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New Foamed Plastic Based on Polyetherols Obtained from 6-Aminouracil, Ethylene Carbonate, and Propylene Oxide

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ABSTRACT: Polyetherols with 1,3-pyrimidine ring including both oxyethylene and oxypropylene groups were obtained in reactions of 6-aminouracil with ethylene carbonate and propylene oxide. The structure of the products was analyzed by spectral methods. Some physical properties of polyetherols were investigated. The polyetherols were used as polyol components to obtain polyurethane foams. Some properties of the foams such as apparent density, absorption of water, linear dimensions stability, thermal resistance, and compression strength were investigated. The foams obtained show an improved thermal stability. © 2012 Wiley Periodicals, Inc. J. Appl. Polym. Sci. 000: 000–000, 2012

KEYWORDS: heteroatom-containing polymers; polyurethanes; thermal properties; mechanical properties

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INTRODUCTION

It has been demonstrated that polyetherols with perhydro-1,3,5-triazine,[1–3] 1,3,5-triazine,[4–8] and purine[9,10] rings can be used as polyol components to obtain polyurethane foams with improved thermal stability. The foams with improved thermal stability can also be synthesized by the introduction of 1,3-pyrimidine ring.[11,12] Polyetherols with 1,3-pyrimidine ring have been obtained in reactions of 6-aminouracil (6-AU) with excess of oxiranes[13] or alkylene carbonates.[14,15] The foams synthesized from polyetherols obtained in reaction of 6-AU with excess of oxiranes (especially propylene oxide, PO) show a very good thermal resistance (the mass loss was equal to 13% at 150°C and 37.5% at 200°C) and good compression strength (0.24 MPa at 10% deformation strain).[11] The foams synthesized from polyetherols obtained in reaction of 6-AU with excess of alkylene carbonates (especially ethylene carbonate, EC) show lower mass loss than these mentioned earlier (6.9% at 150°C and 33.1% at 200°C), but the compression strength is lower (0.14 MPa at 10% deformation strain).[12] In this work, polyetherols containing both oxyethylene and oxypropylene groups in their structure were synthesized and used for the synthesis of polyurethane foams in order to achieve a combination of the advantages of those two cited class of compounds.

EXPERIMENTAL

Synthesis of Polyetherols

Reactions of 6-AU with EC. To a round-bottomed flask (250 cm^3) equipped with a reflux condenser, thermometer, and stirrer, 12.7 g (0.1 mol) of 6-AU (pure; Sigma-Aldrich, Steinheim, Germany), 44.0 g (0.5 mol) or 52.8 g (0.6 mol) of ethylene carbonate (EC; pure, Fluka, Switzerland), and 0.4 g of potassium carbonate (pure, PPH POCh, Gliwice, Poland) as a catalyst were introduced. The mixtures were heated to 140°C with continuous stirring. The reaction course has been controlled by determining the amount of unreacted EC. The reaction was conducted for 18.5 h (including dissolving time of 6-AU—3 h) when 0.5 mol of EC was used and 22 h (including dissolving time of 6-AU—2.8 h) when 0.6 mol of EC was used. The resin-like products were obtained in these reactions.

Reactions of Hydroxyethyl Derivatives of 6-AU with PO. To a 250-cm^3 autoclave reactor equipped with a stirrer 0.1 mol product of reaction of 6-AU with 5 mol of EC (34.7 g) or 6 mol of EC (39.1 g), preweighed amount of PO (pure; Fluka, Buchs, Switzerland; 5.8 g (0.1 mol), 11.6 g (0.2 mol), 17.4 g (0.3 mol), 23.2 g (0.4 mol), 29.0 g (0.5 mol), 34.8 g (0.6 mol), 40.6 g (0.7 mol), and 1 cm^3 of triethylamine (TEA; pure; Fluka, Buchs, Switzerland) as a catalyst were introduced. The mixture was heated up to temperature 50°C and vigorously stirred in order to achieve a homogeneous system. After careful mixing of the components, the mixture was kept at temperature 50–70°C for 12.5–38 h. The end of the reaction was established by determining the epoxy number of the reaction mixture.

