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STUDIES OF POLYMER SOLUTIONS CARRIED OUT IN POLAND

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The field of studies on polymer solutions is very extensive and covers a wide range of varied problems. Such studies may be carried out with various aims in view — to define the properties of the solutions, or to characterise the macromolecules themselves from the properties of the solutions. Because of the variety of problems raised it is not possible to cover completely all work on the study of polymer solutions.

In order that the material presented shall be treated as fully as possible we shall consider only two questions, namely the determination of molecular weight and the study of polydispersity. We shall also examine some interesting papers on the hydrodynamics of solutions, and certain others.

In Polish literature on molecular weight determination, problems of practical importance arouse greatest interest. These papers generally describe experimental studies. The number of theoretical papers on this problem is much smaller.

Most attention has been paid to viscometric studies, and second place, as far as number of papers is concerned, is taken by work on procedures using the ultracentrifuge.

Papers on the determination and calculation of average molecular weight by osmometric and cryoscopic methods and from terminal groups are fairly numerous. There are fewer papers on light scattering by polymer solutions, and they have begun to appear only recently. There are also some papers which discuss the possible use of procedures which have not yet found such extensive application as those noted earlier.

The viscometric method, which requires simple apparatus, is the most convenient method for determining the molecular weight of polymers. It is becoming the most commonly used method of control in industry. The interest of Polish authors in this method is determined to a considerable extent by the practical approach.

Among the large number of papers in this field, mention may be made of the comparatively early determinations, the first to appear in the scientific literature, of the viscometric relationships for polyethylene terephthalate[1]. Tursa and Skwarski determined the molecular weights of a number of low-polymer polyterephthalate fractions by a cryoscopic method and, with the aid of viscometric data, they calculated the constants of the Mark–Houwink equation for the system studied.

Among recent work, mention may be made of a paper by Nadziakiewicz and Jedlińska[2] of the Lodz Synthetic Fibres Institute, devoted to methods of determining the degree of polymerisation of cellulose in solvents in which it does not undergo degradation under the influence of atmospheric oxygen. On the basis of published data and their own studies, they concluded that ethylenediaminecadmium hydroxide has a number of advantages as a solvent for cellulose. Overcoming difficulties in the procedure, associated with the low solubility of some forms of cellulose, the authors confirmed that the viscosity of the solution is linearly dependent on the degree of polymerisation in the range studied, and derived the viscometric equation.

Several other papers[3,4] have been devoted to studies of the dependence of the viscosity of solutions of cellulose in other solvents on the degree of polymerisation.

The viscosity of solutions of other polymers was studied by Kryszewski[5], Skwarski[6], Orszagh[7], Łaszkiewicz[8], and others.

Problems new to the scientific literature in this field were raised by Połowiński[9,10] of the Physical Chemistry of Polymers Faculty in Lodz Polytechnical Institute. Poło-

winski studied the dependence of the viscosity of solutions of graft copolymers on their molecular weight, carrying out the experiments using graft copolymers with accurately determined molecular structure. He suggested that when the solvents used are "good" solvents for the polymers present in the copolymers, the viscometric data can be explained on the basis of the theory of the viscosity of solutions of branched polymers, developed by Berry and Orofino. The premise adopted was confirmed experimentally.

Among these papers mentioned briefly, particular attention may be drawn to several devoted to the development of apparatus. These include papers on viscometers with replaceable capillaries[11], air-tight seals[12], and electronic control of the duration of flow of liquid, giving very high accuracy in the measurements[13], and modifications of a viscometer[14] in which dilution of the solution is carried out.

In addition to these papers, dealing directly or indirectly with the viscometric determination of the molecular weights of polymers, there is a fairly large number of papers describing studies of the viscosity of polymer solutions from another viewpoint. Because of space limitations, we merely cite these papers without giving details[15-31].

Studies using the ultracentrifuge to determine the molecular weight of polymers and the determination of polydispersity by sedimentation analysis have been carried out in Poland in only one research institute, namely the Scientific Research Institute of the "Oświęcim" Chemical Plant. Some papers have been devoted to a discussion of the applicability of the equations derived by various authors, particularly the Flory–Mandelkern equation, for the determination of molecular weights by the centrifuge method[32-34]. In some papers the results of the discussions were compared with the author's own experimental results. The materials studied were polycarbonates and copolymers of butadiene.

