Methods of Preparation of UHMWPE Membranes

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One of the materials that attracts attention as a potential material for membrane formation is ultrahigh molecular weight polyethylene (UHMWPE). The methods used to prepare membranes from semicrystalline (SC) polymers, including UHMWPE, can be divided into two main groups: solvent-free and solvent-based methods.

Keywords: ultrahigh molecular weight polyethylene ; membrane formation ; powder sintering ; thermally induced phase separation

1. Solvent-Free UHMWPE Membrane Formation Methods

1.1. Powder Sintering

The powder sintering method is based on the partial or complete melting of powder particles placed into the desired mold. In this process, the macromolecules on the surface of the molten polymer powders intertwine ^[1] and the sample becomes structurally integral after cooling. Obviously, this method is unsuitable for fine control of the structure of the obtained capillary-porous bodies, as the pore size is directly dependent on the size of the particles used (note that, generally, nearly spherical particles with unimodal size distribution are preferably used in powder sintering).

In spite of this drawback, the proportion of papers focused on powder sintering as a method of ultrahigh molecular weight polyethylene (UHMWPE) membrane formation ^{[2][3][4][5][6][7][8][9]} among all the papers devoted to the problem of UHMWPE membrane preparation is much greater than that for other polymers, such as polypropylene ^[10] and polyvinylidene fluoride ^{[11][12]}. Undoubtedly, powder sintering for the preparation of membranes has advantages because it does not require complex equipment or organic solvents.

The structure and transport properties of the obtained membranes are controlled by:

- Changing the sintering time ^{[3][4]} that determines the duration of interdiffusion of the macromolecules in the molten surfaces of the particles;
- Changing the polymer molecular weight (MW) [4], which determines the rheological properties of the polymer melt;
- Raising the external pressure to 69 MPa $^{[6]}$ and the temperature of sintering from 135 °C $^{[3]}$ to 250 °C $^{[13]}$.

Original methods of UHMWPE powder sintering were proposed in [3][5][7][9][14]. For example, in [3], powder sintering was performed in an inert liquid (80 vol% of glycerol) and in the presence of 2–4 vol% of plasticizer (m-xylene or paraffin oil). In [5][9][14], a dry powder was mixed with NaCl crystals in ratios ranging from 1:1 to 1:9 before sintering. Then, the salt was removed from the membrane (obtained by hot pressing) by sonication in a water bath or washing at 250 bar. In [7], sintering was performed during the rotation of the vessel with the powder.

The membranes obtained by powder sintering from UHMWPE usually have quite high mechanical strength (24–36 MPa) and a high pure water flux (from ~12 ^[4] to ~2500 ^[6] l/m^2 min atm) but relatively low overall porosity compared to microfiltration membranes obtained from other polymers by thermally induced phase separation (TIPS) and immersion precipitation. However, the UHMWPE membranes prepared by powder sintering also have some drawbacks. In particular, most of these membranes are several millimeters thick, while common microfiltration membranes are several hundred micrometers thick. In addition, as shown in ^{[6][8]}, the membranes prepared by sintering have quite a wide pore size distribution. For example, in the membrane with an average pore size of 2.16 μ m, the maximum through-pore size was 13.41 μ m. Thus, the separation performance of the discussed membranes in a typical microfiltration process is rather poor.

Interestingly, the problem of UHMWPE powder sintering was also discussed in ^{[13][15][16][17][18][19][20][21]}, but these studies did not aim to prepare membranes. Rather, in these papers monolithic films or composites were prepared and fundamental aspects of the UHMWPE sintering process were addressed. It was shown ^{[13][15]} that the continuity of the samples after sintering was not only the result of macromolecule reptation at the boundary between the particles but also of the so-called melting explosion process. The latter process leads to a fast sideway motion of the macromolecules when the polymer crystallites melt, thus ensuring entanglement between the macromolecules in different powder particles.

1.2. Stretching

Stretching of monolithic films is widely used to prepare porous structures from a variety of semicrystalline (SC) polymers [22][23]. The behavior of SC polymers in the process of stretching has been the subject of a large number of fundamental studies published recently [24][25][26][27][28][29][30][31][32][33][34][35]. These papers present detailed studies of the mechanism of craze forming and processes that happen in a nano-scale sample (such as lamellae fragmentation ^{[32][33][34]}, lamellae slipping ^{[28][34]}, recrystallization ^[36], microbuckling ^[25], etc.). The features of UHMWPE stretching are discussed in ^{[37][38]} ^{[39][40][41][42][43][44]}.

Monolithic UHMWPE films used as the starting material in membrane formation via stretching are obtained by hot pressing of the powders at a temperature lower than or higher than the polymer melting temperature $\frac{[17][45][46][47][48]}{calendaring}$, calendaring $\frac{[49]}{s}$, skiving off the block $\frac{[50]}{s}$, etc.

