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Solid-state polycondensation (SSP) as a method to obtain high molecular weight polymers

Part II. SYNTHESIS OF POLYLACTIDE AND POLYGLYCOLIDE *via* SSP

Summary — Solid-state polycondensation (SSP) is a competitive method of semicrystalline polymers synthesis to conventional melt polycondensation process. The molecular weight of polymers obtained according to this method is exceptionally high and the process can be carried out under milder conditions than those of the melt state. In this method the side reactions are suppressed, especially those leading to the polymer degradation. In this paper, which is a continuation of our previous publication, application of the SSP method to obtain high molecular weight poly(lactic acid) (PLA) and poly(glycolic acid) (PGA) — polymers of high importance for medicine and environmental protection — is presented and discussed. The two-phase model according to which polycondensation proceeds in the amorphous regions, and the apparatus usually used in this process are also described.

Keywords: solid-state polycondensation, polylactide, polyglycolide, two-phase model.

POLIKONDENSACJA W STANIE STAŁYM (SSP) — METODA OTRZYMYWANIA POLIMERÓW O DUŻYM CIĘŻARZE CZĄSTECZKOWYM. Cz. II. SYNTEZA POLILAKTYDU I POLIGLIKOLIDU METODĄ SSP

Streszczenie — Polikondensacja w stanie stałym (ang. *solid-state polycondensation*, SSP) jest konkurencyjną metodą syntezy polimerów semikrystalicznych w stosunku do tradycyjnej polikondensacji prowadzonej w stopie. Za jej pomocą można otrzymywać polimery o dużym ciężarze cząsteczkowym w łagodniejszych warunkach w stosunku do metody w stopie, co skutkuje ograniczeniem reakcji ubocznych prowadzących do degradacji polimeru. W niniejszej pracy omówiono otrzymywanie metodą SSP wielkocząsteczkowego poli(kwasu mlekowego) (PLA) oraz poli(kwasu glikolowego) (PGA) — polimerów o rosnącym znaczeniu w kontekście ochrony środowiska naturalnego i zastosowań w medycynie. Polikondensacja w stanie stałym zachodzi według modelu dwufazowego. Reakcja między grupami końcowymi łańcuchów zachodzi w fazie amorficznej częściowo krystalicznego polimeru, a powstające produkty uboczne są usuwane z fazy amorficz-

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nej dzięki zastosowaniu przepływu gazu obojętnego lub obniżonego ciśnienia. Opisano również aparaturę jaka jest najczęściej wykorzystywana w tym procesie.

Słowa kluczowe: polikondensacja w stanie stałym, polilaktyd, poliglikolid, model dwufazowy.

INTRODUCTION

The development of synthetic biodegradable polymers, such as poly(α-hydroxyacid)s, is particularly important for constructing medical devices, drug delivery systems, including scaffolds and sutures. Nowadays there are many publications concerning synthesis of aliphatic polyesters like polylactide or polyglycolide which are used mainly in medical or packing applications because of their excellent mechanical properties, biodegradation and compostability [1].

The SSP process enables obtaining higher molecular weight polymers, which cannot be achieved in a typical melt phase process. The resulting quality of the products makes SSP the most attractive method for production of high molecular weight polymers.

