organochlorine pesticide residues contained in plants because they have a high level of sensitivity.

Keywords: Pesticides, Organochlorin, Gas Chromatography

1. INTRODUCTION

Chromatography is a physical method for the separation of distributed components between two phases. Separation with chromatography is based on the difference in the equilibrium of the components of the mixture between the stationary phase and the movement phase [1]. One type of chromatography that is often used is gas chromatography. Gas chromatography is a way to separate a mixture by flowing a current of gas through a stationary phase. The characteristics of samples that can be analyzed with gas chromatography are volatile, have thermal stability at operating temperature, and can first be privatized into volatile materials [2]. Gas chromatography is a dynamic method for the separation and detection of volatile compounds in a mixture. The general use of gas chromatography is to perform qualitative and quantitative analysis of compounds in a mixture [3]. The principle of separation using gas chromatography is based on the volatility properties of each component of the mixture [4]. The method of separating mixtures into these components is based on the interaction of the movement phase and the stationary phase[5]. The phase of motion in gas chromatography is usually helium, hydrogen, or nitrogen gas. The mechanism of gas chromatography is in high-pressure steel cylinders streamed through columns containing stationary phases. The mixture to be separated, usually in the form of a solution, is injected into the gas stream. Then the footage is carried by the carrier gas into the column and inside the column, there is a separation process. The components of the mixture that have been separated one by one leave the column. A detector is placed at the end of the column to detect the type or number of each component of the mixture.

The detection results are recorded with a recorder and called a chromatogram consisting of several peaks. The number of peaks produced states the number of components (compounds) contained in the mixture. If a chromatogram consists of 4 peaks then there are 4 components or 4 compounds in the mixture. While the area of the peak depends on the quantity of a component in the mixture, because the peaks in the chromatogram are triangular, the extent can be calculated based on the height of the width of the peak.

One analysis that can be done using gas chromatography methods is the analysis of pesticide residues. Pesticide is a substance or mixture of both natural and synthetic substances formulated to control and repel pests [7]. The use of pesticides will produce pesticide residues. Pesticide residues are certain substances contained in agricultural produce of foodstuffs or animal feed, either as a direct or indirect result of pesticide use. It is this residue that can damage the environment and spread disease to the surrounding environment [8]. This is because pesticide residues are accumulative in the human body, so it will have a negative impact on the health of humans who consume vegetables that contain pesticide residues continuously. Pesticides that cause the most environmental damage and threaten human health are synthetic pesticides, namely organochlorine. Organochlorine pesticides are a versatile group of chlorinated hydrocarbon chemical compounds. Organochloric pesticides degrade slowly so that they will have a long stay in foodstuffs and the environment [9]. The level of damage caused by organochlorine compounds is higher than other compounds, because these compounds are sensitive to sunlight and do not decompose easily.

Gas chromatography is one of the most sensitive instruments for determining the content of pesticide residues that have stable thermal properties, low polarity and volatile, such as organophosporus, pyretroid and organochlorin. With these specifications, gas chromatography can be widely used to analyze the levels of pesticide residues in a compound [10]. The GC method is preferred in pesticide analysis because it is able to achieve high sensitivity. Analysis of organochlorine pesticide residues can be done by way of extracted samples. The mixture of components that have been extracted will be carried by the carrier gas into the column, resulting in separation. Separation can occur due to differences in boiling points between components to be separated, and because of the interaction of components with columns. Components that have been separated are detected by detectors that have good sensitivity to compounds containing high electronegative elements [11].

This research will focus on analyzing the content of organochlorine pesticide residues in various plants. The purpose of this study was to analyze the levels of organolcorin pesticide residues in a wide variety of plants using the gas chromatography (GC) method.

