Use of MOS Gas Sensors with Temperature Modulation-Specified Detection Point for Potential Identification of Soil Status using Electronic-Nose Principle

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ABSTRACT

This dissertation presents a potential use of array MOS gas sensors in electronic-nose system which driven by temperature modulation-SDP (Specified Detection Point) to identify the soils and their status by capturing the gaseous profiles. We built a self-made e-nose consists of (a) 6 MOS gas sensors which driven and acquired wirelessly to a computer through (b) a PSoC CY8C28445-24PVXI-based interface, and (c) the Principal Component Analysis and Neural Network as pattern recognition tools. We tested the e-nose to identify 2 soils (sandy and loam sand) and the presence of nutrient addition as well. The gaseous compounds are accumulated in a static headspace with termostatting and stirring under controlled condition to optimize the equilibration. The patterns are trained by back-propagation algorithm which employs a log-sigmoid activation function and updates the weights using search-then-converge schedule. PCA results indicate the distinct soil gaseous profiles can distinguish the soil type clearly and to indicate the presence of additional nutrients in soil and their level as well. Moreover, the PCA helps improving the NN classification to differ level of compost addition in soil. An optimum single hidden layer architecture (3-6-3) NN is determined and employed successfully to discriminate among the three categories of compost dose (without, normal, and high).

Keywords: Soil gases, MOS gas sensor, temperature modulation, specified detection point, E-nose application.

1. Introduction.

The logical reasons for this study is that since the existence and content of smell molecules and organic substances in different soil type and the composition of volatile substances of nutrient addition would provide a unique soil gaseous profile (also called fingerprints). It is might resulted from decomposition of organic matters and chemical reactions among others. The smell molecules of soil are known as geosmin and methylisoborneol which mostly produced by bacteria belonging to the most genus Streptomyces that involves a number of enzymes, one of key enzymes is germacradienol synthase (Green, Blincoe ’, & Weeth, 1975; Mei Wang & Cane, 2008; Wang & Cane, 2008). And, the odorous compounds result from decomposition of matter (Scaglia et al., 2011; Vass et al., 2008) and some strong evidences which pointed that resulted gases and volatile organic compounds in the soil atmosphere in vary types and relative concentrations (Peñuelas et al., 2014; Wheatley, Millar, & Griffiths, 1996) might be produced due to fertilizer adding and microbial activity (De Cesare et al., 2011) which influenced by environment conditions (Milchunas, Parton, Bigelow, & Schimel, 1988; Sherlock, Freney, Bacon, Weerden-TJ, & Van der Weerden, 1994; Smith et al., 2003).

Based on qualitative soil gaseous analysis, this dissertation aims to examine the potential of use an array of chosen MOS gas sensors which driven by temperature modulation-SPD in an e-nose-based system for early and rapid indication of soil status/condition. All MOS gas sensors are driven and acquired wirelessly into a computer by a PSoC based interface system through XBee serial communication (IEEE 802.15.4).

We introduced the new technique namely temperature modulation with specified detection point (temperature modulation-SDP) which is able applied to drive the single/array of MOS gas sensor (Sudarmaji & Kitagawa, 2015). Basically, it is similar with general temperature modulation, yet it also modulates the sensing (detector) unit that associated and in same phase with temperature modulation on the heater unit. The SDP means the time of output detection of MOS gas sensor is put at specified point (i.e. at middle of sensing unit modulation). The principle of this technique is shown in Fig. 1. In our first investigation, the rectangular (square) modulation was successfully designed and it led to response more distinct and sloping at lower frequency. It may increase the sensitivity and selectivity either on single or array sensors rather than static temperature. By applying selected modulations on 6 MOS gas sensor and shown with PCA, it provided more than 60% increment of selectivity compared with static temperature in discriminating 3 gases (Toluene, Ethanol and Ammonia).

2. Experimental Material and Method.

2.1. The Self-made E-Nose.

We built self-made e-nose (Fig. 2) that consists of 3 main unit. (1) Sensing unit: 6 MOS gas sensors (TGS2444, TGS2602, TGS825, FISAQ1, FISSB30, and FIS12A) which driven by temperature modulation-SDP and expected to response soil gases and VOCs, and 2 environment sensors (LM35 and HSM30G) to monitor temperature and humidity in sensor chamber. (2) An interface system based on PSoC CY8C28445-24PVXI as modulation generator and data acquisition (PSoC diagram is shown in Fig. 3). And (3) Principal Component Analysis and Neural Network as preprocessing and pattern recognition respectively which are developed under Visual Studio 2011.

