THE USAGE OF MATERIAL QUALITY INDICATORS TO ASSESS THE PROPERTIES OF LINEAR BLOCK-COPOLYURETHANES BASED ON OLIGOETERS

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DOI: 10.17973/MMSJ.2022_11_2022020

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The relationship of the structure with the strength, deformation, thermophysical and tribological characteristics of linear block-copolyurethanes based on oligoeters of regular structure oligooxy tetra-methylene glycol is described by mathematical model. Quality parameters are used to estimate the relationship between the structure and properties of the studied materials. The quality parameters allowed to find optimal composition of initial components.

KEYWORDS

linear block-polyurethane, oligoether, physico-mechanical characteristics, wear, quality parameter

1 INTRODUCTION

Much has been said and written about polyurethanes. Due to their structural and technological capabilities, polyurethanes are among the most universal materials. Therefore, they are rightfully called the materials of the future. Their properties are so diverse that they actually have no boundaries. They work equally well both in usual environment and in extreme conditions [Macala 2017, Balara 2018, Anisimov 2019a,b].

More than 3 million tons of polyurethane materials are processed yearly today, and their world market in 2022 will raise to $74 billion [Valicek 2017, Bielousova 2017, Pandova 2018, Panda 2019].

Polyurethanes are resistant to contact with many chemical liquids, oils, X-rays, fungi, bacteria [Domanski 2016, Panda 2016, Duplakova 2018, Dyadyura 2016 and 2017, Flegner 2019 and 2020]. They are successfully used both in permafrost conditions and in hot countries, in the units of pneumatic-hydraulic devices, in the space industry, in construction and in innovative engineering [Macala 2009, Monkova 2013, Panda 2013 and 2014, Jurko 2016, Thomas 2018, Labun 2021, Ceskovic 2022].

However, the complexity of the chemical structure and structural organization of polyurethanes, as well as a large number of factors that control the relationship between their chemical structure and properties, on the one hand, make them very valuable materials with a widely varying set of properties, and on the other hand, they significantly complicate to systematize the available data on the relationship between their composition and properties [Panda 2017, 2018a,b and 2019]. Therefore, study of the relationship between structure of polyurethanes with strength, deformation, thermophysical and tribological characteristics, as well as mathematical prediction of their behaviour under different operating conditions, taking into account the quality parameters of the material, is of great practical importance.

2 MATERIALS AND METHODS

Block-polyurethanes of different molecular structure are selected as objects of the study. Synthesis was carried out from oligomeric esters (synthesized from adipic acid and glycols of methylene series of different nature: oligoethylene glycol adipate of molecular weight ~2000 ($OEGA_{2000}$), oligobutylene glycol adipate of molecular weight ~500 and ~2000 ($OBGA_{500}$, $OBGA_{2000}$), oligoethylene butylene glycol adipate of molecular weight ~2000 ($OEBGA_{2000}$) and oligomeric ether – oligooxy tetramethylene glycol of molecular weight ~1000 ($OOTMG_{1000}$), synthesized from tetrahydrofuran). Urethane groups were created from 4,4'- Methylene diphenyl diisocyanate (MDI), a low molecular weight glycol – 1,4 butanediol (butylene glycol) (BD) was inserted for obtaining block structure of BPU. Block-copolymer molecule consists of parts that differ in flexibility and repeat [Dyadyura 2016, Flegner 2019 and 2020, Panda 2019 and 2021a,b]. Elastic blocks are formed from flexible parts (oligoeters or oligoesters). Rigid blocks are formed as a result of the self-organization of urethane groups. Molecular weight of all BPU samples is ~50000÷70000 (characteristic viscosity [η] = 0.8÷1.1 dl/g of BPU in dimethylformamide) [ISO 16365-1:2014].

