Characterization of polyurethane networks based on rapeseed oil derived polyol[*)]

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DOI: dx.doi.org/10.14314/polimery.2014.333

Abstract: Bio-based rapeseed oil (RO) polyol was synthesized by transesterification with triethanolamine. Synthesized RO/triethanolamine ester was modified with triethylene glycol (TEG) to obtain a polyol system with higher hydroxyl value, as well as with ε-caprolactone (CL) to increase the carbon chain length and decrease the hydroxyl value. From these polyols there were synthesized polyurethane (PU) networks which showed good mechanical properties. It was established that with increasing urethane groups concentration and increasing crosslinking, polyurethane networks showed higher modulus of elasticity (E) and tensile strength (σ). Polyurethane networks also showed higher elongation at break (ε) with decreasing urethane groups concentration and decreasing crosslinking.

Keywords: rapeseed oil, polyurethane, crosslinking density, cohesion energy density.

Charakterystyka sieci poliuretanowych na bazie poliolu otrzymanego z oleju rzepakowego

Streszczenie: Poliol z oleju rzepakowego (RO) otrzymano metodą transestryfikacji trietanoloaminą. Otrzymany ester RO/trietanoloamina modyfikowano glikolem trietylenowym (TEG), w celu uzyskania układu poliolowego o większej zawartości grup OH, lub ε-kaprolaktonem (CL) w celu zwiększenia długości łańcucha węglowego i zmniejszenia zawartości grup OH. Z otrzymanych polioli syntezowano sieci poliuretanowe (PU), które charakteryzowały się dobrymi właściwościami mechanicznymi. Stwierdzono, że wraz ze wzrostem stężenia grup uretanowych i zwiększeniem usieciowania, sieci PU charakteryzują się większym modułem sprężystości (E) i większą wytrzymałością na rozciąganie (σ). Wykazano również, że zmniejszenie usieciowania i zawartości grup uretanowych w sieciach poliuretanowych wpływa na zwiększenie wydłużenia przy zerwaniu (ε).

Słowa kluczowe: olej rzepakowy, poliuretan, gęstość usieciowania, gęstość energii spójności.

Polyurethanes (PUs) are widely known and used polymers due to their versatility and wide range of applications. PUs can be tailored as foams of various density, elastomers, coatings, sealants and others [1, 2].

One of the major drawbacks of PUs is their dependence on petroleum feedstock. Conventionally PUs are produced from polyether or polyester polyols and isocyanate. Nowadays isocyanates are still produced from petrochemicals, but polyols are also produced from bio-based raw materials [1—3]. Vegetable oils are one of the most important classes of renewable materials. Vegetable oils have low price and are available worldwide and most importantly they are suitable for various chemical transformations [3, 4]. Vegetable oils mainly consist of triglycerides — fatty acids esterified to glycerol [5, 6].

Intermolecular forces play very important role in PU properties. The intermolecular forces (attractions) are hydrogen bonding, London dispersion forces, permanent dipole interactions, ionic bond interactions and Van der Waals forces [1]. Intermolecular forces tend to hold the polymer chains closer together, similar as chemical bonds do. In comparison to chemical bonds, intermolecular forces are much weaker and they more rapidly react to stress or increased temperature. Intermolecular forces can be characterized as cohesion energy density (*CED*).

PU properties depend on the crosslinking of polymer chains and can be modified by varying the crosslinking degree. To characterize crosslinked PU, molecular weight between crosslinks (M_c) can be used. M_c is the parameter characterizing macromolecule architecture [1]. Crosslinking and M_c are interdependent.

The aim of this study was to determine the impact of M_c and thus the crosslinking density, and impact of *CED* on mechanical properties of PUs obtained from rapeseed oil (RO) polyols.

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[*)] Material contained in this article was presented on the 3rd Workshop on Green Chemistry and Nanotechnologies in Polymer Chemistry, 24—26 September 2013, Cracow, Poland.

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EXPERIMENTAL

Materials

Short description and sources of the reagents used for the synthesis of RO polyols and PU networks are given in Table 1. All the reagents were used without any prior treatment.

