

The Effect of Human Size Sorting on the Mineralogy and Chemistry of Ceramic Clays

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The raw materials from which ancient potters formed their pots are most often an intricate mixture of particle sizes with mineralogical variations both within and among the size ranges. Any alteration of the particle size distribution of the raw materials through the removal of coarser size ranges (levigation) and/or the addition of coarse aplastic material (temper), has an effect on the mineralogical and chemical content of the ceramic clays and ultimately the finished ceramic. This paper examines the effect of particle size distribution on the mineralogical and chemical content of natural and levigated clays collected from a single locality in NE Syria.

Over the past three decades bulk chemical characterization studies of ceramics and other clay-based artifacts have become firmly established as a research tool for determining provenience of clay sources or location of the manufacturing site. From the inception of this type of research, however, questions have been raised concerning the utility of bulk compositional analysis of coarse textured ceramics. Whether coarse aplastic material was intentionally added to the clay to improve forming or performance characteristics (tempering) or naturally occurring clays with the proper aplastic composition were selected for the same purpose, the heterogeneity of such a mixture, so the argument goes, would render bulk composition of little utility as a provenience determiner. To counter this line of argument and to improve understanding of the chemical behavior of such mixtures, a fair amount of research has been aimed at the study of the effect of coarse particles in ceramic clays. These studies have ranged from separate analysis of the aplastic fragments (e.g., Blackman 1981), to computer

simulations of the effects of tempering with mineralogically different materials on bulk chemical composition (e.g., Neff et al. 1988, 1989).

Through all of this research, relatively little attention has been devoted to the detailed examination of the chemical effects accompanying the removal of coarser particles in the preparation of clay. This paper reports on research designed to examine the effect of human size sorting (levigation) on the mineralogy and bulk chemical composition of clays used to produce ceramics and unfired clay artifacts and the impact such sorting may have on the ability to form meaningful chemical compositional groups in provenience studies.

Background of the Research

Two research projects focusing on the site of Tell Leilan located on one of the tributaries of the Khabur River in northeastern Syria (Figure 8.1) have dealt with artifacts made from very fine textured clays. Thin section and scanning electron microscopic

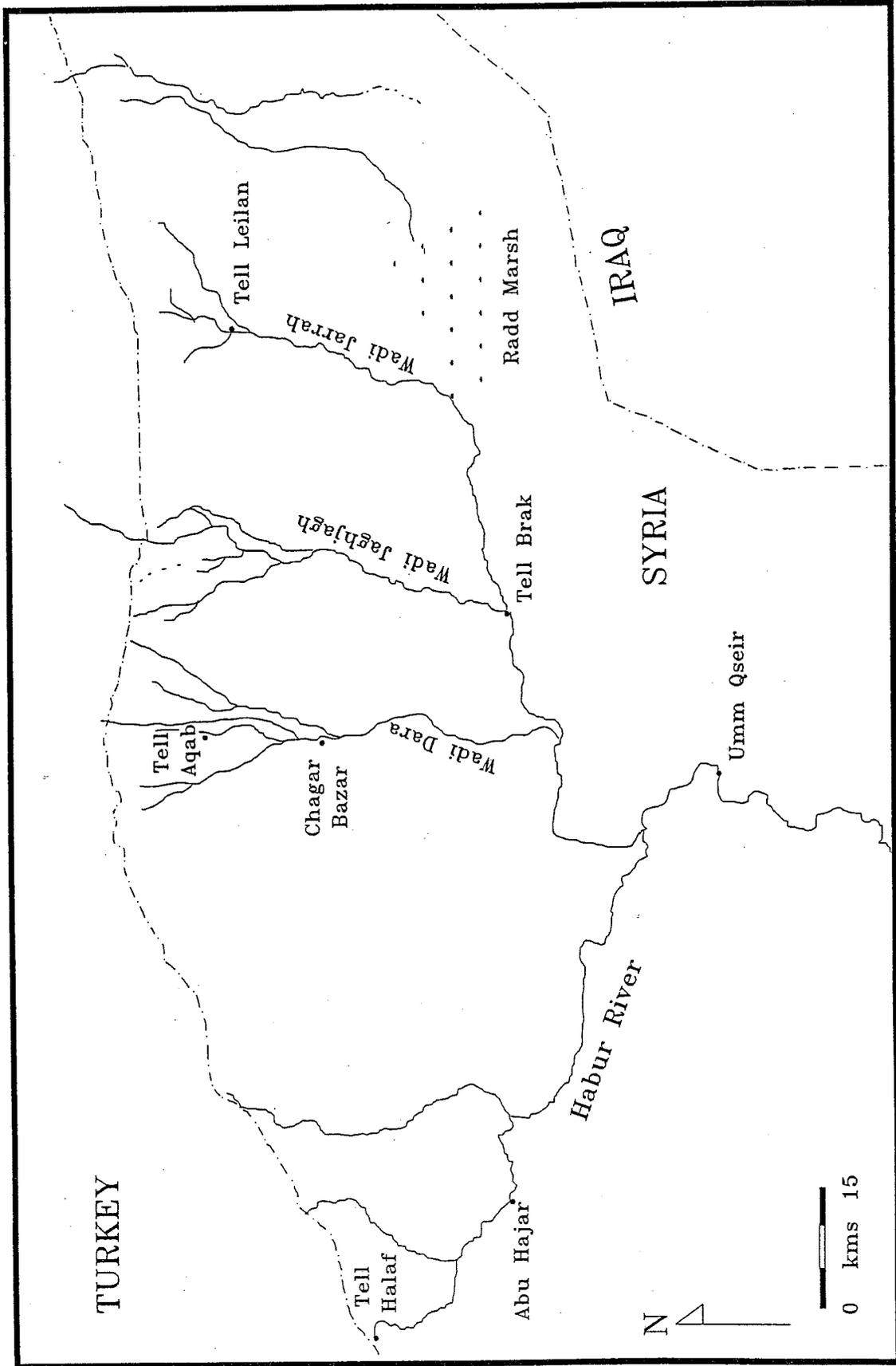


Figure 8.1. Map of the Khabur River drainage in NE Syria.

studied of late 3rd Millennium fine ware ceramics have shown that these ceramics contain few grains with particle sizes greater than 75 microns (Blackman et al. 1991) and low power microscopic examination of tablet and sealing clays have documented similar particle size ranges for these artifacts (Blackman in press). In both projects bulk chemical composition was used to attempt to identify production sites or clay source areas. Since both studies involved the chemical differentiation of clay based artifacts from a rather small geographical region (a radius of about 15 km around the site of Tell Leilan), it was recognized that the extensive analysis of local source clays would be needed to augment the artifact analysis.

