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# Investigating Factors Affecting Stability of Volcanic Ash Soil Aggregates under Heat

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**Abstract:** Volcanic ash soil aggregates can be disaggregated using heat under wet conditions. This study aimed to investigate factors affecting the disaggregation of volcanic ash soil aggregates in a field with organic cattle manure (M plot) and a field with chemical fertilizer (F plot) that were exposed to heat. The two-step wet sieving method, in which aggregates were sieved twice at different water temperatures for different times, was used to investigate the disaggregation caused by heat. It was found that increasing the temperature during the second sieve was more effective in disaggregating aggregates than extending the second-step sieve time. When the water temperature was increased to 80 °C, macroaggregates became more vulnerable, especially those in the F plot. The total carbon (TC) remaining in the soil aggregates was also measured after sieving. Although the TC content in aggregates decreased after sieving, there was only a minor relationship between decreasing TC content and the degree of disaggregation. This suggests that aggregates were not disaggregated by eluting binding agents containing carbon contents, but by partial breakage of the binding agent and/or the peeling of particles with binding agents from the aggregates.

**Keywords:** aggregate stability; temperature dependence; wet sieving; mechanical disaggregation; vulnerability index; volcanic ash soils; andosols

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## 1. Introduction

Physical disinfection methods using heat energy have high potential as alternatives to the commonly used methyl bromide [1]. Therefore, it is important to investigate the temperature dependence of aggregate stability under wet conditions. However, few studies have reported on the effect of increased temperature on this stability. While the proportion of stable aggregates decreased when the water temperature used for wet sieving increased from 20 to 30 °C, the cause of this stability reduction was not identified [2]. Existing methods, such as standard wet sieving (e.g., [3,4]), may not be suitable for evaluating the temperature dependence of aggregate stability because they can only evaluate water resistance at a constant temperature of approximately 20 °C. Thus, a two-step wet sieving procedure was proposed as a modification of the standard method to investigate the temperature dependence of the aggregate stability [5]. In the two-step method, soil aggregates were first wet-sieved at 20 °C to obtain water-stable aggregates at that temperature, and each water-stable aggregate fraction of a certain size was then wet-sieved again at 20, 40, 60, and 80 °C [5]. It was found that aggregates greater than 0.25 mm, referred to as macroaggregates, were more vulnerable to increasing water temperature than those smaller than 0.25 mm, referred to as microaggregates, regardless of fertilization history. When the water temperature increased to 80 °C during the second wet sieve, the aggregates in the field with long-term organic manure were more stable than those in the field with long-term chemical fertilizer. This study showed that aggregate stability was temperature dependent, but it could not address the mechanism for the decrease in aggregate stability with increasing temperature. Aggregates also further disaggregated to some extent when they were re-sieved at 20 °C during the second step of the two-step method [5]. Thus, soil aggregates disaggregated due not only to increasing water temperature, but also by mechanical forces (shaking during sieving). To further evaluate the temperature effect on aggregate stability, the disaggregation should be separated by that caused by heat and that by mechanical force. The degree of disaggregation due to heat can only be quantified by removing the effect of mechanical forces from the entire disaggregation.

The polysaccharide content per unit weight of dry microaggregates in the soil suspension after the second wet-sieving was greater than that in macroaggregates, while a larger proportion of the latter disaggregated compared to the microaggregates after the second sieving at a given temperature [5]. Measuring soil polysaccharide content is insufficient for identifying the cause of disaggregation with temperature. The binding agents of macroaggregates (e.g., plant roots and fungal hyphae) contain not only polysaccharides, but also other types of organic matter. Measuring total carbon (TC) content after wet sieving may allow for identification of the cause of disaggregation due to temperature. TC has been linked to soil aggregate formation because organic matter plays a critical role in the maintenance of soil physical properties, such as soil structures [6]. Soil organic carbon (SOC) was a positive collation between SOC and aggregate associated-C [7], and SOC was present in 250–2000 μm size-class aggregates [8]. Based on these statements, greater carbon content loss from macroaggregates would be able to be observed after the sieving because macroaggregates disaggregated more than microaggregates [5]. However, the role of TC in the stability of aggregates of different sizes and compositions with respect to temperature is not yet understood.

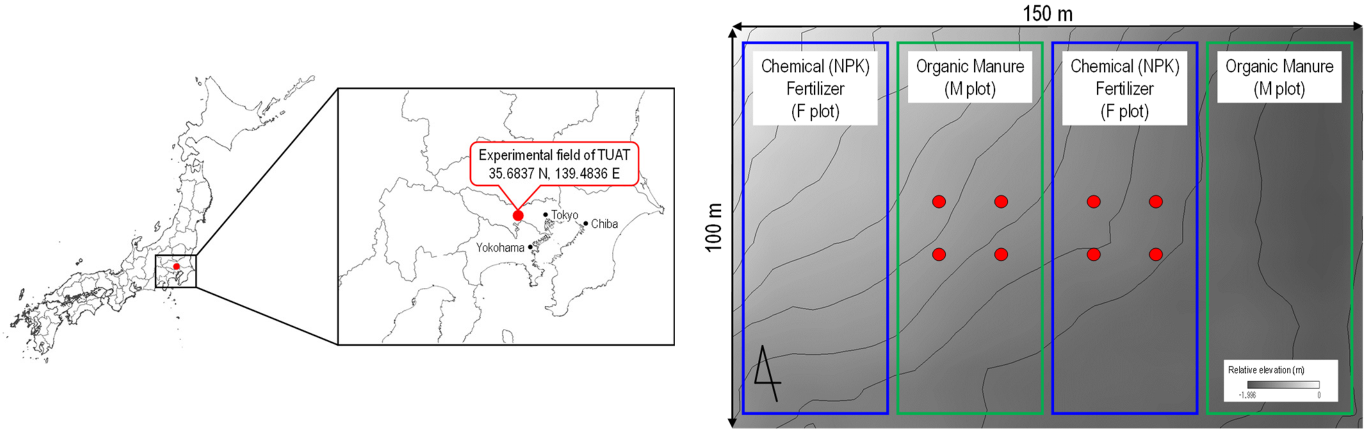
Depending on whether soil is under wet or dry condition, mechanisms of disaggregation due to heat may be different. Increasing the temperature under wet condition promoted the disaggregation of volcanic ash soil aggregates [5]. Under dry condition, studies showed that heat during slash-and-burn could increase or decrease aggregate stability, e.g., [9,10]. Regardless of soil water condition, increasing the temperature may thus alter the aggregate stability. Mechanisms of such phenomena are, however, still not well understood, especially under wet condition. Clarifying the degree and mechanisms of disaggregation due to heat under wet condition can contribute to promote physical disinfection methods based on heat and to manage upland fields appropriately after physical disinfection.

