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# A rapid remote real-time in-situ nitrous oxide measurement system

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**Abstract** In the past half-century, the production of crops and livestock is strongly driven by the increased use of irrigation, agriculture machinery, fertilizer, and pesticide. A balanced amount of fertilizer is needed to increase food production and to meet food security requirements by allowing a stable amount of staple food production. However, the amount of fertilizer needs to be limited to prevent unnecessary greenhouse gas emissions such as $N_2O$. However, the measurement of $N_2O$ gas in agricultural settings is challenging, generally frequiring complex systems. The development and evaluation of a new nitrous oxide ($N_2O$) gas measurement system for agricultural fields is described. This system consists of an Infra- Red (IR) $N_2O$ gas sensors module placed within an acrylic chamber and located in a agricultural field. It is connected to an Internet of Things (IoT) module for recording gas level measurements in the cloud database, thus permitting monitoring of the measurements in real-time using a mobile phone. Firstly, measurements were taken using standard gas to evaluate the characteristic of the sensor module. Susequently, the measurement system was tested in two experimental fields for 4 days with 10 mg urea fertilizer applied in each case. The measurement results using standard gas showed that the IR sensor module produced adequate result compared with the measurement using Gas Chromatography (GC). *In situ* field measurement showed the changes over the 4 days for each. These results indicate that this developed system can be used to monitor $N_2O$ gas levels in agricultural fields.

**Keywords:** Food security, Gas emissions, Internet of things

## Introduction

The increase in the number of greenhouse gases raises concerns that rising temperatures on earth could cause climate change (Griffis *et al.,* 2017; Yue and Gao, 2018, Ting *et al.,* 2021, Moiceanu and Dinca, 2021). On the other hand, an increasing population worldwide, with associated growth in food consumption, places greater demands on agriculture. This has led to efforts to increase agricultural production through the extensive use of fertilizers, which

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can result in the production of high levels of nitrous oxide ($N_2O$) (Millar *et al.,* 2018; Skinner *et al.,* 2019). Several studies have tried to reduce the production of $N_2O$ gas in agricultural fields by using various methods (Harter *et al.,* 2016).

Gas concentration measurement normally involves one of two techniques; gas chromatography (GC) and Infrared (IR) (Hensen *et al.*, 2013). The principle of GC is based on the separation of components of the sample into its constituent. The measurement of $N_2O$ concentration is conducted by injecting $N_2O$ sample gas through a tube or loop into the carrier gas stream of a GC instrument, equipped with an electron capture detector (Wang *et al.,* 2010). To maintain accurate results from the measurements, regular calibration is needed. However, measurements with GC can not be carried out directly *in situ* but it can be conducted on agricultural land using an automated chamber with connection to GC. However, the system required is complex and expensive. The disadvantages of GC measurements are frequently reported they are time-consuming (Brummell and Siciliano, 2011), expensive equipment is required (Stauffer *et al.,* 2008) and errors can occur when taking gas samples (Tokura *et al.*, 2013). By contrast, the IR technique is based on the principle that each gas has a different absorbance characteristic to IR at a specific wavelength. One advantage of an IR sensor is that the sensor does not come in direct contact with the gas, which can be corrosive to the sensor, instead the gas molecule only interacts with light. The IR sensor normally comprises a gas cell/tube which has at one end, an IR light source and on the other side, a detector. The gas to be measured circulate in the tube and the distance that light can pass through the gas is directly proportional to the amount of radiation absorbed (Popa and Udrea, 2019). IR systems can be categorized into two types; open path and closed path systems. In a closed path system, the gas sample is injected into a measurement cell, where an IR beam is focused toward the sample. While, with the open path system, the IR beam is directed towards the sample in the outside environment. Previous researchers have used this open path approach to measure $N_2O$ (Schaefer *et al.,* 2012; Iqbal *et al.,* 2013). Compared with GC, the IR system has slightly better performance in terms of sensitivity (Hensen *et al.,* 2013). However, both techniques provide uncertain accuracy and precision due to overlap with other gases (Iqbal *et al.,* 2013).