Polyurethane Foams Preparation

To a small 250-cm^3 test cup, 10 g of polyetherol, 0.8 wt % of Silicon 5340 (Houdry Hülls) as surfactant, 0−4 wt % of water,

Table I. The Influence of Composition on Foaming Process

		Composition^a (g/100 g of polyetherol)				Foaming process^b			
Polyetherol 6-AU : EC : PO	Composition number	Isocyanate	TEA	Water	Molar ratio OH : NCO	Time of creaming (s)	Time of expanding (s)	Time of drying (s)	Characteristics of freshly prepared foams
1 : 5 : 3	1	144	2.2	4	1.2	15	22	2	Small, regular pores, fragile
	2	112	1.9	2	1.0	18	15	2	Rigid
1 : 5 : 7	3	112	1.3	2	1.4	19	60	19	Small shrink, rigid
	4	132	1.3	2	1.65	17	50	10	Small shrink, small, regular pores, rigid
1 : 6 : 4	5	112	1.3	2	1.2	16	17	1	Small, regular pores, rigid
1 : 6 : 6	6	132	1.3	2	1.6	16	33	1	Small shrink, small, regular pores, rigid

^{a}Amount of surfactant: 0.8 g/100 g of polyetherol, ^{b}Mixing time of composition: 10 s.

and 1.3−2.2 wt % of TEA catalyst were added in relation to polyetherol. After careful mixing of the components, a preweighed amount of 4,4′-diphenylmethane diisocyanate [mixture of di- and triisocyanates (30%) for synthesis; Merck-Schuchardt, Hohenbrunn, Germany] was added. The mixture was vigorously stirred until it started creaming. The amount of diisocyanate was adjusted to OH : NCO molar ratio of 1.0−1.65 (Table I). From the foams thus obtained, samples for testing were cut out after ∼ 48 h.

Analytical Methods

The amount of unreacted EC was determined by reaction with barium hydroxide.[16] The epoxy number was determined by titration, a sample with a dioxane solution of hydrochloric acid.[17] The infrared spectra were recorded on a Fourier transform IR PARAGON 1000 spectrometer (Perkin-Elmer Corp., Norwalk, CT). The samples for IR analysis were prepared as capillary films. The ^{1}H-NMR spectra of products were recorded with a 500 MHz Brucker UltraShield spectrometer. The solvent was d_6-DMSO with HMDSO internal standard. MALDI–ToF (matrix-assisted laser desorption ionization time of flight) spectra were obtained on Voyager-Elite Perseptive Biosystems (USA) mass spectrometer working at linear mode with delayed ion extraction, equipped with nitrogen laser working at 337 nm. The method of laser desorption from matrix was used with 2,5-dihydroxybenzoic or α-cyanocinnamonic acid in THF at 10 mg/cm^3 concentration. The samples were diluted with methanol to 1 mg/cm^3, followed by the addition of 10 mg/cm^3 NaI in acetone. Therefore, in some cases, the molecular ion masses were increased by the mass of Na^+ and CH_3OH.

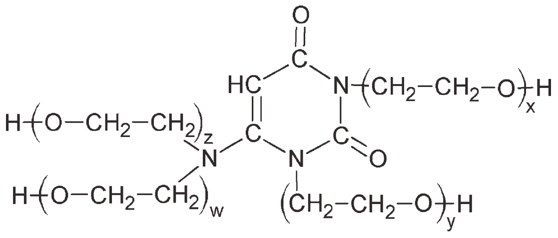
Some other properties of the polyetherols were evaluated, such as refractive index, density (pycnometrically), viscosity (Höpler viscometer),[18] and surface tension, by using the torsion balance method.[19]

The following properties of foams were determined: apparent density,[20] water absorbing capacity,[21] stability of dimensions,[22] and thermal stability as the mass loss after heating at 150, 175, and 200°C for month and compression strength.[23] The thermal analysis of products was conducted with thermogravimetric analyzer TGA/DSC 1 (Mettler Toledo, Spain); recording conditions were as follows: sample weight 1–2 mg, temperature range 20–600°C, recording time 60 min, and nitrogen atmosphere. Thermal investigations[24] of the foams were made using differential calorimeter DSC-type 822^e with the software Star^e system (Mettler Toledo, Spain). Recording conditions were sample weight 10–20 mg, temperature range 0–200°C, heating velocity 10–20°C/min, flow rate 30 cm^3/min, and nitrogen atmosphere.

