

INFRARED ABSORPTION ANALYSIS OF SOME EAST PAKISTANI CLAYS

M. ALIM BISWAS AND A. K. BASAK

Glass and Ceramics Division, East Regional Laboratories, Pakistan Council of Scientific and Industrial Research, Dacca

(Received July 27, 1961)

Infrared absorption spectroscopic study of Bijaipur clay and Mirpur clay (red and black) samples of East Pakistan has been made by the use of a double beam infrared spectrophotometer. Clay samples of requisite particle sizes have been prepared. Characteristic absorptions given by all the three clay samples under examination are identical with those of the clay mineral, kaolinite.

Introduction

The mineralogical composition of clay determines to a great extent its physico-chemical properties and its suitability for uses in industries. Infrared absorption spectroscopy is, at present, a well developed technique for identification of clay minerals present in a clay. This technique, although developed recently, has attracted a number of workers, e.g. Hunt,¹ Keller and Pickett,² Hunt and Turner,³ Adler,⁴ Farmer,⁵ Stubican⁶ who have employed this technique to the study of various clay minerals and related materials. In their publications they have reported the results of infrared absorption analysis of the chief clay minerals and have discussed various related problems. The Indian workers Atma Ram et al.,⁷ and Bishui and Prasad,⁸ have also employed this technique for the mineralogical study of some Indian clays. In a recent review, Nahin⁹ has discussed the present position of infrared absorption analysis of clay minerals and its future application to possible fields.

Infrared absorption analysis has not yet been used for the study of any Pakistani clay. In view of this fact and of the increasing application of this technique to the study of clay minerals, the authors have employed this technique to the mineralogical study of some East Pakistani clays which are present in abundance and can be put to various uses.

Experimental

Preparation of Samples.—Particle size requirement is crucial for obtaining definitive infrared spectra of a mineral. Keller observed that clay or mineral particles having diameters smaller than 5 microns were required for obtaining a good infrared spectrogram. The clay samples under examination were prepared according to the method given by Hunt and Turner.³ About 5 g. of a clay sample was dispersed in 250 ml. of distilled water using about 15 ml. of 0.2 normal sodium oxalate solution. The mixture was vigorously stirred in a long beaker for about 15 minutes and was then poured

into a 250 ml. measuring cylinder and was allowed to stand for two hours. At the end of this time the upper 10 ml. of the suspension were drawn off without disturbing the rest and centrifuged to separate the solid from the liquid. The solid then dried in an oven between 105° and 110°C. for about 24 hours and was kept in a desiccator for future use. A sample, thus prepared, contained requisite clay particles having diameters smaller than 5 microns and about 0.15 - 0.2 g. of such a sample was sufficient for taking infrared spectra.

Some of the samples of the clays under study were prepared in the above manner using concentrated ammonium hydroxide solution as the dispersing agent in accordance with the method given by Mackenzie.^{10,11} These samples gave identical results with the former ones.

Procedure.—A double beamed 'Inracord' Spectrophotometer (Parkin and Elmer, Model 137), was used for recording the infrared spectra of the clay samples under study. An oven dried sample was powdered in a small agate mortar and some of this powder was placed on the sodium chloride window of the spectrophotometer. A few drops of anhydrous isopropyl alcohol was then added to form a paste which was smoothed out on the window with a microscope slide whose edges were polished to prevent scratching. On removal of the slide the alcohol evaporated away and a thin film of clay sample was formed on the window. The window with the thin clay film in it was inserted in the sample beam of the spectrophotometer and the blank sodium chloride window was placed in the reference beam. The film thickness was so adjusted that an initial transmission of 60% was obtained. This method of preparation of a thin film on the sodium chloride window was adopted from that of Hunt.¹

Results and Discussion

Infrared absorption spectra of the clay samples under study are given in Fig. 1 to 3, and the

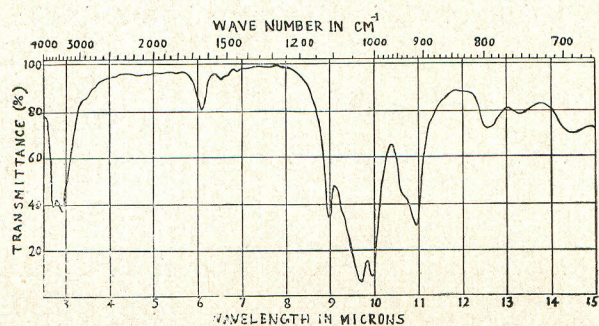


Fig. 1.—Infrared spectra of Bijaipur clay, Mymensingh.

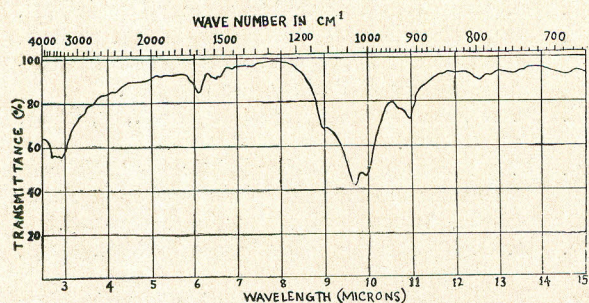


Fig. 2.—Infrared spectra of black clay, Mirpur, Dacca.

