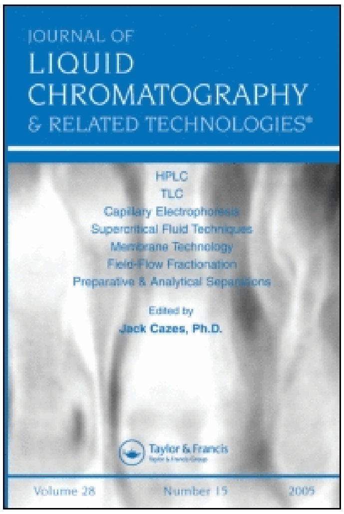

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Polymer Analysis by Size Exclusion Chrokatography

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POLYMER ANALYSIS BY SIZE EXCLUSION CHROMATOGRAPHY

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ABSTRACT

This paper is the review dealing with the applications of size exclusion chromatography to complex characterization of synthetic polymers. It treats, in the first place, the problems associated with the selection of the optimum conditions for the fractionation of various groups of polymers, further the questions of the calibration of separation systems, possibilities of determining structure of polymers by using size exclusion chromatography, and, finally, the most important applications of this separation method to the study of polymerization and degradation of polymers.

INTRODUCTION

The present paper is aimed at showing possibilities of using size exclusion chromatography (SEC) for the analysis of synthetic polymers and at reviewing the most typical applications described in literature. It will be limited to polymers prepared by polymerization or polycondensation of low-molecular weight organic compounds, mostly soluble in organic solvents only. For instance, cellulose and its derivatives, synthetic polypeptides, oligomers and thermoreactive resins will not be treated. Oligomers will likewise not be discussed in particular unless the demonstration of the applications of SEC to the analysis of polymers is concerned. Not even SEC analysis of low-molecular weight compounds which, serving as additives with various functions, are components of polymeric systems will not be a subject of the present review. These restrictions are only introduced for the present purpose since such separation systems may exist in which the separations may be performed over the range of the molecular weights from monomers up to high-molecular weight polymers. In practice however, analysis of low-molecular weight components and oligomers is performed separately, often even by liquid chromatography of a different type. The review will neither include inorganic polymers, such as polyphosphates, however, it will deal with polysiloxanes. The outlined scope is undoubtedly rather casual because a strict distinction between organic and inorganic, synthetic and natural polymers, oligomers and polymers does not objectively exist. In the cases belonging to transient areas between various above mentioned categories, the decisive criterion will be represented by the expediency of the classification into the respective category from the viewpoint of the main topic of the present paper. From the point of view of SEC, the papers will be included in which SEC application is of fundamental importance. The papers will mostly not be cited in which the results of SEC are of minor significance. Some of the papers present more or less limited survey of applications (1-4). A book by a collective of authors (5) belongs to greater recent works.

The present review will treat individual polymers according to the classification common in macromolecular chemistry (6).

SEC is used in the first place to determine the molecular weight distribution (MWD) of polymers. However, also some other potentialities will be pointed out which are provided by SEC in complex analysis of polymers. In order that the above facts might aid the users of SEC to orientate themselves rapidly in solving some concrete problems, general experimental conditions of SEC for the determination of given polymers, calibration methods, investigated for and applied to respective polymers, study of the possibilities of determining branching or, more generally, structure of polymers, investigation of the relationship between the MWD or structure of the polymer and its physicochemical properties, study of the course, i.e., mechanism and kinetics, of polymerization of the studied system by using SEC, study of the degradation of polymeric systems using SEC, comparison of SEC with other methods for fractionating and determining molecular parameters of polymers, applications of SEC to preparative fractionation of polymers and some other possibilities of using SEC for the analysis of polymers manufactured industrially will be discussed.

Theoretical principles of these methods, which can be found in other papers (7-10), will not be discussed here. There are some other papers dealing with certain general possibilities and principles of the applications of SEC to the study of polymerization (11,12), degradation (13-15) and morphology of polymers (16) or pore size distribution in porous materials (17) and particle size distribution (18).

