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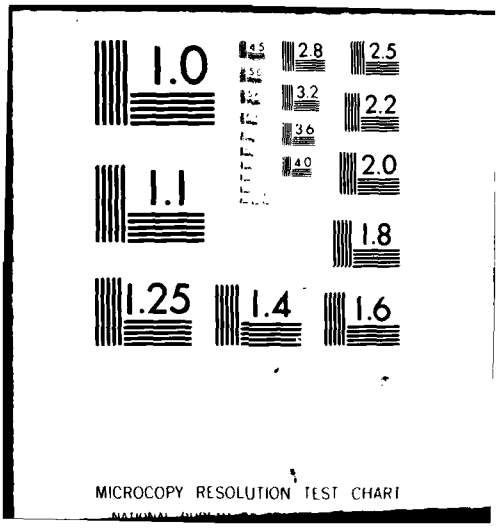
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(6) POWDER CHARACTERIZATION OF WATERWAYS EXPERIMENT STATION NMFC 196500 WATER SAMPLES.

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(10) NORMAND D. CORBIN [redacted] JAMES W. McCAULEY
CERAMICS RESEARCH DIVISION

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1. REPORT NUMBER: AMMRC TR 81-9	2. GOVT ACCESSION NO.: AD-A098391
3. RECIPIENT'S CATALOG NUMBER	
4. TITLE (and Subtitle): POWDER CHARACTERIZATION OF WATERWAYS EXPERIMENT STATION NMFC 196500 WATER SAMPLES	5. TYPE OF REPORT & PERIOD COVERED: Final Report
	6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s): Normand D. Corbin and James W. McCauley	8. CONTRACT OR GRANT NUMBER(s)
9. PERFORMING ORGANIZATION NAME AND ADDRESS: Army Materials and Mechanics Research Center, Watertown, Massachusetts 02172, DRXMR-EO	10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS: Intra-Army Order WESRF 79-288
11. CONTROLLING OFFICE NAME AND ADDRESS: U.S. Army Engineer Waterways Experiment Station, Vicksburg, Mississippi 39180	12. REPORT DATE: February 1981
	13. NUMBER OF PAGES: 18
14. MONITORING AGENCY NAME & ADDRESS(if different from Controlling Office)	15. SECURITY CLASS. (of this report): Unclassified
	15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Approved for public release; distribution unlimited.

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Sediments
Water analysis
Particle size
Emission spectroscopy
X-ray spectroscopy
Chemical analysis
Electron microscopy

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

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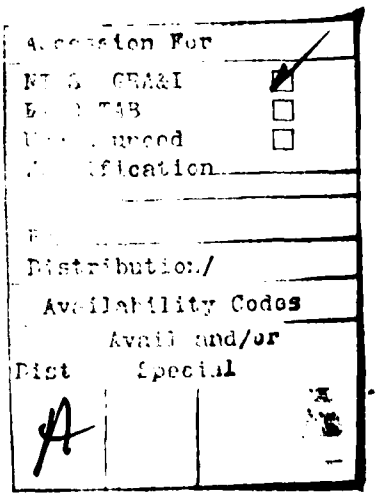
ABSTRACT

The particulate matter of thirty water samples collected near the Waterways Experiment Station in Vicksburg, Mississippi, have been characterized. The investigation included ten samples from each of three different locations: the Mississippi River, Yazu River, and Brown's Lake. Characterization was conducted using particle size distributions, surface areas, scanning electron microscopy, differential thermal analysis, energy dispersive X-ray analysis, X-ray diffraction, and emission spectroscopy. Major differences were noted between all three powder populations.

PREFACE

This final report documents work performed by the Ceramics Research Division of the Metals and Ceramics Laboratory at the Army Materials and Mechanics Research Center, Watertown, Massachusetts, under Intra-Army Order #WESRF 79-288.

The program was administered by the U.S. Army Engineer Waterways Experiment Station in Vicksburg, Mississippi, under technical monitor Dr. K. Thornton. The thirty water samples were collected by Mr. J. Carroll. Also contributing to the work reported herein were Messrs. T. Sheridan (X-ray diffraction), B. Strauss (emission spectroscopy), A. E. Ingram (surface area), A. Connolly (scanning electron microscopy), and C. Bonavita (SEM size analysis).



CONTENTS

SUMMARY

The particulate matter of thirty water samples from the Mississippi River, Yazu River, and Brown's Lake have been characterized. The results are summarized as follows:

Mississippi River - bimodal distribution: 15 wt% averaging 13 μm, 85 wt% averaging ∿0.1 μm; surface area 4.01 m^2/g.

Yazu River - single broad distribution averaging 0.3 μm; surface area 2.36 m^2/g.

Brown's Lake - bimodal distribution: 10 wt% averaging 11 μm, 90 wt% averaging 0.2 μm; surface area 8.07 m^2/g.

OBJECTIVE

To characterize particulates in NMFC 196500 water samples: 30 samples, ten each from the Mississippi River (MR-1 to MR-10), Yazu River (YR-1 to YR-10), and Brown's Lake (BL-1 to BL-10).

APPROACH

Sample Preparations

All 30 samples had to be dried in order to perform the required analysis. The following procedure was used:

1) pipetting off all but 20 ml of water from the settled samples, leaving the undisturbed sediment at the bottom of the jars;

2) shaking, to mix, the remaining 20 ml of water and sample;

3) pouring the mixed sample into a drying dish;

4) rinsing the jar with about 10 ml of distilled water and also pouring into the drying dish;

5) drying in an oven at 90°C for about 8 hours or until the sediment is thoroughly dried; and

6) breaking the dried sample, which is in the form of large thin sheets, with a spatula.

Some tests required combining several samples together to provide enough material for the test. Table 1 shows which samples were analyzed for each test.

Physical Characterization

Particle Size Distribution

Particle size distributions of each sample was determined with a Sedigraph (5000D, Micromeritics Instrument Corporation).

