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Modeling particle size distribution for subaqueous soil survey applications

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Abstract

Coastal environments face a growing number of challenges as a result of a changing climate (e.g., sea level rise, flooding, and erosion). In response, intertidal and subaqueous soils (SAS) are being mapped to provide a soil resource inventory for use and management decisions. An essential part of any soil resource inventory is particle size distribution (PSD) analysis. Coastal soils have elevated levels of salts and sulfides that can complicate PSD analysis, requiring time-intensive pretreatments. We tested a regression model to reduce reliance on labor-intensive methods for PSD analysis. Analysis of 257 SAS samples revealed a strong sand–silt relationship ($p < 0.0001$; $r^2 = 0.975$), allowing for accurate silt and clay prediction from sand content. For samples with >40% sand (70% of the 257 samples), average absolute residuals of predicted silt ranged from 0.80% to 3.58%. Randomized iterative testing (10,000 iterations) showed that as few as 50 samples of the original 257 could be used to develop a model to provide PSD data with <4% absolute error for predicting silt for samples with >40% sand. Accuracy of the model declined for samples with ≤40% sand, especially <20% sand where average absolute residuals ranged from 7% to 8%. We hypothesized that diatom skeletons disrupted the sand–silt relationship in the silt-dominated samples, which contained as many as 9% diatoms. The regression model developed in this study offers a faster, more time- and cost-effective alternative for determining PSD analysis in SAS with >40% sand, aiding large-scale soil survey efforts.

Plain Language Summary

Across the US coasts, large areas of underwater soils (subaqueous soils) are being mapped. This finding provides soil property information for scientists making use and management decisions. A key soil property is the relative abundance of sand-, silt-, and clay-sized particles in a sample, or what is called the particle size

Abbreviations: AAR, average absolute residual; CZSS, coastal zone soil survey; PSA, particle size analysis; PSD, particle size distribution; SAS, subaqueous soils.

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distribution (PSD). Coastal soils usually have high levels of salt that need to be washed out prior to PSD analysis. Our goal was to develop a way to determine PSD without having to perform this washing. Our hypothesis was that the PSD of subaqueous soils is energy-dependent and so should follow a simple model. In faster-moving water, larger particles settle out; in slower-moving water, smaller particles settle. To test our hypothesis, we analyzed 257 samples from a large estuary in Connecticut. A very strong relationship between sand and silt contents allowed us to predict PSD without the washing steps.

1 | INTRODUCTION

The coastal environment, and associated communities, are subjected to a number of stressors and issues including sea level rise, saltwater intrusion, intense storms, hurricanes, increased nutrient loading, and shoreline erosion. Considering that over 80% of the US population lives in coastal states (NOAA, 2024), the USDA-NRCS (2024) initiated the coastal zone soil survey (CZSS) to provide an inventory of these soil resources for use and management decision-making and problem-solving. Characterization is the foundation of any soil inventory, especially particle size distribution (PSD). Since mapping and characterization of subaqueous soils (SAS) has only been a part of soil survey in the last 20 years (M. Stolt et al., 2017), PSD data for SAS are quite limited relative to data for subaerial soils, and the datasets need to be expanded.

The most common approaches to measure PSD are the long-standing hydrometer and pipette methods, which have been in use for nearly a century (Bouyoucos, 1934; Gee & Or, 2002). Given that PSD is such an integral part of soil characterization, methods that are quicker or simpler to perform are being developed and tested including laser diffraction, integral suspension pressure (e.g., PARIO), and electrical conductivity (Hasan & Abuel-Naga, 2024; Leemhuis et al., 2024; Messing et al., 2024; Zhang et al., 2024). The accuracy of these alternative methods is often tested against pipette analysis (Centeri et al., 2015; Faé et al., 2019; Konert & Vandenberghe, 1997), suggesting that the pipette method is the most accurate but also the most labor- and time-intensive. Of the alternative PSD methods, laser diffractometry has been in use for the longest time and is the most common in the literature (Bittelli et al., 2022; Faé et al., 2019; Leemhuis et al., 2024; Loizeau et al., 1994; Messing et al., 2024; Miller & Schaetzl, 2012; Polakowski et al., 2021; Sperazza et al., 2004). Regardless of the approach to measure PSD, there are associated errors that need to be considered as a result of sample preparation and inherent differences in replicate samples, instrument errors, and analyst errors.