3 METHODOLOGY OF RESEARCH

Linear block-copolyurethanes (BPU) of various morphology were chosen as the objects of the study. As oligomeric glycol (OG) the oligoethers synthesized from tetrahydrofuran with a molecular weight of ~1000 ($OOTMG_{1000}$) were used. Oligoeter $OOTMG_{1000}$ is obtained as a result of splitting of tetrahydrofuran by the ether link in the presence of other catalysts. A special feature of oligoethers is the presence in the main lance of macromolecules of the ether link ≡ C - O – C ≡, which is regularly repeated. BPU on the basis of oligoether have high the hydrolytic stability, thermal stability and frost resistance [Pandova 2012 and 2018, Panda 2017, Murcinkova 2017, Baron 2016, Mrkvica 2012, Zaborowski 2007, Chaus 2018, Vagaska 2017 and 2021, Straka 2018a,b, Modrak 2019, Michalik 2014, Olejarova 2017, Prislupcak 2014, Valicek 2016, Rimar 2016]. Urethane groups were formed from 4,4'-diphenylmethane diisocyanate (MDI). To create a block structure of BPU a low molecular weight glycol, 1,4 butanediol (butylene glycol) (BD), was introduced [Pandova 2012 and 2018, Anisimov 2019a,b, Pollak 2020a].

The characteristics of the initial components (with their chemical formulas) for synthesis of investigated polyurethane block-copolymers (BPU OOTMG$_{1000}$) are given in Table 1.

Table 1. Initial components, used for the synthesis of block-copolyurethanes BPU OOTMG$_{1000}$ [Anisimov 2019a,b].

Component name	**Oligooxy tetra-methylene glycol**	**4.41-Methylene diphenyl diisocyanate**	**1.4-Butanediol (butylene glycol)**
Abbreviated designation	OOTMG1000	MDI	BD
Average molecular weight	≈1000	250	90
Purpose	oligoether	isocyanate	chain extension
Chemical formula	HO[-(CH2)4-O-]nH	OCN-C6H4-CH2-C6H4-NCO	HO-(CH2)4-OH
Characteristics	Hydroxyl number – 54.5-57.5 mg KOH/g; acid number – 0.1 mg KOH/g	Tm =39 °C, Tb =157°C, ρ=1190 kg/m3	Tm =30 °C, Tb =203 °C

The samples were obtained in laboratory conditions by a one-stage synthesis in mass at molar ratio of NCO: **OH = 1.0: 1.0**. Morphological structure of material was varied by changing the ratio of butanediol to oligoether during synthesis. With an increase in the amount of butanediol there is an increase in the content of hard blocks (**P$_c$**) in block-copolyurethanes, which were defined as the part of a molecule with a low molecular weight:

$$P_c = \frac{90 \cdot n + 250(n+1)}{a \cdot M_{oe} + 90 \cdot n + 250 \cdot (n+1)} \cdot 100\% \qquad (1)$$

where **P$_c$** – content of hard blocks, %; **M$_{oe}$** – molecular weight of oligoether(1000); **90** – molecular weight of butanediol; **250** – molecular weight of diisocyanate; ***a*** – number of moles of oligoesther; **n** – number of moles of butanediol; **(n+1)** – number of moles of diisocyanate.

Polyurethane block-copolymers with a content of hard blocks (Pc) in the range from 20 to 66% were investigated, that made it possible to investigate polymers with a wide range of properties. Experimental results were obtained using standard methods of physico-mechanical tests, X-ray structural and thermomechanical analyzes as well as special methods for studying the processes of friction and wear of BPU in the absence of lubrication, in water, in the flow of abrasive particles [Macala 2009, Monkova 2013, Cacko 2014, Jurko 2016, Panda 2018a,b,].

4 RESULTS AND DISCUSSION

As usual, it is necessary to conduct a large number of experiments and analyze the results to create materials with preset complex of properties. The amount of conducted experimental studies has great impact on accuracy of the recommendations. In order to create polymeric materials based on block-copolyurethanes with increased wear resistance, it is necessary, at least, to trace the relationship of their structure with strength, deformation, thermophysical and tribological characteristics.

