CO2 LASER POWER OPTIMIZATION OF PHOTOACOUSTIC SPECTROMETER AND IT'S APPLICATION TO DETECT AMMONIA GAS (NH3) CONCENTRATION ON ISOTONIC BEVERAGE CONSUMER'S BREATH

by Bergitta Dwi Annawati

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ABSTRACT

Performance of photoacoustic spectrometer CO$_2$ laser can be optimized by variation of laser voltage with active laser medium gas compositions include gases He, N$_2$ and CO$_2$. Characterization of photoacoustic spectrometer CO$_2$ laser is done by determined resonance curve, quality factor, noise and lowest detection limit. Photoacoustic spectrometer applied to detect ammonia gas concentration in isotonic beverage. photoacoustic spectrometer was applied to detect the concentration of ammonia in the isotonic drinks and measuring result using multicomponent-analysis. The lowest detection limit for ammonia gas on line10R14 obtained at (70 ± 7) ppb with highest laser power obtained at (37 ± 0.1) W and the composition of the active medium gas He, N$_2$ dan CO$_2$ sebesar 30:40:30. The highest concentration of ammonia gas, 0 minute in range of (2.89 ± 0.05) ppm, 30 minutes in range of (4.22 ± 0.05), 60 minutes in range of (5.34 ± 0.05), 120 minutes in range of (5.49 ± 0.05) ppm, 150 minutes in range of (3.62 ± 0.05) ppm and 180 minutes in range of (3.28 ± 0.05) ppm. Changes concentration of ammonia gas result are influenced by the length of time after consuming isotonic beverage.

Keywords: *Photoacoustic Spectrometer, CO$_2$ Laser, Isotonic Beverage, Multi-component Analysis*

1. INTRODUCTION

Isotonic beverages is a drink that has the composition and osmotic pressure equal to the fluid (plasma) blood. Isotonic beverages besides containing water as a substitute for lost body fluids, it also contains minerals as a substitute for mineral lost in sweat and sugar as energy used during activity (Narwidina, 2009).

The use of glass electrodes can separate protein and electrolyte with partial pressure variation (Lang, et al., 1997). Ammonia is released in the metabolism process of amino acids and high toxicity. Ammonia is converted to urea in the liver in the excretion of urine with ammonium release. Ammonia compounds are produced in the mouth does not always enter the bloodstream but can appear on the breathing of exhaled (Curdy, et al., 2007). The author will examine the ammonia gas which is the result of gas blown from volunteers who had consumed a beverage containing isotonic using Photoacoustic Spectroscopy techniques. The highest sensitivity is achieved by using a laser photoacoustic gas system of mid-infrared lasers such as CO and CO$_2$ with high sensitivity that orde ppbv (part per billion volume) and even the order of sub-ppb (Dumitras, 2008).

2. BASIC THEORY

Theory of the photoacoustic gas

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Photoacoustic effect is the process of photoacoustic waves generated in a sample of results from the absorption of photons (West, et al., 1983). Molecule will absorb photon if the photon energy corresponding to the energy difference between level transitions - molecular energy levels.

Molecules that absorb photons will undergo excitation, namely the migration of molecules from the basic level (E_0) to a higher level (E_1), with energy difference ΔE between the two levels is $E_1 - E_0 = hv$, h is Plank constanta and v is absorbed photon frequency.

On CO_2 laser radiation source, the photoacoustic effect is determined by the decay of the most dominant in the fourth process is a process of decay that occurs almost all the non-radiative deexcitation intangibles. The wavelength in this area are owned by the infrared radiation generated by a CO_2 laser radiation.

Photoacoustic Signal Generation

Figure (2.1) describes the mechanism of photoacoustic signal generation in the cells due to radiation absorption modulated. Transfer of kinetic energy of a gas molecule by impacts after absorbing radiation with other molecules that cause an increase in heat energy is increasing the temperature of the gas
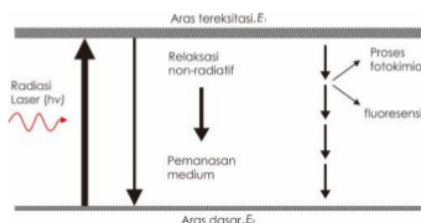

Figure 2.1 Some Type Process Occurred On Ground State Towards Molecular Basis After Radiation Absorbing Laser (hv)

Photoacoustic Signal Generation

Acoustic wave in the photoacoustic cell is detected using a microphone placed in the center of the cell in which the maximum amplitude acoustic wave. Photoacoustic signal proportional to the radiation, properties of the cell (cell constants and responsiveness microphone), absorption coefficient and concentration of the sample in the cell (Wasono, 2008). Pressure modulation at audio frequencies, called acoustic waves are then detected by the microphone (Rosencwaig, 1980).

