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The Analysis of the Anticoagulant's Influence and Its Concentration on Clay Shale Dispersion in Hydrometer Testing

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Abstract. Clay shale is a material that has the characteristic of being easily weathered if it is continuously exposed to the air. Based on this reason, further testing is needed for clay shale material to analyze the material characteristics, one of which is using a hydrometer test. In the hydrometer test, an anticoagulant breaks down the particles. Still, when the test was carried out by various laboratories with different types of samples, it would produce varying test results. This study aims to determine the effect of anticoagulant substances and their concentration on clay shale dispersion in the hydrometer test. The material used was clay shale from the Meteseh, Tembalang District, Semarang. This research method used was a hydrometer test based on British Standard 1377 Part 2 1990 and ASTM D7928-17 using seven anticoagulant agents. The results of the study using the British Standard 1377 Part 2 method showed that sodium polyphosphate, sodium pyrophosphate, and sodium hexametaphosphate were most influential in dispersing clay shale particles, which were characterized by a higher passing percentage of 0.042 mm and 0.002 mm when compared to other anticoagulants. Meanwhile, Calgon, sodium carbonate, sodium tripolyphosphate, and sodium silicate did not significantly affect the clay shale dispersion, indicated by the percentage that passed the sieve, which was not very high. Based on the British Standard 1377 Part 2 1990 and ASTM D7928-17 methods, the most optimum concentration in dispersing clay shale for sodium polyphosphate and sodium pyrophosphate is 5%, while for sodium hexametaphosphate is 6%.

Keywords: clay shale, dispersion, anticoagulant, concentration, hydrometer testing

INTRODUCTION

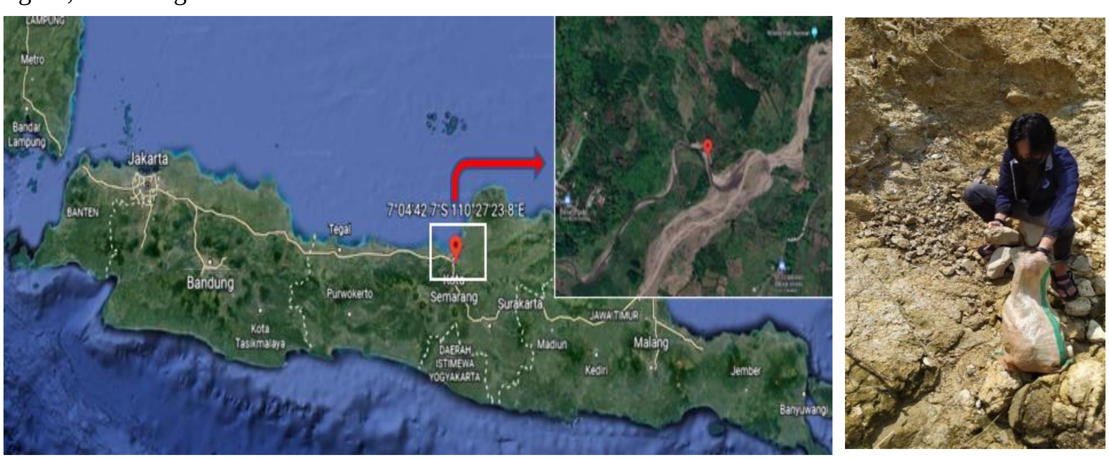
One type of material in Indonesia that frequently poses challenges during construction is clay shale. Clay shale originates from clay minerals and clay rocks [1]. According to Deen (1981), clay shale is a transitional material between soil and rock [2]. Clay shale is known for its susceptibility to weathering when continuously exposed to air. This weathering process can result in a decrease in shear strength, potentially causing landslides in sloped areas or construction sites. Therefore, further testing of clay shale material is required to analyze the characteristics of the material, with one common method being the use of a hydrometer test. The treatment that can be carried out in the hydrometer test is by adding an appropriate concentration of anticoagulant to the existing clay shale. This anticoagulant is used to break up clay shale so that the grain gradation can be analyzed. However, in Indonesia, there needs to be more research on using anticoagulants for clay shale. One of the previous studies regarding clay shale only discussed the effect of clay shale on residual shear strength [3].

