Standardizing Methods for Ball Mill Disaggregation of Slakable Rock and Fine-Grained Soil

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Geotechnical laboratory tests such as grain size analysis, Atterberg limits, residual strength, and swell-consolidation testing on reconstituted specimens require disaggregating a geomaterial sample into its constituent particles. Specimen preparation typically involves hand processing samples with a mortar and rubber-tipped pestle until they pass a designated sieve size. Ball milling is an alternative to hand processing and has the potential to expedite the preparation process and result in more complete disaggregation, leading to more accurate test results. For ball milling to become a validated specimen preparation method and gain wide acceptance it must be standardized. The research presented here seeks to progress the effort towards standardization by evaluating the effects of ball size, ball material, and milling duration on geomaterials including high plasticity clay, elastic silt, shale, claystone, and clayey sandstone. The research also presents results of ball milling a fine aggregate (concrete sand) to assess the potential for grain pulverization in each milling scenario. Ball mill performance is material dependent, but for all materials evaluated in this study, ball milling induced a higher degree of disaggregation than hand processing in all scenarios. Grain pulverization from metal ball milling scenarios was evident, especially in materials with higher sand contents. Parameters obtained from ball milling were normalized by hand processed results, and the trends suggest that ball mill processing causes a greater increase in liquid limit than plastic limit compared to hand processing.

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prepared by

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Research and Development Office
Science and Technology Program

(Final) Report ST-2024-21100-01

Standardizing Methods for Ball Mill Disaggregation of Slakable Rock and Fine-Grained Soil

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Executive Summary

Reclamation has numerous facilities where the local geology contains rock such as shale or claystone that naturally degrade in the presence of water or atmosphere. These types of rock, termed slakable, are notoriously difficult to sample and cannot be tested using typical rock-type physical property testing. Instead, these materials are often processed into a soil-like sample and subjected to conventional soil testing. Fine-grained soils are also common on Reclamation facilities, and when in a desiccated state, can be equally difficult to sample and prepare for laboratory testing. The clay minerals that are present in fine-grained soils and slakable rocks are often susceptible to swelling upon wetting and low shear strength zones adversely affecting slope stability, both of which can lead to challenging geotechnical design considerations. Tests such as grain size analysis, Atterberg limits, residual strength, and swell-consolidation testing on reconstituted specimens all require a disaggregated sample for specimen preparation. These tests are commonly performed for Reclamation laboratory testing programs and within the geotechnical industry.

Disaggregating slakable rock and desiccated soil into their constituent particles prior to specimen preparation typically involves hand processing (i.e., manually grinding) samples with a mortar and rubber-tipped pestle until they pass a designated sieve size. Higher degrees of cementation and/or induration in the sample material require a more significant hand processing effort to fully disaggregate. Furthermore, different operators can vary the disaggregation effort imparted on the sample leading to inconsistencies in the test results, and hand processing may never fully disaggregate the clay-size particles, particularly if the material is indurated or desiccated in-situ. A lack of complete disaggregation can lead to unconservative estimates of the material's physical and engineering properties (e.g., lower clay fraction, lower Liquid Limit (LL)). In addition, hand processing is both time consuming and labor intensive.

Ball milling is an alternative to hand processing and has the potential to expedite the preparation process and result in more complete and repeatable disaggregation, leading to more accurate test results. In order for ball milling to become a validated specimen preparation method and gain wide acceptance it must be standardized. This research sought to develop a standardized ball milling approach for processing slakeable rock and desiccated/indurated soil. The approach to standardization will determine an ideal combination of milling parameters to adequately disaggregate samples with a standard level of disaggregation energy imparted on the sample. Standardization will allow for more widespread adoption of the technique, potentially leading to better estimation of critical material properties. This process will occur first within Reclamation through both the TSC Geotechnical Lab and FCCO and Provo Regional Lab project partners.

This study presents the gradation and plasticity results of samples processed with various ball milling combinations on six natural materials and a concrete sand. As a general trend, finer gradations and higher plasticity values were obtained from increased milling time, increased ball size, and the use of a metal ball vs. rubber ball. For almost all cases, the 12 millimeter (mm) metal ball provides the highest degree of disaggregation; however, as evidenced in the concrete sand and clayey sandstone test results, the metal ball causes significant alteration to the materials natural particle size distribution. While petrographic analysis has not been performed to characterize alteration at the particle level, it is generally inferred that the sand particles are being pulverized into smaller particles, and that some complex clay mineral shapes are being altered as well.

The ball mill outperformed hand processing in obtaining a higher percent fine fraction for almost all milling combinations, even in instances where the ball mill failed to fully disaggregate larger pieces of sample. Ball mill performance is material dependent, but for all materials evaluated in this study, ball milling shifted the LL, Plasticity Index (PI), and clay fraction results outside of the within-laboratory repeatability limit for hand-processed samples. A single ball mill standard process may not exist, and a suitable method for a particular material may require a selection process to identify the right combination of ball material, size, and milling time. The presence of sand in natural materials complicates this selection process as metal balls are clearly altering the composition of the sand grains and should not be used for sandy materials.

The results of this study indicate that ball milling is effective at disaggregating a wide variety of natural geomaterials, but additional testing is required before a standard can be developed. Future research will incorporate petrographic analysis of ball milled and hand-processed samples both pre- and post-processing to confirm the presence of grain alteration. In addition, both hand-processed and milled samples will be tested in triplicate to assess the repeatability of ball milling. To reduce the effects of natural sample variation, several control materials with well-known properties will be evaluated. Additional natural materials will also be evaluated to improve the relationships developed in this study. Longer milling durations will be assessed for harder materials, and the effects of sample volume within the cannister will also be studied.

