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# Some Notes on Methods of Investigation of Coal in Infra-red Spectra

by

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We have already published a paper on the infra-red spectra of extracted coals [1]. The latter contained a fairly large proportion of inorganic matter, and there was some doubt regarding the interpretation of certain bands in the spectra. We have, therefore, started examining infra-red spectra of demineralized coals in order to elucidate the influence of both extraction and demineralization upon the basic structure of coal.

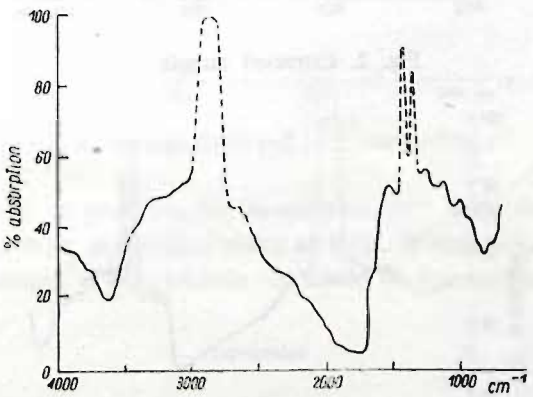



Fig. 1. Original sample

Four samples of brown coal from Konin were examined. Sample 1 was raw coal; Sample 2 was coal extracted with a benzene-ethanol mixture (70:30) as in our previous work [1]. Samples 3 and 4 were demineralized by the Radmacher-Mohrhauer method [2], described below. Sample 4 was subsequently extracted as above. The results of elementary analysis of the samples are tabulated (Table I).

TABLE I

Elementary analysis of samples

| No. | Sample | Elementary analysis C | H | O+N+S | Volatile matter | Moisture | Ash | Extracted matter |
|---|---|---|---|---|---|---|---|---|
| 1 | Raw coal | 66.7 | 5.6 | 27.7 | 52.5 | 11.6 | 9.0 | 14.5 |
| 2 | Extracted coal | 65.0 | 5.5 | 29.5 | 44.0 | 10.4 | 9.7 | — |
| 3 | Demineralized coal (Radmacher-Mohrhauer method) | 65.6 | 5.3 | 29.1 | 54.4 | 4.6 | 0.6 | 31.3 |
| 4 | Demineralized and extracted coal | 64.7 | 5.2 | 30.1 | 50.5 | 8.3 | 0,7 | — |

All results are referred to the ash-free substance.

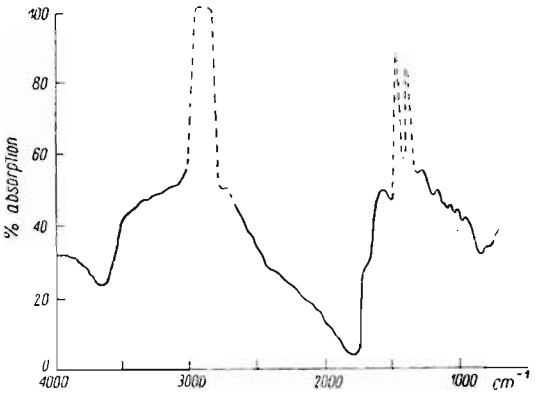
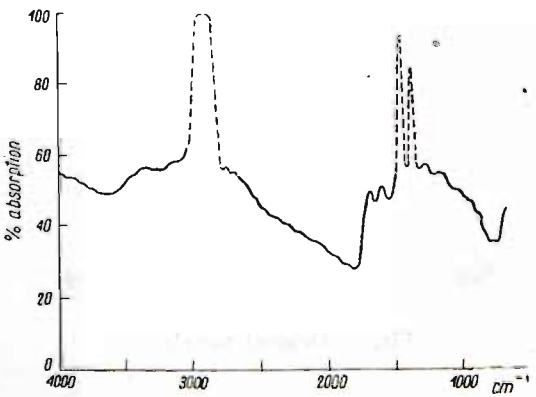



Fig. 2. Extracted sample




Fog. 3. Demineralized sample

The infra-red spectra of Nujol mulls (50:50) of the samples were measured in a Hilger — 800 spectrometer; they are given in Figs. 1—4 (the broken lines represent regions of strong absorption by Nujol). Band frequencies are tabulated (Table II).