Measurement of $N_2O$ on agricultural land is challenging. However, different methods have been proposed to measure gas flux, e.g. micro-meteorological chamber, chamber and mass balance (Hu *et al.,* 2014). The micro-meteorological method requires an expensive and complex measurement system. This method requires readings to be taken at a number of locations within a wide area and the results to be integrated. The measurement is influenced by the atmosphere (Hensen *et al.,* 2013). By contrast, measurement using a chamber does not determine how much $N_2O$ gas comes out of the entire field because it only measures a small plot of land. However, the main idea of the chamber is to have an enclosed measurement area, which is not influenced by the outside air (Kroon *et al.*, 2008). The method of employing a chamber is also less expensive. The mass balance method is used based on input and output from the measurement system by calculating all inputs and outputs such as fertilizer, feed, atmospheric influences and waste. The calculation of gas emissions is performed by deducing the material entering and leaving a system (Denmead, 2008).

The shortcoming of the static chamber system is the gas level measurement still uses GC measurement that are not continuous, not real-time, must be conducted in a laboratory and require a relatively long time to get the results (Jumadi *et al.*, 2019). Therefore, an improvement of the measurement system is needed to overcome these shortcomings. In this study, we have developed a new model measurement system for measuring $N_2O$ to minimize the risk due to greenhouse gases caused by fertilizing agricultural land. This $N_2O$ measurement system could be used for remote, real-time, monitoring, as well as aiding research in reducing greenhouse gasses using IoT technology. The system utilises IR gas sensors linked to the internet and cloud storage to record the result of the measurements. Collecting data in this way has the advantage of permitting easy analysis for further study and facilitating comparison of data from a range of sources across a region. The urgent need for this research was to improve the efficiency of measuring the gases in agricultural environment that cause greenhouses effects, and thus to increase awareness of the consequences of over-fertilizing.

## Materials and methods

### *General information of the experiment site*

The experiment was conducted at the agricultural gardens of Universitas Negeri Makassar, Makassar, South Sulawesi, Indonesia (-5.183192, 119.430099) in the period from December 2019 to January 2020. The site has a tropical climate with an average temperature is 30 $^{o}$C and average rainfall is 549 mm which is located in the tropical climate zone. The climate is classified as Af based on the Koppen-Geiger system. The soil at the experiment site belonged to the class of Typic Haplusters with pH 6.1 and a water content of 45%.

### *Measurement system*

To measure the $N_2O$ gas concentration, an acrylic chamber was made of size 55x55x55 cm and 5 mm thick. A steel ballast was placed in the bottom of the chamber as a holder and so that there was no air leakage. A small fan was placed on top of the chamber to mix the air in the chamber as shown in Figure 1. The measurement system consists of an $N_2O$ sensor module, Internet of Things (IoTs) module and mobile phone as shown in Figure 2. The $N_2O$ IR sensor (SAFEGAS, Shenzhen Yuante Technology Co, Ltd) has a range measurement of 0-100 ppm and a resolution of 0.01 ppm. The sensor device is equipped with a pump to inhale the air into the device and an outlet to release the gas after measurement. The IR sensor measures the $N_2O$ content in the air that is inhaled through this pipe. The IoT module was designed to receive data from the sensor by using the RS485 protocol and to send the result to the Internet. The IoT module consists of Nodemcu ESP8266 and a wifi module.

