*Research article*

# Evaluating Changes in the State of Organic Matter Present in Agricultural Soils based on Loss on Ignition Method

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**Abstract** Loss on ignition (LOI) method is widely regarded as a quick, inexpensive method for evaluating organic matter characteristics in terrestrial soils and littoral sediments. Previous studies examined the LOI method alone to evaluate the state of organic matter, finding that changes in the state of organic matter in littoral sediments could be represented by LOI method-based indices related to combustion temperature: $(LOI_{300}-LOI_{200})/LOI_{600}$ and $LOI_{300}/LOI_{600}$. However, these new indices have not been applied to agricultural soils. In this study, laboratory experiments were conducted to examine the validity of our proposed indices for evaluating the state of organic matter in agricultural soils. Paddy soil, Andosol, and cow manure compost were allowed to decompose over three months. Measurements of pH, redox potential (ORP), and weight LOI at 200, 300, and 600ºC were measured at one and three months after the experiments started. Temporal decreases in ORP were observed, highlighting the decomposition of organic matter present in each material. The $(LOI_{300}-LOI_{200})/LOI_{600}$ metric changed over time for all materials, whereas the $LOI_{300}/LOI_{600}$ metric was almost constant. We concluded that $(LOI_{300}-LOI_{200})/LOI_{600}$ can represent changes in the state of organic matter in agricultural soils. Furthermore, we discovered that $LOI_{200}/LOI_{600}$ can represent changes in the state of organic matter with reasonable accuracy, rather than the more complicated $(LOI_{300}-LOI_{200})/LOI_{600}$. This finding should make LOI measurements easier and more affordable.

**Keywords** loss on ignition, organic matter state, decomposition, combustion, agricultural soils

## INTRODUCTION

Soil organic matter (SOM) is any plant or animal material, including leaf litter, microbial biomass, water-soluble organics, and humus at different stages of decomposition (Stevenson, 1994; Brady and Well, 1999). SOM has a major impact on the physicochemical properties of soils (Tisdall and Oades, 1982). For example, SOM has been shown to affect the cation exchange capacity, structure, water infiltration rate, water-holding capacity, erodibility and conversion, and pesticide adsorption of soil (Schulate, 1995; Ding et al., 2002).

Several methods have been proposed to measure the organic matter of terrestrial soils and littoral sediments. For instance, the Walkley-Black method is a wet oxidation method used to determine the amount of organic matter from the organic carbon content of the soil (Swift, 1996; Kerven et al., 2000). Another method, loss on ignition (LOI), uses dry combustion method to directly determine the amount of organic matter (Cambardella et al., 2001; Konen et al., 2002). Automated CHNS analysis conducted on gas chromatographs has been used to quickly determine the elemental contents (i.e., C, H, N, and S) with high precision (Telek and Marshall, 1974). With advancements in technology, it is now possible to accurately analyze the organic matter characteristics of a sample based on molecular weight measurements, thermogravimetric analysis, and spectrometric analysis (Cuypers et al., 2002; Hong et al., 2010). To the best of our knowledge, the LOI method is the most economical method that is suitable for rapidly analyzing large numbers of samples with the advantage of time and cost savings, it does not require a measurement expert, and it is an accessible method for developing countries.

To date, some authors have evaluated the amount and state of SOM on the basis of the LOI method, alone. For instance, the ratio of weight losses on ignition at 300°C ($LOI_{300}$) and at 600°C ($LOI_{600}$) was found to describe the state of organic matter present in littoral sediment (Hibino et al., 2014). Subsequent refinement revealed that $(LOI_{300}-LOI_{200})/LOI_{600}$ metric more accurately represents the state of organic matter in littoral sediments than $LOI_{300}/LOI_{600}$ metric, especially, changes in the state of organic matter due to decomposition (Touch et al., 2015; Touch et al., 2017). However, these metrics have not been used to evaluate the state of SOM in agricultural soils, so their validity should be tested in that setting.

## OBJECTIVES

This study aims to examine the validities of $LOI_{300}/LOI_{600}$ and $(LOI_{300}-LOI_{200})/LOI_{600}$ metrics in evaluating changes in the state of organic matter present in paddy soil, andosol, and cow manure compost. These materials are allowed to decompose over three months, and changes in their pH and redox potential (ORP) are measured for understanding the decomposition of organic matter present in each material. Then, the association between temporal changes in $LOI_{300}/LOI_{600}$ and $(LOI_{300}-LOI_{200})/LOI_{600}$ metrics and the decomposition of organic matter is investigated for examining the validities of these metrics. Furthermore, this study also aims to propose a more economical metric for examining the state of organic matter present in organic agriculture materials.