Anticoagulant substances are those that prevent particles from clumping together. The concentration of the solution, when mixing anticoagulant substances, is expressed as the ratio of the solvent to the solute, and it plays a critical role in ensuring an effective solution. In hydrometer testing, anticoagulant substances are used as chemical solutions to disperse clumped particles within a soil sample. However, it is important to note that the effectiveness of these substances can vary depending on the type used and the laboratory conducting the tests [4]. ASTM International (2017b) recommends sodium hexametaphosphate with a concentration of 4% as an anticoagulant agent in hydrometer testing [5]. Kaur & Fanourakis (2018) stated that the addition of 7 grams of sodium carbonate to 33 grams of sodium hexametaphosphate produces a substance called Calgon, which can increase the effectiveness of alluvial soil dispersion in Sebokeng Township, Gauteng Province, and black soil in Northwest Province, South Africa [6]. Calgon is also an anticoagulant substance recommended by British Standards (1996) [7]. Emeka (2015) recommends 40 grams of sodium carbonate per 1 liter of distilled water to disperse soil in Ogun State and Kaduna State, Nigeria [8]. Dolzyk & Chmielewska (2014) recommends 3 grams per liter of sodium pyrophosphate as a dispersant agent [9]. Abdulkarim et al. (2021) recommend 29.4 grams of sodium tripolyphosphate per liter of distilled water for dispersing fluvial soils in the Alsatian Upper Rhine Plain, Northeastern France [10]. Kaddah (1975) mentions that 5 grams of sodium polyphosphate per 50 grams of soil per liter of distilled water can increase the dispersion of calcareous soil in Imperial Valley, California [11]. Maharaj & Paige-Green (2013) conducted a hydrometer test using 5 ml of sodium silicate, which resulted in the substance not completely dispersing the soil particles and causing flocculation (sedimentation) [12].

Because there is much literacy from outside Indonesia, more research needs to be carried out in Indonesia. Therefore, this study aims to determine the effect and concentration value of the appropriate anticoagulant substance on clay shale dispersion. The clay shale reviewed in this study was taken from Meteseh Village, Tembalang District, Semarang. The test used was hydrometer testing using British Standard 1377 Part 2 1990 and ASTM D7928-17 methods with various coagulants.

RESEARCH METHODOLOGY

The clay shale material used in this study was taken from Meteseh Village, Tembalang District, Semarang, Central Java. Sampling locations can be seen in Figure 1 (a) with latitude coordinates 7°04'42.7"S and longitude coordinate 110°27'23.8"E. Sampling, as can be seen in Figure 1 (b) around the cliffs near the river, was carried out using a chisel by digging up the surface of the soil where there is clay shale. Based on the sampling method, this sample is a type of disturbed sample. Further, samples taken from the field were tested in the laboratory. The laboratory tests were conducted at the Soil Mechanics Laboratory, Civil Engineering Department, Faculty of Engineering, Universitas Diponegoro, Semarang.



(a) (b)

FIGURE 1. Field Sampling (a) Sampling Locations (*Google Earth*, 2022) [13]; (b) Sampling of disturbed soil samples

The properties index of clay shale was carried out to analyze the physical properties. The test includes the Atterberg limit based on the ASTM D4318-17el standard [14] and Specific gravity based on ASTM D854-02 [15]. The data from each property index is then processed and analyzed to determine the sample type. Soil classification analysis was performed using the USCS and AASTHO Classification Systems.

Before laboratory testing, it is necessary to pre-treat the clay shale samples to prevent weathering of the clay shale samples. This pre-treatment was done by soaking the clay shale samples collected in the field in a water bath before being used for testing. This was done because clay shale will weather quickly when placed in an open place. Clay shale samples from pre-treatment were filtered on sieve No.10. Clay shale samples that passed sieve No. 10 were used for further testing. Clay shale samples that passed sieve No. 10 were weighed according to the requirements of the tests carried out.

Furthermore, hydrometer testing was carried out based on British Standard 1377 Part 2 1990 [7] to obtain the best anticoagulant substance and the optimum concentration of the essence. In addition, hydrometer testing was also carried out based on the ASTM D7928-17 standard [5] to serve as a comparison or a control. The differences in the working methods of these two standards were that in the hydrometer test based on British Standard 1377 Part 2 1990, a wet sieve was carried out, wet filtering of the results of the stirrer sample of clay shale and anticoagulant solution on sieve No. 200, whereas in the hydrometer test according to ASTM D7928-17, a water bath was carried out or in other words soaking the clay shale samples with a solution of anticoagulant for 16 hours before stirring.