This study aimed to investigate the causes of disaggregation of volcanic ash soil aggregates in fields that have different fertilization histories using the two-step wet sieving method. To achieve this objective, the sieve time of the second step was varied and a new coefficient, referred to as the vulnerability index $R_{st(Tw,t)}$, was introduced to distinguish mechanical disaggregation from that by heat. The TC content of the soil aggregates after two-step wet sieving was then measured to investigate the cause of disaggregation by heat.

## 2. Materials and Methods

### 2.1. Soil Sampling Site Description

Well-aggregated volcanic ash soil, called Andosol [11], was sampled from the experimental field of the Tokyo University of Agriculture and Technology (TUAT) (35.6837° N, 139.4836° E) (Figure 1a). Andosols cover approximately 0.84% (124 million hectares) of the Earth's ice-free surface. It is one of the most common soils found in cultivated lands in Japan [12]. This experimental field had been under fertilizer management since 1997 by the TUAT Field Science Center when aggregate samples were collected. The field was divided into organic manure (cattle manure) and chemical fertilization (NPK fertilizer) areas (Figure 1b). The mean annual air temperature is 15.4 °C with an average annual rainfall of 1598.9 mm according to data from the Automated Meteorological Data Acquisition System of the Japan Meteorological Agency near the study site for 1991–2020 [13]. Winter wheat, maize, and soybeans were rotated in the field. The field was plowed to a shallow depth before each planting. Cattle manure application areas were called M plots, whereas NPK fertilizer application areas were called F plots (Figure 1b). Soil samples were collected from the locations denoted by the red dots in Figure 1b from both the M and F plots. As there was a gentle slope from the northwest corner to the southeast corner (Figure 1b), soil samples were collected from the central parts of each plot to eliminate the effect of elevation differences. Disturbed soil samples were collected at a depth of 5–10 cm from each sampling location to avoid the surface layers where various disturbances and residues associated with agricultural practices were evident.




(a) (b)

**Figure 1.** (**a**) The location of the experimental field of Tokyo University of Agriculture and Technology (TUAT) in Japan and (**b**) sampling site description. The gray scale contour lines represent the relative elevation. Sections surrounded by the blue solid line represent chemical fertilizer plots (F plot), while sections surrounded by the green solid line represent organic manure plots (M plot). Closed red circles denote the sampling locations.

Plant residues were removed from the soil samples using a 4000-µm mesh sieve. For each plot, all soil samples collected from the four sampling locations were mixed well to create a composite sample. Each composite soil sample was divided into three parts for aggregate analysis in triplicate. The samples were not air-dried. Therefore, they were not broken by slaking during wet sieving. The soil samples were stored in plastic bags and refrigerated at 3 °C after sampling. The soil contained 57.68% sand (2–0.02 mm), 33.04% silt (0.02–0.002 mm), and 9.28% clay (less than 0.002 mm). The soil collected from plots M and F had pH values of 6.4 ± 0.07 and 5.7 ± 0.07, respectively.

### 2.2. *Assessment of Disaggregation Due to Heat*

#### 2.2.1. Two-Step Wet Sieving Method

The standard wet sieving method (e.g., [3,4]) has been widely used to determine aggregate stability in water. A two-step wet sieving method was proposed to evaluate the temperature dependence of the aggregate stability by modifying the standard method [5]. Aggregates in six size classes were first obtained using the standard wet sieving method, where aggregates were sieved at 20 °C for 40 min. The six size classes corresponded to those greater than 2000 (>2000), 1000–2000, 500–1000, 250–500, 100–250, and those smaller than 100 (<100) µm. Aggregates in each class, except for those <100 µm, were wet-sieved again under a given, usually increased, temperature (the second step of the two-step process). The detailed procedure of the two-step wet sieving method was summarized in our previous study [5]. In this study, this method was used with different "sieving times" during the second step to evaluate whether the degree of disaggregation was affected by sieve time. In this study, the water temperature of the second sieving of the two-step method, $T_w$, was set to 20, 40, and 80 °C, and three different sieving times were used: half (20 min), the same (40 min), and twice (80 min) the standard sieve time. The dry weight of the aggregates remaining on each sieve was measured after oven drying at 105 °C for 24 h. All experiments were performed in triplicate, average values were used for further evaluation, and the data were evaluated using statistical analyses at $p < 0.05$, with the R software.