POLYALKENES

Solubility of polyalkenes - polyethylene (PE), polypropylene (PP), polyisobutylene (PIB) and others - for the most part at increased temperatures only, dictates the experimental arrangement and the conditions of SEC, including the instrumentation (19-25). 2,4,6-Trichlorobenzene (TCB) is mostly used as a solvent at 125°-150°C, less usual is o-dichlorobenzene (DCB) and perchloroethylene (24) was applied at 110°C quite exceptionally, p-xylene at 115°C (26), tetrachloromethane (TCM) for PIB (27) and tetrahydrofuran (THF) for low-molecular weight PIB (28,29).

From the viewpoint of the packing of the separation columns, problems associated with thermal stability of cross-linked polymeric gels at the analysis of PE (30) and some problems of adsorption of PIB and PE (26,27) on inorganic supports were solved and the possibilities of deactivating surfaces of these supports were searched. Narrow fractions or unfractionated samples of PE (24,31-37), PP (38-40) or PIB (41,42) are usually used to calibrate separation SEC systems. A principle of universal calibration was tested and confirmed for samples of both linear and branched PE (34,43-59), PP (45,46,60-62) and PIB (47,63) (in the last instance at temperatures of 25° and 150°C). The constants of Mark-Houwink equation, required for the interpretation of experimental data by using the universal calibration curve, were determined for PE in TCB at 135°C in a comparative work (64). Q factor (21,65) was used in addition to the above procedures for the calibration and the interpretation of SEC data.

The universal calibration combined with other methods for determining molecular parameters, such as viscometry, light-scattering and osmometry was applied to study the branching of PE (35,44,49,52-57,66-80). The measurement of the rate of sedimentation (81) was similarly utilized for the correlation. In addition to long-chain branching, which affects the hydrodynamic volume of macromolecules in solution, the content of short chains of fractionated PE (53) was determined from the total content of methyl end-groups by combining SEC with infrared spectrometry (IR). Variable contents of long chains even in relatively narrow fractions of PE can obviously lead to erroneous SEC results (82) if they are neglected unjustifiably.

SEC method was used to study the relationships between MWD and some physical properties of PE (32,83) and PP (84), to investigate PE fractionation in the course of its crystallization from solutions (85) and to observe statistical cross-linking of PE (86). MWD was correlated with thermodynamic properties of PE (87), shear stability of PIB used as an oil additive (88,89) and with rheological properties of PE melts (90) and PIB solutions (91). SEC was further used to study polymerization of PE initiated by γ-irradiation (92). Interesting results were obtained by studying the course of the degradation by nitric acid in the case of both PE (36,93-100) and PP (94).

In this respect the SEC results also contributed to the elucidation of the structure of the initial PE (96) and its morphology (98). The course of the degradation of PIB was also studied at laminar flow (101) and under the action of ultrasound (102). The variations in MWD were further studied at rapid overheating of PE (103), at thermal degradation of PE in the atmosphere of nitrogen containing a small amount of oxygen (51) and at radiolysis of PE at which occurs its cross-linking (86,104). The accuracy of SEC results was verified by comparing with the results obtained by other methods, such as column fractionation using elution gradient in the case of PE (33,49, 57,75,82), PP (38-40) and PIB (41,42,105), light-scattering, osmometry and aventually also viscometry for PE (57,75,106,107) and PP (40,65) and sedimentation for PE (81). The accuracy or the reproducibility of SEC was determined for PE in the range of approximately ±10-15 % rel. (108,109) with using for the determination in some instances samples recognized as international standards (110-112). Preparative SEC was employed to fractionate samples of PP and PE (113-115). In a number of other cases, SEC was used to study MWD of samples of, for the most part commercial, PE (21,22,26,54,79,87,106,116-122), PP (21, 84,123) and PIB (124). The determination of MWD was also described for some other polyalkenes, such as poly-1-butene (21), poly-1-pentene (121) and poly-1-octadecene (21). The compositions of multicomponent mixtures of polymers of which one is PIB (28,29) was determined using SEC as well as the distribution of double bonds in PIB by means of the specific detection (125).

POLYDIENES

Polybutadiene (PBD), polyisoprene (PI) and their copolymers have mostly been determined by means of SEC. THF or trichloromethane is used as a solvent for SEC analyses most frequently, DCB (126) is less usual, always at room temperature. The formation of gel-like microparticles in the solution means an experimental complication and that is why the solutions are, prior to the injection, filtered through metallic membrane filters (127). Narrow fractions obtained, for instance, by preparative SEC fractionation are used for calibration in the case of PBD (128). A universal calibration curve was used to evaluate MWD od samples of PI (129-131) and PBD (28,29,131-136).