Table 1. WATERWAYS EXPERIMENT STATION

Sample	Sedigraph Traces	Surface Area*	SEM	Emission Spec*	XRD	DTA*
BL						
1	x					
2	x		x			
3	x	x		x		x
4	x				x	
5	x	x		x		x
6	x	x		x		x
7	x	x		x		x
8	x	x		x		x
9	x	x		x		x
10	x					
YR						
1	x					
2	x		x			
3	x	x		x		x
4	x				x	
5	x	x		x		x
6	x	x		x		x
7	x	x		x		x
8	x	x		x		x
9	x	x		x		x
10	x					
MR*						
1, 2	x	x	x	x		x
3, 4	x				x	
5, 6	x	x		x		x
7, 8	x	x		x		x
9, 10	x	x		x		x

*Samples combined for analysis

Most samples were run separately in the Sedigraph; however, MR samples had to be doubled up to obtain enough material for analysis. The samples were very difficult to disperse in water with the result that the initial data was affected by problems with flocculation and agglomeration of particles. The following procedure was adopted which produced very good results.

1) A surfactant consisting of a 0.1 weight percent solution of sodium pyrophosphate in deionized water was used.

2) The sample and solution were mixed with a magnetic stirrer for approximately 20 minutes.

3) The mixtures were then further diluted with the surfactant to near the limit of Sedigraph sensitivity in order to remedy flocculation problems.

4) The sample and solution mixture was put into an ultrasonic bath for 20 minutes.

5) A solution of NaOH was added until the mixture pH was about 10.5.

6) The mixture was reagitated for an additional 10 minutes in an ultrasonic mixer.

To use the Sedigraph a value for the density of the particles making up the samples must be known. We assumed a value of 2.60 g/cc, because it was thought to be representative of the average density for alpha quartz and clay materials.

Surface Area

The surface area was determined by the multiple point BET method.[1] The samples were prepared by drying as previously described. Several samples had to be combined to obtain enough sediment for an accurate surface area determination.

Scanning Electron Microscopy (SEM)

In order to observe individual particles under the SEM, a *very* dilute solution of each sample had to be specially prepared, using the same technique as for the Sedigraph analysis. This resulted in the breakdown of the large, agglomerated dry fragments. A drop of this solution was then placed on a warmed SEM sample holder. The water was allowed to evaporate, leaving a fairly well dispersed sediment sample. This sample was then coated with a thin (~400 Å) gold-palladium alloy to prevent charge buildup on the sample.

The frequency percent by number histogram distributions were derived from photographs of the samples at 2000X. The method for measuring the distribution used several different photographs of each sample and an overlay grid. The size of the particle at regular grid intervals was recorded with up to 100 counts taken on each photograph.

Chemical Characterization

Emission Spectroscopy

Semiquantitative emission spectroscopy was conducted on all three sediment samples. The samples for emission spectroscopy were dried, as previously described, and consisted of mixtures of several samples.

X-Ray Diffraction

X-ray diffraction analysis was performed by using CuKα radiation with a nickel filter, setting the sensitivity to 500 cps and starting at a two theta angle of five degrees. In all cases the samples were smear mounted on glass slides. (Recessed plastic holders were tried but the results were of poor quality.)

SEM Energy Dispersive X-Ray Analysis

The preparations of samples for this analysis are identical to the SEM sample preparation.

Using energy dispersive X-ray analysis (EDAX) on the scanning electron microscope, semiquantitative chemical analyses were obtained on individual particles within the samples. Two measurements were made on each sample: one on a large particle and the other on the fine matrix phase.

1. BRUNAUER, S., EMMETT, P. H., and TELLER, E. *The Adsorption of Gases in Multi-Molecular Layers.* J. Am. Chem. Soc., v. 60, 1938, p. 309.

The measurements are recorded as electron volts (energy of characteristic X-rays) versus counts, with the energy levels yielding unambiguous elemental analysis, while their relative concentrations are obtained by peak (count) height measurements. The characteristic voltages (eV) of the following elements were used in the analysis: 1.48 Al, 1.73 Si, 2.10 K, 6.30 Fe, and 7.95 Cu. Other elements, 2.0 eV Au and 2.75 eV Pd, were present due to their use in sample preparation.

Differential Thermal Analysis

Differential thermal analysis (DTA) was carried out using a DuPont 990 Thermal Analyzer. A sample weight of 0.03 g (30 mg) was used with a heating rate of 5°C/min against an αAl_2O_3 reference sample of similar weight. The samples were heated from room temperature to 940°C to trace any organic peaks occurring in the sample. Upon reaching 940°C the furnace was allowed to cool to 400°C. The sample was then reheated to 700°C. For this second heating the instrument sensitivity was increased to observe the weak alpha-to-beta quartz transformation peak at ∿600°C.

RESULTS AND DISCUSSIONS

Physical Characterization

Macroscopic Characterization

The as-received samples contain some fairly large [>0.1 mm (100 μm)] fragments of multicolored material. These fragments appeared stringy in nature and clogged up the tubing used for particle size analysis. When dried, the samples had brown to gray color tones with the MR samples darkest and BL samples the lightest. Under low-power (100X) magnification, the samples appeared to have large, clear particles with sharp corners lying on brown powdery material. The possibility of two different phases, each having a different size, became immediately apparent.

Particle Size Distribution

Figure 1 illustrates the experimental range (experimental variation) of results obtained on all samples. The data are plotted as cumulative mass percentage less than a a particular size versus log of equivalent spherical diameter. The effects of Brownian motion are pronounced for particles less than 1.0 μm, making the experimental variation of results for these particles noticeably larger. Roughly 25 wt% of all samples had an equivalent spherical diameter (ESD) ∿0.1 μm. None of the samples had particles larger than 40 μm.

Other graphical representations were produced using an average distribution for each sediment sample. By taking the slope along the average cumulative mass percent curves [Δ wt% ÷ Δ log ESD (μm)], a frequency curve can be obtained as shown in Figure 2. Figure 3 presents lognormal plots of the three samples, using an average of the original Sedigraph data. The straight lines show the populations within each sample if lognormal distributions are assumed. The idealized coarse and fine population lognormal distribution lines are calculated in the following way: the actual frequency distribution median values and standard deviations are iteratively estimated from the frequency distribution until idealized frequency distribution curves closely approximate the actual data.

