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Evaluating Soil Particle Analysis Methods for Tropical Andosols in Indonesia

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ABSTRACT

Choosing the right method to quantify soil particles (sand, silt, clay) is essential to predict and manage soil characteristics in relation to e.g. nutrient or water retention. However, separating silt and clay particles for volcanic soils is difficult. In this study, we investigated the effect of three different soil particle analysis methods, namely the regular pipette method, Na-resin dispersion, and ultrasonication with high energy level (1500 J ml^{-1}) on the distribution of soil particle size in tropical volcanic soils. Ultrasonication with high energy level was founded to be the best method to fully separate silt and clay fraction compared to Na-resin and regular pipette method. In conversion, the dispersion of soil particles using regular pipette method was suboptimal. In the ultrasonication method, a step that is important to achieve full dispersion is pH buffer > 8 in soil suspension. This ultrasonication method was founded to have good correlation with Na-resin dispersion method (R = 0.88). This study suggested that conventional particle size analysis may not fully separate sand, silt, and clay particles correctly in soils rich in amorphous materials and oxides, and we proposed ultrasonication high energy level with pH buffer > 8 method as an alternative method.

Keywords: soil particles, clay, volcanic soils, dispersion

1. INTRODUCTION

Soil particle size analysis is a fundamental technique in soil science. Understanding the distribution of sand, silt, and clay particles is essential for application in many fields, such as agriculture or environment because it provides critical insights into soil texture, structure, and related physical and chemical properties. In the soil particle analysis, dispersion of clay and silt particle is challenging. Clay particles are properties that significantly influence soil behavior due to their fine size (< 2 µm) and high surface area (Uehara & Gillman, 1981), such as cation exchange capacity (Curtin & Rostad, 1997), carbon storage (Six et al., 2002), estimating water content (Nanzyo et al., 1993), and so forth. Due to their important role, quantifying these particles are essential to predict and manage soil.

One of the important steps to quantify soil particles is dispersion of soil aggregates. However, the dispersion of soil aggregated volcanic soils which contains abundant amorphous materials (e.g. short-range order minerals, aluminum-iron oxides) is difficult due to their interaction with organic matter, or irreversible drying forming stable sand and silt size aggregates (Huygens et al., 2005; Nanzyo et al., 1993; Uehara & Gillman, 1981). Conventional methods, such as pipette and hydrometer techniques are widely used in laboratory.

However, some limitation with these methods are incomplete extraction of clay particles and re-flocculation, leading to inaccurate assessment of clay particles, particularly in volcanic soils. Some modifications were reported to be applied to optimize the dispersion process, such as replacing sodium hexametaphosphate with sodium hydroxide (Zhang et al., 2005), increasing the concentration of dispersant, particularly sodium hexametaphosphate (Mishra et al., 2011), or manipulating shaking time (Ogunwole et al., 2001). Some modifications improve the dispersion, but the method specifically for tropical volcanic soils is not yet included in those experiments.

Other modification or methods to separate of silt and clay particles are by using saturated sodium treatment and ultrasonication with high energy level (Asano & Wagai, 2014; Rouiller et al., 1972; Silva et al., 2015). Those authors used either one of those two approaches or both approaches. These methods have been significantly improved the dispersion of soil aggregates, particularly extracting clay particles in Andisols in Japan and Hawaii. The objective of this research is to compare the effectivity of the three soil particle analysis methods, i.e. saturated sodium treatment (Na-resin), ultrasonication with high energy level, and standard pipette method in separating sand, silt, and clay particles in tropical volcanic soils in Indonesia.

2. MATERIAL AND METHODS

2.1 Site description and soil sampling

Soil samples were taken from Mount Tangkuban Perahu and Mount Burangrang regions, West Java, Indonesia. The soils in these regions were derived from basaltic-andesitic (ash) tuff from Holocene to Late Pleistocene activities (BBSDLP, 2017; Dam et al., 1996) and were classified as Andosols and Cambisols (Anindita et al., 2022). These regions have a mean annual temperature 19-21°C and a mean annual precipitation 2000 – 3000 mm per year. The samples were derived from six different locations (NF-y, AG-y, PF-i, AG-i, PF-o, AG-o) representing different parent material (NF-y) and land use (natural/secondary forest: NF-y, PF-i, PF-o; agricultural land: AG-y, AG-i, AG-o) (Anindita et al., 2022). Soils were collected from the 0 – 20, 20 – 40, and subsoil (50 – 70 or 60 – 80) cm depths. These soils were air-dried and sieved to < 2 mm.

