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# **SOME REMARKS ON FINE-GRAIN SAMPLE PREPARATION FOR TL DATING**

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More than one decade has passed since the basic work on the preparation of fine grain TL samples (Zimmerman, 1971), and several aspects concerning the mineralogy of baked clays have not yet been considered in practical dating. For instance, changes in mineral composition occurring on firing, sintering and vitrification have so far not been accounted for. Also, the effect of clay composition on TL properties, such as signal height and the plateau, has received little study. The present report is concerned with three aspects of Zimmerman's early work.

## **AIMS OF INVESTIGATION**

1. The methods of preparing samples of fine grains (1-8  $\mu\text{m}$ ) for TL dating follow closely the basic working techniques of clay mineralogy, especially the separation of fine grains by sedimentation which is related to heavy mineral separation using an Attenberg Cylinder. In applying these techniques to fired fabrics, it has been assumed that individual minerals can be isolated by breaking apart the ceramic matrix. We investigated the validity of this assumption and the extent to which firing temperature influences the result of the procedure.
2. For the fine grain technique, the grains to be selected for TL measurement should have received the full infinite matrix dose rate. According to calculations by Bell (1980) of the alpha dose delivered to quartz, attenuation becomes significant in grains larger than 5  $\mu\text{m}$ . A sedimentation technique has been established to ensure that larger particles are not contained in the TL sample. Examination of samples of fired clay using a scanning electron microscope (SEM) has shown the grain size range that is actually achieved using this procedure.
3. Grain size selection is almost always paralleled by mineral selection. This can be shown by comparison of the chemical composition of fine and coarse fractions.

## **EXPERIMENTAL RESULTS**

### **The effect of firing temperature**

Sedimentation experiments were performed using samples of a non-calcareous clay which had been fired at 600, 800, 1000 and 1200°C. Firing was performed following procedures similar to those used in pottery manufacture in order to ensure a similar degree of sintering. The oven temperature was gradually increased by 100°C every half hour, starting from 300°C. The maximum

temperature was held for 30 min and the samples remained in the oven overnight to cool. After crushing in a vice, fine grain samples were obtained by sedimentation in acetone following the standard procedure ( Zimmerman, 1971). The sedimented samples were viewed in a SEM, and the images shown in Figure 1 indicate the following:

- The grain-size distribution at a firing temperature of 600°C is substantially homogeneous, the diameter of the largest grains is less than 10  $\mu\text{m}$ , and there are no particles less than 1  $\mu\text{m}$ . The grain size distribution corresponds exactly to the predictions which had been made on the basis of Stoke's law ( Zimmerman, 1971).
- The same is still true for the firing temperature of 800°C, but considerable changes occur at higher firing temperatures.
- In the sample fired at 1000°C a noticeable proportion of particles of approx. 2  $\mu\text{m}$  is visible positioned between very large grains. The distribution appears heterogeneous, and there are already vitrified structures detectable that are due to mineral species decomposing at this temperature.
- Fragments of the above type also form the predominant constituent in the 1200°C fired sample. The shape of the grain boundaries and the absence of internal structure of the grains indicate that they are no longer individual mineral grains isolated from the matrix, but fragments of a vitrified continuum. It can be shown that the mean size of fragments at the firing temperature of 1200°C is higher compared with that for lower firing temperatures, although the same sedimentation process has been used.

### **Maximum grain size**

The series of SEM images demonstrates that sedimentation of fine grains in acetone does not guarantee a homogeneous size distribution. Depending on firing temperature and clay composition, a broad distribution may be obtained which contains a proportion of grains with a diameter beyond the critical size. This would not necessarily require a grain size correction (Bell, 1980; Mejdahl, 1979), because not every particle is an individual TL-emitting mineral, but may consist of an agglomerate of very small minerals, each one having received unattenuated dose. However, to exclude errors due to grain size, a narrow distribution of grain sizes would be desirable, preferably with an average grain size of less than 5  $\mu\text{m}$ .

Wet-sifting was used to obtain a narrow grain size distribution independent of firing temperature and clay composition. As before, a suspension of the ceramic material was prepared, filtered through sieves of 10 and 5  $\mu\text{m}$  mesh-width. SEM images of the 5-10  $\mu\text{m}$  fraction revealed a narrower size distribution, and particles of a size less than 3  $\mu\text{m}$ , said to carry spurious light emission, were no longer present. The mean grain size obtained in this procedure was determined by measuring 50 individual particles at a magnification of 2000. The mean diameters obtained for four brick samples #1- #4 of 4.9 $\mu\text{m}$ , 4.4 $\mu\text{m}$ , 4.2 $\mu\text{m}$  and 4.7 $\mu\text{m}$  respectively, are slightly below the low mesh-width, which seems to be due to the particular form of the nylon mesh used.