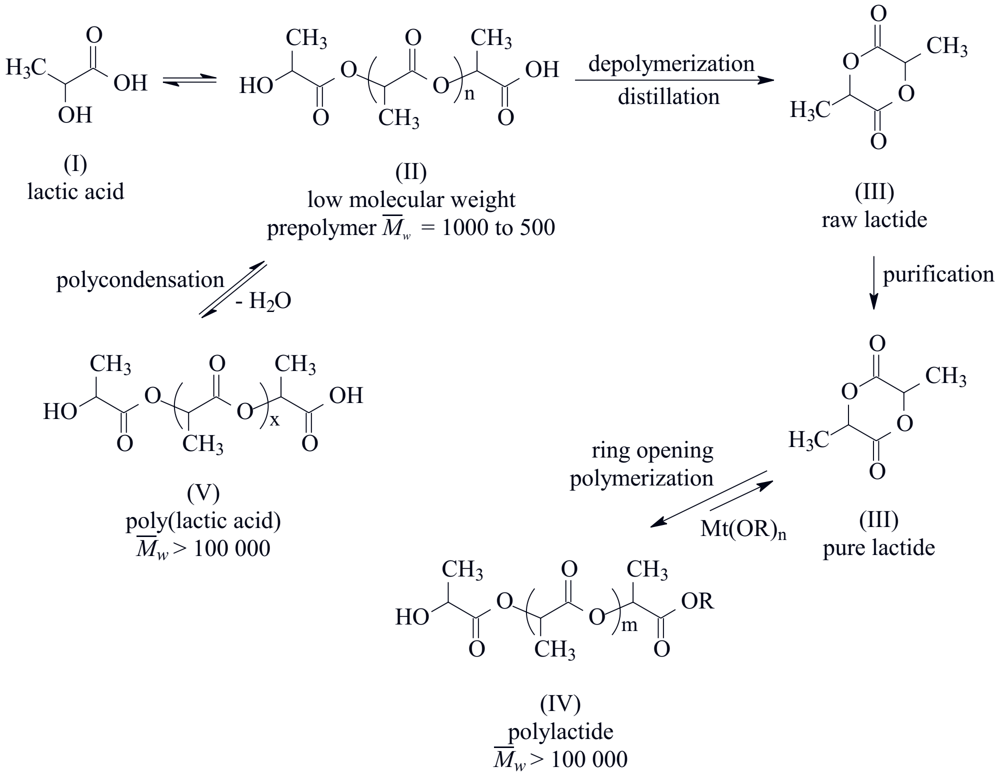
POLYLACTIDE AND POLYGLYCOLIDE OBTAINED BY SSP PROCESS

Both polylactide and poly(lactic acid) (PLA) are polymers composed of the same type of repeating units. However, they are obtained by different methods and as a consequence contain different end groups. The first method, shown in Scheme A, is a direct polycondensation of lactic acid (2-hydroxypropionic acid) [formula (I) in Scheme A] and if side reactions are inhibited, the brittle, glassy PLA of low molecular weight [formula (II) in Scheme A] including secondary alcohol and carboxyl end groups is produced but it is unusable for any applications unless its molecular weight is increased. Therefore, it is further subjected to SSP to obtain high molecular weight PLA [formula (V) in Scheme A].

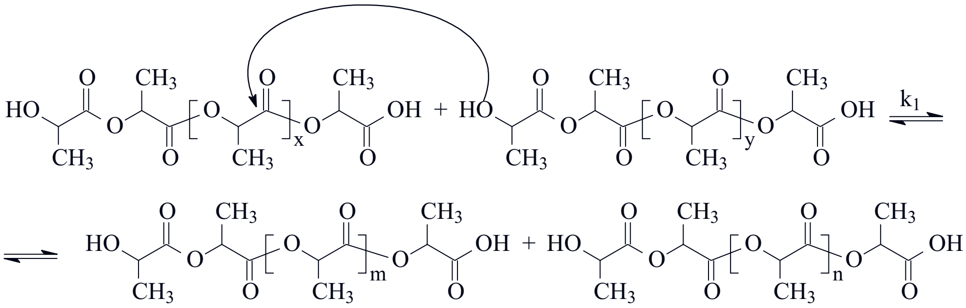
The second method to obtain PLA is polymerization of lactide, usually catalyzed by covalent metal alkoxides [$Mt(OR)_n$] like $Sn(Oct)_2$. As a product PLA with secondary alcohol and ester end groups is formed [formula (IV) in Scheme A]. Dilactide cyclic dimer of lactic acid, commonly known as lactide [formula (III) in Scheme A], is obtained by depolymerization combined with distillation under reduced pressure of low molecular weight prepolymer [formula (II) in Scheme A].

The polymerization method was the only one to produce pure, high molecular weight PLA until Mitsui Toatsu Chemicals commercialized a process in which lactic acid is azeotropically dehydrated in a refluxing, high boiling, aprotic solvent under reduced pressure to obtain poly(lactic acid) with molecular weight greater than 300 000 [3, 4].