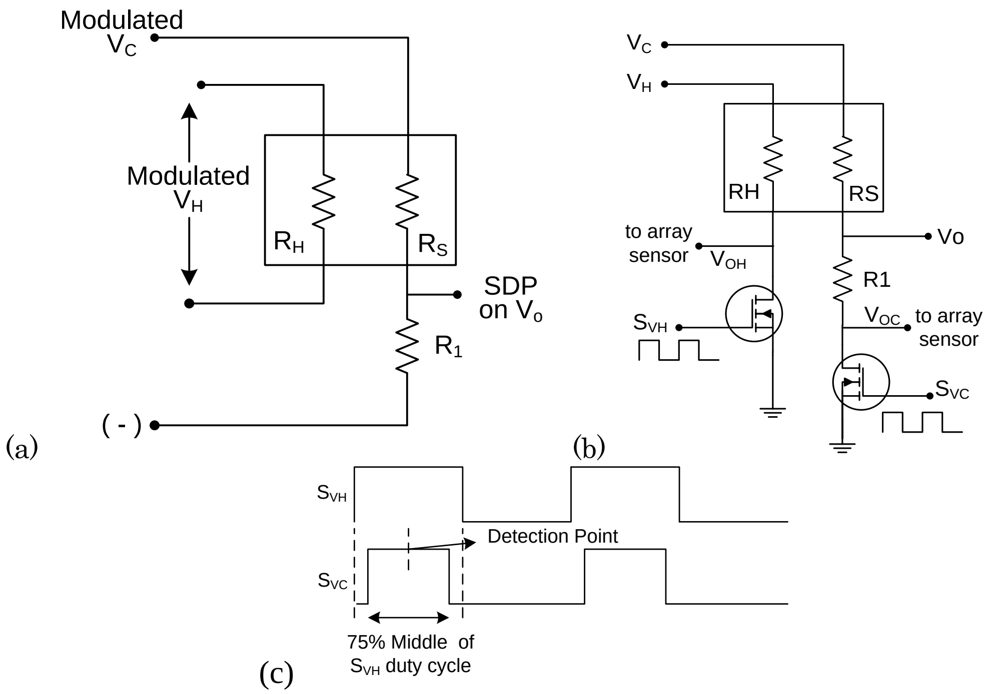


Fig. 1. Temperature modulation-SDP: (a) the principle, (b) the schematic of temperature modulation-SDP for single/array TGS sensor, and the modulation signal. V_H is heater voltage, V_C is sensing circuit voltage, S_{VH} is modulation signal for V_H, and S_{VC} is modulation signal for V_C (Sudarmaji & Kitagawa, 2015).

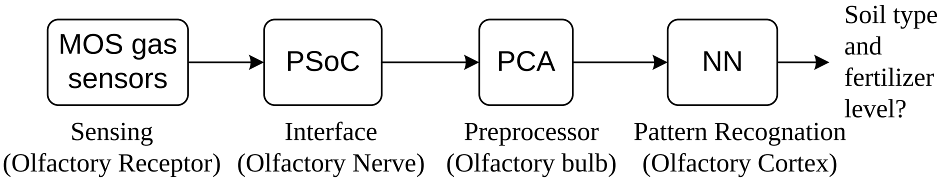


Fig. 2. Measurement diagram of soil vapor fingerprint based on e-nose principle.