### **Quartz enrichment**

During the development of fine grain dating the change of composition during sedimentation was studied - in particular, whether the quartz content could be enriched (Zimmerman, 1971). Recent work has shown that quartz can be enriched using magnetic techniques (Janer and Junger, 1982; Kitis and Charalambous, 1985). It is also known from mineralogical techniques, as well as from alpha-

composition of a fine grain fraction, which has been prepared from a bulk material, is not equal to the composition of the latter. The effect of sedimentation and wet-sifting was investigated using XRF on three samples of fired bricks ( ref. L4.5, VV12/11 and Bdf1.0) and the results given in table 1 reveal that there are no significant differences in composition between sedimentation and wet-sifting. Both procedures do, however, significantly change the original composition as a consequence of density separation, which is the desired effect in sedimentation, but obviously cannot be totally avoided in wet-sifting. The most obvious change occurs in potassium-oxide, followed by calcium-oxide. These elements are enriched in sedimented/wet-sifted samples; silicon is hardly altered, and aluminium possibly depleted. It may be concluded from these observations that, using both methods of sample preparation, the potassium content is enriched, which would be in logical agreement with the method of coarse-grain feldspar separation from pottery (Mejdahl, 1985). X-ray diffraction was, unfortunately, not sufficiently sensitive to detect the type of mineral. Thus the present measurements cannot confirm the enrichment of quartz during sample preparation, which was the original aim.

#### ACKNOWLEDGEMENT

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**[PR] Reviewer's Comments (S R Sutton)**

This experiment demonstrates two things concerning the conventional technique for fine grain sample preparation. First, the ultimate grain size distribution can be dependent on the firing history of the material. Second, wet-sifting can be successful in size-separation of these small grains. It is erroneously implied that for accurate dating, such a size separation is required to produce a sample of homogeneous grain size.

The basic assumption of the fine grain dating method is that the measured TL-emitting grains have received the infinite medium dose-rates from alphas and betas corresponding to the bulk sample radioactive element content. Consequently, grain size distributions are irrelevant so long as (1) all measured TL grains are small enough to have experienced negligible alpha attenuation, and (2) the TL grains did not result from the break up of larger grains during vicing. Vitrification may or may not lead to systematic errors. In all cases, one needs to know the TL and radioactivity distributions in order to determine the validity of the resulting glass would tend to be homogeneous in both TL and radioactivity and the infinite medium assumption would apply regardless of the grain size used (of course, laboratory irradiation of such material is another matter). Partial vitrification may prohibit mineral grains from being isolated by vicing but the TL may be dominated by small crystals included within larger glassy fragments.

Clearly, the greatest problem associated with material fired to a high temperature is that it is hard and there will be a greater probability of breaking up large TL grains during vicing. A combination of cathodoluminescence and electron microscopy on the present samples might provide some information on the firing temperature at which such breakup becomes significant.

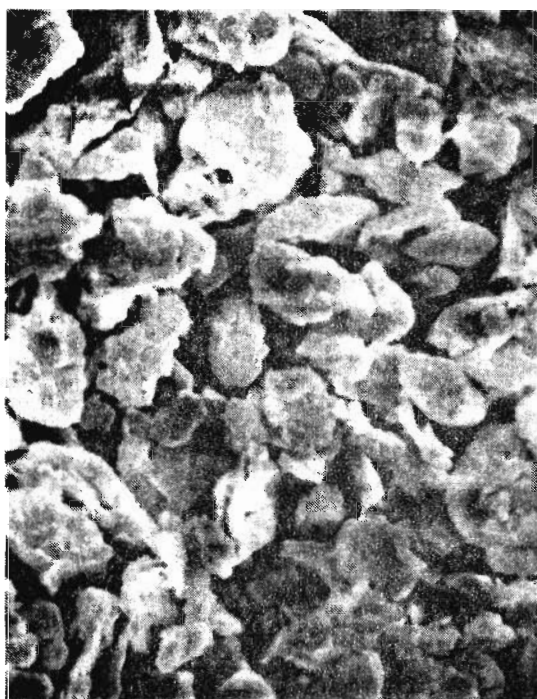
**TABLE 1: CHEMICAL COMPOSITION BEFORE AND AFTER FINE GRAIN SAMPLE PREPARATION**