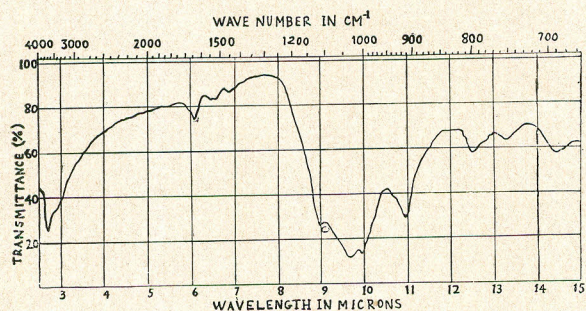


Fig. 3.—Infrared spectra of red clay, Mirpur, Dacca.

position of individual absorption band centres with their relative intensities are given in Table 1. The results of the chemical analysis of the clay samples are given in Table 2.

The main absorption characteristics in these infrared spectra of the clay samples under study,

TABLE 1.—ABSORPTION BANDS OF BIJAIPUR AND MIRPUR CLAYS.

Absorption bands of Bijaipur clay at microns	Absorption bands of Mirpur Clay (Red variety) at microns	Absorption bands of Mirpur clay (black variety) at microns
2.72 (s)*	2.72 (s)*	2.72 (s)*
2.91 (s)	2.95 (w)	2.93 (s)
6.09 (w)	6.10 (w)	6.07 (w)
6.51 (vww)	6.50 (vww)	6.48 (vww)
8.98 (s)	8.97 (s)	8.97 (s)
9.70 (s)	9.69 (s)	9.69 (s)
9.95 (s)	9.95 (s)	9.94 (s)
10.70 (w)	10.68 (w)	10.70 (w)
10.94 (s)	10.95 (s)	10.94 (s)
12.51 (w)	12.61 (w)	12.55 (w)
12.71 (vw)	12.76 (vw)	12.79 (vw)
13.29 (vww)	13.26 (vww)	13.21 (vww)
14.44 (vw)	14.56 (vw)	14.45 (vw)

* (s), (w), (vw) and (vww) correspond to intensities of strong, weak, very weak and very very weak denominations, respectively.

TABLE 2.—CHEMICAL ANALYSES OF BIJAIPUR AND MIRPUR CLAYS.

Clay	SiO ₂ (%)	Al ₂ O ₃ (%)	Fe ₂ O ₃ (%)	CaO (%)	Na ₂ O (%)	K ₂ O (%)	TiO ₂ (%)	Ignition Loss (%)
Bijaipur clay	57.09	30.04	0.32	0.04	0.21	0.11	trace	11.99
Mirpur clay (Red variety)	55.96	23.07	9.57	0.14	0.23	0.12	trace	10.21
Mirpur clay (Black variety)	48.79	26.46	2.50	0.21	0.06	0.04	trace	21.50

are similar. In all the three spectra absorption peaks are all identical. Bijaipur clay has given the strongest absorption peaks while the other two samples of Mirpur clay have produced absorption of lesser intensities. The spectrogram of Bijaipur clay is identical with those of standard kaolinite^{1, 8} mineral and consists of the diagnostic doublet in the region 9.65 to 9.90 microns, a shoulder like formation near 10.70 micron, and a singlet at about 10.95 micron. Although absorption spectrogram of Bijaipur clay differs from those of Mirpur clay samples in absorption intensities, they are fundamentally similar and are of the same mineral. The three clay samples, therefore, contains the same kaolinite clay mineral, but the Mirpur clay samples contain varying proportions of impurities. Chemical analyses of these three clay samples given in Table 2 have also indicated that Mirpur clay (both red and black varieties) contains varying proportion of impurities while Bijaipur clay contains the least amount of impurities and is the purest of the clay samples under study.

Acknowledgement.—The authors are thankful to Dr. M. Q. Khuda, Director, East Regional Laboratories for his keen interest and encouragement during the progress of the work and for his kind permission to publish the results. Thanks are

also due Dr. M. A. Ali of these laboratories for his help in taking the infrared spectra.

References

1. J. M. Hunt, M. P. Wishered and L. C. Bonham, *Anal. Chem.*, **22**, 1478-97 (1950).
2. W. D. Keller and E. E. Pickett, *Am. Mineralogist*, **34**, 855-64 (1949).
3. J. M. Hunt and D. S. Turner, *Anal. Chem.*, **25**, 1169-74 (1953).
4. H. H. Adler, *Am. petrol. Inst. Proj. 49, Prelim. Rept. 8*, (New York) pp. 1-72.
5. V. C. Farmer, *Mineral. Mag.*, **31**, 829-45 (1958).
6. V. Stubican, *Mineral Mag.*, **32**, 38-52 (1959).
7. Atma Ram and B. M. Bishui and J. Prasad, *Central Glass and Ceramics Research Institute (India) Bulletin* **7**, 3-10 (1960).
8. B. M. Bishui and J. Prasad, *ibid.*, **7**, 1960, pp. 97-109.
9. P. G. Nahin, *Proceeding of the First National Conference on Clays and Technology* (San Francisco), *Bulletin* 169, 1955, pp. 112-118.
10. R. C. Mackenzie, *Clay Minerals Bull.*, 3(15), 1956, pp. 4-6.
11. B. M. Bishui and J. Prasad, *Central Glass and Ceramic Research Institute (India) Bulletin*, **6**, 76-85 (1959).