There were seven anticoagulant substances used in this study: sodium polyphosphate, sodium pyrophosphate, sodium hexametaphosphate, sodium silicate, sodium carbonate, sodium tripolyphosphate, and Calgon. Each of these substances was dissolved according to the concentration of each with a ratio of 1 liter of distilled water. As an example, for a concentration of 4%, it means that 40 grams of anticoagulant was used, which was dissolved in 1 liter of distilled water.

RESULTS AND DISCUSSION

Soil Properties Index Testing (Physical Properties)

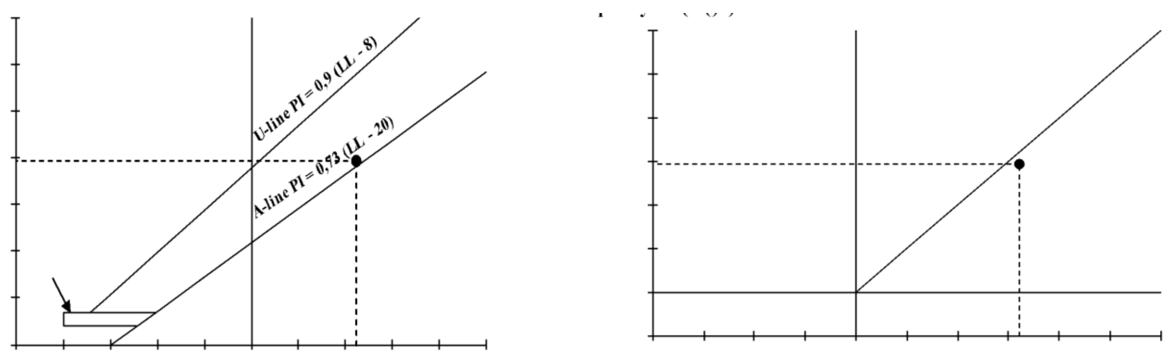
Atterberg limit test for clay shale (oven dried) and clay shale (not oven dried) with test parameters including liquid limit (LL), plastic limit (PL), and plasticity index (PI) can be seen in Table 1. It can be seen that the liquid limit value of clay shale (oven-dried) is 67.52%, whereas the liquid limit value of clay shale (not oven-dried) is 72.21%. Thus, the ratio of oven-dried and not oven-dried Liquid Limit is 0.935. Based on Das & Shoban (2014) [16], clay shale samples can be categorized as inorganic soils if the Liquid Limit ratio is more than 0.75.

Based on Burmeister (1949), who discussed the relationship between soil plasticity values based on the Plasticity Index Classification, if the Plasticity Index (PI) is in the range of 20 – 40, the clay shale samples are classified as soils with high plasticity [17]. The Plasticity Index sample of this study is 39.38 (between 20 – 40), which can be seen in Table 1; it can be categorized as soils with high plasticity. Meanwhile, for the Specific Gravity (Gs) test, the Gs value for the not oven-dried clay shale sample was 2.717. Based on Hardiyatmo (2002) [18], regarding the Classification of Soil Types based on Specific Gravity, the clay shale sample is an Inorganic Clay soil type.

TABLE 1. Atterberg Limit and Specific Gravity Test Results

Variable	*Oven Dried*	*Not Oven Dried*
LL (%)	67.52	72.21
PL (%)	28.22	32.83
PI	39.30	39.38
Gs	-	2.717

Furthermore, the soil classification in this study was also analyzed using the Unified Soil Classification System (USCS) and the American Association of State Highway and Transportation Officials (AASHTO) classification systems. The Liquid Limit (LL) and Plasticity Index (PI) values of the clay shale, not oven-dried sample, were then plotted on the USCS classification system graph [16] as shown in Figure 2 (a). Based on the Atterberg limit test results, when the liquid limit value is 72.21%, a straight line is drawn to meet the plasticity index value of 39.38. The meeting point is above the A-Line and below the U-Line. Then, because the value of the liquid limit (LL) is above 50%, the clay shale sample belongs to the CH group, namely inorganic clay with high plasticity. In addition, the LL and PI values of the clay shale, not oven-dried sample, were plotted on the AASHTO classification system graph [16], as shown in Figure 2 (b). Based on the Atterberg limit test results, the meeting point of the LL and PI values is below the slope due to the PI ≤ LL - 30 so that according to the AASHTO classification system, the clay shale sample belongs to group A-7-5a, namely clay with high plasticity. Based on soil classification carried out using correlation with literature based on Atterberg Limit (ratio of LL and PI) and Specific Gravity (Gs) values, as well as USCS and AASHTO classifications, it can be concluded that the clay shale in this study is an inorganic clay soil with high plasticity. The high content of soil organic matter has a threatening effect on the remains of forest plant material falling to the soil surface [19]. The advantage of organic materials is that they can improve the physical, chemical, and biological properties of soil [20].



