# Federal Interagency Sedimentation Project Call for Ideas 2021

## Idea Title:

The Role of Chemical and Physical Dispersive Pre-treatment Methods on Particle Size Distribution Using Laser Diffraction.

# Principal Investigator:

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## Additional Investigators:

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# PI Location and Study Location:

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#### Introduction:

Laser diffraction methods to size particles have gained wide popularity and acceptance due to its measurement resolution, reproducibility, and rapidity, especially compared with traditional sieve and sedimentation methods. It is well understood that the measurement of particle "size" cannot be decoupled from the instrument or technique used to obtain the measurement due to differences in the properties being measured (Konert and Vandenberghe, 1997; Buurman et al., 2001; Blott and Pye, 2006; Rodriguez and Uriate, 2009; Ferro and Mirabile, 2009; Di Stefano, et al., 2010, and many others). Furthermore, while standard operating procedures have been published through the International Standards Organization (ISO) (2009) and the American Society for Testing and Materials (ASTM) (2000) for laser diffraction methods, they are somewhat general and provide little guidance on sample preparation. According to Merkus (1997), the primary errors associated with sample preparation using laser diffraction are reported to be poor subsampling and poor dispersion, each of which are a function of particle size. Sample preparation may imply a combination of procedures aimed at achieving sample representativeness with low variability between subsamples. These procedures may include removal of certain constituents (e.g., organic carbon and calcium carbonate), sampling and subsampling, and dispersion. This proposal seeks to quantify changes in the size distribution brought by differences in sample preparation, namely disaggregation methods of the silt and clay fraction prior to analysis.

# Background:

The US Army Corps of Engineers (USACE) collected and analyzed bed sediments from the Lower Mississippi River in 1932, 1989, and 2013 as part of the Corp's continuing potamology program. The purpose of the most recent study was to characterize changes in sediment texture in response to the meander cutoff program and ongoing river control structures and protective works since the 1930's. The latest data collection and analysis effort was intended to match that of the previous studies.

Data analysis for the 2013 study was performed at the Corp's Engineer Research and Development Center, Coastal and Hydraulics Laboratory (ERDC-CHL), Vicksburg, Mississippi. An independent quality control check led by the United States Geological Survey's Kentucky Water Science Center Sediment Laboratory (USGS-KY) revealed differences in certain sediment size classes, attributed early to differences in measurement techniques between the two labs. Whereas the USGS-KY lab used the traditional sieve-pipet method (SP), the CHL sediment lab used a combination of sieving and laser diffraction (LD) to size particles. To investigate further, a polydisperse natural sediment sample was analyzed by ten sediment laboratories in a pilot project to quantify the variability resulting from their preferred sizing methods. Table 1 lists the participating laboratories and their respective methodologies (as of 2015).

TABLE 1. LIST OF PARTICIPATING AGENCIES AND EQUIPMENT USED TO OBTAIN PARTICLE SIZE DISTRIBUTIONS.

Agency/Laboratory/Location	ID	Method	Equipment
USGS Center for Sediment Forensics and Excellence, Reston, VA	A	Laser diffraction	Beckman Coulter, LS 13-320
USGS Hydrological Research Lab, Salinas CA	В	Laser diffraction	Beckman Coulter, LS 13-320
USGS Grand Canyon Monitoring & Research Center, Flagstaff, AZ	С	Laser diffraction	Beckman Coulter, LS 13-320
USGS CVO Sediment Lab, Vancouver, WA	D	Wet sieve/Sedigraph	SediGraph 5120 (Micrometrics) 1997 model
USGS Water Science Center, Iowa City, IA	E	Wet sieve/Sedimentation	Sieves, visual accumulation tube, graduated settling column
USGS Woods Hole Coastal & Marine Sci. Ctr., Woods Hole, MA	F	Electroresistance ESZ	Coulter Multisizer 3
Sequoia Scientific, Inc., Bellevue, WA	G	Laser diffraction	LISST-100X
USGS Water Science Center, Louisville, KY	н	Wet sieve/Sedimentation	Sieves, graduated sedimentation column
USGS Water Science Center, Albuquerque, NM	ı	Wet sieve/Sedimentation	Sieves, graduated sedimentation column

TABLE 2. AVERAGED GRAIN SIZE STATISTICS FOR EACH LAB (A-J; SEE TABLE 1 FOR REFERENCE) DERIVED FROM THE CUMULATIVE DISTRIBUTIONS.