T a b l e 1. Reagents used for the synthesis of RO polyols and PU

Materials	Source	Description
Rapeseed oil	Iecavnieks & Co Ltd, Latvia	iodine value = 117 I_2 mg/100 g, acid value = 2.1 mg KOH/g, saponification value = 192 mg KOH/g
Triethanolamine (TEA)	BASF, Germany	99.5 %
Zinc acetate dehydrate	Sigma Aldrich, Germany	≥98 %
Triethylene glycol (TEG)	Sigma Aldrich, Germany	99 %
Stannous octoate	Sigma Aldrich, Germany	95 %
ε-Caprolactone (CL)	Sigma Aldrich, Germany	99 %
Toluene anhydrous	Sigma Aldrich, Germany	≥99.7 %
4,4'-Methylene diphenyl diisocyanate (MDI) Voratec SD 100	Dow Chemical, Belgium	NCO content = 33.6 wt %, functionality f_n = 2.0, M = 250 g/mol
A polymeric MDI Suprasec 2651	Huntsman, Belgium	NCO content = 32.2 wt %, f_n = 2.3, M = 303 g/mol
A polymeric MDI Lupranate M20	BASF, Germany	NCO content = 31.5 wt %, f_n = 2.7, M = 360 g/mol

Synthesis of RO/TEA polyol and polyol systems

RO polyols were synthesized using transesterification with triethanolamine (TEA). Transesterification with TEA was carried out at 170 ± 5 °C for 2 h and 0.15 wt % of zinc acetate was used as a catalyst. A more detailed description of RO polyol synthesis is given in our previous work [7]. The completion of the reaction was tested by the solubility of the obtained product in ethanol at the volume ratio (resin:ethanol = 1:1), as well as by a change in refractive index of the synthesis mixture. The synthesized RO/TEA esters had the hydroxyl value 375 mg KOH/g and viscosity 156 mPa · s at 25 °C.

The main chain of RO/TEA polyol was extended by modifying obtained RO/TEA polyol with ε-caprolactone (CL) to introduce [-$(CH_2)_5$-COO-] links in the structure. The mixture of RO/TEA polyol (28 g) and CL (12 g) was heated for 36 h at 120 °C, and 0.05 wt % of stannous octoate was used as a catalyst. Since we synthesized only 20 g of RO/TEA/CL no further testing of this polyol was carried out.

The second polyol system, denoted as RO/TEA/TEG was obtained by mixing 80 wt % of RO/TEA and 20 wt % of TEG at the room temperature.

Preparation of isocyanate mixture

To obtain PU networks we used the previously described RO polyol systems — RO/TEA, RO/TEA/TEG and RO/TEA/CL. The polyol component was reacted with various isocyanates separately or with isocyanate mixture to obtain a variation in M_c of PU network. The isocyanate mixtures were prepared using MDI, a polymeric MDI with trade name Suprasec 2651, and polymeric MDI named Lupranate M20. The functionality (f_n) of isocyanates or isocyanate mixtures was 2.0, 2.2, 2.3, 2.4, 2.5, 2.6 and 2.7. The necessary calculations to obtain isocyanate mixture with required functionality were carried out using equation:

$$f_n = \frac{m_1 f_{n_1} + m_2 f_{n_2}}{m_1 + m_2} \tag{1}$$

where: f_n — acquired functionality of the isocyanate system, m_1, m_2 — mass of the mixed isocyanates, f_{n_1}, f_{n_2} — functionality of the isocyanates.

PU networks synthesis

The PU samples were cast from a solution of 50 % anhydrous toluene at the temperature of 20—25 °C. The functional group ratio NCO:OH was 1:1. When the conversion of NCO groups reached 60—70 %, the solution was poured on a polyoxymethylene support and left at the temperature of 70 °C for 2 h for conditioning. In preliminary tests for each series the NCO group concentration was determined using chemical analysis methods, and then NCO group conversion was calculated. After the conditioning the PU films were left at the room temperature for 3 days before stress-strain determination. The obtained films had the thickness of 150—250 μm.

Methods of testing

Progress of RO polyols synthesis reaction was monitored by examination of the changes in the refractivity index (n_D^{20}) of the reaction mixture. Refractive index was tested using digital refractometer PAL-RI (Atago, USA). During RO/TEA polyol synthesis samples were taken every 5 min, cooled down to the room temperature and then n_D^{20} was measured.

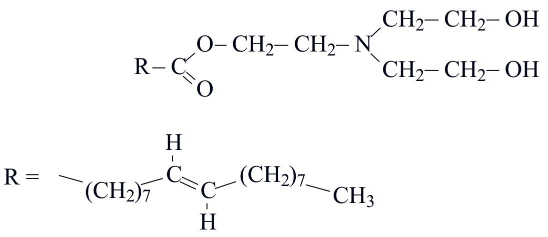
The stress-strain characteristics of PU films were tested according to the requirements of ASTM D882-10 standard. The films' stress-strain characteristics were determined by tensile testing of 5—7 specimens on a Zwick/Roell at the deformation rate 7 mm/min.