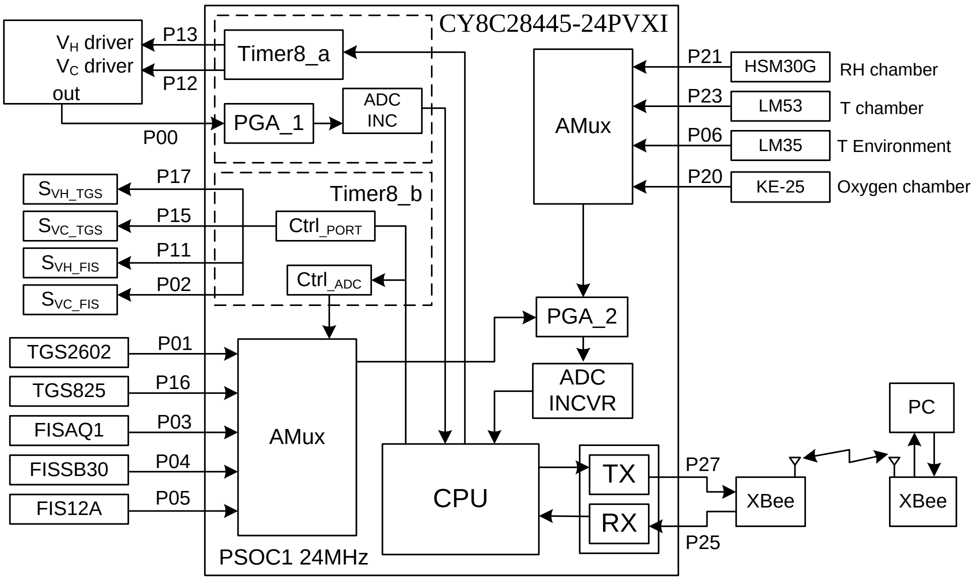


Fig. 3. Diagram block of system based on PSOC CY8C28445-24PVXI with pins configuration (Sudarmaji & Kitagawa, 2015).

2.2. Soil Preparation and Sample Handling.

The two soil types (sandy loam and sand soil) were derived from the top 15 cm and land without prior soil management. Sandy clay loam soil was taken from land around Kanazawa University (36°32'46.3380"N, 136°42'11.5452"E), while sand soil was taken from around coastal area of Uchinada Beach (36°38'39.19"N, 136°37'37.88"E), a sand hill on Sea of Japan, which is located about 17 km from Kanazawa University. The collected soil samples were crushed and sieved manually at <2 mm after plant derbies, turfs, and gravels were carefully removed. As soil treatments, we added an amount of fermentation compost. The compost is given at average and high doses as recommended in practical application, i.e. 20 and 30 ton ha^{-1} DM (Dry Matter) respectively (Haber, Deller, Flaig, Schulz, & Reinhold, 2010). Thus, we added it at rate 0, 15, and 22.5 mg/g soil sample corresponding nearly to 0, 20, and 30 ton ha^{-1} DM respectively by considering that it is generally assumed that in 1 ha soil area, 15 cm deep, contains 2Mkg despite bulk density of soil varies considerably (Conklin, 2014; King, 1911).

We prepared the samples in static headspace (SH) into solution since soil contains many soluble substances in water and it has bigger diffusion coefficient than solid, thus leads shorter diffusion and consequently equilibration times. We determined the mass of soil sample using Eq. 1 to define the mass of pure water and compost addition, where m_s expresses mass of soil (g), V_v is volume of headspace vial (ml), ρ_s is bulk density of soil (sandy loam = 1.44 g/ml and sand = 1.51 g/ml) (Yu et al., 1993), ρ_w is density of pure water =0.998 g/ml, β (V_G/V_S) is phase ratio in SH, and w_c is water content (in fractional number). Table 1 resumes the properties of parameters used and calculation results.

$$m_s = \frac{V_v \times \rho_s \times \rho_w}{(\beta + 1) \times (\rho_w + w_c \times \rho_s)} \qquad \text{Eq. 1}$$

Table 1. Properties of samples of soil, fertilizer, water, and static headspace condition.

Properties of SH	Value
Volume of SH Vial	90 ml
Bulk density of sandy loam soil	1.44 g/ml
Bulk density of sand soil	1.52 g/ml
Phase ratio	1.5
Water content	1
Density of pure water	0.998 g/ml
Mass of sandy loam soil	21.22 g
- mass of compost adding at 20 ton/ha	0.318 g
- mass of compost adding at 30 ton/ha	0.477 g
Mass of sand soil	21.63 g
- mass of compost adding at 20 ton/ha	0.324 g
- mass of compost adding at 30 ton/ha	0.287 g

We optimized the headspace equilibration by both agitating (i.e. stirring) and termostatting concurrently for all samples on the same phase ratio. We set 30 minutes, 60℃, and 200 rpm of equilibration time, temperature, and stirring frequency respectively. We utilized The Corning PC-4200D to heat and stir the sample in the SH vial. We used 90 ml glass container with sealed cap as headspace vial which is put inside the 500 ml open beaker filled with 100 ml water (Fig. 4). It aims to maintain the equilibrium relative humidity the same as the soil sample. And, the headspacing was conducted inside a room with controlled-temperature. By those ways, all soil samples were under the same treatments and environmental conditions.