The initial working hypothesis assumed that since the major north-south tributaries of the Khabur River drain different geological formations, the alluvial clays along these tributaries should reflect this difference in a distinctive bulk chemical composition. The Khabur River and its tributaries head in the mountains to the north in Turkey and flow out onto what is today a relatively flat plain. The north to south gradient, once the streams enter the plain, is only about .3 meters per kilometer, creating a distinctly low energy environment. Geomorphological studies on this plain and to the west on the Euphrates River drainage indicate several cycles of active fluvial aggradation and downcutting from Pleistocene through recent times (Galactionov et al. 1966). Given the post-Pleistocene geological history of the plain, it was thought that surface or near surface deposits of well sorted alluvium should exist to serve as sources for the fine clays used to make the artifacts under study.

In 1987 and 1989 a clay resource survey was undertaken, in conjunction with archaeological and soils surveys carried out under the auspices of the Tell Leilan Project directed by Dr. Harvey Weiss of Yale University. The goal of this survey was to locate, sample, and chemically characterize the naturally well sorted clay deposits of the region to provide a data base for comparison with ceramic and unfired clay artifacts. The clay resource survey concentrated on the Wadi Jarrah and its tributaries within a 15 km radius of Tell Leilan (Figure 8.2) and on the many "borrow" pits associated with road construction and repair in the region.

Examination of the profiles in the "borrow" pits showed that much of the region is underlain with sand and small gravel deposits, probably associated with wetter periods of terracing in the late Pleistocene (Galactionov et al. 1966). These near surface sand and gravel deposits and the lack of significant post-Pleistocene downcutting to expose deposits below the sands and gravels restricted both ancient and modern access to the surficial deposits. In none of the 60+ loci

examined were any extensive, naturally well sorted deposits of silts or clays encountered. The alluvial deposits, overlying the sands and gravels and available for exploitation are predominately poorly sorted mixtures with sand components that range up to 15% by weight. The source "clays" used for ceramic or clay object manufacture, therefore must have started with this poorly sorted alluvium.

The lack of well sorted silt/clay deposits—at least within the 15 km radius of Tell Leilan that was extensively surveyed—strongly indicates human intervention was necessary to produce the uniformly fine raw materials used for sealings and tablets and needed to produce the ceramic fine wares. Simple gravitational settling—levigation—seems the most likely pretreatment for the poorly sorted starting materials. Personal observation of the preparation of mud for interior plastering (in Iran) indicated that large quantities can be prepared by the following process. Near a water supply, a pit is dug into the alluvium and filled with water. Enough of the alluvium is added back to the pit to form a slurry. The slurry is stirred and the pit is left undisturbed until the water evaporates. This simple process results in a layered deposit from which various size fractions can be selectively removed depending on the plaster requirements.

The sorting process outlined above, while quick and efficient, is imperfect and depending on the thickness of the slurry, varying amounts of the coarse silt and finer sand sizes are included in or excluded from the "fine fraction". Given the imperfect nature of the size sorting, careful examination of the effects of grain size variation on bulk chemistry was necessary to assess its impact on compositional groups.

One sampling locus, CS-12, a four meter high meander scarp on the Wadi Jarrah about 4 km south of Tall Leilan (Figure 8.2) was selected for detailed study. In section this exposure consisted of 2.5 meters of finely laminated beds of fine sands, silts, and clays interbedded with layers of coarse sand heavily stained with manganese oxide. This deposit was extensively sampled and forms the basis of the research reported in this paper.

Sample Preparation

In an attempt to simulate the assumed levigation process, the raw alluvium "clay" from sample locus CS-12 was subdivided and a subsample of about 150 grams taken for treatment. The subsample was initially wet sieved through a 75 micron brass sieve and the sand fraction was taken to consist of all particles greater than 75 microns. No attempt was made to further subdivide the sand fraction into size categories. All material passing through the sieve was taken to be

