

## **DILATOMETRIC STUDIES OF LEH CLAY PART II**

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Clay from the Leh area of J&K State (India) was separated into various size fractions by the method of sedimentation and with Sharple's supercentrifuge process. The results of dilatometric studies of the fractions are discussed in detail.

The thermal expansion of refractories and ceramic materials has been the subject of numerous papers [1-3]. Although a large number of Indian clays from various localities have been investigated in considerable detail by various workers [4-8], these studies have not covered the clays from the Leh area of J&K State (India). The main aim of this investigation is to report on the nature of these clays as revealed by dilatometric studies. This paper is a continuation of a previous communication [9].

### **Experimental**

The clay under investigation was collected from the Leh area (J&K State), which lies between  $77^{\circ} 30'$  and  $77^{\circ} 45'$  E and  $34^{\circ} 0'$ , and  $34^{\circ} 15'$  N, and is situated at an altitude of 11,600 ft. The clay (so.gr. 2.23) occurs in the form of beds which vary from 25 cm to 1 m in thickness; its colour is light olive-grey [10] 5Y 6/1, it has a fine texture and it slakes easily.

The representative clay sample was powdered and separated into various size fractions by the procedure given earlier [9]. The numbers assigned to each fraction are given in Table 1 and the results of chemical analysis of all the fractions are presented in Table 2.

The dilatometric and the differential thermal analysis studies were carried out on a MOM derivatograph (Model OD-103) at a linear heating rate of 10 deg/min.

**Table 1**

Sample no.	Size of fraction
1	Whole clay
2	> 10 $\mu\text{m}$
3	10–5 $\mu\text{m}$
4	5–2 $\mu\text{m}$
5	2–0.5 $\mu\text{m}$
6	<0.5 $\mu\text{m}$

**Table 2** Chemical analysis of the six fractions, samples 1–6

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6
SiO <sub>2</sub>	51.71%	68.70%	67.34%	61.11%	45.60%	42.86%
Al <sub>2</sub> O <sub>3</sub>	21.19%	16.10%	16.58%	17.58%	22.68%	23.17%
Fe <sub>2</sub> O <sub>3</sub>	5.98%	2.55%	3.02%	6.82%	9.59%	10.34%
TiO <sub>2</sub>	0.13%	0.20%	0.11%	0.12%	0.12%	0.10%
CaO	2.79%	1.91%	1.67%	0.98%	0.32%	0.22%
MgO	4.00%	2.04%	2.63%	4.73%	9.07%	9.40%
Na <sub>2</sub> O	2.21%	3.17%	2.97%	0.95%	0.25%	0.12%
K <sub>2</sub> O	2.96%	1.06%	1.56%	2.40%	3.81%	4.31%
L.O.I.	8.73%	3.80%	4.00%	5.50%	8.02%	10.10%

L.O.I. = Loss on ignition

## Results and discussion

The linear change upon the heating of sample 1, i.e. the whole clay, is depicted in Fig. 1a; it shows a small initial shrinkage up to 150°, because of the removal of sorbed water. The moderate expansion up to 750° is the combined result of the expansion of quartz during transition and the exfoliation of the micaceous mineral (illite) and chlorite. The dehydration of illite is followed by the dehydration of chlorite. Illite and chlorite shrink after dehydration, which is manifested by the curve after 750°. The downward trend of the curve continues up to 900°. At about 860°, calcite present in the sample begins to dissociate, giving highly reactive lime as residue. At a temperature slightly above 900°, this lime reacts with alumina and silica in a solid-state reaction, to yield calcium aluminosilicate; the crystallization then occurring checks the contraction in the sample [11]. Although the calcite present in the sample is insufficient to cause a reversal in the linear change curve, crystallization does cause a discontinuity in the linear change curve between 910 and 1000°. These results were confirmed by the DTA data. Figure 2a presents the DTA curve for sample 1.

