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Letter to the Editor

Loss on Ignition-Based Indices for Evaluating Organic Matter Characteristics of Littoral Sediments

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ABSTRACT

Understanding organic matter characteristics (*e.g.*, amount and state) of sediments is necessary for evaluating both the sediment and water environments; however, methods that have been proposed to investigate these characteristics are relatively expensive. This study attempted to evaluate the organic matter characteristics of littoral sediments by employing solely the loss on ignition (LOI) method, which is the most economical and accessible method in developing countries. Different types of sediments were first oven dried at 100 °C and then continuously burned at 200, 300, 400, 500, and 600 °C for 4 h at each temperature. The mass LOI at each temperature was calculated and compared with the 100 °C oven-dried weight. Our results suggested that the mass LOI across the temperature range of 200–300 °C ($LOI_{200-300}$):mass LOI at 600 °C (LOI_{600}) ratio ($LOI_{200-300}/LOI_{600}$) could represent different types of sediments as well as the organic matter:organic C ratio. Compared with the sediments unaffected by wastewater, the sediments that were more influenced by the inflow of domestic wastewater had a higher $LOI_{200-300}/LOI_{600}$. Interestingly, $LOI_{200-300}/LOI_{600}$ could also represent changes in the organic matter characteristics as a function of organic matter decomposition in sediments. In conclusion, the organic matter characteristics of littoral sediments could be evaluated using solely the LOI method, particularly with $LOI_{200-300}/LOI_{600}$.

Key Words: C loss, decomposition, mass loss on ignition, organic matter state, sediment type

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The organic matter present in littoral sediments plays an important role in marine ecosystems and can deteriorate the marine environment upon its decomposition. Aller (1980) reported that the decomposition of organic matter within sediments consumes oxidants (*e.g.*, Mn^{4+}, Fe^{3+}, and SO_4^{2-}) and releases reduced substances (*e.g.*, Mn^{2+}, Fe^{2+}, and S^{2-}). The reduced substances cause a decrease in the redox potential of both sediments and the water at sea bottom. Consequently, the water at sea bottom becomes anoxic and the bioactivity therein is inhibited. Hence, understanding the organic matter characteristics (*e.g.*, amount and state) of sediments is necessary for evaluating both the sediment and water environments.

Several methods have been proposed for evaluating the amount of organic matter in terrestrial soils and littoral sediments. The Walkley-Black method (Walkley and Black, 1934), a routine wet oxidation method, has been used to determine the amount of organic matter from organic carbon (C) content using a conversion factor (Broadbent, 1953; Heanes, 1984; Swift, 1996; Kerven *et al.*, 2000). Loss on ignition (LOI), a dry combustion method, is a simple and economical method that has been used for the past few decades to directly determine the amount of organic matter (Cambardella *et al.*, 2001; Konen *et al.*, 2002). Unfortunately, it provides a crude estimate and there is inherent difficulty in determining the optimal temperatures and durations for the combustion (Ben-Dor and Banin, 1989; Abella and Zimmer, 2007). Many researchers, including Salehi *et al.* (2011), refined the LOI method to provide a more accurate determination of organic matter content in terrestrial soils. The automated CHNS analysis, involving gas chromatographic analysis, has been used to determine the elemental contents (*i.e.*, C, H, N, and S) with high precision and less time (Sharp, 1974; Telek and Marshall, 1974; Hirota and Szyper, 1975). However, sample pretreatment (*e.g.*, acidification of the sample) is required for separating organic and inorganic C from the sample. Unfortunately, the

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acidification of the sample causes errors in the obtained results because other components besides inorganic C are also removed (Roberts *et al.*, 1973; Dean, 1974).