The background, methods, results, and discussion of this research are presented in Appendix A. The manuscript in Appendix A, entitled "Evaluating Methods for Ball Mill Disaggregation of Slakable Rock and Fine-Grained Soil," has been accepted for publication in ASTM's *Geotechnical Testing Journal* and has also been selected for presentation at ASTM's 2025 Symposium on Shear Testing of Soils. This manuscript documents all aspects of the research from this project. Specific details of facility names were not included in the manuscript. A follow up research proposal has been submitted to S&T during the FY24 call for proposals to pursue the remainder of this research and push for standardization.

Appendix A

Evaluating Methods for Ball Mill Disaggregation of Slakable Rock and Fine-Grained Soil

Richard G. Bearce, Ph.D., P.E.[1], Robert V. Rinehart, Ph.D., P.E.[2]

ABSTRACT

Geotechnical laboratory tests such as grain size analysis, Atterberg limits, and residual strength and swell-consolidation testing on remolded specimens require disaggregating a sample into its constituent particles. Specimen preparation typically involves hand processing samples with a mortar and rubber-tipped pestle until they pass a designated sieve size. Ball milling is an alternative to hand processing and has the potential to expedite the preparation process and result in more complete disaggregation, leading to more accurate test results. For ball milling to become a validated specimen preparation method and gain wide acceptance it must be standardized. The research presented here seeks progress the effort to standardization by evaluating the effects of ball size, ball material, and milling duration on geomaterials including high plasticity clay, elastic silt, shale, claystone, and clayey sandstone. The research also presents results of ball milling a fine aggregate (concrete sand) to assess the potential for grain pulverization in each milling scenario. Ball mill performance is material dependent, but for all materials evaluated in this study, ball milling induced a higher degree of disaggregation than hand processing in all scenarios. Grain pulverization from metal ball milling scenarios was

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evident, especially in materials with higher sand contents. Parameters obtained from ball milling were normalized by hand processed results, and the trends suggest that ball mill processing causes a greater increase in liquid limit than plastic limit compared to hand processing.

Keywords

Ball milling, gradation, liquid limit, plasticity index, clay fraction, sample preparation

Introduction

The clay minerals that are present in fine-grained soils and slakable rocks are often susceptible to swelling upon wetting and low shear strength zones adversely affecting slope stability, both of which can lead to challenging geotechnical design considerations. It is important to provide accurate and representative test results in order to inform these designs. Laboratory testing of these materials typically includes grain size distribution and Atterberg limits and can also include residual strength and swell potential testing. To prepare specimens for these tests, materials must be disaggregated into their constituent particles. Liquid limit (LL), plastic limit (PL), and plasticity index (PI) testing per ASTM D4318 specifies that dry samples should be disaggregated using a mortar and rubber-tipped pestle or similar method that does not cause breakdown of individual particles. The material is screened over a 425 µm (No.40) sieve, and the retained material is subjected to additional mortar and pestle processing until the sample is judged to be fully disaggregated. This specimen preparation process can introduce considerable uncertainty, as higher degrees of cementation and/or induration in the sample material require a more significant hand processing effort to fully disaggregate. Furthermore, different operators can vary the disaggregation effort imparted on the sample, and hand processing may never fully disaggregate the clay-size particles, particularly if the material is indurated or desiccated in-situ. A lack of complete disaggregation can lead to unconservative estimates of the material's physical and engineering properties, e.g., lower clay fraction, lower liquid limit. In addition, hand processing is both time consuming and labor intensive.

Ball milling is an alternative to hand processing which is less labor-intensive and largely automated, but there is no established standard for processing fine-grained soils and slakable rock via ball mill. The use of a ball mill to fully disaggregate clay minerals is thought to be a superior approach and has a well-established background in literature. Mesri and Cepeda-Diaz (1986) used a ball mill on air-dried shale to prepare samples for remolded direct shear testing and milled the sample until 100% passed a 75 μm (No. 200) sieve. This same general sample preparation approach was used by Stark and Eid (1994, 1997), Eid (2001), Mesri and Shahien (2003), and Stark and Hussein (2013). All of these studies confirm increases in LL and clay fraction due to ball milling, presumably due to more complete disaggregation.

Stark *et al.* (2005) discusses several advantages to ball mill disaggregation. Ball milling facilitates the measurement of residual strength of over consolidated clays, mudstones, claystones, and shales in the laboratory by expediting and more thoroughly completing the disaggregation process. This results in smaller shear displacements (and thus less time required) to achieve a residual strength condition in laboratory ring shear testing. Because the ball milled samples are known to have higher clay fractions and LLs, the researchers propose a correction factor for comparing LL and clay fraction results between ball milled and hand-processed samples. The authors show that LLs from ball milled samples were 1.1 to 1.7 times higher than the hand-processed LL, and the correction factor increased with increasing LL. The ratio of ball milled clay fraction to hand-processed clay fraction ranged from 1.1 to 2 with higher correction factors at lower LL values.

The previously discussed studies use materials that are processed until 100% passes the 75 μm (No. 200) sieve. This approach is generally accepted if the sample is known to contain only fines and does not contain larger particles, e.g., sand, that can be broken down and altered by the ball milling process; however, petrographic analysis would be required to confirm this conclusion. Taha (2009) discusses the benefits of adding nanoparticles to soil mixtures to improve the properties of the soil. These nano-soils were originally natural soil that was ball milled down to the point of pulverization, documenting that over-milling of geomaterials can result in changes to natural composition and texture of the soil.

This research seeks to advance the understanding of ball milling variables on slakeable rock and desiccated/indurated soil. A better and more thoroughly documented understanding of the variables effecting ball milling is required to develop a standard test method for the technique. The approach to standardization will determine an ideal combination of milling parameters to adequately disaggregate samples with a standard level of disaggregation energy imparted on the sample. Standardization will allow for more widespread adoption of the technique, potentially leading to better and more efficient estimation of critical material properties.