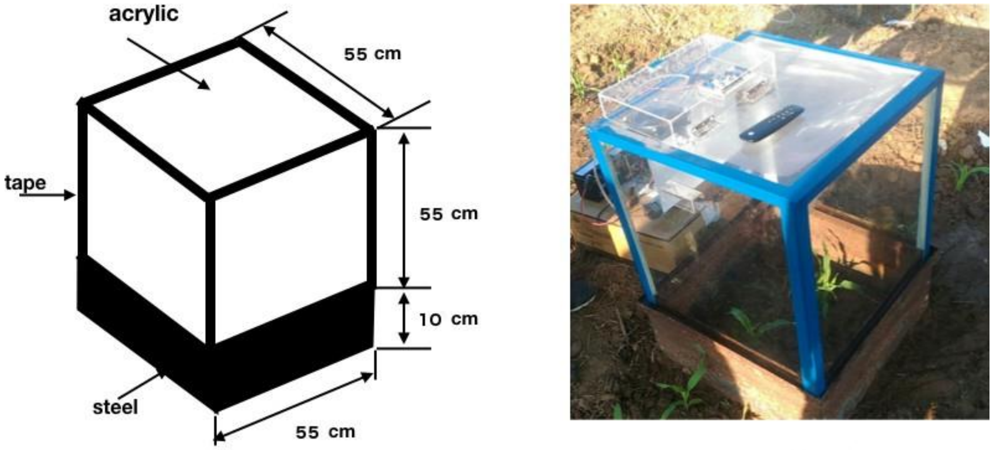

**Figure 1.** The chamber size and the photograph of the real chamber on the farm site

### *Hardware and software architecture*

The architecture of hardware and software of the developed system is shown in Figure 3. It divided into 4 layers. The developed system adopted the IoT architecture which consists of a sensing layer, network layer, service layer and application layer. In the sensing layer, Arduino mini and $N_2O$ sensor module were used to gather measurement result and send it using RS 485 protocol at 9600 bps to the IoT module on the network layer to be saved in the cloud system (Firebase, Inc.). Furthermore, the android application gathers the measurement result from the cloud system to be displayed on the screen. The android application to monitor the measurement result in real-time was developed by using MIT App Inventor using Android 9 as the operating system.

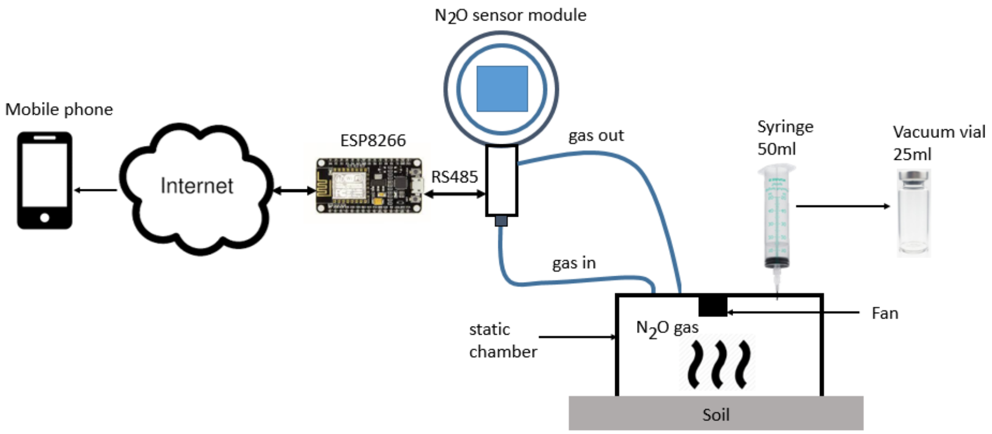

**Figure 2.** Diagram of the experimental components

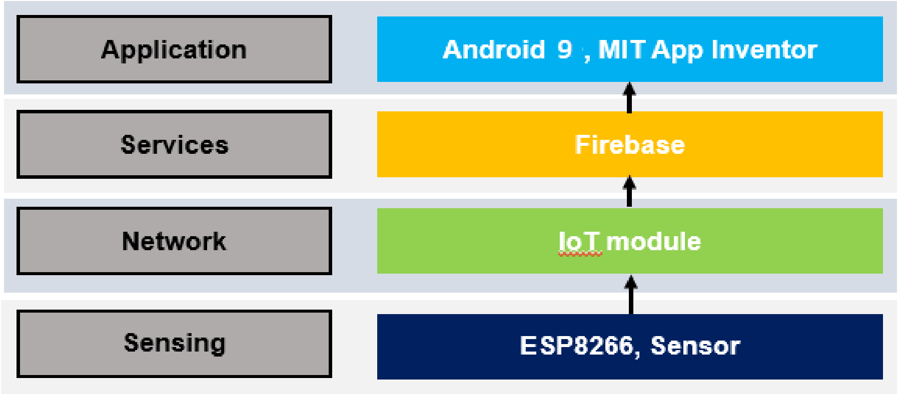

**Figure 3.** The architecture diagram of the developed hardware and the application

### *Measurement method*

#### Using standard gas

Measurements using standard gas were carried out to see the characteristics of the sensor module. The $N_2O$ measurement module was tested using standard $N_2O$ 1 and 2 ppm gases (supplied by PT SHC Gas Indonesia) which consists of a mixture of $N_2O$ and Argon gas. The measurement was done by taking gas as much as 10 mL using a syringe in a tube containing a capacity of ± 1.5 $m^3$. 1 and 2 ppm standard $N_2O$ gas were injected into the $N_2O$ sensor module using a three-way stopcock connector. Furthermore, the gas is allowed to flow through connected the input hose and output hose. The measurement was conducted for 20 seconds which provided 20 values of measurement results. As a comparison, a GC measurement method was also applied to these standard gases.