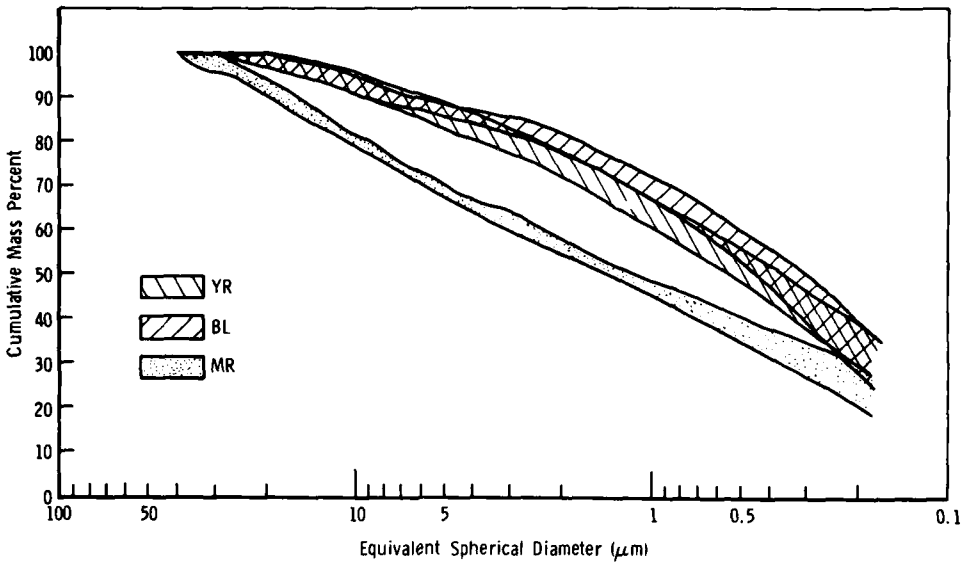


Figure 1. Ranges of X-ray Sedigraph traces of WES samples.

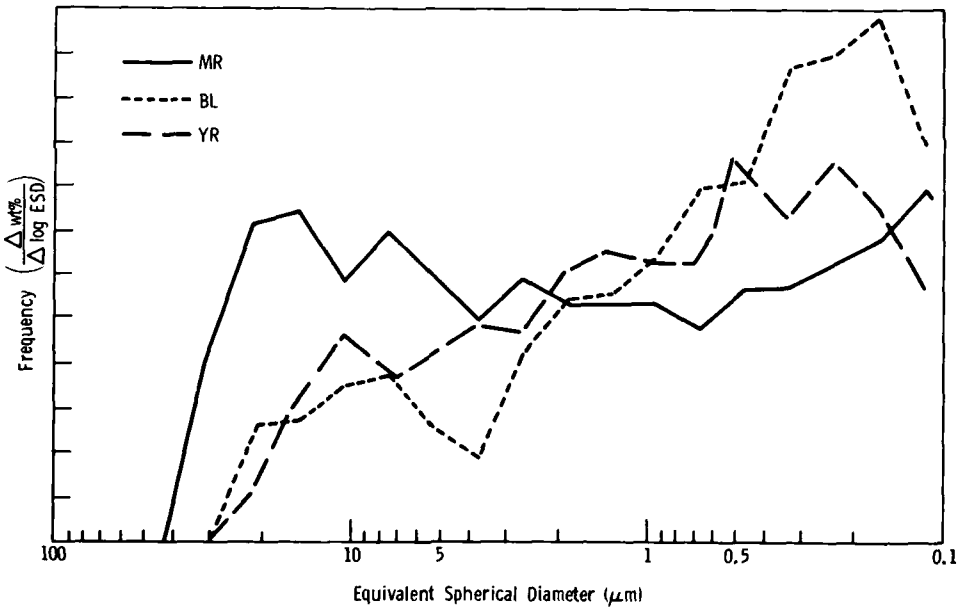
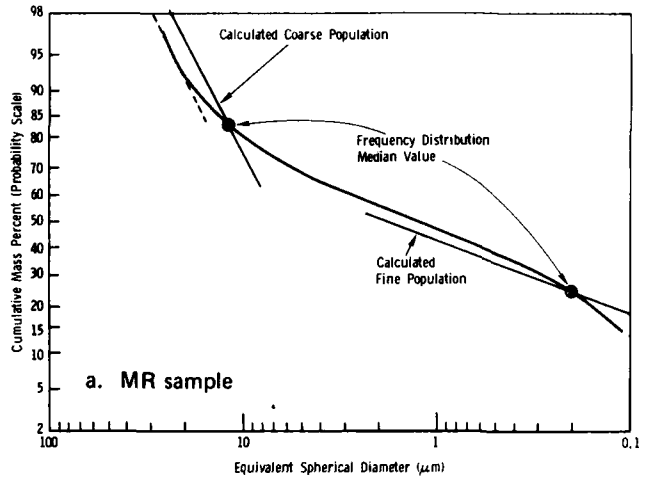
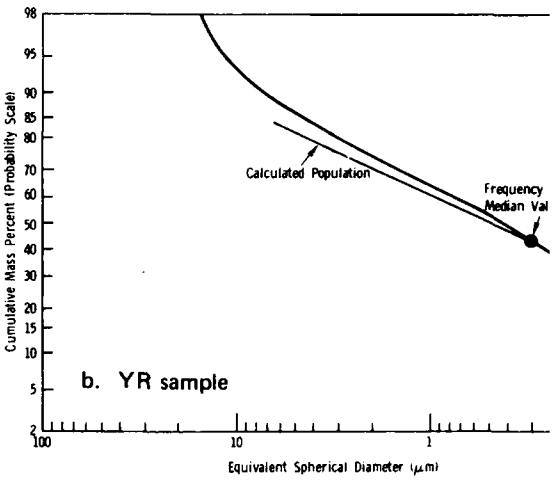


Figure 2. Unsmoothed frequency distribution curves calculated from Sedigraph traces.

Using the lognormal distribution equations (presented in Table 2), and the *idealized* lognormal population means and standard deviations (Table 3), a smooth frequency distribution plot can be determined (Figure 4). This matches very well with the frequency plot from the raw data (Figure 2).

The idealized Sedigraph results, summarized in Table 3, clearly show differences between all three samples. Samples MR and BL contain two distinct populations: coarse fractions having medians about 11 μm and fine fractions with medians of about 0.2 μm.





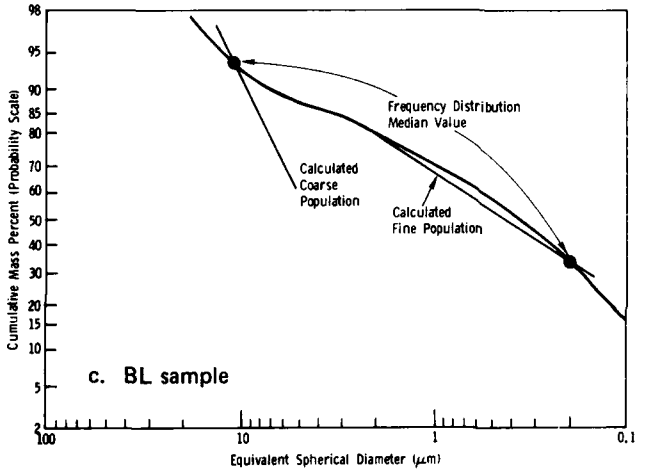


Figure 3. Actual and calculated lognormal distributic samples plotted on log-probability paper.