If the photon radiation is modulated at audio frequencies, there will be gas temperature modulation with a frequency equal to the modulation frequency. Pressure modulation at audio frequencies is called acoustic waves are then detected by the microphone (Rosencwaig, 1980).

Transfer of kinetic energy of a gas molecule impacts after absorbing radiation with other molecules that cause an increase in heat energy that is an increase in the gas temperature.

Presupposed a beam of radiation with a radiation power I_0 through a homogeneous gas density in a cell cylindrical symmetry. Power I_0 is transmitted through the cell is given by Lambert- Beer's law :

$$I_{trans} = I_0 e^{-\beta l}$$

(1)

with β (cm^{-1}) dan l (cm) in sequence states the sample absorption coefficient per unit length and the length of the sample. From equation (1) can be lowered much power is absorbed by the sample:

$$I_{abs} = I_0 - I_{trans} = I_0\left[1 - e^{-\beta l}\right]$$

(2)

The low absorption $\beta l \ll 1$, with take the first-order equation and ignores the contribution of higher order of Fourier expansion to equation (2), equation amplitude proportional to the pressure and the amount of radiation absorption occurs in the gas molecules (Sauren, 1992):

$$I_{abs} \approx I_0 \beta l$$

(3)

Absorption coefficient per unit length for a specific absorption coefficient β can be expressed in α absorption per unit pressure α (cm^{-1}atm^{-1}) and the molecular absorption cross section σ (cm^2/mol)

$$\beta = \alpha p_a = \sigma N_a = \sigma C N_0$$

(4)

with p_a partial pressure of absorbing molecules in the sample (atm^{-1}), N_a number of molecules per unit volume absorber cm^{-3}), fractional constants C (dimensionless) and N_0 total number of molecules per unit volume (cm^{-3}), by substituting equation (3) into equation (4) is obtained:

$$I_{abs} = I_0 \sigma C N_0 l = I_0 \alpha p_a l$$

(5)

If large electrical photoacoustic signal is proportional to the microphone responsivity R (mV/Pascal) and the amount of power absorbed by the gas (Quist, 1991)

$$S \propto I_{abs} R$$

(6)

Then substitution between equations (5) to equation (6) and gives the pro- portionality factor is represented by the cell constant F, FA signal can be expressed as :

$$S = FRI_0 \alpha C$$

(7)

with F does not depend on I_0 and α, but only depends on the geometry of the cell. Can be defined that at the resonant frequency $(\omega = \omega_0)$, F is a constant equal to the ratio between the pressure response of the cell (Pascal = N/m^2) and laser power (Watts) and the absorption coefficient (cm^{-1}),.

The structure of the CO$_2$ molecular vibration energy levels

CO_2 laser is a type of molecular gas laser with CO2 gas as the laser active medium. CO_2 laser radiation emission occurs due to the displacement of cedar vibrational-rotational CO_2 molecules with a wavelength of 9 μm to 11 μm. The composition of the CO_2 molecule is composed of two covalent bonds because the electron pair of the C and O atoms shared by two atoms.

Gas Measurement blown Using Photoacoustic Spectroscopy With Multicomponent Method

The most important characteristics of the photoacoustic system that can measure in a multicomponent gas, high sensitivity for measurement of low concentration (*ppb*), a high selectivity for the detection of a specific gas contained in a multicomponent gas mixture and can be performed *in-situ* (Gondal, et al., 2004). In a multicomponent gas measurement, the photoacoustic signal measured at each set of wavelengths are selected based on the absorption spectrum of the individual components.