(a) (b)

FIGURE 2. Classification System of Soil (a) Unified Soil Classification System (USCS) Plasticity Chart; (b) Association of State Highway and Transportation Officials (AASHTO) Plasticity Chart

Hydrometer Testing

Hydrometer Test according to British Standard 1377 Part 2 1990

a. The Phase-1 test used seven anticoagulant agents (according to British Standard 1377 Part 2 1990)

In the first phase, hydrometer testing based on British Standard 1377 Part 2 1990 was carried out using seven anticoagulant substances, including sodium hexametaphosphate, Calgon, sodium tripolyphosphate, sodium polyphosphate, sodium pyrophosphate, sodium carbonate, and sodium silicate with the same concentration of 4% as much as 250 ml of anticoagulant solution.

Based on the test, it was found that sodium polyphosphate, sodium pyrophosphate, and sodium hexametaphosphate have the highest pass percentage on a sieve diameter of 0.042 mm, in 62%, 58%, and 58% and on a sieve diameter of 0.002 mm, in 43%, 43%, and 31%, respectively. Therefore, it can be concluded that these anticoagulants can break down clay shale particles better than the other four anticoagulants. This is by research from Kaddah (1975) [11] and Dolzyk & Chmielewska (2014) [9], who recommend these anticoagulant substances. Whereas for Calgon, sodium carbonate, sodium tripolyphosphate, and sodium silicate, the percentage of passing the 0.042 mm sieve was not very high, only 50% or less, so it can be concluded that these four substances are not good at breaking down clay shale particles. Due to this reason, the three best anticoagulants were selected, sodium polyphosphate, sodium pyrophosphate, and sodium hexametaphosphate, for use in the next stage of testing with various concentrations. The results of phase 1 testing using seven anticoagulant substances with a concentration of 4% can be seen in Figure 3.

Moreover, Phase-2 hydrometer testing based on British Standard 1377 Part 2 1990 was carried out using the three best anticoagulant substances, sodium hexametaphosphate, sodium pyrophosphate, and sodium polyphosphate with varying concentrations of 2%, 3%, 5%, and 6% to find out which attention is the most optimum on each of these anticoagulant substances in breaking down clay shale particles, which is described as follows.

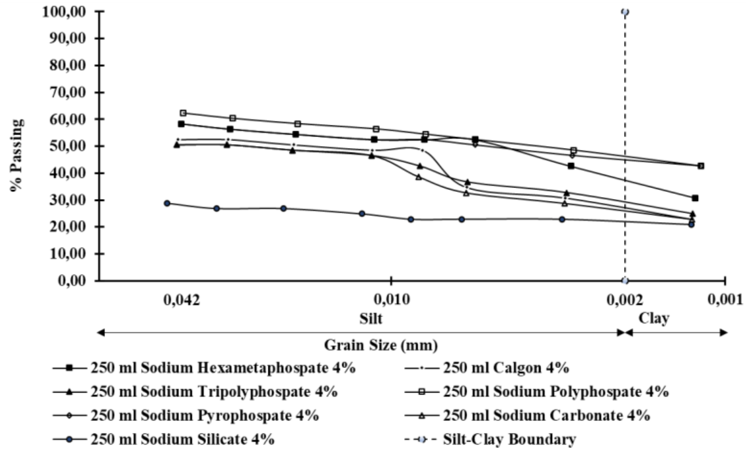


FIGURE 3. Gradation Chart of Clay Shale Grain Size Using British Standard 1377 Part 2 1990 Method Phase-1

b. The Phase-2 test used sodium hexametaphosphate with varying concentrations of anticoagulants (based on British Standard 1377 Part 2 1990).

Based on hydrometer testing using sodium hexametaphosphate as an anticoagulant, it was found that the optimum concentration for sodium hexametaphosphate was 6% because it could produce the highest pass percentage, 66% on a sieve diameter of 0.042 mm and 41% on a sieve diameter of 0.002 mm when compared to other concentrations. The lowest passing percentage for sodium hexametaphosphate occurred at a concentration of 2%, 41% on a sieve diameter of 0.042 mm, and 19% on a sieve diameter of 0.002 mm. The results of stage 2 testing using the anticoagulant substance sodium hexametaphosphate can be seen in Figure 4.