Dynamic mechanical analysis (DMA) was used to study mechanical properties of PU films. We used a Mettler Toledo DMA/SDTA 861^e dynamic mechanical analyzer with a tensile mode at the heating rate of 3 °C/min and frequency of 1 Hz. The tensile storage modulus (E') was measured as a function of temperature.

RESULTS AND DISCUSSION

Polyol and polyol system characterization

RO is one of the most widespread oil crops worldwide. RO mainly consists of unsaturated fatty acids: 61 % of oleic acid, 21 % of linoleic acid, 9 % of linolenic acid and saturated fatty acid, 4 % of palmitic acid and other saturated and unsaturated fatty acids [8].

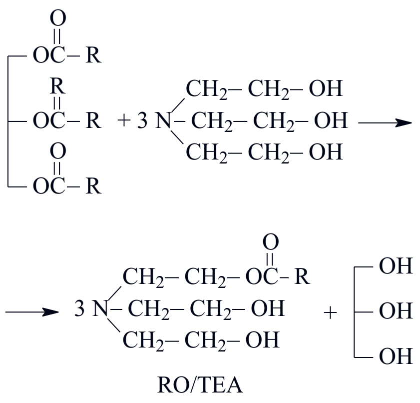


(I)

$$R-C(=O)-O-CH_2-CH_2-N(CH_2-CH_2-OH)_2$$

$$R = -(CH_2)_7-CH=CH-(CH_2)_7-CH_3$$

The RO/TEA polyol can be characterized as a short chain polyol (chain length between OH groups — 5 atoms) with high dangling chain content. Dangling chains are fatty acid radicals, and they can act as plasticizers in PU networks [4]. The idealized structure of RO/TEA polyol synthesized using transesterification is shown by formula (I). However, transesterification of RO does not occur according to the idealized reaction as presented in Scheme A. The polyol may also contain monoglycerides, diglycerides, glycerol and triethanolamine. The formation of these by-products increases when RO/TEA polyol is synthesized at molar ratio of the oil and alkanolamine lower than 1:3.



Scheme A

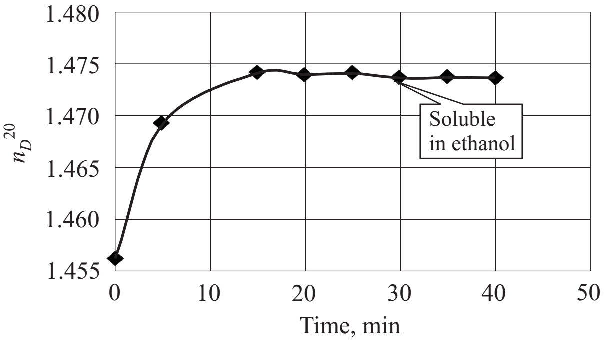


Fig. 1. Changes in the refraction coefficient (n_D^{20}) during RO/TEA polyol synthesis

The progress the RO/TEA polyol reaction was judged by the measurements of n_D^{20} during the reaction and results are shown in Fig. 1. RO/TEA polyol synthesis is complete and there is no free RO when n_D^{20} has reached the point when its value does not change significantly.

The synthesized RO/TEA polyol had the hydroxyl value of 375 mg KOH/g and functionality f_n = 2.25 OH groups/mol. The functionality ($f_{n\ OH}$) is the second most important characteristic of a polyol and is defined as an average number of hydroxyl groups in a molecule of polyol [1].

RO/TEA polyol is characterized with catalytic activity in urethane forming reactions because it contains a tertiary amine atom. It is well known fact that tertiary amine catalyzes the urethane forming reaction [1]. Also RO/TEA polyol contains primary hydroxyls which are more than 3 times more reactive with isocyanate component than secondary hydroxyls [9]. Due to the primary hydroxyls and tertiary amine atom present in RO/TEA polyol and the use of a hydrophobic solvent, we decreased the possibility of moisture water reaction with isocyanate component instead of polyol component. It is known that reaction between NCO-R and water leads to production of CO_2 [1], but our obtained PU films did not contain air bubbles, that also confirms the fact that reaction between water and NCO-R was not significant. However, the possibility of the reaction between water and NCO-R can not be completely ruled out.

