

HEAVY MINERALS SEPARATION AND THEIR STUDY BY THE OPTICAL AND SCANNING ELECTRON MICROSCOPY

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The heavy mineralogical investigation of the soil sample involved from the Khurrianwala soil series suggests a mixed parentage dominated by schists of low, e.g., chlorite to high, e.g., kyanite, grade together with mafic igneous, e.g., clinopyroxene. Investigations of the quartz grains at submicroscopic level revealed the solution and precipitation of $\text{SiO}_2 \cdot n\text{H}_2\text{O}$, alongwith V-shaped etching as the main shaping mechanisms of minerals.

INTRODUCTION

The soils are formed mainly due to the weathering of rocks composed of specific minerals suit. The soil minerals inherited from rocks are characterised to understand soil processes. To concentrate specific minerals phases, minerals grain are separated according to their specific gravity. Heavy minerals ($\text{SG} > 2.95 \text{ g cm}^{-3}$) are resistant to weathering and provide information on source rock for soils. The isolation of heavy minerals is possible by sinking the specific density minerals in liquids of known specific gravity and isolation may be performed through density separation. A search of the literature suggested the use of tetrabromoethane $\text{SG} = 2.95 \text{ g cm}^{-3}$ (Jackson, 1956). The density of tetrabromoethane matches closely to the lower limit of the heavy minerals. The technique is very well explained by Jackson, (1956).

Microtextural features of heavy/

light minerals give considerable insight into the environmental history of the soil parent material and some indication of the processes of soil environment. The prints of physical and chemical processes, especially dissolution and precipitation are preserved on the heavy minerals surfaces. Glacial, fluvial, aeolian, and chemical agencies impose their recognisable characteristics feature and etch pattern on grain surfaces (Krinsley and Doornkamp, 1973). A set of distinctive sand grains microtextures has been recognised by Krinsley and Donahue (1968), Krinsley and Margolis (1969) providing a means for distinguishing grains which have been acted upon by chemical and mechanical processes in various environments. Micro-morphology of these minerals also allow the estimation of the relative age of the soil (Douglas and Platt, 1977; and White, 1981).

No published data have been found on the heavy minerals of the soils of Pakistan. It is, therefore,

planned to study the heavy minerals from some soil profiles of Pakistan in order to gain an insight into the past environmental history of the profiles and the parent material.

MATERIALS AND METHODS

A subsurface soil sample collected at the depth of 65–70cm of the Khurrianwala soil series (*Natric Camborthid*) was air-dried and sieved through 2mm sieve. The physical and chemical measurements (Richards, 1954) suggest the soil was saline-sodic. The texture was silty clay loam. All measurements were made on duplicate air-dried soil sample and results are presented on an oven-dried basis.

The fine sand fraction (200–63 μm) was obtained by wet sieving and used for the separation of heavy and light minerals. Five grams of the fine sand fraction was dispersed in tetrabromoethane (SG 2.95) in a tapered centrifuge tube. The mixture of the fine sand fraction and tetrabromoethane was centrifuged at 1500 rpm. The operation was repeated thrice, each time redispersing light and heavy fractions. A plunger was then used to seal the heavy minerals in the tapered part of the tube and the light minerals poured into a filter paper (Whatman 541) under hydraulic suction. The light and heavy fractions were washed with acetone and placed in an oven to dry at 105°C. The percentage of the light and heavy fractions were calculated on oven drying basis.

The heavy minerals were then studied under an optical micro-

scope both immersing in methyl salicylate (Refractive Index = 1.54) in a watch glass and also in a permanent mount on a glass slide using Canada Balsam (R.I. = 1.54). Some individual heavy and light minerals grains were also separated to study their microtextural features by the scanning electron microscopy (SEM).

Studies by SEM: The heavy/light minerals were identified first under the optical microscope and hand-picked, dried and stored separately. The adhesive material from a 10cm long (1 cm wide) sellotape was dissolved in 10ml of trichloroethane and a drop of the solution was placed on a carbon stub. The dried heavy minerals were then sprinkled on this stub. The mounted stub was coated under vacuum with a layer of gold/palladium (20–30 nm) thick to prevent charge build up on the specimen, and to hold the surface of the specimen at a constant electric potential. A current of 10 mA was maintained during the coating, and a time of 7–10 minutes was found suitable. A polaron E-5000 sputter coating unit was used in the study. The gold/palladium coated stub was then used on the SEM (Hitachi S-520). Photographs were taken at X20, X200 and X2000 magnifications on a 0.5mm grid. Only representative features and those of specific nature were magnified further.