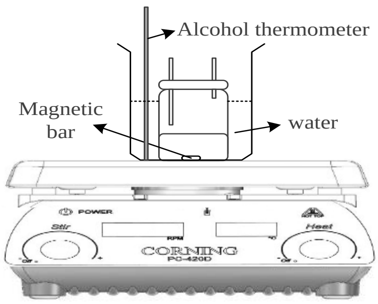


Fig. 4. Headspace conditioning with heating and stirring using The Corning PC-420D in SH sampling, the layout of Corning modified from (Corning Inc., 2007).

2.3. Measurement Procedures.

The measurement of soil gaseous profiles are performed using close measurement method by switching between the reference gas (filtered air with silica gel) as baseline and analyte gas (soil gaseous compounds). The flow direction and rate of gas are controlled by 3-way valve and The Koflok mass flow controller (MFC) respectively. The

MFCs are set at 0.3 lpm. As shown in Fig. 5 the reference gas flows through point a (valve-1), point c (valve-2), and point e (valve-3), while the analyte gas flows through point b (valve-1), point d (valve-2), and point e (valve-3). The purging of sensor chamber was in open measurement mode by disconnecting the hose of inlet pump from valve-2, directing the valve-3 to point f, and turning on the purge pump.

The temperature modulation was set on 0.25 Hz; 75% duty cycle to drive all MOS gas sensors, except TGS2444. As initial action at first time turning on, the system turned on operating in reference measurement mode for one hour to allow the MOS gas sensors reach stabilized. The gas sensors are expressed in resistance and the profiles is defined by its Sensitivity (S), where R_0 is sensor resistance of air and R_g is sensor resistance of analyte gas exposure (Eq. 2) (Arshak, Moore, Lyons, Harris, & Clifford, 2004; Huang, Liu, Shao, Pi, & Yu, 2003). Concisely, the overall steps of measurement is shown in Fig. 6.

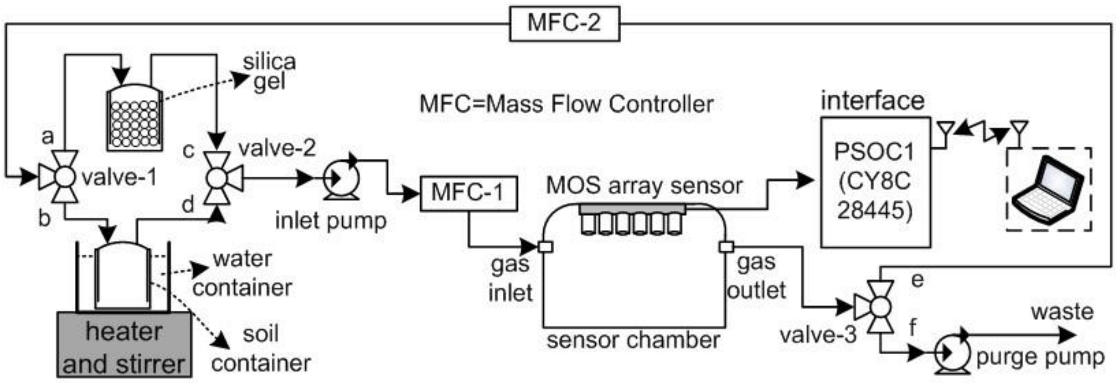


Fig. 5. Experimental setup to capture the soil gaseous compounds using static headspace extraction in sample flow system (close) measurement.

