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16. Конгрес на хемичарите и технолозите на Македонија, Скопје, 28-30 октомври, 1999

MINERALS FROM MACEDONIA: I. ANALYTICAL APPLICATION OF POWDER X-RAY DIFFRACTION PATTERNS OF CALCITE AND ARAGONITE

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Abstract: The study of powder X-ray diagrams of calcite and aragonite has shown that the appearance of a rather intense diffraction maximum at 2θ = 27.620 ° in the aragonite debyegram (absent in the calcite diagram) as well as the existence of an extremely strong maximum at 2θ = 31.970 ° in the calcite diagram (where aragonite does not exhibit diffraction) make it possible to detect impurities of aragonite in calcite and vice versa. The comparison of the data estimated by both FT IR and powder X-ray diffraction methods has shown that the X-ray diffraction method is more sensitive.

Key words: Macedonia, minerals, calcite, aragonite, analytical applications, powder XRD

1. Introduction

Since carbonate minerals, especially the polymorphic forms of calcite and aragonite (their chemical composition is identical and they both are calcium carbonate) may appear in common mineral agregates, it is advantageous to have a rapid and simple method for detecting impurities of these minerals in one another. In that sense, as a part of the broader research of the structural and spectroscopic characteristics of metal minerals originating from Macedonia, we have previously studied the analytical application of the FT IR spectra and partly of the powder X-ray diffraction patterns of the carbonate minerals calcite and aragonite [1]. It should be noted that the IR and Raman spectra of calcite and aragonite were previously studied [2, 3] including the suggested method on how to distinguish them using FT IR and FT Raman spectroscopy [4].

As in other methods, the detection of the presence of mineral impurities by powder X-ray diffraction in this work was based on the appearance of selected analytical maxima in the diagram region where other constituents of the studied system do not exhibit diffraction. Namely, the analysis of the powder X-ray diagrams of calcite and aragonite has shown that appropriate analytical bands are indeed present in their X-ray diagrams.

2. Experimental

The calcite mineral was from Zletovo while the aragonite mineral sample was taken from the R`anovo ore deposit in Macedonia. They were very carefully picked under a microscope from the ore samples.

The appropriate artificial mixtures of calcite and aragonite were prepared by mixing certain amounts of pure minerals, the total mass of the samples always being 2 mg.

The X-ray powder diagrams were recorded on a Philips Analytical Diffractometer type PW3710 using Cu$K\alpha$ radiation.

3. Results and discussion

In Figures 1 and 2 are shown the powder X-ray diagrams of aragonite and calcite, respectively, whereas the diagram of the corresponding calcite sample with aragonite impurities is presented in Fig. 3.

The study of powder X-ray diagrams of calcite and aragonite has shown that the appearance of rather intense diffraction maximum at $2\theta = 27.620\ ^{\circ}$ in the aragonite debyegram (absent in the calcite diagram) as well as the existence of a very strong maximum at $2\theta = 31.970\ ^{\circ}$ in the calcite diagram (where aragonite does not exhibit diffraction) enables detecting impurities of aragonite in calcite and vice versa.

In order to determine the presence of aragonite impurities in the calcite samples the series of synthetic calcite : aragonite mixtures have been prepared and their powder X-ray diffraction diagrams have been analyzed. The analysis has shown that the limit of detection of impurities of aragonite in calcite is between 1 % and 2 % (Fig. 3).

The presence of an extremely strong maximum at $2\theta = 31.970\ ^{\circ}$ in the calcite diagram (where, as it was mentioned, aragonite does not exhibit diffraction) (see Figs. 1 and 2) makes it possible to detect the impurities of calcite in aragonite even in the cases when the presence of the latter is lower than 1 %.

The comparison of the data previously estimated by FT IR spectroscopy [1] and the data obtained using powder X-ray diffraction methods (this work) has shown that the X-ray diffraction is more sensitive method for determination of the impurities of calcite or aragonite in the calcite-aragonite mineral mixtures.