Correlated with the results of other absolute methods for determining molecular parameters, SEC results were used to study branching of PBD (131,137). The determinations of MWD and of the distribution of terminal carboxyl and hydroxyl end-groups of PBD (128, 138-146) are exceptional applications of SEC. Here the SEC separation is, however, complicated by adsorption of PBD containing carboxyl or hydroxyl end-groups on the column packing (143,144). This adsorption usually is rather irreproducible (146). As long as the carboxyl groups are esterified, the adsorption is suppressed (138,139,146). In any case, the effective hydrodynamic volume of the chains containing functional groups differ, however, from that of the chains without functional groups and this fact must be taken into consideration on interpreting experimental data (143).

In order to determine the distribution of functional groups, dual detection (142) may be used, e.g., by combining SEC with IR spectrometry (128) or UV (ultraviolet) detection (138,139). PBD with functional groups usually are prepolymers with very low molecular weights in the range of oligomers (140-142,145,147-149). A specific detector was described, making it possible to determine the distribution of unsaturation of PBD (125). Valuev et al. (150) described the determination of MWD of stereoregular PI. SEC was used to study cyclization of PI (151), to investigate changes in

MWD for PBD that are induced by alkyllithium complexes with alkyl halides (152).

SEC results were correlated with the results of other fractionation methods (126,128,143,144,146, 153-155) or those of the methods for determining absolute molecular parameters, such as osmometry (128,132, 153) and light-scattering (153). Some other papers were devoted to optimalization of experimental conditions of SEC analysis of PBD (19,156-159), including effects of various solvents and temperature (20), to the study of process polymerization of PBD (160) or PI (160,161) and to the determination of MWD of PBD in a mixture with PIB (28,29) or MWD of PBD elastomer in a mixture with oil (162,163). One paper concerned the testing of reliability of SEC for the determination of MWD in samples of PBD (164) and one preparative fractionation of PBD (114).

Polychloropropene has been studied up to now from the viewpoint of utilization of universal calibration and determination of branching (165), in process control (166) and in the investigation of the concentration effect depending on the quality of the solvent used (167).

POLYACRYLIC ACID, SUBSTITUTED POLYACRYLIC ACID AND THEIR DERIVATIVES

Polyacrylic acid (PAA), substituted polyacrylic acid and their derivatives have been, despite their considerable industrial and commercial importance studied by using SEC by only a few authors. Several works were performed to define general experimental conditions of SEC for polyacrylonitrile (PAN) (168, 169), polymethyl methacrylate (PMMA) (170) and other acrylates (171). THF or chloroform (129) is used as a solvent for PMMA most frequently, dimethylformamide (DMF) for PAN or less frequently dimethyl sulphoxide (169). Experimental complications have been met in the case of the analysis of polyalkyl thiomethacrylates (172) since a reaction probably occurred between the separated polymer and the column packing, and the polymer also adsorbed on the hydrophilized surface of the refractometer cell, which made the use of SEC very difficult. Validity of universal calibration was confirmed for PMMA (129,173-175) and poly(2-methoxyethyl methacrylate)(PMEMA) (173,176).

A low-angle laser light-scattering has recently been applied to monitor directly molecular weights of PMMA in the eluate leaving the SEC separation system (177). PAN samples (178) were used to carry out a comparative study on different calibration methods, the influence was studied of different solvents on the calibration curve for PAN (179), and the effect of the addition of an electrolyte into the eluent on variations of the calibration curve, caused by the change in the expansion of ionogenic PAN owing to internal electrostatic forces (180). The possibility of determining the distribution of functional groups by means of the IR detector was demonstrated for PMMA (181).