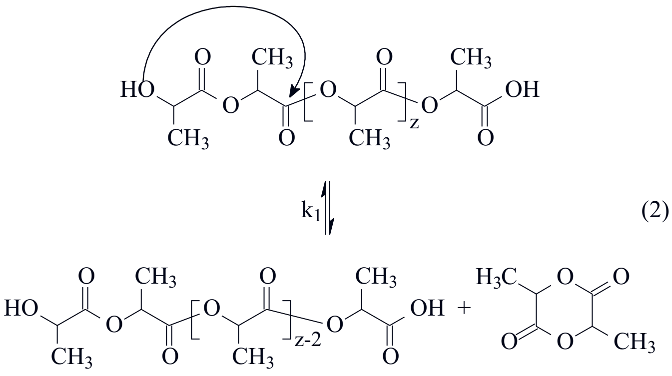
In contrast to polyesters obtained by heteropolycondensation like poly(ethylene terephthalate) (PET), in PLA



Scheme A. Two reaction pathways leading from lactic acid to polylactide and poly(lactic acid) (according to [2]) synthesis only one type of the reaction proceeds to form high molecular weight polymer — reaction of carboxylic with hydroxylic groups. Side reactions such as transesterification lead only to different molecular weight distribution:


(1)

or molecular weight reduction by formation of the cyclic product — lactide [5, 6]:


(2)

It should be emphasized that industrial application of SSP refers primarily to production of polyamides and polyesters. SSP is used mainly to produce poly(ethylene terephthalate) [7] and polyamides 6 and 66 [8]. Molecular weights of other polyesters such as poly(butylene terephthalate) (PBT) [9], poly(trimethylene terephthalate) (PTT) [10] and poly(ethylene naphthalene) (PEN) [11—13] can also be increased with the SSP method.

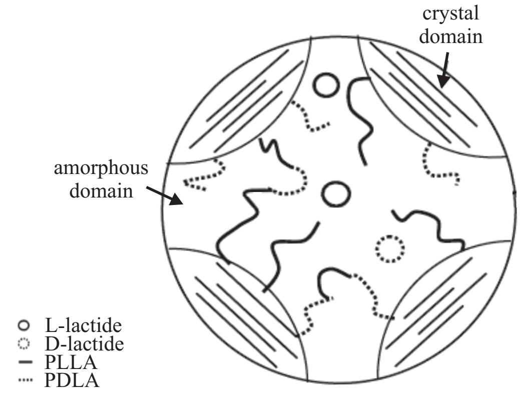


Fig. 1. Scheme of the two-phase model of SSP (according to [14])

Recently it has become accepted, that SSP of L-lactic acid (LLA) can also give poly(L-lactic acid) (PLLA) with a significantly high molecular weight (500 000) which is comparable with that of PLLA obtained by the lactide polymerization.

SSP proceeds according to the two-phase model [14—16]. Scheme of this model is presented in Figure 1. In this model the reaction progresses stepwise in the amorphous regions of the semicrystalline polymer. End groups, by-product and low molecular weight oligomers are excluded from crystalline regions.

According to the two-phase model, the diffusion of end groups occurs in the amorphous phase through translation of oligomers, motion of terminal segments or through exchange reactions. That allows reactive end groups to shorten the distance between them, which is then suitable for the reaction.

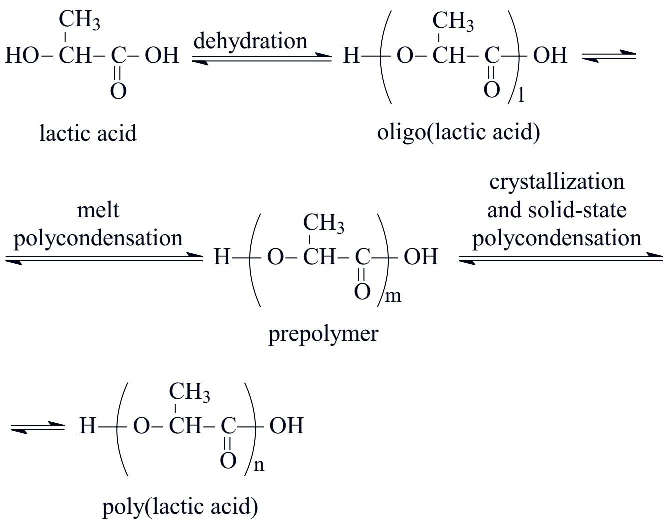
Gostoli *et al.* [17] and Meyer [18] proposed a general model for polycondensation in the solid state. They made the following assumptions: polymer end groups, monomers, condensate, and catalysts are present exclusively in the amorphous phase. Upon crystallization, these species are expelled from the ordered crystalline phase.