CO_2 laser is a laser source that can measure gas mixtures include ethylene, acetone and ammonia. Before measuring the absorbance at the sample, the instrument is calibrated against three types of gases, namely ethylene, acetone and ammonia. The results of the calibration matrix that is the relationship between the concentration of gas that sought to normalize the signal in each laser line that is chosen is to ethylene 10P14, 10P20 to acetone and 10R14 for ammonia which is shown as follows:

$$\begin{bmatrix} \left(\frac{S}{P}\right)_1 \\ \left(\frac{S}{P}\right)_2 \\ \left(\frac{S}{P}\right)_3 \end{bmatrix} = \begin{pmatrix} K_{11} & K_{12} & K_{13} \\ K_{21} & K_{22} & K_{23} \\ K_{31} & K_{32} & K_{33} \end{pmatrix} \begin{pmatrix} C_1 \\ C_2 \\ C_3 \end{pmatrix}. \qquad (8)$$

with $(S/P)_1$, $(S/P)_2$, dan $(S/P)_3$ each is normalized photoacoustic signal on line 10P14, 10P20 and 10R14 for ethylene gas (C_1), acetone gas (C_2), and ammonia gas (C_3). While K_{ij} the calibration factor between the uptake of laser *i* lines for gas concentration *j* (Mitrayana, *et al.*, 2014).

Human breath gas is a gas blow heterogeneous. In healthy people, the first part of a breath, approximately 150 ml in the form of gas blown from the upper respiratory tract (mouth), a bellows gas is not in contact with the lung alveoli. The next section, approximately 350 ml gaseous blowing coming from the lungs, where gas exchange between the blood and the air breathing occurs. This gas is used as a marker gas (Dumitras, 2012).

Tilden states that if each gas in a mixture absorbs the CO_2 laser the same line, the classical approach can be used to determine the concentration of the components of a mixture by solving simultaneous equations (Murniati, 2001). Multicomponent equation is used :

$$\left(S/P\right)_1 = k_{11}C_1 + k_{12}C_2 + k_{13}C_3$$
$$\left(S/P\right)_2 = k_{21}C_1 + k_{22}C_2 + k_{23}C_3 , \qquad (9)$$
$$\left(S/P\right)_3 = k_{31}C_1 + k_{32}C_2 + k_{33}C_3$$

with k_{ij} can get by any gas gradient that is used in some variation of laser absorption lines. Then to get the calibration to convert the normalized signal to the gas concentration, do the inverse of the values that are already known, then obtained:

$$\begin{bmatrix} C_1 \\ C_2 \\ C_3 \end{bmatrix} = \begin{bmatrix} k_{11} & k_{12} & k_{13} \\ k_{21} & k_{22} & k_{23} \\ k_{31} & k_{32} & k_{33} \end{bmatrix}^{-1} \begin{bmatrix} \left(S/P\right)_1 \\ \left(S/P\right)_2 \\ \left(S/P\right)_3 \end{bmatrix} \qquad (10)$$

Amonia Gas (NH_3)

Ammonia dissolves readily in water to form ammonium hydroxide (Lewis, 1993). Ammonium hydroxide produces heat when interacting with moist surfaces such as mucous membranes. The corrosive nature of ammonia may cause damage to the mucous membranes of the oral cavity and respiratory tract (Willems, 2001).

Contained in the human plasma and ammonium ions (Shakhashiri, 2008). Based on the WHO in 2003, beverages and foods containing ammonium compound around 0,001 - 3,2%.

3. METHODS

Material Research

The research material used is CO_2, N_2 and He as CO_2 laser active medium. Scrubber KOH and CaCl2 to absorb water vapor and CO_2 in the gas blow is detected. Sample gas blown human breath of volunteers who consumed a drink containing isotonic or electrolyte.

Experiment configuration

Bellows breathing gases expelled through the mouth with a gas-making scheme shown in figure (3.1). As shown in the figure the whole gas sample to be examined flowed into photoacoustic cell through *scrubber* KOH and CaCl$_2$. This is done in order to absorb water vapor and CO_2 which can interfere with the performance of gas absorption biomarker in CO_2 lasers. Recorded data then processed off-line. By calculating and making the calibration of the matrix inversion signal results gases, the concentration of the multicomponent gas standards respiratory outcomes can be determined.

Measurement of the concentration of ammonia gas in the gas blowing humans, is necessary to calibrate the instrument. Calibration carried out on three standard gas is ethylene, acetone and ammonia. The results of this calibration will be obtained matrix relationship between the concentration of gas that sought to signal to each laser line is selected, that is 10P14 for ethylene, 10P20 for acetone, and 10R14 for ammonia.

