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Perbandingan beberapa detektor kromatografi gas dan aplikasinya dalam analisis pangan

Comparison of gas chromatography detectors and its application in food analysis

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ABSTRACT

Gas Chromatography (GC) is an instrument used for analyzing gaseous compounds based upon differences of boiling point and polarity. The mobile phase of GC is an inert (unreactive) gas. GC consists of several components, one of them is a detector. Detector provides an electronic signal that is recorded, and component concentration shown in the chromatogram. A detector has interrelated properties to the other. The properties of the detector are considered under three main headings: (1) types of detector, (2) sensitivity, and (3) ease of operation. Ideal detector for one application is not necessarily ideal for another application. Application of GC coupled with a detector in food analysis are also informed. Detectors that are discussed are Thermal Conductivity Detector (TCD), Flame Ionization Detector (FID), Electron Capture Detector (ECD), and Mass Spectrometer (MS).

Keywords: comparison of detectors, detectors, gas chromatography, GC detectors, food analysis

ABSTRAK

Kromatografi gas merupakan instrumen yang digunakan untuk menganalisis komponen gas berdasarkan perbedaan titik didih dan polaritas. Fase gerak pada kromatografi gas merupakan gas inert (tidak reaktif). Kromatografi gas terdiri atas beberapa komponen, salah satunya adalah detektor. Detektor memberikan sinyal elektronik yang terbaca sebagai konsentrasi komponen pada kromatogram. Beberapa pertimbangan dalam pemilihan detektor adalah: (1) jenis detektor, (2) sensitivitas, dan (3) kemudahan dalam penggunaan. Detektor yang ideal untuk suatu penggunaan belum tentu ideal pada penggunaan lainnya. Pada *review* ini juga dijelaskan tentang penggunaan kromatografi gas dengan detektor berbeda-beda pada analisis pangan. Detektor yang akan dibahas adalah *Thermal Conductivity Detector (TCD), Flame Ionization Detector (FID), Electron Capture Detector (ECD), and Mass Spectrometer (MS).*

Kata kunci: perbandingan detektor, detektor, kromatografi gas, detektor kromatografi gas, analisis pangan

Introduction

Gas Chromatography (GC) is an instrument used for analyzing gaseous compounds or compounds that can be vaporized based upon differences of boiling point/vapor pressure and polarity (Jwaili, 2019). In GC, the mobile phase is an inert gas (i.e. nitrogen, helium, hydrogen, argon). Inert gas is a type of gas that is unreactive. The stationary phase is a layer of immobilized liquid coated on solid supports or solid adsorbents inside a column (Baiulescu & Ilie, 2013; Li & Liu 2018; Buse, *et al.*, 2019). GC is divided into two types, based on its stationary phase. GC with liquid stationary phase called gas-liquid chromatography (GLC), while GC with solid stationary phase called gas-solid chromatography (GSC).

Separation using two different phases (partition chromatography) invented by Martin and Synge in 1941. Martin and Synge used two different liquid phases to separate amino acids. GC was developed in 1952. That year, James and Martin developed and demonstrated separation using gas-

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liquid phases to separate volatile fatty acids. At that time, this method was called “vapor chromatography”, but known today as GC (Gordon, 1990).

GC consists of several components. Components in GC are: (1) carrier gas flow controller, (2) sample injection port, (3) separation column, (4) column oven, (5) detectors, and (6) integrator chart recorder (Aniszweski, 2007). Its principle is introducing the sample by an injection port. A carrier gas - which is the mobile phase, passes through the injector and brings the sample onto the column. Separation takes place in the column, which leads to a detector. Detector provides an electronic signal that is recorded and shown in a chromatogram (Li & Liu, 2019).

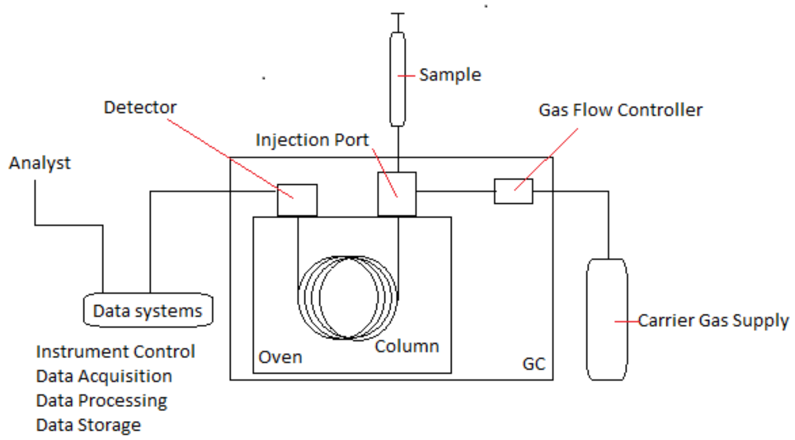

Figure 1. Schematic of gas chromatography system (Modified from Obeidat, 2021)