To date, only a small number of publications have reported UHMWPE membrane formation by stretching ^{[40][51][52]}. Many more papers have focused on UHMWPE membrane formation by a combination of TIPS and stretching.

The structure of the membranes is usually controlled by changing the stretching temperature, draw ratio and method (oneand two-axis stretching).

2. Solvent-Based Methods of UHMWPE Membrane Formation

Among the numerous methods of membrane formation from SC polymers through solutions, only the TIPS method is suitable for UHMWPE membrane preparation. This method was proposed in 1981 ^[53] and nowadays is one of the most common methods of membrane preparation from SC polymers. A typical TIPS process starts with the preparation of a homogeneous mixture of a polymer and a diluent (usually a low MW liquid) at an elevated temperature. Then, a polymer solution is shaped into a desired form (such as a flat sheet or hollow fiber) with a casting knife, extruder, etc. Later, the thermal energy is removed from the sample either on air or in an inert liquid medium. At low temperatures, the solution becomes thermodynamically unstable and thus cooling of the mixture induces phase separation and polymer crystallization to produce a porous structure. Then, the solvent is removed from the pores of the formed membrane by extraction or annealing in vacuo.

2.1. Conventional Physico-Chemical Basis of the TIPS Method

The first attempt to formalize the TIPS process of an SC polymer mixture with a solvent and to connect the obtaining structures with the thermal behavior of the mixtures based on temperature-composition phase diagrams was made in 1985 by W.C. Hiatt et al. ^[54]. However, a series of papers entitled "Microporous membrane formation" published in the early 1990s by D.R. Lloyd et al. ^{[55][56][57][58][59][60][61]} received much greater recognition. In this series of papers, the authors plotted experimental phase diagrams, discussed the mechanisms of membrane formation and studied the effect of membrane formation process parameters (such as cooling rate, solvent and polymer nature, concentration and MW of the latter, presence of a nucleating agent, etc.) on the structure of the obtained membranes. The understanding of structure formation in mixtures of SC polymers with low MW liquids during their TIPS, proposed by Lloyd et al., has undergone only some minor changes and has survived to this day in an almost unchanged form. For example, this understanding has been briefly summarized in recent reviews ^{[62][63][64][65][66].}

2.2. Alternative Physico-Chemical Basis of the TIPS Method

An alternative view of the process of structure formation during TIPS was proposed and presented in Refs. ^{[67][68][69][70][71]} ^[72]. This point of view was developed due to adding one more boundary curve to the phase diagrams. The curve reflects the dependence of the polymer swelling degree on temperature or is the same as the dependence of the low MW component solubility in the SC polymer amorphous regions on temperature. Although this curve was for the first time plotted on a phase diagram by R.B. Richards in 1946 ^[73], its significance for describing the thermal behavior of SC polymer mixtures with low MW substances has not been given due attention. This curve has been experimentally constructed since then only in a few papers ^{[67][68][69][70][71][72][74][75][76][77]}. However, it was also plotted on the schematic phase diagrams in a recent review ^[78] and was shown as a hypothetical extrapolation of the binodal curve in Refs. ^{[79][80]} ^{[81][82][83]}. The absence of this curve on the published phase diagrams is explained by the fact that the common methods used to plot phase diagrams, such as differential scanning calorimetry (DSC) and the cloud point method, are not suitable for finding its position. At the same time the published data on the temperature dependence of the swelling degree of a polymer ^{[84][85]}, obviously, can be used to reconstruct the position of this curve on a temperature-composition phase diagram. Importantly, the presence of this curve drastically changes the thermodynamic meaning of the phase diagrams. Presence of this curve enables one to delineate on the phase diagram regions where uniform and nonuniform gels exist.

An extract from the IUPAC goldbook ^[86] states that a "gel is a non-fluid colloidal network or polymer network that is expanded throughout its whole volume by a fluid that can contain a polymer network formed through the physical aggregation of polymer chains, caused by hydrogen bonds, crystallization, helix formation, complexation, etc., that results in regions of local order acting as the network junction points. The resulting swollen network may be termed a thermoreversible gel if the regions of local order are thermally reversible". Bearing that in mind, one can assert that the crystallization of an SC polymer is a process of gel formation and gel in this case is a swollen SC polymer network. In addition, the swelling degree of an SC polymer in a solvent is usually known to decrease with a temperature reduction. Therefore, when the gel is cooled, one more process is expected to occur—deswelling of the polymer that results in separation of the liquid that was dissolved in the polymer amorphous regions.