Materials and Methods

Materials

This study assessed the variability in ball milling methods using six natural materials from Bureau of Reclamation project sites including a high plasticity clay, an elastic silt, two shales, a claystone, and a clayey sandstone (Table 1). In addition, a commercially obtained ASTM C33 fine aggregate (concrete sand) was ball milled and tested to assess the potential for pulverization of sand-size mineral grains. The rock samples in this research were received in the lab as HQ-size (61 mm diameter) core while the soil samples were received as air-dried bulk samples from test pits. Unconfined compressive strength (ASTM D7012), indirect tensile strength (ASTM D3967) and slake durability (ASTM D4644) data are provided for rock samples where available (Table 1).

The high plasticity clay is Quaternary slope wash (Qsw) material obtained from the foundation of an earthen embankment dam in the western portion of the Central Valley in California. The Qsw is a surficial cover developed on Panoche Formation rocks and is overconsolidated in-situ, presumably due to desiccation. The elastic silt was obtained from the core of an earthen embankment dam in Oregon, located in the western Cascades physiographic province. The material was sourced from required excavation during construction and from a nearby borrow area and is composed of highly weathered volcanic tuff. Petrographic examination revealed that the material contains a significant amount of the clay mineral halloysite. The first shale, termed Shale 1, was obtained from a pipeline project in northern New Mexico and comes from the Mancos Shale formation. This shale was moderately weathered and interbedded with sandstone. The clayey sandstone was obtained from the same project as Shale 1. The local geology is characterized as predominately shale with interbedded lenses of sandstone. This sandstone was of particular interest because a petrographic analysis from a prior investigation revealed clay minerals in the rock matrix. In addition, the material exhibited plasticity (ASTM D4318) with a hand-processed sample. Shale 2 was obtained from beneath a spillway chute of a dam in Kansas and comes from the Graneros Shale formation. It had a low degree of weathering. The claystone was obtained from a foundation site near a dam in southern New Mexico and is from the McRae formation. The claystone was the most highly weathered of any intact rock specimens and existed in thin, high-angle lenses between sandstone layers. For this reason, the claystone samples used in this research likely contain a sand fraction.

Test Methods

Bulk ~8 kg samples of each material were passed through a jaw crusher to coarsely process the material to 100% passing a 9.5 mm (No. 5) sieve. Each coarsely processed bulk sample was reduced per ASTM C702, yielding a total of sixteen ~500 g samples. Each sample was ball milled in a matrix-based approach to evaluate combinations of ball material, ball size, and milling duration on the various materials. In addition, one sample was hand-processed per guidance in ASTM D4318 to establish a baseline for each material. The six natural materials were milled for times of 5, 10, and 30 minutes and the concrete sand was milled for durations of 5, 10, 30, and 60 minutes.

Milling was performed using a SPEX 8000M Mixer/Mill and SPEX hardened steel vial set with internal volume of 65 cm^3. The steel vials were filled with coarse sample to approximately 30% of their volume, which was approximately 20-30 g depending on material. This variability in mass was affected by material unit weight and size/shape of coarsely processed particles. The balls used in this testing included 12 mm and 6 mm diameter hardened steel balls included with the vial set and 12 mm and 6 mm fluoroelastomer rubber balls with a durometer rating of 75A (hard). To assess the potential for sample contamination, the rubber balls were tested for 60 minutes on C33 sand and found to undergo negligible mass loss, indicating that the ball material is not being abraded into the sample.

All six natural materials were subjected to grain size distribution testing in accordance with USBR 5330-89 (combined sieve and hydrometer test) and Atterberg limit testing in accordance with ASTM D4318. The concrete sand was subjected to gradation analysis only.

TABLE 1: Summary of materials used in this research.

Material[1]	Geologic Classification	Compressive Strength[2] (MPa)	Tensile Strength[2] (Mpa)	2-Cycle Slake Durability	USCS (Hand)
CH	Quaternary Slope Wash	-	-	-	CH
MH	Weathered Tuff	-	-	-	MH
Clayey Sandstone	Mancos Shale Formation	4.4	0.56	95	SC
Shale 1	Mancos Shale Formation	5.6	0.36	40.2	CL
Shale 2	Graneros Shale Formation	8.5	0.89	90.8	CL
Claystone	McRae Formation	-	-	-	CL-ML

[1] Material classifications for soil are based on hand-processed USCS results. Classifications for rock are based on logs from field geologists

[2] Compressive and tensile strength results based on average values from testing on intact rock specimens.

Results and Discussion

The effectiveness of the various milling combinations on each material is assessed in terms of gradation, LL and PI. For the purpose of this research, more effective disaggregation is characterized by higher LL and PI values and higher clay fraction, defined as material finer than 2 μm. Methods utilizing a metal ball are evaluated in the context of potential grain pulverization evaluated in terms of a fining of the sand fraction of the sample, i.e., material retained on the 75 μm sieve. As all samples were processed and tested by the same laboratory, repeatability limits are plotted on hand-processed results based on ranges and material types reported in ASTM D4318, D6913, and D7928.

Gradation results from milling the concrete sand are shown in Figure 1. Compared to the hand-processed result (assumed to be representative given the lack of aggregated fine particles), it is apparent that the 12 mm metal ball significantly alters the gradation of the sand by pulverizing the mineral grains, especially at longer milling durations. Gradation results from milling with the 6 mm metal ball indicate that longer milling time is associated with higher fines content, but much less so than when milling with the 12 mm ball. This is presumably due to the smaller ball abrading but not fully pulverizing the sand grains. The gradations for both the 12 mm and 6 mm rubber balls are relatively similar but do exhibit a slight trend in increased fines content with increased milling time. While no single method falls within the in-lab repeatability limit on the hand-processed plot, results from the rubber balls most closely match the hand-processed result. Scatter in the results is attributed to natural variation in samples.