## METHODOLOGY

### Materials and Experimental Procedures

The paddy soil used in experiments was collected from a rice field (Ebina, Kanagawa, Japan) during the agricultural off-season. Approximately 150 mm of the surface soil was collected and transported to the laboratory. Approximately 200 mm of the surface soil of an andosol was collected from a farmland (Nakai, Kanagawa, Japan) during the agricultural off-season. Finally, commercial cow manure compost was purchased and used in this study. All of the materials were used in the experiment without any pre-treatment. Each material was placed in the experimental device as shown in Fig. 1.

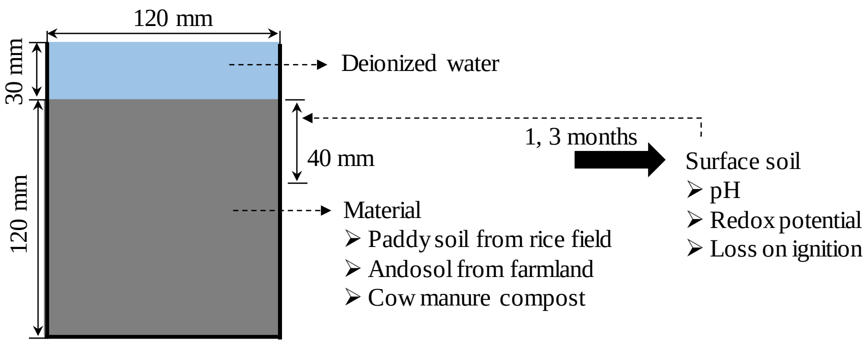



**Fig. 1 Diagram of the experimental device and some procedural details used in this study**

The experimental device comprised a cylindrical bottle with an inner diameter of 120 mm and a height of 150 mm. The bottle was filled with each material to a depth of 120 mm. The bottle was then filled the rest of the way with deionized water (Fig. 1), and placed under ambient conditions for three months. After one and three months, the top 40 mm of the surface soil was collected from the bottle for measurements of pH, ORP, and weight LOI.

### Soil Analysis

Each collected sample (paddy soil, andosol, or cow manure compost) was mixed before pH and ORP were measured. A D-50 pH/ORP meter (Horiba, Japan) was inserted directly into the sample. After the pH and ORP measurements were completed, samples were prepared for the measurement of weight LOI. Each sample was oven-dried at 50 ± 5°C for more than three days, and the dried weight was then measured. A temperature of 50°C is suitable for drying samples before determination of organic matter content (Takata et al., 2017). An electronic muffle furnace (Advantec, KL-420) was used to burn each of the dried samples for 4 h at each of three temperatures: 200, 300, and 600°C according to Saito et al. (1977). The sample was weight after burning at each temperature. LOI for each stage was determined by comparing these values with the 50 °C oven-dried weight. The measurements of LOI were performed in triplicate and had an error of ± 6 $mg.g^{-1}$.

## RESULTS AND DISCUSSION

### Temporal Decomposition of Organic Matter

Figure 2 shows the temporal changes in pH and ORP for each material while under ambient conditions. The ORP values started at 304±4 mV, 318±1 mV, and 96±9 mV for the andosol, paddy soil, and cow manure compost, respectively. The OPR of the paddy soil decreased to 93 mV over one month, and to −241 mV over three months.  Decreases in the ORP of andosols and cow manure compost were also found. After three months, the ORP was −226 mV for the andosol, and −437 mV for cow manure compost.