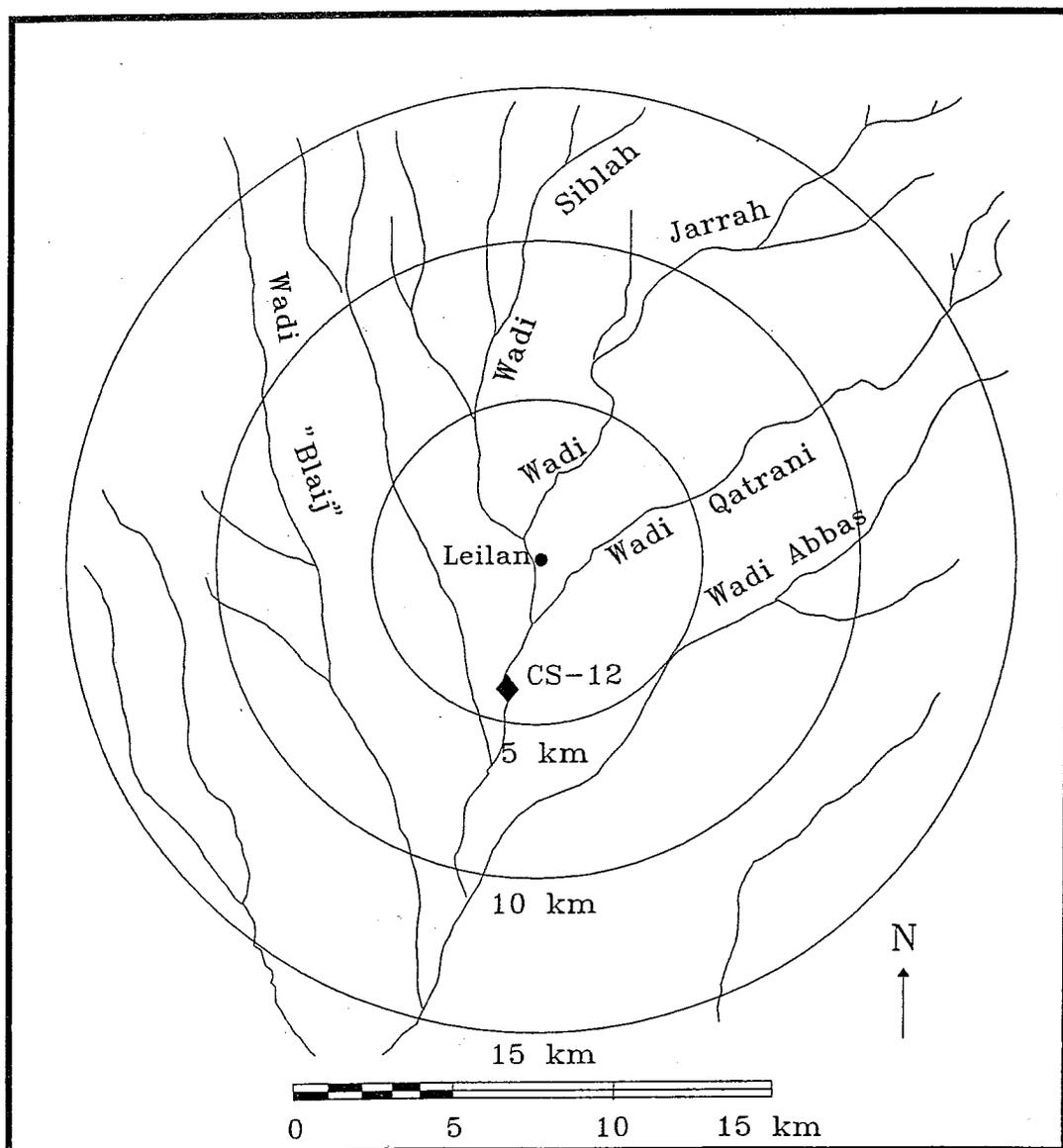


Figure 8.2. Map of the region within a 15 kilometer radius of Tell Leilan, Syria showing the location of sampling locus CS-12.

silt and clay sizes and was allowed to evaporate to dryness in a large diameter, shallow basin. The initial separation resulted in a split that contained 38% sand sizes and 62% silt/clay sizes by weight.

The silt/clay split was disaggregated, homogenized, and a subsample taken for analysis. The remaining silt/clay fraction was then resuspended in a 1,000 ml graduated cylinder, stirred, and allowed to settle for 15 minutes. At the end of this period the supernatant was poured off and the fraction that had settled out was oven dried at 100° C for 24 hours. The fraction that remained in suspension was allowed to settle for 24 hours at which time the supernatant was clear. The supernatant was again poured off and the

fine fraction was dried for 24 hours also at 100° C. This split consisted of about 85% coarse fraction and 15% fine fraction.

These four size fractions: (1) greater than 75 microns; (2) less than 75 microns; (3) that which settled out in fifteen minutes; and (4) that which remained suspended are hereafter referred to as the sand, silt/clay, silt, and clay fractions respectively. It is recognized that these fractions are only incomplete size separations. Microscopically some of the "sand" grains could be seen to be aggregates of the finer sizes, many cemented with calcite. Likewise, many of the "silt" grains were flocculated clay particles and smaller calcite cemented aggregates. The imperfect

nature of the size separation is not critical as it was intended to simulate techniques available to ancient potters not to provide perfect size categories. The samples to be analyzed, therefore consisted of the unprocessed alluvium as it came from the ground, the sand fraction, the combined silt/clay fraction, the silt fraction, and the clay fraction.

Mineralogy of the Sand, Silt, and Clay Fractions

X-ray diffraction analysis was carried out on the sand, silt, and clay fractions to identify the major mineral phases. Sample preparation consisted of grinding subsamples of each fraction to a uniform fineness with an agate mortar and pestle. The samples were packed into the cavity in the sample holder with no attempt to avoid preferential orientation. The analysis was performed using copper radiation at power settings of 50 volts and 40 ma. The tube and detector electronics were allowed to equilibrate for one hour before analysis began and tube power and detector settings were held constant between all diffraction runs.

The sand, silt, and clay size fractions each proved to be mixtures of the same mineral phases consisting of varying amounts of quartz, calcite, dolomite, feldspar, and clay minerals. No other mineral phases were present in quantities large enough to be identified with certainty. The feldspar appears to be a plagioclase with little or no K-spar detectable. The clay minerals are represented by broad, poorly shaped peaks that appear to be poorly crystalline or mixed layer illite.

Absolute amounts of each phase in each fraction could not be calculated. However, since all machine settings were constant between diffraction runs, peak intensities could be used to compare individual mineral phases among the size fractions by ratioing to the fraction with the most intense peak. Table 8.1

shows the results of this analysis. Relative to the silt and sand fractions, the clay size particles contained the greatest amounts of the clay mineral (illite?) and the least quartz, calcite, dolomite and feldspar. The silt fraction was highest in quartz and feldspar, while the sand fraction was highest in the carbonates—calcite and dolomite—with low amounts of feldspar. Both the silt and sand fractions also had relatively high amounts of clay minerals. As mentioned above, optical microscopic examination of the sand fraction showed numerous clay and silt size particles cemented with calcite. Nearly all the quartz and carbonate grains also retained a coating of clay particles not removed during the wet sieving or settling process. These two occurrences of clay size particles in the sand fraction explains the presence of the clay minerals in this fraction. In the silt fraction, the presence of clay minerals are probably due to a combination of flocculated clay size particles and a finer version of the carbonate cemented particles observed in the sand fraction.