Table. 2 presents the raw results of physico-mechanical characteristics of polyurethane block-copolymers based on oligoesters of a regular structure (BPU OOTMG1000).

According to Table. 2, with increasing amount of butanediol and, accordingly, the content of hard blocks, the hardness (HA) of polyurethane block-copolymers increases rapidly. The same rapid growth is observed for the conditional tensile strength (ƒp), modulus of tensile elasticity (Ep), Vicat softening temperature (Tp), degree of crystallinity (Sk). The level of elongation (εp) decreases with increasing content of rigid blocks.

Table 2. Physico-mechanical characteristics of block-copolyurethanes based on OOTMG$_{1000}$:BD:MDI of various morphological structure

Parameters	**The amount of butanediol, mol**						
	0	**0.5**	**1.0**	**2.0**	**3.0**	**4.0**	**5.0**
Content of hard blocks, **P$_c$**, %	20	30	37	48	56	62	66
Conditional tensile strength, **ƒ$_p$**, MPa	10	38	53	62.5	61.5	62	63
Elastic modulus, **E$_p$**, MPa	10	15	30	90	220	300	375
Elongation at rupture, **ε$_p$**, %	150 0	780	665	490	445	430 0	425
Residual elongation, **ε**, %	110	20	27	90	155	180	205
Shore hardness, scale **A**, conventional units	60	76	88	95	97	98	98
Vicat softening temperature, **T$_p$**, K	333	363	384	433	473	468	463
Degree of crystallinity, C$_к$, %	0	0	0	1.8	3.8	8.3	15.2
Wearing intensity during friction without lubrication, **I$_c$**·10^4, g/km (P=0.2 MPa, V=0.4 m/s)	18	2.5	3.5	4.0	4.5	5.0	5.5
Coefficient of friction without lubrication, **ƒ$_c$** (P=0.2 MPa, V=0.4 m/s)	2.0	1.2	0.95	1	1.05	1.1	1.15
Wearing intensity during friction in water, **I$_w$**·10^4, g/km (P=0.2 MPa, V=2.3 m/s)	33	24	18	19	20	21	22
Coefficient of friction in water, **ƒ$_w$** (P=0.2 MPa, V=2.3 m/s)	0.8	0.35	0.3	0.33	0.36	0.39	0.4
Intensity of gas abrasive wearing, **K$_g$**·103, g/kg (V=76 m/s, α=45°)	0	8	15	30	45	62	80

The dependency of the residual elongation (ε) from Pc is extreme with a minimum in the region of Pc ~30%. In addition, the dependences ƒp, Ep, HA та Cк in the region of Pc ~ 50% have a noticeable inflection. As for the tribological characteristics of BPU, in the concentration range Pc = 30-50% minimum values of wear intensity and friction coefficient are observed both in the presence of water and without lubrication, and the intensity of gas abrasive wear has a peculiar plateau. Graphical analysis of the whole set of characteristics is difficult, although it gives the most complete information about their relationship. In addition, the graphical method of analysis provides low accuracy of results and also leads to a large number of errors due to the human factor, which is unacceptable. To simplify the analysis of the relationship between the properties of the described materials, a mathematical model that describes the relationship between all the presented properties of materials and their structure has been developed. The described system is a set of functions of one argument - Pc. Functions data source is given in tables. By approximating each individual dependence, we obtain a set of regression equations that will describe the relationship between all parameters. The input data is entered in the form of column vectors, variables are marked with the lower index "exp", after the transition to approximating functions, the index "exp" is discarded. The "linfit" function of the Mathcad package is used to approximate each individual function. There is a fragment of the Mathsad document (Fig. 1), which approximates the dependence of wear without lubrication on the concentration of hard blocks Ic (Pc).