### In situ measurement

Measurements were made by applying 10 grams (e.q. 150 kg-N ha−1) of urea fertilizer by perforating the soil approximately 2-3 cm and covering it with soil on an experimental plot of land on which the chamber was placed at around 10 a.m. Ten minutes after the application of the fertilizer, during which there was good mixing of the air by the fan in the chamber, the first measurement of $N_2O$ was made. The input and output pipes were placed in the chamber so that the measurement was only of the air contained in the chamber. Before fertilization was carried out, gas measurement without fertilization, as a control, was performed. Measurements using $N_2O$ sensor were carried out by taking the measurement results every 1 second for 2 minutes to get 120 values. On the second day after fertilization (day 2) between 09:00 a.m and 02:00 p.m, $N_2O$ gas measurement was again conducted. In addition, the temperature in the chamber was determined using a thermometer. The measurement was continued for the next two days (day 3 and day 4) using a similar procedure as the method on day 2. This measurement was given the name as experiment 1. One day after experiment 1 was conducted, a similar experiment and method were carried out at the same location but on a second experimental plot of land which is 3–4 meters away from the location of experiment 1 as a comparison namely experiment 2. The temperature inside the chamber during measurements were taken at 40 $^{o}$C on the first experiment and 38 $^{o}$C on the second experiment. The result was recorded as a CSV file for analysis purposes. The measurement was conducted twice to ensure that the measurement is reproducible. Previous studies have used an automated chamber that can be used for continuous system (Denmead, 1979; Ambus and Robertson, 1998; Denmead *et al.*, 2010; Jørgensen *et al.*, 2012). However, in this study, a static measurement was taken and will not be used for continuous measurements. This measurement system will be developed for mobile measurements which will measure at one point and move to other points on the same land.

## *Statistical analysis*

Statistical analysis was performed to determine the characteristics and performance of the sensor module *in situ*, in agricultural setting by comparing two measurement results from the two experimental plots using one-way repeated-measures analysis of variance (ANOVA) as shown in Figure 4. The ANOVA one-way test was performed to determine whether there is a statistical difference between two unrelated sample groups. However, in this study, the measurement for the control was excluded because the results values were all zero, which means that the sensor did not detect any $N_20$ gas. Consequently, they could not be included in ANOVA calculations because the data generated does not meet the requirements of the technique. Before performing ANOVA calculations, outlier identification was carried out to check the values of data that are outside the range using a boxplot diagram. Then, a normality test was performed using a QQ plot. The QQ plot provides a graph of the correlation of data with the normal distribution. Data that were not normally distributed were transformed to become normal using the log function and Tukey transformation. Subsequently, the Mauchly's test, to evaluate whether the sphericity assumption has been violated, was carried out. A multiple pair-comparison test was performed using TukeyHSD (Tukey Honest Significant Difference) was applied to check the difference between specific pairs of days. The next step was to perform a Post-hoc test by conducting pairwise paired t-tests. The Bonferonni multiple correction method was used to adjust the p value. In addition, normality tests were carried out with the Shapiro test for the residu of the ANOVA test.

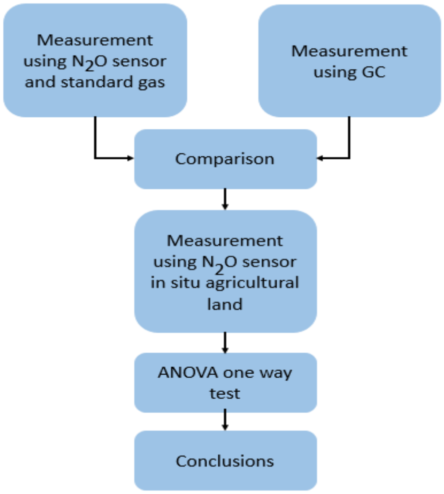

**Figure 4.** The methodological diagram of the study

## Results

### *Using standard gas*

The measurement result using standard gas 1 ppm and 2 ppm is shown in Figure 5. The boxplot shows that the median values of the measurement result were close to the standard value of the gas injected into the $N_2O$ gas sensor. The measurement results showed a varying value between 0 and 1.35 for a standard gas with a concentration 1 ppm and varying between 1.17 ppm and 2.61 ppm for a standard gas with a concentration of 2 ppm. Measurement results using GC were 1.05 ppm for the standard gas with a concentration of 1 ppm and 2.09 ppm for the standard gas with a concentration of 2 ppm. By using GC, 1 measurement value is obtained. Meanwhile, using IR sensors obtained several measurements were performed and the values obtained were very close to the value in each case. In order to get an estimated real value, statistical analysis should be applied such as mean, median or linear regression. Based on this result, *in situ* field measurement was conducted to obtain real measurement results.