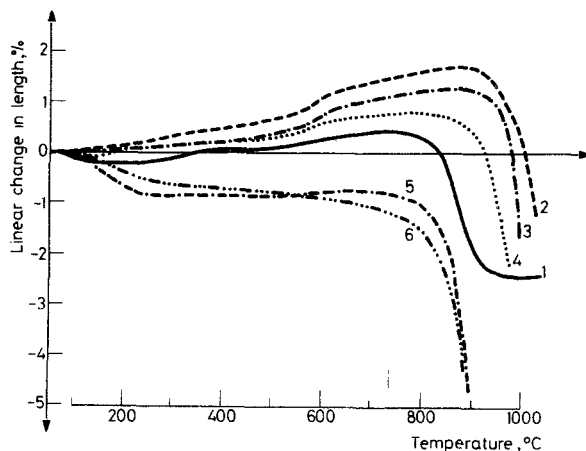


Fig. 1 Dilatometric curves of different fractions of Leh clay

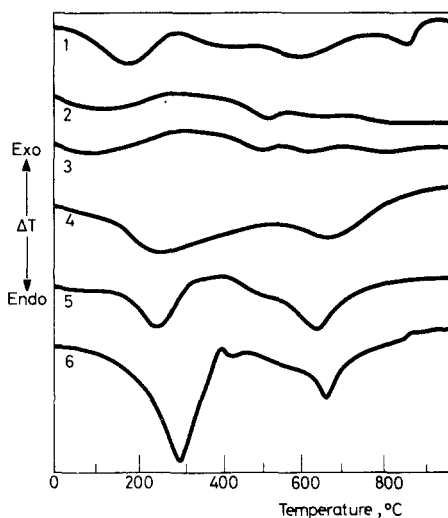


Fig. 2 Differential thermal analysis curves of different clay fractions from Leh area

The dilatometric analysis curves of samples 2, 3 and 4 in Fig. 1b-d do not show appreciable change up to 150°, revealing the presence of very little interlayer water in them. After 150°, all three samples exhibit a slow rate of expansion up to 500°, indicative of the presence of micaceous mineral. Between 500 and 900°, samples 2 and 3 display a sharp increase in their dilation curves, whereas this thermal effect continues only up to 850° in the case of sample 4. The significant increase observed in the region 550–650° is characteristic of the presence of free quartz, which

undergoes the  $\alpha \rightleftharpoons \beta$  transition in this range [12]. The moderate expansion observed beyond 650° may be assigned to the exfoliation of the micaceous mineral illite and chlorite [13]. The shrinkage due to the dehydration of these minerals is insufficient to counterbalance the exfoliation effect. The maximum expansion observed for samples 2, 3 and 4 was 1.75%, 1.50% and 0.80% linear, respectively. The variation in the percentage linear change behaviour arises from differences in the quartz and the mineral components present in these fractions. At about 900° (850° in the case of sample 4), fluxes from mica and chlorite start to form liquid and filling the pores; the samples shrink, which is manifested by the downward trend in their expansion/contraction curves. Here it is observed that sample 3 shows a greater shrinkage than sample 2, but in turn shrinks less than sample 4. This is because the non-plastic component quartz inhibits the liquid formation to an extent proportional to its content in the sample. These observations are in close agreement with the DTA data in Fig. 2b–d. The linear percentage expansion contraction curve for sample 5 (Fig. 1e) reveals a shrinkage in the temperature range 100–300°, due to the loss of interlayer water, after which a period of volume stability is observed. The slight upward trend in the dilatometric curve between 500 and 700° may be attributed to the combined effect of quartz inversion and the exfoliation of micaceous and chlorite minerals. The loss of interlayer water and the dehydration of illite and chlorite in sample 6 are illustrated by the dilatometric curve (Fig. 1f) in the temperature range 100–700°. The exfoliation of these mineral components in this case is not sufficient to counterbalance their dehydration effect [9]. Above 800°, the fluxes from the mica form an appreciable amount of liquid, which is characterized by the sharp and rapid downward trend in the curves of these two samples (5 and 6). The principal feature in the dilatometric curves of these two samples is the shrinkage between 800 and 900°, which amounts to about 5% linear. The DTA curves of these samples in Fig. 2e, f also show results in close agreement with the dilatometric analysis curves.

## Conclusion

Thus, from the dilatometric analysis curves it transpires that samples 2–4 are highly siliceous, containing low percentages of interlayer water and the mineral components illite and chlorite, whereas samples 5 and 6 are rich in mineral components and alkalis. The original clay sample 1 contains a small amount of calcite as impurity in addition to free quartz. These results are in close agreement with those of the chemical analysis of the fractions given in Table 2.

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**Zusammenfassung** — Ton aus dem Leh-Gebiet des J&K Staates (Indien) wurde mittels Sedimentation und nach dem Ultrazentrifugenverfahren nach Sharple in Fraktionen unterschiedlicher Größe zerlegt. Die Ergebnisse von dilatometrischen Untersuchungen dieser Fraktionen werden detailliert diskutiert.

**Резюме** — Глина месторождения Лех (Индия) была разделена методом седиментации и сверхскоростного центрифугирования на различные по размеру фракции. Детально обсуждены результаты dilatометрических исследований отдельных фракций.