Polyurethane Foams with the Contribution of Products of Hydroxyalkylation of *N,N'*-Bis(2-hydroxyalkyl)oxamides with Alkylene Carbonates—Obtaining and Properties

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ABSTRACT: In the reactions of *N,N'*-bis(2-hydroxyethyl)oxamide (BHEOD) with an excess of ethylene carbonate (EC) and *N,N'*-bis(2-hydroxypropyl)oxamide (BHPOD) with an excess of propylene carbonate (PC), the hydroxyethoxy and hydroxypropoxy derivatives of oxamide (OD) were obtained, respectively, distinguished by an increased thermal stability. First time, these derivatives were used as polyol components to obtain foamed polyurethane plastics with the contribution of 4,4'-diisocyanate diphenylmethane (MDI). The rigid polyurethane foams of a slight water uptake, good stability of dimensions, enhanced thermal stability, and compression strength were obtained. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 124: 755–764, 2012

Key words: *N,N'*-bis(2-hydroxyethyl)oxamide; *N,N'*-bis(2-hydroxypropyl)oxamide; hydroxyalkoxylation; polyurethane foams; thermal stability

INTRODUCTION

During the reaction of parabanic acid (I) with alkylene carbonates (ACs), an acid ring opening and formation of linear oligomers—estroamideimidetetraols (II) occur which are characterized by an increased thermal stability (maximum decomposition temperature of 240–260°C)[1,2]:


$$(1)$$
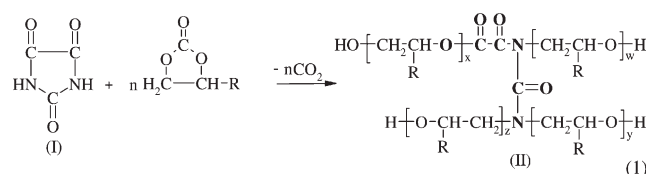
where: R = H-, CH₃-, $n = x + y + z + w$.

The reason for the increased thermal stability is not related to the presence of the above-mentioned acid's ring but the product's structure, that is, oxamidester and carbamide groups connected by an imide bond, is responsible for that.[1,2]

The reaction products of oxamic acid (III) with ACs show the increased thermal stability; their maximum decomposition temperature is 260–315°C.[3,4]

$$(2)$$
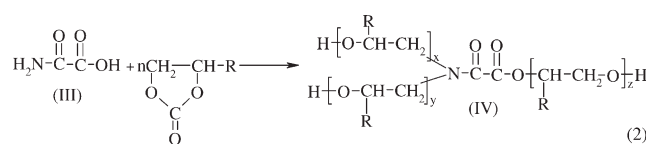
where: R = H—, CH₃—, $x + y + z = 9, 12, 15$.

Similarly, hydroxyalkoxy derivatives of urea (VI), obtained in the reactions of urea (V, $o = p = 0$) or *N,N'*-bis(2-hydroxyalkyl)urea (V, $o = p = 1$) with an excess of ACs, also demonstrate the increased thermal stability (temperature of maximum decomposition 240–380°C).[5–7]

$$(3)$$
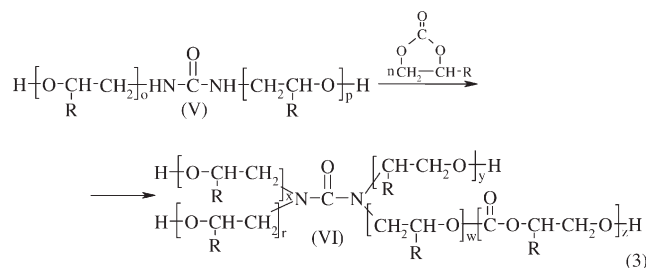
where: R = H—, CH₃—, $x, z, w \geq 0$; $o, p = 0$ or 1.

Hydroxyalkoxy derivatives of parabanic acid, oxamic acid, and urea proved to be proper polyol

components for obtaining rigid foamed polyurethane plastics.[8]

In particular, the foams obtained with contribution of hydroxypropoxy derivatives of parabanic acid and oxamic acid are distinguished by good physical properties as well as the increased thermal stability.[5,6,9–11]

In the structure of parabanic acid one can also distinguish a structural fragment of oxamide (OD); the reaction products of OD and N,N'-bis(2-hydroxyalkyl)oxamides (BHAOD) with ACs are also characterized by the increased thermal stability.[12–14] Use of the hydroxyalkylation products of OD with ACs as polyol components resulted in obtaining rigid foamed polyurethane plastics,[14,15] of the properties similar to the foams obtained with the contribution of hydroxyalkyl derivatives of parabanic acid and oxamic acid. Similarly as in the case of hydroxypropoxy derivatives of the above-mentioned compounds, also with the contribution of hydroxypropoxy derivatives of OD the obtained foams have better physical properties (water uptake, stability of dimensions) and an enhanced thermal stability.[14,15]

It should be emphasized that the polyurethane foams obtained with contribution of hydroxyalkyl urea derivatives obtained with N,N'-bis(2-hydroxyalkyl)ureas and ACs have improved properties compared to the foams obtained with contribution of urea and ACs reaction products.[5,11] A similar dependence was expected in the case of hydroxyalkyl derivatives OD and BHAOD.