RESULTS AND DISCUSSION

Initially, operating conditions of a reaction were determined. As the starting step of synthesis of polyetherols containing both oxyethylene and oxypropylene groups, reactions of 6-AU with carbonates were chosen, because 6-AU reacts faster with carbonates than with oxiranes[13–15] and the reaction with carbonates does not require solvent. Because of higher reactivity of EC in comparison with PC,[15,25,26] oxyethylene groups were introduced first into a 6-AU ring. Obtained resin products react with PO in the presence of TEA as a catalyst (without solvent). Thus, oxypropylene groups were introduced using PO. It was also useful due to relatively low temperature of the reaction (60–70°C, while reaction with PC requires 180°C).

Reactions of 6-AU with EC were conducted at temperature 140°C, using starting molar ratio 1 : 5 and 1 : 6 and using potassium carbonate as a catalyst (4 g/mol 6-AU). The IR and ^{1}H-NMR spectra of obtained compounds are practically the same as spectra of hydroxyethyl derivatives of 6-AU described in previous studies.[14] Thus the structure of the products can be described by formula (I):



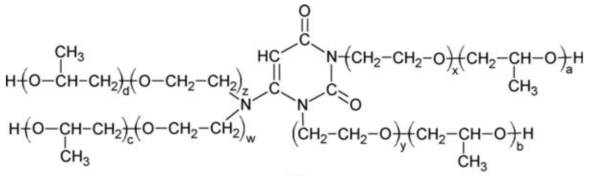
(I)

where $w + x + y + z \leq 5$ or 6.

Table II. Elemental Analysis of Polyetherols

Polyetherol 6-AU : EC : PO	Elemental analysis C (wt %) Calc.	Found	H (wt %) Calc.	Found	N (wt %) Calc.	Found
1 : 5 : 1	50.36	49.47	7.71	7.74	10.36	9.54
1 : 5 : 3	52.96	52.90	8.31	7.86	8.05	8.45
1 : 5 : 5	54.62	54.43	8.69	9.03	6.59	6.23
1 : 5 : 7	55.76	55.98	8.96	9.25	5.57	5.30
1 : 6 : 2	52.06	52.62	8.14	8.44	8.27	7.74
1 : 6 : 4	53.92	52.37	8.57	8.77	6.73	6.14
1 : 6 : 6	55.19	54.96	8.86	9.35	5.68	5.60

Then obtained hydroxyethyl derivatives of 6-AU were subjected to reaction with PO. Syntheses were carried out in autoclave reactor at temperature 50–70°C in the presence of TEA as a catalyst. As a result, polyetherols were obtained for initial molar ratio of reagents equal to 1 : 5 : 1, 1 : 5 : 3, 1 : 5 : 5, 1 : 5 : 7, 1 : 6 : 2, 1 : 6 : 4, and 1 : 6 : 6. Elemental analysis (Table II) confirms composition of polyetherols. Their structure (II) was investigated using IR, ^{1}H-NMR, and MALDI–ToF spectra.



(II)

where $x + y + z + w \leq 5$ and $1 \leq a + b + c + d \leq 7x + y + z + w \leq 6$ and $2 \leq a + b + c + d \leq 6$

In IR spectrum (Figure 1) besides bands of methylene (2930, 2870, 1450, and 1122 cm^{-1}) and hydroxyl groups (3350, 1420, and 1260 cm^{-1}), the stretching and deformation bands of methyl groups were also found (2966 and 1373 cm^{-1}, when number of moles of attached PO rises then intensity of bands rises too), and band of C—O stretching vibrations in secondary alcohols (1080 cm^{-1}) appeared.

