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## Note

### Electron-microscopic investigation of the sorbents used in gas-liquid chromatography\*

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The properties of the solid support considerably influence the distribution of the stationary liquid phase (SLP)<sup>1</sup>. Thus investigations in this area are of both practical and theoretical interest.

In the present work the SLP distribution pattern on the microporous diatomite support with surfaces featuring hydrophobic and hydrophilic properties has been studied by scanning electron microscopy (SEM). Non-polar squalane and polar PEG-400 liquid phases were applied *in vacuo* to the Chromaton N AW and N AW DMCS supports in amounts of 10% and 16% respectively of the solid support weight<sup>2</sup>.

After the preliminary conditioning of the column with the sorbents, their chromatographic properties were studied and then the outer and inner surface areas of the sorbent grains were examined by SEM.

The presence and the distribution pattern of the SLP on the solid support was determined by examining the changes in the relief of the initial solid support and also by studying the microcracks formed inside the adsorbed liquid layer under the effect of the electron beam<sup>3</sup>. Thus it was found that inside the grains of the support with the hydrophilic surface the polar SLP (PEG-400) is distributed in a continuous thin layer over the whole bulk of the grain, *i.e.*, uniformly over the inner and outer surfaces of the sorbent grain (Fig. 1). In contrast, in the case of the solid support with hydrophobic properties the polar phase is mainly situated at the outer surface in the form of a thick, non-uniform layer (Fig. 2) and the SLP distribution along the diameter of the sorbent grain is non-uniform.

The introduction of the detergent stearic acid (0.8%), which improves the wettability, changes the distribution of the polar phase at the silanized solid support: the SLP impregnates the whole sorbent grain, forming a thick layer at the outer and inner support surfaces (Fig. 3). An analogous situation is observed for squalane at the hydrophobic support surface (Fig. 4).

The experimentally obtained regular SLP distribution pattern on the diatomite support agrees with that expected theoretically. This, in accordance with the thermodynamic model proposed by Giddings<sup>4</sup>, when the support surface is easily wettable

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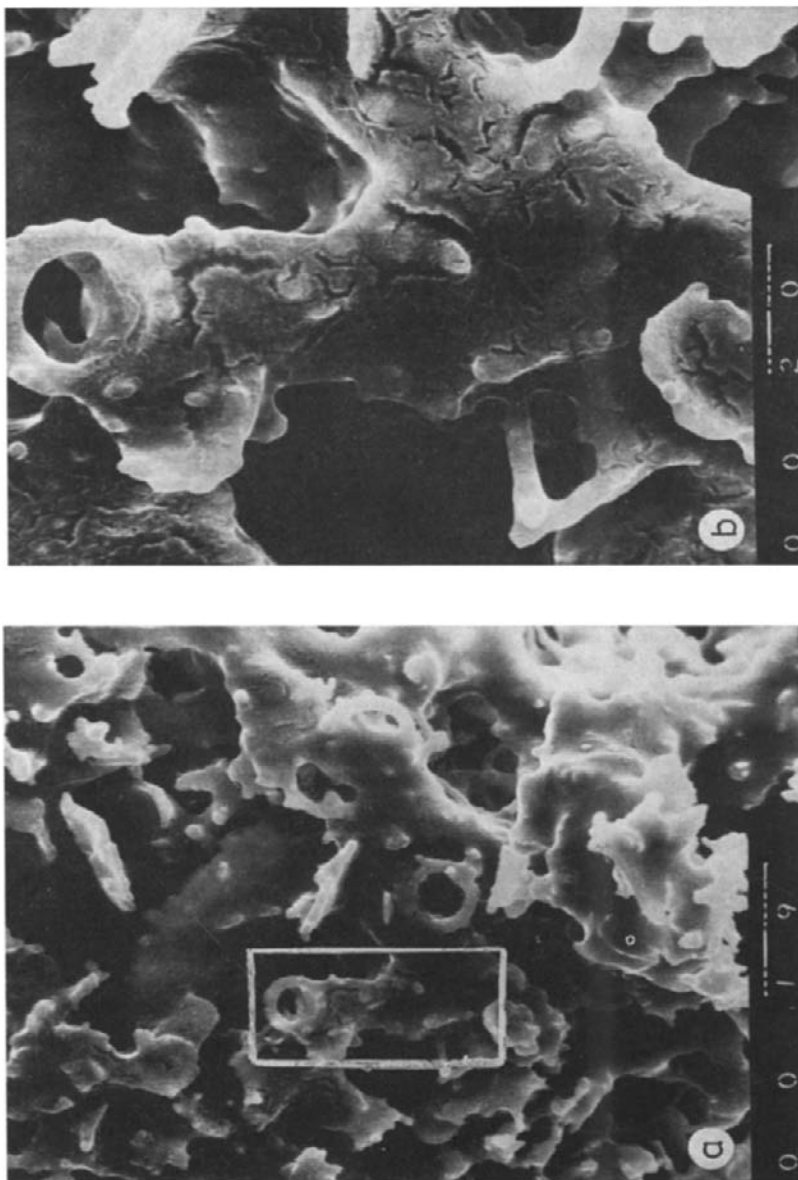


Fig. 1. Microphotographs of the sorbent Chromaton N AW + 16% (w/w) PEG-400: a, outer surface area,  $\times 1000$ ; b, fragment,  $\times 3000$ .