#### 2.2.2. Assessment of the Effect of Water Temperature and Sieve Time

The proportion of aggregates remaining on each sieve after the second step was computed for each size class, as follows:

$$K^{i}_{st(T_w,t)} = \frac{W^{i'}_{(T_w,t)}}{W^{i}_{(T_w,t)}} \quad (1)$$

where $W^{i'}_{(T_w,t)}$ is the dry weight of the aggregates remaining after the second-step sieve; $W^{i}_{(T_w,t)}$ is the dry weight of aggregates in the aggregate size, $i$ (μm), after the first-step sieve; $T_W$ is the water temperature of the second-step sieve (°C); and $t$ is the sieve time of the second step (min). $K^{i}_{st(T_w,\ t)}$ represents the stability of the soil aggregates with increasing water temperature. A similar concept, referred to as the "proportional coefficient of each aggregate size", was also proposed, in which the weight of aggregates in each aggregate size after dry sieving is divided by the total weight of the initial soil [14].

To evaluate the degree of disaggregation due to heat only, $K^{i}_{st(T_w,\ t)}$ was standardized with the results of wet-sieving at 20 °C for each sieve time, which represented disaggregation due to mechanical forces only. These standardized values were defined as $R^{i}_{st(T_w,\ t)}$ as follows:

$$R^{i}_{st(T_w,\ t)} = \frac{K^{i}_{st(T_w,t)}}{K^{i}_{st(20,t)}} \quad (2)$$

where $K^{i}_{st(20,\ t)}$ is $K^{i}_{st(T_w,\ t)}$ obtained at 20 °C from the second step for a given sieve time, $t$. This ratio is referred to as the vulnerability index in the remainder of this study.

#### 2.2.3. Analysis of Total Carbon Content of Aggregates in Each Size Class

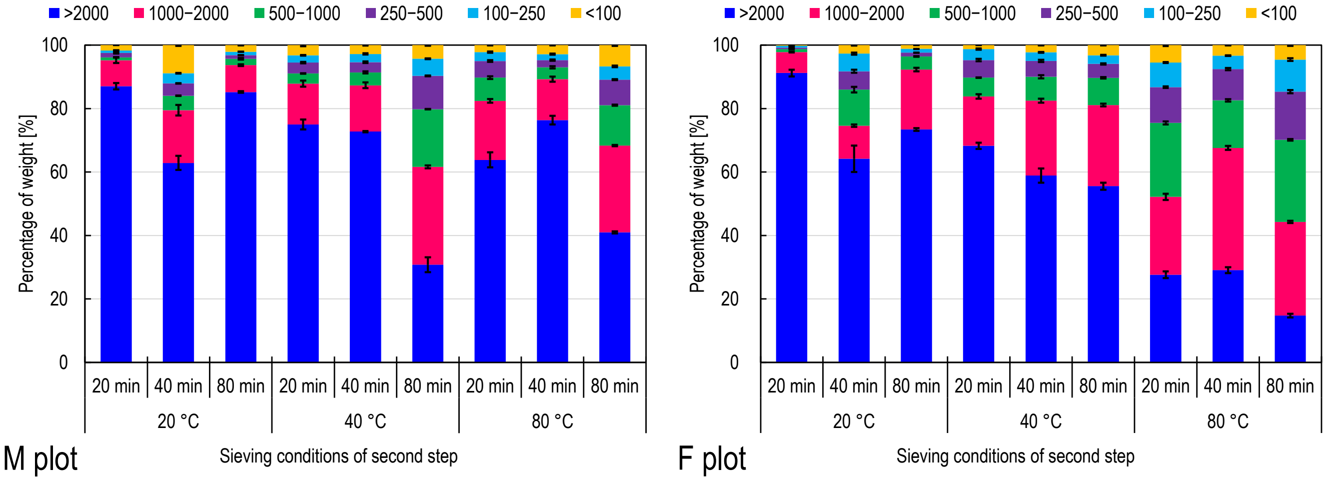
To measure TC content, soil aggregates in each size class before and after the second sieving were oven-dried for 24 h at 105 °C. The samples were then powdered using a mortar and pestle. Approximately 20 mg of the powdered samples were analyzed to obtain the TC content using an NC analyzer (NC-TR22, SUMIGRAPH). All experiments were performed in triplicate, the average values were used for further evaluation, and the data were evaluated using statistical analyses at $p < 0.05$, with the R software.