Analysis of PSD is complicated in coastal soils because of salts. This is especially an issue in estuarine SAS, which are continuously exposed to the sulfate- and halide salt-rich overlying water column, often at concentrations of 600 mM (35 ppt) or more (Schmitt, 2008). In estuarine subaqueous environments, halide salts are trapped in the porewater, and the sulfate is often reduced to sulfide, forming Fe mono- or disulfide (M. H. Stolt & Rabenhorst, 2011; M. Stolt et al., 2017). Sulfate salts are produced when the SAS samples are dried for analysis and the sulfides are oxidized. Issues with the salts are twofold: (1) flocculation of clay-sized particles due to electrostatic forces between salts and clay particles (Chibowski et al., 2011), which causes erroneous measures of finer particles in the sedimentation column; and (2) errors in clay content measures as dissolved salts are measured as part of the clay-sized fraction, leading to an overestimation of the percent clay (Bradley, 2001; Demas, 1998; Gregory & O'Melia, 1989; M. H. Stolt & Rabenhorst, 2011; Sutherland et al., 2015). To minimize the effects, salts are removed through washing and centrifuging or with week-long dialysis tubing osmosis until salt concentrations are below 10 mM (Gee & Or, 2002). Salt removal is time-intensive and has the potential of additional error if fine material is lost during decanting or removing samples from the dialysis tubing. Incorrect measurement of PSD of SAS may lead to interpretation issues relative to dredging, erodibility, cation exchange capacity, and shellfish suitability (Balduff, 2007; Barko & Smart, 1986; Erich et al., 2010; M. H. Stolt & Rabenhorst, 2011). Thus, there is a need for a less time-consuming approach to measure the PSD of SAS without introducing additional error. This is especially relevant given the current CZSS efforts to deliver a soil inventory of the coastal soils of the United States (USDA-NRCS, 2024).

In aquatic ecosystems, PSD is primarily controlled by the amount of energy in the water, with coarser materials settling out in high-energy environments and finer materials settling out in lower-energy environments (Balduff, 2007; Bradley, 2001; Demas, 1998; M. H. Stolt & Rabenhorst, 2011; M. Stolt et al., 2017). Balduff (2007) noted that this results in a much narrower range in SAS particle distributions compared to soils from nearby subaerial soils. Because of the relationship between system energy and PSD, we hypothesized that there should be a relatively simple model that explains the variability in percent sand relative to percent silt in SAS. Once

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such a model is developed for a given project or study area, the analyst would be able to determine silt and clay content and thus the texture class of SAS samples based on sand contents alone. This method would predict silt content from sand content, and clay content would be determined by the remaining portion of the PSD not contributed by silt and sand. Such a method would minimize the need for pipette, laser, or hydrometer analysis, and thus, salt removal would not be necessary. In order to validate this as a potential method for PSD analysis in future characterization efforts, we tested this approach on 257 SAS samples collected from a large estuary (Long Island Sound).

2 | METHODOLOGY

Long Island Sound extends across the entire coastal shoreline of Connecticut (185 km). Sampling efforts were focused on water depths to 4 m in nearshore subaqueous environments by NRCS soil scientists as a part of their Long Island Sound soil survey efforts (USDA-NRCS, 2022). Note that 106 pedons were sampled via vibracore to 200+ cm or refusal, and typical field properties were described and recorded (horizon designation, field texture class, fluidity, boundary distinction, 3% and 30% H_2O_2 reaction class, pH, electrical conductivity, odor, and parent material). A total of 257 mineral horizons were randomly selected and analyzed for PSD from a range of SAS pedons distributed over the entire survey area.