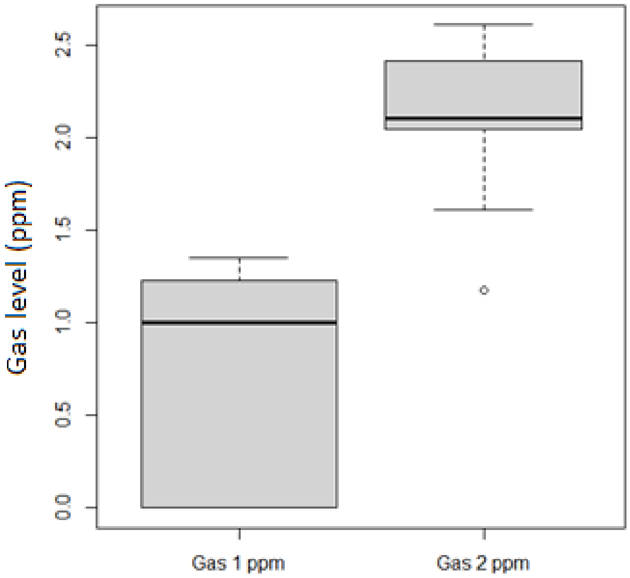

**Figure 5.** Measurement results of the developed system using standard gases 1 ppm and 2 ppm

### *In-situ measurement*

Boxplot charts of measurements results using the developed system in the in-situ filed experiments are shown Figure 6. There was an indication of a significant increase in concentration on day 3 and then a decrease on day 4 for experiment 1. The same pattern of results were also shown for experiment 2 where there was an increase in gas concentration on day 3 (not as large as experiment 1) and significant  decrease slightly on day 4. This reveals that both experiments show the same trend.