## 3. Results and Discussion

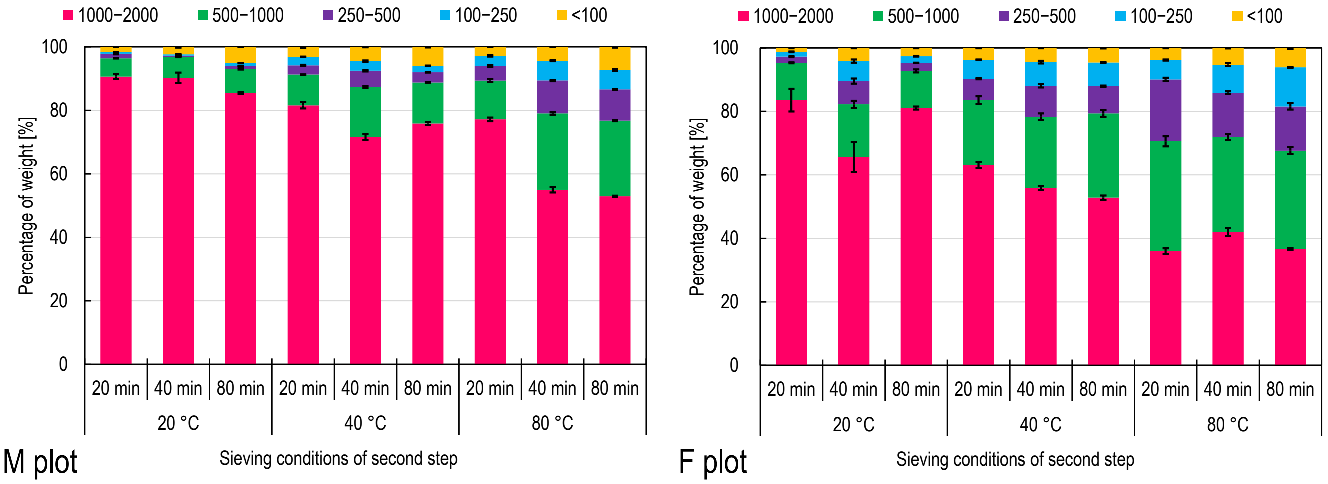
### *3.1. Factors for Disaggregation*

#### 3.1.1. Aggregate Size Distribution under Different Sieving Conditions

Figure 2 shows the aggregate size distributions under different sieving conditions for the M and F plots. While disaggregation was generally enhanced by increasing the water temperature during the second sieve, the proportions of the aggregates remaining after the second sieve increased as the aggregate size decreased, regardless of fertilizer application (Figure 2a–e). The M plot aggregates in all size fractions, except for the 100–250 μm fraction, had greater proportions than those of the F plot aggregates (Figure 2a–e), indicating that the latter were more vulnerable to temperature increases. The M plot aggregates were more stable under heat because the soils fertilized with organic manure had higher carbon contents than those fertilized with chemical fertilizers. Soil carbon can strongly bind to soil particles and form soil aggregates (e.g., [15,16]). The application of organic manure induces microorganisms to produce polysaccharides, which are possible binding agents for soil particles (e.g., [17,18]).




(**a**)

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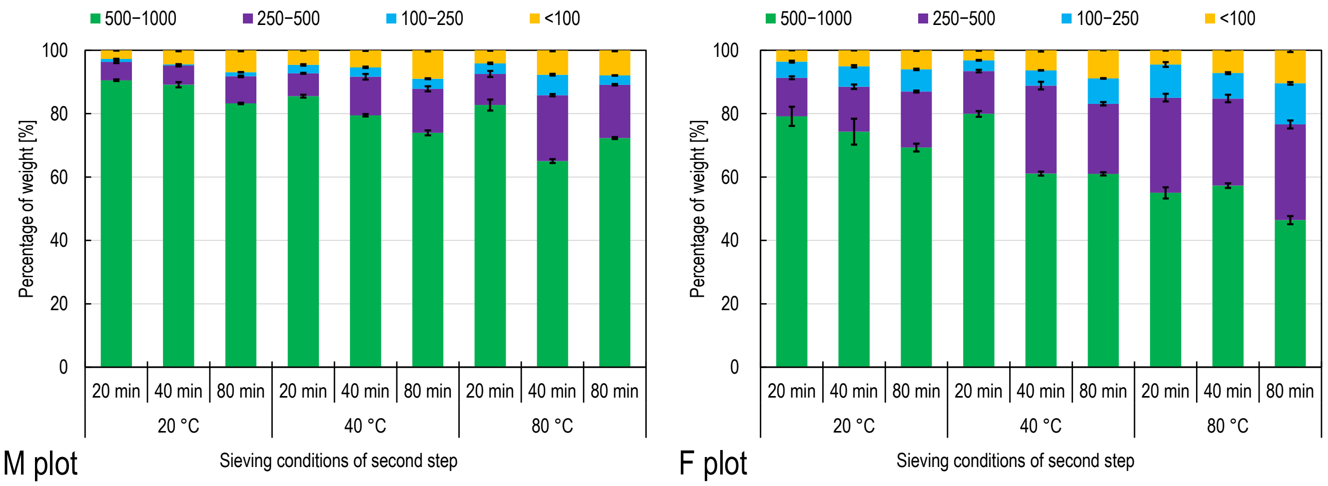
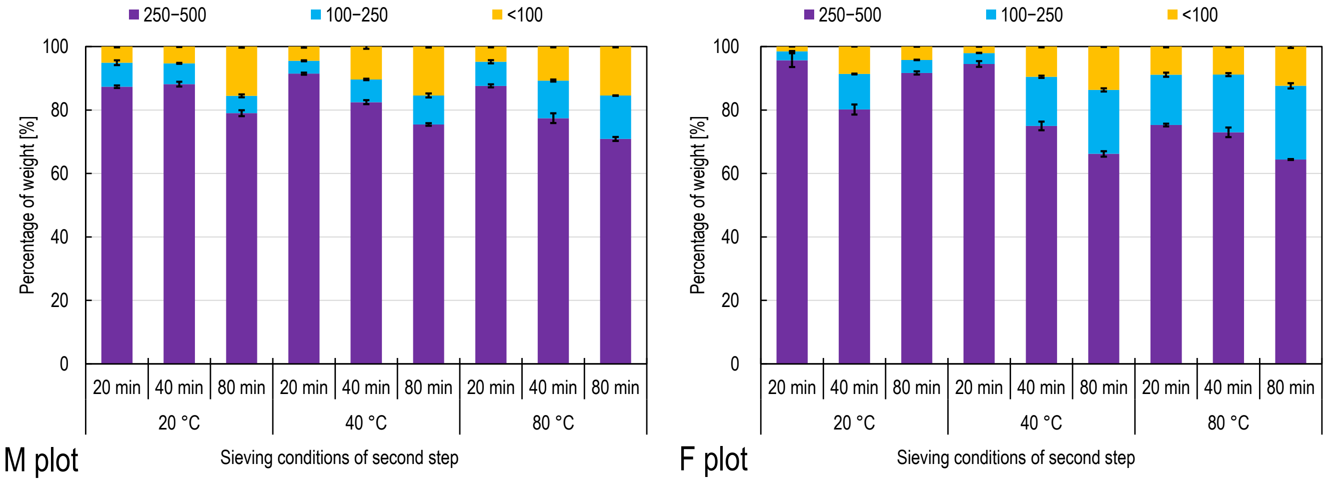