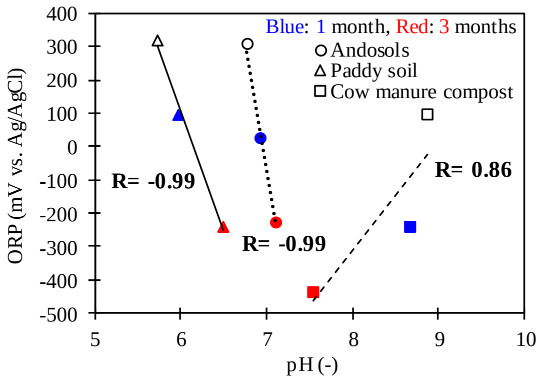
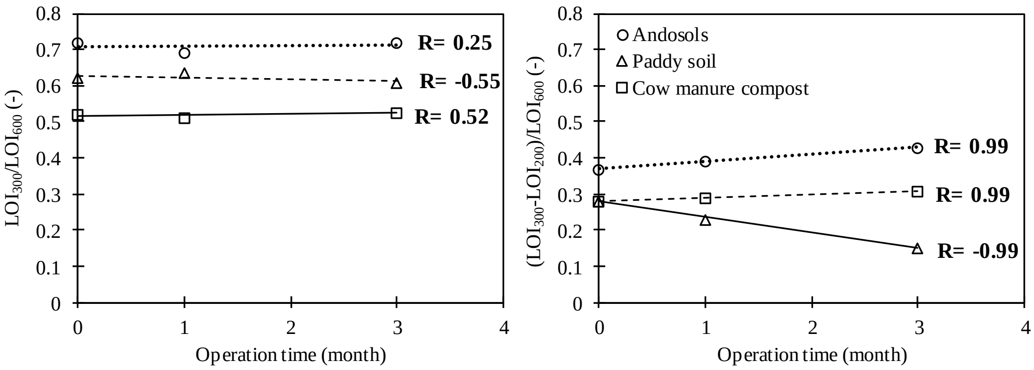



**Fig. 2 Temporal changes in pH and redox potential (ORP)**

Variations in pH and ORP can represent the decomposition of organic matter present in littoral sediments (Touch et al. 2015). A decrease in ORP indicates the decomposition of organic matter because oxidants are used in the decomposition processes. Thus, our results showed clear signs of decomposition of organic matter over time, as was found by Touch et al. (2015).

### Validities of $LOI_{300}/LOI_{600}$ and $(LOI_{300}-LOI_{200})/LOI_{600}$

As revealed by Fig. 2, the decomposition of organic matter present in each material decomposed over time. This means that a correlation between the proposed indices and operation time should be observed. Figure 3 depicts the temporal patterns of $LOI_{300}/LOI_{600}$ and $(LOI_{300}-LOI_{200})/LOI_{600}$ metrics. Low correlation between $LOI_{300}/LOI_{600}$ metric and operation time was confirmed, with this matric remaining nearly constant for each material over the span of experiment (Fig. 3a). This finding suggests that $LOI_{300}/LOI_{600}$ metric is unable to reflect the decomposition of organic matter present in andosol, paddy soil, and cow manure compost.




**(a) $LOI_{300}/LOI_{600}$** **(b) $(LOI_{300}-LOI_{200})/LOI_{600}$**

**Fig. 3 Temporal variations of $LOI_{300}/LOI_{600}$ and $(LOI_{300}-LOI_{200})/LOI_{600}$**

On the other hand, the $(LOI_{300}-LOI_{200})/LOI_{600}$ metric for each material had a strong correlation (R = 0.99 for all samples) with operation time (Fig. 3b). This demonstrates that $(LOI_{300}-LOI_{200})/LOI_{600}$ metric can indicate the decomposition of organic matter present in these materials. When combined with the findings of previous research (Touch et al., 2017), we concluded that the $(LOI_{300}-LOI_{200})/LOI_{600}$ metric can represent the decomposition of organic matter either in littoral sediments or in agricultural soils, including compost materials.

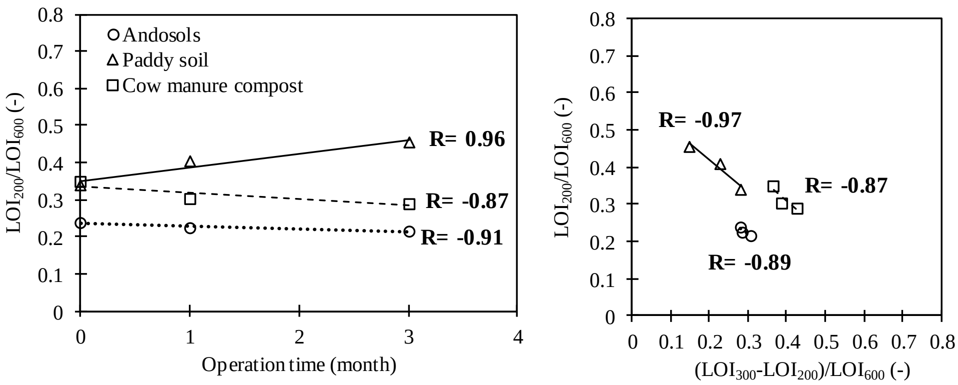
Interestingly, different tendencies in the variation of $(LOI_{300}-LOI_{200})/LOI_{600}$ metric were observed among the materials used. Specifically, $(LOI_{300}-LOI_{200})/LOI_{600}$ metric increased in the andosol and cow manure compost, while it decreased in the case of paddy soil (Fig. 3b). This reflects differences in organic matter decomposition characteristics among these materials due to a difference in the initial pH-ORP (Fig. 2) and organic matter content (e.g., Fig. 3a) of each material.