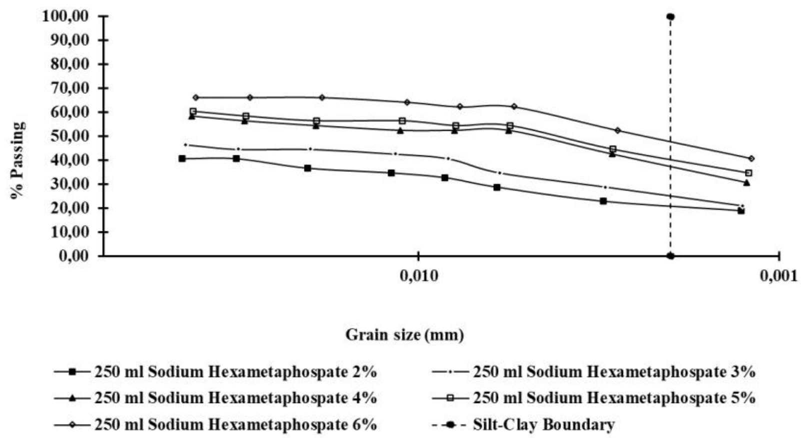


FIGURE 4. Gradation Chart of Clay Shale Granules using British Standard 1377 Part 2 1990 Method using Sodium Hexametaphosphate Phase-2

c. The Phase-2 test used sodium pyrophosphate with varying concentrations of anticoagulants (based on British Standard 1377 Part 2 1990).

Based on the hydrometer test using sodium pyrophosphate as an anticoagulant, it was found that the optimum concentration for sodium pyrophosphate was 5% because it produced the highest passing percentage, 66% on a sieve diameter of 0.042 mm and 47% on a sieve diameter of 0.002 mm when compared to other concentrations. Meanwhile, the lowest passing percentage for sodium pyrophosphate occurred at a concentration of 2%, namely 49% on a sieve diameter of 0.042 mm and 25% on a sieve diameter of 0.002 mm. The results of stage 2 testing using the anticoagulant substance sodium pyrophosphate can be seen in Figure 5.

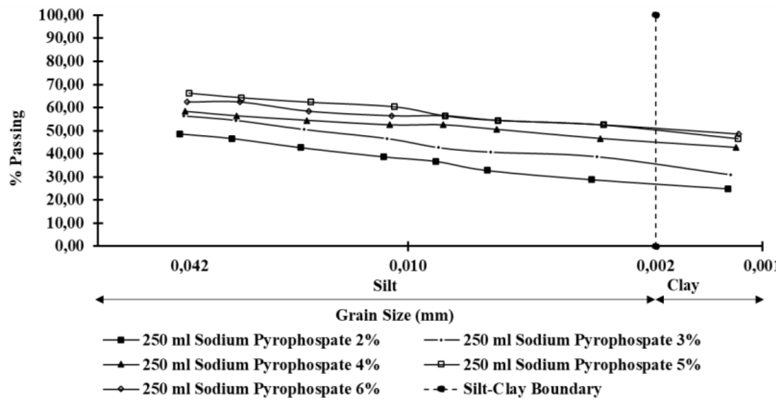


FIGURE 5. Gradation Chart of Clay Shale Granules using British Standard 1377 Part 2 1990 Method using Sodium Pyrophosphate Phase-2

d. The Phase 2 test used sodium polyphosphate with varying concentrations of anticoagulants (based on British Standard 1377 Part 2 1990).

Based on hydrometer testing using sodium polyphosphate anticoagulant, it was found that the optimum concentration for sodium polyphosphate was 5% because it could produce the highest passing percentage, 70% on a sieve diameter of 0.041 mm and 49% on a sieve diameter of 0.002 mm when compared to other concentrations. The lowest passing percentage for sodium polyphosphate occurred at a concentration of 2%, 51% on a sieve diameter of 0.041 mm, and 33% on a sieve diameter of 0.002 mm. The results of stage 2 testing using the anticoagulant substance sodium polyphosphate can be seen in Figure 6.