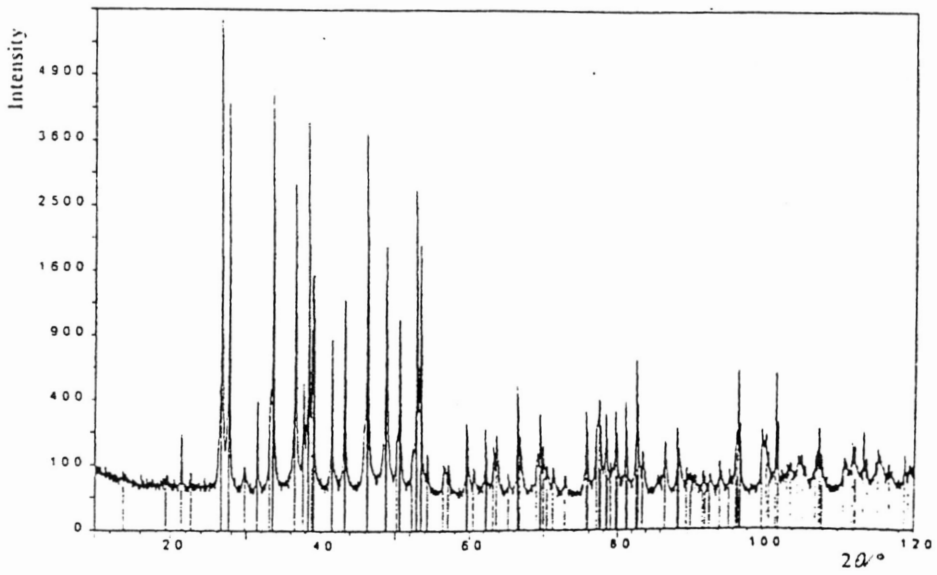

Fig. 1. Powder X-ray patterns of aragonite.

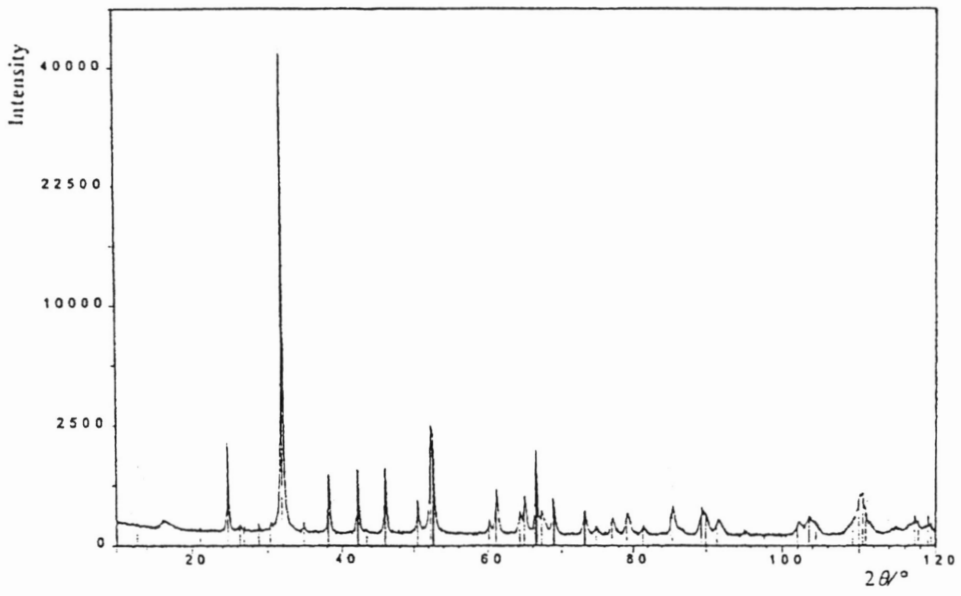

Fig. 2. Powder X-ray patterns of calcite.