In these studies, N,N'-bis(2-hydroxyethyl)oxamide (BHEOD) and N,N'-bis(2-hydroxypropyl)oxamide (BHPOD) were subject to the reactions with a large excess of ethylene carbonate (EC) and propylene carbonate (PC), respectively. The resulting derivatives were used as polyol components for obtaining foamed polyurethane plastics, selected properties of such plastics were tested and then compared to the properties of the foams obtained with the contribution of the hydroxyalkylation products with ACs of OD itself.

EXPERIMENTAL

Syntheses

Synthesis of BHEOD and BHPOD

BHEOD was obtained according to Ref. 16 and BHPOD obtained according to Ref. 13.

Reactions of BHEOD and BHPOD with excess of EC and PC

In a 250-cm^3 three-necked round bottom flask 26.4 g (0.15 mol) BHEOD or 25.5 g (0.125 mol) BHPOD the appropriate amount of EC or PC (pure, Fluka, Swit-

zerland), were placed to reach the molar ratio of reagents of 1 : 8 and 1 : 12, and 1.86 g potassium carbonate (12.42 g/mol BHEOD, 0.09 mol/mol BHEOD) in the case of EC and 1.68 g diazabicyclo[2.2.2]octane (DABCO) (13.44 g/mol BHPOD, 0.12 mol/mol BHPOD) in the case of PC was added. The reaction mixture was protected from moisture with tube filled with magnesium sulfate mounted on top of reflux condenser and stirred mechanically at 160 or 180°C to dissolve BHAOD in AC. Progress of reaction was followed by determination of unreacted AC.[17]

Foam preparation

Attempts of foaming the modified polyols were carried out in small 250-cm^3 test cups at room temperature. To 5 g of a polyol, 0.1 g of surfactant (Silicon 5340, Houdry Hülls), 0.5–2.0 wt % of triethylamine (TEA) catalyst (pure, Avocado, Germany), and 2 wt % of water were added. After careful mixing of the components, a preweighed amount of 4,4'-diisocyanate diphenylmethane (pure, Merck, Germany) was added, calculated as described in Ref. 18. The amounts of diisocyanate and water were adjusted to maintain OH : NCO molar ratio varying from of 1 : 1.5 to 1 : 2.6. Each composition was vigorously mixed until it started to cream (see Table III). The samples for testing were cut out from the foams thus obtained after ~ 48 h.

Analytical methods

^{1}H-NMR spectra of products were recorded with 500 MHz spectrometer (Bruker, Germany) in deuterated dimethyl sulfoxide (d_6-DMSO), and hexamethyldisiloxane (HMDS) reference.

Chromatographic analysis of by-products, that is, ethylene glycol (EG) and products of its consecutive reactions with EC (diethylene glycol (DEG), triethylene glycol (TRIEG), tetraethylene glycol (TETRAEG)) and propylene glycol (PG) and products of its consecutive reactions with PC (dipropylene glycols (DPG), tripropylene glycols (TRIPG)), and oxazolidinones—N-(2-hydroxyethyl)oxazolidinone (OXON)[19] and 3-(2-hydroxypropyl)-5-methyloxazolidinone (IPOX) were performed with gas chromatograph HP 4890A (Hewlett–Packard, Ringoes, NJ) with FID detector and HP1 30 m × 0.53 mm column packed with crosslinked methylsiloxane film of 1.5 μm thickness. Initial temperature was 50°C, heating rate: 20 °C/min, end temperature: 220°C, time of heating at 220°C: 6 min, loader temperature: 250°C, detector temperature: 300°C. The samples were dissolved in methanol (0.01M). Internal reference was cyclohexanone. Percentage of diols

and polyols were calculated according to calibration curves as described in Ref. 1.

Thermal analyses (DTG and TG) of hydroxyalkoxy derivatives of BHAOD were performed in ceramic crucible, at temperature range of 20–600°C, on ~ 2 mg sample, under a nitrogen atmosphere with Termowaga TGA/DSC 1 derivatograph, Mettler.

The following properties of hydroxyalkoxy derivatives of BHAOD have been determined: pycnometer density,[20] refractive index, Höppler viscosity,[21] and surface tension by ring detach method.[22] All measurements were made in temperature range 20–80°C.

The following properties of foams were determined: apparent density,[23] water uptake,[24] stability of dimension,[25] glass transition temperature (by DSC), thermal stability as the weight loss after heating at 150, 175, and 200°C for a month, and the compressive strength.[26]

The differential scanning calorimetry (DSC) measurements of foams were made using a DSC822^e Mettler Toledo instrument, in 20–200°C temperature range and 10 deg/min heating rate under nitrogen atmosphere. The results were recorded as heat flow in [W/g] versus temperature.