Most authors have dealt with methods for determining the average molecular weight.

In the field of osmometry, the following may be mentioned: a methodological paper by Marciniak and Szota[35] on measurements at elevated temperatures; a paper by Robalewski[36] on the "membrane balloon" effect in osmometry; the previously mentioned paper by Brzeziński[20] on the determination of the second virial coefficient and the molecular weight of an isotactic fraction of polypentene; a paper by Skwarski[37] on the osmometric determination of the molecular weights of *N*-substituted polyamides; and papers by Kalfus[38] on the errors in osmometric measurements, and modifications to apparatus preventing the membrane balloon effect.

Cryoscopy is now generally accepted as a convenient method for determining very low molecular weights[39-42] and those of polymers which have not been thoroughly studied.

Other methods for the quantitative determination of the molecular weight of polymers are for the most part modifications of chemical methods of determination from terminal groups[43,44], for example using tracer atoms or electrochemistry.

There are other noteworthy papers describing new techniques. These include a paper by Kryszewski[45], demonstrating the possibility of calculating the constants of the viscometric equation from the results of X-ray diffraction studies, and a paper by Łabudzińska[46] describing the construction of a simple apparatus of her own design, which makes it possible to determine the molecular weight and the second virial coefficient with sufficient accuracy from light scattering measurements. The method was tested using polystyrene solutions.

In general terms, we can say that none of the Polish research centres have carried out systematic studies on the determination of molecular weights as the main aim of their research work. Most of the papers cited deal with developments following from earlier work, for example on the kinetics of polymerisation processes or the study of the dependence of the properties of polymers on their molecular weight.

This approach is revealed in the nature of papers in this field. Polish authors have first of all developed measurement procedures, or introduced simplifying modifications, or have found systems and corresponding constants for studying a large number of interesting materials for which insufficient published data were available.

The situation is quite different when we come to the characterisation of polydispersity. Although in this field also studies involving the investigation of reaction mechanisms and other work on kinetics are being carried out, there is a large number of papers devoted exclusively to methods of fractionation and methods of determining molecular weight distribution functions.

Of the analytical methods characterising polydispersity, only two have aroused great interest: sedimentation analysis and turbidimetric titration. As already noted, only one research centre — The Scientific Research Institute of the "Oświęcim" Chemical Plant — has examined the use of the ultracentrifuge for the fractionation of polymers.

Of the papers which have been published there, two deal with the determination of the molecular weight distribution function for butadiene-styrene rubber (Kalfus and coworkers[47]) and stereoregular polybutadiene (Mitoraj[48,49]). The authors discuss the influence of the polymerisation parameters on the distribution function, and also the influence of the distribution function on the physicomechanical properties.

Analogous papers deal with the relationship between polymerisation parameters and the results of sedimentation analysis for polystyrene[50] and with extensive studies of the relationship between the polydispersity and properties of rubber[51].

All these papers are clearly experimental in character; a more theoretical approach is adopted in two papers by Kalfus[52,53], in which, contrary to the views of Schulz and Horbach, he puts forward a simplified method for evaluating the results of sedimentation analysis, involving standardisation and calibration.

Finally, one paper[54] has been devoted to purely methodological problems. The authors suggest the use of a preparative centrifuge for the fractionation of polymers. After centrifuging, the specimens are frozen and separated into fractions mechanically.

Again only one research centre in Poland — The Synthetic Materials Institute — has examined the use of turbidimetric titration to characterise polydispersity. This subject is discussed in several papers by Krozer and coworkers[55-57]. The authors, who used these methods to characterise polycarbonates, state that the degree of dispersion of the precipitate obtained is independent of mole-

cular weight, so that the turbidimetric curves obtained can be used to characterise the polydispersity. These methods are also being applied to other materials to permit comparison with results obtained by other methods.

Polish authors have shown much greater interest in methods for the preparative determination of polydispersity; there are a fairly large number of papers dealing with fractionation, and this work is being carried out in various research institutes. Considerable attention is being paid to fractionation by the separation of two liquid phases.

Here, mention must be made of work by Basiński and coworkers[58-60] on the fractionation of various vegetable tanning agents by the separation of two liquid phases in systems with mixed solvents.