Analysis of ^{1}H-NMR spectra (Figure 2) showed that methyl groups are present in products of normal and abnormal addition[27,28] (signals at chemical shift 1.0 and 1.2 ppm, respectively) and in TEA (at 1.1 ppm; products of reaction include small amount of catalyst).

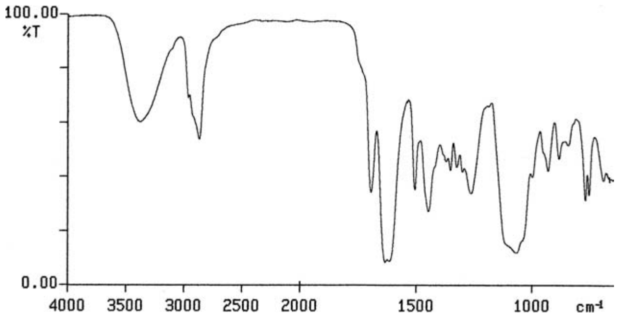
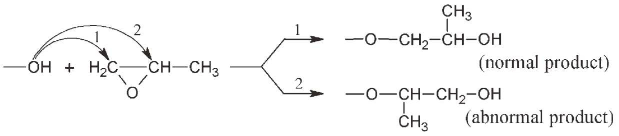


Figure 1. IR spectrum of the product of reaction of 1 mol of 6-AU with 6 mol of EC and 2 mol of PO.



(III)

Moreover, in the ^{1}H-NMR spectra of products (Figure 2), signals of protons of methylene and methine groups at chemical shift 3.2–3.9 ppm are present, and the proton resonances of H—C= groups at chemical shift 4.2–4.3 ppm and protons of hydroxyl groups at chemical shift 4.4–4.8 ppm (the splitting of signals is associated with various surrounding of the groups) can also be easily attributed. In the spectra, the signals at 4.4–4.8 ppm did not disappear completely after D_2O was added. These observations led to conclusion that unsaturated fragments are present in the product. They are formed at the ends of chains as a result of elimination of water in the first step of synthesis (reaction of 6-AU with EC) due to the high-temperature process (140°C). The height of the signal indicates that the amount of these structures is relatively small.

$$-O-CH_2-CH_2-OH \xrightarrow[-H_2O]{} -O-CH=CH_2$$

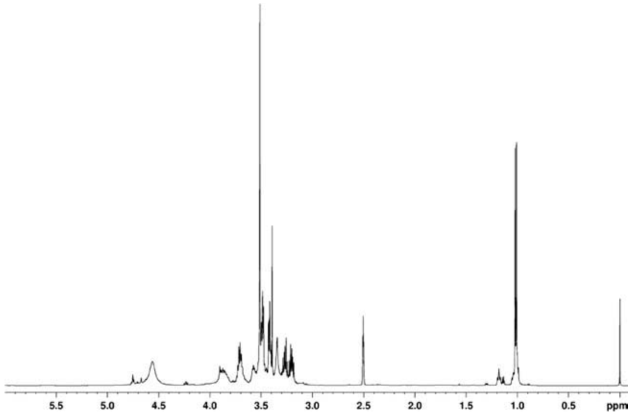


Figure 2. ^{1}H-NMR spectrum of the product of reaction of 1 mol of 6-AU with 6 mol of EC and 2 mol of PO.

Table III. Interpretation of MALDI–ToF Spectrum of the Product of Reaction of 1 mol of 6-AU with 6 mol of EC and 2 mol PO