Analysis of data from the results of experiments performed using software Origin Pro to obtain graph calibration for each pure gas ethylene, acetone and ammonia in the entire absorption line laser. The graph of the results to be obtained calibration data of proportionality between the normalization signal on a line to the pure gas concentrations tested for each gas ethylene, acetone and ammonia. After obtained matrix with using MS Excel software will be obtained inverse matrix.
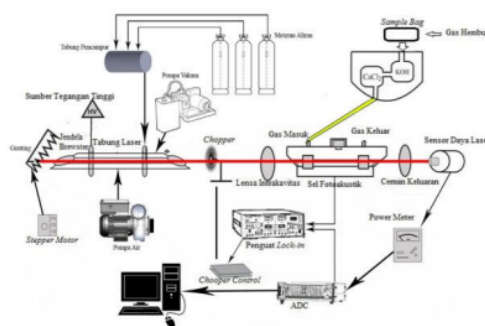
Figure 3.1 Schematic experimental setup in the study using a CO$_2$ laser photoacoustic spectrometer intrakavitas

After that ethylene gas uptake from sample on 10P14 will be scanned within 10 minutes followed aseton gas on 10P20 and ammonia gas on 10R14. Absorbed accoustic signal from ethylene gas, acetone gas, ammonia gas will be seen on *LabVIEW Software* along with power laser radiation which is then normalized signal obtained. The normalized signals will be processed to find the value of the concentration of ammonia gas footage volunteers with different timescales.

Research Tools

The equipment used can be grouped into two parts:

1) Photoaccoustic spectrometer consists of several components:
   a) Laser power supply high voltage DC current (HV) HCN 350-20.000 model with a maximum voltage of 20 kV and a maximum current of 15 mA.
   b) Vacuum pump.
   c) Water pump serves for cooling the laser tube.
   d) *Grating* and *stepper motor* for choose CO$_2$ laser line.
   e) *Chopper* along with the control to modulate CO$_2$ laser beam.
   f) CO$_2$ laser tube flowing system as the place of discharge for medium active laser.
   g) Photoaccoustic cell H type generates a photoaccoustic signal.
   h) *Outcoupling mirror* as a laser beam output.
   i) Power meter to detect CO$_2$ laser power output.
   j) Thermometer to determine temperature around the laser tube.
   k) Lock-in brace EG&G model 5210 to record and amplify signals as well as functioning as signal filter.
   l) ADC to convert analog signal to digital.
   m) One unit PC Pentium IV 2.4 GHz 1 GHz memory using LabVIEW 8.6 software to computerize SFA system.
2) Sampling system and gas flow
   a) Regulator gas holder CO$_2$, N$_2$, He.
   b) Mass flow meter Brooks 5860 model along with microprocessor control and reader unit Brooks 0152 model.

c) *Scrubber* used is KOH and $CaCl_2$ whose function is to absorb water vapor and CO_2 in the gas blow on breath sample

Work Procedure and Data Collection

The study is divided into two parts, namely the photoacoustic spectrometer characterization and application of CO_2 laser photoaccoustic spectrometer to detect the concentration of ammonia gas in the gas blow volunteers who consume isotonic beverages using KOH scrubber and CaCl2.

Performance Characterization of Photoacoustic Spectrometers

Characterization of CO2 laser photoacoustic spectrometer performance as follows:

a. Streamlining optics and CO_2 laser power optimization

b. Make factor resonance curve and quality factor (*Q*)

$$Q = \frac{f_{res}}{\Delta f}$$

c. Measurement of noise and background signal.

d. Determine lowest detection limit

$$BDT = \frac{C}{S_n/N}$$

e. Making the curve linearity signal line laser absorption of CO_2 to the variation in gas concentration of acetone, ethylene, and ammonia standard using multicomponent method.

Measurement of Concentration Levels Snapshot Ammonia Gas In Gas blow consuming Isotonic Beverages

Measurement of ammonia concentration of the gas blown done in a way that is :

1. Sampling gas blow on 15 volunteers aged 20-35 years conducted by blowing into a sample bag through the scrubber KOH and $CaCl_2$, due to reducing carbon dioxide and water vapor contained in the respiratory.