				D							J2
D <sub>10</sub>	3	4	*	*	*	3	5	*	*	12	10
D <sub>50</sub>	44	44	*	29	47	36	42	49	52	80	70
$D_{90}$	224	197	178	190	176	194	151	207	216	255	301

The results showed moderate variability between laboratories using various methods. The variability can be attributed to a number of factors including 1) the potential inhomogeneity of sub-samples split from the primary sample, 2) differences in the techniques utilized by each method to measure grain size, and 3) differences in sample preparation prior to analysis.

Sample preparation may have a considerable effect on size distributions. Stojanovic (2010) reported that poor sample preparation attributes to half of the problems encountered when measuring different samples. Techniques and procedures for sample splitting and disaggregation may vary between laboratories allowing for an uncontrolled source of error.

# Purpose and Scope:

Samples containing significant quantities of mud require additional mechanical and chemical dispersion techniques such as ultrasonication (Gee and Bauder, 1986) and adding surfactants (Chappel, 1998) to disaggregate and prevent the reformation of clay flocs. Care must be taken, however, as excessive ultrasonic energy can break up particles (Chappel, 1998) or cause air bubbles to become trapped in the dispersing liquid, especially if using a chemical dispersant (Sabin, 2011; Mingard et al., 2009). Chappel (1998) suggested that ultrasonic action prior to measurement should be limited to 6 minutes. However, DiStenfano et al. (2010) did not find significant differences in PSDs using different ultrasonication times between 228 soil samples. The experiment will help to resolve these discrepancies in findings. While the effects of sonication time on PSDs have been examined, to the author's knowledge the effects of sonication intensity have not. The results will be used to help refine sample preparation procedures for LD measurements.

## Technical Requirements:

The proposed experiment intends to test the effects of dispersant and sonication treatments on PSDs. The testing will occur in two phases: Phase 1 will evaluate the performance of a cone splitter following USGS procedures (Wilde et al., 2014). Splits from a primary sample of known concentration will be analyzed for sub-sample concentration and size distribution variability. Phase 2 will assess different dispersing and disaggregation methods using known masses of fine sand-mud mixtures. The test cases will investigate the effects of two dispersant concentrations, two sonication times, and two sonication intensities.

#### Deliverables:

Peer-reviewed publication within an open access journal.

#### Timeline:

The proposed duration of the project is expected to be on the order of six months. However, work may not commence immediately upon receipt of funds, which may be delayed by scheduling constraints with prior work obligations and field deployments, particularly for the engineering technician conducting the laboratory tests. The project is expected to be completed with a journal paper submitted for peer review at least by the end of the fiscal year.

Month 1: Planning and sample preparation

Months 2-3: Experiment execution

Months 3-5: Data analysis and reporting

Month 6: Publication submission

## Budget:

# The requested funds are \$45K.

The experiment leverages in-house instruments, materials, and expertise. The laser diffraction system used is the newly acquired Malvern Mastersizer 3000. The USGS cone splitter and filtration equipment are already on hand. The materials proposed for the experiment include Vicksburg silt, which has a narrow and predictable size distribution, and stock supplies of mud from previous sediment collection efforts. Therefore, no equipment or material purchases are needed.

## The cost estimate is itemized as follows:

O/A Labor 3%	\$ 1,150.61	FTE Labor
Mob/Demob & Planning Labor	\$ 2,024.04	FTE Labor
Labor in Field	\$ -	FTE Labor
Data Processing Labor	\$ 4,048.09	FTE Labor
Sample Analysis Labor	\$ 12,041.16	FTE Labor
Report writing Labor	\$ 16,192.34	FTE Labor
	\$ 4,048.09	
Purchases	\$ 250.00	Purchase Orders for Materials and Services
FTE Pd/Travel	\$ -	FTE Travel
Contract Labor/ Boat Rental	\$ -	Contract Labor/Travel/
Equipment Usage	\$ -	Fleet Vehicles, Vessels, Instrumentation
Overhead	\$ 2,475.49	
<b>Publication Fees (estimated)</b>	\$ 2,500	

TOTAL \$ 44,729.82

# Proposed funding allocation and major phases

Planning, sample preparation, and experiment execution	\$15,500
Data processing and reporting	\$20,000
Tech transfer, publication fees, virtual presentation of results	\$10,000

# Unique Qualifications:

The ERDC-CHL sediment laboratory is certified for quality control by the USGS Water Science Center, Louisville, Kentucky. The sediment laboratory has processed thousands of sediment samples for concentration and size gradation, often using a combination of dry/wet sieve and laser diffraction techniques. The laboratory has recently acquired the latest Malvern laser diffraction instrument at a cost of \$50,000. The PI and Co-PI have advanced degrees in geology and the earth sciences and utilize sediment characterization in their regular research involving erosion behavior and transport dynamics, particularly with cohesive sediments.

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