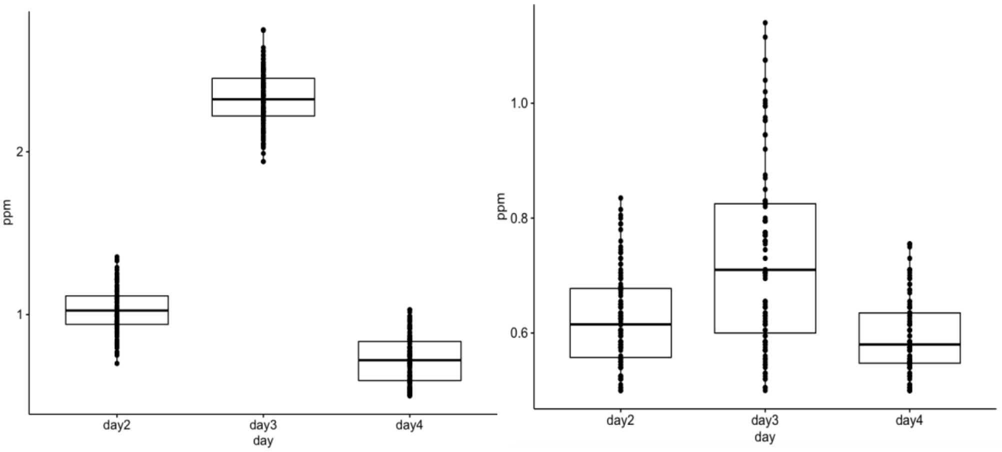

**Figure 6.** The raw measurement results for (a) experiment 1 and (b) experiment 2

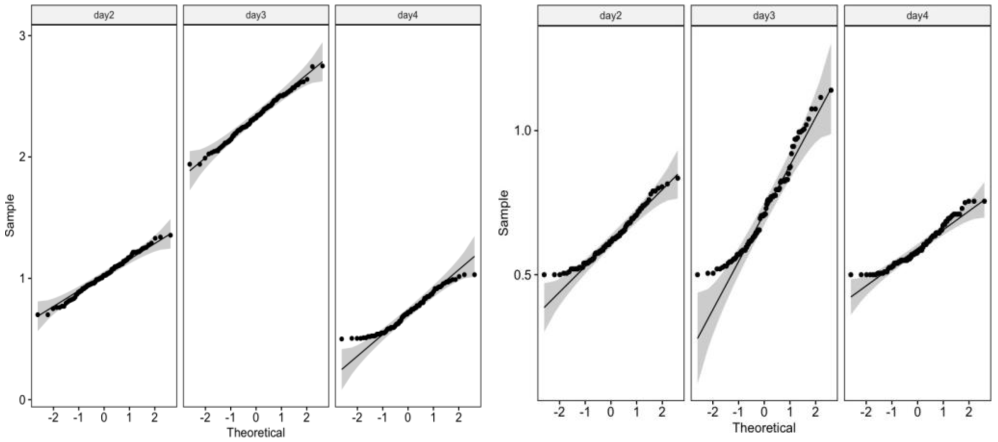

**Figure 7.** QQ plot for checking the correlation between the measurement result and the normal distribution

In order to perform ANOVA one-way test, the normality assumption was checked using the QQ plot as shown in Figure 7. The graph depicts the QQ plot of the measurement results for experiment 1 and experiment 2. On day 4 of experiment 1, it looks that some of the points did not fall along the reference line indicating that the data is not normally distributed. This also happened in experiment 2 on all trial days. For that reason, data transformation was performed to produce normally distributed data for one-way ANOVA calculations.

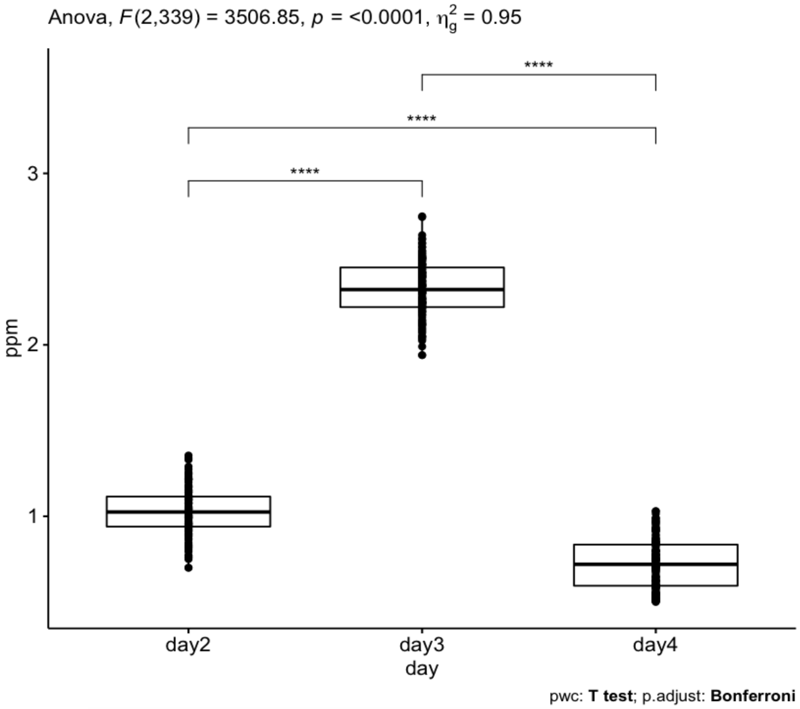

**Figure 8.** Result of repeated measures ANOVA for experiment 1 with normalised data (p=3.21e-227)

From transformed measurement data in the two experimental sites, the results of ANOVA calculation are shown in Figures 8, and 9. The picture of the results of experiment 1 showed an increase in $N_2O$ gas levels on day 2 and day 3 day of measurement. The latter decreased slightly on the day 3 day of measurement. The ANOVA calculation for experiment 1 revealed the F value with degrees of freedom (df) (2,339) was greater than the critical value which was 3506.85 and p value was <0.0001 which is smaller than the significance level of 0.05. It explained that there was a significant difference in experiment 1 of $N_2O$ gas levels for the three trial days. To determine statistically different day pairs, Tukey multiple pairwise- comparison was applied. The results demonstrate that all pairs show the value of p adj is equal to 0. It showed significant differences between all pairs of days. The second experiment is shown in Figure 8 which revealed, the same trend for experiment 2, namely a significant increase in gas levels on day 2 and 3. The negative values are produced on the boxplot graph and the raw measurement data are transformed to normal distribution. However, on day 4 there was decreased in gas level by 0.1 ppm. It revealed that the F value was 43.55 and the p value was <0.0001. It revealed that in experiment 2 there was significant differences in the three days of measurement of $N_2O$ gas levels. Tukey multiple pairs-comparison test results showed all p adj values equal to 0 on all pairs of days which confirmed differences in measurements for each pair. The trend and rate of the gas level on the day 2, 3 and 4 and the difference in measurement values in experiment 1 and experiment 2 were due to different soil conditions such as humidity, temperature, the presence of animal dung and the remains of dead plants (Jumadi *et al.,* 2019). The results from both experiments are shown in Table 1.