Run	Signal position	Probable structure of molecular ion	Calc. molecular weight (g/mol)
1	276.3	AU + 2EO + PO – $2H_2O + K^+$	276.3
2	282.3	AU + 3EO + PO – $2H_2O + H^+$	282.3
3	300.2	AU + 3EO + PO – $H_2O + H^+$	300.3
4	326.3	AU + 4EO + PO – $2H_2O + H^+$	326.4
5	369.3	AU + 5EO + PO – $2H_2O$	369.4
6	388.3	AU + 5EO + PO – $H_2O + H^+$	388.4
7	413.3	AU + 6EO + PO – $2H_2O$	413.4
8	465.2	AU + 5EO + 2PO + H^+	464.5
9	503.4	AU + 5EO + 3PO – H_2O	503.6
10	523.3	AU + 5EO + 3PO + H^+	522.6
11	548.2	AU + 6EO + 3PO – H_2O	547.7
12	561.3	AU + 5EO + 4PO – H_2O	561.7
13	567.3	AU + 6EO + 3PO + H^+	566.6
14	581.3	AU + 5EO + 4PO + H^+	580.7
15	592.3	AU + 7EO + 3PO – H_2O	591.7
16	605.3	AU + 7EO + 4PO – H_2O	605.7
17	636.2	AU + 6EO + 3PO + $CH_3OH + K^+$	636.7
18	650.3	AU + 7EO + 4PO – H_2O	649.8
19	666.5	AU+5EO+2PO+CH_3OH+K^+	666.4
20	696.4	AU + 5EO + 6PO + H^+	696.8
21	727.3	AU + 7EO + 5PO + H^+	726.9
22	771.3	AU + 8EO + 5PO + H^+	771.3

AU is a fragmentation product of 6-AU; EO/PO is an oxyethylene/oxypropylene group, and the number preceding the symbol indicates the number of 6-AU molecules or oxyethylene/oxypropylene groups in a structure.

In the mass spectra of polyetherols, molecular peaks differing by $m/z = 44$ (oxyethylene subunits) and by $m/z = 58$ (oxypropylene subunits) are observed. It evidences that polyetherols containing oxyalkylene chains of various length are formed (Table III). The spectra show that unsaturated structures are formed (Table III). Much of the unsaturated structures are created in the conditions of registration of the spectrum: high temperature favors the elimination of water from the hydroxypropyl groups. Analysis of the ^{1}H-NMR spectra with D_2O shows that the small amount of unsaturated structures is present. Moreover, molecular ions masses increased by the mass of K^+ are recorded (from catalyst of reaction of 6-AU with EC—K_2CO_3; Table III nos. 1, 17, and 19).

Some physicochemical properties of obtained polyetherols, such as refraction index, density, viscosity, and surface tension (Table IV), were determined. Typical relationships of these parameters versus temperature were observed. The refractive index, density, and surface tension show linear dependence, while viscosity changes exponentially (Table IV). The values of the quantities decrease when initial molar ratio of 6-AU to EC and PO rises too.

Table IV. Some Physical Properties of Polyetherols Describing Their Dependence on Temperature, in the Range 20–80°C

Polyetherol 6-AU : EC : PO	Refraction index n_{20}^D (–) $n = aT + b$			Density d (g/cm^3) $d = aT + b$			Viscosity η (N s/m^2)·10^3 $\eta = a + b\,e^{-T/c}$				Surface tension τ (N/m)·10^3 $T = aT + b$		
	A × 10^3	b	r	a·10^4	b	r	a	b × 10^{-3}	c	r	a ;× 10^2	b	r
1 : 5 : 1	–3.20	1.5341	0.9977	–6.80	1.2567	0.9999	584.50	499.46	8.62	0.9997	–1.282	5.8554	0.9620
1 : 5 : 3	–3.21	1.5197	0.9997	–7.28	1.2056	0.9999	231.03	144.87	9.35	0.9998	–1.321	5.4321	0.9946
1 : 5 : 5	–3.50	1.5079	0.9997	–7.36	1.1650	0.9999	119.47	46.29	9.71	0.9994	–0.986	4.8357	0.9984
1 : 5 : 7	–3.48	1.4983	0.9992	–7.53	1.1385	0.9999	62.81	16.41	10.50	0.9992	–1.011	4.6454	0.9994
1 : 6 : 2	–3.10	1.5183	0.9995	–7.26	1.2186	0.9999	183.09	77.61	9.98	0.9998	–1.075	5.3375	0.9990
1 : 6 : 4	–3.30	1.5082	0.9999	–7.49	1.1800	0.9999	120.09	40.14	9.95	0.9994	–1.196	5.0368	0.9979
1 : 6 : 6	–3.34	1.4997	0.9985	–7.59	1.1529	0.9999	68.91	20.82	10.60	0.9998	–1.754	5.0025	0.9960