Table 2. LOGNORMAL PARAMETERS AND EQUATIONS

d_g (c) = geometric median diameter for coarse population
d_g (f) = geometric median diameter for fine population
σ_g (c) = geometric standard deviation for coarse population
σ_g (f) = geometric standard deviation for fine population
f (c) = weight fraction of sample in coarse population
f (f) = weight fraction of sample in fine population
f (c) + f (f) = 1

1) Normal Gaussian distribution equation:

$$f(x) = \frac{1}{\sigma\sqrt{2\pi}} \exp\left[-(x-\mu)^2/2\sigma^2\right]$$

2) Lognormal Gaussian distribution equation used for analysis of powders less than 100 μm:

$$f(x) = \frac{1}{(\log \sigma_g)\sqrt{2\pi}} \exp -[2-(\log d_g)]^2/2(\log \sigma_g)^2$$

3) Lognormal bimodal Gaussian distribution equation used for analysis of powders less than 100 μm:

$$f(x) = \frac{f(c)}{(\log \sigma_g(c))\sqrt{2\pi}} \exp -[2-(\log d_g(c))]^2/2(\log \sigma_g(c))^2 + \frac{f(f)}{(\log \sigma_g(f))\sqrt{2\pi}} \exp -[2-\log d_g(f)]^2/2(\log \sigma_g(f))^2$$

Table 3. SUMMARY OF SEDIGRAPH RESULTS

	Mode 1			Mode 2		
	d_g (c)	σ_g (c)	Percent of Sample*	d_g (f)	σ_g (f)	Percent Sampl
BL	11 μm	1.78	10	0.2 μm	6.3	90
YR				0.3 μm	15.85	100
MR	12 μm	1.99	15	0.2 μm	19.95	85

*Estimated

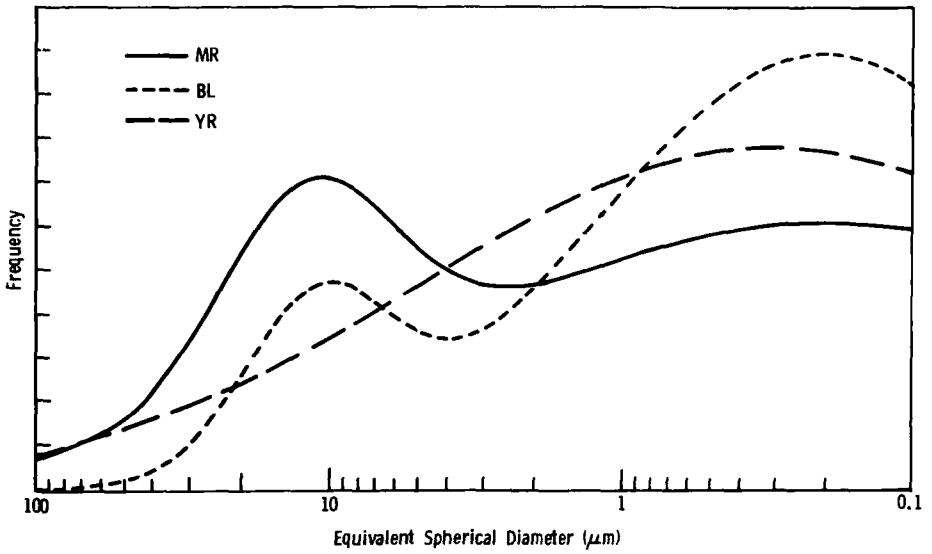


Figure 4. Idealized lognormal frequency distribution curves.

The YR sample appears to have only one very broad population present, with a median value of 0.3 μm. These idealized results, however, are slightly different from data extracted from Figure 2. From this figure the following results are deduced:*

BL: two populations: 10 wt% averaging 11 μm; 90 wt% averaging ∿0.16 μm;

YR: single broad distribution averaging 0.3 μm; and

MR: two populations: 15 wt% averaging 13 μm; 85 wt% averaging ∿0.1 μm (the experimental limit of the Sedigraph).

Surface Area

The surface area results are given in Table 4. Ideal spherical diameters assuming a density of 2.6 g/cc were calculated and are also listed in the table. Interpretation of surface area values of multimode populations is extremely difficult. At first glance the results do not seem to compare with the Sedigraph data; however, closer inspection of Figure 4 reveals reasonable rationale. The high surface area of the BL sample can be ascribed to the large population of the fine fraction as compared to the other two.

Table 4. SURFACE AREA DATA

			X-Ray Sedigraph	
	S_W (m^2/g)	d (S_W°), μm	d_g (f), μm	Percent of Population
BL	8.07	0.286	0.2	90
YR	2.36	0.977	0.3	100
MR	4.01	0.576	0.2	85

*Equivalent spherical diameter assuming 100% monospheres and ρ = 2.6 g/cc

*The percentages are deduced from the total area under the curve, not from relative peak heights.

The MR sample has a relatively large fraction of larger size particles, suggesting a low surface area; however, the large amount of the <0.1 μm material contributes significantly to the total surface area. The surface irregularity (deviation from sphericity) of the individual particles can also significantly affect the results.

SEM Analysis

Scanning electron microscopy (SEM) photographs ranging in magnifications from 20X to 10,000X are shown in Figures 5, 6, and 7. The 1,000X magnification photographs

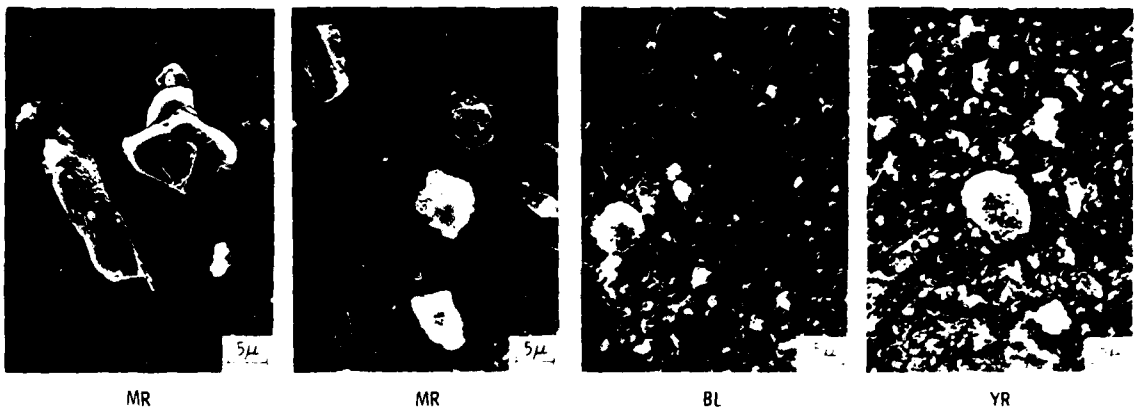


Figure 5. SEM photographs of large particles.