SAMPLE		TYPE OF TREATMENT		
		none	wet-sifted	sedimented
			5-10 $\mu$ m	2/20min
L 4.5	CaO	3.00	3.25	3.25
	K <sub>2</sub> O	4.38	5.20	6.09
	SiO <sub>2</sub>	70.80	71.56	71.63
	Al <sub>2</sub> O <sub>3</sub>	15.44	12.51	12.51
	TiO <sub>2</sub>	0.86	0.84	0.92
	Fe <sub>2</sub> O <sub>3</sub>	6.09	6.55	6.55
	MnO	0.07	0.09	0.07
	Na <sub>2</sub> O	0.94	0.92	0.83
	MgO	2.54	3.00	3.34
VV 12/11	CaO	2.35	2.87	2.65
	K <sub>2</sub> O	5.55	8.37	7.26
	SiO <sub>2</sub>	64.94	67.74	66.48
	Al <sub>2</sub> O <sub>3</sub>	16.45	11.26	13.62
	TiO <sub>2</sub>	0.90	0.87	0.93
	Fe <sub>2</sub> O <sub>3</sub>	7.76	8.76	8.87
	MnO	0.12	0.13	0.13
	Na <sub>2</sub> O	1.54	1.30	1.25
	MgO	3.11	4.18	3.43
Bdf 1.0	CaO	0.98	1.08	1.06
	K <sub>2</sub> O	2.50	3.43	3.55
	SiO <sub>2</sub>	67.67	67.76	69.62
	Al <sub>2</sub> O <sub>3</sub>	18.07	18.13	17.26
	TiO <sub>2</sub>	0.82	0.91	0.93
	Fe <sub>2</sub> O <sub>3</sub>	5.93	8.08	8.12
	MnO	0.11	0.15	0.15
	Na <sub>2</sub> O	0.83	0.88	0.88
	MgO	2.29	2.33	2.52

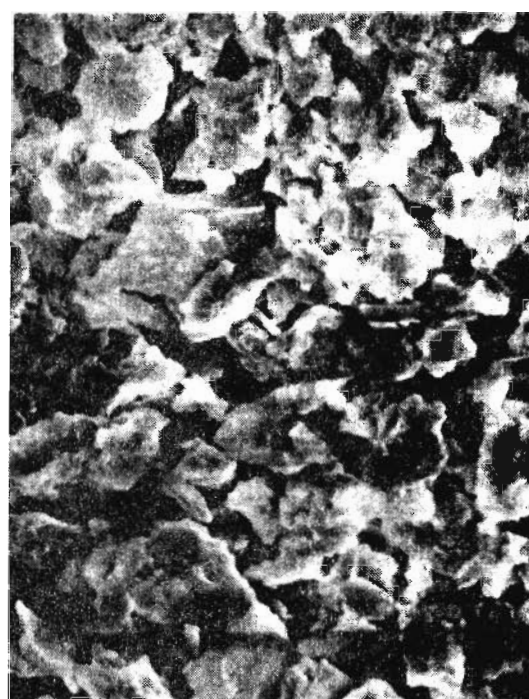
**Notes.** Values are in wt.% and are means of three samples.

Errors: Na<sub>2</sub>O  $\pm$ 5%; Mg $\pm$ 10% (determined by AAS), and others  $\pm$ 3% (determined by XRF).

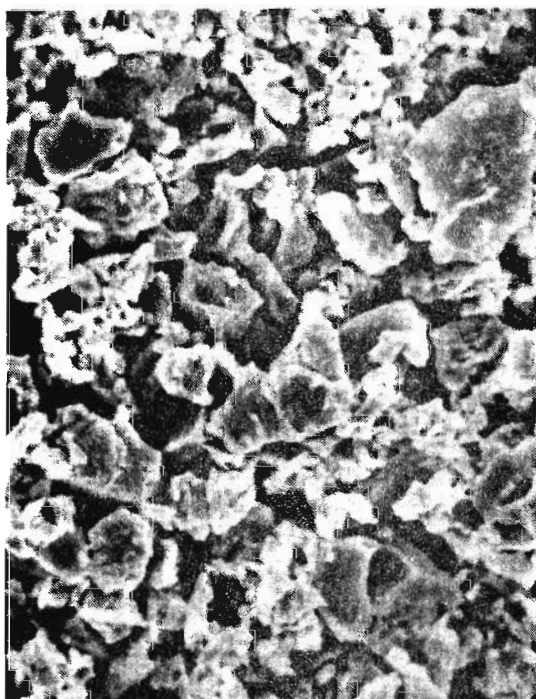
Figure 1.



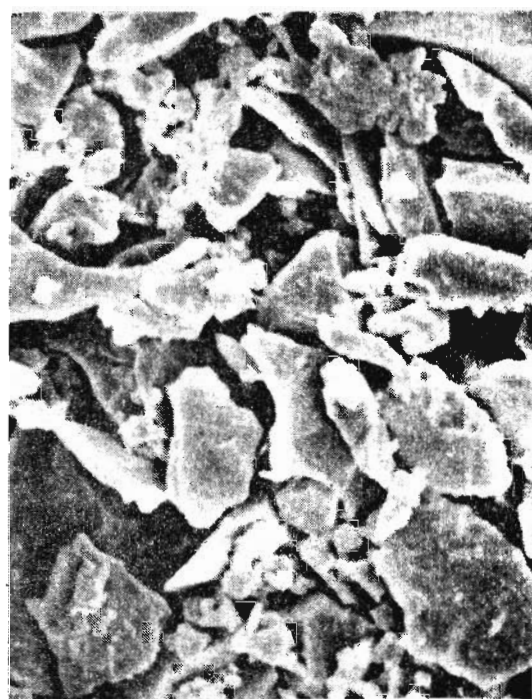
a: 600°C



b: 800°C



c: 1000°C



d: 1200°C

scale |~~~~~| 10μm