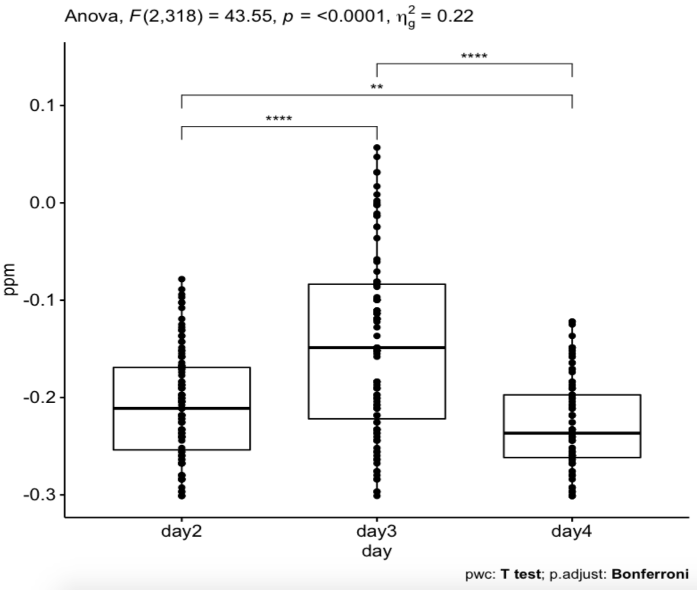

**Figure 9.** The repeated measures ANOVA for experiment 2 with normalised data (p=3.58e-18)

**Table 1.** Results values of ANOVA one way test for all experiments

| **Activities/Value** | **df** | **F** | **p** | **$\eta_g$** |
|---|---|---|---|---|
| **Experiment 1** | 2.339 | 3505.85 | <0.0001 | 0.95 |
| **Experiment 2** | 2.318 | 43.55 | <0.0001 | 0.22 |

The normality of the plot of residuals and residuals versus fits plots of the two $N_2O$ gas measurement experiments are shown in Figure 10 and 11. The plots of residuals of the two experiments are expressed. It showed that almost all points fall along the reference line. It revealed that all experiments were assumed to be normally distributed. Besides, from the two plots of residuals versus fits, no evidence of a relationship between residuals and fitted values is evident, so it is assumed that the experimental results were homogeneous.