The mineralogy of these three size fractions, while similar in the phases present, are quite different in the relative proportions of each phase. This difference in phase proportions should be reflected in the bulk chemistry of each fraction and in differing mixtures of the three primary size components.

Chemical Analysis

The bulk chemical analysis was carried out by instrumental neutron activation analysis at the NIST Reactor in Gaithersburg, Maryland. Twenty-nine elements were sought in the analysis and twenty seven were routinely quantified using an analytical protocol similar to that described in Blackman (1984:23-26). The samples were dried for 24 hours at 100° C, allowed to cool to ambient temperature in a desiccator, and two replicate samples of each size category, clay, silt, sand, clay/silt, and the unprocessed sand/silt/clay sample were weighed into

Table 8.1. Summary of the X-ray Diffraction Analysis of the Clay, Silt, and Sand Fractions of CS-12.

Phase	dÅ	hkl	Relative Intensity		
			Clay	Silt	Sand
Quartz	3.343	101	42	100	54
Calcite	3.030	104	55	75	100
Dolomite	2.886	104	11	44	100
Feldspar	3.24	002?	13	100	16
Illite?	4.48	020?	100	63	63

Note: The intensities reported are relative to the most intense peak in the three size fractions.

sample containers. Table 8.2 presents the data for the average concentration of the two replicate samples in each of the size fractions.

The alkali elements, sodium, potassium, rubidium, and cesium, do not all show the same distribution in the various size fractions (Figure 8.3). While potassium, rubidium, and cesium are concentrated in the clay fraction and depleted in the silt (the sand having intermediate values), sodium shows the opposite trend being concentrated in the silt and depleted in the clay fraction. The alkaline earth elements, calcium, strontium, and barium, also display disparate behavior (Figure 8.4). Calcium, with concentrations about equal in the silt and sand, is depleted in the clay fraction, while the minor constituents, strontium and barium show the opposite trend being concentrated in the clay fraction and depleted in the silt and sand. The transition metals, iron, scandium, cobalt and zinc, have distributions comparable to potassium (Figure 8.5). All are more concentrated in the clay fraction, depleted in the silt, and intermediate in the sand. Chromium is an exception, like sodium it is greatly enriched in the silt and depleted in the clay fraction.

The rare earth elements and thorium (Figure 8.6) show a slight enrichment in the clay fraction and similar values in the silt and sand. Zirconium and hafnium, like sodium and chromium, show significant enrichment in the silt and depletion in the clay fraction (Figure 8.7).

The Effect of the Mineralogy on the Bulk Chemistry

The distribution of the elements that show enrichment in the clay fraction, depletion in the silt, and intermediate values in the sand is explainable by examining the distribution of major mineral phases among the size fractions. Potassium is a major constituent of the illitic clay minerals that are concentrated in the clay fraction relative to the silt and sand. The trace elements, rubidium and cesium are likely substituting for potassium in the same clay minerals.

The reduced amounts of the carbonate mineral phases (calcite and dolomite) in the clay fraction and their dominance in the sand accounts for the lower

Table 8.2. Average Elemental Concentrations in the Size Fractions of Sample CS-12.

Element	Clay	Silt	Clay/Silt	Sand	Sand/Silt/Clay
Na (%)	0.230	0.553	0.547	0.349	0.458
K (%)	1.19	0.837	0.970	0.892	0.955
Rb	79.3	47.6	55.7	57.9	54.6
Cs	4.70	2.52	2.89	3.26	2.99
Ca (%)	9.37	13.9	12.2	13.7	13.2
Sr	816.	716.	650.	634.	712.
Ba	385.	321.	327.	321.	298.
As	11.4	7.82	8.40	8.71	8.42
Sb	0.939	0.589	0.660	0.669	0.696
Sc	16.7	11.1	12.0	12.9	12.4
Cr	189.	388.	307.	225.	283.
Fe (%)	4.87	3.40	3.54	3.77	3.82
Co	38.0	28.5	30.4	30.7	21.0
Zn	103.	65.2	71.8	73.2	78.5
La	30.9	26.1	27.3	27.5	27.7
Ce	64.0	52.8	54.9	53.6	54.0
Nd	24.6	20.3	22.3	22.9	22.7
Sm	5.03	4.46	4.61	4.67	4.62
Eu	1.07	0.980	1.01	1.04	1.02
Tb	0.713	0.646	0.615	0.644	0.619
Yb	2.34	2.43	2.28	2.28	2.41
Lu	0.351	0.351	0.351	0.333	0.328
Th	8.10	6.65	6.90	6.80	6.80
U	2.27	1.76	1.58	2.00	1.89

Units are parts per million except where indicated %.