The amount and state (decomposition degree) of organic matter can be identified using the above mentioned methods. Bader (1954) and Nissenbaum and Kaplan (1972) showed that the C:N ratio increases during organic matter decomposition due to the production of the fulvic and humic acids. It has also been reported that the soil organic C (SOC):soil organic matter (SOM) ratio decreases with increasing depth in terrestrial soils (Périé and Ouimet, 2008; Bianchi *et al.*, 2008; Wang *et al.*, 2013). In other words, different organic matter states can be identified using the SOC:SOM ratio. With the advancements in technology, it is now possible to accurately analyze the organic matter characteristics, based on molecular weight measurements, thermogravimetric analysis, and spectrometric analysis (Cuypers *et al.*, 2002; Hong *et al.*, 2010). However, these analyses involve a high cost, limiting the analyses to a small number of samples. To the best of our knowledge, currently, both C:N and SOC:SOM ratios are widely used. This suggests that the LOI method should be combined with the CHNS analysis in order to examine the organic matter characteristics. As reviewed earlier, many researchers have refined the LOI method for obtaining a more accurate determination of organic matter content. Therefore, we did not aim to propose a more accurate LOI method, but examined the potential of using solely the LOI method to represent different types of littoral sediments affected/unaffected by the inflow of domestic wastewater. Another objective was to present an explanation for proposing indices based on the ignition characteristics of the littoral sediments using the LOI method, in order to examine the organic matter state in sediments. The LOI method is the most economical method that is suitable for analyzing a larger number of samples with the advantage of time and cost savings, does not require a measurement expert, and is an accessible method in developing countries.

We collected surface seafloor sediments (0–40 cm) from two coastal regions in the Hiroshima Prefecture (Japan). One is the Fukuyama inner harbor (34°28′50″ N, 133°22′55″ E), an enclosed coastal region strongly influenced by the inflow of domestic wastewater. The wastewater from a combined sewer system discharges into the harbor approximately 60 times a year (around 25 000 m^3 d^{-1} during low rainfall and 250 000 m^3 d^{-1} during high rainfall). As such, the sediment deposited in this harbor (FS) mainly comprised the organic matter derived from the wastewater. The *n*-hexane-extracted substance content of FS at 0.3 and 1.3 km from the wastewater outlet was approximately 20 and 10 mg g^{-1}, respectively. The FS at 0.3 km from the wastewater outlet was collected and used in this study. Strong decomposition of organic matter occurs within FS, creating malodorous gases, such as hydrogen sulfide (H_2S) and ammonia, as byproducts. The other coastal region is the Kure Bay (34°13′34″ N, 132°29′41″ E), a coastal region that has no direct inflow of wastewater. The sediment deposited in this bay (KS) mainly comprised the organic matter derived from plankton decay and the KS used in this study had an *n*-hexane-extracted substance content of around 0.7 mg g^{-1}. The FS and KS had specifically different characteristics (affected and unaffected by the inflow of domestic wastewater), which allowed us to examine the potential of using solely the LOI method to study the types and the organic matter characteristics of littoral sediments. Sediments were collected from five stations throughout each of the two coastal regions with an HR-type core sampler (Cat. No. 5172, Rigo, Japan) and an Ekman-Berge bottom sampler (Cat. No. 5114-B, Rigo, Japan). A total of 34 FS samples and 17 KS samples were analyzed. The sediments collected using the Ekman-Berge bottom sampler were placed in a clean plastic bucket and then transported to the laboratory under ambient temperature. The FS samples were passed through a 2-mm sieve to remove coarse debris and other large terrestrial deposits. The sediments were stored in a room at 25 ± 1 °C, and homogenized before use. For the sediments collected using the HR-type core sampler, the sediment core was sliced every 20 mm.

To examine the potential of using solely the LOI method for evaluating the decomposition of organic matter within sediments, approximately 250 mL of FS was placed in a 500-mL plastic bottle, and then tap water was added to the sediment. The bottles were placed into a 40-L container filled with tap water and taken out of the container for sediment quality analysis at days 7, 10, 14, 22, and 28. Details of the experimental procedures can be found in Touch *et al.* (2015).

Prior to its use in the LOI tests, the sediment was oven dried at 100 ± 5 °C for more than 24 h and the dry weight was measured. A temperature of 50 °C has usually been used for drying samples before determination of organic matter content. The drying of sediments at 100 ± 5 °C in this study allowed us to determine the water content of the sediments. Another reason for using 100 ± 5 °C was that only a small amount of organic matter in the sediment burned at 50–100 °C and this organic matter had less than 2% C. An