Mallon and Ray [14] developed a comprehensive model to handle the reactions in polymers undergoing polycondensation reactions in the solid state. The polymer crystalline fraction is modeled as containing only repeated units, thus concentrating end groups and by-product in the amorphous phase. In addition, by using a general framework for the equations, many previously neglected effects were included; for example, variable crystallinity and gas phase mass transfer effects.

There are several reports concerning synthesis of poly(lactic acid) *via* the SSP method [1, 19—21].

As a starting material, water solution of lactic acid is used and the process is conducted in three stages. After removing water by distillation, an oligomer is obtained, which is then condensed in the melt state in the presence of a catalyst to obtain a prepolymer. The prepolymer is then crystallized at 105 °C (p ≈ 70 Pa) and then it is submitted to SSP carried out at 150 °C under reduced pressure (p ≈ 70 Pa). According to this procedure, shown in Scheme B, high molecular weight PLA can be obtained.

As it was presented by Moon *et al.* [22, 23] high molecular weight PLLA can be obtained by the melt/so-



Scheme B. Synthesis of high molecular weight poly(lactic acid) via SSP (according to [15])

lid-state polycondensation of LLA in the presence of a tin(II) chloride dihydrate/*p*-toluenesulfonic acid catalytic system. In this process, a prepolymer of 20 000 molecular weight was prepared by typical melt-polycondensation, then crystallized at 105 °C for 1 h, and heated at 140 or 150 °C for 10—30 h under reduced pressure for further polycondensation in the solid state. Characteristics of products of the melt/solid-state polycondensation at various stages for PLLA are presented in Table 1.

T a b l e 1. Characteristics of typical products of the melt/solid-state polycondensation at various stages for poly(L-lactic acid)

Stage	T, °C	p, Pa	*Time*, h	$\overline{M}_w$ [a]	$\overline{M}_w/\overline{M}_n$ [a]	T_g [b], °C	T_m [b], °C	Crystallinity[c], %
Melt polycondensation[d]	180	1350	5	1300	1.9	48	157	53
Crystallization[e]	105	70	1	1600	1.6	nd[h]	159	63
	105	70	2	1500	1.5	nd[h]	158	66
Solid-state polycondensation[f]	150	70	10	32 000	3.4	nd[h]	178	85
Solid-state polycondensation[g]	150	70	10	26 000	4.7	nd[h]	179	89

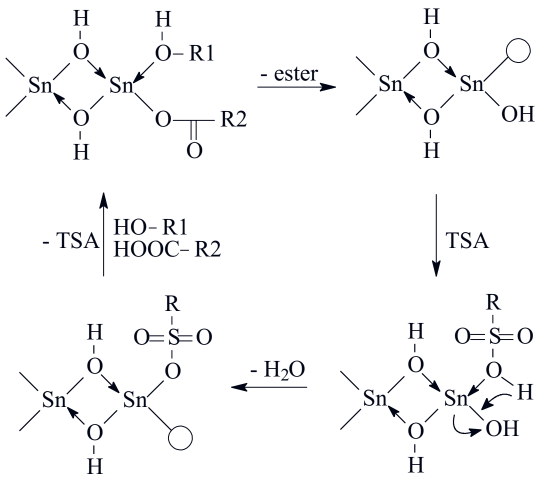
[a] Determined by GPC relative to polystyrene standards with chloroform as the eluent; [b] Measured by DSC (heating rate 10 °C/min); [c] Based on enthalpy of fusion; measured by DSC (first scan); [d] For PLLA (20 g) with $SnCl_2 \cdot 2\,H_2O$ (60 mg) and TSA (50 mg); [e] For the melt polycondensate (*ca.* 4 g); [f] After the heat-treatment for 1 h; [g] After the heat-treatment for 2 h; [h] nd = not detected.