Thus, contrary to conventional opinion, crossing the crystallization line on a phase diagram leads not only to the crystallization of the polymer that fixes the structure formed. Rather, it results in two continuous processes: gel formation (formation of a three-dimensional network of crystallites connected by tie chains and entangled loops) and solvent separation from the amorphous regions of the gel. The latter process is also accompanied by the relaxation of the polymeric matrix. The structure of the formed capillary-porous body depends on the relaxation rate. If the relaxation rate is sufficiently high, the amorphous regions shrink, the polymer deswells and the separated solvent increases the volume of the polymer-lean phase droplets formed during the liquid–liquid phase separation. If the relaxation rate is not high enough, polymer deswelling results in the formation of small droplets of the solvent inside the amorphous regions of the polymer. The foregoing explains the formation of small pores in the walls of the cellular or bicontinuous structure observed in ^{[67][87]}

2.3. Problem of Dope Solution Preparation

The researchers developing new technologies for the preparation of UHMWPE membranes via TIPS encounter difficulties already at the stage of solvent selection. There are several reasons for this. First, there are no solvents capable of dissolving pure polyolefins (including PE) at room temperature. Aromatic (toluene, xylenes and other alkylbenzenes) and aliphatic (n-alkanes, branched and cyclic alkanes) hydrocarbons, which are considered to be the best solvents for PE, as well as some chlorine derivatives (for example, 1,3,5-trichlorobenzene) form homogeneous mixtures with relatively low MW polyethylenes (LDPE, high-density polyethylene (HDPE)) at temperatures of ~70 to 100 °C, depending on the polymer concentration and nature. Second, the thermodynamic affinity of low MW liquids for polyethylenes (PE) is known to decrease with an increase in the polymer MW ^[90]. Therefore, solvents widely used for membrane preparation from common polyolefins, but having a somewhat worse thermodynamic affinity for the polymer (soybean oil ^[87], DOP ^[91], etc. ^[92]), cannot form molecular mixtures with UHMWPE at technologically reasonable temperatures. Third, due to the high MW of the discussed polymer, the viscosity of its solutions in low MW liquids is significantly higher than that of the solutions of common polyethylenes with the same concentration. Thus, it is rather difficult (even for a solvent with good thermodynamic affinity for PE) to obtain homogeneous mixtures of components within a reasonable time.

In more than 60% of the cited works, the authors use liquid paraffin (LP), mineral, white and paraffin oils as the solvent for UHMWPE. Interestingly, these are essentially different names for the same object—a liquid mixture of linear and/or branched saturated hydrocarbons. Moreover, as a rule, the fractional composition of the solvent is not disclosed. Only a few works ^{[93][94][95][96][97][98][99]} specify the MW range of the components in LP, usually ranging from 150 to 300 g/mol. In ^[95], the authors used a mixture of hydrocarbons with an average molecular weight of 500 g/mol, which should probably be classified not as LP but as paraffin wax. Only a few studies have employed other substances as solvents for UHMWPE. In particular, it was proposed to use decalin, xylene, naphthenic oil, diphenyl ether, etc. A slightly larger variety of potential solvents have been proposed in patents ^{[100][101][102]}, for example, dioctyl phthalate, dibutyl phthalate, aromatic oils, etc. However, LP is considered the best choice.

2.4. Investigation of Thermal Behavior of UHMWPE Mixtures with Various Solvents

Traditionally, the first stage in the development of new technologies for the preparation of membranes via TIPS is usually the study of the thermal behavior of the chosen mixture. The main result of such a study is the temperature–composition

phase diagram. This practice, however, is much less common when preparing UHMWPE membranes ^{[93][95][96][97][99][103]} ^{[104][105][106][107]}, probably because it is difficult to construct phase diagrams for mixtures containing such a high MW polymer.

Even for the most commonly used mixture of UHMWPE with LP there is no consensus on what type of phase diagram describes the thermal behavior of this system. The phase diagram for the mixture of UHMWPE with LP plotted in ^[95], does not contain liquid equilibrium binodal. Moreover, to make sure no liquid decomposition occurs in the system, the authors obtained a diagram using two methods: DSC, which fixes only the process of polymer crystallization, and light scattering, which can observe turbidity resulting either from liquid decomposition or from polymer crystallization. The fact that the temperature values obtained by different methods are essentially the same led the authors of ^[95] to conclude that the only process responsible for the structure formation upon cooling of UHMWPE mixtures with LP is polymer crystallization. The same opinion is shared by the authors of ^[108]. In the cited paper, the phase diagram of the UHMWPE mixture with LP was plotted in the polymer composition range of 5 to 35 wt%.

