

## Notes

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### Improvement of the properties of the Teflon support

The effectiveness of a Teflon support is usually not lower than that of diatomite supports, but its mechanical properties are worse<sup>1</sup>. The properties of the proposed support "Polychrom"<sup>2</sup> made from polytetrafluoroethylene type 4D<sup>3</sup> (surface area 10 m<sup>2</sup>/g, bulk weight 0.57 g/cm<sup>3</sup>) and its effectiveness (height equivalent of the theoretical plate 1.5–2.5 mm) approach those of the Chromosorb T<sup>4</sup>.

We have investigated two methods of improving the mechanical properties of the Teflon support without causing a decrease in the effectiveness of separation. The first method consists in carrying out an additional thermal treatment of the Teflon support; the second in joining the particles of the polytetrafluoroethylene by means of soluble fluorinated copolymers.

The study of the support was made on a chromatographic apparatus with a hot wire detector and U-type stainless steel columns 1.8 m long and 4 mm in inner diameter. The temperature of the column and the detector was maintained at 70°. The space velocity of the helium carrier gas was approximately 30 ml/min.

The thermal treatment of the support was carried out at various temperatures from 280 to 390° and with various heating periods from 5 to 30 min. After the thermal treatment the support readily turned into a powder without additional grinding and was then passed through a sieve. Fig. 1 shows the dependence of the height equivalent of the theoretical plate on the duration of the thermal treatment of the support at 300°. When the thermal treatment period was 10 min, the hardness of the support considerably improved<sup>5</sup>. After treatment, the specific surface area of the support was somewhat decreased while the bulk weight was increased. Apparently it took the first 7 min to heat the polymer through, and after this (from 10 to 15 min) the thermal treatment itself took place. The polytetrafluoroethylene particles were photographed under an electron microscope after various periods of thermal treatment. As may be seen from Fig. 2, the pores of randomly selected particles of polytetrafluoroethylene (size 1–2 microns) are mostly 0.1–0.2 microns in diameter (1000–2000 Å); this agrees well with the pore size determined by other methods. The particles of the Teflon support ( $25 \times 10^5$ – $50 \times 10^5$  Å) apparently consist of a large number of single particles joined together by the action of electrostatic forces and form large aggregates. As a result of the thermal treatment of the support, the single particles join together. In the process the number of pores decreases as Fig. 2 shows, whereupon the surface area of the support decreases while its bulk weight increases. The size of the pores remains practically unchanged (Fig. 2C).

The mechanical strength of the support is also increased when the particles of polytetrafluoroethylene are joined together by impregnating the support with an acetone solution of tetrafluoroethylene–vinylidene fluoride copolymer. The copolymer is deposited on the untreated Teflon powder by a method similar to impregnation of

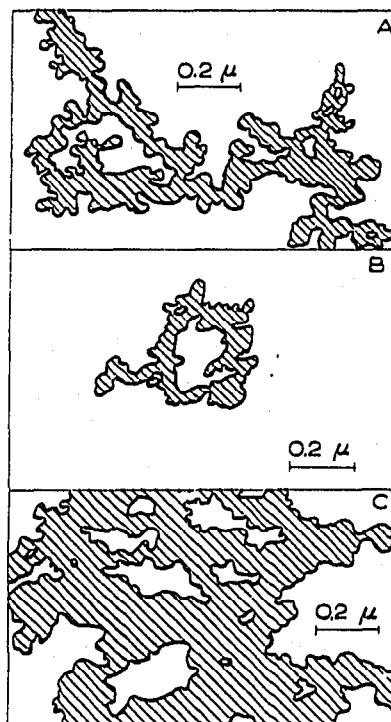
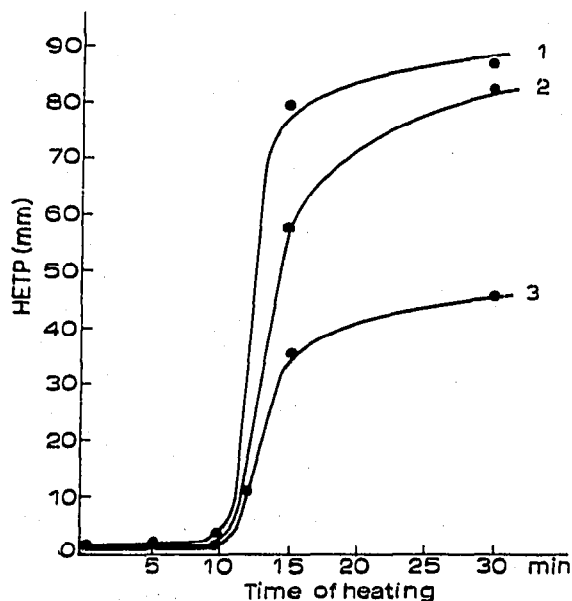


Fig. 1. Dependence of the height equivalent of the theoretical plate on the period of thermal treatment of the Teflon support at 300°. 1 = Benzene; 2 = toluene; 3 = chlorobenzene.