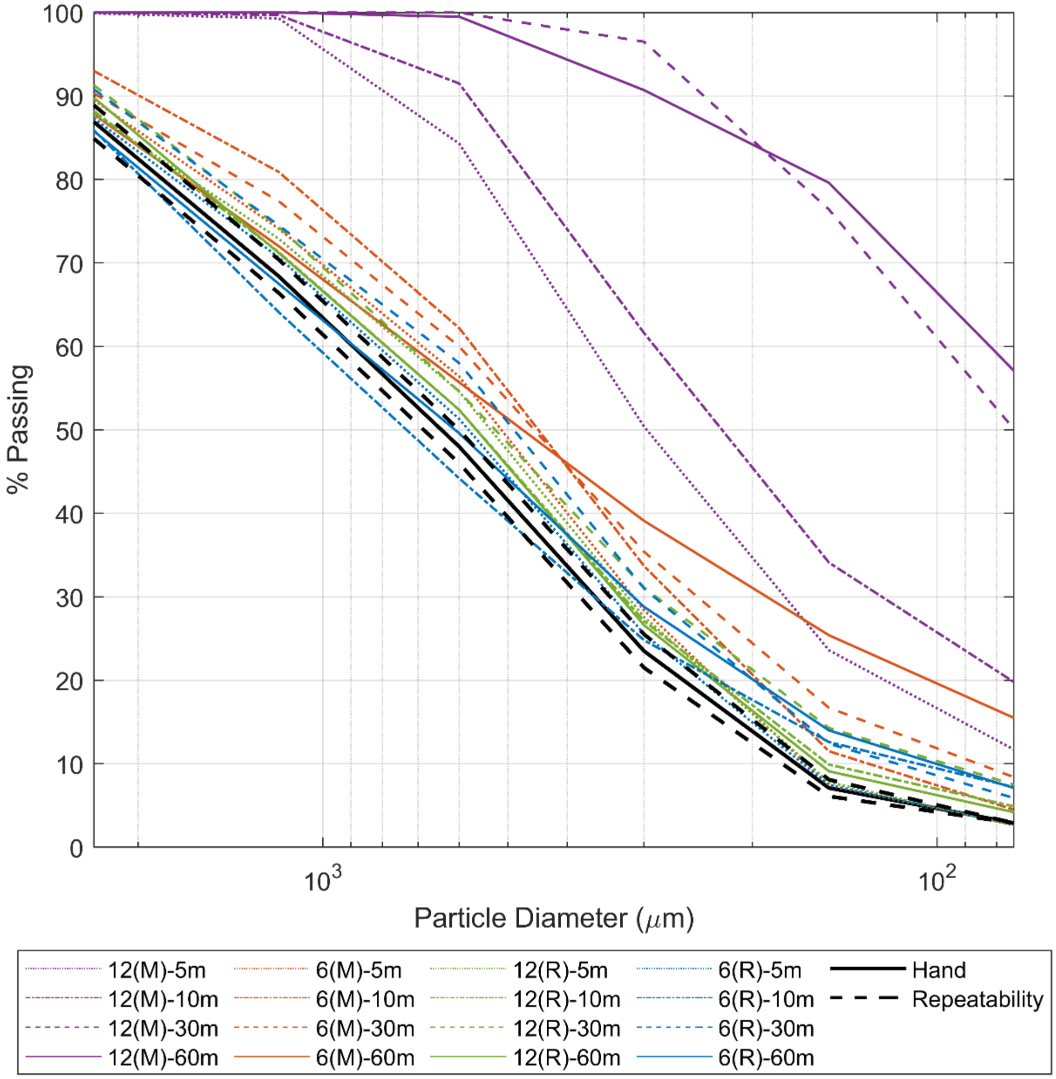

FIGURE 1: Gradation results for C33 sand. Legend entries are defined as “ball size (ball material) – milling duration.”

Gradation and plasticity results for each combination of natural material and milling method are shown in Figures 2 and 3, respectively. The milling processes affected the gradation and plasticity of each material differently. For the high plasticity clay, gradation results were within the hand-processed repeatability limit for all milling combinations down to the sand fraction but diverged significantly in the fines portion of the material (Figure 2a). The highest portion of fines was obtained using the 12 mm rubber ball for 60 minutes, but all milling combinations resulted in a higher fines portion than hand-processing. Including the LL and PI results (Figure 3a), the hand-processed sample classified as CH, while the milled samples classified as CH or CL depending on the ball milling method. In general, 5-minute milling times were less effective than hand processing while 30-minute milling times had the highest increase in plasticity compared to the hand-processed result. Only the 5-minute 12 mm metal ball result fell within the hand-processed in-lab repeatability limit.

The elastic silt had widely spanning gradation results due to milling, especially at particle sizes greater than 75 μm (Figure 2b). 30-minute milling durations with the 12 mm metal and rubber balls resulted in equal or better disaggregation compared to hand processing over the entire range of particle sizes. 5- and 10-minute milling durations, specifically for the rubber balls, showed significantly coarser gradation at particle sizes greater than 75 μm; however, all milling combinations resulted in higher fines portions than hand processing. The hand-processed sample classified as MH and although the classification did not change with any milling method, there is a notable increase in PI for all methods (Figure 3b). There is a general increase in LL with increased milling time with 10- and 30-minute combinations being equal to or more effective than hand-processed. 5-minute milling times were generally less effective than hand-processed, but the 5-minute large and small rubber ball results fell within the repeatability limit.

Shale 1 gradation curves have a relatively similar shape across all methods, with the hand-processed result representing a coarser limit for most particle sizes. The 12 mm metal ball provided the highest degree of disaggregation but also showed a significant increase in percent passing at particle sizes greater than 75 µm compared to the hand-processed result. Most other milling combinations were clustered together and had slightly higher percent passing than hand processing. Some of the 5- and 10-minute milling combinations have lower percent passing than hand-processed, especially in the 9-75 µm range. The hand-processed sample classified as CL and did not change classification with any milling method; however, there is a notable increase in both LL and PI for all methods (Figure 3c), and no results fall within the repeatability limit. Increased milling time had a significant effect on the increase in LL and PI, and in general, 12 mm metal and rubber balls appear to outperform their 6 mm counterparts.