However, an analysis of other papers ^{[93][99][109]} indicates that even such a basic question regarding the type of phase diagram of a UHMWPE mixture with LP is still controversial. In particular, in ^[99], despite the fact that, the plotted diagram does not contain binodal, the authors speak of upper critical solution temperature (UCST)-type phase behavior in the system. The potential possibility of liquid decomposition in UHMWPE mixtures with LP has also been reported in Refs. ^[93]

2.5. Combination of TIPS and Stretching

Despite the fact that the TIPS method, as a way of obtaining porous materials, took shape only in the early 1990s, it was in the early 1980s, researchers began to study the behavior of porous bodies obtained upon cooling of a polymeric solution and subsequent drying of porous bodies upon their drawing ^{[110][111]}. However, these papers did not aim to obtain membranes and, hence, study the transport properties of the obtained materials. A combination of the TIPS method and stretching for the preparation of UHMWPE composite membranes was used in ^[112]. In the cited paper, hollow fibers were obtained using an extruder from a mixture of UHMWPE and mineral oil. Then the solvent was extracted and the obtained hollow fibers were dried and subjected to uniaxial drawing in the draw ratio range from 1 to 6. This paper shows that as the draw ratio becomes bigger, the flux, porosity, bubble point, and tensile strength of the membrane strength increase, while elongation at break decreases. The authors achieved a pure water flux of ~350 l/m² h with a maximum pore size of ~0.45 µm and satisfactory mechanical properties. In a later work ^[113], this method was used to obtain hybrid membranes consisting of UHMWPE mixtures with poly(vinylidene fluoride) (PVDF).

Interestingly, stretching can be carried out not only for samples that have already undergone structure formation by TIPS and extraction, as in ^{[110][111][112][113]}, but also directly in the presence of a solvent. The authors of ^[114] compared the results of the biaxial drawing of a "gel film" consisting of 70 wt% UHMWPE and 30 wt% LP and "extracted film," obtained from the same mixture, but with the solvent extracted before drawing. It was shown that when the process was carried out in the presence of a solvent, cavitation nuclei were eliminated, which made the porosity of the resulting membrane dependent on the volume fraction of the solvent in the initial mixture rather than on the draw ratio. As a result, the membranes obtained in the absence of a solvent had higher porosity and ionic conductivity but a wider pore size distribution and worse mechanical properties than those obtained in the presence of a solvent. The extraction of membranes in the presence of a solvent was also carried out in Ref. ^[115].

Works ^{[116][117]} also deserve special attention. In these papers, stretching was carried out not only in the presence of a solvent but also at an elevated temperature. Thus, contrary to the methods discussed above, TIPS proceeded simultaneously with drawing. In these papers, however, little attention was paid to the mechanism of structure formation and it was only stated that an increase in the draw ratio made the pores larger ^[47], and the porosity passed through an extremum at the temperature of the highest crystallization rate of UHMWPE with an increase in the drawing temperature.

3. Advantages and Disadvantages of UHMWPE Membrane Formation Methods

It can be concluded that UHMWPE membranes are nowadays prepared mainly by four methods: by powder sintering, by stretching of monolithic films, by the TIPS method and by the combined method including TIPS and stretching. The advantages and disadvantages of each of the methods, as well as the approximate proportion of the papers devoted to UHMWPE membrane formation by each of the methods, are summarized in **Table 1**. One can see that almost 70% of the papers and patents (from all the works devoted to UHMWPE membrane formation) deal with the TIPS method (~46%) and its combination with stretching (~23%). Interestingly, an analysis of patent literature shows that, in industry, UHMWPE

membranes are generally obtained by the latter method, while no patents report the use of standalone TIPS techniques for UHMWPE membrane formation.

Method of UHMWPE Membrane Preparation	Advantages	Disadvantages	Proportion of Papers Devoted to the Formation of Membranes by the Respective Method
Powder sintering	No shrinkage, simplicity of hardware design, no need to prepare a homogeneos solution, high tensile strength	Low porosity, wide pore size distribution, low selectivity, difficulty in control of the porous structure and properties	~18
Stretching	No need to prepare a homogeneous solution, high tensile strength	High thermal shrinkage, difficulty in control of the porous structure and properties	~5
TIPS	High porosity, good possibilities of control of the porous structure and properties, possibility for modification by inclusion of fillers in the dope solution	Problem of homogeneous solution preparation, lower tensile strength, high shrinkage, low surface porosity	~46
Combination of TIPS and stretching	High porosity, excellent possibilities of control of the porous structure and properties, possibility for modification by inclusion of fillers in the dope solution, high tensile strength	Problem of homogeneous solution preparation, high shrinkage	-23
Other	-	-	~8

Table 1. Conditions for preparing homogeneous UHMWPE solutions.

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