The paper concerning the effect of solvent polarity on the separation of PAA in DMF (182) and the paper studying polymer-polymer-solvent interactions during the separation of PMMA (183,184) are of fundamental significance for the elucidation of some interactions occurring in SEC of polymers. The relationship between MWD and shear stability of polyalkyl methacrylate used as an oil additive (88,89) and temperature resistance of PMMA as a component of lackers (185) and films (186) were studied. The possibilities were described of studying mechanism and kinetics of polymerization of PMMA by means of SEC (187-189). Using SEC, the variations in the MWD caused by degradation effects of UV light on polyalkylacrylates and polyalkyl methacrylates (190) and thermal degradation of PMMA were studied. The results of SEC were compared with those of other fractionation methods for PAN (169). Samples of polybutyl methacrylate (193) and PMMA (114) were fractionated with the aid of preparative SEC. The feasibility of SEC application to the determination of the particle size distribution in aqueous dispersions of PMMA (194) is undoubtedly very interesting.

POLYVINYL HALIDES, POLYVINYL ESTERS, POLYVINYL ALCOHOL

SEC was exploited in some instances for the determination of average molecular weights and MWD of polyvinyl chloride (PVC), polyvinyl acetate (PVAc) and polyvinyl alcohol (PVA). Fractionated samples of PVC were taken for the calibration of SEC separation systems and, on the basis of the calibration curves thus obtained, average molecular weights of unfractionated polymers (195,196) were then calculated. On interpreting SEC data of PVC samples, the principle of universal calibration (133,174,175,178,197-208) was also applied, in some instances by combining SEC with an automatic through-flow viscometer (209,210). Miscellaneous calibration methods have recently been compared (178,211). Universal calibration was employed to determine the degree of PVC branching (202,204, 205). Using this procedure, the number of long chains could be estimated. The total degree of branching was determined by reducing PVC to polyethylene and measuring absorption in IR spectrum (202,204) at 1378 cm^{-1}. Several authors (197,202,204,205,208,212,213) studied some anomalies in properties of PVC in solution. The presence of aggregates in THF was observed in such a degree that they appeared in SEC chromatogram as bimodal distribution (a small separate peak in the high-molecular weight range (204). On heating the solutions prior to the separation by SEC, the aggregates were disturbed and bimodality disappeared. These experiments were later supplemented by measurements of light-scattering and an attempt at quantitative interpretation (205) of which the conclusion was drawn that the aggregates in THF solutions under study can be characterized as conglomerates in which one crystallinic centre contains 10-14 different macromolecules. It was further found (202) that at lower concentrations of injected samples and on heating at lower temperatures, the above anomalies did not occur. Andersson et al. (197) reported that the deviations from the linear dependence of the intrinsic viscosity on the molecular weight were caused, in their case, by aggregation and not by branching. The presence of aggregates need not always appear as chromatogram bimodality, the values of molecular weights calculated from SEC data (208) can only be increased. The aggregates can also be eliminated by ultrasound action combined with surface active substances (208).

A valuable contribution to applications of SEC to the study of PVC properties was made in the paper (212) suggesting determination of tacticity of PVC by SEC method. Formation of molecular aggregates of PVC is caused by stereoregular sequences the distribution of which even affects the degree of the degradation. When selecting optimum experimental conditions, the ratio of aggregates (204,205) and the percentage of syndiotacticity can be evaluated quantitatively. Abdel-Alim and Hamielec (212) and Sörvik (214) found a very good quantitative agreement between the results of the determination of the content of syndiotactic structures by nuclear magnetic resonance (NMR) method and by SEC. SEC was utilized to study PVC adsorption on $CaCO_3$ (215-217) and kinetics of mechanico-chemical degradation of PVC and the variations in MWD occurring at this degradation (218).

Comparison of miscellaneous methods for the determination of molecular weights and MWD with SEC provided a satisfactory agreement of the experimental results (197,199,204-206,208,217-221). SEC method was compared with light-scattering (204-208,211,220,221), osmometry (206-208,211,220,221), viscometry (199,211) and fractional precipitation (197,219). SEC method finally found the application at determining MWD of commercial PVC samples (208,221-223), different additives (221), and on preparative scale at fractionation (114).

MWD of PVAc samples has been investigated with the aid of SEC in several instances; applicability of universal calibration was tested and the degree of branching determined (137,224,225), various calibration methods were correlated (178) and low-molecular weight samples (226) and particles (227) were characterized.

The only important paper has been published that concerned SEC fractionation of PVA samples soluble in water (228). In consideration of appreciable PVA adsorption on unmodified silica gel used as column packing, deactivated silica gel had to be used.