Fig. 2. Electron microscope photographs of Teflon particles of "Polychrom" type support at 300° and after different periods of thermal treatment. Magnification: 100,000 ×. (A) = 8 min; (B) = 10 min; (C) = 15 min.

the support with a stationary phase. The amounts of the deposited copolymer (based on weight % of the support) were 0.1, 0.5, 1, 3 and 5 %. The dependence of the specific surface area of the support on the amount of the copolymer deposited on the support is shown in Fig. 3. In the case of small amounts of copolymer the surface area of the Teflon support decreases more rapidly owing to preferential filling-in of the small pores<sup>6</sup>. The mechanical strength of the support after deposition of up to 1 % of the

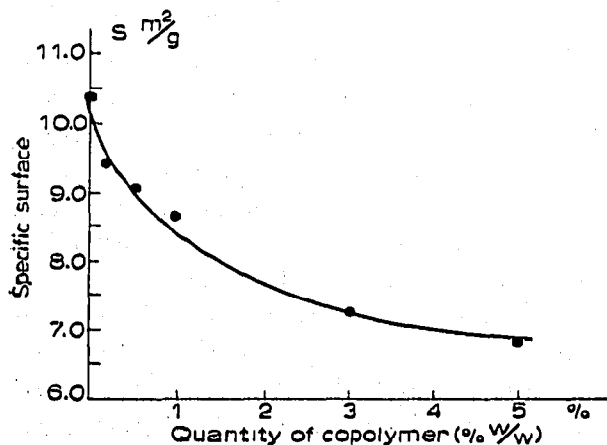


Fig. 3. Dependence of the specific surface area of the Teflon support on the amount of deposited copolymer (based on the weight % of the support).

copolymer remains practically unchanged, but it improves considerably when the amount of the copolymer exceeds 3 %.

Fig. 4 shows the differential dependence of the volume of the pores according to the radii after impregnation with the copolymer in amounts of 1 and 5 %. The total volume of the pores of the support decreases from 0.370 cm<sup>3</sup>/g for 1 % of the copolymer to 0.243 cm<sup>3</sup>/g for 5 % of the copolymer. Most pores of the support are from 500–2000 Å in dimension, the number of small pores (with diameters less than 300 Å) being few. As a result of filling of small pores with the acetone solution of the copolymer and subsequent formation of a copolymer film following the evaporation of the solvent, the single particles of the support are joined together, and their mechanical strength improves. With small amounts of the copolymer, the height equivalent of the theoretical plate first somewhat increases (for example, in the case of propanol, from 1.4 to 2 mm); at a concentration of 1–5 % it remains practically independent of the amount of the copolymer deposited on the support.

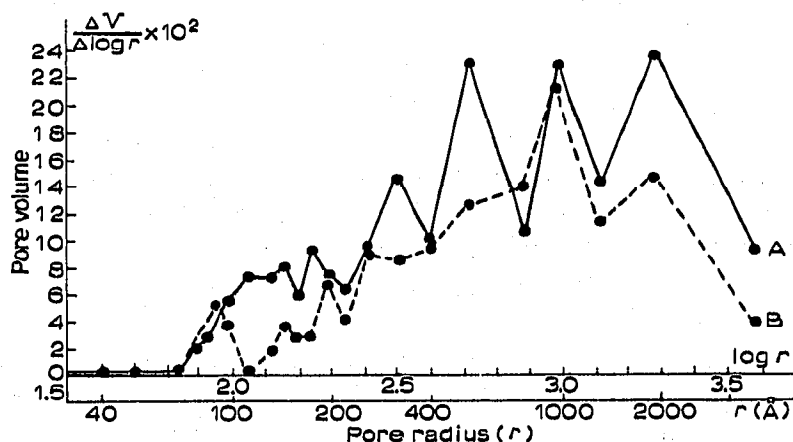


Fig. 4. Differential dependence on the distribution of the volume of the pores according to the radii for Teflon supports with 1 % (A) and 5 % (B) of copolymer.

The method described makes it possible to prepare Teflon supports of sufficiently high chromatographic effectiveness with mechanical strength approaching that of the usual diatomite supports of the Chromosorb W type.

*Karpov Physicochemical Institute,  
Moscow (U.S.S.R.)*

V. BRAZHNIKOV  
L. MOSEVA  
K. SAKODYNSKI

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