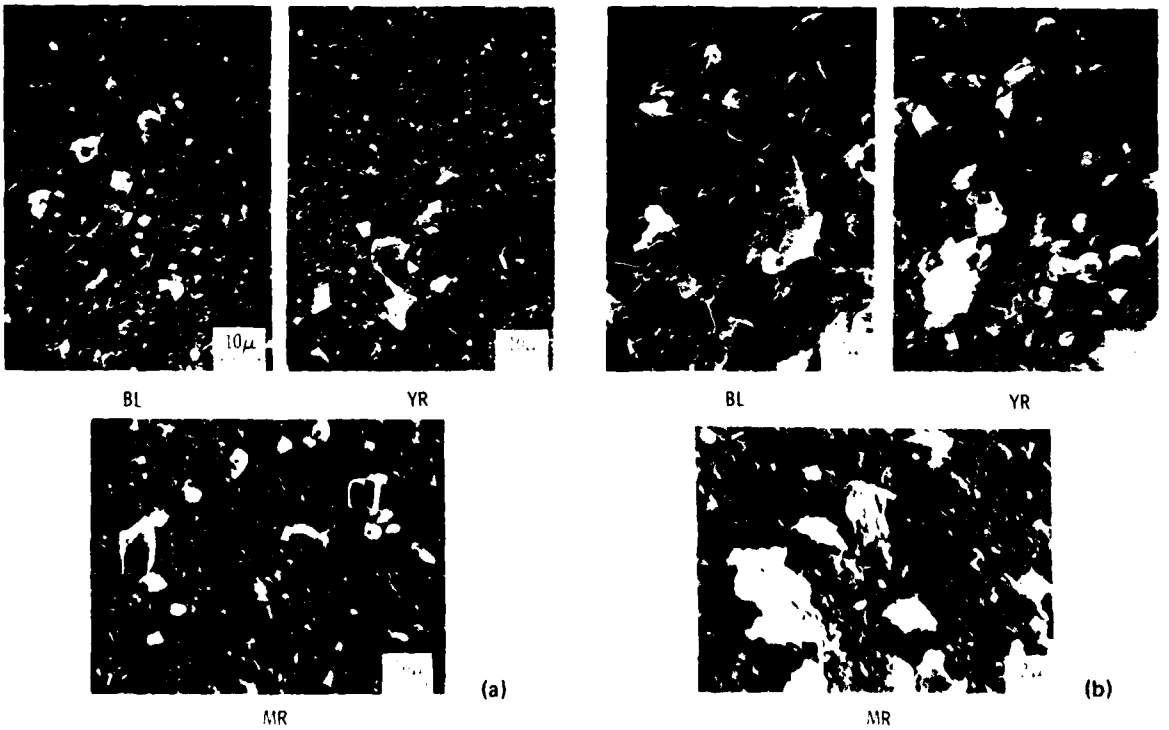


Figure 6. SEM photographs of matrices.

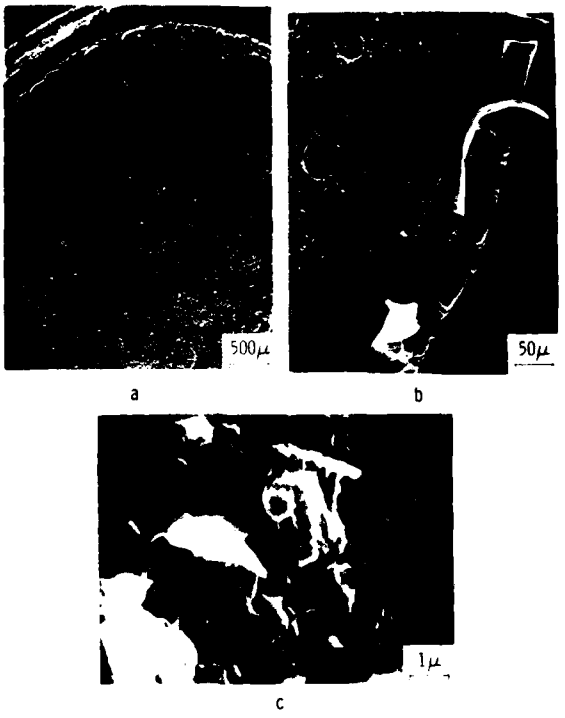


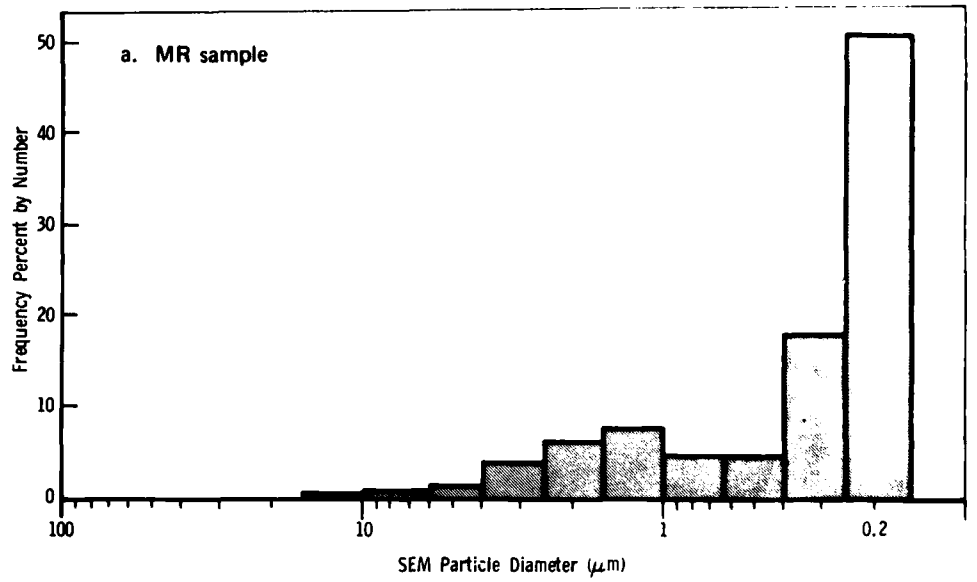
Figure 7. MR sample (a); YR sample showing organic rim (b); and MR sample showing platy structure of matrix (c).