RESULTS AND DISCUSSION

The results of the present study will be described in two sections i.e., 1) heavy minerals study by the optical microscopy; and 2) SEM studies. **Heavy-minerals study by the optical microscopy:** The heavy minerals

fraction as seen under the optical microscope contain apatite, biotite, chlorite, clinozoisite, epidote, garnet, green amphibole, kyanite and tourmaline as common grains; topaz and zircon were found occasionally and glaucophane, hypersthene, clinopyroxenes, and staurolite were found rarely. The analyses of the heavy minerals suggested mixed parentage dominated by schists of low, e.g., chlorite to high, e.g., kyanite, grade together with mafic igneous, e.g., clinopyroxenes.

Microtextural investigations by SEM: Only one light mineral species i.e., quartz has been investigated by SEM. Multiple microtextural features and heterogeneity were observed (by SEM) on the quartz surfaces. Briefly, the microtextural features observed were: 1) Angular, irregular and rounded shapes, 2) Conchoidal fractures, 3) Si-precipitation, 4) Rough grain surfaces, 5) Oriented V-shaped, dish shaped and crescentic chipping and upturned plates. Only few will be described here.

Fig. 1a shows a composite entire quartz grain with a rough surface which indicates that this grain probably has experienced much chemical weathering compared to other grains. In the same fig. smooth surface is also evident, this suggests that the roughness around the smooth surface may be increased by differential chemical weathering, which relates to difference in chemical resistance within the grain (Krinsley and Doornkamp, 1973) or this may be the application of the principle of Sorby

(1863) and Riecke (1895) whereby a mineral grain under stress has an increased solubility. If a grain is stressed inhomogeneously it will be dissolved at stressed points and precipitation may occur on the surface of lower stress. This grain was found to have its surface almost completely fretted. The detailed examination of the surface roughness revealed three points i.e., 1) the rough surface is covered by oriented crystal faces due to intense etching (fig. 1b), 2) two orientations of superficial crystals showing well developed prisms and rhombohedral faces (fig. 1c) and 3) Si-precipitation as fibres and smooth areas on the crystal surfaces (fig. 1b).

The rate of growth of quartz crystals varies with crystallographic directions and is most rapid in the direction of the c-axis (Van-Praag 1947 & 1949). In another study Ballman and Laudise (1963) have determined the ranking of growth rates of the more common faces. They suggested that the basal plane is not a naturally occurring face in quartz, but is often used in quartz synthesis because of its rapid growth rate. From the observations of the quartz crystal faces growth it is suggested that the closely spaced quartz crystals with different crystallographic planes could grow into the large composite crystal, and in each plane the crystallographic direction is identical for adjacent sub-units are able to grow into single continuous lattice structure, this is evident in fig. 1b.

Examination of another rough surface quartz grain (fig. 1d) also

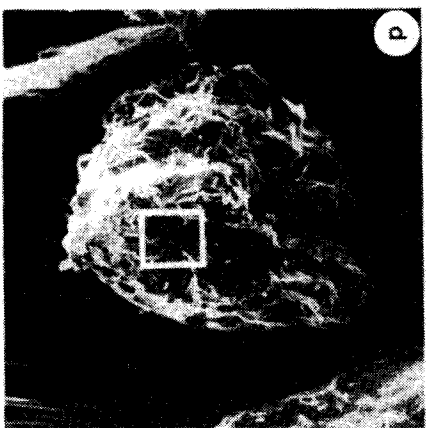
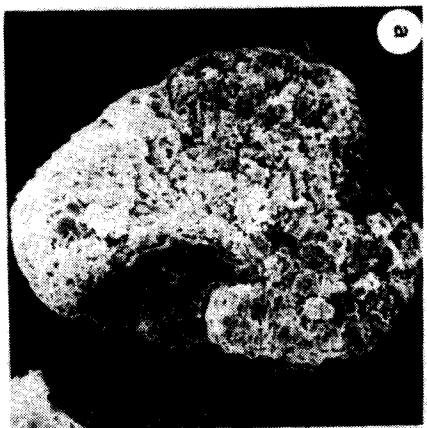


Fig. 1. a. Rough surface of a composite quartz grain. b. Oriented crystals faces and Si-precipitation. c. Two orientations of etched (?) superficial crystal showing well developed prisms and rhombohedral faces. d. Rough surface of a quartz grain. e. Details from "d" showing granular crystals.