Shale 2 was notably more difficult and time consuming to hand process than most other materials and had higher uniaxial compressive and tensile strength values compared to Shale 1. The 12 mm metal ball with 30-minute milling time was the only method to have a finer gradation than the hand-processed sample at every interval, but the 10-minute milling time was very close to achieving this condition (Figure 2d). Gradations from all other milling methods had lower percent passing than those from hand-processed samples at larger particle sizes. Between 37 and 9 µm, gradations for each milling method surpassed the hand-processed curve to achieve proportionally more fines. The hand-processed sample classified as CL and did not change classification with any milling method. Similar to Shale 1, a notable increase in both LL and PI was observed for all methods (Figure 3d), and the 12 mm rubber ball with 5-minute milling time was the only result within the repeatability limit. The largest increase in LL and PI was associated with longer milling durations and metal balls.

The claystone was similar to Shale 2 in that it was especially difficult and time consuming to hand process, and only the 30-minute 12 mm metal ball gradation was finer than hand-processed over the entire range of particle sizes. Strength testing was not performed on this material because it occurred in thin, high angle layers that were insufficient for specimen preparation. The gradations from processing with the 6mm metal ball and both rubber balls were significantly coarser than those for the hand-processed samples until the 37 μm data point, after which the gradations began transitioning to finer than hand-processed (Figure 2e). The hand-processed sample classified as CL and did not change classification with any milling method (Figure 3e). The claystone experienced a considerable increase in both LL and PI that was most heavily influenced by milling time with only the 6 mm metal ball with 5-minute milling time falling within the in-lab repeatability limit.

The results for the clayey sandstone indicate that the 12 mm metal ball is likely pulverizing sand grains and altering its natural composition. This is illustrated in Figure 2f where the metal ball gradation curves are shifted to an overall finer gradation compared to the other milling methods. With the exception of the 30-minute 12mm metal ball, the gradation curves were relatively similar in shape with the hand-processed result serving as a coarser bound. The hand-processed sample classified as CL-ML. All three milling durations with the 12 mm metal ball pushed the material to classify as CL, but it is likely that the additional contributing fines are the result of material pulverization. The 30-minute milling times for both the 6 mm metal ball and the 12 mm rubber ball are on the boundary to CL classification, with the 12 mm rubber ball inducing a slightly higher LL. Under all milling combinations, the clayey sandstone had higher LL values. For shorter milling times, PI values were equal to hand-processed and increased slightly with increased milling time. ASTM D4318 does not specify repeatability limits for SC, so no bounding box is shown in Figure 3f.

Hand processing is not a consistent means of test specimen preparation because the amount of time and effort required to obtain sufficient sample quantity for index testing is highly material dependent. The claystone and Shale 2 were difficult to hand process and required multiple cycles of processing for material retained on the 425 µm (No. 40) sieve. For several milling combinations, the ball mill was not able to match the hand-processed gradation at larger particle sizes. Post-milling inspection of these two materials revealed that the higher percent retained at larger particle size was largely due to sample that had not been fully disaggregated. The milling approaches in this study each impart a relatively constant amount of energy on the sample being processed, especially in comparison to the hand processing approach. The ball mill can likely match the hand-processed result for harder materials by increasing the milling time. In addition, the sample quantity in the milling container can be reduced, but the effects of sample to cannister volume ratio were not studied in this research.