$$X := P_{c.exp} \quad Y := f_{r.exp} \quad S(X) := \begin{pmatrix} 1 & X & X^2 & X^3 \end{pmatrix}^T \qquad a := \mathrm{linfit}(X, Y, S) = \begin{pmatrix} -109.481 \\ 8.341 \\ -0.134 \\ 7.074 \times 10^{-4} \end{pmatrix}$$

$$y(x) := a_4 \cdot x^3 + a_3 \cdot x^2 + a_2 \cdot x + a_1$$

$$f_r(P_c) := y(P_c)$$

$$f_r(P_c) \text{ float}, 3 \rightarrow 8.34 \cdot P_c + -0.134 \cdot P_c^2 + 0.000707 \cdot P_c^3 - 109.0$$

Figure 1. Mathcad approximation block for Ic(Pc)

Figure 1 shows that the variables Pc.exp and Ic.exp are first encoded in X and Y, respectively. This operation is performed to unify the variables of the approximation block, which includes calculating the vector of the coefficients of the regression equation a, using the functions S(X) and linfit (X, Y, S (X)) and creating the form of the regression equation in function y(x). After that, the results of the block are recoded into a more perceptible form and displayed on the screen as a ready-made regression equation. Coefficients with small numerical values are not discarded, because they usually stand for members of a polynomial with high exponents and significantly affect the accuracy of the approximation.

The results of the approximation of the function Ic(Pc) are presented graphically in Fig. 2.

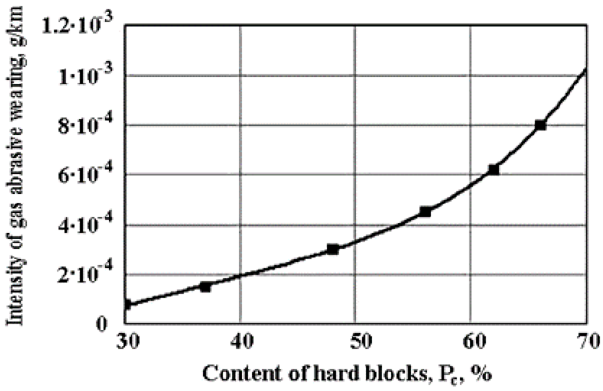

Figure 2. Results of approximation of the function Ic(Pc)

For each individual function, the optimal degree of the polynomial can vary depending on the complexity of the behaviour of the function. Whenever possible, polynomials of lower degrees are used. Taking into account that the number of experimental points is 7, the greatest appropriate degree of the polynomial is 6, in which the curve accurately passes through all the experimental points. The usage of approximating functions of another type (not polynomial) is also not appropriate in this case, because then there are additional limitations on the method of solving the basic system of equations. After approximating all the functions, we obtain the following system of algebraic equations:

$$f_r(P_c)=8{,}34 \cdot P_c - 0{,}134 \cdot P_c^2 + 0{,}0007 \cdot P_c^3 + 109$$

$$E_p(P_c)=-6{,}87 \cdot P_c + 0{,}08 \cdot P_c^2 + 0{,}0013 \cdot P_c^3 + 107$$

$$\varepsilon_p(P_c)=-514 \cdot P_c + 15{,}6 \cdot P_c^2 - 0{,}212 \cdot P_c^3 + 0{,}001 \cdot P_c^4 + 7081$$

$$\varepsilon(P_c)=-77 \cdot P_c + 2{,}09 \cdot P_c^2 - 0{,}0219 \cdot P_c^3 + 7{,}91 \cdot 10^{-4} \cdot P_c^4 + 978$$

$$H_A(P_c)=-3{,}18 \cdot P_c + 0{,}226 \cdot P_c^2 - 4{,}26 \cdot 10^{-3} \cdot P_c^3 + 2{,}56 \cdot 10^{-5} \cdot P_c^4 + 63$$

$$T_p(P_c)=-834 \cdot P_c + 55{,}5 \cdot P_c^2 - 1{,}89 \cdot P_c^3 + 0{,}035 \cdot P_c^4 + 3{,}34 \cdot 10^{-4} \cdot P_c^5 + 1{,}29 \cdot 10^{-6} \cdot P_c^6 + 5338$$