The microscopic observation of foams was performed at optical microscope Nikon Eclipse LV100 POL, camera Digital Sight DS-5Mc, objective 2,5 or 5 × L Plan. Microscopy measurements were performed in the Biophysical Laboratory of the Department of Physic of Rzeszow University of Technology. The Laboratory has been constructed in the frame of UE Polish Integrated Regional Operation Programme.

RESULTS AND DISCUSSION

In the reactions of BHAOD with a 6 and 10M excess of EC or PC at temperature of 160 or 180°C, respectively, in the presence of potassium carbonate or DABCO as a catalyst (Table I), the following hydroxyalkoxy derivatives of OD of a general formula (VII) were obtained:
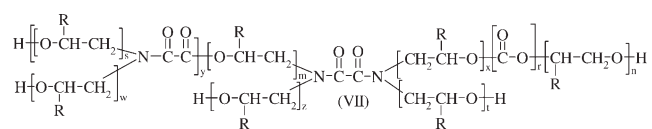
where: R = CH$_3$—, H—, $m \geq 1$, x, y, $r \geq 0$.

The structure of the obtained products was proposed on the basis of an instrumental analysis (spectra ^{1}H-NMR and IR) as described in the study.[14] The reactions of BHAOD with EC and PC proceed mainly with the release of carbon dioxide while only the partial build-up of the carbonate groups into the product's structure (VII, $r \geq 0$) occur. In the course of the reaction of BHAOD with ACs, dimerization of

TABLE I
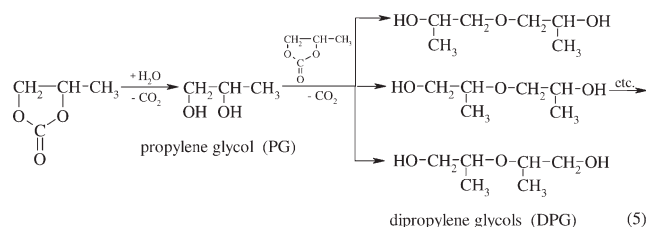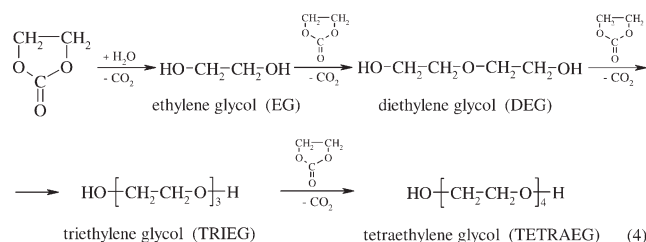**Reaction Conditions of BHAOD with ACs and Percentage of By-Products**

Run	Kind of ACs	Initial molar ratio	Amount of catalyst [mole/mole BHAOD]	Temp. (°C)	Time of reaction (h)	Molar ratio BHAOD : x in post-reaction mixture (from mass balance)	Percentage of oxazolidinone in post-reaction mixtures (wt %)	Percentage of glycols in post-reaction mixtures (wt %)			
								AG^a	DAG^a	TRIAG^a	TETRAEG
1	EC	1 : 6	0.09 K$_2$CO$_3$	160	6	1 : 5.73	4.13	0	0	0	1.9
2		1 : 10	0.09 K$_2$CO$_3$	160	14	1 : 9.96	2.66	0	0	0	0
3	PC	1 : 6	0.12 DABCO	180	8	1 : 4.57	1.19	0	7.56	1.23	–
4		1 : 10	0.12 DABCO	180	12	1 : 7.66	1.80	0	15.38	7.92	–

x, oxyalkylene unit.
^{a} Means E or P for EC and PC, respectively.

hydroxyalkoxy derivatives of OD takes place and in the structure of such obtained products, apart from OD groups, also oxamidester groups (VII, $y \geq 0$) appear. However, the contribution of such dimerization is insignificant which is demonstrated by a low content of oxazolidinones in the composition of the obtained products (Table I). Oxazolidinones are formed in the reaction of 2-aminoethanol or 1-aminopropane-2-ol, as released during the dimerization, with EC (*N*-(2-hydroxyethyl)oxazolidinone, OXON) and PC (3-(2-hydroxypropyl)-5-methyloxazolidinone, IPOX) respectively.[12–14] The content of OXON is maximum 4.2 wt % and it decreases with the increase of an excess of EC used to react with BHEOD (Table I). However, the content of IPOX increases together with a growth of the excess of PC but is not higher than 1.8 wt % (Table I). Different values of OXON and IPOX determined in the products of BHEOD with EC reaction and BHPOD with PC reaction, respectively, follow from different dimerization degree of hyroxyalkyl OD derivatives. Hydroxyethyl groups make less space hindrance than hydroxypropyl ones and therefore hyroxyethyl OD derivatives undergo condensation easier what explain bigger OXON contents in the products of BHEOD and EC reaction.