2. EXPERIMENTAL

2.1 Analysis of Red Chili Pesticide Residues with Gas Chromatography

In studies that have been conducted [12] with the aim of knowing the levels of organochlorine pesticide residues in large red chili samples. The study was conducted using gas chromatography (Type 450-GC variant) with Electron Capture Detector (ECD) detector. Organic solvents used to extract organochlorine pesticide residues are acetone, dichloromethane, and petroleum ether by the same ratio. The carrier gas in pesticide residue analysis is nitrogen gas and is regulated at a flow rate of 1.5 mL/min. Determination of pesticide residues of red chili samples is carried out in 3 stages, namely sample preparation, measurement, and calculation of yamg data is done 3 times.

2.2 Determination of Pesticide Residue Levels in Commercial Teas Using Gas Chromatography

Research conducted by [13] aims to analyze α -endosulfan compounds which are one type of organochlorine pesticide. Tea samples derived from 7 (seven) trademarks with Code: I - VII were randomly taken from 3 (three) local markets with Codes 1-3 in West Java, Indonesia. Pesticide residue analysis includes three stages: extraction, purification and analysis using Gas Chromatography (GC) which is equipped with electron capture detectors (ECD) Solvents used in this study namely acetone, dichloromethtan, n-hexan, dietileter and florisil. While the motion phase used in this study is helium and nitrogen gas.

2.3 Analysis of Pesticide Residues in Rice Commodities in Several Cities

The research conducted by [14] aims to analyze pesticide residues on rice commodities from Cianjur, Semarang and Surabaya. The method used in this study is gas chromatography with an electron capture detector (ECD). The phase of motion used in this study is nitrogen gas.

2.4 Pesticide Residues in Rice Production Centers in Central Java

Research conducted by [15] aims to identify pesticide residues in rice crop production centers in Central Java. Plant samples of 10 g each are put into a soxhlet paper tube, extracted with an acetone solvent of 100 ml for 6 hours at 80°C. After 6 hours the extraction results are evaporated to be somewhat dry in the evaporator at a temperature of 45°C. Pesticide residues obtained from evaporation are transferred into a 150 ml divided funnel with the help of a 25 ml n-hexant solvent, then extracted with a 25 ml acetoneile solvent 3 times. The n-hexane layer is at the top while the acetoneile layer is at the bottom in the FAAS. Next, it will be analyzed using GC instruments equipped with ECD (Electron Capture Detector) and FPD (Flame Photometric Detector) detectors.

2.5 Multi-Residue Analysis of Pesticides on Carrots Chromatography Gas

The study conducted by [16] aimed for the multi-residue analysis of pesticides on carrot samples with gas chromatography. The pesticide that will be analyzed in this study is organochlorine. The instrument used in the study is gc also equipped with a highly selective detector i.e. MS (Mass Spectrometry).

3. RESULTS AND DISCUSSION

3.1 Analysis of Red Chili Pesticide Residues with Gas Chromatography

Based on the results of research on Red Chili Pesticide Residues obtained data as in the following table.

Peptiside Organochlorine	Retention Time	Standard Peak Area
Lindan	8.28	3148.7
Heptaklor	8.66	19231.7
Aldrin	9.19	15689.4
Endosulfan	11.31	10284.5
Dieldrin	13.32	3990.7
Endrin	13.02	8158.6
DDT	15.25	4415.0

Table 1 shows the peak area and standard retention times of organochlorine pesticide mixtures. According to Table 1, lindan has the smallest retention time of 8.28 minutes, while DDT has the largest retention time of 15.25 minutes. This standard retention time is used as a reference when identifying components in a sample. Retention time is the time that indicates the length of time a component compound is held in a column [18]. Identification of components in a sample can be done by comparing sample retention times and standard retention times. For example, the peak that comes out near 8:28 on a sample chromatogram can be identified as lindan.

Table 2. Levels of Organochlorine Pepctiside Residue in Red Peppers [19].

Residue Organochlorine	Retention Time	Standard Peak Area
Lindan	0.0103	0.2

Heptaklor	0.0014	0.05
Aldrin	0.0028	0.1
Endrin	0.0052	0.05

In table 2. Describing the results of analysis of organochlorine pesticide residues in large red peppers showed that the red chili samples analyzed contained residues of lindan, aldrin, heptaklor and endrin pesticides with consecutive levels of 0.0103, 0.0014, 0.0028 and 0.0052 mg/L. The results when compared to SNI 7313 in 2008 are still below the Maximum Residue Limit (BMR), so the red chili is still safe for consumption.