There are several detectors coupled with GC, such as Thermal Conductivity Detector (TCD), Flame Ionization Detector (FID), Electron Capture Detector (ECD), and Mass Spectrometer (MS) that are discussed here. A detector has various properties that are interrelated from the other. By comparing each detector, it is possible to consider the ‘ideal’ detector for analysis. However, the ideal detector for one application is not necessarily ideal for another application.

It is proposed to consider the ideal GC detector for analysis by their properties under three main headings: (1) types of detector, (2) sensitivity, and (3) ease of operation. It is also proposed to inform application of GC coupled with the detector in food analysis.

Detectors

In GC, the detector is located at the end of the column. Detector provides an electronic signal by detecting an analyte carried by carrier gas, which provides a measurement of the analyte. Any component different from the carrier gas can be detected by the detector (Ševčík, 1976).

Detector primarily consists of two parts, sensor and signal conditioning electronics. The sensor should be as close as possible to the column to optimize detection. Signal conditioning electronics is an equipment used to digitize the signal and the signal will be shown in a chromatogram (Scott, 1996).

An ideal detector requires high sensitivity and low detection limit in order to provide high resolution signal for all analytes. An ideal detector also needs good stability and reproducibility. Another important characteristic is that the detector should be easy to operate and reliable (Ševčík, 1976; Scott, 1996). Each detector has different properties, it is not possible for a detector to meet all of an ideal requirement. In order to choose an ideal detector for an analysis, the subsection below will discuss some properties of common GC detectors.

Type of Detectors

There are five ways to classify detectors: (1) universal vs. selective, (2) concentration-sensitive vs. mass-sensitive, (3) destructive vs. non-destructive, (4) bulk property vs. solute property, (5) analog vs. digital. Most important classification is the first three.

a. Universal vs. selective

Detectors classified into two general categories, universal and selective (specific). Universal detectors are able to detect most compounds. TCD is an example of a universal detector. Selective detectors detect specific compounds based on their properties. It has specificity to detect specific atoms or functional groups, such as ECD that is selective to electronegative compounds (McNair & Miller, 2011; Poole, 2012). Universal detectors are recommended for analysis. Choosing a desirable detector depends on the analyte. With an universal detector, all components in a sample can be quantified. It is needed for non-targeted analysis with unknown compounds, it can be used to know all components in a sample. However, using selective detectors is recommended to analyze specific targeted compounds.

b. Concentration-sensitive vs. mass-sensitive

Detectors are also classified by their responses. A concentration-sensitive detector responds to the concentration of analyte carried by carrier gas passing through it. Most common concentration-sensitive detectors are TCD and ECD. Concentration-sensitive detectors peak changes with the change of flow rate. Peaks increase as the flow rate decreases. Some detectors respond to the mass of analyte or ions passing through the detector. It is called mass-sensitive detectors. Because it detects mass, its peak doesn't change with flow rate. Example of a mass-sensitive detector is FID (Ševčík, 1976; McNair & Miller, 2011; Poole, 2012).

c. Destructive vs. non-destructive

Another classification of detectors are destructive and non-destructive. Destructive detectors consume analytes by chemical reaction (i.e. flame) while in non-destructive detectors, the analyte passes through the detector without chemical change (Andersson, 2014). Non-destructive detectors are highly recommended, because the analyte can be recovered for further analysis. FID uses flame so it is categorized as a destructive detector. TCD is a non-destructive detector, it detects thermal conductivity changes (McNair & Miller, 2011; Poole, 2012; Kellogg, 2017).

d. Bulk property vs. solute property

Bulk property detector and solute property detector are less common classification of GC. Bulk property detector detects bulk physical properties of analyte, such as refractive index or dielectric constant. It measures the physical properties difference of solute in carrier gas compared to the carrier gas only. Bulk property detectors have low sensitivity, and depend

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on ambient condition, temperature, and pressure. Most common bulk property detectors are TCD and ECD. Solute property detectors detect unique physical or chemical properties, such as fluorescence (Scott, 1996; McNair & Miller, 2011).

e. Analog vs. digital

This classification is based on detector sensors. Digital sensors only work at either 0% or 100% while analog sensors detect any value between 0% and 100%. By this description, measurement using a detector with an analog sensor is more precise than a detector with a digital sensor. Usually, common GC detectors use analog sensors (McNair & Miller, 2011).