$$S = \frac{R_0}{R_g} \qquad \text{Eq. 2}$$

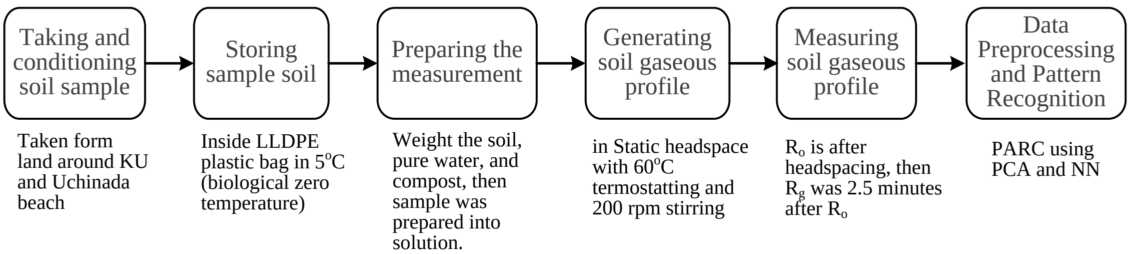


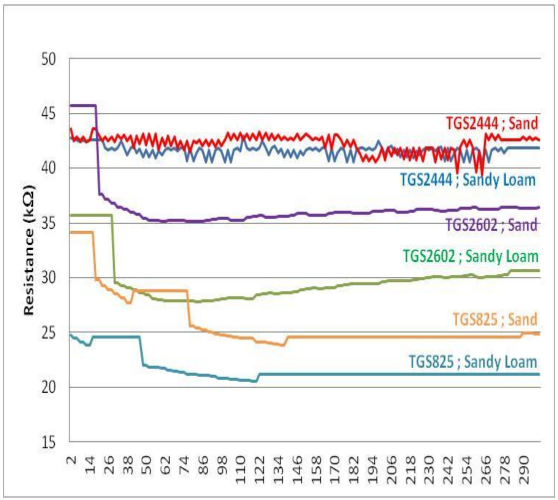
Fig. 6. Measurement steps to indicate the nutrient level based on soil gaseous profiles.

3. Results and Discussion.

Initially, we observed R_g for 5 minutes after R_0 measurement to determine the response of each sensor and obtain the best starting measurement time for R_g measurement since we assumed the gas distribution is not spread evenly. Significantly, we found that overall sensors reached a stable state after ±150 s (±2.5 minutes) which strongly indicate they sensing stably the flow of gas that have been spread evenly in the close measurement system. Therefore we took this time be the starting point of R_g measurement.

Fig. 7 also shows that most sensor has similar response (except TGS2602 and

TGS2444) to the flow and distribution of gas produced in the headspace, but reaches a different stability time. Particularly on TGS2444, even though it seem most distinguish (seem unstable and more ripples) among the others, yet it still showed its typical response. When it is expressed in ppm (part per million) using graphical calibration in its datasheet (Figaro Engineering Inc., 2011), the values lie around 2 ppm. While the resistance of TGS2620 suddenly dropped then toward its stability response.



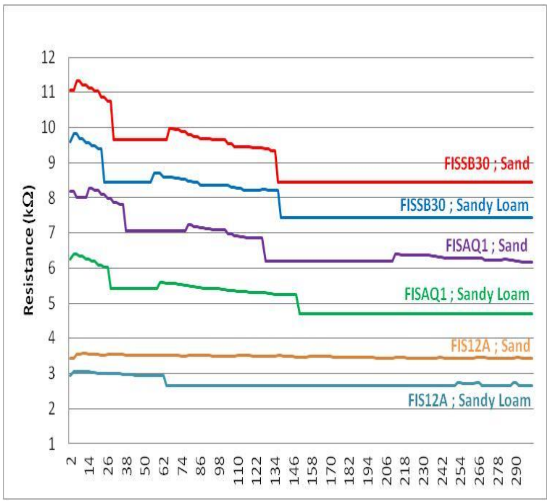


Fig. 7. The response of TGSs (2444, 2602, 825) and FISs (AQ1, SB30, 12A) to soil samples (sandy loam soil and sand soil) without compost addition under 0.25 Hz; 75% modulation in 5 minutes.

Individual soil gaseous profiles on each soil type shown in Fig. 8(a). The chart reveals that the highest concentration during the headspace process was hydrogen sulfide (H_2S). It highly indicated there much acid sulfate materials in soil samples. This gas is produced by some bacterial actions upon organic matter with the aid of the sulfates oxygen contained as an oxidation in low oxygen level (like flooded soil) which depends on ambient conditions such as temperature, humidity, and the concentration of certain metal ions (Chou et al., 2014; Elion, 1927). And, soils may absorb amounts of H_2S from the air through atmospheric deposition, migration of mobilized pore water, or sulfuric material from spills and leaks, then retaining most of it in the form of elemental sulfur as sediment (Chou et al., 2014). The result also shows that the sandy loam soil provided higher concentration than sand soil since it contained higher organic matter.