Table V. Thermal Resistance of Polyetherols Based on Thermal Analysis

Polyetherol 6-AU : EC : PO	$T_{5\%}$ (°C)	$T_{10\%}$ (°C)	$T_{25\%}$ (°C)	$T_{50\%}$ (°C)
1 : 5 : 1	110	130	165	290
1 : 5 : 3	150	180	210	270
1 : 5 : 5	105	150	185	220
1 : 5 : 7	150	170	200	240
1 : 6 : 2	100	140	190	250
1 : 6 : 4	135	160	195	240
1 : 6 : 6	155	180	210	260

Thermal stability of products of reaction of hydroxyethyl derivatives of 6-AU with PO was investigated using thermal analysis techniques (Table V). It shows that 5% mass loss at temperature range 100–150°C and 50% mass loss at the range 220–290°C occur. Thus, thermal stability of the polyetherols is similar to that of polyetherols obtained from 6-AU and oxiranes[13] and to that of polyetherols obtained from 6-AU and alkylene carbonates.[14,15]

Further, polyurethane foams were synthesized from polyetherols, and their properties were investigated. Foaming was carried out on the laboratory scale, and composition of foaming samples was determined by methods presented previously.[11,12] 4,4′-Diphenylmethane diisocyanate was used as an isocyanate component with water as a foaming factor. Silicon surfactant and TEA catalyst were used. It was observed that the best foams with regular small pores were obtained using 2 wt % of foaming factor (water), 1.3 or 1.9 wt % of catalyst (TEA), 0.8 wt % of surfactant (silicon), and such amount of isocyante that the molar ratio of OH : NCO was equal to 1.0–1.2 for polyetherols with shorter oxyalkylene chains (products of reaction 6-AU : EC : PO = 1 : 5 : 3 and 1 : 6 : 4) and 1.4–1.65 for polyetherols with longer oxyalkylene chains (products of reaction 6-AU : EC : PO = 1 : 5 : 7 and 1 : 6 : 6). Foaming conditions of selected polyurethane foams (characterized by small regular pores, good thermal, and mechanical properties) are presented in Table I.

Apparent density of the foams is in the range 29.5–47.2 kg/m^3 (Table VI), and it is comparable with apparent density of foams obtained from polyetherols based on oxiranes[11] and alkylene carbonates.[12] All the foams can be characterized as rigid.

Absorption of water after 24 h of exposition varies within the range 3.6–9.1 wt % (Table VI). The smallest absorption (3.6–6.3 wt %) shows the foams made from polyetherols of larger oxyalkylene chains, obtained at an initial molar ratio 6-AU : EC : PO equal to 1 : 5 : 7 (Table VI, compositions no. 3, 4) and 1 : 6 : 6 (Table VI, composition no. 6). Absorption of water of the foams is comparable to the absorption of foams made from polyetherols synthesized from carbonates (3.1–15.2 wt %),[12] and it is a little lower than absorption of foams made from polyetherols synthesized from oxiranes (5.8–32.5 wt %).[11]

Dimension stability measurements show that the foams have both positive and negative dimension change after thermal treatment at temperature 150°C (Table VI). The smallest dimension change was observed for composition no. 1.