POLYSTYRENE AND DERIVATIVES

The majority of papers dealing with SEC of polymers on both theoretical and applied lines are based on polystyrene (PS) standards that are commercially available. On using non-polar solvents, such as TCM, adsorption occurred on polar, for the most part inorganic gels. This is also the reason why the properties of different column packings and eventually effects of their surface modifications on a suppression of PS adsorption (26,27,120,229-232) were investigated. Coupling of an automatic through-flow viscometer (137,209,210,233) means a contribution in view of the calibration of the separation systems. Branching of different type was studied with PS (137, 233-237) samples along with morphology of PS samples (238,239) and kinetics and mechanism of styrene polymerization, initiated by various systems (187-189, 234,235,240-246).

PS degradation is treated in the paper aimed at a special type of modelled atmospheric ageing of PS (247). Further papers concern the study of PS degradation owing to ultrasound action (248,249) and benzoyl peroxide (249). Particular view is here adopted by the paper (250) describing shear degradation of PS standards with extremely high molecular weights under the conditions of the separation by SEC. Comparisons of SEC in column with thin-layer chromatography (251) and with column fractionation (144) should be cited. Interesting conclusions were drawn in the papers (252,253) dealing with exact evaluation of MWD of PS standards NBS 705 and 706. Further papers studied MWD and particle size distribution in PS emulsions (194,254), separation of radioactive and inactive

PS samples by SEC method (255,256) and SEC applications to the samples from industrial production (257).

Poly-α-methylstyrene (PMS) samples were investigated by SEC method in view of the applicability of universal calibration (61,62), determination of branching (258), investigation of the course of the polymerization (259-262), comparison of miscellaneous methods for the determination of MWD (263), and specification of the optimum experimental conditions for high resolution SEC (264).

OTHER VINYL POLYMERS

Of other polymers of vinyl type it is polyvinylpyridine (PVP) (265-267) that was particularly studied by SEC method. When DMF was used as a solvent, PVP adsorption occurred on silica gel column packings and PVP deposition from the solution occurred on styrene-divinylbenzene gels, probably owing to PVP incompatibility with the column packing (265,266). SEC was applied to study the course of PVP polymerization (267). It was further utilized in order to characterize MWD of some vinylaromatic polymers (poly-1-vinylnaphthalene, poly-2-vinylnaphthalene and poly-4-vinylbiphenyl) (268), to study a formation of polyvinylcarborane induced by γ-irradiation (269) and polymerization of vinyl ketones (270).