It appears on the basis of gas chromatography testing that hydroxyethoxy derivatives of OD contain in their composition a by-product TETRAEG, formed in the reaction of EC with water and during the consecutive reactions of the formed EG with EC. The content of glycol however does not exceed 2 wt %. (Table I). Contribution of poly(propylene glycols) is distinctly higher and reaches 23 wt % (Table I).

ethylene glycol (EG)   diethylene glycol (DEG)

triethylene glycol (TRIEG)   tetraethylene glycol (TETRAEG)   (4)

propylene glycol (PG)

dipropylene glycols (DPG)   (5)

EC is more reactive than PC and the reactions of BHEOD with EC run shorter, because reactions of primary hydroxyl groups with secondary amine groups run easier. In the case of reactions with PC, reaction of secondary hydroxyl groups with second-

ary amine groups run more difficult. Duration of the reactions is much longer, and PG forming in the reaction of water with PC, reacts forming consecutive poly(propylene glycols). Therefore, much bigger contribution of poly(propylene glycols) in the reaction of BHPOD with PC products compares with the contribution of poly(ethylene glycols) in products of the reaction of BHEOD with EC.

As it follows from our earlier investigations, too much quantity of low molecular substances (polyglycols) can worsen properties of polyurethane foams, that is, they have not abrasion resistance and show worse mechanical properties. Despite the presence of big quantity of poly(propylene glycols), the foams obtained with contribution of hydroxypropyl OD derivatives show much better properties compared to the properties of the foams obtained with contribution of hydroxyethyl OD derivatives, in which the influence of glycols can be neglected.

Thermogravimetric testing demonstrated an enhanced thermal stability of the obtained hydroxyalkoxy derivatives of OD. In the DTG curve of the reaction, products of BHEOD with EC excess two peaks are noticeable. The first one at 250°C comes from a decomposition of the carbonate groups,[27] while the second one at 350°C results from a decomposition of the OD and oxamidester[3] groups (Table II). Similarly, two peaks are noticeable in the DTG curve of the reaction product BHPOD with a 6*M* excess of PC, but the maximum occurs at a slightly lower temperature of 220(240) and 300°C (Table II).

Certain physical properties of the obtained hydroxyalkoxy derivatives of OD were investigated like: density, surface tension, viscosity, and light refractive index (Fig. 1) versus temperature function in the range of 20–80°C. It was found that together with a temperature growth, values of the light refractive index, density, and surface tension decrease linearly and values of the viscosity exponentially (Fig. 1). So, they are subject to changes in a way typical for polyols used conventionally for the preparation of polyurethane foams.[28]

Hydroxyalkoxy derivatives of OD were used as polyol components to obtain polyurethane foams with the contribution of MDI as isocyanate component. It has been predetermined during the testing that the optimal foaming is with 2 wt % of water (Table III). The amount of water is selected experimentally. Using too little water amount during foaming, foams of a low foaming degree are obtained, whereas application of too much water amount results in foams possessing irregular pores. The foams of the best properties were obtained with the contribution of the product in which the initial molar ratio of BHAOD to AC is 1 : 6 (BEOD6, BPOD6) for the isocyanate factor 1.5 (Table III, comp. 1 and 8), while in the case of using polyol

TABLE II
Thermal Stability of Reaction Products of BHAOD with ACs

Entry	Kind of AC	Initial molar ratio, BHAOD : AC	$T_{5\%}$ (°C)	$T_{10\%}$ (°C)	$T_{20\%}$ (°C)	$T_{50\%}$ (°C)	Temperature of max. decomposition (°C)
1.	EC	1 : 6	160	180	220	260	250 and 360
2.		1 : 10	90	160	190	240	220 and 350
3.	PC	1 : 6	135	160	180	255	220 and 300
4.		1 : 10	130	160	180	250	240 and 300

$T_{x\%}$, temperature at which the weight loss is $x\%$.

obtained with a 10M excess of EC or PC (BEOD10, BPOD10), the isocyanate factor amounting 2.3 proved to be adequate (Table III, comp. 4 and 12).

The creaming time of the foamed compositions was within 13–36 s or shorter when to obtain the foamed compositions, the derivatives prepared at a 10M EC excess and 6M PC excess were applied (Table III). If polyols with BHPOD and PC were used, the growth time of the foams was within 30–59 s (Table III, comp. 5–13). It was shorter when polyols with BHEOD were used and was in the range of 7–18 s (Table III, comp. 1–4). Drying time of the foams obtained from polyols with BHEOD and EC was usually short; the foams were almost dry on completion of the growth (Table III), and in the case of

foams obtained with hydroxypropoxy derivatives of OD—after 3–27 s (Table III, comp. 5–13).

The foams obtained with the contribution of hydroxyethyl derivatives of OD expand faster and after expanding are dry, whereas expanding time of the foams obtained with the contribution of hydroxypropyl derivatives is longer, and they became dry during 30 s. That is connected with higher reactivity of primary hydroxyl groups in the reaction with isocyanate, as compared with the reactivity of secondary hydroxyl groups, which takes place during the foam growth.