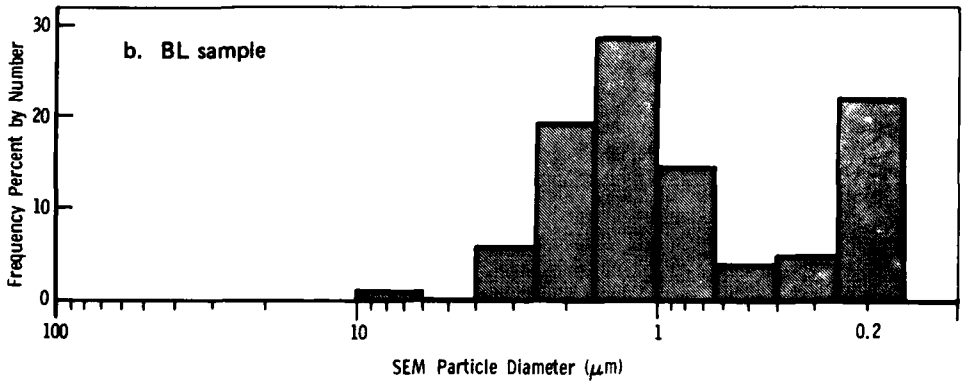
(Figure 6a) show the MR sample containing larger particles (∿12 μm) than BL or YR. The MR sample also appears to have two very distinct populations present, one having particles near 10 μm while the other contains particles less than 0.1 μm, confirming the Sedigraph results. The BL and YR samples also contain particles over a broad range of sizes, with the largest being 10 μm. These two samples contain more intermediate sized particles, near 4 μm, than MR, which gives them a coarser appearance. Figure 6b shows 5,000X photographs of the matrix phase for each sample.

The platy structure of particles in the 1-μm size range can be readily observed in Figure 7c. The 200X photograph (Figure 7b) shows the outer cracked rim which forms when the sample is prepared. It is presumed the rim contains predominantly organic matter from the sample.

The large particles in Figure 5 are determined to be quartz by energy-dispersive X-ray analysis. Particle size distributions were determined by using the SEM photographs. Figure 8 presents histograms plotted as the number percent of particles in a size range versus particle diameter. The frequency block at 0.2 μm represents all particles counted that were less than this value. Again the multimode characteristics of the powder distribution can be observed. MR has the largest percent of particles less than 0.2 μm, while BL has the largest percent of particles in the 1.0-μm range. All three samples show two populations present, one near 1.5 μm and the other at less than 0.2 μm.

Comparison of these histograms to the Sedigraph frequency distribution plots in Figure 4 reveal an interesting relationship, often overlooked. While the microscope particle size distributions frequency is in number percent, the Sedigraph distribution





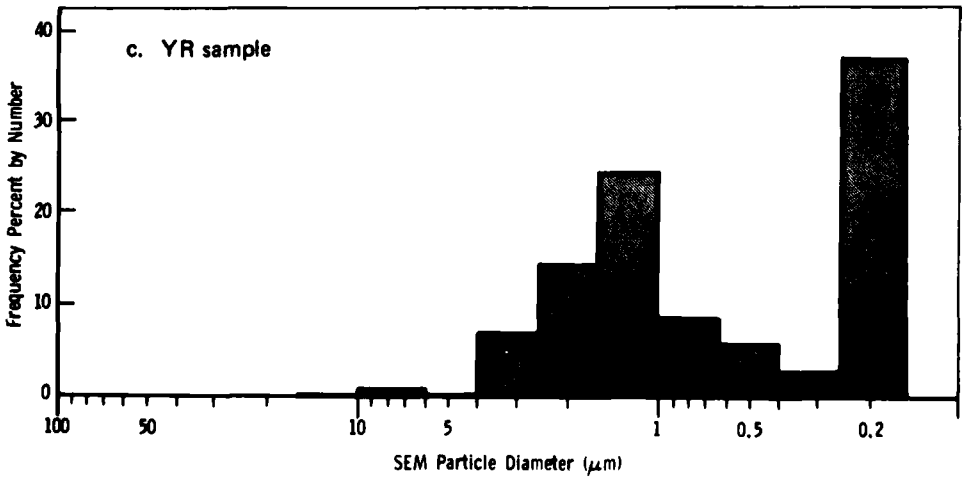


Figure 8. Histograms of percent by number from SEM of samples.

is in terms of weight percent. The weight percent distribution can easily be converted into a number percent distribution, assuming ideal lognormal single-mode distribution. In these terms, the microscope and Sedigraph distributions agree within reasonable limits. The following example will illustrate this point: assume a population of 1001 spheres consisting of one 10-μm particle and a thousand 1-μm particles. The total weight of the small particles is equal to the weight of the single large particle. Therefore, the coarse fraction makes up 50% by weight of the sample. In the number percent calculation, the large particle is counted only once so it makes up only 0.1% by number of the sample.

Chemical Characterization

Emission Spectroscopy

Semiquantitative emission spectroscopy results on all three samples are listed in Table 5. No major differences were noted in the major chemistry. Silicon, iron, and aluminum are the major elements, while calcium, magnesium, and sodium also significantly contribute to the overall chemistry. Differences appear to consist of BL and MR being higher in calcium, whereas BL has more sodium than YR and MR.