(**c**)

**Figure 2.** cont.

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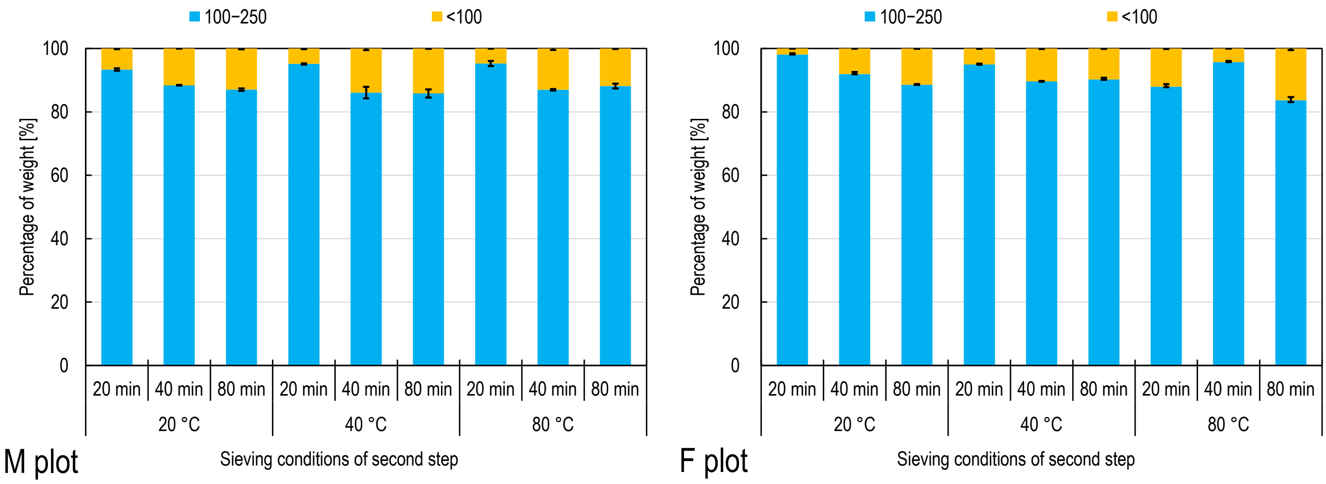



(**e**)

**Figure 2.** Aggregate size distributions after the second sieve during the two-step wet sieving for half (20 min), the same (40 min), and twice (80 min) the standard sieve time and at a given temperature for each aggregate size class obtained with the standard wet sieving: (**a**) >2000 μm, (**b**) 1000–2000 μm, (**c**) 500–1000 μm, (**d**) 250–500 μm, and (**e**) <250 μm. Temperatures of 20, 40, and 80 °C were used. Vertical bars show the standard deviations (S.D.) of means. S.D. bars may not be visible for small S.D.s.

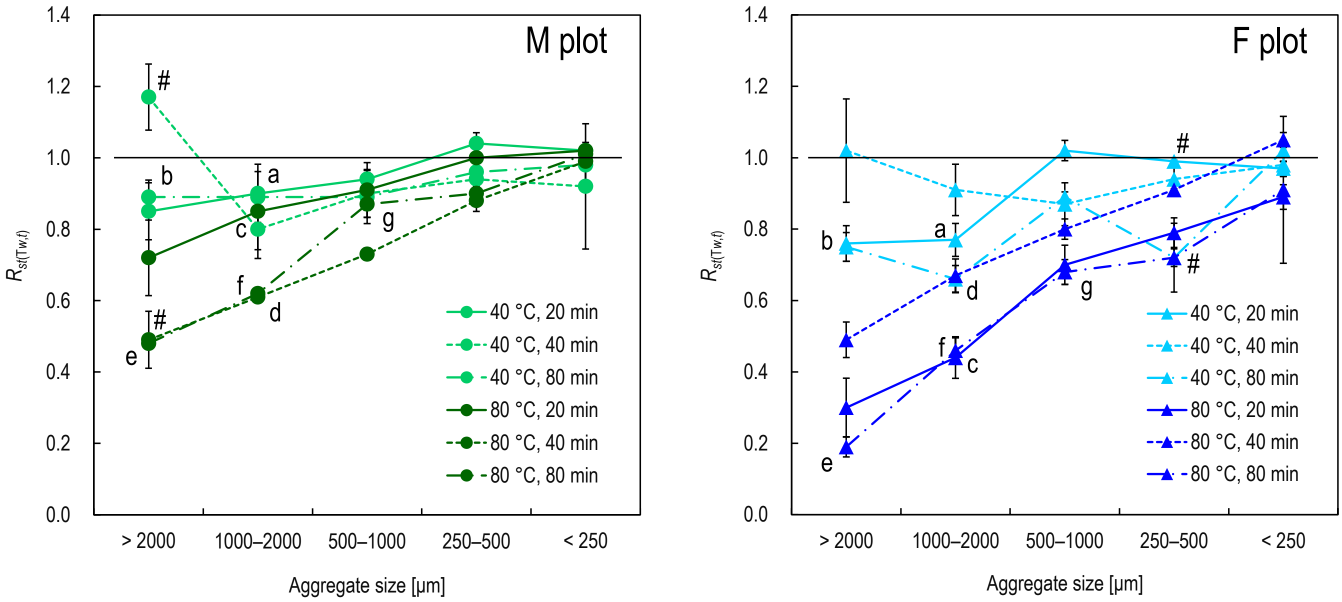
Smaller aggregates (i.e., microaggregates (<250 μm)) were more stable than their larger counterparts (i.e., macroaggregates (>250 μm)) regardless of fertilization management. This may be attributed to differences in the binding agents between macro and microaggregates [17,19]. The microaggregates appeared to be strongly bound.