The following selected properties of polyurethane foams were determined: apparent density, water uptake, dimension stability, thermal stability, and
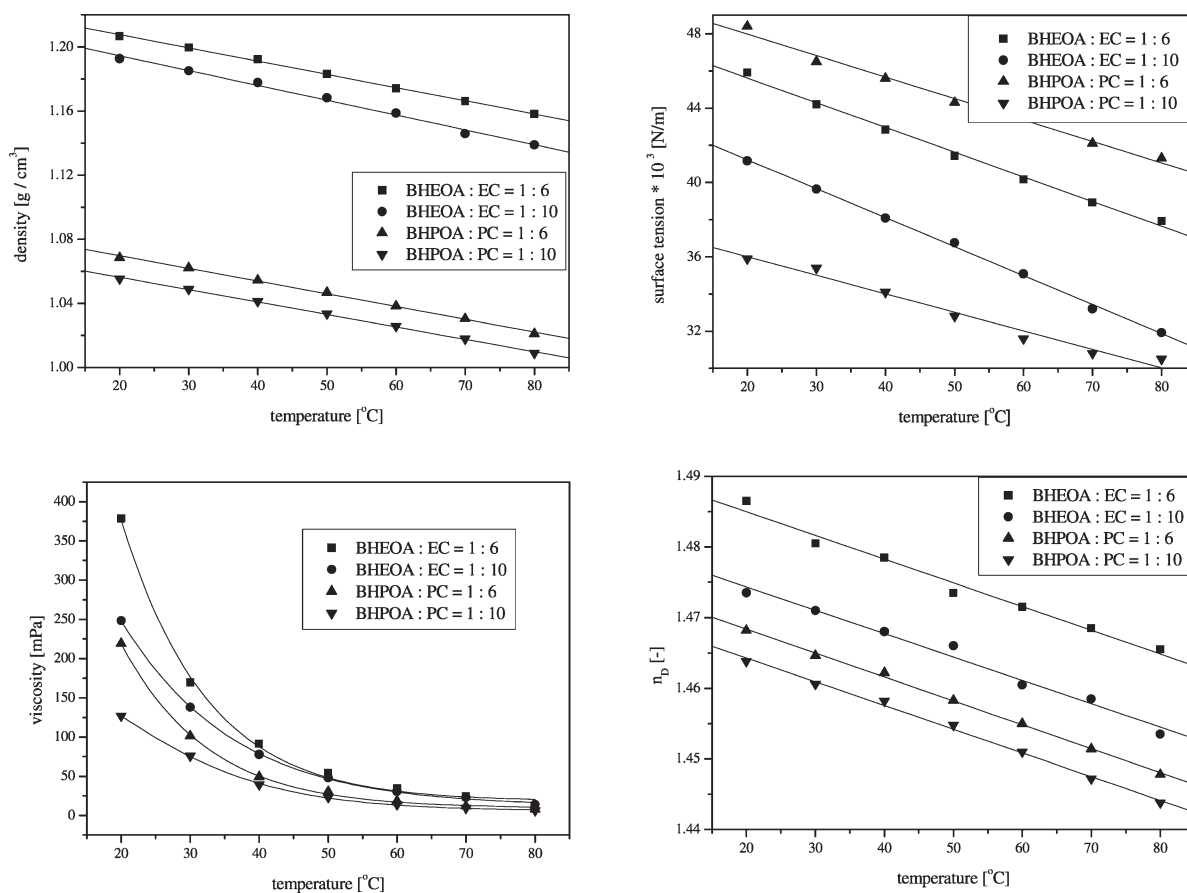

Figure 1 Physical properties of reactions products of BHAOD with EC and PC in function of temperature.

TABLE III
Parameters of Foaming Process

| Initial molar ratio BHAOD : AC in polyol | Kind of AC | Composition No. | Composition (g/100 g of polyol) | | | Molar ratio OH : NCO | Foaming process | | | Properties of foams just prepared |
| | | | isocyanate[a] | Water | Catalyst[b] | | Time (s) | | | |
							Creaming[c]	Expanding[d]	Drying[e]	
1 : 6	EC	1	200	2	0.50	1 : 1.57	36	18	0	Rigid
		2	240	2	0.50	1 : 1.88	26	15	0	Rigid
		3	268	2	0.50	1 : 2.10	25	12	0	Rigid
		4	212	2	0.50	1 : 2.31	17	7	0	Rigid
		5	240	2	1.49	1 : 2.28	23	40	0	Rigid, big pores
1 : 10		6	180	1	1.49	1 : 1.71	13	30	0	Rigid, big pores
1 : 6		7	180	0	1.00	1 : 1.71	15	35	0	Brittle, rigid
		8	160	0	0.50	1 : 1.52	25	35	2	Rigid
		9	140	0	0.00	1 : 1.33	15	42	8	Brittle, rigid
		10	120	0	0.50	1 : 1.58	28	39	27	Rigid
		11	180	0	0.50	1 : 2.37	29	59	15	Rigid
1 : 10	PC	12	180	0	1.00	1 : 2.37	25	43	8	Rigid
		13	200	1	1.00	1 : 2.63	28	47	3	Rigid

[a] 4,4'-diphenylmethane diisocyanate.
[b] Triethylamine.
[c] Time of creaming; the time elapsed from the moment of mixing to the start of volume expansion.
[d] Time of expanding; the time from the start of expansion to the moment of reaching the sample final volume.
[e] Time of drying; the time from reaching by the sample its final volume to the moment of loosing its surface adhesion to powdered substances.