There are many detectors with different classification. Most common detectors coupled with GC and their properties are described below.

Thermal Conductivity Detector (TCD)

TCD was one of the earliest detectors developed for GC. It is also known as a katharometer or hot wire detector. The basic principle of TCD is detecting the thermal conductivity properties between carrier gas and sample by monitoring the changes in electric conductivity of the filament (Ševčík, 1976; Budiman, *et al.*, 2015). TCD is a universal detector that gives response to all analytes that have different thermal conductivity and heat capacity from the carrier gas (Poole, 2012; Yin, *et al.*, 2016; Kellogg, 2017). TCD response to thermal conductivity, so it is very sensitive to flow rate (Frink & Armstrong, 2016).

In analysis, usually two TCD were used. One is used as reference to monitor the thermal conductivity of carrier gas, the other used to monitor the thermal conductivity of analyte (Scott, 1996). TCD has low sensitivity. Higher sensitivity of TCD can be obtained by using the right carrier gas. Helium is an ideal carrier gas for TCD, because thermal conductivity difference between helium and most compounds is greater than other carrier gas (Poole, 2012). Due to low sensitivity of traditional TCD, the volume of TCD is reduced and demonstrated as µTCD (Zao, *et al.*, 2020).

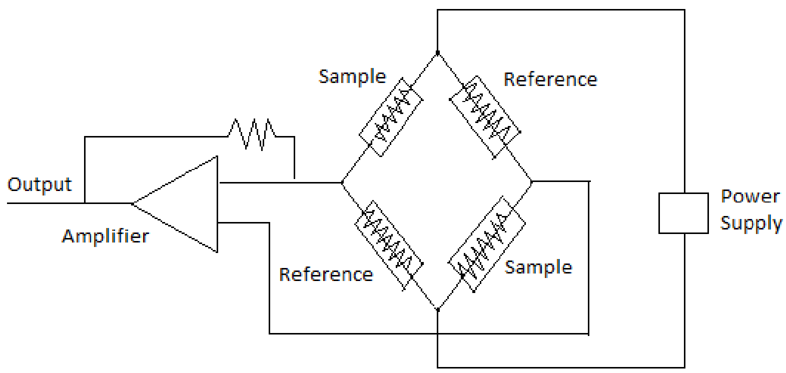

Figure 2. Schematic of thermal conductivity detector (Modified from Mcminn, et al., 2000).

TCD consists of a power supply, detector block, sample + carrier gas (from column), and pure carrier gas (reference gas). TCD uses CAT (Constant Average Temperature) mode in supply power that can extend TCD linear range several times, increase, and improve its sensitivity and response speed. Detector block consists of four inline filaments (two for the sample side and two for reference) that are installed in its own thermostatically controlled and those filaments can be heated electrically so it can be hotter (Wei, 2016; Coning & Swinley, 2019). From column also known as column effluent consists of sample and carrier gas (helium or hydrogen). Reference gas used to monitor the change of TCD signal by motion of the ship (Yamagishi, *et al.*, 2012).

Flame Ionization Detector (FID)

FID is a universal detector that is the most common GC detector for detecting organic compounds (i.e. hydrocarbon), due to high sensitivity of carbon-containing compounds. FID responds to all organic compounds that are combused in oxy hydrogen flame. FID also has low cross sensitivity to inorganic compounds (Poole, 2012; Budiman, *et al.*, 2015; Lenz, *et al.*, 2016; Bai, *et al.*, 2018; Ponphaiboon, *et al.*, 2018). Although FID is easy to use and unaffected by flow rate, FID requires three separate gas supplies and destroys the sample (destructive). FID requires hydrogen for combustion, helium or nitrogen for carrier gas, and oxygen or air for the combustion agent (Scott, 1996; Buse, *et al.*, 2019).

The principle of FID is ionization of organic compounds by combustion of the compound in hydrogen air flame (Budiman, *et al.*, 2015). The gas sample subsequently in hydrogen flame and the hydrocarbon that are from the sample are being ionized by the flame and then extracted by an electrical field. The result from this process is the electrical current that is generated, equal to the total carbon content of the sample (Hill & McMill 1992).

