Pakistan J. Sci. Ind. Res., Vol. 28, No. 2, April, 1985

MODIFIED X-RAY DIFFRACTION METHOD, INCORPORATING MASS ABSORPTION CORRECTION, FOR THE QUANTITATIVE DETERMINATION OF CALCITE AND DOLOMITE IN SEDIMENTS

Muhammed Atique Ahmed Baig*

Department of Geology, The University Southhampton, SO_9 5NH England

(Received 4 February, 1984; revised 16 January 1985)

A method of X-ray diffraction, incorporating mass absorption, for the quantitative determination of calcite and dolomite (ferroan), using rock powder pellets of the Oxford Clay sediments, presented in this paper, has produced satisfactory results. Ratios of the peak heights for calcite at 29.43°, 2 θ, and for dolomite (ferroan), at 30.84°, 2 θ, peak positions obtained by X-ray diffraction, corrected for mass absorption and the values of carbon dioxide determined by dividing the total contents of calcite + dolomite, by a factor of 2.2, have been used in this method. The method, presented in this paper has been found fairly rapid and produces accurate and reproducible results.

INTRODUCTION

Many workers including Molina [1], have published methods for the quantitative determination of carbonate minerals found in sedimentary rocks. But none of these methods is applicable when more than one carbonate mineral e.g., calcite and dolomite, is present in the same sample of sediments and their quantitative analysis is desired. The present author has experienced this problem in the quantitative analysis of sediments of the Oxford clay formation of Warlingham Borehole from southern England. On X-ray diffraction, presence of calcite and dolomite (ferroan), in these sediments was indicated by their diagnostic peak positions on the X-ray diffractograms, at 29.43°, 2 θ (3.03 Å) and 30.84°, 2 θ (2.72 Å), respectively, in association with other minerals, as is shown in Fig. 1. To overcome this problem, the present author has modified the X-ray diffraction method published by Cosgrove and Sulaiman [2], for the simultaneous quantitative determination of calcite and dolomite in sediments. In this method, two parameters as pre-requisites are required for the calculation of calcite and dolomite abundances, which are as follows:

1. Ratios of calcite/dolomite peak heights, obtained by X-ray diffraction and corrected for mass absorption.
2. The amount of carbon dioxide determined by an accurate method, representing the contents of calcite and dolomite.

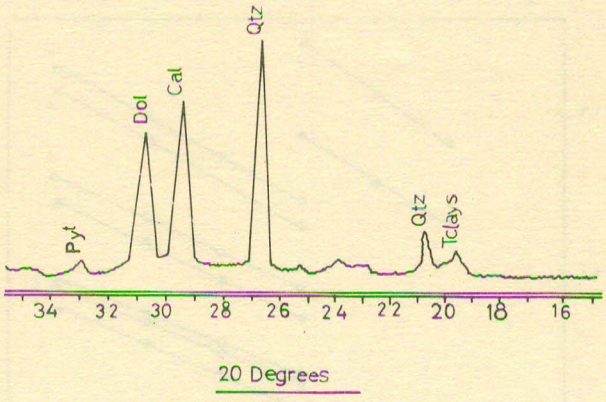

Fig. 1. X.R.D. trace of bulk-rock sample (BR-115) of Oxford clay, showing peak positions of calcite and dolomite (ferroan), in association with other minerals.

MATERIAL AND METHODS

Warlingham borehole core samples of the Oxford Clay and Kellaways Formations, provided by the Institute of Geological Sciences, London, have been used in the present study.

Before determination of the ratios of the X.R.D. peak values the X-ray diffractometer was calibrated by running pellets of the rock standards of a known proportions of calcite and dolomite.

* Present address: Department of Geology, Sind University, Jamshoro, Sind.

Rock standards: Rock standards used include coccolitic limestone and dolomite (ferroan) reported by Bellamy [3], being identical in the content of calcite (coccolithic) and dolomite (ferroan) present in the Oxfar Clay sediments, as confirmed by the X-ray diffraction.