The results showed that disaggregation was enhanced by extending the sieve time during the second sieving of the two-step method. Macroaggregates revealed greater disaggregation with extended sieve times than that of microaggregates. Macroaggregates also showed higher disruption with increasing temperatures than microaggregates [20]. When increasing the sieve time, soil aggregates in the M plot were more stable than those in the F plot. In contrast, in the F plot, disaggregation increased by approximately 20% when the sieve time increased from 20 to 80 min. For aggregates smaller than 2000 μm in both plots, disaggregation was slightly enhanced by extending the sieve time from 20 to 80 min. Approximately 90% of the microaggregates in both plots remained, regardless of increases in the sieve time and water temperature during the second sieving.

3.1.2. Assessment of Disaggregation Due to Heat Only

As shown in Figure 2, some disaggregation occurred in all aggregate size classes when the second step of sieving was performed at 20 °C, even after 20 min. This disaggregation is considered to be due to mechanical forces during sieving. By comparing the proportion of disaggregation at a given temperature and sieve time with that at 20 °C, we can separate the disaggregation due to heat only. A vulnerability index was used in this study, whereby the ratio of the disaggregation degree at 20 °C to a given temperature was determined.

Figure 3 shows that, in general, the $R_{st(40,t)}$ values are closer to unity than the $R_{st(80,t)}$ values regardless of fertilization history for all the sieve times. This result is expected, since when the water temperature increased, disaggregation was enhanced. When the water temperature of 40 °C was used for the second sieve, the difference in the $R_{st(40,t)}$ values between the aggregates in the M plot and those in the F plot was not significant for all the aggregate size classes, except for the fractions greater than 1000 µm. When the 80 min sieve time was used, the $R_{st(40,80)}$ values for the F plot were smaller than those of the M plot, indicating that increasing the sieve time for the aggregates in the F plot likely promoted disaggregation due to heat.




(**a**) (**b**)

**Figure 3.** Vulnerability index, $R_{st(Tw,t)}$, for each aggregate size fraction. Vertical bars show the standard deviations (S.D.) of means. S.D. bars may not be visible for small S.D.s. Box (**a**) shows indexes of the M plot, and those of the F plot are depicted in (**b**). In both figures, water temperatures at 40 °C are indicated by light colors, while deep colors in the plots show results obtained for a water temperature of 80 °C. The sieve time for 20, 40, and 80 min are shown as solid lines, dotted lines, and dashed-dotted lines, respectively. In the figures, the number sign (#) indicates a significant difference ($p < 0.05$) between the different water temperatures under the same sieve time in the same plot, while lowercase letters show the significant difference ($p < 0.05$) between the M and F plots under the same sieving conditions.

When 80 °C water was used during the second sieve, there was a clear decreasing trend in $R_{st(80,t)}$ with the aggregate size, regardless of fertilization history (Figure 3). The aggregates in the F plot had much smaller $R_{st(80,t)}$ values than those in the M plot, except when the second sieve time was 40 min. For the aggregates in the F plot, the $R_{st(80,80)}$ values approximated 0.2, indicating that the disaggregation of 80% of aggregates greater than 2000 µm did not occur due to mechanical forces, but instead due to heat. This demonstrates their vulnerability to temperature increases. In contrast, for the aggregates in the M plot, the $R_{st(80,80)}$ values were approximately 0.5, indicating that aggregates greater than 2000 µm were considerably more resilient to heat. These results generally support studies showing that soil aggregates in fields where organic manure had been applied were more stable than those from fields with chemical fertilizer (e.g., [5,15,16]).

These results show that temperature increase under wet condition would enhance disaggregation, regardless of temperature and its duration. Soil management, such as organic manure application, to enhance soil aggregate formation is therefore necessary after upland field soil is disinfected by heat.

### 3.2. Total Carbon (TC) Contents in Soil Aggregates

Measuring the total carbon (TC) content after wet sieving may clearly reveal the cause of disaggregation due to temperature. Figure 4 shows the TC content in each aggregate size fraction after the second sieve under different sieving conditions. The TC contents generally decreased as the aggregate size became smaller among the results with the same sieving temperature but different sieving times. Thus, the larger aggregates contained more carbon than the smaller aggregates after the second sieve. The aggregates in the M plot revealed approximately 2% higher TC contents than those in the F plot for all aggregate size fractions, regardless of the sieving temperature and time. While the TC contents were generally higher when the second sieve was performed at 40 °C, the overall results obtained for different sieving temperatures showed that there was no clear trend. Although a number of studies stated that a positive relation between increasing organic matters in soils and soil aggregate stability (e.g., [7,8,14–17,20–25]) and total organic carbon was known to decrease with increasing temperature [20], no obvious trend was observed between disaggregation and decreasing TC content in aggregates after the wet sieving.