FID divided into seven parts like, column, flame tip (anode), flame, collector (cathode), power supply, tower body, and igniter (Poy, 1997). The samples from GC column were premixed with hydrogen make-up gas and will be heated with air or oxygen in flame tip. Burning process in the flame tip will decompose the organic sample. Flame is the main part in FID and the ionization mechanism of organic compounds happened here. The result of the ionization mechanism will be collected by a collector (cathode). Collector cathode consists of a positively charged ring. FID using electronic power supply to supply a stable polarizing voltage. Tower body is the outer part of FID and its functionality is for protection of the inner part (Gill & Hartmann, 1967; El-Naggar, 2006).

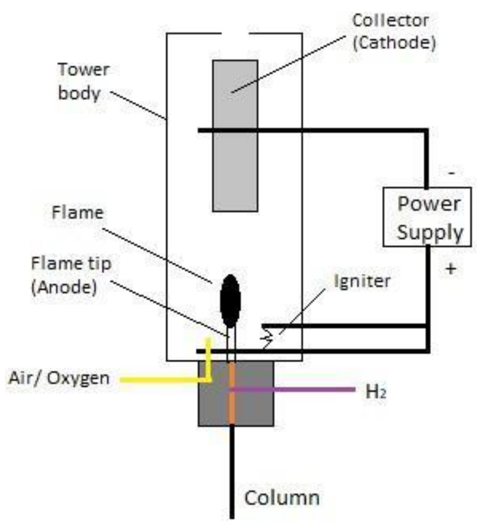

Figure 3. Schematic of flame ionization detector (Modified from Gebruers, et al., 2009).

Electron Capture Detector (ECD)

ECD, also known as electron affinity detector or the electron absorption detector is a concentration, sensitive and specific detector. ECD is used to detect electron affine compounds. ECD has high sensitivity to electronegative compounds (i.e. halogens) but also has a low detection limit. To produce electrons and ions, a low energy β-ray source is used (Lovelock & Lipsky, 1960; Scott, 1996; Schedl, *et al.*, 2018).

Using different carrier gas may affect various properties of electrons, such as thermalization rate. Carrier gas may also affect ionization of molecules complicating ECD signal. ECD usually uses a mixture of methane and argon gas (Scott, 1996). ECD consists of an ionization chamber containing a radioactive source and two polarized electrodes. ECD utilizes a radioactive emitter for ionizing particles. ECD detects the concentration of an analyte based on electrons adsorbed (Dressler, 1986).

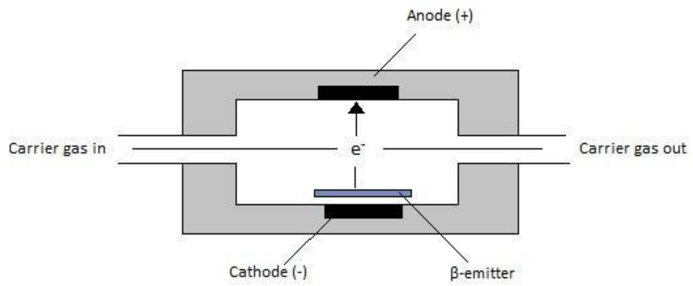

Figure 4. Schematic of Electron Capture Detector (Modified from Harvey, 2019).

Mass Spectrometer (MS)

MS is one of the GC detectors that is frequently used to detect organic and inorganic compounds due to high sensitivity, selectivity, possibility to identify compounds by molecular mass, and this detector provides straightforward analysis (Milman, 2011; Jumhawan, *et al.*, 2015; Lim, *et al.*, 2018). MS can offer detailed structural information in a component by separating the component (Skooog, *et al.*, 2017).

This detector consists of six parts, such as inlet system, ion source, mass analyzer, ion detector, vacuum system, and recorder (Rubakhin & Sweedler, 2010). Inlet system is a system where the sample was injected and the sample will be continued to stream or carrier gas (Baiulescu & Ilie, 2013; Li & Liu 2018). Ion source is a device of MS that can allow ionization. There are many types of ion source in MS analysis, such as chemical ionization, thermal ionization and field ionization (Sparkman, 2000). In a mass analyzer, the ions are separated by their mass or charge ratio and after the ion being separated, the ion will be detected by an ion detector and the signal will be transferred to a data system. Vacuum systems are being used to maintain the low pressure. Vacuum system will minimize, scatter, and neutralize the ion molecule reaction. After that, the recorder will record their relative abundance of each ion by making a peak (Wheeler, *et al.*, 1997).