2.2 Laboratory analysis

Particle size analysis were conducted to separate three different fractions, i.e. sand, silt, and clay. We determined those fractions following three different methods, i.e. standard pipette method, dispersion using Na^+ resins, and dispersion using high energy of ultrasonic energy levels. The theoritical approach of dispersion silt and clay using Na^+ resin was converted in a practical methods by Rouiller et al. (1972). In principle this method used sonication to separate sand fraction, and Na^+ resin was used to separate silt and clay particles. High energy of ultrasonic dispersion was conducted following Silva et al. (2015) with some modification.

2.2.1 Standard pipette method

In this method, soils were sieved (< 2mm) and weighed (20 gr). To remove $CaCO_3$ and organic matter, 100 ml HCl (0.2 N) were added and followed by 33 ml of H_2O_2 (30%) after 15 minutes, let it rest for a night. In the next day, the samples were put in warm water bath and 66 ml H_2O_2 (30%) were added. In addition, 15 ml H_2O_2 were repeatedly added until the foam formation stopped. Then, put the samples in the hot plate for 15 min at 220°C to break down H_2O_2 that was added. Dilute the content by adding demineralized water until 800 – 1000 ml and waited until the soils were deposited (i.e. clean liquid) which usually takes 1 – 3 days.

The clean liquid was removed by suction and demineralized water was added again (i.e. "washing the soils"). This step was repeated until sufficiently washed (i.e. conductivity is < 400 µS/cm). Furthermore, 100 ml of peptization liquid was added and the samples was shaked for about 18 hours. To measure sand particles, soil samples were sieved using 50 µm sieving to separate sand and silt+clay.

The suspension (> 50 µm) was collected, put in the oven for 12 h at 105°C, and weighed to calculate sand fraction. The silt and clay particles were placed in the 1 L beaker (cylinder) and were shaked for 1 min. The sub-samples were taken with a two-way pipette of 25 ml, transferred to a metal cup, put in the oven at 105°C for 12 h, and weighed to measure silt particles. On the other hand, the cylinder beakers were placed into sedimentation bath (25°C) over one night. Each sample in the cylinder beakers were shaked for 1 min and left the samples for 6 hour 47 minutes in sedimentation bath. After that time, the samples were taken using 2-way pipette sampling, transferred to a metal cup, put in the oven for a night, and weighed to measure clay fraction.

2.2.2 Dispersion using Na-resin

To prepare the saturated Na-resin, 1000 ml of resins (protonated Amberlite IR 120; > 500 µm) were washed 3 x 1 hour with 2 – 3 M HCl by shaking to remove the supernatant. Furthermore, the resins were rinsed using deionized water in the rinsing column until the conductivity of the outflow water is similar to deionized water. Resins were then saturated 3 x 1 hour using 2 - 3 M NaOH by shaking and rinsed them again by HCl.

Soil samples were sieved (< 2 mm), weighed 10 grams, agitated with 200 ml of deionized water overnight. The suspension was sieved through a 50 µm and. Sonication was applied to the collected suspension (> 50 µm) and this step was repeated until clean sand particles were obtained. For the suspension (< 50 µm), few drops of $SrCl_2$ were added to flocculate the suspension. The clear supernatant was removed, and the flocculate was collected, then the flocculate was centrifuged for 10 minutes, and again the clear supernatant was discarded.

Furthermore, 100 ml of Na^+ resin was added to the flocculate. If the dispersion is not sufficient, Na^+ resin was added again, and the samples was shaked overnight. The soil-resin mixture was poured onto 200 µm sieve superimposed on the 50 µm sieve. The dispersed samples (silt and clay) were collected and determined following the standard pipette method. Each particle size fraction is weighed and expressed related to the soil dry weight.