3.2 Determination of Pesticide Residue Levels in Commercial Teas Using Gas Chromatography

Based on the results of research on Pesticide Residues in Tea obtained data as in the following table.

Table 3. Content Of A-Endosulfan In Commercial Tea In West Java – Indonesia^[20]

Kode**	Kandungan Senyawa Dalam Teh (ng/g)		
Sampel	α-endosulfan*	bifenthrin*	
1-1	4.87	4.29	
1-2	9.57	<mdl< td=""></mdl<>	
1-3	17.04	2.19	
H-1	<mdl< td=""><td colspan="2">1.38</td></mdl<>	1.38	
11-2	<mdl< td=""><td>1.49</td></mdl<>	1.49	
11 - 3	<mdl< td=""><td>1.41</td></mdl<>	1.41	
III - 1	<mdl< td=""><td>4.26</td></mdl<>	4.26	
111-2	<mdl< td=""><td>7.41</td></mdl<>	7.41	
III - 3	<mdl< td=""><td colspan="2">3.47</td></mdl<>	3.47	
IV - 1	<mdl< td=""><td colspan="2"><mdl< td=""></mdl<></td></mdl<>	<mdl< td=""></mdl<>	
IV - 2	3.87	<mdl 1.72</mdl 	
IV - 3	11.35		
V-1	<mdl< td=""><td colspan="2">1.42</td></mdl<>	1.42	
V-2	4.48	5.55	
V-3	1.53	4.64	
VI - 1	5.20	1.51	
VI - 2	18.12	2.08	
VI - 3	9.76	1.14	
VII - 1	2.48	<mdl< td=""></mdl<>	
VII-2	8.93	<mdl< td=""></mdl<>	
VII-3	20.16	<mdl< td=""></mdl<>	

*BMR α -endosulfan: 30 μ g/g (ppm) and biphentrin: $0.1-25~\mu$ g/g (ppm), MDL = 0.5~ng/g (ppb). ** I – VII : Tea trademark code, 1 – 3 : local Market code

The results of the analysis obtained are listed in Table 3 which shows the content of α -endosulfan compounds between < MDL s.d 20.16 ng/g (ppb) and bifenthrin compounds between <MDL s.d 7.41ng/g. BMR for α -endosulfan of 30 μ g/g. This shows that the analytical methods applied in commercial teas proved to be easy to use and highly sensitive. Analysis of pesticide residues in the commercial with GC-ECD with a method of analysis that has determined its performance, provides information

that commercial tea circulating in the local market, especially West Java Indonesia, is safe to consume.

3.3 Analysis of Pesticide Residues in Rice Commodities in Several Cities

Based on the results of research on Pesticide Residues in Rice Commodities obtained data as in the following table.

Table 4. Positive Results Of Organochlorine Pesticide Testing^[21]

10.2 1017	Konsentrusi residu (mg/kg)					
Asalisa	Sarabaya		Senarang		Clasjur	
	BL.	BI	BL	BI	BL	BI
ORGANOKLORIN						
BHC (Linden)	0,0075	×3				
Aldrin		*	0,0044	0,0048		
Heptakler	0,0349	8	-	0,0082		(*)
Dielárja		95	25	100	200	
Endoselfan			20	100	4.1	

The data in Table 4 illustrates that the levels of organochlorine pesticide residues detected are Lindan and Heptaklor. Rice commodities from the city of Cianjur do not contain residues of organochlorine pesticides.

3.4 Pesticide Residues in Rice Production Centers in Central Java

Based on the results of research on Pesticide Residues in the Production Center can be seen in Figure 1.