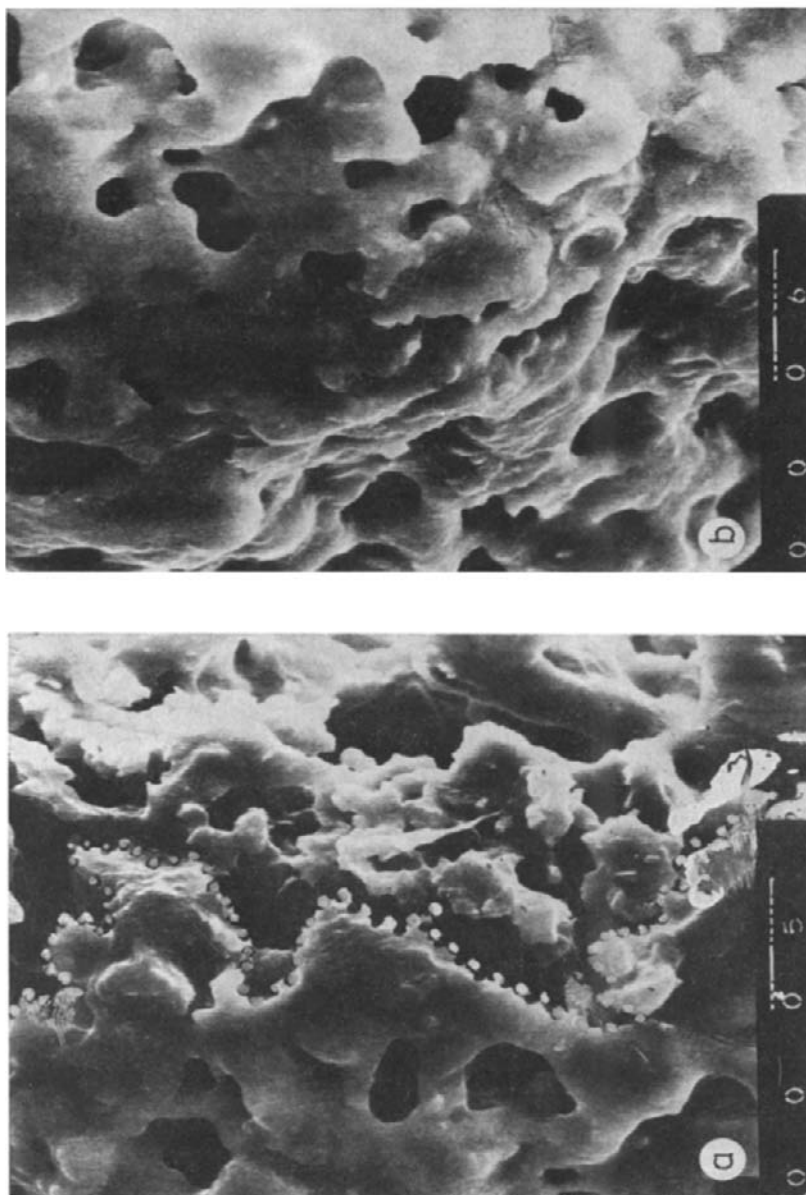


Fig. 2. Microphotographs of the sorbent Chromaton N AW DMCS + 16% PI:G-400, Details as in Fig. 1.

the main SLP content is a capillary liquid, filling the pores and hollows of the grain, *i.e.*, at the support surface a continuous thin film is formed. When the surface of the support is not readily wettable then the SLP is present in the form of individual drops ("islands")<sup>5</sup>. As far as we are aware the present work is the first direct experimental confirmation of this theory for conventional, unbounded SLPs for the system "polar phase-hydrophobic solid support".

In practice, the SLP is concentrated at the outer support grain surface and this kind of distribution can be considered to be "microdrop-like".

The results obtained by SEM on the SLP distribution pattern agree well with the chromatographic data on efficiency. Thus, when the support surface is not readily wettable and the SLP is distributed as a thick layer, the efficiency of the column is 1.5 times lower than that of a column filled with the sorbent where the SLP forms a thin coating and the support surface is easily wettable by the SLP.