In the Physical Chemistry of Polymers Faculty of Lodz Polytechnical Institute, the method involving the separation of two liquid phases is used for the fractionation of synthetic polymers which crystallise particularly readily.

Two systems suitable for the fractionation of polyamides have been found[61-67]. The first is the phenol–ethylene glycol–water system, and the second, which is more suitable from the experimental viewpoint since the rate of separation of the layers is high, is the phenol–tetrachloroethane–n-heptane system. The latter system has also been used with good results for polyethylene terephthalate[68,69].

A similar method involving a slightly different procedure was used to study the fractionation of polycarbonates[70]. Although polycarbonates crystallise slowly and with difficulty from melts, they crystallise readily from solution. For this reason, separation into liquid fractions is important. The authors chose the dioxan–n-heptane system, in which equilibrium is first established between the liquid phases, after which a precipitate forms. The fractionation of polycarbonates with the formation of solid precipitates was studied by Brzeziński and Wajnryb[71] using the chloroform–petroleum ether system.

A combined extraction–precipitation method is now being used in the Physical Chemistry of Polymers Faculty to characterise low-molecular-weight epoxy-resins[72].

The fractionation of poly(vinyl chloride) in the cyclohexanone–glycol system was studied by Ulińska and coworkers[73].

A group of papers has been devoted to the fractional dissolution procedure.

Fractionation involving the extraction of fine fibres of viscose rayon was studied by Skwarski[74]. The use of extraction in a fractionating column has been developed for several materials. Polyethylene has been fractionated in a Francis column at elevated temperature in the xylene–Cellosolve system using graphite as carrier[75].

The column method has also been used for the fractionation of polycarbonates[76], the results obtained being similar to those obtained by precipitation.

Pentone has been fractionated by extraction in a column at elevated temperature (117°) in the cyclohexanone–diethylene glycol system using sand as carrier[77].

A number of papers have been devoted to total fractionation. This method was developed for poly(vinyl alcohol) using the acetone–water system[78].

One joint experimental–theoretical paper[79] and one purely theoretical paper[80] have been devoted to studies of the selectivity of the method.

In addition, there are a large number of papers which deal with the problems of fractionation only indirectly. Some of these papers deserve special mention.

Sibiński and Gralewicz[81] studied the change in polydispersity taking place during the pre-ripening of alkali cellulose. The work is of technical importance, the material studied being cord cellulose.

Turska and Skwarski[82-84] studied chiefly the changes in the polydispersity of polyethylene terephthalate during polycondensation and thermal degradation. They confirmed that during polycondensation, with change in the degree of completion of the reaction, the distribution function changes in accordance with Flory's theory, whereas during thermal degradation it leads to products with greater molecular uniformity; the function obtained corresponds more closely to the theory of Korshak and Slonimskii. The changes in the polydispersity of polyethylene terephthalate under the influence of irradiation have also been studied[85]; the distribution found corresponds most closely to Flory's ideas. An analogous result was obtained in the heat treatment of stabilised polyamides[86].

A joint paper[87] was devoted to the dependence of the mechanical properties of butadiene–styrene rubber on the molecular weight distribution function. It was found that a relationship exists between the polydispersity of the specimens studied and the maximal elasticity, tensile strength, and other mechanical parameters. The influence of the conditions of the polymerisation of styrene on the molecular weight distribution was studied by Kalfus and coworkers[88]. Kamińska[89] carried out analogous studies for polyvinyl chloride.

In many papers on kinetics, which in view of their specialised subject matter will not be discussed here, the distribution functions were determined in order to characterise the product completely.

Summarising these problems related to the fractionation of polymer solutions, it may be concluded that the work carried out in Poland in this field is predominantly experimental in character. An undoubted achievement is the large number of new systems which have been developed and verified. It is worth emphasising that the new fractionation systems have considerable advantages from the practical viewpoint, since they generally reduce considerably the duration of the fractionation with an appreciable saving of time.

In addition to papers dealing with methods for characterising polydispersity, there are also in this field a fairly large number of studies of another type, devoted to an analysis of the changes taking place during the formation of polymers, and also on the determination of the dependence of the physicomechanical properties of polymers on the distribution function. Since it has been established many times that there is a close relationship between the polydispersity of a polymer and the possibility of its practical application, and also since the polydispersity undergoes marked changes under the influence of various parameters of the polymerisation processes, the studies devoted to these problems are extremely interesting.