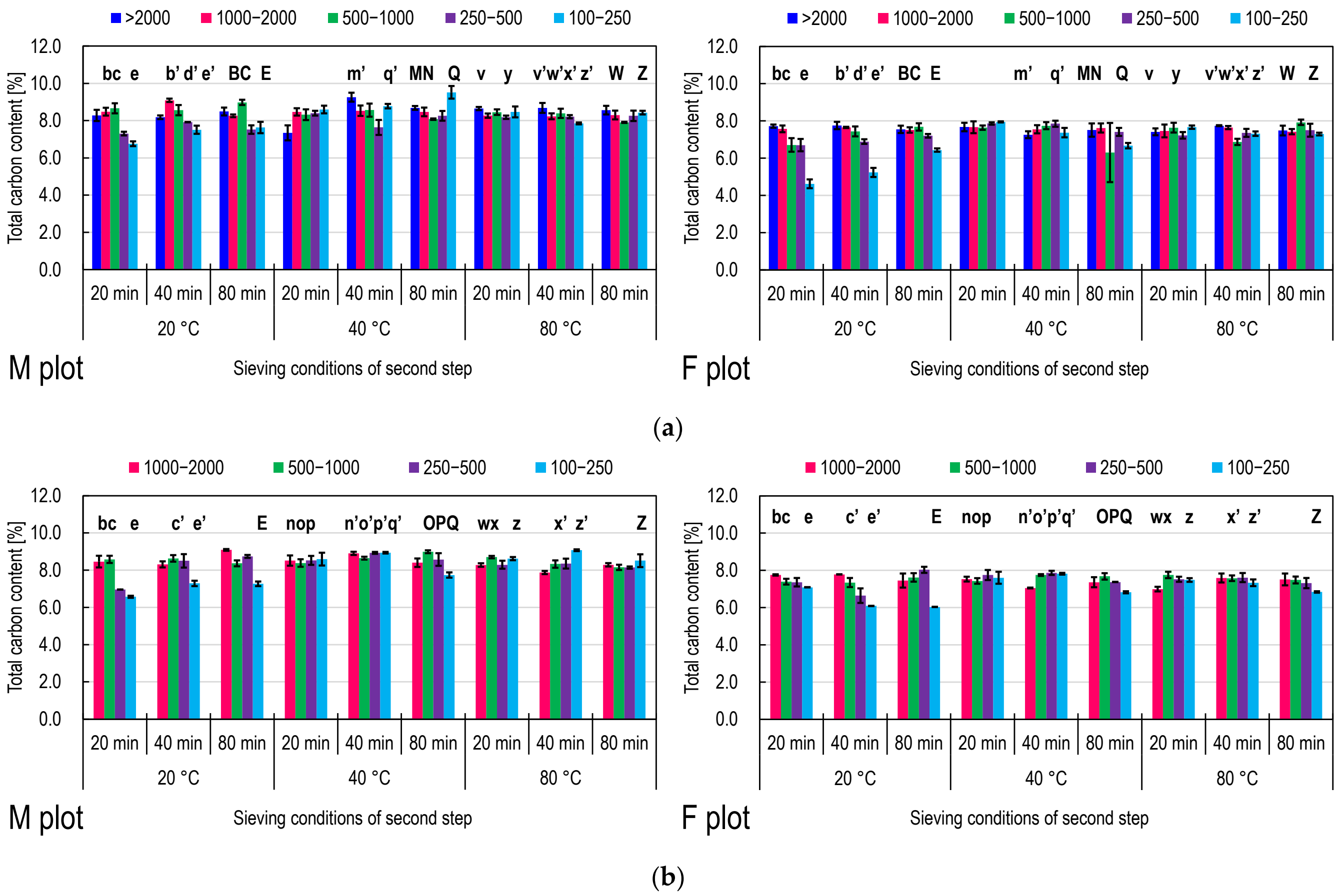
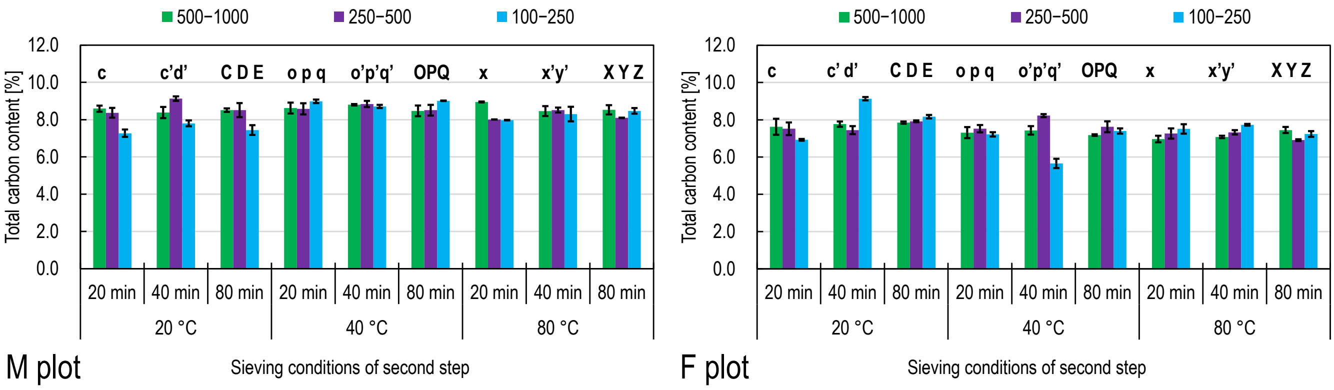