In conclusion, for the first time it is shown that, when the solid support surface is not readily wettable, the conventional, unbounded SLP is non-uniformly distributed along the sorbent grain diameter, being mainly concentrating at the outer grain surface and penetrating inside only to a small extent. When the surface is easily

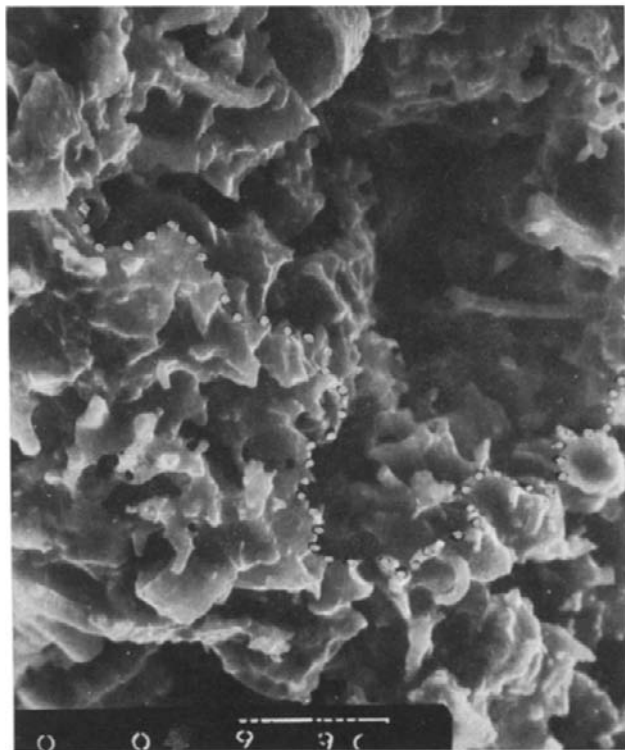


Fig. 3. Microphotograph of the sorbent Chromaton N AW DMCS + 16% PEG-400 and 0.8% stearic acid showing grain cleavage surface,  $\times 1000$ .

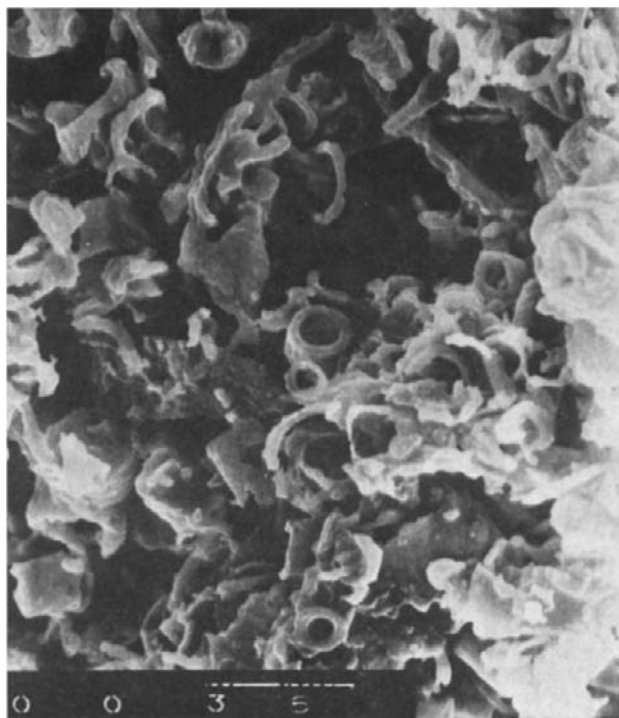


Fig. 4. Microphotograph of the sorbent Chromaton N AW DMCS + 16% squalane showing the inner surface area,  $\times 1000$ .

wettable, the support grains are completely impregnated by the liquid phase. The presence of a detergent causes a change in the SLP distribution pattern on the solid support.

#### REFERENCES

- 1 V. G. Berezkin, V. P. Pahomov and K. I. Sakodinsky, *Solid Support in Gas Chromatography* (in Russian), Khimija, Moscow, 1975, p. 200.
- 2 A. A. Zhukhovitskii, M. L. Sazonov, M. Kh. Lunskii and V. Yusfin, *J. Chromatogr.*, 58 (1971) 87.
- 3 W. A. Aue, C. R. Hastings, J. M. Augl, M. K. Norr and J. V. Larsen, *J. Chromatogr.*, 56 (1971) 295.
- 4 J. C. Giddings, *Anal. Chem.*, 34 (1962) 458.
- 5 V. G. Berezkin and V. M. Fateeva, *Zh. Anal. Khim.*, 25 (1970) 2023.