The thermal stability measurements were performed at temperature 150, 175, and 200°C during 30 days by registering the loss of mass (Table VI). The gradual loss of mass has been observed. The highest loss of mass was always observed after the first day of exposition. All the foams after thermal treatment become more rigid. The smallest mass loss was observed for composition nos. 1, 4, and 6 (Table VI). For the rest of foams, higher mass loss was observed, up to 52.43 wt % in temperature 200°C (Table VI, composition no. 3). Some of the foams show small distortion of their shape after 1 day of thermal treatment temperature 150°C (Table I, composition nos. 3 and 4), larger distortion of their shape in temperature 175°C (Table I, composition nos. 3 and 4), and quite large at temperature 200°C (Table I, composition nos. 3, 4, and 5). Composition nos. 1, 2, and 6 did not show distortion of their shape after thermal treatment. The studies show that thermal stability of polyurethane foams made from polyetherols obtained from 6-AU, EC, and PO (composition nos. 1, 4 and 6) is comparable with thermal stability of foams made from polyetherols obtained in reaction 6-AU with alkylene carbonates[12] and slightly better than thermal

Table VI. Some Properties of Selected Polyurethane Foams

					Linear dimensions stability at temperature 150°C						Thermal stability of the polyurethane foams expressed as the mass loss of foams (wt %) after heating for 30 days at temperature		
		Absorption of water (wt %)			Length increase (%)		Width increase (%)		Depth increase (%)				
Composition number	Density (kg/m^3)	After 5 min	After 3 h	After 24 h	After 20 h	After 40 h	After 20 h	After 40 h	After 20 h	After 40 h	150°C	175°C	200°C
1	29.5	2.76	5.93	8.24	0	–1.17	–0.05	–0.75	0	1.52	7.89	24.66	37.84
2	44.45	4.1	7.38	9.11	–5.04	–5.45	–3.98	–5.05	–2.34	–2.34	15.46	33.22	45.66
3	44.86	3.49	4.39	6.26	–0.28	0.84	3.27	6.54	3.51	7.00	19.73	38.04	52.43
4	39.61	1.29	2.35	3.59	–0.90	0.38	0.57	3.45	–3.84	–3.06	12.97	26.92	40.56
5	47.12	3.67	5.57	7.69	7.14	9.52	7.69	10.26	3.70	6.48	19.98	36.83	48.10
6	42.27	2.57	4.15	5.28	–0.29	2.03	2.36	4.72	–1.94	0	12.80	29.03	41.40

Table VII. Thermal Resistance and Heat Stability Based on Thermal Analysis and DSC and Compression Strength at 10% Strain of Selected Polyurethane Foams

Composition number	Thermal resistance and heat stability $T_{10\%}$ (°C)	$T_{25\%}$ (°C)	$T_{50\%}$ (°C)	T_g (°C)	Compression strength σ_{10} (MPa)
2	200	260	400	101	0.22
4	255	285	330	133	0.30
6	235	280	330	138	0.34

stability of foams made from polyetherols synthesized from 6-AU and oxiranes.[11]

The thermal analysis of the selected polyurethane foams show that 10% mass loss is observed at temperature range 200–255°C and 50% mass loss at temperature range 330–400°C (Table VII). The mentioned results confirm an improved thermal stability of the obtained foams. Next, determined by the DSC method, glass transition of some polyurethane foams is included in the range 101–138°C (Table VII) what shows that the obtained foams are rigid.

Mechanical properties of the foams made from polyetherols obtained in reactions of 6-AU with EC and PO (Table VII) were evaluated on the basis of compression strength measurements. The investigations confirmed that compression strength of the foams (composition nos. 4, 6) is significantly larger than compression strength of foams made from polyetherols obtained from 6-AU and carbonates.[12] It is even larger than compression strength of polyetherols obtained from 6-AU and oxiranes.[11]

CONCLUSIONS

In reaction of 6-AU with EC and next with PO, polyetherols with 1,3-pyrimidine ring are obtained, containing oxyethylene and oxypropylene groups in their structure.

The products are highly thermally resistant, and thus they can be used as polyol components to obtain polyurethane foams with improved thermal stability. The foams obtained from these polyetherols show better thermal stability and higher compression strength than foams based on polyetherols obtained in reaction of 6-AU with oxiranes[11] and foams based on polyetherols synthesized from 6-AU and carbonates.[12] Other properties of foams such as apparent density, water uptake, and dimension stability are similar to the properties of the commercial polyurethane foams.

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