However, we also observed that there is an overlapping response in differing level of compost addition (Fig. 8 (b)), especially between in dose 20T/ha and 30T/ha, in which this phenomena also shown in the other sensors. However, it may be reduced by new dimension projecting using PCA as commonly used in E-nose.

3.1. Performance of Discrimination of Nutrient Level in Soil.

PCA projects variables onto fewer dimensions, original data can be condensed to a few variables reflecting the most relevant analytical information (Hines, Boilot, Gardner, & Gongora, 2003). This offers an advantage that the classification of unknowns is processed much faster, thus reducing detection time. We put three principal components (PCs) to distinguish between headspace volatiles released from soil samples and input of neural network since they represent more than 90% of divergence samples data (Table 2).

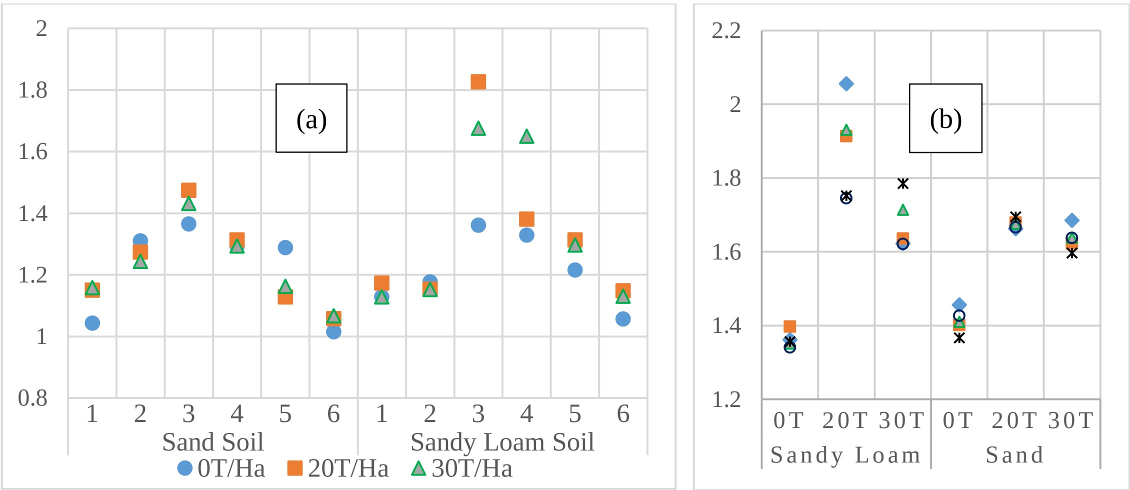


Fig. 8. (a) Individual Sensitivity of sensor, average of 5 replicates, to 3 level of compost adding in different soil, 1:TGS2444, 2:TGS2602, 3: TGS825, 4: FISAQ1, 5: FISSB30, and 6: FIS12A, (b) Experiment result of TGS 825 responses to compost dose (Ton/Ha) in sandy loam and sand soil for 5 replicates.

Table 2. Cumulative proportion of 3 PCs resulted from 6 sensors used.

PC	PCs proportion			
	SL*	S*	SL+S*	Soil diff*
PC1	64.27%	75.61%	66.53%	52.69%
PC2	86.34%	88.96%	80.69%	78.32%
PC3	93.73%	93.73%	89.18%	90.38%

* SL=Sandy Loam; S=Sand; Soil diff=differing between sandy loam and sand soil.

Overall by Fig. 9(a), (b), (c), and (d) shows that the principal components 1 and 2, which account about 64% and 83% cumulatively of the variance in the input variables, allow to discriminate distinctly type of soil and to differ between soil condition whether with or without compost (nutrient) addition, even in discrimination regardless of soil type. It was only for sandy loam soil (Fig. 9 (a)) the level of compost were able to be classified clearly into three groups as predefined previously while for sand soil (Fig. 9 (b)) there were miss-identification between soil with dose 20T/Ha and to 30T/Ha. Moreover, it also might there no clear classification when identifying soil with dose 20T/Ha and to 30T/Ha in regardless of soil type (Fig. 9(c)).