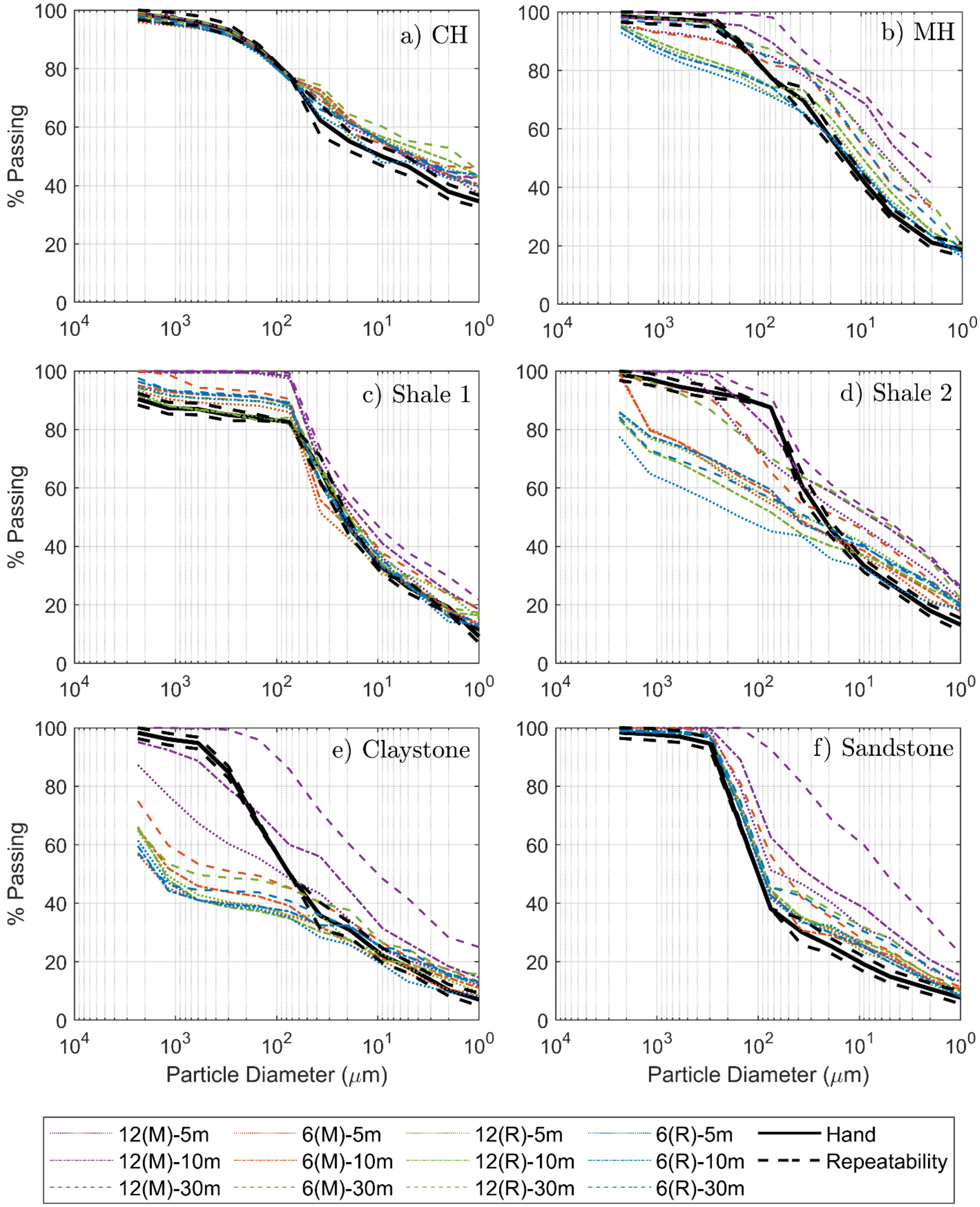

FIGURE 2: Gradation results for all six natural materials.

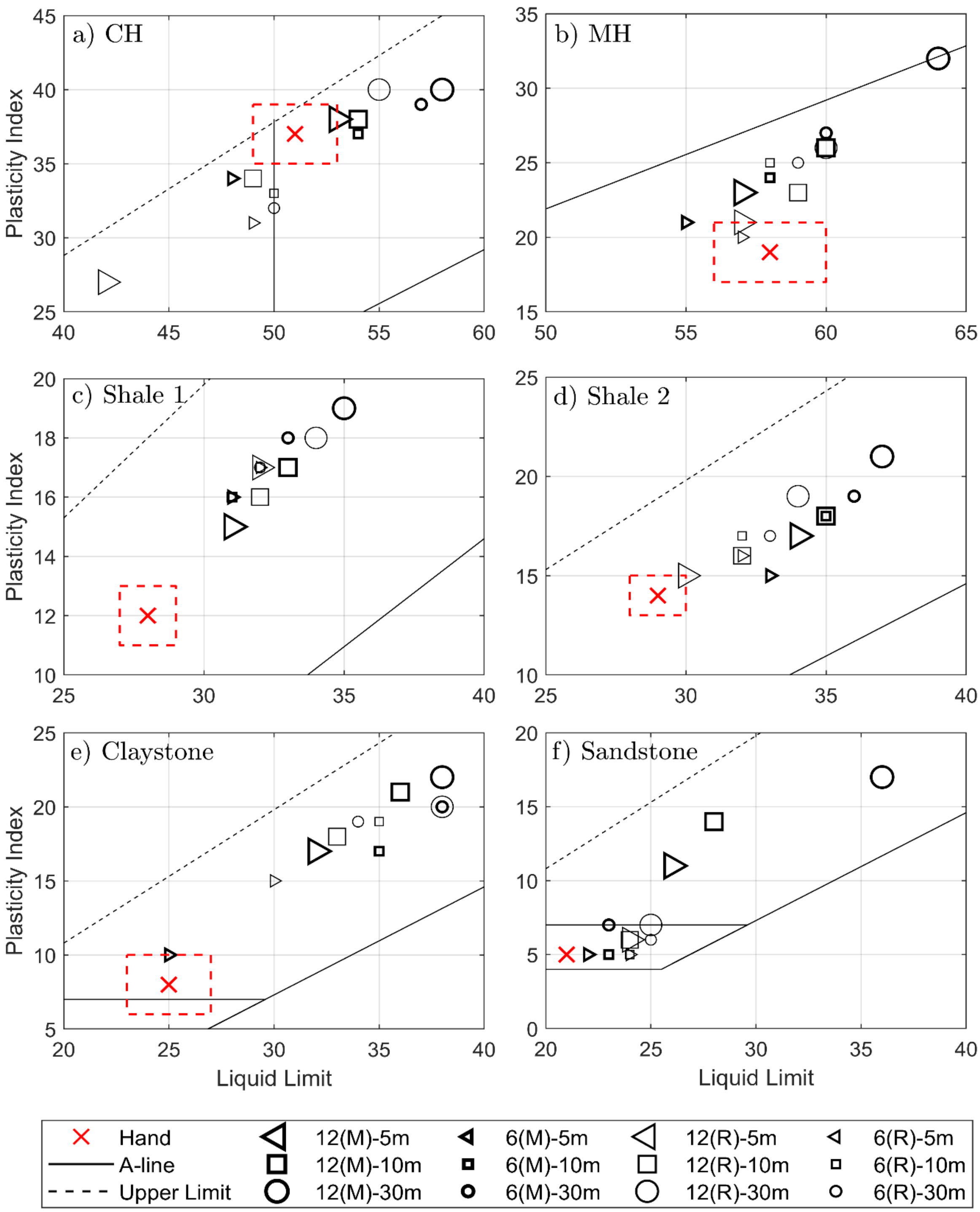

FIGURE 3: Liquid Limit and Plasticity Index results for all six natural materials (note that scales vary between each figure). The dashed red line around the hand-processed point represents the in-lab repeatability limits per ASTM D4318.