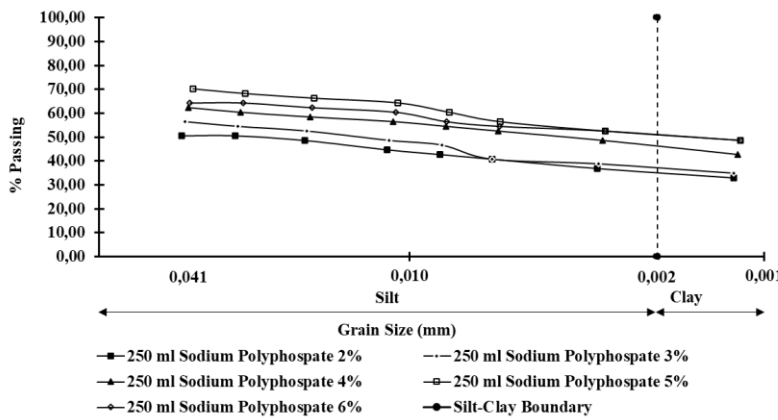


FIGURE 6. Gradation Chart of Clay Shale Granules using British Standard 1377 Part 2 1990 Method using Sodium Polyphosphate Phase-2

Furthermore, based on the hydrometer test stage 2 with the British Standard 1377 Part 2 1990 method, it can be analyzed that the best anticoagulant substances are sodium polyphosphate 5%; sodium pyrophosphate 5 %; and sodium hexametaphosphate 6%. Sodium polyphosphate 5% has a passing percentage of 70% on a sieve diameter of 0.042 mm and 49% on a sieve diameter of 0.002 mm. Meanwhile, sodium pyrophosphate 5 % has a pass percentage of 66% on a filter diameter of 0.042 mm and 47% on a filter diameter of 0.002 mm. Moreover, sodium hexametaphosphate 6% has a passing percentage of 66% on a filter diameter of 0.043 mm and 41% on a filter diameter of 0.002 mm, as shown in Figure 7.

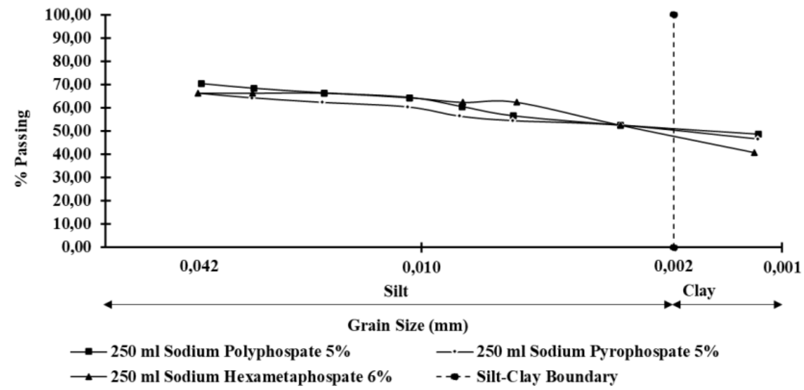


FIGURE 7. Graph of Final Gradation Results of Clay Shale Granules with the British Standard 1377 Part 2 1990 Method Phase-2

Based on the hydrometer test using the British Standard 1377 Part 2 1990 method that has been carried out, the graphs shown in Figures 3 to Figure 7 are graphs of clay shale samples that pass sieve No. 200. Clay soil itself is soil that passes the No. Sieve. 200. To ensure soil classification based on analysis of soil distribution, Table 2 presents data on the weight of the clay shale sample retained on sieve No. 200, which was converted to a retained percentage of 50 grams of clay shale sample. This data was a soil sample tested using the three best anticoagulants and their optimum concentrations: sodium polyphosphate 5%, sodium pyrophosphate 5%, and sodium hexametaphosphate 6%. According to Table 2, it can be concluded that the highest passing percentages are sodium polyphosphate 5%, sodium pyrophosphate 5%, and sodium hexametaphosphate 6%.