TABLE II

Band frequencies in cm [2]

| | | |
|---|---|---|
| 1 | Raw coal | c. 3300 (*s, b*), 1690 (*sh*), 1560 (*m, b*), 1280 (*w*), 1170 (*w*), 1070 (*vw*), 1030 (*w*), (960 (*w*) |
| 2 | Extracted coal | c. 3300 (*s, b*), 1690 (*sh*), 1560 (*m, b*), 1280 (*w*), 1170 (*w*), 1030 (*w*), 960 (*sh*) |
| 3 | Demineralized coal | c. 3300 (*w, b*), 1690 (*m*), 1600 (*m*), 1280 (*vw*), 1170 (*vw*), 1030 (*sh*), 960 (*sh*) |
| 4 | Deminalized and extracted coal | c. 3300 (*m, b*), 1690 (*m*), 1600 (*m*), 1280 (*w*), 1170 (*w*), 1030 (*vw*), 960 (*sh*) |

Abbreviations: *b*—broad, *s*—strong, *m*—medium intensity, *w*— weak, *vw*—very weak, *sh*—shoulder.

**Demineralization of the samples**

A sample of the coal of grain diameter less than 0.2 mm was placed in a vessel of plastic and treated with hydrofluoric acid (pure for analysis) for 1 hour at room temperature. The acid was then decanted and the sample was treated with conc.

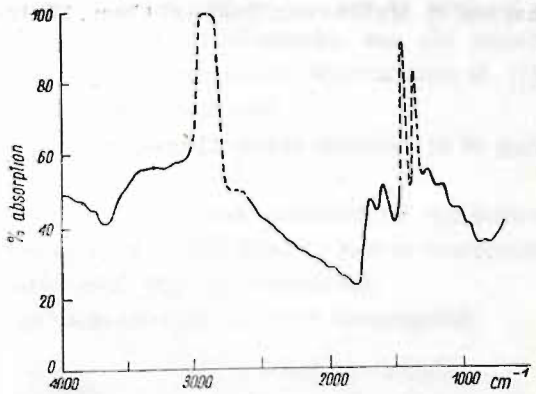



Fig. 4. Demineralized and extracted sample

HCl (*d* 1.19) in two 50 ml portions, for 30 minutes each. Then the coal was filtered off and washed with 0.5 l of distilled water at 80°C. If necessary, the washing was continued until no traces of the chloride ion could be detected in the filtrate.

**Discussion**

As it appears from the spectra, extraction does not affect the basic structure of coal; only minor changes can be observed in the "fingerprint" region (1300—100 $cm^{-1}$). However, the bands in the spectra of extracted samples are more pronounced as compared with those of unextracted ones, thus it seems that extraction may be a useful procedure in the investigation of coal structure.

Demineralization when carried out with hydrofluoric acid leads to more significant changes in the spectra. The OH absorption intensity in the region near 3300 $cm^{-1}$ diminishes and a fairly strong band replaces the shoulder at 1690 $cm^{-1}$.

This would suggest that the number of free carbonyl groups increases, and the number of hydroxyl groups decreases in the course of demineralization with HF. There is also a change in the region of aromatic skeletal vibrations; we observe a band 1600 $cm^{-1}$ in the spectra of demineralized samples instead of that at 1560 $cm^{-1}$ in the spectra of the samples that were not treated with HF.

Since the analytical results show that the elementary composition of the coal is only slightly altered by the action of HF, the changes in the spectra are most probably due to some internal rearrangement in the structure of the coal.

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