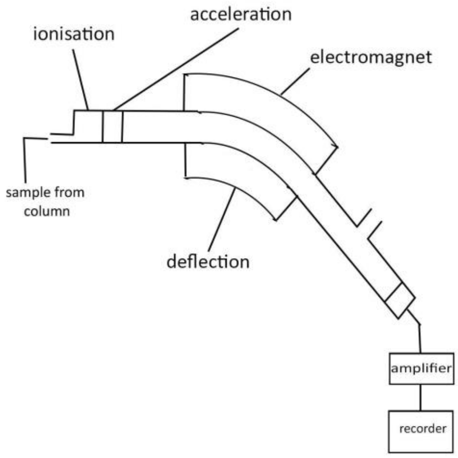

Figure 5. Schematic of mass spectrometer (Modified from Radauscher, 2015).

Its principle is to produce ions from analytes. Ions separated according to their mass-to-charge ratio. Selected ions are detected in the detector. Detector will convert to an electronic signal and it is recorded (Wheeler, *et al.*, 1997).

Sensitivity (Minimum Detectable Concentration)

Sensitivity is one of the most important properties of a detector. Each detector has a minimum detectable signal, which represents the minimum gas concentration that could be detected. Minimum detectable gas concentration expressed as detector sensitivity. Sensitivity of a detector expressed in many units, such as ppm (equal to µg/mL and ng/µL) or ppb (equal to ng/mL and pg/µL) (Scott, 1996).

Each detector has different sensitivity and it is related to the carrier gas that is used. For example, TCD is one of the less sensitive detectors, but helium can be used as carrier gas to make it more sensitive (Poole, 2012). Different analytes may also affect detector sensitivity. It means, detector sensitivity is not constant. As already described, a lower detection limit indicates higher sensitivity. It is better to use a detector that has higher sensitivity. Sensitivity of the detector that is described is shown in the table down below.

Table 1. Detection limit of GC detectors, according to Scott (1996); McNair & Miller (2011).

Detector	Lowest detection limit (g/mL)	Highest detection limit (g/mL)
TCD	10^{-6}	10^{-3}
FID	10^{-12}	$5x10^{-4}$
ECD	10^{-13}	10^{-8}
MS	10^{-12}	10^{-7}

TCD is the less insensitive detector compared to FID, ECD, and MS. However, TCD is commonly used in analysis, because it is a universal detector and non-destructive. According to Budiman, *et al.* (2015), sensitivity of TCD is low compared to FID in detecting propane. But, for detecting compounds that FID couldn't detect, TCD is still a good alternative. Although ECD has high sensitivity, the detection is selective to high electronegative compounds (Scott, 1996). It means

ECD couldn't detect most compounds that universal detectors could. In order to analyze non-targeted analytes, ECD is not a good choice.

Ease of Operation

Ease of operation also has a significant role beside sensitivity. An ideal detector should be easy to operate, high sensitivity, and selectivity. Every detector has different characteristics and factors that affect its characteristics are described below.

Thermal Conductivity Detector (TCD)

TCD is one of the oldest detectors that are still common to use. It is a universal detector which can detect almost all compounds (Poole, 2012). However, many detectors are better than TCD. TCD detecting thermal conductivity differences. There are several things that can affect sensitivity and sensor performance. TCD is sensitive to temperature fluctuation, heat loss, and flow rate. Thermal noise can reduce sensitivity and stability (McWilliam, 2007; Frink & Armstrong, 2016, Lotfi, *et al.*, 2019).

Flame Ionization Detector (FID)

FID is a simple detector and can be used to analyze organic compounds. FID are unaffected by gas flow rate. However, FID uses 3 different gasses: carrier gas, oxygen/air, and hydrogen and is affected by temperature. FID responses increase as hydrogen flow rate increases (Scott, 1996; El-Naggar, 2005; Scortichini, *et al.*, 2020).

Electron Capture Detector (ECD)

ECD detects electronegative compounds. ECD is a complex detector. It is sensitive to detector temperature and impurities. Impurities in air or impurities by electrode contamination will affect detector sensitivity and reliability. electrode contamination, which affects detector sensitivity and reliability. Oxygen and water molecules reduce ECD sensitivity (Dressler, 1986; Scott, 1996).

Mass Spectrometry (MS)

MS is a flexible detector due to its ability to detect organic and inorganic compounds. MS also provides structural information so it can simplify the users to understand the information (Milman, 2011; Jasi m, *et al.*, 2015; Jumhawan, *et al.*, 2015; Lim, *et al.*, 2018).