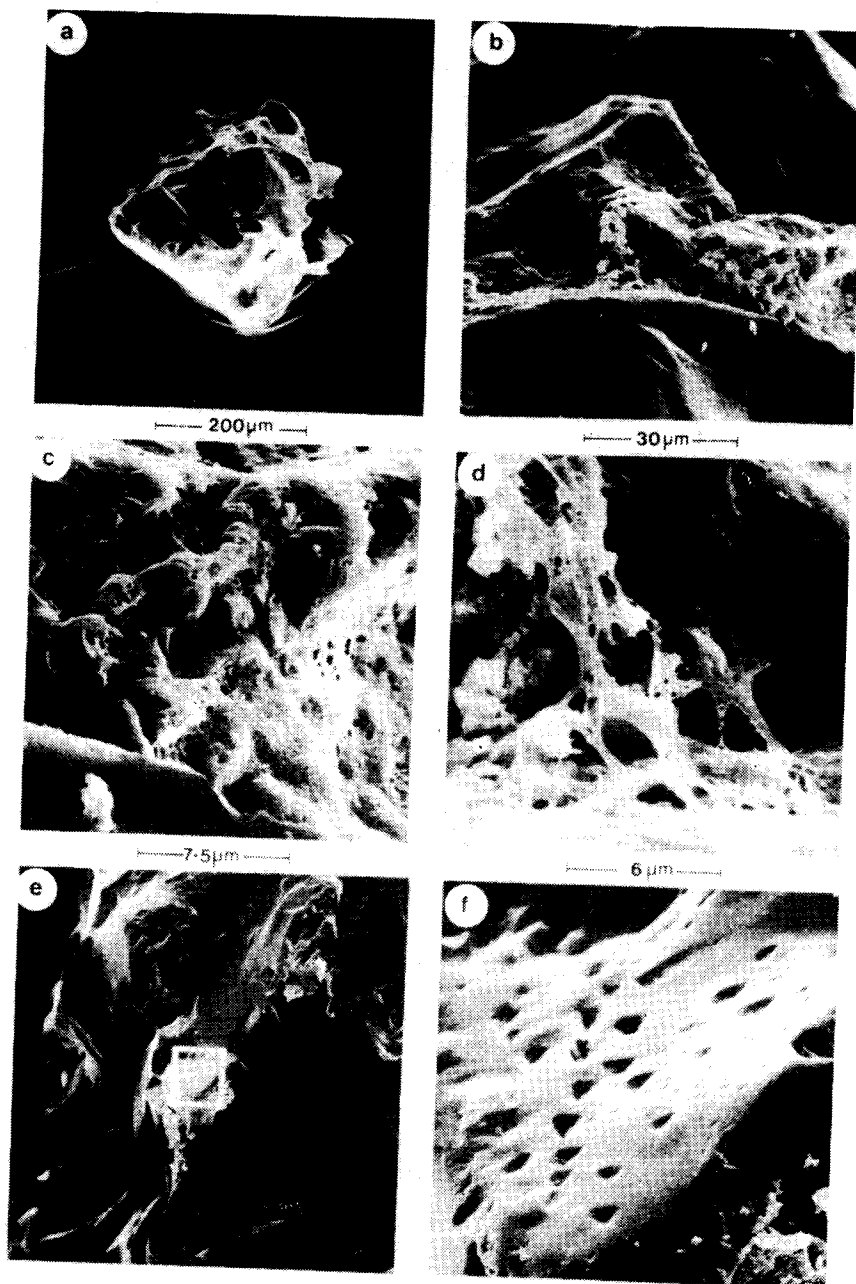


Fig. 2. a. Quartz grain with Si-precipitation.
 b. Si-precipitation and solution pits.
 c. Smooth Si-coatings with solution pits.
 d. Si-precipitation revealing cellular stranded linkage.
 e. Quartz grain with oriented V-etch pits.
 f. Details of oriented V-etch pits and smoothing of surfaces by Si-precipitation.

revealed the crystals growth. A selected view has been shown in fig. 1e, which suggest granular development of the crystals.