The resultant molecular weight of PLLA, according to the authors, exceeded 500 000 which is comparable with that of PLLA obtained by ring opening polymerization (ROP) of lactide. The authors proposed a plausible reaction mechanism, presented in Scheme C, in which together with chain growth the increase in crystallinity can continue until the crystallinity exceeds 43—45 % [22, 24].

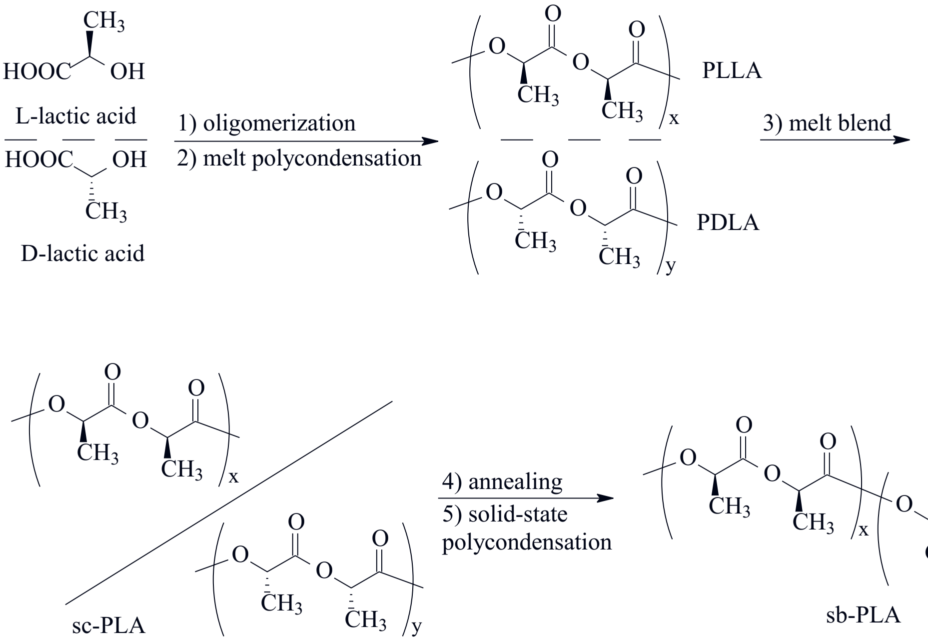
Xu *et al.* [25] presented the effect of crystallization time of PLLA prepolymer on the molecular weight of the resultant polymer. In this process, prepolymer with a molecular weight of 18 000 was firstly prepared in a melt polycondensation process. The prepolymer was crystallized at 105 °C for different times, and then heated at 135 °C for 15—50 h for further SSP process. The results showed that the molecular weight of PLLA reached the maximum value under the condition of the crystallization time of 30 min and SSP of 35 h [25].

Qian *et al.* [20] claimed that the SSP process can be carried out without continuous vacuum supply or purging by inert gases. They used in this new process a desiccant for removing water as the by-product of polycondensation. PLLA obtained according to this process was characterized by molecular weight higher than 300 000, small dispersity index, low monomer content and high crystallinity.

Recently, Fukushima and Kimura [26, 27] revealed that stereocomplexed PLA with high molecular weight can be obtained by simultaneous SSP of the powdery prepolymers of PLLA and poly(D-lactic acid) (PDLA). Ordinary melt polycondensations of L- and D-lactic acids gave the PLLA and PDLA prepolymers having moderate molecular weight which were pulverized for blending in 1:1 ratio. The resultant powder blends were then subjected to SSP at 130—160 °C for 30 h under reduced pressure of 70 Pa. In the last step, this melt-blend is subjected to



Scheme C. Hypothetical polycondensation mechanism by the action of the tin(II) chloride dihydrate/p-toluenesulfonic acid catalytic system (according to [22])