Finally, we determined the performance of NN as decision unit of e-nose to classify the level of nutrient addition in soil based on indicator the error (MSE) achieved resulted from the training process. We designed the architecture of MLPNN that comprises 3 layer (single hidden layer). We determined the optimum number of neuron in hidden layer by Singular Value Decomposition (SVD) analysis of its output in each training dataset (Tamura, 1997). By input from 3 PCs and considering resulted SVD value, we choose 6 neuron in hidden layer to differ among the pre-described three categorized fertilizer levels in soil sample, thus the neuron number architecture of MLPNN is 3-6-3 of respectively input, hidden, and output layer.

In learning, we took the learning parameters of BP as follow: maximum epoch is 10^4, error target is 10^{-5}, initial learning rate is 0.8 and the constant of search time in search-then-converge annealing learning rate is 700. We also trained the NN by input directly from sensors output (without preprocessing/PCA) with the same hidden layer (6-6-3 NN architecture). The achieved MSE of training results (Table 3) show that PCA helps improving the NN classification to differ level of compost addition in soil. The all application of trained data shows successful recognitions to indicate level of nutrient addition in soil as well.

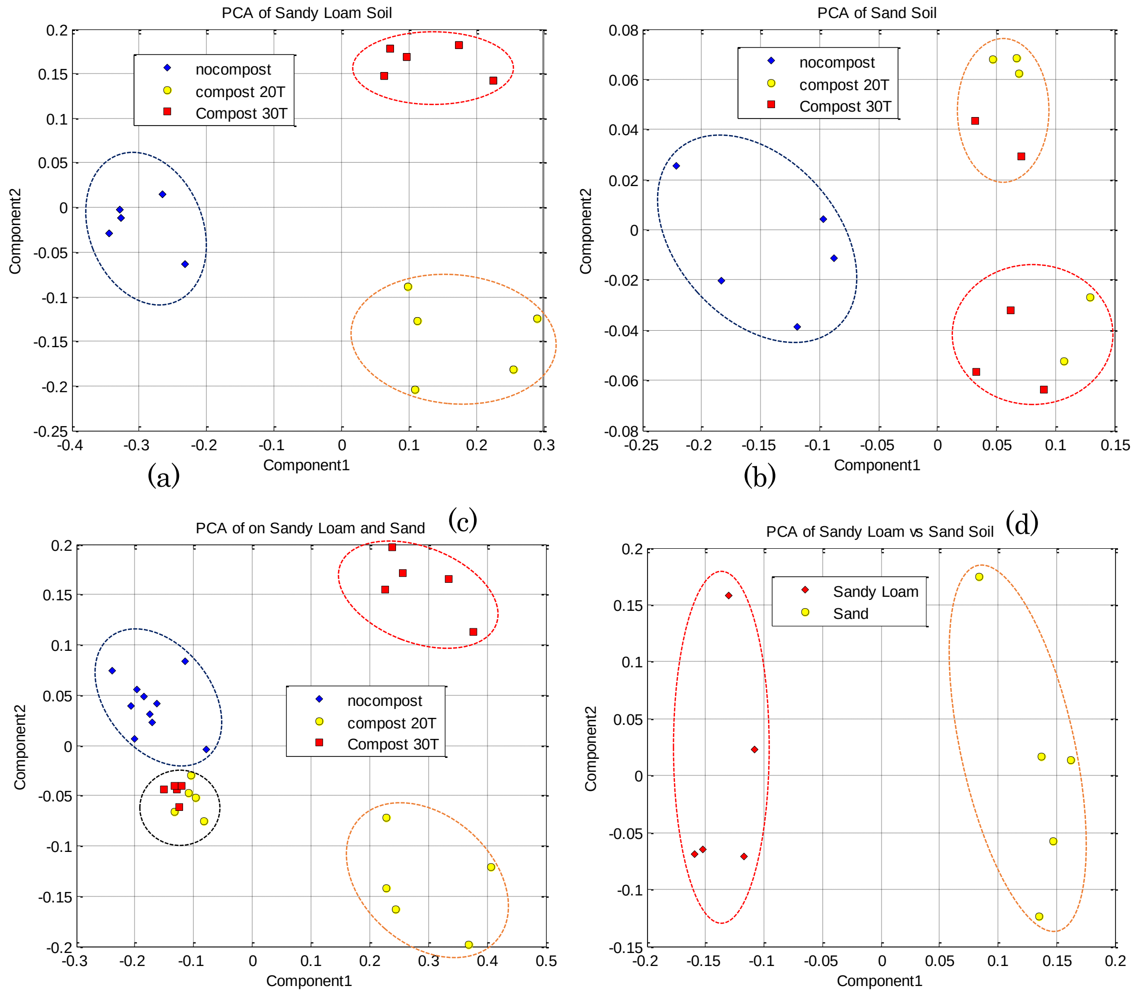


Fig. 9. Soil gaseous pattern projection mapped in 2 PCs for each soil sample to differ the level of compost addition of (a) sandy loam soil, (b) sand soil, (c) regardless of soil type by merging divergence data of both sandy loam and sand soil, while (d) PCA result in differing between sandy loam and sand soil both without compost addition.

Table 3. MSE achieved by 6 neuron of hidden layer to discriminate 3 level of compost addition in soil.

Soil type	MSE of with PCA	MSE of without PCA
Sand	4.204e-04	3.490e-03
Sandy Loam	1.226e-04	5.024e-04
Regardless of type	2.678e-03	4.080e-03

4. Conclusions and Future Work.