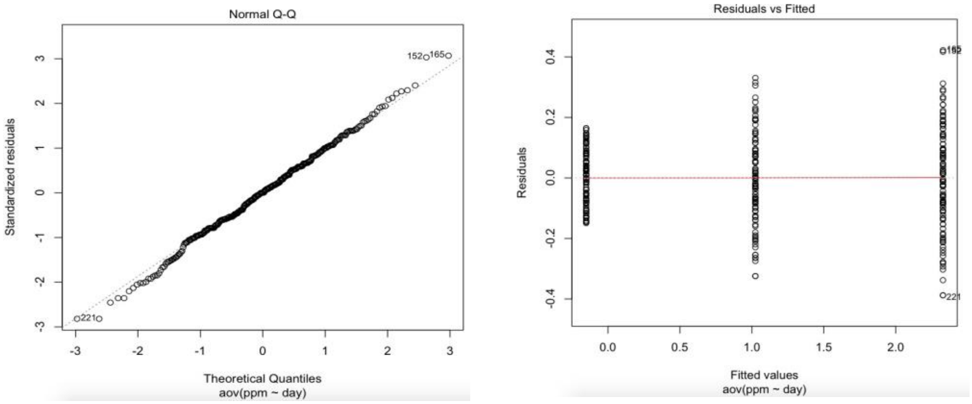

**Figure 10.** Residuals of repeated measures ANOVA for experiment 1

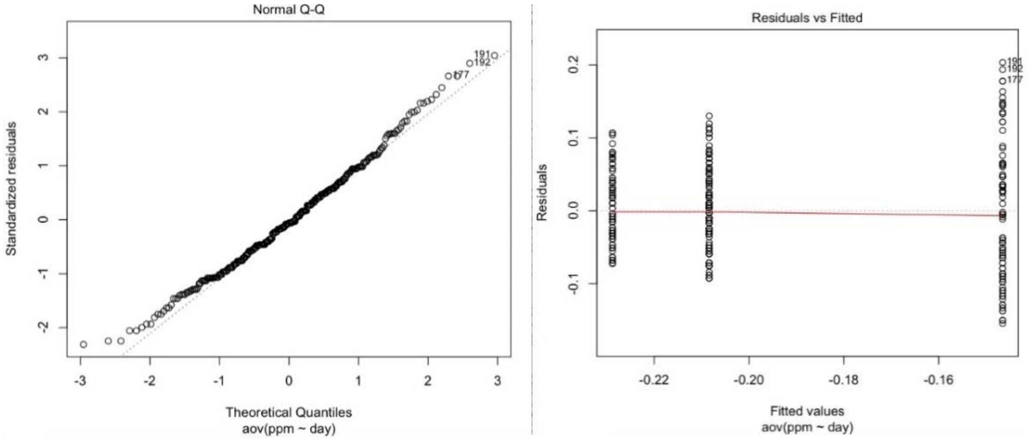

**Figure 11.** Residuals of repeated measures ANOVA for experiment 2

## Discussion

Fields on agricultural land that are not fertilized produce very little $N_2O$ gas. The application of nitrogen-containing fertilizers, such as urea and ammonium (NH+) to a field stimulates the production and emission of $N_2O$ gas (Luo *et al.,* 2007; Jumadi *et al.,* 2020). In this study, an increase in $N_2O$ gas was seen on days 2 and 3 after applying fertilizer. One of the factors that cause an increase in $N_2O$ gas level was the nitrification process which results in the change of $NH_4$ into nitrate producing high levels of $N_2O$ gas. On the following day, $NH_4$ was no longer available or only available in smaller quantiites. Consequently, the nitrification process did not occur again or decreased. In addition, measurement using the IR sensor are also affected by changes in the ambient temperature and pressure due to the response of components within the sensor that are sensitive to environmental factors.

This preliminary study has shown that a developed real-time measurement system can be used to remotely monitor $N_2O$ gas levels in agricultural land. Measurements using the IR sensor produced results that show statistical variation, reason for this include the response of the sensor to changes in temperature. Therefore, the use of statistics is needed to obtain estimates of measurement results. Measurements on agricultural fields are influenced by many additional things including the weather and initial soil conditions. The gas concentration fluctuation observed in this study occurs because of the nitrification process. Previous research has also carried out *in-situ* gas measurements using infrared-spectroscopy (Brummel and Siciliano, 2011). This study used soil gas probe that was inserted into the ground. The main difference with the research study described that the development allowed the measurement of $N_20$ gas in real time.

Several studies have used IoT system to control and monitor in agriculture (Doshi *et al,* 2019; Ayaz *et al.,* 2019). However, in this study, the application of the IoT feature to measuring $N_2O$ gas levels facilitates a gas monitoring process that runs in real-time and can be monitored anywhere using a mobile phone. This has significant practical advantages compared with measurements using GC that must be done in the laboratory. Another advantage of adding this IoT feature is the ability to transfer and record measurement result in a cloud database that can also be accessed anytime and anywhere, thereby reducing workloads and cost, thus increasing the efficiency of the measurement process. Furthermore, real time analysis can be performed or the saved measurement results can be used for further analysis to find a picture of the nitrogen (N) content of the soil.

Future research should take more samples on planted land to get more comprehensive dataset. In addition, measurements should be made with different soil and plant conditions. In the longer term, it is expected that the results will help farmers to correctly regulate their application of fertilizer to maximise efficiency and minimise production of unnecessary greenhouses gases.

It concluded that a measurement system for measuring *in-situ* $N_2O$ has been developed. The system was created using an IR sensor module and IoT technology to monitor the level of $N_2O$ on the agricultural land. The measurement system used a chamber to hold the gases mitted from the soil. A plastic pipe was inserted in to the chamber to permit the entry of air. The sensor module detected the gas level from the air inhaled by a pump from the plastic pipe. The result was sent to IoT module for reading and recording in the Cloud. The result showed an increasing level of $N_2O$ when the soil is fertilized. There were significant differences in measurements on the second, third and fourth days for the two experiments. This fluctuation occured because of the nitrfication process which resulted in the conversion of $NH_4$ to nitrate which produces $N_2O$ gas. Thus, this preliminary study is shown that the developed measurement system can be used to remotely monitor $N_2O$ gas levels in real-time agricultural land. Future research should take more a greater number of samples on planted land. In addition, measurements should be made with different soil and plant conditions.

## Acknowledgements

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