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An Examination of the Sulphur-binding Capacity of Charcoal by Means of X-Ray and Electron Ray Diffraction

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In an earlier investigation Wetterholm and Davidson [1] discussed briefly the properties of charcoal (degree of carbonisation, specific surface etc.) and its influence on the capacity for binding sulphur so as to make it impossible to extract it with solvents such as carbon disulphide and hot aniline. Juza and Blanke [2] and Wibaut *et al.*[3] have given some evidence that surface compounds can be formed in the charcoal-sulphur system. Wetterholm and Davidson [1] could show by extraction with hot aniline that the content of sulphur in the system decreased slowly with the extraction time. Thus it seemed probable that the system could not be regarded as a chemical compound. Instead, the sulphur may, during the preparation of charcoal-sulphur, be distilled into the capillaries of the charcoal. (It must be difficult to detect any sulphur in these capillaries). This is also confirmed by Wibaut,[3] that at a temperature of 1000° C in vacuum the sulphur can be distilled off except for a very small portion.

In order to elucidate the structure of the charcoal-sulphur system, electro-optical and X-ray investigations have been made on pure charcoal, mixtures of charcoal and sulphur, and the charcoal-sulphur »compound» (extracted with either hot aniline or carbon disulphide). The last two samples contained 13 % of sulphur.

In the *electro-optical investigation* the ordinary technique was used (V. K. Zworykin *et al.* Electron Optics and the Electron Microscope, New York 1945). The samples were suspended both in water and in octyl alcohol. The magnification was 1,200 and 20,000 times. No difference was observed between the charcoal-sulphur »compound» and the charcoal. Thus, the particles retain their form and size after the absorption of sulphur.

The electron microscope was then used to give diffraction diagrams of the samples. These are shown in Figs. 1 and 2. Charcoal gives three diffuse rings

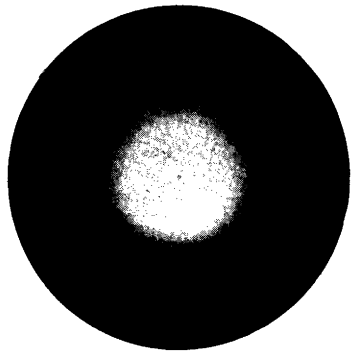

Fig. 1. Electron ray diffraction diagram of charcoal.

Fig. 2. Electron ray diffraction diagram of charcoal-sulphur compound (approx. 13 % S). The preparation has been subjected to extraction with aniline.

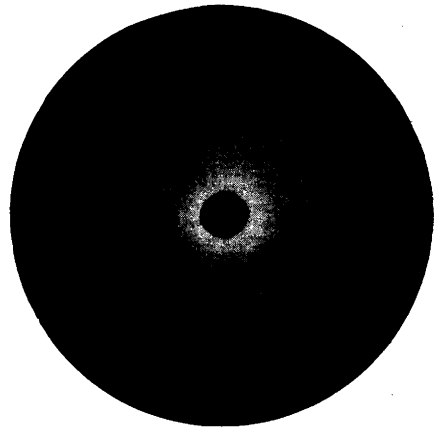

Fig. 3. X-ray diffraction diagram of charcoal.

(Fig. 1). The charcoal-sulphur samples, however, can show either diffuse rings only or both these and a number of sharp rings (Fig. 2). The sharp rings of the various preparations did not always correspond, and they also varied in number. The existence of the sharp rings show that a crystalline structure

Fig. 4. X-ray diffraction diagram of sulphur.

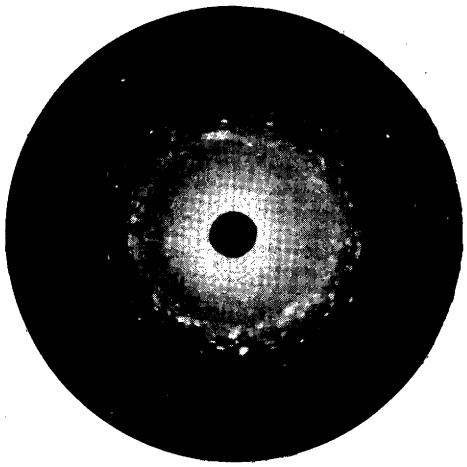

Fig. 5. X-ray diffraction diagram of a mixture of charcoal and sulphur (13.0 % S). The preparation has not been heated.

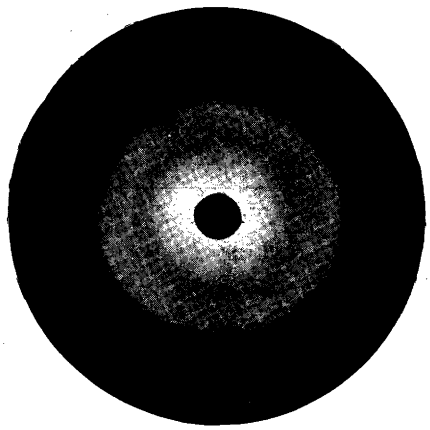

Fig. 6. X-ray diffraction diagram of charcoal-sulphur compound (approx. 13 % S). The preparation has been heated and then extracted with hot aniline.

Fig. 7. X-ray diffraction diagram of charcoal-sulphur compound (approx. 13 % S). The preparation has been heated and then extracted with carbon disulphide.

must be present in the charcoal-sulphur system. In the sample consisting of the charcoal-sulphur mixture, the sulphur evaporated in the microscope and a pure charcoal picture was obtained. A comparison could therefore not be made.

For the *X-ray investigation* CuKα-radiation was used. The samples were compressed into a small rod with 0.8 mm. diameter. With this powder diagrams were obtained (Figs. 3—7). Charcoal gives a very diffuse ring (Fig. 3) whereas sulphur produces a diagram with interference points (Fig. 4). The sulphur is thus distinctly crystalline. The mixture of charcoal and sulphur shows a diffraction diagram which contains the diffraction pictures of both charcoal and sulphur (Fig. 5). The charcoal-sulphur »compound» gives diffraction diagrams containing only one diffuse ring. This ring has, however, the same radius as that of the charcoal but is a little more distinct (Figs. 6 and 7). This is certainly due to some ordered structure in this compound. Crystalline sulphur alone gives quite a different diagram (Fig. 4).

From the investigations performed it seems very probable that on heating charcoal and sulphur some sort of a chemical compound with crystalline structure is formed. It was not possible to detect any change in the diffraction diagrams due to the method of extraction used, in spite of small differences in sulphur content after extraction (Wetterholm and Davidson).[1]

SUMMARY

A compound obtained by heating charcoal and sulphur has been investigated by means of electron diffraction and X-rays. There is evidence that a definite chemical compound with crystalline structure is formed.

This investigation has been made in connection with research work carried out at the request of *Nitroglycerin AB.*, Gyttorp. We are very indebted to the Head of the Institute, Professor The Svedberg, for his kind interest in this work and for the facilities placed at our disposal.

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