2.2.3 Dispersion using high ultrasonic energy levels

Soils were weighed equivalent to 10 gr dry soil and put them in the 400 ml beaker. To remove organic matter, 50 ml H_2O_2 was added to the samples and gradually added 5-10 ml H_2O_2 until foam formation ceased in a hot water bath. The samples were left until they were settled, then remove the clean supernatant. The samples were transferred into a 400 ml beaker and demineralized water was added until the volume of suspension was about 80 ml. Sodium hexametaphosphate (20 ml, with concentration 0.4408 g L^{-1}) was added and the samples were buffered at pH 8 using sodium hydroxide. Furthermore, ultrasonic with energy level 1500 J ml^{-1} as suggested by (Silva et al., 2015) was applied to disperse the soils. The temperature were maintained below 35°C using ice bath to keep cavitation efficiency (Christensen, 2001). Sand, silt, and clay particles were determined following the procedure standard pipette method. We kept the pH suspension above 8 during the pipetting step.

3. RESULT AND DISCUSSION

The difference of sand, silt, and clay particles using three different methods are presented in Table 1. Most of sand particles were found to be higher using pipette method compared to dispersion using Na-resin and higher ultrasonic energy level. More silt particles were also detected in pipette method than the other two methods. On the other hand, more clay particles was founded using ultrasonic energy level > Na-resin > pipette method.

Furthermore, we also found that by using ultrasonication and Na-resin method, four (i.e. AG-y, PF-i, AG-o, and PF-o) out of six soils were classified to have clay soil texture. NF-y and AG-i were mainly detected to have loam and silty clay loam, respectively. These results indicated that Na-resin and ultrasonic energy levels are more effective on dispersing the macro- and meso- aggregates (53 – 250 µm), and it was also supported by the good correlation between Na-resin and ultrasonic high energy levels (R = 0.88) (Fig. 1).

NF-y soil showed to have lower amount of clay particles in those three methods because the soils in NF-y is still young, as indicated by vitric properties (Anindita et al., 2022). In young soil, most of material still exist as sand and silt sized particles which are closed to the original parent rock fragments. Weathering has not progressed enough to break down minerals into secondary clay minerals. In AG-i soil, ultrasonication method could isolate more clay particles than the other two methods, but less clay particle were detected than other soils. The reason is possibly because the higher Al_o content in this soil compared to other soils (Table 1). Al-oxides can form a strong bond with organic matter and clay enhancing aggregate stability. More energy levels might be required to fully separate silt and clay particles.

Table 1 Soil particle analysis using three different methods (pipette method, Na-resin, and ultrasonic dispersion), pH, and oxalate extracted-aluminum in studied soils

	Pipette method			Na-resin^1			Ultrasonic			pH^1		$\text{Al}_o^{1,2}$
	sand	silt	clay	sand	silt	clay	sand	silt	clay	KCl	H_2O	
					%							g kg^{-1} soil
Primary forest (NF-y)												
0-20	51.1	37.8	12.1	42.8	38.5	18.7	35.6	47.0	17.4	3.9	4.1	3.5
20-40	47.9	39.4	12.9	40.0	40.2	19.8	36.1	46.0	17.9	3.9	4.4	2.3
60-80	40.1	45.6	14.4	29.2	41.3	29.5	25.9	59.5	14.6	4.0	4.3	4.8
Agricultural land (AG-y)												
0-20	18.1	25.5	56.4	8.2	23.6	68.2	2.3	21.7	76.1	4.4	5.1	14.4
20-40	26.8	26.5	46.7	3.1	33.8	63.1	2.3	22.2	75.4	4.4	5.0	16.5
60-80	26.5	33.5	40.1	5.7	32.9	61.4	4.4	27.8	67.8	4.3	4.8	22.3
Pine forest (PF-i)												
0-20	9.5	21.3	69.3	8	24.7	67.3	6.9	23.1	69.9	3.7	4.5	8.1
20-40	6.5	21.9	71.7	6.1	28.4	71.6	3.8	23.2	73.0	3.7	4.4	10.4
70-90	22.9	43.1	34.2	6.4	29.3	64.4	3.3	29.9	66.8	3.6	4.4	13.7
Agricultural land (AG-i)												
0-20	13.2	74.0	12.7	25.0	47.8	27.2	16.4	51.7	31.9	4.7	5.4	31.4
20-40	50.0	35.65	14.4	12.1	52.3	35.6	16.5	46.5	37.0	5.3	5.8	33.3
50-70	66.2	28.1	5.7	12.5	40.4	47.1	10.9	34.2	55.0	5.5	5.9	36.4
Pine forest (PF-o)												
0-20	31.9	46.5	21.5	9.5	35.0	55.5	5.0	29.2	65.9	4.6	4.7	28.5
20-40	28.8	60.0	11.2	10.7	42.9	46.6	6.5	34.2	59.3	4.7	4.9	27.7
60-80	27.5	52.0	20.4	10.8	40.4	48.8	6.4	35.0	58.6	4.4	4.4	25.9
Agricultural land (AG-o)												
0-20	17.1	52.8	30.2	14.5	30.6	54.9	7.0	35.0	58.0	4.5	5.4	17.1
20-40	19.4	45.7	35.0	12.7	37.3	50.0	5.9	32.2	61.8	4.7	5.6	15.6
60-80	10.2	40.5	49.4	4.9	32.3	62.8	3.6	25.5	70.9	4.7	5.8	9.1