Fig. 2a shows a quartz grain, plastering of $\text{SiO}_2 \cdot n\text{H}_2\text{O}$ and some solution pits covered the topographical features of the quartz grain. Smooth Si-precipitation with solution pits were also observed (fig. 2b). The SEM observation suggested that Si-precipitation covered the topographical features, then re-exposed through solution pits (fig. 2b, c, d). On some quartz grains oriented V-shaped pits were also seen (fig. 2e, f). The smooth surface of the V's suggested the precipitation, which must have commenced after the patterns formation. The similar action of water evaporation in a hot environment during the day/night cycle has been considered by Kuenen and Pedrok (1962). They concluded that the pH of the water rises due to the presence of dissolved evaporites during the evening which leads to the removal of a small amount of Si from the quartz grain surfaces. The removed Si is redeposited on the grain surfaces as an irregular layer of either opal or silicic acid which smooths out or subdues the preexisting mechanical fracture pattern. Deposition of Si is predominantly on the flat surfaces, but eventually all parts of the grain become covered and irregular rounding of the grain may take place. Margolis and Krinsley, (1971) suggested that silica precipitation occurs as an amorphous layer on grain surfaces where the solution

evaporates, resulting in a progressive rounding of sharp edges and subduing of irregular mechanical fracture patterns. Silica precipitation pattern allows differentiation of tropical desert sand from coastal sand dunes and periglacial sands.

REFERENCES

- Ballman, A. A. and R. A. Laudise. 1963. Hydrothermal growth. In Gilman, J.J. (ed). The art and science of growing crystals. John Wiley and Sons, New York, N.Y., P. 1-493.
- Douglas, L.A. and D.W. Platt. 1977. Surface morphology of quartz and age of soils. Soil Sci. Soc. Am. J., 41:641-645.
- Jackson, M.L. 1956. Soil chemical analysis-advanced course. p. 477-485.
- Krinsley, D.H. and J. Donahue. 1968. Environmental interpretation of sand grain surface textures by electron microscopy. Geol. Soc. Am. Bull., 79:743-748.
- Krinsley, D. H. and J. C. Doornkamp. 1973. Atlas of quartz sand surface texture. Cambridge University Press, London.
- Krinsley, D.H. and S.V. Margolis. 1969. A study of quartz sand grain surface textures with the electron microscope. New York Acad. Sci. Trans. Ser., 31(2): 457-477.
- Kuenen, Ph. H. and W.G. Pedrok. 1962. Frosting and defrosting of quartz grain. J. Geol., 70:648-658.
- Margolis, S.V. and D.H. Krinsley.

1971. Submicroscopic frosting of aeolian and subaqueous quartz sand grains. *Geol. Soc. Am. Bull.*, 82:3395-3406.
- Richards, L.A. (ed) 1954. *Diagnosis and Improvement of Saline and Alkali Soils*. U.S.D.A. Handb. 60 (Washington D.C.).
- Riecke, E. 1895. Über das Gleichgewicht zwischen einem festen homogen deformierten Körper und einer flüssigen Phase: *Annalen der Physik*, 54:741-748.
- Sorby, H.C. 1863. Über Kalkstein-Geshiebe mit Eindrücken. *Neues Jahrb Mineral Abh.*, p. 801-807.
- Van Praag, G. 1947. Synthetic quartz crystals. *Geol. Mag.*, 84:98-100.
- Van Praag, G. 1949. The hydrothermal crystallisation of vitreous silica at constant temperature. *Discussion of the Faraday Soc.*, 5:338-341.
- White, K.L. 1981. Sand grain micromorphology and soil age. *Soil Sci. Soc. Am. J.*, 45:975-978.