$$C_k(P_c)=22{,}1 \cdot P_c - 1{,}29 \cdot P_c^2 + 0{,}0378 \cdot P_c^3 + 5{,}78 \cdot 10^{-4} \cdot P_c^4 + 4{,}29 \cdot 10^{-6} \cdot P_c^5 - 1{,}15 \cdot 10^{-8} \cdot P_c^6 - 147$$

$$f_c(P_c)=-0{,}34 \cdot P_c + 6{,}13 \cdot 10^{-3} \cdot P_c^2 - 3{,}82 \cdot 10^{-5} \cdot P_c^3 + 6.13$$

$$I_c(P_c)=-9{,}75 \cdot 10^{-3} \cdot P_c + 5{,}34 \cdot 10^{-4} \cdot P_c^2 - 1{,}53 \cdot 10^{-5} \cdot P_c^3 + 2{,}41 \cdot 10^{-7} \cdot P_c^4$$
$$-1{,}99 \cdot 10^{-9} \cdot P_c^5 + 6{,}76 \cdot 10^{-12} \cdot P_c^6 + 0.0728$$

$$f_w(P_c)=-0{,}232 \cdot P_c - 1{,}28 \cdot P_c^2 + 3{,}83 \cdot 10^{-4} \cdot P_c^3 - 1{,}09 \cdot 10^{-5} \cdot P_c^4$$
$$+1{,}27 \cdot 10^{-7} \cdot P_c^5 - 5{,}5 \cdot 10^{-10} \cdot P_c^6 + 4{,}25$$

$$I_w(P_c)=-0{,}232 \cdot P_c - 6{,}42 \cdot 10^{-6} \cdot P_c^2 - 3{,}62 \cdot 10^{-8} \cdot P_c^3 + 46$$

$$Kg_{45}(P_c)=-6.41 \cdot 10^{-4} \cdot P_c + 0{,}296 \cdot P_c^2 - 5{,}23 \cdot 10^{-3} \cdot P_c^3 + 3{,}54 \cdot 10^{-4} \cdot P_c^4 + 46$$

Only two variables occur simultaneously in each equation due to the fact, that the equations are result of approximation of flat function. The total number of equations is 12. The system of equations works in such intervals (2).

P_c = 20..66%	**T_p = 330..480**	
f_r = 10..63	**C_k = 0..16**	
E_p = 10..375	**f_c = 1..2**	
ε_c = 425..1500	**I_c = 2..18**	**(2)**
ε = 20..205	**f_w = 0,3..0,8**	
H_A = 60..100	**I_w = 18..34**	
	Kg_{45} = 0..80	

There are many ways to use this system in practice. One of the most important is the task of finding the best combination of properties of the material and its structure from the standpoint of a somewhat parameter that would reflect needed set of properties, i.e. quality of material. It is possible to calculate all the characteristics of the material for given Pc or any other characteristic of the material (inside mentioned above range). However, on practice it is often necessary to obtain a material with a set of target properties. In this case, it is necessary to use weights for each parameter. There are simple examples of using weights to obtain a material with preset properties.

Let's introduce the quality index of the material Q (quality), which is an integrated assessment of all given set of characteristics [Bozek 2021].

It should notice that for adequate usage of Q all functions should be reduced to same interval of variation, for example 0..1, otherwise the influence of some functions will prevail over others. Or introduce weight coefficient in order to increase impact of a specific function. The presence of a sign "-" before one of the parameters indicates that its growth leads to a decrease in the quality of the material. Thus, the greater value of wear Ic is reached, the worse quality of the material is.