Scheme D. Synthesis of stereoblock poly(lactic acid) (sb-PLA) by melt/solid-state polycondensation (according to [20])

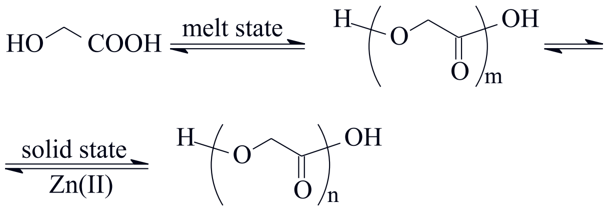
SSP at the temperature where the dehydrative condensation was allowed to promote chain extension in the amorphous phase with the stereocomplex crystals preserved. Finally, stereoblock poly(lactic acid) of high-molecular weight was obtained. The stereoblock poly(lactic acid) synthesized by this way, summarized in Scheme D, showed a higher melting temperature in consequence of the controlled block lengths and the resulting higher molecular weight [27].

It was found that the SSP process can be applied to obtain blend compositions of amorphous poly(D,L-lactic acid) and crystalline PLLA of various molecular weights [28]. The blends were prepared in one step by melt/solid-state polycondensation in the presence of either $SnCl_2 \cdot 2\,H_2O$/*p*-toluene sulfonic acid (PTSA) or Sb_2O_3 as a catalyst. The polymers prepared in the presence of Sb_2O_3 were characterized by two- or three-fold higher molecular weight with respect to the comparable polymers prepared from a pure LLA source.

Katiyar and Nanawati [29] described the novel procedure to prepare high molecular weight PLA nanocomposites using the SSP technique. The proposed procedure is a step ahead beyond conventional *in situ* synthesis of PLA nanocomposites with either LLA or L-lactide as starting materials. Thus, this method is an alternative approach to yield high molecular weight PLA nanocomposites with tailored morphology at the nano-level and further, the product can be used directly for the end applications.

Shinno *et al.* [30] proposed the SSP process in which the monomer removal technique is used. The fundamental reaction dynamics in the solid state two-step postpolymerization of L-lactide is reported to have proved how effectively the polymerization of L-lactide can be promoted by crystallization of PLLA. Presented data would contribute to determination of the reaction conditions for the solid-state postpolymerization of L-lactide to reduce the remaining monomer in the product.

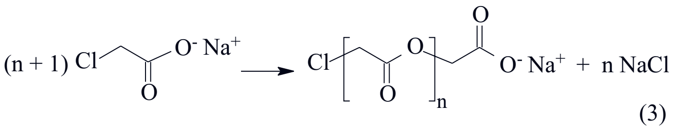
Moon *et al.* [31] reported that polyglactin, a copolymer of glycolic acid (GA) and LLA, can be prepared by solid-state polycondensation carried out at 170 °C for 10—20 h. Starting from the crushed melt polycondensate with the particle diameter of 180—250 μm, the highest molecular weight obtained was 80 000.



Scheme E. Melt/solid-state polycondensation of glycolic acid promoted by catalysis of Zn(II) (according to [32])

A competitive method to ROP of glycolide of obtaining high molecular weight poly(glycolic acid) (PGA) was reported by Takahashi *et al.* [32] and is presented in Scheme E. At first, a solid prepolymer was prepared by melt polycondensation of glycolic acid at 190 °C, and then it was subjected to SSP at the same temperature, to increase its molecular weight. It was found that in contrast to PLA zinc acetate dihydrate was the best catalyst. The weight-average molecular weight of PGA reached M_w = 90 000, which was similar to that of PGA prepared by the conventional ROP of glycolide. This process can afford a facile route to large scale synthesis of PGA. However, the main drawback of this process was brown color of the product.

Schwarz and Epple [33] applied the SSP process for synthesis of polyglycolide [poly(glycolic acid), PGA] applying sodium chloroacetate as a monomer according to equation:



(3)

This reaction leads to a composite of PGA with NaCl.