Application in Food Analysis

Food analysis using GC usually for analyzing volatile or vaporized compounds. Each detector used for different analysis, due to their selectivity with some compounds. Universal detectors are more applicable to many analysis than specific detectors.

Thermal Conductivity Detector (TCD)

TCD is a universal detector that can detect both organics and inorganics. TCD is a good detector for unknown samples, for initial investigation (McNair & McMiller, 2011). Because TCD has low sensitivity, usually in food analysis GC is coupled with another detector. GC-TCD is suitable for water analysis. In food analysis, TCD coupled with headspace gas chromatography (HSGC) is used to determine water in honey and compared to barrier discharge ionization detector (BID). TCD can be used to analyze, although BID can trace better (Frink & Armstrong, 2016).

Flame Ionization Detector (FID)

FID is considered as a universal detector. However, FID has high sensitivity with carbon-containing compounds and less sensitive with compounds without carbon. There are many food analyses using FID. For example, FID can be used to identify lauric acid in modified coconut oil and palm oil (Pruksatrakul, *et al.*, 2017; Ponphaiboon, *et al.*, 2018) and determine volatile compounds in virgin olive oil and wines (Aparcio-Ruiz, *et al.*, 2018; Barbará, *et al.*, 2020). FID can detect fatty acids in bovine colostrum (Yurchenko, *et al.*, 2016), GC-FID can also be used to analyze metabolics fingerprints in food. FID can be used to authenticate the Asian palm civet coffee (Jumhawan, *et al.*, 2015).

Electron Capture Detector (ECD)

ECD is a detector that can detect electron affine compounds due to high sensitivity and selectivity to high electronegative compounds (Scott, 1996). This detection used hydrogen as carrier gas and can be used to analyse the pesticide and residue in fruits and vegetables with high water content, also fish (Stocka, *et al.*, 2016; Farina, *et al.*, 2017; Yousefi, *et al.*, 2017; Lukyanova, *et al.*, 2018; Freitas, *et al.*, 2018). In food analysis, ECD could be used to detect volatile compounds in beer (da Silva, *et al.*, 2015), and analyze metabolites in sugarcane (Biswas, *et al.*, 2019).

Mass Spectrometry (MS)

MS is classified as a universal detector that can detect organic and inorganic compounds due to high sensitivity, selectivity, and capability to identify compounds by molecular mass (Milman, 2011; Jumhawan, *et al.*, 2015). MS can be used to analyze fatty acids (Pontoh, 2016), volatile compounds (Breme & Guggenbuhl, 2014; Irawan, *et al.*, 2018; Bueno, *et al.*, 2019), aldehydes (Ogihara, 2018), alcohol compound (Hasan, *et al.*, 2019). MS also can be used to detect the spoilage, contaminant, adulteration, and microplastic analysis in food industry and environmental (Nasiri, *et al.*, 2016; Wang, *et al.*, 2016; Georgiou & Danezis 2017; Bai, *et al.*, 2019; Matsui, *et al.*, 2020).

Combination

Sometimes a combination of instruments may be used in food analysis. For example, Raafat (2018) using combined GC-MS, GC-FID, and RP-HPLC for analyzing phytochemicals of *Juglans regia* oil and kernel. Using combined instruments is shown to be an efficient method. Biswas, *et al.*, (2019) use GC-ECD to analyze fipronil in sugarcane and use GC-MS/MS for confirmation.

Table 2. Example of food analysis using GC.

Type	Detection	Example	Sample	Sources
GC-TCD	Universal	Water	Honey	Frink & Armstrong, 2016
GC-FID	Hydrocarbon	Fatty acid	Modified coconut oil	Pruksatrakul, *et al.*, 2017
			Palm oil	Ponphaiboon, *et al.*, 2018
		Volatile compound	Wine (aroma)	Nicolli, *et al.*, 2018

GC-ECD	Electronegative compound	Volatile compound	Beer	da Silva, *et al.*, 2015
		Fipronil	Sugarcane	Biswas, *et al.*, 2019
GC-MS	Universal	Volatile compound	Red-berries yoghurt drink	Breme & Guggenbuhl, 2014
			Julang-jaling	Irawan, *et al*, 2018
			Raw meat	Bueno, *et al.*, 2019
		Fatty acid	Coconut oil	Pontoh, 2016

Conclusions

Most important thing about a detector is detector selectivity. In analysis, make sure that the detector used can detect analytes. However, sometimes there are several detectors that can detect the same compounds. In order to choose an ideal detector, find a detector that can detect compounds wanted, has high sensitivity and convenience.

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