Calibration of X.R D. with rock standards: The Philips PW-1010 X-ray diffractometer was calibrated by running pellets of rock standards for which the values of mass abosrption coefficient were obtained while molybdenum tube programme was in progress. Each pellet was scanned four times from 28°,20 to 33°, 2θ. The mean value of the four readings thus obtained was taken as the final reading. The Calibration line thus produced is shown in fig. 2.

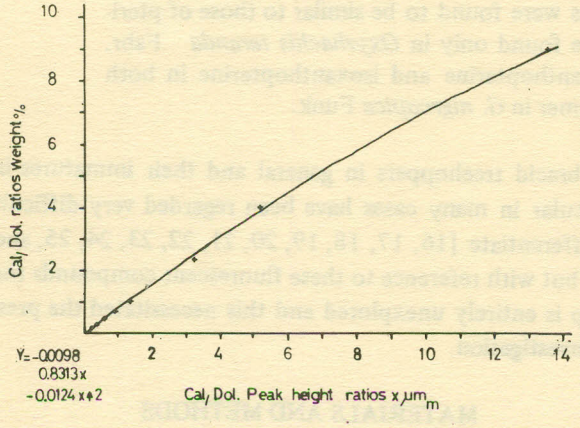

Fig. 2. Calibration line showing very strong positive correlation between weight percent ratios of calcite/dolomite and X.R.D. peak heights ratios of calcite/dolomite of the rock standards, corrected for the mass absorption correction.

Determination of calcite/dolomite X.R.D peak height ratios for Oxford clay sediments: Pellets of the Oxford Clay sediments were prepared for the determination of X.R.D peak heights of calcite and dolomite. Each pellet was scanned on the X-ray diffractometer having the same settings as described for the X-ray diffraction of rock powder pellets of rock standards. Peak heights for calcite and dolomite (ferroan) were recorded at their diagnostic X.R.D peak positions. Mass absorption correction was incorporated by multiplying the mean value of the peak heights obtained with the value of mass absorption coefficient given by the X-ray spectrometry, peak height ratios of calcite and dolomite were calculated.

The abundances of calcite and dolomite (ferroan), calcuated in the present study, when added together and ploted against the total contents of carbonates, determined by o.1N HC1 method, have shown a very strong positive correlation (r=+1,00), highly significant at an 0.01% level of signifcance, as shown in Fig. 3, confirming a satisfactory estimation of the abundance of calcite and dolomite (ferroan).

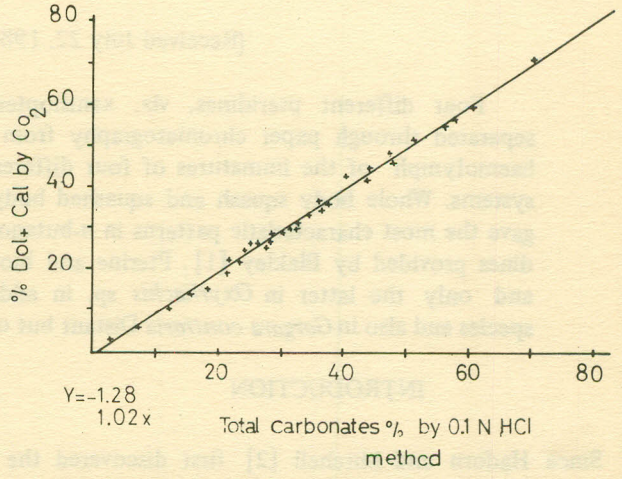

Fig. 3. Very strong positive correlation (r = +1.00), shown by the determined abundances, of total carbonates by 0.1N HC1 method and the total of calcite and dolomite (ferroan), by X-ray diffraction method.

DISCUSSIONS

The X-ray diffraction method described here offers many advantages. It is rapid and gives accurate and reproducible results. It does not require extra time for the preparation of sample for carbonate determination, the pellets of rock powder originally prepared for X-ray spectrometry, serving the purpose. These pellets automatically provide values of the mass absorption coefficient during the analysis of trace elements by X-ray Fluorescence Spectrometer, in the molybdenum tube programme.

CONCLUSION

It is suggested that the X-ray diffraction method, presented in this paper, could be used satisfactorily for the simultaneous quantitative determination of calcite and dolomite, when both occur in the same sample.