TABLE IV
Properties of Foams

| Comp. no.[a]	Density (kg/m³)	Water Uptake (vol. %)			Linear dimension change after heating at 150°C (linear %)					
		After 5 min	After 3 h	After 24 h	Length		Width		Thickness	
					After 20 h	After 40 h	After 20 h	After 40 h	After 20 h	After 40 h
1.	37.79	3.80	9.39	9.63	0.00	1.17	0.00	0.00	0.39	0.19
4.	30.86	4.92	7.60	10.77	2.15	3.01	1.39	1.74	0.00	0.00
8.	42.8	3.03	3.24	4.94	0.38	0.76	0.85	1.28	1.23	1.23
12.	39.51	2.00	2.56	3.60	0.00	0.62	0.86	0.86	0.43	0.43
13.	38.52	2.20	2.99	3.51	0.58	0.87	0.80	0.80	0.56	0.56

[a] Comp. no. according to Table III.

glass transition temperature. Apparent density of all foams is similar and is in the range of 31–38 kg/m³ (Table IV). The water uptake of all foams increases versus time but after 24 h it does not exceed 11 wt %. A lower water uptake is demonstrated by the foams obtained from polyols with BHPOD—it is 3.6 vol. % (Table IV, comp. 12 and 13). In the case of the polyol foams with BHEOD the lowest water uptake, that is, 9.6 vol. % after 24 h, is specific for the foam obtained with the contribution of BEOD6 (Table IV, comp. 4). Water uptake of the foams depends on the contribution of open pores in their structure. Much more water uptake of foams obtained with the contribution of hydroxyethyl derivatives of OD indicates that the foams contain more open pores.

Observations of the foams under an optical microscope have evidenced that their structure contains mainly closed pores of a regular distribution. In the case of foams obtained with the contribution of polyols with BHEOD, wall thickness of the pores is 15–31 μm and size of the pores is in the range of 0.11–0.23 mm. The foams obtained from hydroxypropoxy derivatives have pores of slightly thicker walls of 31–55 μm and bigger sizes of 0.11–0.28 mm. Furthermore, it was noted that pores of the foams prepared with the contribution of hydroxypropoxy derivatives of OD have sharp-edged shapes while those of the foams obtained with BHPOD and PC are more circular (Fig. 2). The differences in pore structure in the case of foams obtained with contribution of hydroxyethyl and hydroxypropyl OD derivatives result from differences in the structures of the derivatives. The presence of methyl groups in hydroxypropyl OD derivatives chains causes that pores are more circular, larger, and have thicker walls as compared with foams obtained with contribution of hydroxyethyl OD derivatives.

The obtained foams are characterized by good thermal stability of dimensions and change of dimensions usually does not exceed three linear percent. The best thermal stability of dimensions is demonstrated by the foams obtained with BEOD6 and BPOD10 (Table IV, comp. 1 and 12); change of

the dimensions does not exceed 1.2 and 0.9 linear percent, respectively.

The glass transition temperature (TG) of the obtained foamed polyurethane plastics determined by the DSC method is 96–119°C (Table V), therefore these foams can be classified as the rigid ones.[25] The
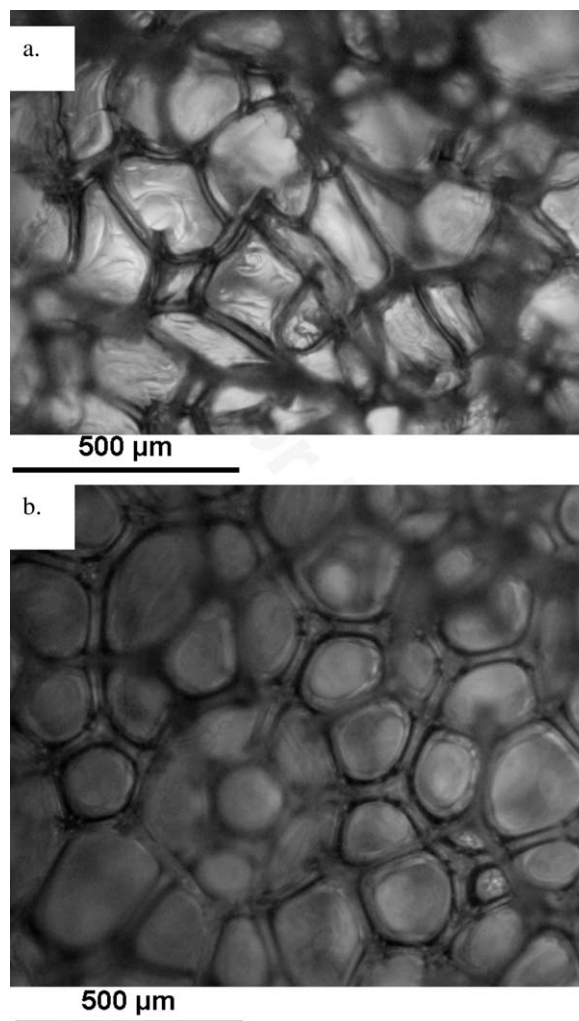
Figure 2 Morphology of foam obtained from polyols obtained from BHAOD and 10M excess of: (a) EC, (b) PC, magnification 25×.