**Figure 4.** cont.

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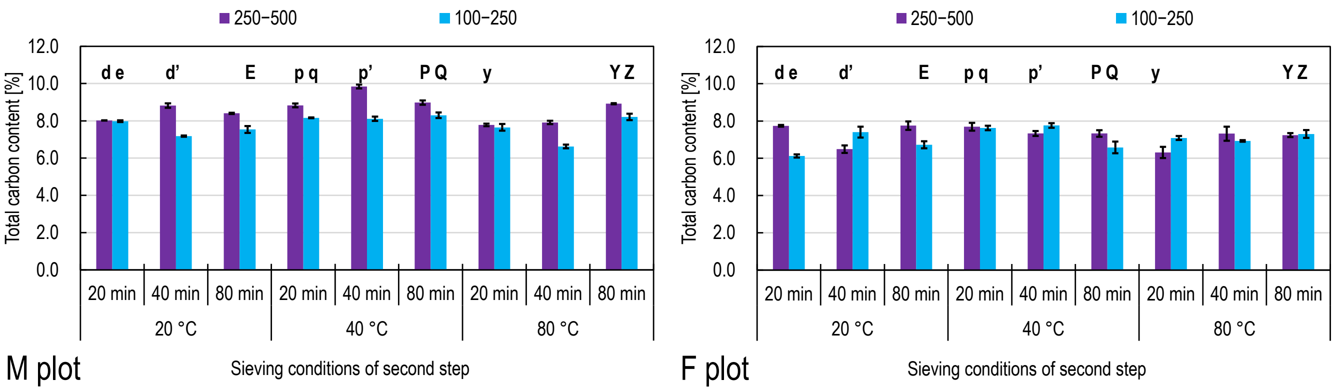


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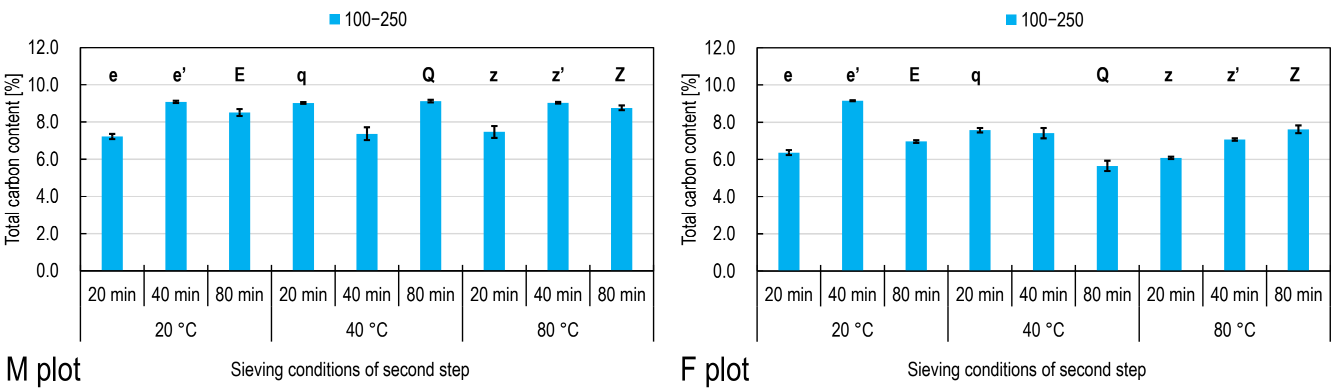


**(e)**

**Figure 4.** Total carbon content in soil aggregates for each aggregate-size class after two-step wet sieving ($n$ = 3). Vertical bars show the standard deviations (S.D.) of means. S.D. bars may not be visible for small S.D.s. The result obtained for >2000, 1000–2000, 500–1000, 250–500, and <250 µm aggregate size classes are shown in (**a**–**e**), respectively. The graphs of the M plot are shown in the left columns, while those of the F plot are shown in the right columns. The letters in the figure indicate the significant difference ($p < 0.05$) for the same sieve time between the plots. The letters (a, m, v), (b, n, w), (c, o, x), (d, p, y), and (e, q, z) indicate the significant difference ($p < 0.05$) of aggregates size for >2000, 1000–2000, 500–1000, 250–500, and <250 µm, respectively. The lowercase letters, lowercase letters with apostrophes, and capital letters denote the significant difference ($p < 0.05$) of sieve time of the second step for 20, 40, and 80 min, respectively. The letters a–e, m–q, and v–z indicate the water temperature at 20, 40, and 80 °C, respectively.

Our previous study documented that no relation was observed between polysaccharides content in soil suspension after the second sieving and disaggregation rate during the wet sieving [5]. This study was able to demonstrate further that decreasing the TC content in aggregates after the wet sieving was not related to disaggregation during the two-step wet sieving. These results could be summarized in that disaggregation did not occur by losing binding agents, such as plant roots, fungal hyphae, and polysaccharides (e.g., [17]), which are expected to have higher carbon contents, from the aggregates by two-step wet sieving. This means binding agents with higher carbon contents were not lost from soil particles, and instead they lost their ability to bind to the particles. Therefore, disaggregation during two-step wet sieving may have been caused by partial breakage of the binding agent and/or peeling of particles with binding agents from the aggregates.

## 4. Conclusive Remarks

This study investigated the causes of disaggregation using the two-step wet-sieving method. Even when the second step was performed at 20 °C, a certain degree of disaggregation was observed, which relates to disaggregation by mechanical forces. By changing the second-step sieve time, it was possible to evaluate the disaggregation due to heat only by comparing the rate of disaggregation with that obtained at 20 °C. The following conclusions can be drawn from this study:

(1) It was found that increasing the temperature during the second sieve was more effective in disaggregating the aggregates than extending the second-step sieve time for both macro and microaggregates.
(2) When the water temperature was increased to 80 °C, macroaggregates became more vulnerable, especially those with a history of chemical fertilizers.
(3) Disaggregation could have been caused by binding agents not eluting into the soil suspension during two-step wet sieving. Instead, they lost their ability to bind to the particles.

**Author Contributions:** R.S. designed and performed the experiments; R.S. and H.S. analyzed the data; H.T. and Y.K. contributed to the discussion of results and editing of the paper; and R.S. and H.S. wrote the paper. All authors have read and agreed to the published version of the manuscript.

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