TABLE 2. Weight of Clay Shale Sample Retained in Sieve No. 200 (British Standard 1377 Part 2 method 1990)

Anticoagulant Agents	% Concentration	Holding Weight (gram)	% Restrained	% Passed
Sodium Polyphospate	5	9,51	13,52	86,48
Sodium Pyrophosphate	5	10,12	14,22	85,78
Sodium Hexametaphosphate	6	9,17	15,96	84,04

Hydrometer Testing according to ASTM D7928-17

Hydrometer testing based on ASTM D7928-17 was carried out using the three best anticoagulant substances from the hydrometer test based on British Standard 1377 Part 2 1990: sodium polyphosphate, sodium pyrophosphate, and sodium hexametaphosphate. The testing was according to recommendations from ASTM International (2017b) with varying concentrations of 2%, 3%, 4%, 5%, and 6% as controlling or comparison to British Standard 1377 Part 2 1990 method. Based on hydrometer testing using the ASTM D7928-17 method using sodium hexametaphosphate anticoagulant, it was found that the optimum concentration for sodium hexametaphosphate is 6%. This is because it can produce the highest passing percentage of 56% at 0.043 mm sieve diameter and 37% at 0.002 mm sieve diameter compared to other concentrations. The lowest passing rate for sodium hexametaphosphate occurred at a concentration of 2%, 43% on a sieve diameter of 0.043 mm, and 21% on a sieve diameter of 0.002 mm. The results of the ASTM D7928-17 hydrometer test using the anticoagulant sodium hexametaphosphate can be seen in Figure 8.

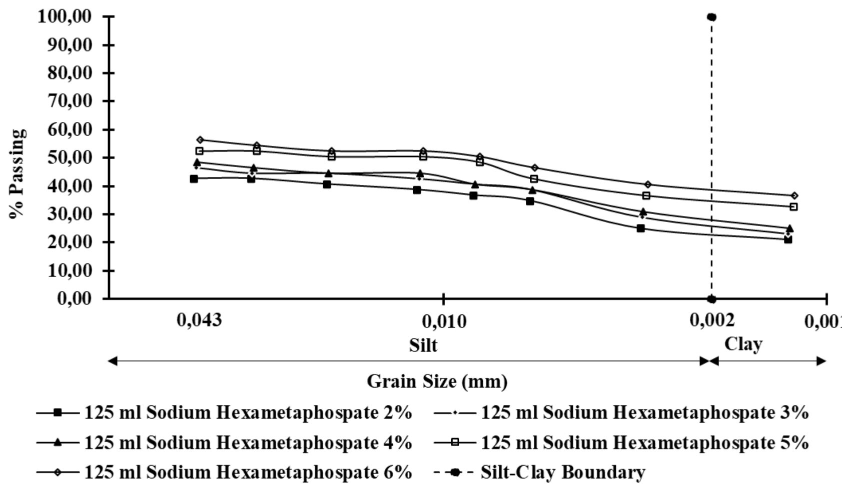


FIGURE 8. Gradation Chart of Clay Shale Granules Using ASTM D7928-17 Method using Sodium Hexametaphosphate

Based on hydrometer testing using the ASTM D7928-17 method using sodium pyrophosphate as an anticoagulant, it was found that the optimum concentration for sodium pyrophosphate was 5%. This is because it produced the highest passing percentage, 56% on a diameter of 0.043 mm and 45% on a diameter of 0.002 mm, compared to other concentrations. Meanwhile, the lowest passing rate for sodium pyrophosphate occurred at a concentration of 2%, 45% on a sieve diameter of 0.043 mm, and 31% on a sieve diameter of 0.002 mm. The results of the ASTM D7928-17 hydrometer test using sodium pyrophosphate can be seen in Figure 9.

Moreover, based on the hydrometer test using the ASTM D7928-17 method using sodium polyphosphate as an anticoagulant, it was found that the optimum concentration for sodium polyphosphate was 5%. This is because it produced the highest pass percentage, 58% on a diameter of 0.043 mm and 45% on a diameter of 0.002 mm, compared to other concentrations. Meanwhile, the lowest passing rate for sodium polyphosphate occurred at a concentration of 2%, 47% on a sieve diameter of 0.043 mm, and 29% on a sieve diameter of 0.002 mm. The results of the ASTM D7928-17 hydrometer test using sodium polyphosphate can be seen in Figure 10.