Comp. no[a]	$T_{5\%}$ (°C)	$T_{10\%}$ (°C)	$T_{20\%}$ (°C)	$T_{50\%}$ (°C)	T_{max} (°C)	Glass transition T_g (°C)
1.	200	220	240	330	250	104
4.	210	235	255	330	260 and 310	96
8.	180	220	245	340	260 and 340	105
12.	200	240	270	340	325	119

$T_{x\%}$, temperature at which the weight loss is $x\%$.
[a] Comp. no. according to Table III.

glass transition temperature decreases together with an increase of EC excess used in the reaction with BHEOD, that is, together with an increase of oxyethylene groups in the used polyol component (Table V, comp. 1 and 4). In the case of foams obtained with hydroxypropoxy derivatives of OD, a contrary result was observed (Table V, comp. 8 and 12).

Thermogravimetric analysis confirmed the presumed enhanced thermal stability of the obtained polyurethane foams. A 5% weight loss of the foam occurs not before temperature of approximately 210°C and 180°C for the foams obtained with BHEOD and BHPOD, respectively. The maximum temperature of decomposition is higher for the foams obtained with hydroxypropoxy derivatives of OD and is approximately 325°C (Table V, comp. 12). A slightly lower maximum temperature of the decomposition (260°C) is demonstrated by the foams obtained with BHEOD and EC (Table V, comp. 4).

To investigate a thermal stability of the obtained polyurethane foams they were exposed to the temperatures of 150, 175, and 200°C until constant mass was reached (30 days). After about 20 days it was evidenced that weight losses in the foams were insignificant (Fig. 3). The lowest weight loss is demonstrated in the foams prepared with the contribution of hydroxypropoxy derivatives of OD. The lowest weight losses are presented by the foam prepared with BPOD10; they are 9.5, 25.5, and 37.5 wt % at the temperature of 150, 175, and 200°C, respectively, (Fig. 3, comp. 12). A slightly higher weight loss is exhibited by the foams obtained with BHEOD and EC contribution (Fig. 3, comp. 1 and 4).

The obtained foams as well as their samples heated up for 30 days at temperature of 150 and 175°C were subject to strength testing to determine resistance of the foams.

Two processes take place during temperature exposition of the foams, that is, further crosslinking and degradation. Contribution of individual processes depends on temperature of heating up. At 150°C the main role plays further crosslinking, whereas the higher is temperature of heating up the larger participation of foams decomposition process is observed. Larger degree of crosslinking makes the foams show higher compressive strength.

The foams with BHPOD and PC contribution demonstrate an enhanced compression strength compared to the conventional foams (Table VI, comp. 12). The compression strength increases after the temperature exposure at 150°C. Heating up at a higher temperature causes a decrease of such strength (Table VI).

Comparing properties of the foams obtained with the contribution of hydroxyalkylation products of OD itself and of BHAOD ACs, it is noted that an apparent density of all foams is similar (Table IV).[15] The water uptake in the foams obtained with the contribution of hydroxyethoxy derivatives of OD and BHEOD is similar, too; in the case of foams obtained with BHPOD and PC (Table IV) it is however distinctly lower (almost by a half) as compared to the foams with OD and PC.[15] Dimension stability of the foams obtained with BHAOD and ACs is higher compared to the foams with OD and ACs, while the smallest change of dimensions is shown by the foams obtained with the contribution of BHPOD and PC reaction products. Thermal stability of the foams tested thermogravimetrically and by heating up at the temperature of 150, 175, and 200°C is similar, however it is slightly higher in the case of foams obtained with the contribution of BHAOD and ACs. The compression strength is distinctly higher in the case of foams obtained with BHPOD and PC.