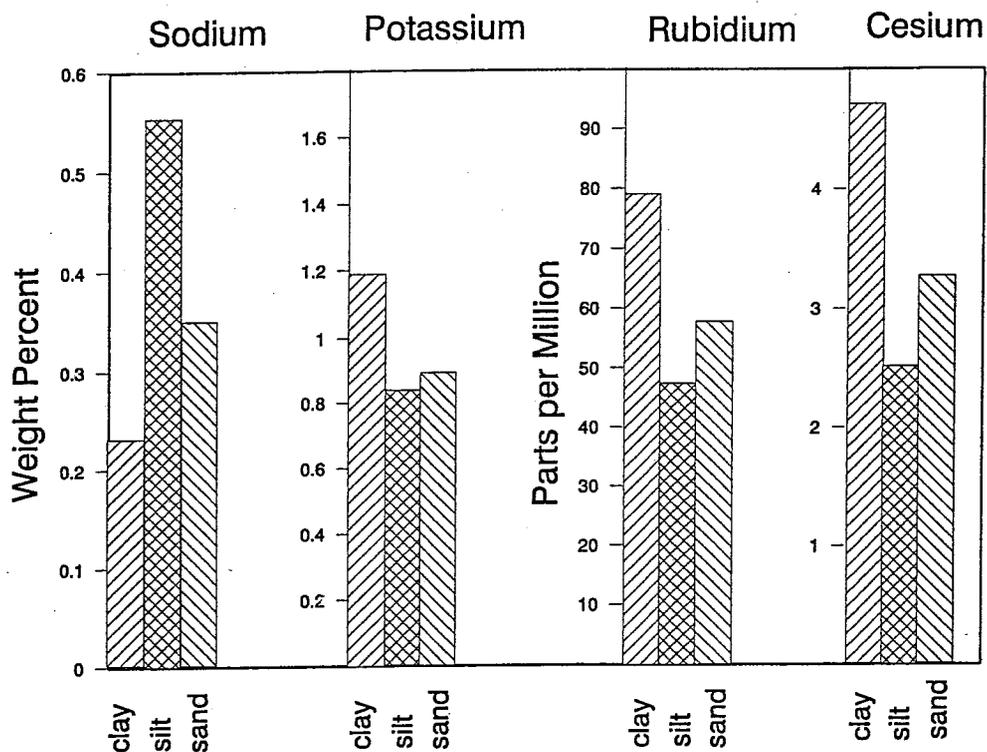


Figure 8.3. Plot of the concentrations of the alkali elements in the clay, silt, and sand size fractions of alluvial "clay" sample CS-12.

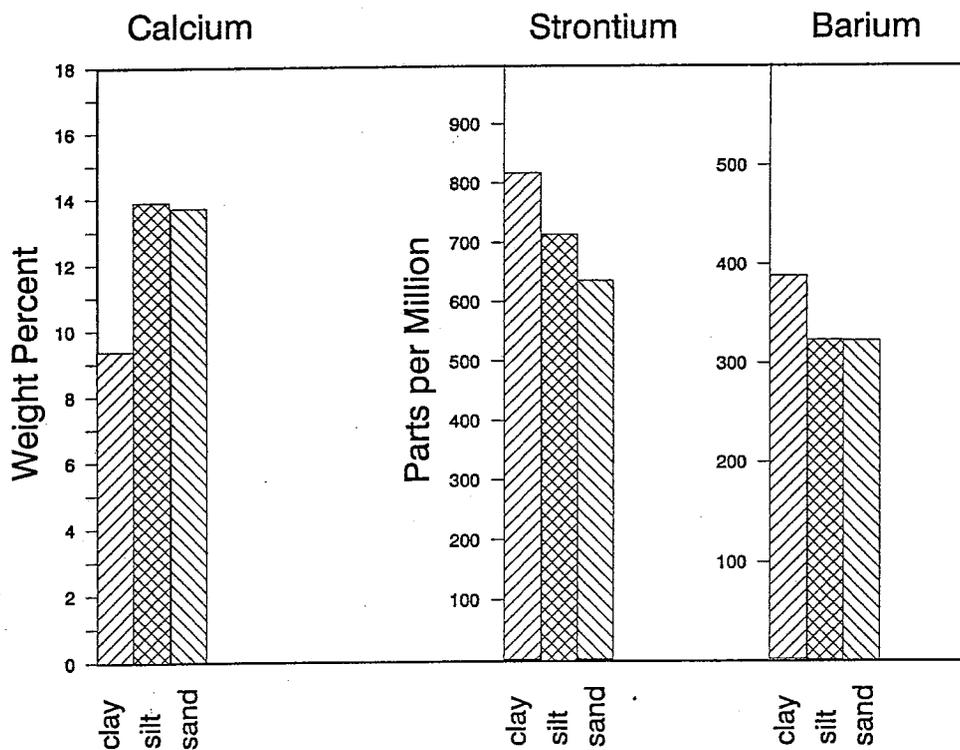


Figure 8.4. Plot of the concentrations of the alkaline earth elements in the clay, silt, and sand size fractions of alluvial "clay" sample CS-12.

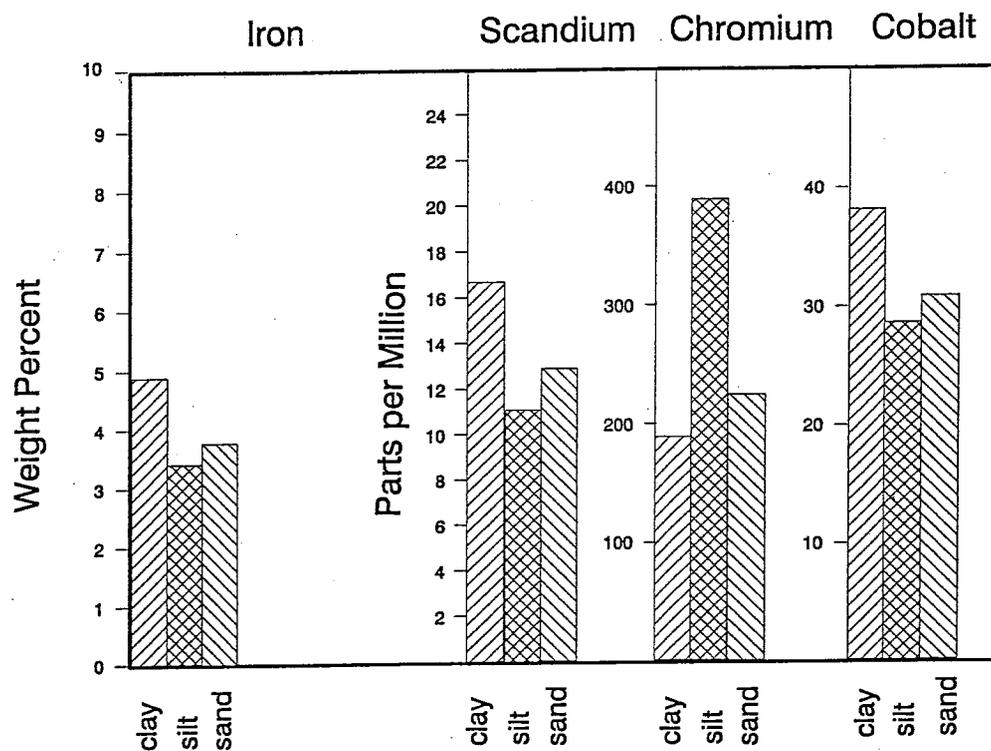


Figure 8.5. Plot of the concentrations of the transition metal elements in the clay, silt, and sand size fractions of alluvial "clay" sample CS-12.