While taking account of the pure research aspect, which in this field makes it possible to obtain information on phenomena and relationships which have not been clear

until now, it is necessary to emphasise the practical importance of this field of study. A knowledge of the changes associated with polydispersity, which can take place during an industrial process, may give important information for the correction and control of this process. Control of the change in distribution functions of the product obtained or a re-treated product, even with rapid methods of study (with which difficulties are still associated) can hardly provide extensive possibilities in industry. On the other hand, it would undoubtedly be very useful from the industrial viewpoint to be able to control a process for the production or processing of polymers by altering the molecular weight distribution as desired. To achieve this, thorough development of the theoretical principles is obviously necessary. This is one of the reasons why I consider that the study of the relationship between polymerisation processes and the polydispersity and properties of macromolecular products is a very important field of research.

The very important relationship between work which at first sight concerns purely theoretical problems and work concerning industrial problems is also clearly shown in another field, which is represented fairly extensively in Poland; this work is described in theoretical papers and in a small number of experimental papers devoted to work carried out in the Technical Physics Laboratory of the General Chemistry Institute, on the hydrodynamics of polymer solutions. The authors, Ziabicki and Takserman-Krozer, have for a number of years been making a thorough study of a single problem, which has still not been solved. This problem concerns the behaviour of macromolecules in fields with other than transverse velocity gradients. As far as polymer solutions are concerned, the theory of their behaviour in the field of a transverse velocity gradient, like that for capillary viscometers, has for long been extensively treated in the scientific literature.

It should be noted, however, that there exist a number of important phenomena to which this theory cannot be applied, such as the formation of jets from the spinning solution in the production of synthetic fibres. The authors cited are therefore gradually beginning to develop a more general theory. The first papers in this field are purely theoretical[90-93] and begin with the simplest systems, that is a field with longitudinal velocity gradient and an immobile rigid ellipsoidal molecule. The first papers examine problems of the viscosity of a dilute solution and the orientation of this type of rigid molecule. The subsequent papers deal with the more complicated case of flexible macromolecules in solution and the viscosity of dilute solutions of these macromolecules in a field with longitudinal gradient.

The next stage, published jointly by both authors[94-99], expands previous discussions, but from a slightly different viewpoint. This series of papers is devoted to the problem of the restoration and breakdown of liquid fibres. Of these papers, only the last is confirmed experimentally by fast cinematography.

The earlier papers examine theoretically possible mechanisms for the restoration and breakdown of the jet under the influence of various parameters.

Subsequent papers by Takserman-Krozer[100-107] examine and summarise earlier studies of the properties of solutions of flexible chain macromolecules in the field of a general velocity gradient. On the basis of theoretical discussions, the author reaches general solutions characterising the distribution function, the orientation and deformation of the macromolecules, the time dependences, and other quantities.

Despite the fact that other work by these authors has not yet solved the problem raised, it can be confidently stated that an important step forward has been taken in this field.

These papers also provide an interesting example of work which is clearly theoretical in character but which should undoubtedly have a considerable influence on methods for organising the industrial process of fibre formation.

Another field which although not represented by a large number of papers has nevertheless aroused the interest of a number of authors is the study of polyelectrolyte solutions. Here mention may be made of the work of Ulińska, Wojtczak, Huppental, and others in the General Chemistry Faculty of the Copernicus University in Torun[108-116], devoted primarily to the study of various properties of polymethacrylic acid solutions, namely its adsorption, analytical methods for its determination, etc.

Bolewski[117] is studying polyelectrolyte solutions from another viewpoint; he is investigating the influence of the ionic strength of the solution on its properties.

Kryszewski[118,119], who is concerned chiefly with other problems, has also devoted two interesting papers to polyelectrolyte solutions. It can be seen that this field is attracting fairly serious attention from Polish authors working on polymers.

As already noted, the general character of research work in Poland, devoted to the study of polymer solutions, can be described as involving attempts to apply science in the national economy. Even papers which appear purely theoretical are generally of value as providing a theoretical basis for future industrial processes. This is as it should be.

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