COPOLYMERS

The determination of MWD of copolymers by SEC is complicated by the fact that besides polydispersity of molecular weights occurs the possibility of the distribution of the copolymer composition. In many cases conclusions of the experimental studies are only qualitative, limited to publication and brief description of the chromatograms obtained, without any quantitative evaluation (158,271-276). Major part of experimental works deal with block copolymers styrene-butadiene or styrene-isoprene (89,127,130,131,145,149, 155,162,163,271,274,276-293), styrene-methyl methacrylate (181,294), α-methyl-styrene-butadiene (295-298), styrene-maleic anhydride (299), styrene-vinyl stearate (300), styrene-vinyl chloride (301), α-methylstyrene-methacrylonitrile (158) and terpolymers butadiene-acrylic acid-acrylonitrile (302,303), acrylonitrile-butadiene-styrene (179) and vinyl acetate-styrene-acrylonitrile (178). In all the cases mentioned above, a differential refractometer-UV detector dual detection can be applied to both evaluation of MWD and distribution of the composition. SEC method was further used to analyze ethylene-propylene (45,46,94,125,304,305), ethylene-butene (59,94), vinyl chloride-vinylidene chloride (306), vinyl chloride-vinyl acetate (307-313), glycol methacrylate-glycol dimethacrylate (314), PMMA grafted to cellulose (315), styrene-divinylbenzene (316,317), ethylene-vinyl acetate (67,318,319), ethylene-isobutyl acrylate (318,319), butyl methacrylate-methyl methacrylate (307), THF-propylene oxide (272), THF-epoxides (275), styrene-acrylonitrile (320-322) and some others (136,180,323-328). In several cases the IR detector (181,271,273,313,320) was used for the specific detection. General scheme of the procedure for the determination of MWD and distribution of the composition of copolymers was proposed by Owens and Cobler (329). Two works (330,331) summarize fundamental theoretical principles of the determination of MWD of copolymers using different methods, including SEC. A few papers (278-281) presented theoretical analysis of the interpretation of SEC data of block copolymers. According to these papers it is possible to evaluate by means of a linear interpolation between parallel and non-parallel calibration curves of the corresponding homopolymers MWD of di-, at the maximum triblock copolymers. Runyon et al. (288) employed this method experimentally and evaluated point-by-point the simultaneous chromatogram of the differential refractometer and the UV detector for styrene-butadiene copolymer. Even in the case of copolymers, a number of papers have been devoted to the problems associated with the universal calibration (45,46,59,130, 131,136,155,178-180,203,282,286,291-294,299,308-313,316, 317,325,326). Influence of the addition of an electrolyte on the universal calibration curve of copolymers that represent polyelectrolytes was studied in several works (180,323,326). Evaluation of MWD of copolymers was made possible by using an automatic through-flow viscometer coupled with a chromatograph (130,291). Universal calibration was preferred experimentally to the linear interpolation between calibration curves of particular homopolymers (155,286). A discontinuous procedure for the evaluation of SEC data (181,276,294,300,332) has also been applied. Composition of copolymers was determined by IR spectrometry in separated fractions. Several papers were concerned with the study of branching of various copolymers (67,130,131,283,294,301, 316,317). Even in the case of copolymers several works were devoted to the determination of the distribution of different functional and terminal groups (145,181) by using an IR detector and a specific detector based on the reaction of ozone with double bonds (125). The course of copolymerization of different comonomeric pairs was studied under various conditions (275,294, 295,301,314,325,333). Degradation of styrene-butadiene copolymer (285) was studied along with structure of olefin copolymers with the aid of degradation by fumic nitric acid and MWD of the degradation products was determined using SEC method (94). The results of SEC were compared with those of thin-layer chromatography for styrene-butadiene copolymer (155) and viscometry for vinyl chloride-vinylidene chloride copolymer (306). Preparative SEC was also exploited for fractionation of copolymers (289,300,309,310,326,334-336). A necessity of filtering styrene-butadiene copolymers prior to SEC separation in order to prevent column pluging (127) and a necessity of correcting chromatograms of copolymers for variations in increments of refractive indices caused by variations in chemical composition (149) is an important knowledge from the viewpoint of experimental work. Content of gel was determined in irradiated copolymers (318,319). Peculiar applications of SEC were described for copolymers important for lacquer´s purposes (307,337,338), for elastomers with oil additives (162,163) and for liquid rubbers (302,303).

POLYALKYLENE AND POLYARYLENE OXIDES

Several fundamental works described principles for the selection of the experimental conditions suited for SEC separations of polyalkylene and polyphenylene oxides (339-348) of different type, covering a wide range of the samples under investigation from high molecular weights down to oligomers. Applicability of universal calibration was tested successfully in two works (349,350).

Properties of polyalkylene oxides (351) in solution were studied in detail along with the influence of the interactions of polyalkylene oxides both with solvent and gel on SEC separation (182,339,352). With the aid of SEC the course of polymerization (353) and degradation (342) was studied. The comparison was described of SEC and thin-layer chromatography (354) in order to determine also the distribution of functional groups of polyalkylene oxides. Additionally the SEC results were compared with the results of light-scattering and viscometry (348,350) and with those of dialysis (355). SEC was also used for preparative fractionation of polyalkylene oxides (193,356).

POLYESTERS, POLYCARBONATES

This section will treat SEC applications to the analysis of polyethylene terephthalate (PET) (174, 357-360), polytetramethylene terephthalate (361), polycarbonates (20,362-368) and various other polyesters (174,369-376). In view of the selection of the optimum experimental conditions for SEC, of particular interest is the work (359) dealing with the selection of suitable solvent for the separation of PET since its degradation was observed (359) in m-cresol used at higher temperatures. Mixed solvents, e.g., tetrachloromethane with 0.5 % of nitrobenzene (359), which are usable at room temperature, appeared advantageous since PET degradation does not occur.