X-Ray Diffraction

The BL sample appeared to be the least crystalline material, due to few non-alpha quartz diffraction peaks. Table 6 lists the approximate relative intensities and d-spacings for the samples. Phase identification was assisted by the JCPDS*-Johnson/Vand Powder Diffraction Search/Match Computer Program. The results are listed in Table 7. Alpha quartz (SiO_2) was the most abundant crystalline phase present in all

Table 5. SEMIQUANTITATIVE EMISSION SPECTROSCOPY*

	BL	YR	MR
Si	>10	>10	>10
Fe	5-10	5-10	5-10
Al	~5	~5	~5
Ca	1-5	~1	~5
Mg	1-5	1-5	1-5
Na	~5	~1	~1
Ba	~0.5	~0.5	~0.5
Ti	~0.5	~0.5	~0.5
W	~0.5	~0.5	~0.5
Mn	0.1-0.5	~0.1	0.1-0.5
B	~0.05	~0.01	~0.05
Cr	~0.05	~0.05	~0.05
Cu	~0.05	~0.05	~0.05
Ni	~0.05	~0.05	~0.05
V	~0.05	~0.05	~0.05

*Approximate weight percent of total sample

Table 6. X-RAY POWDER DIFFRACTION DATA

BL		YR		MR	
d, Å	I	d, Å	I	d, Å	I
13.291	41	14.418	25	14.096	7
10.464	26	10.282	3	10.282	6
7.138	10	7.036	16	7.025	8
4.216*	12	4.928	8	6.237	6
3.548	16	4.193*	16	4.955	3
3.326*	100	3.534	20	4.418	7
2.447*	15	3.311*	100	4.210*	24
1.991*	10	3.140	20	3.986	6
1.812*	10	2.441*	7	3.312*	100
		2.269*	6	3.175	13
		1.976*	10	3.089	7
		1.811*	12	2.969	5
		1.666	8	2.876	5
		1.537*	10	2.557	8
		1.372*	8	2.442*	17
				2.270*	10
				2.225*	7
				2.115*	8
				1.973*	8
				1.811*	19
				1.664*	10
				1.537*	15
				1.469*	6
				1.372*	17

*αSiO2 (PDF #5-0490)

*Joint Committee on Powder Diffraction Standards, 1601 Park Lane, Swarthmore, PA 19081.

samples. Analysis of remaining non-SiO_2 peaks revealed at least two other phases: one a chlorite-type clay and the other difficult to determine accurately. The percentages of each phase present are only estimates due to possible preferred orientation of the clay particles and the presence of noncrystalline clays and organics.

Energy-Dispersive X-Ray Analysis

Energy-dispersive X-ray analysis results are listed in Table 8. Figure 9 is an example of the EDAX analysis. In all samples the matrix has an Al/Si ratio from 10 to 100 while the large particle ratio is much smaller, 0.2 to 3.0. Although this analysis was conducted on a very small number (certainly not a representative sample) of large particles, some tentative conclusion can be made. The large particles in the BL material are predominantly quartz, whereas the high aluminum content of the YR large particle may mean the presence of corundum or altered plagioclase feldspar. The large particulates in MR could be made up of quartz and altered orthoclase feldspar.

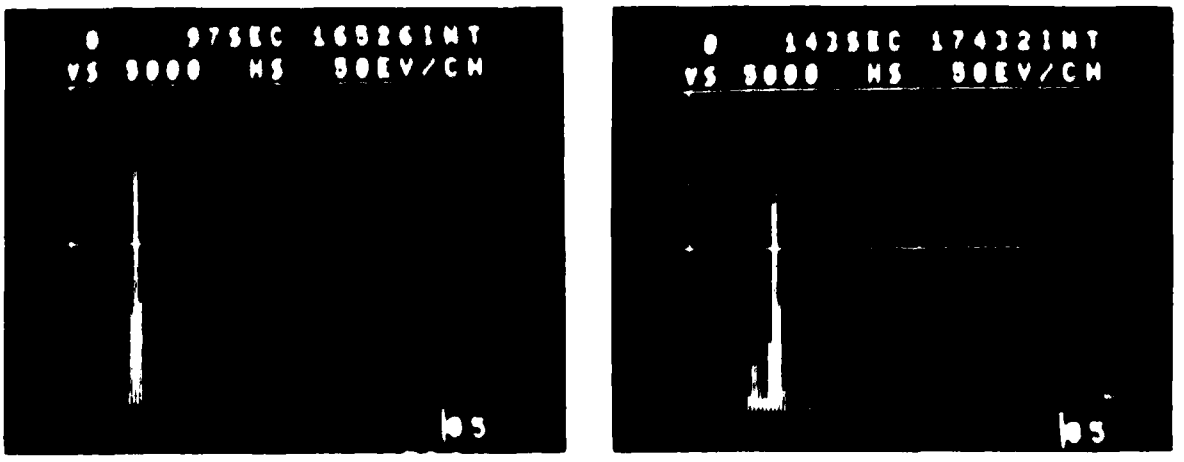
Table 7. X-RAY DIFFRACTION COMPUTER-ASSISTED ANALYSIS

BL	65%	Alpha Quartz SiO_2
	35%	Chlorites (Polygorskite, Bavalite) $(Fe, Mg, Al)_5(Si, Al)_8O_{20}(OH)_2 \cdot XH_2O$
YR	70%	Alpha Quartz SiO_2
	15%	Zeolite(?) (Gismondine) $Ca(Al_2Si_2O_8) \cdot 4\ H_2O$
	15%	Chlorites (Polygorskite, Bavalite) $(Fe, Mg, Al)_5(Si, Al)_8O_{20}(OH)_2 \cdot XH_2O$
MR	80%	Alpha Quartz SiO_2
	9%	Montmorillonite $Na_{0.33}(Al_{1.67}Mg_{0.33})\ Si_4O_{10}(OH)_2$
	11%	Chlorites (Polygorskite, Bavalite) $(Fe, Mg, Al)_5(Si, Al)_8O_{20}(OH)_2 \cdot XH_2O$

Table 8. NONDISPERSIVE X-RAY ANALYSIS*

	BL		YR		MR	
	Large Particle	Matrix	Large Particle	Matrix	Large Particle	Matrix
Al	1.5	8.0	6.5	7.2	3.1	9.5
Si	7.0	0.4	2.0	0.7	3.8	-
K	0.4	-	0.1	-	1.2	-
Fe	-	0.1	0.1	0.1	-	-
Cu	-	0.1	0.1	0.1	-	0.1
Al/Si	0.214	20.0	3.25	10.3	0.816	>100