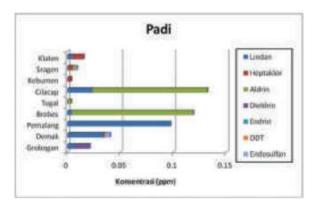


Figure 1. Composition of Organochlorine Residues in Rice Plants in Rice Production

Centers in Central Java [22]

In Figure 1 it can be seen that the domineerant organochlorine compounds found in rice plants are aldrin and lindan, mainly found in Cilacap, Brebes, and Pemalang counties.

3.5 Multi-Residue Analysis of Pesticides on Carrots Chromatography Gas

The standard solution of organochlorine with selected chromatography conditions has a chromatogram that can be seen in the following image.

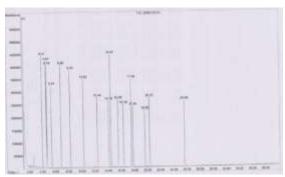


Figure 2. GC-MS chromatogram standard solution of organochlorine with a concentration of 7.0 ppm under selected conditions^[23]

Based on the results of research on Pesticide Residues in Rice Commodities obtained data as in the following table.

Table 5. Retention Time of Organochlorine Peptides Peaks In Gc-Ms Chromatogram^[23]

No	Waktu Retensi (t _R , menit)	Nama Pestisida	Kelas Pestisida	
1	3.213	Etophorphos	Organo fosfat	
2	3.921	α-Lindane	Organoklorin	
2 3 4	4.537	β-Lindane	Organoklorin	
4	4.643	γ-Lindane	Organoklorin	
5	5.144	Disulfoton	Organofosfat	
6	5.226	δ-Lindane	Organoklorin	
7	6.531	Methyl parathion	Organofosfat	
8	6.771	Heptachlor	Organoklorin	
9	7.055	Ronnel	Organofosfat	
10	8.144	Aldrin	Organoklorin	
11	8.582	Chlorpyrifos	Organofosfat	
12	10.123	Heptachlor epoxide	Organoklorin	
13	12.198	Endosulfan I	Organoklorin	
14	13.908	Dieldrin	Organoklorin	
15	14.192	p',p'-DDE	Organoklorin	
16	15.338	Endrin	Organoklorin	
17	16.089	Endosulfan II	Organoklorin	
18	17.24	p',p'-DDD	Organoklorin	
19	19.113	Endosufan sulphate	Organoklorin	
20	20.044	o,p'-DDT	Organoklorin	
21	25.286	Methoxychlor	Organoklorin	

Based on Table 5 it can be seen that the peaks of analytes detected have similarities with data on the library of 93%-99% for organochlorines and organophosphates. These results suggest that this method has the ability to measure the analyte in question specifically and selectively despite the other components in the sample matrix.

4. CONCLUSION

A number of studies conducted on the analysis of organochlorine pesticide residues in plants with Gas Chromatography Methods obtained the results that: (1) Levels of organochlorine pepctiside residues in red peppers showed that the red chili samples analyzed contained pesticide residues lindan, aldrin, heptaklor and endrin with consecutive levels of 0.0103, 0.0014, 0.0028 and 0.0052mg/L, (2) Pesticide Residue Levels in Commercial Teas showed that the samples contained α -endosulfan compounds between < MDL s.d 20.16 ng/g (ppb) and bifenthrin compounds. between <MDL s.d 7.41ng/g, (3) Pesticide Residue Levels in Rice Commodities illustrate that the levels of organochlorine pesticide residues detected are Lindan and Heptaklor, (4) The results of analysis of pesticide residues in rice production centers in Central Java can be seen that the dominant organochlorine compounds found in rice plants are aldrin and lindan, (5) Multi-residue levels of pesticides in carrots can be seen that the peaks of analyte detected have similarities. with data on the library of 93%-99% for organochlorines and organophosphates.

Based on the results of analysis from several literature studies that show that using gas chromatography methods (GC) can analyze the levels of organochlorine pesticide residues from various plants well because it is able to achieve high sensitivity. Thus, the use of gas chromatography method (GC) is effective and efficient for use in other plants.

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