2. Sample gas blow stored in an vacuum bag sample. Snapshot blowing gas is passed into the photoaccoustic cell flow through the pump.

After that, do scan signal of gas amonia uptake from sample on 10P14, 10P20 dan 10R14 line within 5 minutes. Acoustic signals of ammonia absorbed gas will be seen on LabVIEW Software along with power laser radiation which is then normalized signal obtained. The normalized signals will be processed to find the value of footage ethylene gas concentrations, acetone and ammonia

4. RESULT AND DISCUSSION

CO_2 Laser Power Optimization

In this study, a large operating voltage and current of the laser used and the current 8,15-9,40 kV range of 11,45 to 15,80 mA, because the value of laser discharge occurs in the discharge tube. Furthermore, the composition ratio of the active laser medium gas consisting of He, N_2 and CO_2. Rated the best comparison would

produce optimum laser power. Gas composition ratio of the active laser medium is He: N_2: CO_2 by 30: 40: 30. Comparison of the three active medium gas pressure affect CO_2 laser output power (Jelvani, 2012).

Scanning CO_2 Laser Line

In this research, the CO_2 laser line spectrum consisting of three groups in sequence from left to right 9P, 10R, 10P with laser power intrakavitas maximum of (37 ± 0.1) W.

Figure (4.1) explains that absorbs ammonia gas 10R14 strong in line with the normalized signal at 2.5 mVolt / W with intrakavitas laser power of 20 watts. Ammonia gas absorbs strongly in line 10R14 and 10P32. Uptake was strongest in 10R14 has a length of 10.29 μm (Konjevic, 1979).
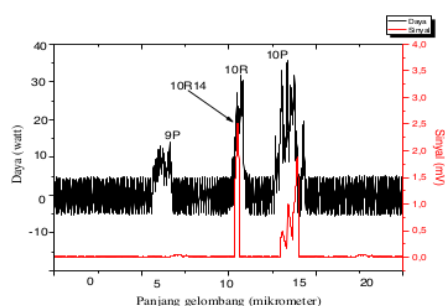
Figure 4.1. Ammonia Absorption Lines

Figure (4.2) is a graph of the CO_2 laser power stability on the line 10R14 what showed no drop in power, which means that the power used is stable. CO_2 laser power stability in line 10R14 obtained at (25 ± 2) watts.
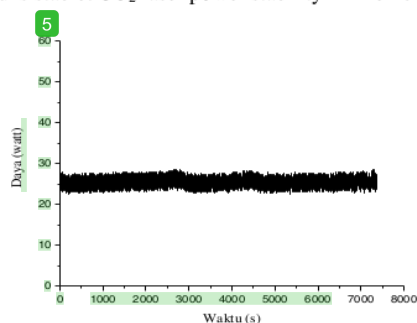

Figure 4.2. CO₂ laser power stability on the line 10R14

Resonance Curve and Quality Factor Q

Figure (4.4) shows the resonance curve photoacoustic cell for standard ammonia gas at 10R14 laser line with the value of the resonant frequency f_{es} (1650 ± 5) Hz with a quality factor Q of (14.3 ± 0.6).
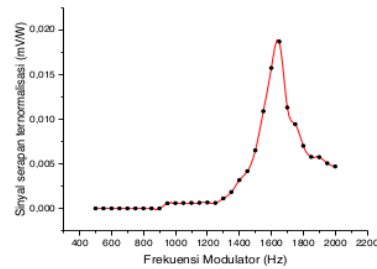
Figure 4.4 Resonance Curve laser lines for ammonia gas

Noise Measurement and Detection Limit Low (BDT)

Figure (4.5) shows a graph of signal *noise* and noise values obtained by $(1.9 \pm 0.4) \times 10^{-3}$ mV/$\sqrt{Hz}$.
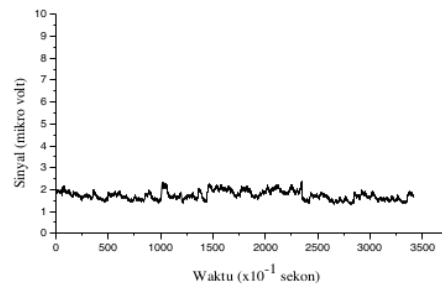

Figure 4.5. Signal noise photoacoustic cell

Kepekaan spektrometer fotoakustik Laser CO_2 diperoleh dengan mencari batas deteksi terendah (BDT). Pada penelitian ini diperoleh BDT pada gas amonia standar sebesar (70 ± 7) *ppb*.