[1] Data taken from (Anindita et al., 2022)

[2] Al_o = oxalate-extracted aluminum

A higher amount of clay particles was recovered using ultrasonic treatment, likely due to multiple steps in this method that are considered effective in dispersing soil aggregates. Firstly, organic matter in soils was removed. Organic matter, more specifically microbial and fungal secretions, plays a crucial role in soil aggregation by acting as binding agent, improving soil structure and stabilizing soil aggregates (Lehmann & Kleber, 2015; Totsche et al., 2018).

In soils containing abundant non-crystalline materials, such as Andisols, microaggregates are highly stable. Highly reactive surface of these materials with organic matter can create strong bond with organic matter (i.e. organo-mineral complexes) forming stable microaggregates (Burgeon et al., 2021). As the organic matter was removed, soil aggregates are more susceptible to dispersion. Furthermore, the suspension was buffered at pH above 8. Previous study, Silva et al. (2015) used pH above 9 or 10. The increase of pH suspension during pipetting step was intended to increase the occurence of de-protonation of zero point of net charge (ZPNC). This process will increase net negative charge and reduces electrostatic attraction that can re-aggregate particles (Uehara & Gillman, 1981).

As presented in the Table 1, the ΔpH (pH KCl – pH H_2O) in our studied soils are about 0 to -0.8. The ΔpH can be used to rapidly measure surface charge, where ΔpH < 0 indicates a net positive charge (i.e. more exchangeable acidic cations, ΔpH > 0 denotes a net negative charge, ΔpH ~ 0 indicates that the soil is close to the point of zero charge (Uehara & Gillman, 1981). The increase of pH suspension is an important step since our study showed that the process of dispersion is suboptimal without the increase of pH suspension. Lastly, ultrasonic treatment with high energy level was applied to disperse the samples.

Ultrasonication uses high-frequency sound waves to create cavitation bubbles that exert strong shear forces, breaking down the stable aggregates. Field et al. (2006) suggested that higher energy was required to disperse the strong bond in microaggregates. The energy level, 100 J ml^{-1}, could dispersed only a weak-bound aggregates while a strong bound required at least 1600 J ml^{-1} (Silva et al., 2015). Asano & Wagai (2014) reported a sonication 5000 J ml^{-1} to maximum disperse Andosols in Japan, whereas (Poeplau et al., 2013) used sonication 22 J ml^{-1} to disperse labile macroaggregates of loamy clay and loamy sand soils in an experimental farm in Sweden and Switzerland.