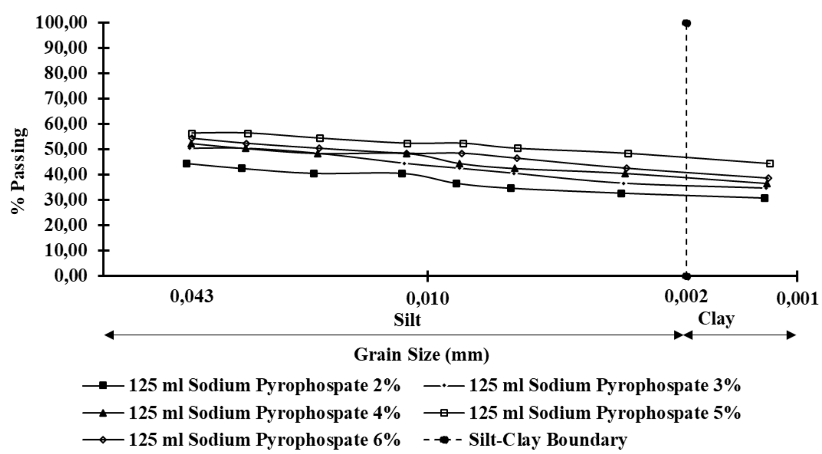


FIGURE 9. Gradation Chart of Clay Shale Granules Using ASTM D7928-17 Method using Sodium Pyrophosphate

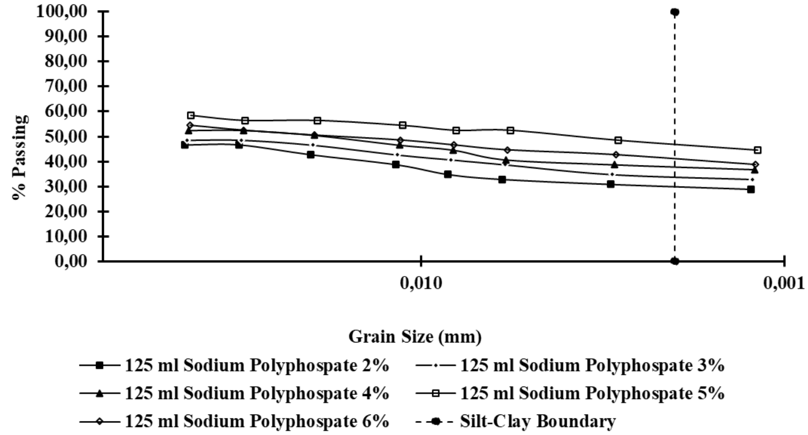


FIGURE 10. Gradation Chart of Clay Shale Granules Using ASTM D7928-17 Method using Sodium Polyphosphate

Based on the hydrometer test using the ASTM D7928-17 method, the results of the hydrometer test based on ASTM D7928-17 are similar and tend to be close to the development of the hydrometer test based on British Standard 1377 Part 2 1990. Therefore, the hydrometer test carried out in this study is valid. However, the passing percentage value in the hydrometer test using the British Standard 1377 Part 2 1990 method is higher than in the hydrometer test using the ASTM D7928-17 method. Thus, it can be concluded that in hydrometer testing on clay shale soil, it is recommended to use the British Standard method 1377 Part 2 1990 to get a better pass percentage and particle distribution. A comparison of the hydrometer test results using the British Standard 1377 Part 2 1990 method and ASTM D7928-17 method can be seen in Table 3.

TABLE 3. Comparison of hydrometer test results using British Standard 1377 Part 2 1990 method and ASTM D7928-17 method

Anticoagulant Agents	% Passed 0.043 Sieve Diameter	
	British Standard 1377 Part 2 1990	ASTM D7928-17
Sodium Polyphospate 5%	70	58
Sodium Pyrophosphate 5%	66	56
Sodium Hexametaphosphate 6%	66	56

CONCLUSION

Sodium polyphosphate, sodium pyrophosphate, and sodium hexametaphosphate are the most influential anticoagulant agents in dispersing clay shale particles, which were characterized by passing percentages of 0.042 mm and 0.002 mm higher when compared to the other four anticoagulants. Unlike Calgon, sodium carbonate, sodium tripolyphosphate, and sodium silicate did not have much effect on dispersing clay shale particles, which were characterized by passing percentages of 0.042 mm and 0.002 mm, which were not very high. The optimum concentration for sodium polyphosphate and sodium pyrophosphate is 5% because it can produce the highest passing percentage in both the British Standard 1377 Part 2 1990 and ASTM D7928-17 methods when compared to other concentrations. The optimum concentration for sodium hexametaphosphate is 6% because it can produce the highest passing percentage both in the British Standard 1377 Part 2 1990 and ASTM D7928-17 methods when compared to other concentrations. Therefore, the sequence of the best anticoagulants for dispersing clay shale is sodium polyphosphate 5%, sodium pyrophosphate 5%, and sodium hexametaphosphate 6% in the hydrometer test using the British Standard 1377 Part 2 1990 (British Standards, 1996) and ASTM D7928-17 (ASTM International, 2017b).

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