To assess the relative effectiveness of each ball mill combination, results were normalized by the corresponding hand-processed result after the approach in Stark *et al.* (2005). These relationships are shown in Equations 1, 2, and 3.

$$\widehat{\mathrm{LL}}=\frac{\mathrm{LL_M}}{\mathrm{LL_H}} \tag{1}$$

where:

$\widehat{LL}$ = normalized liquid limit

LL_M = liquid limit obtained from a ball milling method

LL_H = liquid limit obtained from a hand-processed sample

$$\widehat{PI}=\frac{PI_M}{PI_H} \tag{2}$$

where:

$\widehat{PI}$ = normalized plasticity index

PI_M = plasticity index obtained from a ball milling method

PI_H = plasticity index obtained from a hand-processed sample

$$\hat{C}=\frac{C_M}{C_H} \tag{3}$$

where:

$\hat{C}$ = normalized clay content

C_M = clay content from gradation tested with a ball milled sample

PI_H = clay content from gradation tested with a hand-processed sample

These normalized properties serve as scaling factors to compare the effectiveness of each milling method across material type. Where $\widehat{LL}$, $\widehat{PI}$, or $\hat{C} = 1$, the milling method produces a result equivalent to hand processing, but when these parameters have values greater than 1, a higher degree of disaggregation (and potentially pulverization) is achieved. A value lower than 1 indicates that the method was less effective than hand processing. Establishing these correction factors over a range of materials is important because the majority of historic test data was conducted with hand-processed samples.

Stark *et al.* (2005) found that the ratio of ball milled clay fraction to hand-processed clay fraction ranged from 1.1 to 2 for the materials evaluated. In this study, $\hat{C}$ was found to be both material and milling method dependent (Table 2). Shale 1 had a maximum $\hat{C}$ of 1.55 with the 30-minute 12 mm metal ball, but on average, had the smallest $\hat{C}$ values with 5-minute milling combinations having $\hat{C}<1$. With the exception of the 5-minute 6 mm rubber ball on claystone, all other milling combinations had $\hat{C}\geq 1$. The highest values of $\hat{C}$ were obtained from the 30-minute 12 mm metal ball method for all materials but the CH, where maximum $\hat{C}$ occurred with the 30-minute 12 mm rubber ball method. In general, $\hat{C}$ values in this study were consistent with the range observed by Stark *et al.* (2005). Values outside of this range are attributed to lack of disaggregation for $\hat{C}<1$ and addition of fines due to pulverization for $\hat{C}\geq 2$. The pulverization effect is most prominent in the clayey sandstone and claystone, both of which have known sand content.

A plot of $\widehat{LL}$ vs. $\widehat{PI}$ illustrates the difference in plasticity caused by milling for each material and milling combination (Figure 4). All six natural materials achieved a higher degree of disaggregation due to milling. Rock samples had $\widehat{LL}$ and $\widehat{PI} \geq 1$ for all milling combinations. While the natural soils (CH and MH) had some milling methods where $\widehat{LL}$ and/or $\widehat{PI} > 1$, they were also the only materials to also have some values less than 1. Milling combinations deemed less effective than hand processing were primarily associated with 5-minute milling times.

Excluding short duration milling that was less effective than hand processing, $\widehat{LL}$ ranged from 1.0 to 1.7, which is consistent with the 1.1 to 1.7 range reported by Stark *et al.* (2005). $\widehat{PI}$ ranged from 1.0 to 3.4, and both $\widehat{LL}$ and $\widehat{PI}$ were highly material dependent. The sandstone had the highest values of both $\widehat{LL}$ and $\widehat{PI}$, but this fit is notably skewed by the 12mm metal ball data which is likely introducing additional fines via grain pulverization. The two shales have $\widehat{LL}$ and $\widehat{PI}$ ranges that are relatively similar, with Shale 1 plotting slightly higher $\widehat{PI}$ values. The natural soils have relatively low $\widehat{LL}$ values. The CH material also has relatively low $\widehat{PI}$ values, but the MH had the highest $\widehat{PI}$ values of any material besides the sandstone.

When comparing these normalized results across material, it is important to consider the slope of the best-fit lines. A shallow slope indicates that the LL and PL are increasing at more similar rates (recall PI = LL - PL) while a steeper slope indicates that the LL is increasing relatively more rapidly than the PL. This range in slopes exhibited by the linear fits proposed in Figure 4 further illustrates the material dependence on milling method. In all cases, the best-fit slope was greater than 1, indicating that the ball milling process achieves a greater increase in LL than PL.

TABLE 2: Normalized clay fraction, $\hat{C}$, for each material and milling combination.

Milling Method	$\hat{C}$					
	CH	MH	Shale 1	Shale 2	Claystone	Clayey Sandstone
12(M)-5m	1.16	1.54	0.98	1.72	1.53	1.63
12(M)-10m	1.13	1.95	1.27	1.98	1.79	1.93
12(M)-30m	1.18	2.37	1.55	2.04	2.76	3.18
6(M)-5m	1.15	1.20	0.88	1.19	1.02	1.34
6(M)-10m	1.15	1.30	0.96	1.36	1.41	1.27
6(M)-30m	1.23	1.58	1.22	1.58	1.69	1.42
12(R)-5m	1.21	1.20	0.99	1.50	1.34	1.15
12(R)-10m	1.28	1.17	0.91	1.41	1.49	1.43
12(R)-30m	1.40	1.63	1.24	1.96	1.63	1.67
6(R)-5m	1.12	1.10	0.74	1.15	0.94	1.24
6(R)-10m	1.18	1.11	0.83	1.55	1.55	1.24
6(R)-30m	1.16	1.37	0.90	1.51	1.48	1.71