Kalibrasi dan Linearitas

Based on the results of linearity on the lines of laser 10R14, 10P14 and 10P20, can be written in the form of a multicomponent matrix equation as follows:

$$\begin{bmatrix} C_1 \\ C_2 \\ C_3 \end{bmatrix} = \begin{bmatrix} 36{,}27871 & -2{,}34198 & -0{,}74398 \\ -26{,}97680 & 68{,}48097 & -34{,}6367 \\ -0{,}241022 & -1{,}83573 & 46{,}40933 \end{bmatrix}^{-1} \begin{bmatrix} \left(S/P\right)_1 \\ \left(S/P\right)_2 \\ \left(S/P\right)_3 \end{bmatrix}$$

Ammonia Gas Concentration Measurement Results

Figure 4.9 is the result of measurement of ammonia gas concentration of each volunteer using multicomponent methods with time variations. Variations in the time used before the interval of consumption, 0, 30, 60, 180 minutes.
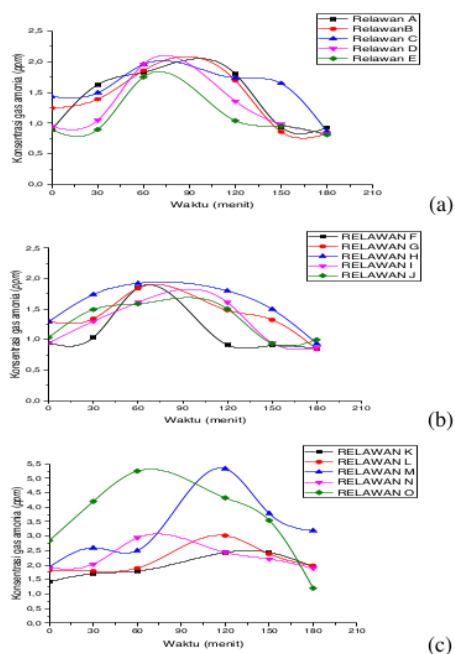
Figure 4.9 Graph ammonia gas concentration (a) Volunteers A-E, (b) Volunteers F-J, (c) Volunteers K-O

Figure 4.9 shows the concentration values of each different sample. Based on time variations, an average value of ammonia gas concentration was produced for 0 minutes (1.34 ± 0.05) ppm, 30 minutes for (1.67 ± 0.05) ppm, 60 minutes for (2.22 ± 0) , 05) ppm, 120 minutes for (2.21 ± 0.05) ppm, 150 minutes for (1.37 ± 0.05) ppm, and 180 minutes for (1.43 ± 0.05) ppm. In isotonic solutions, there is an ammonia gas content with a limit of pK 8,923. Using glass electrodes, research has been conducted to calculate the content of ammonia gas in an isotonic solution of 150.15 M (Lang, 1997).

The difference can also be due to the higher age, the metabolism in the body decreases (Son'kin, 2012). Hibbard and Killard (2011) state that there is no significant difference between the concentration of gas in the human body and gender. The lowest ammonia gas concentration of each sample was when volunteered before consuming isotonic drinks. After the volunteers consume isotonic drinks the ammonia gas concentration changes. Increasing the concentration of ammonia gas has increased in 60 minutes to 150 minutes. Then experiencing a phase decrease in the concentration level of ammonia gas after 150 minutes.

5. CONCLUTION

1. Spektrometerfotoakustik has a lower limit of detection for ammonia gas (70 + 7) *ppb* in line 10R14. Intrakavitas power peak of (37 ± 0.1) W with a quality factor of (14.3 ± 0.6).

2. CO2 laser photoacoustic spectrometer with multicomponent analysis methods capable of measuring the concentration of ammonia gas in volunteers consuming isotonic beverage with the highest value for each of the following time 0 minutes of (2.89 ± 0.05) *ppm*, 30 minutes of (4.22 ± 0.05), 60 minutes of (5.34 ± 0.05) *ppm*, 120 minutes of (5.49 ± 0.05) *ppm*, 150 minutes of (3.62 ± 0.05) *ppm*, dan 180 menit sebesar (3.28 ± 0.05) *ppm*.

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