Of different methods, calibrations with various polyester (376) and polycarbonate (366) fractions, calibrations using Q factor (370) and even universal calibration applied to PET and polyesters (174) and polycarbonates (362,364) were described. SEC was also used to investigate polymerization of polyesters or polycarbonates (368,371). PET photodegradation (357) was also studied. The results of SEC were compared with those of light-scattering, viscometry and osmometry (174,366,370). Preparative SEC fractionation of polycarbonates and PET (357,365) was reported. Furthermore, some polyesters (373) of practical importance were analyzed.

POLYAMIDES

Utilization of SEC for the study of polyamides is complicated by the problems associated with their solubility. The following solvents were tested for SEC of polyamides: o-chlorophenol (377) at 100°C, however, adsorption of low-molecular weight components on styrene-divinylbenzene gels destorting final values of the MWD was observed; hexamethylphosphorotriamide (378); m-cresol (379,380) at 120°-130°C, however, under these conditions degradation of polyamides during SEC (381, 382) can occur; m-cresol/chlorobenzene (381,382), a mixed solvent for which the optimum conditions were found to be 1:1 ratio and temperature of 43°C; m-cresol/chloroform mixed solvent at room temperature; 96 % sulphuric acid (columns packed with silica gel) for aromatic polyamides (383). Validity of universal calibration was verified experimentally under various conditions (379,381,382). It was even confirmed for the cited system of aromatic polyamides in 96 % sulphuric acid and PS in THF in the same separation system (383). The principle of universal calibration was applied in order to determine branching of polyamide Nylon66 (379). In this paper, good results were also obtained even by calibrating by means of Q factor. Polymerization of polyamide (380-382) was investigated and commercially manufactured polyamides (384-386) were analyzed with the aid of SEC. Interesting results were obtained on studying polyamides which have character of polyelectrolytes (387,388) at various concentrations and ionic strengths.

POLYURETHANES

In one experimental work (389) the possibilities are shown of applying SEC to the analysis of polyurethanes. Universal calibration was verified for polyurethanes by Vakhtina et al. (390). The relationships between MWD and distribution of functional groups of polyurethanes were studied using SEC (391). Other works were aimed at the applications of SEC as a control analytical method for preparing polyurethanes (392) and for analyzing initial components for the preparation of polyurethanes (393).

POLYSILOXANES

Polydimethylsiloxanes, polydipropylsiloxanes, polydiphenylsiloxanes and some others were studied. In these cases a scale of solvents for SEC separations is fairly wide; THF, trichloroethylene, toluene, benzene and chloroform were applied. Early published works were of qualitative character only, defining fundamental possibilities of using SEC (394, 395). Validity of universal calibration was not confirmed in one case using THF as a solvent (396). In other cases, the results were positive (129,397-401) even under the conditions applying thermodynamically poor solvent (402-406).

SEC was used to study different procedures for preparing polyphenylsiloxane (407,408) and anionic copolymerization of organic cyclic siloxanes (409). Chemical composition of copolymer fraction after SEC separation was studied using NMR. Variations in MWD of polysiloxane solutions were studied using degradation action of ultrasound (410,411) with an IR detector. The results of SEC were correlated with those of other methods for determining molecular weights and MWD, such as precipitation fractionation and gas chromatography (412), reversed phase chromatography (413), ultracentrifugation (398), viscometry and light-scattering (396). Two papers on preparative SEC fractionation (114,414) were published and another one on the study of polymer-polymer-solvent interactions (183).

POLYSULPHONES

Several works (144,172,364,415-417) have studied polysulphones with the aid of SEC method. One of the first papers (417) specified general conditions for the application of THF (at a temperature of 40°C) as a solvent, the SEC results were compared with light-scattering and osmometry. Universal calibration was tested and its validity confirmed (364). A comparison of column fractionation with SEC was also studied for polysulphone samples (144). Experimental complications (172) were met and explained by polysulphone reaction with column packing. Later work by other authors (415,416) negates these complications as well as their explanation.

OTHER POLYMERS

Some works investigated course of polymerization and MWD of polyphenylacetylene (418-420) and MWD of polyfurfuryl alcohol (421-424) which is used for preparing defined graphite. Particular papers described SEC applications to the studies of conformations of poly-(α-phenylethylisocyanide) in solution (425), branching of polymethylindenes (426), preparation of cyclocopolymers of maleic anhydride (427), MWD of polybromotrifluoroethylene (428) and some fluoroalkylene ether polymers (429).

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