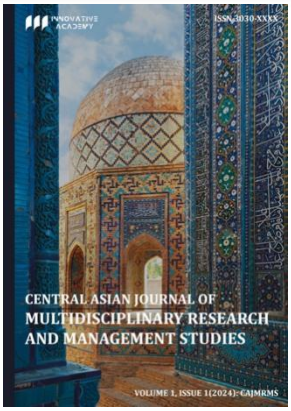

ANALYSIS OF POLYSACCHARIDES BY LIQUID CHROMATOGRAPHY

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ABSTRACT

The chromatographic properties and elution properties of industrial samples of XTZ and Na-KMS are studied by the method of two-detector exclusion liquid chromatography. It is shown that the use of a multi-angle laser beam scattering detector and a refractometric detector provides not only the accuracy of the molecular mass value of the polymer under investigation, but also a qualitative assessment of the degree of intermolecular interaction in aqueous-salt solvents.

In the last decade, exclusion liquid chromatography has become increasingly popular due to the complex use of high-performance sorbents and recording devices for chromatographic analysis, including multi-angle, refractometric and viscometer detectors (1-8).

It is known that the inclusion of a detector with a single detector and the simultaneous use of three detectors in chromatographic analysis, which allows to study such important physicochemical $_{\text{parameters}}$ (parameters) of polymer molecules and the specific viscosity of the polymer in the system polymer-solvent (1,2,4,6,7.), allows to study the absolute expression of the average molecular weights (M_{W,M_N}, M Z) and the inertial radius of macromolecules (R g) of the exclusion liquid chromatography with the help of two detectors. Previously, we (10) described the use of size exclusion liquid chromatography in the determination of cellulose sulfate and Na-KMS with a polymer sorbent in a universal column using PSS suprema (FRG) aqueous saline eluent ·

A notable advantage of the actual work is the use of two detectors in the analysis of Na KMS and XTZ industrial samples, with one detector being a multi-angle scattering of laser light.

The liquid chromatography was performed using a Merck-Hitachi L-6000A plunger pump, Shodex Pl-101 refractometric detector, DAWN HSP laser beam multi-angle scattering detector, Watt Technology (SSHA) Rheodine 2104 probe hand-held injector, eluent depositor and 25^0 PL Aquagel-OHMixed chromatographic tubes connected in series. The inner diameter and length of each tube were 300 and 7.5 mm, respectively. The volume of the test sample was

100 µl, using an aqueous solution of NaCl 0.1 mol/l as the eluent. The volumetric flow rate of the eluent was 60 ml/h.

The polymer solutions are filtered through a 0.22 µm filter before being sent to the chromatographic column. The samples are analyzed using multi-angle laser beam scattering at a wavelength of 632.8 nm.

Na-KMS experiment The refractive index of aqueous 0.1M NaCl solution is 0.147 (9.11). Na KMS samples were supplied for analysis by "Carbonam ¨ 000

Discussing the results of the samples .

It is known that water-soluble polyethers of cellulose, such as Na KMS, exhibit polyelectrolyte properties in aqueous solutions. In size exclusion liquid chromatography, polyelectrolyte effects (V_R) are observed in the form of a concentration dependence of the volume fraction and the asymmetry of the chromatograms, i.e., the chromatographic peak of the Gaussian distribution is avoided. Figure 1 shows the corresponding gel chromatogram of NaKMS obtained at the concentrations found for testing in water of 0.1; 0.5; 2.5; 5 mg/ml and $M_m = 4.8 \cdot 10^4$.

The figure shows the asymmetric profile and multimodal distribution of the gel chromatogram.

It can be seen that the polyelectrolyte swelling effect is present in a given chromatographic system, given that the $V_{R\,=\,decrease}$ in the first peak is due to the decrease in the concentration of the solution.