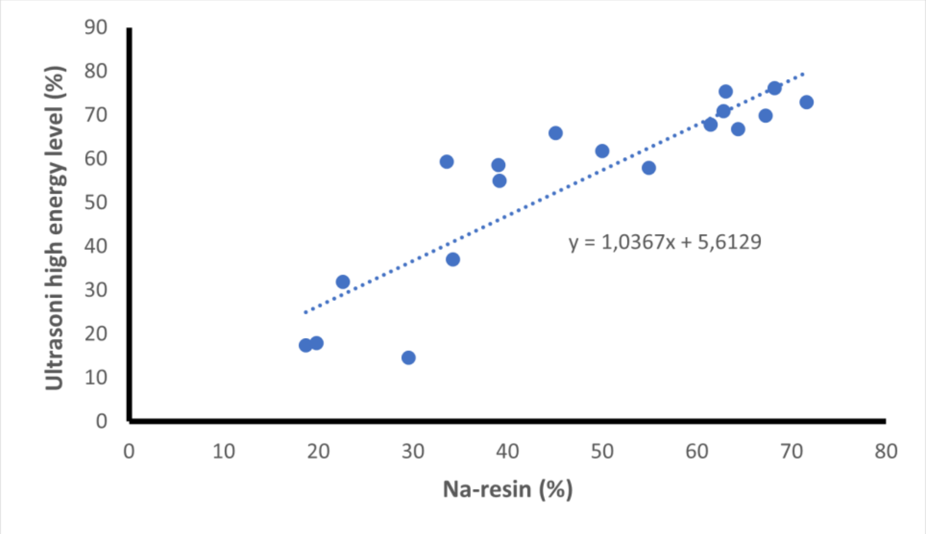

Figure 1. Correlation of clay particles using ultrasonic high energy levels and Na-resin dispersion methods

Thus, the accurate energy level might be different between soils and regions. More investigation, particularly about mineralogical characteristics that affect the stability in relation to ultrasonication need to be conducted. However, we found that the energy level of 1500 J ml^{-1} was sufficient for most of of the studied soils. High energy of ultrasonic treatment have been confirmed to a potential damage of soil surface (Kaiser et al., 2012). Silva et al. (2015) reported that ultrasonication treatment of 1600 J ml^{-1} yielded an “etched” surface or crater-like feature, particularly in sand particles, but it might be considered negligible since most of their soils did not show evidence of damage.

The application of sodium saturation treatment (i.e. Na-resin) to disperse soil aggregates and extract clay particles has been conducted in previous research (Asano &

Wagai, 2014; Delfosse et al., 2005). The idea is that the application sodium ions (Na^+) will replace other cations, e.g. Ca^{2+}, Mg^{2+}, K^+ on soil colloids, leading to dispersion. The dispersion occurs due to reduction of flocculation since Na^+ has a larger hydration radius than Ca^{2+} and Mg^{2+}, and the increase of electrostatic repulsion. In our study, the application of Na-resin method showed a good correlation with ultrasonication high energy level (Fig. 1). Although the Na-resin method recovered less clay particles than ultrasonication with high energy level, the Na-resin method does not require the removal of organic matter from the soil. This is very useful approach to analyze organic matter fractionation where we want to separate soil fractions without removing the organic matter in soils. (Asano & Wagai, 2014) showed a maximum dispersion using sodium saturation treatment followed by ultrasonication (max. 5 kJ ml^{-1}) in allophanic Andisols. However, the damage of particles after using such a high energy needs to be considered. On the other hand, the regular pipette method cannot fully separate sand, silt, and clay particles than the other two method. The reasons are possibly because the pipette method relies on gentle mechanical stirring and chemical dispersant (e.g. sodium hexametaphosphate) that are not fully break down aggregates, particularly those bound by oxides or organic matter, and there is a chance the clay particles to flocculate (re-aggregate) after the dispersion leading to underestimation of clay fraction.

4. CONCLUSION

This research highlights that dispersion using Na-resin and ultrasonic with high energy levels extracted more clay particles, suggesting a better method to quantify clay particles than regular pipette method, particularly in the soils with amorphous materials or oxides, such as Andisols and Oxisols. Combination of pH buffer > 8 and ultrasonication is the best method, but the required energy level might be different between region, hence adjustment is needed. Accurate soil texture analysis is essential for various scientific and practical reason, particularly in soil science, agronomy, environmental studies, and geotechnical engineering. Incomplete clay separation may lead to misclassification of soil types, inaccurate understanding of soil behaviour, or imprecise results in modelling soil development (i.e. soil particles influences water retention, drainage, aeration, fertility). The right method of soil particle analysis ensures precise results and better decision-making in soil research.

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