The NaCl salt can be removed by extraction with water leading to a highly porous polymeric material. The special porous structure of SSP prepared PGA should be advantageous for biomedical materials, *e.g.* resorbable bone fixation devices or bone substituent materials. The polymer chains grow during the reaction, but prolonged heating leads to a subsequent decrease in chain length due to thermal degradation. PGA from the solid-state process is identical with PGA conventionally prepared by ROP, but molecular weight of the product is smaller. It should be noticed that no low molecular weight oligomers were detected in the product.

EQUIPMENT FOR SSP

SSP processes are characterized among others by relatively low operating temperature, uncomplicated equipment and oxygen-free atmosphere. The latter is reached through application of vacuum or assisted by the flow of inert gases. The application scale, as well as starting material, are major factors influencing the choice of one of three mentioned earlier reaction pathways. For small scale the use of the vacuum process is preferred, whereas on a larger scale better is the flow of inert gas which is used widely on an industrial or bench scale.

Mostly the SSP process in vacuum is carried out periodically applying various dryers (*e.g.* tumble dryers, rotary dryers, double-cone dryers or paddle dryers). While in the continuous SSP process the gas flow method is commonly preferred, which can be carried out in production lines with capacities of more than 600 t/24 h. The periodic process is restricted to the production on a smaller scale which is limited by reactor volume between 20 and 44 m^3. Apart from it, also waste polymer may by applied [34].

The SSP reactions can be conducted in various apparatuses (*e.g.* plug and batch flow reactors), depending on the scale. The schemes of typical devices are shown in Figure 2. They can be performed for example in glass tubes [35, 36], in fluidized- and fixed-bed reactors (Fig. 2a) [37, 38], in rotating flasks [39], in tumbler dryers (Fig. 2b) [38, 40], in an inert liquid medium [41—43], in vertical reactors with stirring blades [44], and in rotating blenders [45], *etc*. The system should contain an appropriate apparatus to remove volatile reaction by-products, oligomers and any atmospheric oxygen (*e.g.* bag filters, gas wash-

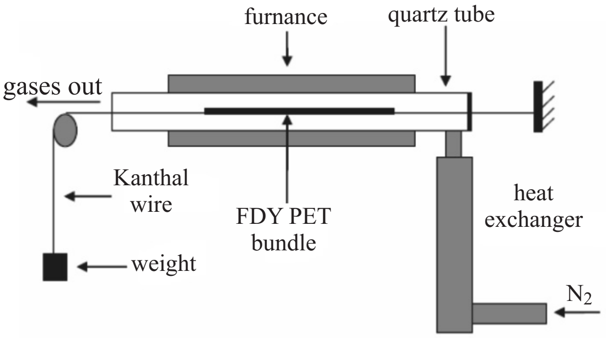


Fig. 3. Schematic diagram of the apparatus used for SSP of fully drawn filament yarn (FDY) polyester

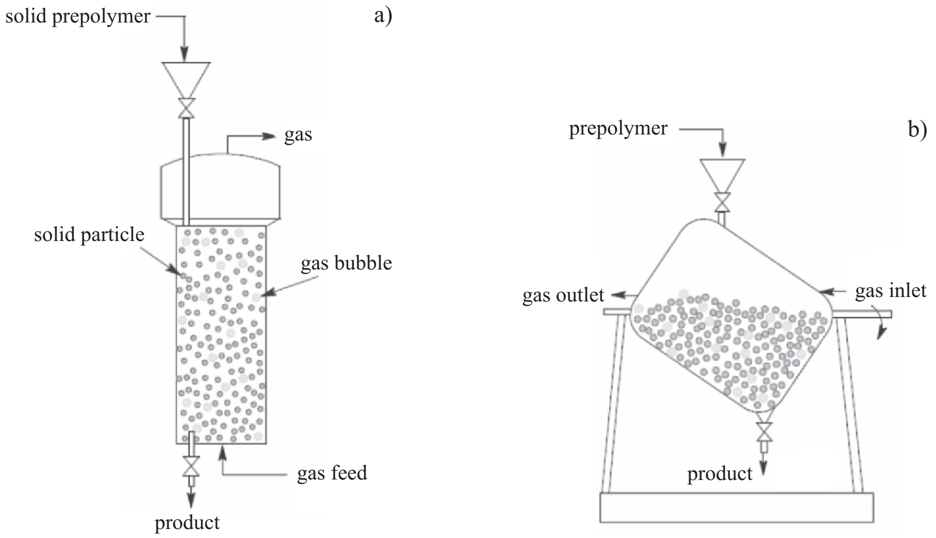


Fig. 2. Typical reactors for SSP (according to [37]): a) fluidized bed reactor, b) tumbler dryer

ing, catalytic gas cleaning) [43]. Mechanical stirring is usually applied in SSP processes, to enhance heat and mass transfer and to prevent particles agglomeration [46].