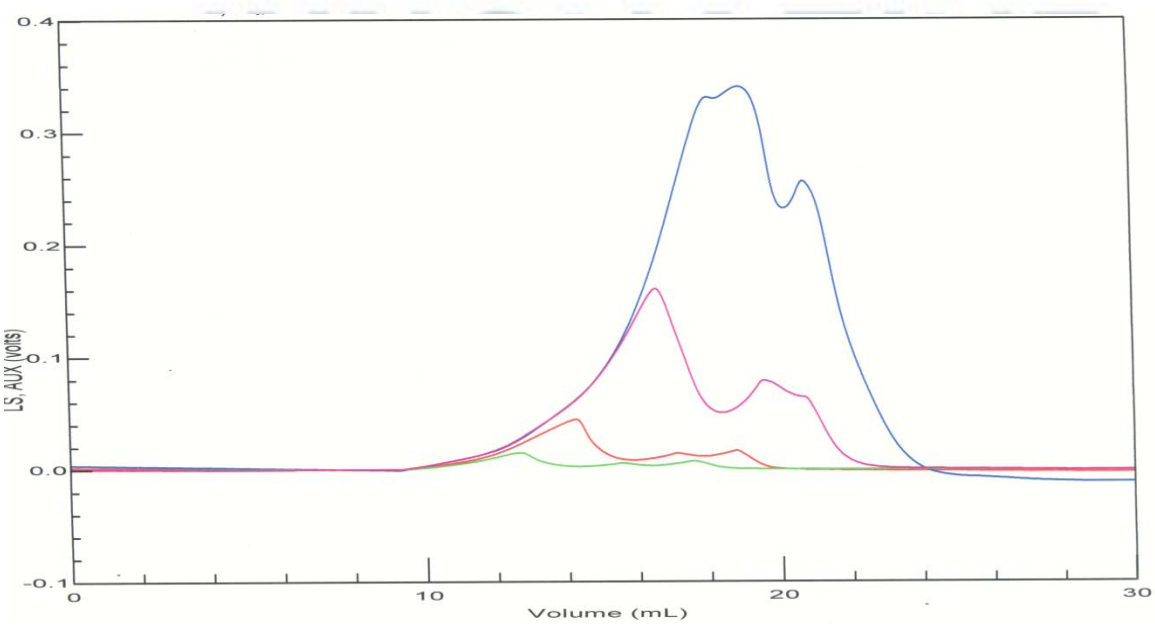


Figure 1: Gel chromatogram of Na-KMS obtained at different concentrations (mixtures) sent for testing:

a) 5; b) 2.5; c) 0.5; d) 0.1

Eluent: H_2O.

PL Aquagel – OH Mixed – two chromatographic speaker system as eluent

The use of a 0.1 mol/l NaCl aqueous solution completely eliminates (quenches) the indicated effect and, as a result, the gel chromatogram of the sample takes on a Gaussian shape. (Figure 2)