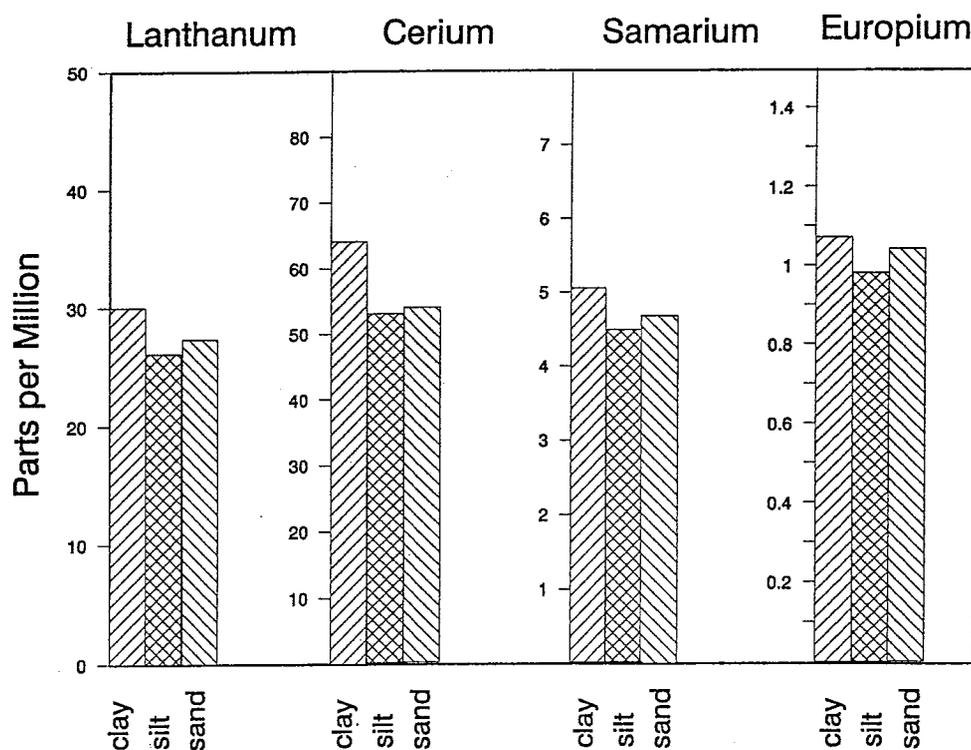


Figure 8.6. Plot of the concentrations of the rare earth elements in the clay, silt, and sand size fractions of alluvial "clay" sample CS-12.

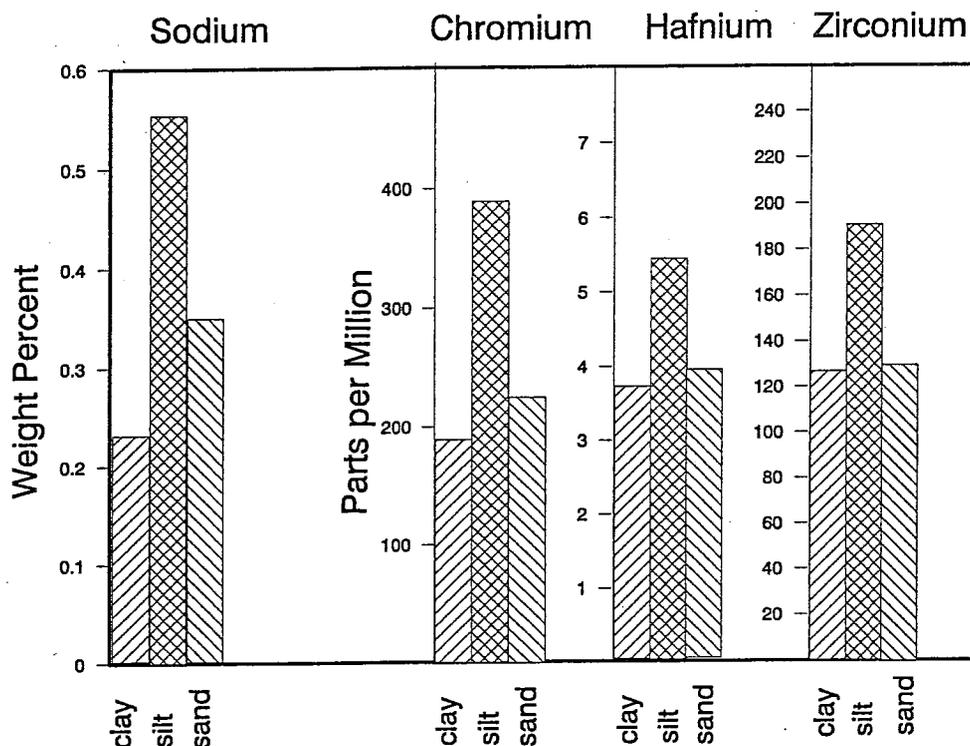


Figure 8.7. Plot of the concentrations of sodium, chromium, hafnium, and zirconium in the clay, silt, and sand size fractions of alluvial "clay" sample CS-12.

calcium concentrations in the clay and higher concentrations in the sand. The reduced carbonate concentrations in the clays are a result of the greater solubility of very fine clay carbonate grains due to increased surface area. When the dissolved calcium reprecipitates as a carbonate, this occurs as cementation of aggregates of clay and silt size particles or as crystallization on pre-existing calcite grains. Both processes have the net result of increasing the size of the calcium containing grains and redistributing calcium to the silt and sand size range. The silt fraction with calcium concentrations comparable to the sand, but with much lower amounts of calcite and dolomite, must have a third calcium containing mineral phase. The plagioclase feldspar, highly concentrated in the silt relative to the clay and sand, would seem the likely candidate. The combination of higher amounts of carbonates (CaCO_3 and $\text{CaMg}(\text{CO}_3)_2$) in the sand and these same carbonate minerals plus higher relative amounts of quartz (SiO_2) in the silts serve as diluents, depressing the concentrations of the transition metals and the rare earth elements in the silt and sand.