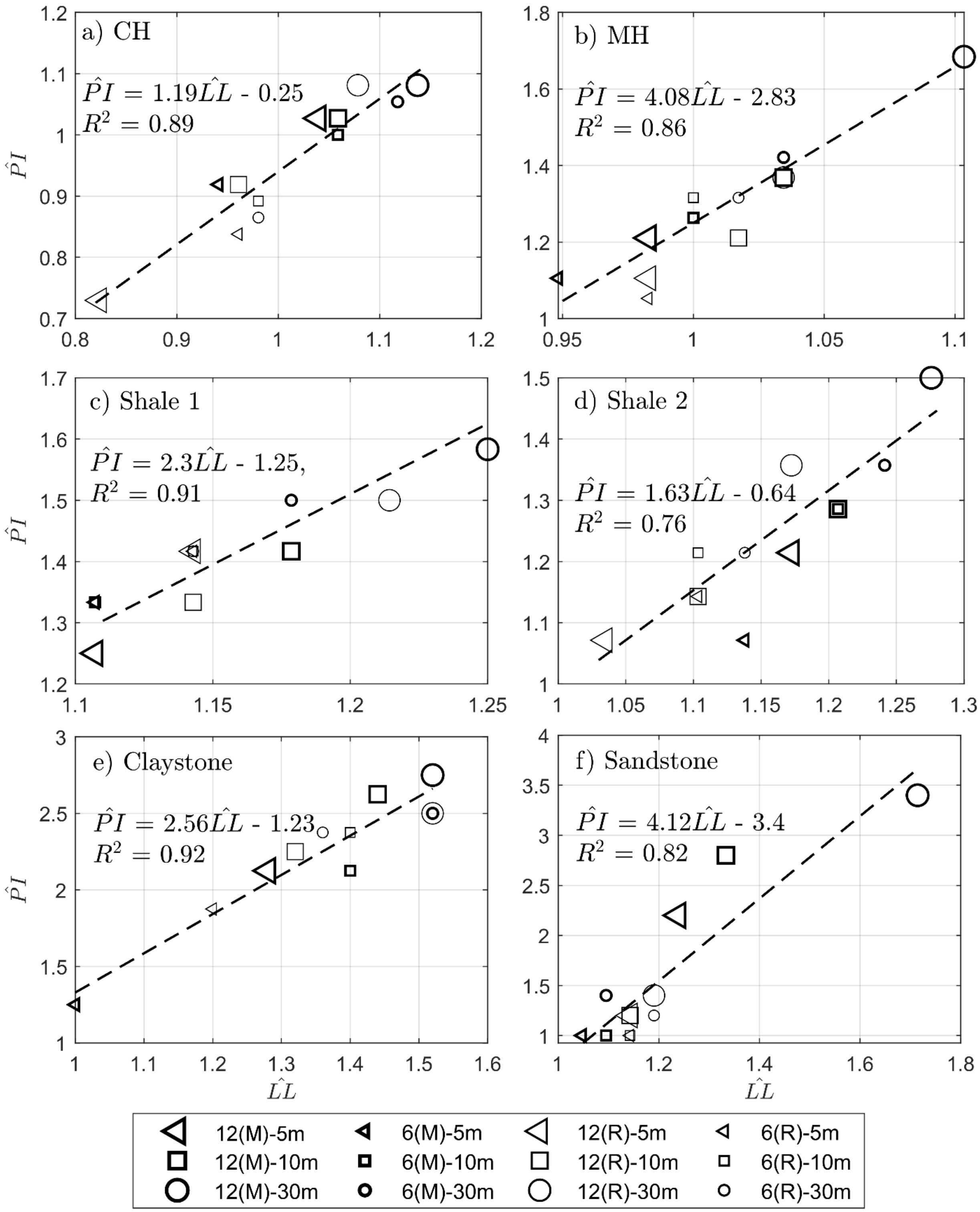

FIGURE 4: Normalized LL and PI data for all natural materials.

Conclusions

This study presents the gradation and plasticity results of samples processed with various ball milling combinations on six natural materials and a concrete sand. As a general trend, finer gradations and higher plasticity values were obtained from increased milling time, increased ball size, and the use of a metal ball vs. rubber ball. For almost all cases, the 12 mm metal ball provides the highest degree of disaggregation; however, as evidenced in the concrete sand and clayey sandstone test results, the metal ball causes significant alteration to the materials natural particle size distribution. While petrographic analysis has not been performed to characterize alteration at the particle level, it is generally inferred that the sand particles are being pulverized into smaller particles, and that some complex clay mineral shapes are being altered as well.

The ball mill outperformed hand processing in obtaining a higher percent fine fraction for almost all milling combinations, even in instances where the ball mill failed to fully disaggregate larger pieces of sample. Ball mill performance is material dependent, but for all materials evaluated in this study, ball milling shifted the LL, PI, and clay fraction results outside of the within-laboratory repeatability limit for hand-processed samples. A single ball mill standard process may not exist, and a suitable method for a particular material may require a selection process to identify the right combination of ball material, size, and milling time. The presence of sand in natural materials complicates this selection process as metal balls are clearly altering the composition of the sand grains and should not be used for sandy materials.

Normalized parameters $\widehat{LL}$, $\widehat{PI}$, and $\hat{C}$ are material dependent but found to be consistent with ranges reported in literature. A plot of $\widehat{LL}$ vs. $\widehat{PI}$ for all materials illustrates trends in the degree of disaggregation achieved by each milling method. Comparing the slopes of best-fit lines through the $\widehat{LL}$ vs. $\widehat{PI}$ data, a higher slope indicates that LL is increasing at a faster rate than PL.

All of the fits in this study had slopes greater than 1, indicating that ball mill processing causes a greater increase in LL than PL compared to hand processing.

The results of this study indicate that ball milling is effective at disaggregating a wide variety of natural geomaterials, but additional testing is required before a standard can be developed. Future research will incorporate petrographic analysis of ball milled and hand-processed samples both pre- and post-processing to confirm the presence of grain alteration. In addition, both hand-processed and milled samples will be tested in triplicate to assess the repeatability of ball milling. To reduce the effects of natural sample variation, several control materials with well-known properties will be evaluated. Additional natural materials will also be evaluated to improve the relationships developed in this study. Longer milling durations will be assessed for harder materials, and the effects of sample volume within the cannister will also be studied.

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