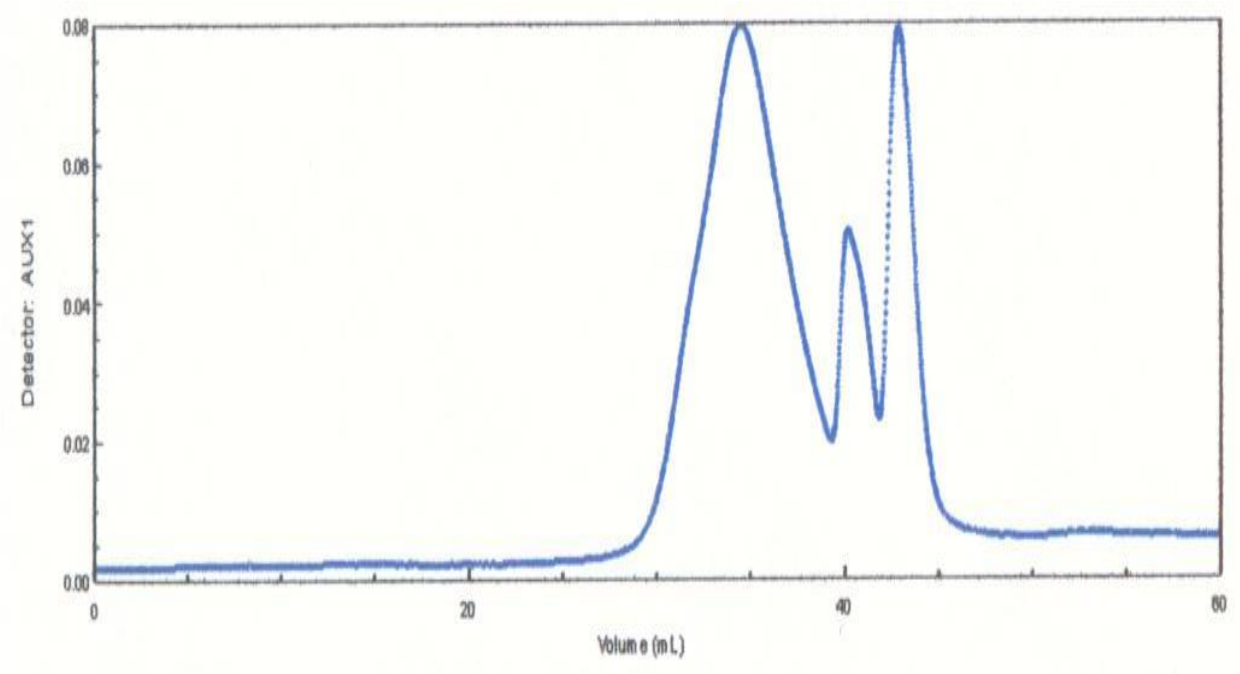


Figure 2. Gel chromatogram of Na KMS obtained in the absence of polyelectrolyte swelling effect: aqueous solution 0.1M NaCl.

In order to determine the effect of the nature of the solvent on the elution behavior of Na-KMS, it is carried out in water with NaN_3, NaCl, $NaNO_3$ as eluent at a concentration of 1 mol/l. As determined in (8), the analysis of NaKMS by exclusion liquid chromatography in 0.1M $NaNO_3$ is complicated by the presence of small amounts of associates formed as a result of intermolecular interactions in the solution being used.

propose to first prepare a solution of Na-KMS in water, and then add the required amount of $NaNO_3$ to the polymer solution.

obtained measurement data in NaKMS exclusion liquid chromatography with $NaNO_3$ and NaN_3 eluates in conjunction with the work .

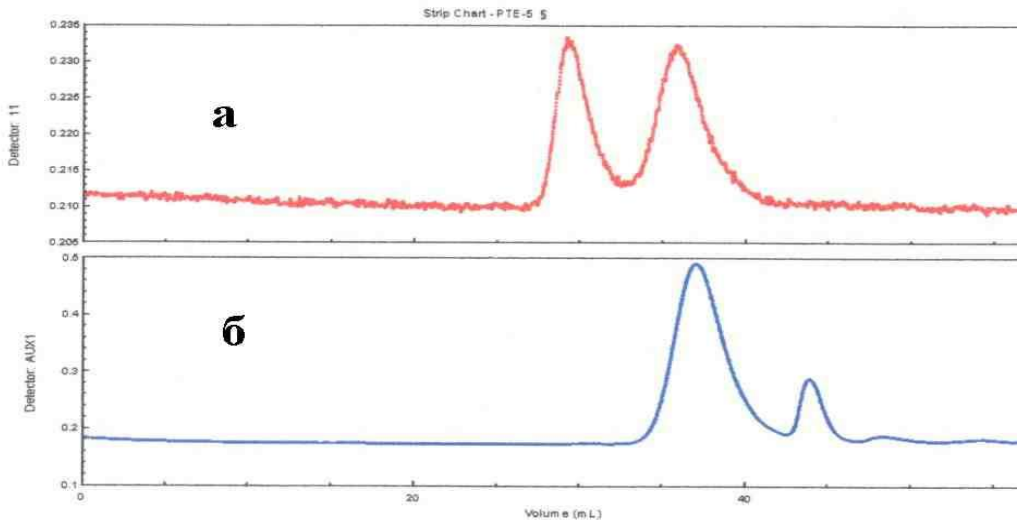
Multi-angle laser beam scattering detector

Figure 3 shows the multi-angle scattering of laser light (a) and the gel chromatogram of Na KMS $M_W = 2.18 \cdot 10^4$ generated using a refractometer.

Multi-angle scattering of laser light was obtained using (a) and a refractometer (b).

Eluent: aqueous solution 0.1M $NaNO_3$.

As can be seen from the figure, the multi-angle scattering detector of the laser beam registers 2 chromatographic peaks.



(Fig. 3 a) The first of them is separated from the main peak, which indicates the presence of a small amount of Na KMS associates in the solution. The multi-angle scattering detector of laser light is highly sensitive to large macromolecules in the solution and the product

The refractometer registers a small amount of associates, while the refractometer is not sensitive to such macromolecular interactions. Similar results were obtained with 0.1M $NaNO_3$. However, when 0.1M NaCl was used as the eluent of the aqueous solution, the first peak disappeared (Figure 4).

We can conclude that this is the path of intermolecular associations in a given eluent.

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