2.1 | Particle size analysis (PSA)

Bulk samples were air-dried and gently ground with a mortar and pestle to disrupt clods >2 mm. Dried and ground samples were passed through a 2-mm (#10) sieve to remove coarse fragments. A portion of each sample was dried at 105˚C and weighed to determine soil moisture content, and heated to 550˚C for 5 h (loss on ignition) to determine soil organic matter (SOM) content. For samples with >4% SOM, organic matter was removed with 30% hydrogen peroxide prior to PSD analysis (Gee & Or, 2002). Replicate samples were processed to determine oven-dry weights for calculating PSD by weight. Processed samples were added to centrifuge bottles, filled with deionized (DI) water, shaken by hand, and centrifuged at 2500 RPM for 7 min. The supernatant was decanted, and additional water was added to the soil, shaken, and centrifuged again. This was repeated (two to five times, depending on the sample) until the sample was washed of chloride salts. We tested for salts by adding a drop (0.05 mL) of 0.017 N silver nitrate to approximately 2 mL of supernatant (Nóbrega et al., 2023). We assumed that the sample was free of salts if no white precipitate formed.

Core Ideas

- Salts and sulfides in estuarine subaqueous soils can complicate measuring particle size distributions.
- Particle size distribution of subaqueous soils is dictated by the energy (flow rate) of the overlying water column.
- Particle size distribution can be effectively modeled by total sand content for large-scale subaqueous soil surveys.
- Finer-textured subaqueous soils may have a large quantity of silt-sized diatom skeletons that affect particle size distribution relationships.

PSD was determined for all mineral samples following the procedures described by Gee and Or (2002) after removing salts (and organic matter when needed). Samples were treated with a sodium hexametaphosphate solution and placed on a horizontal shaker for 24 h to disperse the primary particles. Sands were separated from the silt and clay fraction by wet-sieving using a 53-μm (#270) sieve, oven-dried, and the fractions separated through a nest of standard-sized sieves (1000, 500, 250, 105, and 53 μm). Silt and clay particles were collected in sedimentation columns and determined by the pipette method (Gee & Or, 2002).

2.2 | Diatom quantification

Diatoms are single-celled aquatic organisms whose silica-based skeletons (frustules) often fall in the silt-sized range (Battarbee et al., 2002). To estimate their abundance, we randomly selected 30 mineral horizons from the 257 samples. For each horizon, 1–2 g of air-dried soil was rinsed with DI water through a 53-μm sieve to filter out very fine sand and coarser-sized particles. The entire <53 μm slurry was retained and homogenized, and a 1 mL aliquot was pipetted onto a glass slide and gently dried on a hot plate.

The dried slurry was analyzed using a Zeiss 470916–9903/38 standard microscope equipped with a bright-field optical system and magnifications ranging from 100× to 400×. We adapted the line-point intercept grain-count technique of Galehouse (1971). A single fixed point in the center of the eyepiece reticle served as the counting probe, while the mechanical stage advanced in parallel transects from right to left. Diatoms were identified by valve outline, raphe structure, and striae pattern following Battarbee (1986). Fragmented or weathered frustules were counted if at least one diagnostic feature could be recognized; otherwise, they were logged as silt. Every particle crossing the probe was recorded until

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300 intercepts were tallied and recorded as either mineral or diatom.

2.3 | Statistical analysis

JASP 0.19.0 (JASP Team, 2024) was utilized for exploratory data analysis. R version 4.4.2 (R Core Team, 2020) was utilized for figure creation, linear regression modeling, and residual calculation. A linear regression was established with total sand as the independent variable and total silt as the dependent. As a measure of model error, we calculated average absolute silt content residuals (|actual silt content − predicted silt content|) across 10% sand intervals (e.g., 10%–20%, 20%–30%). To determine the minimum sample size required for a reliable predictive model in future studies, we conducted random subsampling without replacement, testing model sample sizes from 10 to 250 in increments of 10. For each model sample size, we randomly selected subsets from the full dataset ($n = 257$) and fit a linear regression model to the silt and sand content. Absolute residuals were calculated for all points in the full dataset. R code was used to sample data (see Supporting Information for code). This process was repeated 10,000 times per model sample size, yielding mean and maximum average absolute residuals (AARs) for each iteration.