Interesting modification of the SSP process of obtaining polyester yarn was recently proposed by Agrawal and Mhaisgawali [47]. Post-extrusion solid-state polycondensation (SSP) of a commercial fully drawn filament yarn (FDY) of poly(ethylene terephthalate) was carried out at 220—240 °C for 30 min to 2 h under inert atmosphere (nitrogen) (Fig. 3). After the treatment, molecular weights of the polyester filaments were slightly increased from 16 700 to 26 100. Application of tension during SSP was found to improve also the mechanical properties of the SSP yarn by a small value.

CONCLUSIONS

Solid-state polycondensation is characterized by some important advantages, which make this technique attractive for obtaining aliphatic polyesters. The SSP is a competing process to ROP of lactide and glycolide, taking into account that these cyclic monomers are obtained by polycondensation of lactic or glycolic acid to prepolymer and then by depolymerization combined with distillation under reduced pressure. An additional step needed is the cyclic monomers purification. Melt techniques usually do not lead to high conversion that prevent thermal degradation of the product and also various problems stemming from viscosity increase, such as stirring, heat and mass transfer and reactor handling. Besides, melt polycondensation process stops at a low or medium molecular weight product whereas high molecular weights may be reached *via* SSP. Products of SSP have improved properties because cyclization and other side reactions are suppressed, due to low SSP operating temperatures. The polymers usually have greater thermal stability than those prepared in the melt phase. Increase of the molecular weight of products during SSP is accompanied by an increase in their crystallinity. This affects positively the SSP rate and implies more effective confinement of the amorphous phase. Thus, high concentration and homogeneous distribution of reactive chain ends in the amorphous regions are observed. Moreover, the process is environmentally friendly, because no organic solvents are used.

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Materiały Polimerowe

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Przewodniczący Konferencji: prof. dr hab. inż. Tadeusz Spychaj

Tematyka konferencji będzie obejmować następujące zagadnienia:

— Kompozyty i kompozycje polimerowe (kompozyty i nanokompozyty; nowe materiały termoplastyczne, elastomerowe, chemo- i termoutwardzalne, materiały powłokowe i klejowe, modyfikatory i środki pomocnicze).

— Polimery a środowisko (polimery biodegradowalne, biomateriały polimerowe, polimery i żywice w układach wodnych, recykling materiałów polimerowych).

Program naukowy konferencji obejmuje: wykłady na zaproszenie Komitetu Naukowego (30 min), komunikaty sekcyjne (15 min), komunikaty młodych pracowników i doktorantów/studentów (10 min), sesje plakatowe.

Publikacje i materiały konferencyjne — Dwustronicowe skróty wystąpień zostaną wydane w formie materiałów konferencyjnych. Prezentowane prace (po recenzji) będą publikowane w czasopismach: *Polimery*, *Polish Journal of Chemical Technology*, *Przemysł Chemiczny*.

Terminy — Udział w konferencji należy zgłosić do **15 stycznia 2013 r.**, a skrót wystąpienia nadesłać do **31 stycznia 2013 r.**

Koszty

Opłata normalna: do 31 marca 2013 r. — 1100 zł, a po tym terminie — 1200 zł.

Opłata ulgowa: doktorant/student — 850 zł, osoba towarzysząca — 700 zł (do 31 marca 2013 r., a po tym terminie odpowiednio 950 zł i 700 zł).

Opłata obejmuje: koszty konferencyjne, wyżywienie (obiady i kolacje) oraz program towarzyszący.

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Biuro konferencji: dr inż. Katarzyna Wilpiszewska — Sekretarz konferencji

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