## New Index for Evaluating the Decomposition of Organic Matter

To explore the source of the difference in the performance between $(LOI_{300}-LOI_{200})/LOI_{600}$ metric and $LOI_{200}/LOI_{600}$ metric, Figure 4 shows the behavior of $LOI_{200}/LOI_{600}$ metric. This metric had a strong correlation (R > 0.87) with operation time for each material (Fig. 4a), but was not as strong as $(LOI_{300}-LOI_{200})/LOI_{600}$ metric (R = 0.99). Furthermore, it was observed that $LOI_{200}/LOI_{600}$ metric had a strong correlation (R > 0.87) with $(LOI_{300}-LOI_{200})/LOI_{600}$ metric (Fig. 4b), suggesting that $LOI_{200}/LOI_{600}$ metric can be used instead of $(LOI_{300}-LOI_{200})/LOI_{600}$ metric. This means that $LOI_{200}/LOI_{600}$ metric can be viewed as a new index for evaluating the decomposition of organic matter in these materials. This may provide an advantage in terms of cost and time savings, because only the weight losses on ignition at 200 and 600° C must be measured for this simplified metric.

As noted earlier, we observed different tendencies in the variation of $(LOI_{300}-LOI_{200})/LOI_{600}$ metric (Fig. 3b). According to Cuypers et al. (2002), the burning temperature of organic matter increases as the organic matter becomes more humified. For the cow manure compost, $(LOI_{300}-LOI_{200})/LOI_{600}$ metric increased (Fig. 3b), while $LOI_{200}/LOI_{600}$ metric decreased (Fig. 4a). This suggests a release of the organic matter burned at 200−300°C ($OM_{200-300}$) and the loss of the organic matter burned at 50−200°C ($OM_{50-200}$). The paddy soils, however registered a formation of $OM_{50-200}$ and a loss of $OM_{200-300}$.

Generally, cow manure compost contains the organic matter that readily decomposes, while the organic matter in paddy soils and andosols tends to be humified. Hence, these results reflect decomposition of $OM_{200-300}$, leading to the release of $OM_{50-200}$ in paddy soils, while the humification of $OM_{50-200}$ leads to the formation of $OM_{200-300}$ in cow manure compost. It can be said that the tendency of the temporal change in each metric reflects the decomposition characteristic of organic matter.




**(a) Operation time and $LOI_{200}/LOI_{600}$** **(b) $(LOI_{300}-LOI_{200})/LOI_{600}$ and $LOI_{200}/LOI_{600}$**

**Fig. 4 Relations between operation time and $(LOI_{300}-LOI_{200})/LOI_{600}$ with $LOI_{200}/LOI_{600}$**

## CONCLUSION

Our laboratory experiments examined the validities of $LOI_{300}/LOI_{600}$ and $(LOI_{300}-LOI_{200})/LOI_{600}$ metric in evaluating changes in the state of organic matter present in paddy soil, andosol, and cow manure compost. Temporal changes in $LOI_{300}/LOI_{600}$ metric were not observed, while $(LOI_{300}-LOI_{200})/LOI_{600}$ metric had a strong correlation with operation time. This reinforced the idea that $(LOI_{300}-LOI_{200})/LOI_{600}$ metric can represent the decomposition of organic matter, either in littoral sediments or agricultural soils, including compost materials. Furthermore, we found out that $LOI_{200}/LOI_{600}$ metric had a strong correlation with $(LOI_{300}-LOI_{200})/LOI_{600}$, suggesting that this matric can be used as a simpler proxy for $(LOI_{300}-LOI_{200})/LOI_{600}$ metric. Finally, the proposed metrics, i.e., $(LOI_{300}-LOI_{200})/LOI_{600}$ and $LOI_{200}/LOI_{600}$, were used to identify the decomposition and humification of organic matter. For example, the humification of organic matter caused a decrease in $LOI_{200}/LOI_{600}$ metric and an increase in $(LOI_{300}-LOI_{200})/LOI_{600}$ metric. The tendency of the temporal change in each metric reflected the decomposition characteristic of organic matter.

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