An application of new technique, namely temperature modulation-SDP (Specified Detection Point), on MOS gas sensors for soils identification based on volatiles profiles using biological system (e-nose) is presented. The 6 commercial MOS gas sensors used which driven by this technique are promising used for indicating the presence of additional nutrients in soil and their level as well since they could response and provide (unique) soil gaseous profiles resulted from a static headspace optimized by termostatting and stirring in certain condition. The temperature modulation-SDP in the e-nose system could differentiate clearly the soil type and indicate the presence of nutrient addition in soil. The optimum architecture of MLPNN with single hidden layer was 3-6-3 by PCA as prior data preprocessor which it leads better identification. However, the PCA result also shows there is miss-classification when discriminating the soil with normal dose (20 Ton/Ha) and high dose (30 Ton/Ha). Therefore, it is strongly needed further investigations on other/many MOS gas sensors and their correlation or calibration to the parameters of soil nutrient. The gas sensors with this particular technique also offers a potential for replacing existing techniques in soil environmental fields for a quick and in-situ application. Depending on the applications and the type of sample to be analyzed, the choice of sensor array can be crucial for the good performance of the system

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平成 28 年 1 月 29 日

学位論文審査報告書（甲）

１．学位論文題目（外国語の場合は和訳を付けること。）

Use of MOS Gas Sensors with Temperature Modulation-Specified Detection Point for Potential Identification of Soil Status Using Electronic-Nose Principle

和訳：最適温度変調検出法により高精度化されたイーノーズの土壌状態評価への応用

２．論文提出者　(1) 所　　属　電子情報科学　専攻

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３．審査結果の要旨（600～650 字）

平成 28 年 1 月 26 日に開催した第 1 回学位論文審査委員会、平成 28 年 1 月 29 日に実施した口頭発表と第 2 回学位論文審査委員会で審査した結果、以下の通り判定した。

現代の農業においては、勘や経験に基づく記録や管理ではなく、事実の記録に基づくきめ細やかな管理により、収量、品質の向上及び環境負荷低減を図ることが求められている。特に情報技術を活用した農業生産の管理が重要な課題となっている。

本論文では、無線ネットワーク技術を農業生産の管理に応用することを目的とした土壌状態の自動分析手法を提案し、試作システムによりその有効性を実証している。

金属酸化物半導体ガスセンサアレイを用いたイーノーズ（においセンサ）を、土壌に含まれる主要栄養素などに関わる物質の分析に適用するため、高精度化された成分分離手法として、最適温度変調検出法を新規に提案している。さらに、この手法を用いて得られたセンサアレイのデータに、主成分分析とニューラルネットワークを適用し、土壌の施肥状態の判定に成功した。以上の研究成果は、農業生産管理に電子情報技術を利用するための基礎となるものであり、博士論文に値するものと判定する。

４．審査結果　(1) 判　　定（いずれかに○印）　○ 合　格　・　不合格

(2) 授与学位　博　士（学　術）