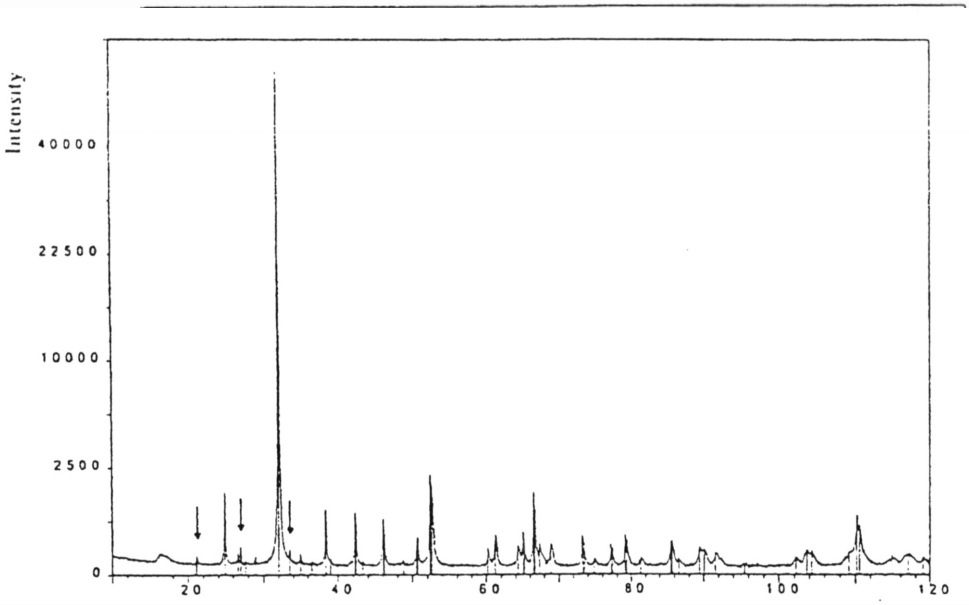

Fig. 3. Powder X-ray patterns of mineral mixture containing 98% calcite and 2% aragonite. The aragonite maxima are denoted by arrows.

4. Conclusion

The analysis has shown that the powder X-ray diffraction is a more sensitive method for the detection of the impurities of calcite or aragonite in the calcite-aragonite mineral mixtures than the FT IR spectroscopy.

5. References

[1] G. Jovanovski, B. Šoptrajanov, V. Stefov, B. Jovanovski, B. Kaitner, *XVI Croatian Meeting od Chemists and Chemical Engineers*, Split, 1999, Book of Abstracts, E6, 184.

[2] D. R. Tylor, R. S. Growther, P. Sharrock, J. Wu, R. D. Soloway, *Hepatology*, **22**, 488 (1995).

[3] R. Frech, E. C. Wang, J. B. Bates, *Spectrochim. Acta*, **36A**, 915 (1980).

[4] J. Tao, Y. Xu, Z. Xu, J. Wu, D. Xu, *XVIth International Conference on Raman Spectroscopy*, Cape Town, 1998, Proceedings, 716.

МИНЕРАЛИ ОД МАКЕДОНИЈА: I. АНАЛИТИЧКА ПРИМЕНА НА РЕНДГЕНОГРАМИТЕ НА СПРАШЕНИ ОБРАСЦИ ОД КАЛЦИТ И АРАГОНИТ

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Карбонатните минерали понекогаш се појавуваат во заеднички агрегати. Заради тоа е потребно да се развиваат едноставни (но брзи и точни) методи за определување на многу мали количества на една или повеќе компоненти во мешаниот природен минерален образец.

Продолжувајќи го испитувањето на структурните карактеристики на металните минерали по потекло од Македонија, работено е и на аналитичката примена на рендгенската дифракција за определување на примеси од калцит во арагонит и обратно.

Слично како и при примената на другите методи, определувањето на присуството на минерални нечистотии со помош на рендгенска дифракција беше засновано на избирање на аналитички максимуми во оние подрачја од рендгенограмите каде што другата компонента од системот не покажува дифракциона активност. Анализата на синтетичките смеси од двата минерала со различен однос арагонит : калцит покажа дека долната граница на детекција на нечистотии од арагонит во калцит изнесува измеѓу 1 % и 2 %, а границата за определување на нечистотии од калцит во арагонит е дури под 1 %.

Споредбата на резултатите од аналитичката примена на рендгенската дифракција за определување на нечистотии од калцит или арагонит во калцит : арагонитни минерални смеси покажа дека рендгенската дифракција е поосетлива метода од Фуриетрансформната инфрацрвена спектроскопија.

Клучни зборови: Македонија, минерали, калцит, арагонит, аналитичка примена, рендгенска дифракција на спрачени обрасци.