Furthermore, there was compared properties of polyurethane foams obtained with contribution of hydroxyalkyl OD derivatives with the properties foams obtained with contribution of hydroxyalkyl derivatives of urea.[6,11] The experimental conditions, hydroxyl number, and functionality of the polyols are similar. The water uptake is smaller and dimension stability is higher in the foams obtained with the contribution of hydroxyalkoxy derivatives of OD than in foams obtained with contribution of hydroxyalkoxylated derivatives of urea. Comparing thermal stability of the foams obtained with contribution of N,N'-bis(2-hydroxyethyl)urea and EC reaction products with those of the foams obtained with contribution of BHEOD and EC reaction products it should be emphasized that it is similar ($T_{max.}$ = 240–260°C), while thermal stability of the foams obtained with

contribution polyols obtained in the reactions of N,N'-bis(2-hydroxypropyl)urea (BHPU) with PC ($T_{max.} = 260°C$)[11] and BHPOD with PC ($T_{max.} = 255–355°C$) is definitely higher in the case of BHPOD. Compressive strength is higher in the case of the foams obtained with the contribution of hydrox-
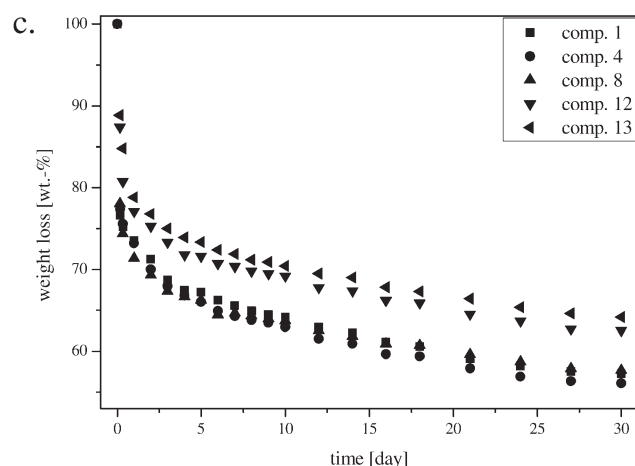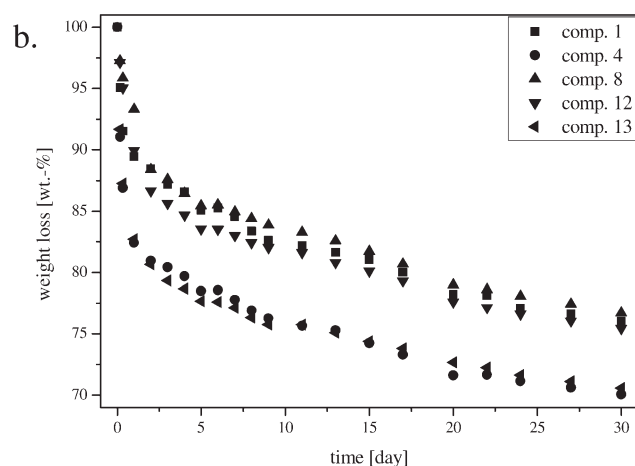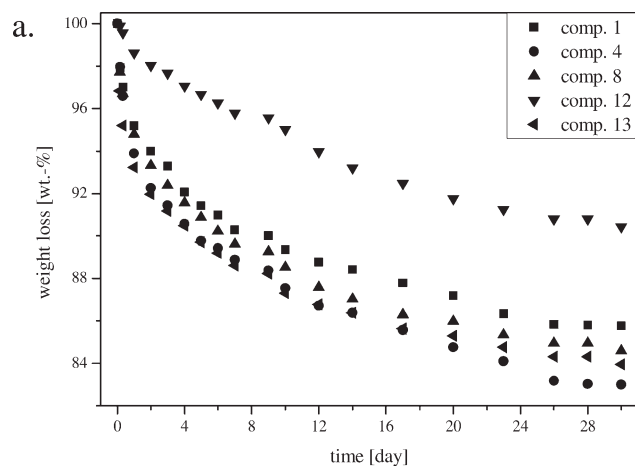
Figure 3 Thermal stability of compositions as the weight loss after heating at (a) 150, (b) 175, and (c) 200°C for a month.

TABLE VI
Compressive Strength of Polyurethane Foams (MPa)

Comp.[a] no.	Compressive strength σ_M (MPa)	Compressive strength in (MPa) after exposition in temperature 150°C	Increase of compressive strength in (%) after exposition in temperature 150°C
1	0.14	0.30	112
4	0.16	0.34	115
8	0.16	0.32	102
12	0.64	0.82	28

[a] Comp. no. according to Table III.

yalkyl OD derivatives, wherein in the case of the foams obtained with the contribution of hydroxypropyl derivatives the differences are definitely higher (for foams from BHPU σ_M = 0.01–0.04 MPa,[11] for foams from BHPOD σ_M = 0.14–0.64 MPa).

CONCLUSIONS

Hydroxyethoxy and hydroxypropoxy derivatives of OD obtained with the contribution of BHEOD and N,N'-bis(2-hydroxypropyl)oxamide and excess of EC or PC are characterized by an enhanced thermal stability. They demonstrate properties of typical polyols used in obtaining polyurethane foams. The foams obtained with the contribution of hydroxyethoxy derivatives of OD show good stability of dimensions and an increased thermal stability. Foams of the best properties were obtained with the contribution of hydroxypropoxy derivatives. They are featured by an insignificant water uptake, good stability of dimensions, increased thermal stability, and compression strength, especially when polyol with a 10M excess of PC was used. The foams obtained with the reaction products of BHAOD and excess of ACs are characterized by even better properties than the foams obtained with the contribution of hydroxyalkylation products with OD ACs.

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