3 | RESULTS AND DISCUSSION

The PSD in the ternary textural triangle diagram shows a simple pattern of an increase in silt with a decrease in sand, while the largest range in clay (2%–31%) occurs at the highest silt contents (Figure 1). Similar results were reported by Balduff (2007) for SAS in Chincoteague Bay (a large estuary in Virginia). The relationship between total sand content and percent silt was highly significant ($p < 0.0001$; $r^2 = 0.975$; Figure 2). In contrast, using silt as a predictor of clay was relatively ineffective, with an r^2 value of only 0.376. When using sand as a predictor, AAR values of predicted silt ranged from <1% to 8%, with a general increase going from the greatest amount of sand to the least. For sand contents >20%, AAR values were 4.5% or less, but for sand contents <20%, the values nearly doubled (Figure 3), suggesting confounding factors in explaining variability in the PSD of the finer fractions. Some variation in PSD is expected when utilizing different methods. For example, reports suggest that there is a 55%–95% agreement between different methods (laser diffraction, hydrometer, and pipette) for assigning particle size classes (Bittelli et al., 2022; Coates & Hulse, 1985; Elfaki et al., 2015; Faé et al., 2019; Polakowski et al., 2021; Yang et al., 2019). Miller and Schaetzl (2012), for example, reported that over 11% of the replicate samples measured using laser diffractometry showed a different texture class. Intra-method variation is another source of error, with typical variation in percent silt between 1% and 4% expected for the pipette method (Centeri et al., 2015; Coates & Hulse, 1985; Indorante et al., 1990; Mota et al., 2019). Centeri (2015) examined the differences in percent measured silt between two professionals determining PSD via pipette and reported a mean absolute difference of 1.53 ± 2.28% (2 standard deviations).

Given the importance of accurate PSD data, a common question in many of the investigations of PSD methodology is how much error is acceptable (Centeri et al., 2015;

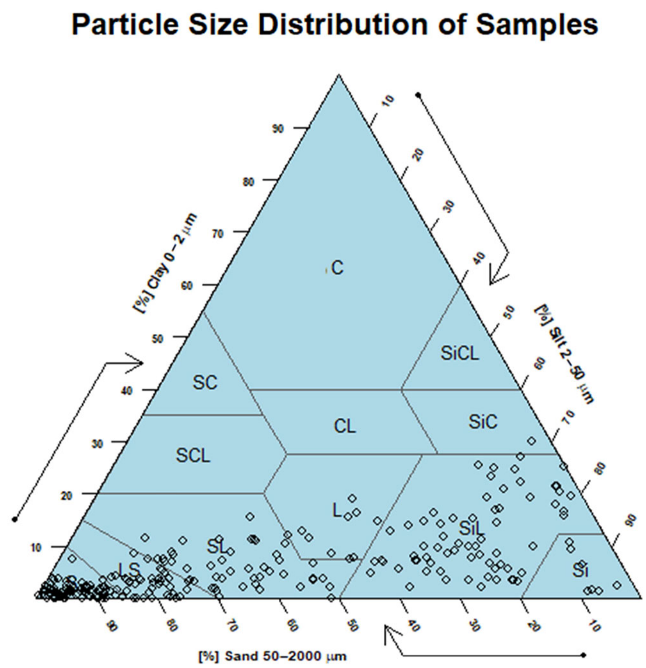

FIGURE 1 Particle size distribution of all sampled horizons ($n = 257$). Each marker on the plot is a single sample.