The opposite trend, i.e., higher concentrations in the silt and lower concentrations in the clay and sand, displayed by the elements sodium, chromium, hafnium and zirconium, run counter to the apparent

dilution effect noted for the other elements. The presence of mineral phases containing these elements and preferentially concentrated in the silt fraction would seem the likely explanation. The relative concentration of a sodic plagioclase feldspar in the silt would explain the sodium enrichment in the silt. The chromium enrichment in the silt can be most easily understood by the presence of small amounts (too low in abundance to be detected by x-ray diffraction) of a chromium bearing mineral, probably chromite (FeCr_2O_3). Chromite's high density ($D = 5.09$) would cause it to settle more rapidly during the gravitational separation of the silt and clay fraction and thus even the finer sizes would be concentrated in the silt fraction. The relative enrichment in chromium in the silt fraction over the clay fraction is 200 ppm, which would result from a mere 0.05 weight percent enrichment in chromite.

The enrichment of zirconium and hafnium in the silt can be similarly explained by enrichment in the heavy mineral zircon (ZrSiO_4). Zircon has a density of 4.6 to 4.7 and would be expected to behave in much the same way as chromite in a gravitation separation. Zircon typically has Hf/Zr ratio of 0.01 to 0.04 (Deer et al. 1966:14). The observed Hf/Zr ratio in the enriched silts is 0.026, well within the published ratios. Although zircon frequently contains rare earths and actinides as minor constituents, the small

amounts of zircon necessary to account for the enrichment in the silts (less than 0.01 weight percent) would not be sufficient to affect the concentrations of rare earths or actinides.

Effects of Size Sorting on the Bulk Chemistry

The effect of natural or human size sorting on the bulk chemistry of the resultant raw materials as it bears on the ability to form meaningful chemical composition groups can be examined in light of the documented differences in chemistry and mineralogy presented above. To facilitate comparison of the size fractions, the concentration values for each element were normalized by dividing by the concentration in the unprocessed sand/silt/clay sample. These normalized concentrations are arranged in Table 8.3 to show the effect of the removal of first the sand fraction and then the silt fraction.

Table 8.3 shows that removal of the sand size fraction from sample CS-12 produces concentration changes of less than 5% for 15 of the 27 elements determined. An additional 8 elements show changes in the 5–10% range, while only 4 elements, sodium, cobalt, zirconium, and uranium show concentration differences greater than 10% (columns 2 and 3 in Table 8.3). The overall effect is to produce variation well within the range noted for fine ware ceramics (Blackman et al. 1991) and for unfired clay artifacts (Blackman in press) from this region. It would, therefore, appear that for alluvial deposits of the mineralogical composition documented here, removal of varying amounts of the sand size fraction does not seriously compromise one's ability to identify chemically meaningful groups of artifacts.

Table 8.3 also shows, however, that removal of all or part of the silt size fraction in deposits of this type has serious implications for chemical characterization studies. All elements, excepting the rare earths, show concentration changes in the range of 20–40%, with chromium, zirconium, hafnium, sodium, and calcium decreasing and the others increasing. Figures 8.8 and 8.9 graphically demonstrate the impact on chemical composition of silt size removal. In Figure 8.8 the calcium vs. chromium concentrations, plotted for each of the different size fractions, are representative of the behavior of the elements concentrated in the silt fraction. In Figure 8.9 the calcium vs. scandium concentrations are representative of the behavior of the elements concentrated in the clay fraction. The arrows in both figures indicate the directions taken by the concentration values as first sand and then silt sizes are removed from the mixture.

Calcium, in both plots, decreases only slightly as the removal of calcium carbonate rich sand is balanced by the calcium content in the silts. The decrease in calcium concentration accelerates, however, as the carbonates and plagioclase are removed with the silt fraction. Chromium shows only a slight increase with sand removal, but a sharp decrease in concentration as chromite is removed with the silt fraction. Scandium, on the other hand decreases slightly with sand removal as the increased

Table 8.3. Average Elemental Concentrations in the Clay, Clay/Silt, and Sand/Silt/Clay Size Fractions of Sample CS-12 Normalized to the Unprocessed Sand/Silt/Clay Sample Concentration.

Element	Sand/Silt/Clay	Clay/Silt	Clay
Na	1.00	1.19	0.50
K	1.00	1.02	1.25
Rb	1.00	1.02	1.45
Cs	1.00	0.97	1.57
Ca	1.00	0.92	0.70
Sr	1.00	0.91	1.15
Ba	1.00	1.10	1.29
As	1.00	1.00	1.35
Sb	1.00	0.95	1.35
Sc	1.00	0.97	1.35
Cr	1.00	1.08	0.67
Fe	1.00	0.93	1.27
Co	1.00	1.45	1.83
Zn	1.00	0.91	1.31
La	1.00	0.99	1.12
Ce	1.00	1.02	1.19
Nd	1.00	0.98	1.08
Sm	1.00	1.00	1.09
Eu	1.00	0.99	1.05
Tb	1.00	0.99	1.15
Yb	1.00	0.95	0.97
Lu	1.00	1.07	1.07
Th	1.00	1.01	1.19
U	1.00	0.83	1.20
Cr	1.00	1.15	0.97
Hf	1.00	1.08	0.82
Ta	1.00	1.01	1.14