$$Q(P_c)=Q_{fm} \cdot (P_c) + Q_{fr} \cdot (P_c) + Q_{ab} \cdot (P_c)$$

$$Q_{fm}(P_c)=f_r(P_c)+E_p(P_c)+\varepsilon_p(P_c)+\varepsilon(P_c)+H_A(P_c)+T_p(P_c)+C_k(P_c)$$

$$Q_{fr}(P_c)=f_c(P_c)+I_c(P_c)+f_w(P_c)+I_w(P_c)$$

$$Q_{ab}(P_c)=Kg_{45}(P_c)$$

$$q(P_c)=I_c(P_c)+2H_A(P_c)$$

where Q – integral quality parameter; Qfm – quality parameter, which takes into account only physico-mechanical characteristics; Qfr – quality parameter, which takes into account only tribological characteristics; Qab – quality parameter, which takes into account only characteristics of gas-abrasive wearing; q – quality parameter, which takes into account only wearing without lubrication and hardness, but hardness impact is more important. It is possible to find the maximum values of quality indicators by traditional methods of finding extrema, and find the appropriate value of Pc, and then all the other parameters. To clarity the situation, not only the points of maximum quality are presented, but also all the curves of quality parameters in the entire range of Pc (Fig. 3).

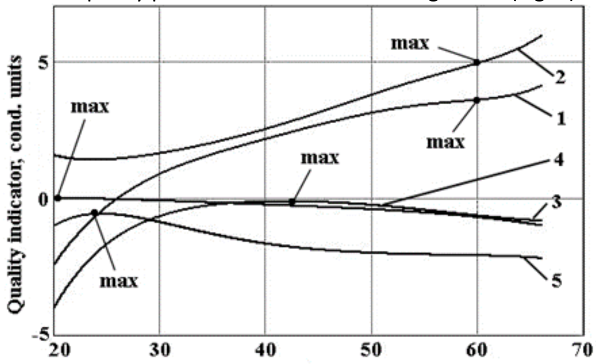

Figure 3. Graphical representation of quality parameters curves
1 – integral quality parameter;
2 – quality parameter, which takes into account only physico-mechanical characteristics;
3 – quality parameter, which takes into account only tribological characteristics;
4 – quality parameter, which takes into account only characteristics of gas-abrasive wearing;
5 – quality parameter, which takes into account only wearing without lubrication and hardness, but hardness impact is more important.

Fig. 3 clearly shows that the maximum values of quality parameters depending on the operating conditions correspond to different contents of rigid blocks Pc, and, consequently, to their different molecular structure of the studied polyurethanes. So, for operating conditions where it is necessary to provide a high complex of strength characteristics, it is rational to use BPU on the basis of OOTMG1000 with the maximum maintenance of hard blocks (Pc > 50%). For usage as wear-resistant materials, the synthesis of polyurethanes with the formation of cluster of hard blocks in the range of 30-50% is recommended. It is clear that the structure of the composition varies significantly depending on the parameters by which the evaluation is performed, and it can be used when developing new materials.

5 CONCLUSIONS

It has been established that the development of recommendations for a reasonable choice of linear block-copolyurethanes, is mainly intuitive. It is proposed to assess the relationship between structure and properties of investigated materials using quality parameters. Using polyurethane block-copolymers based on OOTMG1000, a mathematical model has been developed that clearly describes the relationship of the morphological parameter of the structural organization Pc with strength, deformation, thermophysical and tribological characteristics. Thus, polyurethane block copolymers based on OOTMG1000 with Pc1 = 30% can be successfully used in industrial conditions, where, first of all, it is necessary to provide high deformation characteristics, and BPU with Pc> 50% provide sufficient frame rigidity under conditions of high loading and temperature influence. The obtained dependencies allow to predict the behaviour of investigated materials under different operating conditions, taking into account the quality parameters of the material. Also, it can be a scientific basis for the development of a branded range of BPUs with increased wear and abrasive resistance for various industries.

ACKNOWLEDGMENTS

This work was supported by the project VEGA 1/0226/21 of Scientific Grant Agency of the Ministry of education, science, research and sport of the Slovak Republic and the SAS.

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