*Peak heights in arbitrary units - relative quantities



a. BL sample - large particle

b. BL sample - matrix material

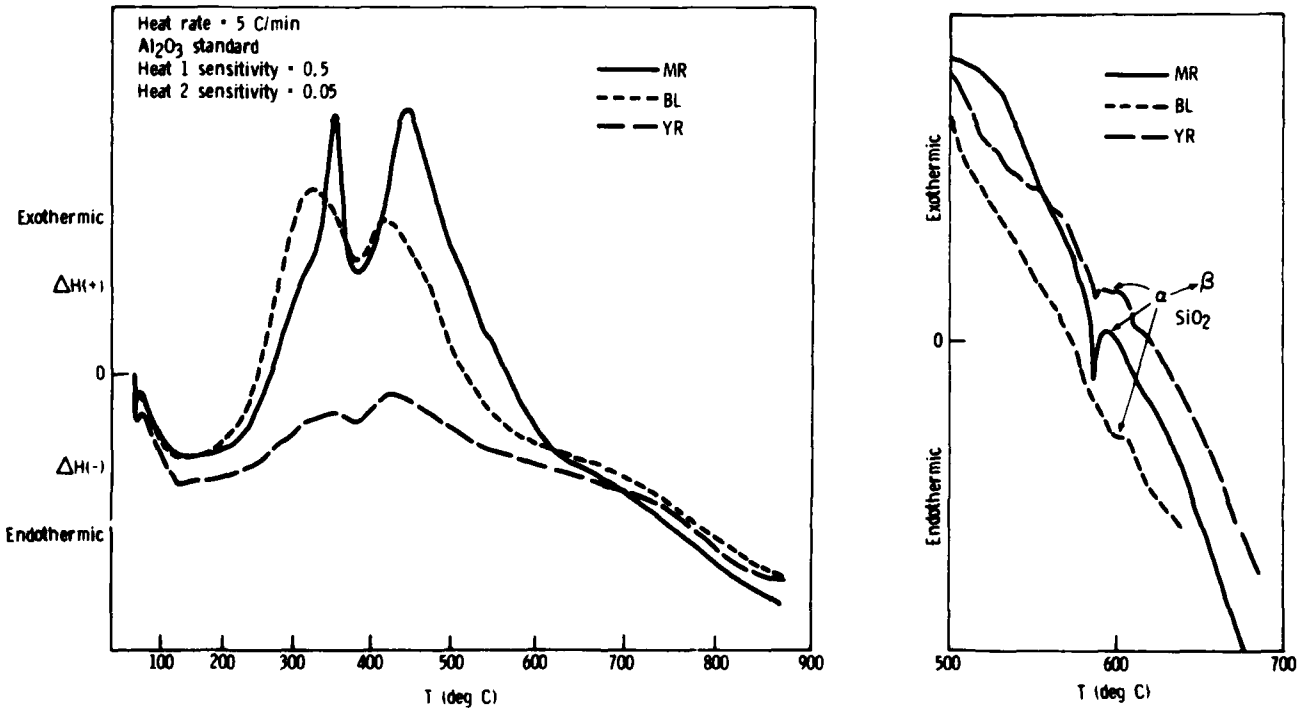
Figure 9. Energy-dispersive X-ray analysis.

Differential Thermal Analysis

Since clay materials differ in their characteristic DTA curves, this method of analysis was chosen to aid in determination of the clay phases. The resulting traces are shown in Figure 10. These samples have very different patterns than expected. Clays typically have several endothermic peaks, one at low temperatures due to water loss and one near 570°C due to a dehydroxylation reaction. However, as seen in the diagram, no endothermic peaks seem to occur, only exothermic. The exothermic peaks are due to burning organic materials within the sample. These reactions are strong enough to overwhelm any possible clay peaks. Unaltered carbonaceous remains (algal remains and/or peaty components) burn out between 300°C and 350°C. At 380°C volatile components are liberated from organics while at 500°C free carbon burns.[2]

The alpha-to-beta quartz transformation peak was obtained by reheating the sample after the organics were burned off. The occurrence of this peak implies that the samples probably contain at least two crystalline phases, quartz and an unidentifiable clay material. From these scans we are able to obtain the relative amounts of organics within each sample. The MR sample contains the most organics and YR the least.

Since in the absence of air we could not obtain the exothermic peaks due to carbonaceous oxidation, several scans were run in an argon atmosphere. These results provided no further information.



a. First heating

b. Second heating

Figure 10. DTA scans.

2. GRIMSHAW, R. W., and ROBERTS, A. L. *Carbonaceous Materials, Chapter XVI* in Differential Thermal Investigation of Clays, Robert C. MacKenzie, ed., London Mineralogical Society (Clay Minerals Group), 1966, p. 404-417.

CONCLUSIONS

Table 9 summarizes the conclusions detailed below.

Table 9. SUMMARY OF RESULTS

Population	BL 2 Modes	YR 1 Broad Mode	MR 2 Modes
Large Fraction			
Population, %	10	-	15
Median Size, μm	11	-	13
Width	1.78	-	1.99
Composition	Quartz	Quartz and Corundum	Quartz and Orthoclase Feldspar
Fine Fraction			
Population, %	90	100	85
Median Size, μm	0.2	0.3	<0.1
Width	6.3	15.85	19.95
Composition	Clay	Clay	Clay
Surface Area, sq m/g	8.07	2.36	4.01
Organic Matter	Intermediate	Least	Largest

The Brown's Lake sample is bimodally distributed with 10 wt% of the total particulate population being large, nearly spherical quartz grains having a median size of 11 μm and the remaining 90 wt% consisting of a very broad distribution of an iron-rich chlorite-like clay with a median size near 0.2 μm. Differential thermal analysis suggests the presence of a fairly large quantity of carbonaceous material. X-ray diffraction analysis also indicates that the clay phase is poorly crystallized or X-ray amorphous.

The Yazu River material is a broadly distributed powder with a median near 0.3 μm. The larger size fraction is predominantly quartz with smaller amounts of corundum or altered plagioclase feldspar, while the very fine particles are essentially a poorly crystallized chloritic clay material. This sample contains a much larger proportion of particles in the 1- to 5-μm range than the other two (BL and MR) populations. It also contains the least amount of organic matter.

The Mississippi River sample also has a bimodal distribution. Fifteen weight percent of the total population consists of irregularly shaped quartz or altered orthoclase feldspar grains with a median size of 13 μm, while the remaining 85 wt% is a fine montmorillonite or chlorite-type clay with a median size of less than 0.1 μm. The SEM analysis clearly shows that there is a more discernible size difference between the large and fine fractions of this material than the other two samples. This sample also contains the most organic matter and clay phases which exhibit X-ray diffraction patterns suggesting a more crystalline material.

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