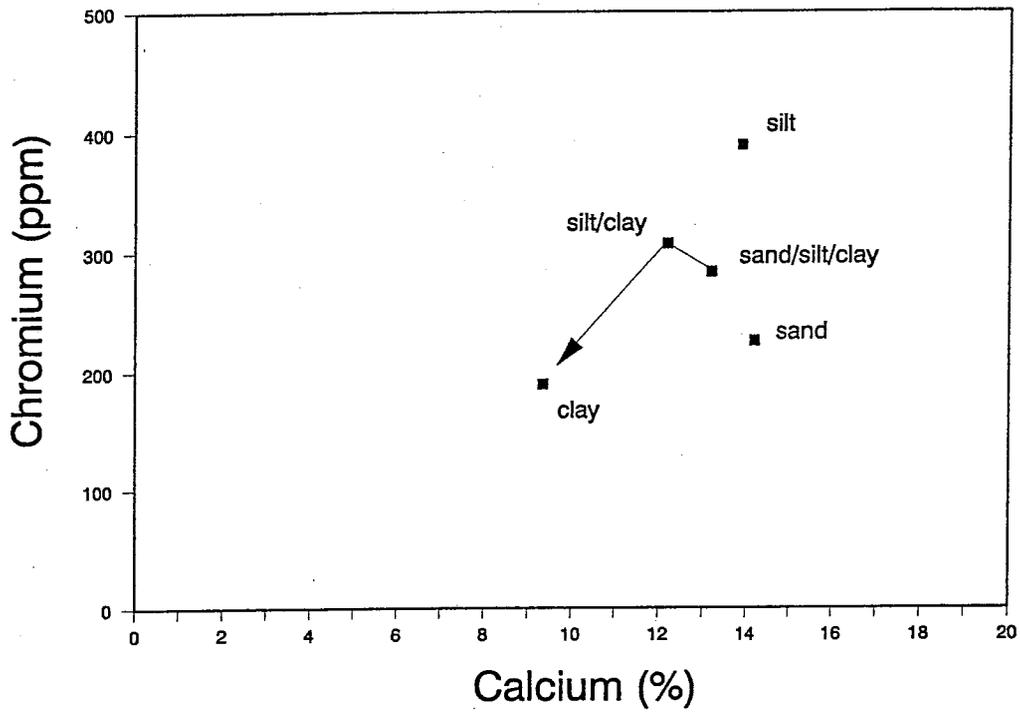


Figure 8.8. Plot of calcium vs. chromium showing the concentration trends with the removal of sand and silt.

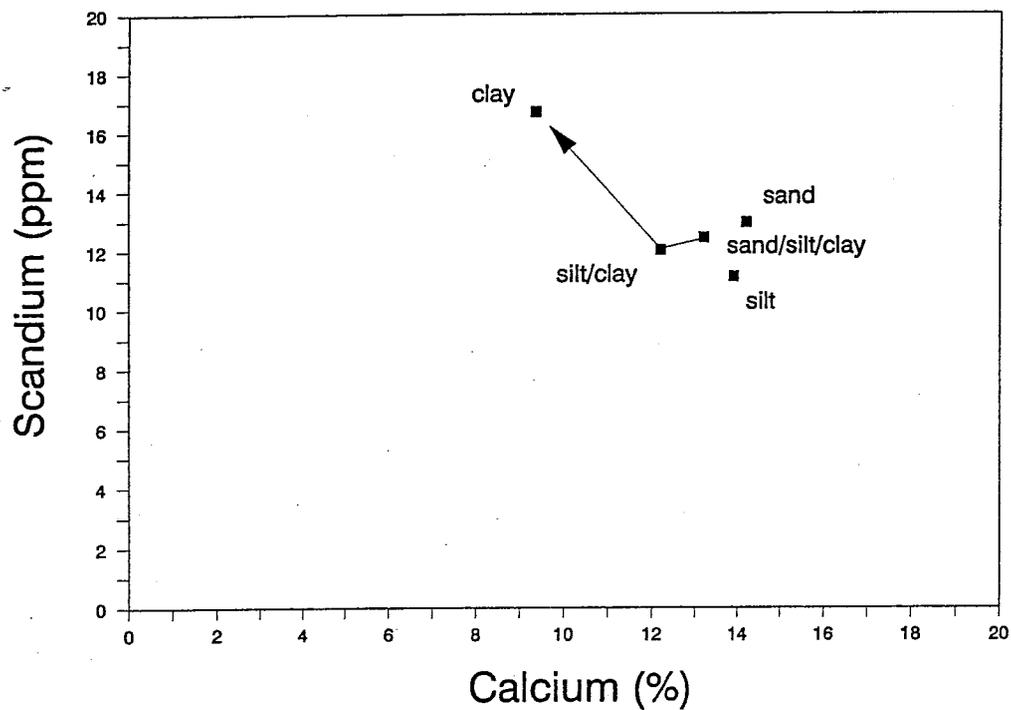


Figure 8.9. Plot of calcium vs. scandium showing the concentration trends with the removal of sand and silt.

dilution effect of the combined carbonates and quartz in the silt exert more influence on the mass of the sample. The scandium concentrations then increase sharply as these diluents are removed with the silt size particles.

Conclusions

As shown in Table 8.3 and Figures 8.8 and 8.9, concentration changes in CS-12 alluvium are relatively minor when the levigation process removes only sand size particles, with abrupt changes in the concentration of most elements occurring only as the silt sizes are removed. This finding would seem to indicate that removal of the sand size particles through levigation would have little effect on the ability to define coherent chemical compositional groups. However, since the sample preparation technique used sieving to separate the sand from the silt/clay fraction, it is possible that the heavy minerals, presumed to be responsible for the

increased concentrations of chromium, hafnium, and zirconium in the silt fraction, were preferentially concentrated in the silts. Had gravity settling been used these same minerals would probably have settled out with the coarsest silt to fine sand sizes shifting their influence on the concentrations of these elements to the fine sand removal phase of levigation. Major concentration fluctuations for these elements would then be expected to occur between different levigation events as more or less of the fine sand and coarse silt sizes were removed or retained.

Clearly the concentration fluctuations related to particle size noted here are specific to a single type of alluvial deposit and can, only with great caution, be extended to deposits of different mineralogy and geological history. This study should, however, serve to point out the need to be aware that particle size related elemental concentration differences do exist and to institute methodological controls to monitor for them.

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