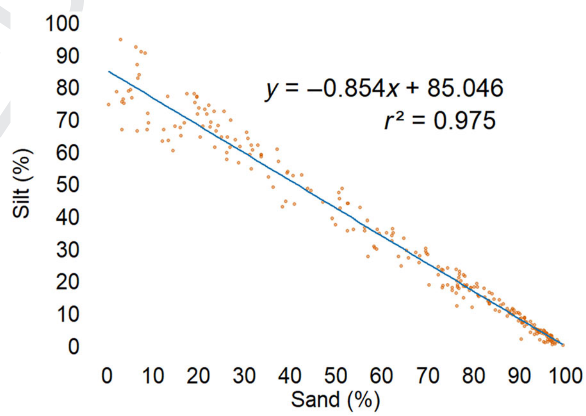

FIGURE 2 Linear relationship between sand content (%) and silt content (%) of mineral soil samples collected from Long Island Sound subaqueous soils ($n = 257$). A significant negative relationship ($p < 0.0001$) was found between sand and silt content.

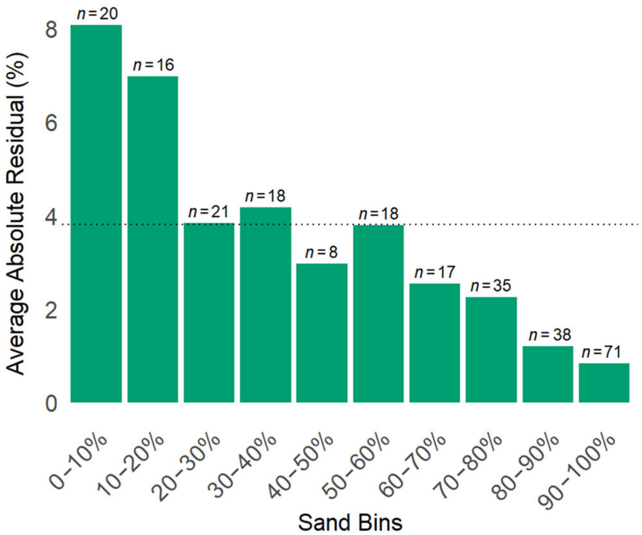

FIGURE 3 Average absolute residuals of predicted silt when utilizing the linear relationship presented in Figure 2. Samples were binned into 10 categories based on their sand content (%): 0–10, 10–20, 20–30, 30–40, 40–50, 50–60, 60–70, 70–80, 80–90, and 90–100. Dotted line (3.81%) indicates the average error using the pipette method for measuring percent silt between two professionals as reported by Centeri (2015). Sand bins 0%–10%, 10%–20%, 20%–30%, and 30%–40% have a higher error rate than 3.81%, indicating that silt contents in soils with ≤40% are not as well predicted by our model.

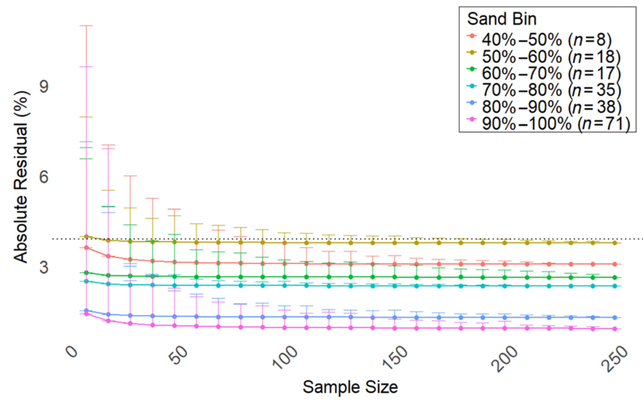

FIGURE 4 Results of random subsampling of our dataset to determine the minimum sample size for modeling purposes. Sand bins are grouped in 10% increments (40%–50%, 50%–60%, 60%–70%, 70%–80%, 80%–90%, and 90%–100%). Average absolute residual values for each bin are represented by the circles, and error bars show the maximum average absolute residual across 10,000 random subsamples. The sample size increased in increments of 10, ranging from 10 to 250 samples. Dotted line (3.81%) indicates the average error using the pipette method for measuring percent silt between two professionals as reported by Centeri (2015). All sand bins have an average absolute residual of <3.81% and a maximum average absolute residual of <4% when the sample size is 50 or greater.

Coates & Hulse, 1985; Faé et al., 2019; Yang et al., 2019). Since we used the pipette analysis to develop and test our model, we followed Centeri et al. (2015) and chose 3.81% as the maximum allowable average error in percent silt corresponding to the upper limit of 2 standard deviations of intra-method variability. Categorizing sand content into bins having increments of 10% (e.g., 0%–10%, 10%–20%), samples with at least 40% sand had AARs ranging from 0.80% to 3.58% (Figure 3), with an overall mean of 1.00%. In addition, none of the 187 (70%) samples with >40% sand differed in texture class between the modeled data and the pipette data. Thus, error analysis suggests that for SAS samples collected in Long Island Sound that have at least 40% sand, PSD can be accurately determined by measuring the sample's sand contents and predicting silt contents from the regression equation. The advantage of this approach is that time-intensive pipette analysis and pretreatments to remove salts are not required. Flocculation as a result of elevated concentrations of salts effectively only occurs in the clay fraction because the clay fraction is the dominant source of cation exchange capacity (Chibowski et al., 2011; Gregory & O'Melia, 1989; Sutherland et al., 2015). Thus, because salt removal is not necessary for sand content determination, this model saves a considerable amount of time without introducing more error than would already be expected from using traditional methods. If slightly more variability can be accepted (4.15%), PSD of samples with at least 20% sand may be ascertained almost as accurately.

Over 250 samples were analyzed to create the predictive PSD model for the 15,000-ha study area that was investigated and mapped in Long Island Sound. Did we need all 257 samples to create an effective model, or could we have used fewer samples? To answer this question, we utilized R to randomly subsample without replacement from the original 257 sample set a given number of samples from 250 to 10 at 10 sample intervals (e.g., n = 250, n = 240, n = 230) with 10,000 iterations (Figure 4). The average and maximum AAR values showed little change with a decreasing sample size up to 50 samples. At 50 samples, the maximum AAR value was still <4%, suggesting that as few as 50 samples (20% of the total samples) could be used to create an effective model for determining PSD of samples with >40% sand in future studies.

We questioned why the amount of variation increased with a decrease in sand content, especially in samples with <20% sand. Some of this could be a function of flocculation of the clay fraction in the haline waters (Chibowski et al., 2011; Gregory & O'Melia, 1989; Sutherland et al., 2015). We also suspected that the number of diatoms in the samples may be an issue since diatom skeletons occur primarily in the silt fraction. For example, soils classified as silt contained nearly 9% diatom skeletons among their mineral particles (Figure 5). The greater abundance of diatoms in the samples with silt loam and silt textural classes may be why the

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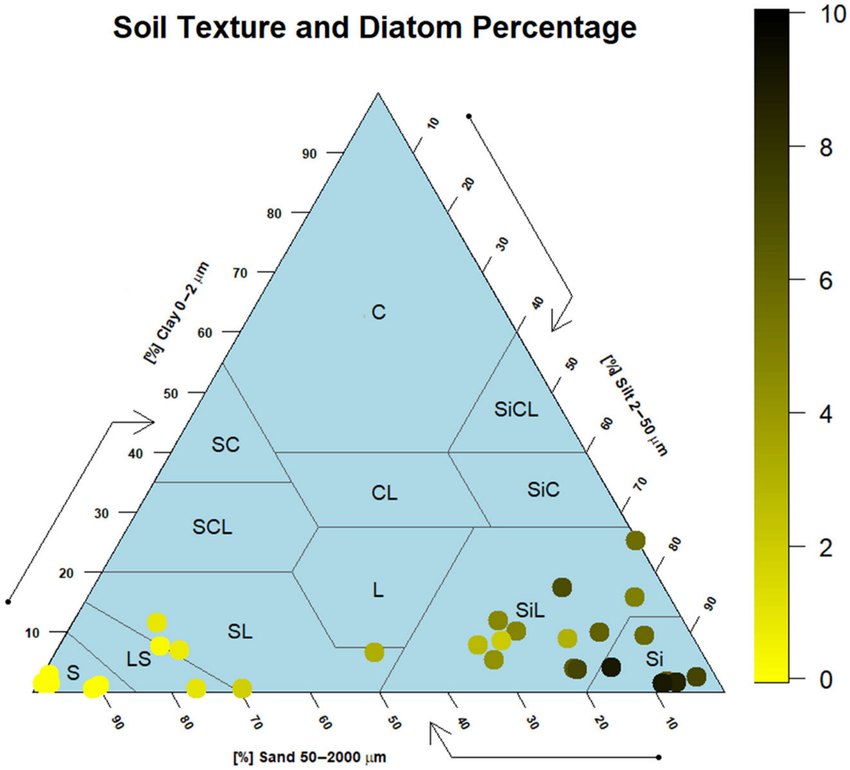

FIGURE 5 Number of diatoms relative to different USDA soil texture classes. Each circle on the plot is one sample (n = 30), and the color gradient represents varying diatom content (%) of the total mineral primary particles across the soil samples.

predicted silt residuals are so much higher than those in samples with more sand. Diatoms are less dense than most primary soil particles (Miklasz & Denny, 2010; Skopp, 2000), and the sinking speed of diatoms can vary significantly by species (Miklasz & Denny, 2010). Most marine diatoms are elongate in shape rather than the relatively spherical mineral particles assumed in Stokes' law. Thus, diatoms are expected to settle more slowly in the water column, requiring less energy to remain suspended compared to similarly sized mineral silt particles (Miklasz & Denny, 2010). As a result, an abundance of diatom skeletons may disrupt the silt–sand relationship expected in subaqueous environments, particularly in lower-energy aqueous environments.

4 | CONCLUSIONS

This study demonstrates a clear relationship between sand and silt contents in the PSD of SAS of the Long Island Sound. Our study found that for soil samples with > 40% sand, measures of total sand can be used to accurately determine PSD of the silt fraction. In SAS samples with lower sand content, the presence of diatom skeletons appears to result in greater variability in PSD, reducing the model's predictive accuracy. Diatoms remain suspended for longer periods and settle more slowly than similar-sized mineral particles, which further complicates PSD modeling in these soils. For these finer-textured samples, pipette, hydrometer, or other alternative methods, such as laser diffraction, are likely required for accurate PSD determinations.

Considering that 70% of the 257 horizons we measured PSD had >40% total sand, being able to model PSD for sandy loam, loamy sand, and sand-textured SAS samples by only measuring the sand fraction greatly reduces the need for more time-consuming analysis and pretreatments. Our findings have practical implications for soil survey field offices or soils laboratories that are providing characterization data for survey efforts of sandy SAS, especially those in high-energy estuarine environments where salts can complicate analyses. Compared to other alternative methods of PSA, our proposed method of PSA yields results with similar accuracy but without the need for costly instruments such as laser diffractometers or pressure transducers. Current yearly work plans for nationwide coastal surveys include goals of mapping SAS in North Carolina, Florida, Mississippi, Texas, Alabama, and Louisiana, encompassing over 125,000 ha (USDA-NRCS, 2024). Our study found that for large datasets, as few as 20% of samples can be used to develop an effective PSD model. Given the potential time and cost savings, this model can be a valuable tool for soil surveyors when the goal is rapid, broad-scale soil mapping in subaqueous environments.

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AUTHOR CONTRIBUTIONS

Joseph V. Manetta: Data curation; formal analysis; investigation; methodology; validation; visualization; writing—original draft; writing—review and editing. **Mark H. Stolt**: Conceptualization; funding acquisition; investigation; methodology; project administration; resources; software; supervision; validation; visualization; writing—review and editing.

CONFLICT OF INTEREST STATEMENT

The authors declare no conflicts of interest.

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