To obtain PU networks with significant differences in properties we modified the obtained RO/TEA polyol. The modification was carried out by adding TEG to the RO/TEA polyol. In the result we obtained a RO/TEA/TEG polyol system with hydroxyl value of 449 mg KOH/g and $f_{n\ OH}$ = 2.14 (Table 2). Also, the RO/TEA polyol was modified to introduce CL links in the RO/TEA polyol main chain and increase the carbon chain length in the main chain of polyol (Fig. 1). That will yield more elastomeric PU networks. The hydroxyl value of RO/TEA/CL was 264 mg KOH/g, $f_{n\ OH}$ = 2.25 (Table 2).

Table 2. Hydroxyl and urethane groups concentration in polyol systems used to obtain PU networks

PU series	Hydroxyl value mg KOH/g	f_n	Urethane group concentration mol/1000 g
RO/TEA	375	2.25	3.74 ± 0.04
RO/TEA/TEG	449	2.14	4.08 ± 0.05
RO/TEA/CL	264	2.25	3.08 ± 0.03

The hydroxyl value of the polyol is the most important parameter characterizing polyol. Higher hydroxyl value allows forming more urethane linkages. We calculated the urethane group concentration to help characterize and explain the properties of PU networks obtained from RO polyols. At first the OH group equivalent in 100 g of polyol was calculated:

$$\text{OH group equivalent} = \frac{\text{OH\%}}{17\ \text{g}\cdot\text{mol}^{-1}} \tag{2}$$

where: OH % — weight percent of hydroxyl group in polyol, 17 g · mol^{-1} — molar mass of hydroxyl group.

Then the urethane groups concentration (UGC) in 1000 g of PU was calculated:

$$\text{UGC} = \frac{1000\cdot\text{OH group equivalent}}{\text{mass of polyol and isocyanate}}\cdot 1.1\ \text{(mol/1000 g)} \tag{3}$$

where: 1.1 — the isocyanate index.

The urethane group concentration was calculated for each RO polyol/polyols system with different isocyanates or mixtures of isocyanates. The urethane group concentrations for each PU series are presented in Table 2. Since in one PU network series the difference between urethane group concentrations was small, we decided to display the results as urethane group concentration for the whole PU network series with standard deviation (Table 2). The PU network series were named after the polyol used.

PU network and their *CED*

PUs are a class of widely spread polymers that contain urethane groups in their structure regardless of the rest of the molecule [10].

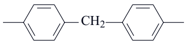
In our study *CED* was calculated for different groups present in PU structure using group contribution method by Fedors [11]. The *CED* of polymer is calculated according to equation:

$$CED = \frac{\Delta E_v}{V} \tag{4}$$

where: ΔE_v — the energy of vaporization, V — molar volume.

The calculated values of *CED* are presented in Table 3.

Table 3. CED of groups in PU structure

Group Name	Structure	*M*, g/mol	*CED*, J/cm^3
Urethane	-NH-C(O)O-	59.02	1172
Ester	-C(O)O	28.01	1001
Ether	-O-	16.00	883
Methylene diphenyl		166.23	569
Unsaturated hydrocarbons	-CH=	13.02	318
Hydrocarbon	-CH$_2$-	14.03	306

Each group has a cohesive energy that defines how much energy is required to tear it apart. As we can see from the data in Table 3, the highest *CED* (1172 J/cm^3) is for urethane group, and then ester and ether groups follow. The urethane, ester and ether groups also play a significant role in hydrogen bonding. The total value of *CED* has an effect on the strength of the PU. In this study we did not calculate the total *CED* for the whole PU macromolecule due to its complexity. Since urethane group has the highest *CED*, we can expect that PU networks with higher urethane group concentration will also show better mechanical properties.

PU properties can be modified by varying the crosslinking degree. In our study we did not use any chain extenders, but we used various isocyanates and mixtures of isocyanates with different functionalities to obtain PU networks with different crosslinking.

Crosslinking and M_c are interdependent. To characterize crosslinked PU, M_c can be used. M_c is the parameter characterizing macromolecules' architecture [1]. The theoretical M_c value was calculated for each RO polyol system reaction with different isocyanate mixtures using equation:

$$M_c = \frac{\sum(m_1 + m_2 + m_3 + \ldots + m_n)}{\frac{(f_{n_1}-2)\cdot m_1}{M_1} + \frac{(f_{n_2}-2)\cdot m_2}{M_2} + \frac{(f_{n_3}-2)\cdot m_3}{M_3} + \ldots + \frac{(f_{n_n}-2)\cdot m_n}{M_n}} \tag{5}$$

where: $\Sigma(m_1 + m_2 + m_3 + \ldots + m_n)$ — total mass of all the components present in PU; $m_1, m_2, m_3 \ldots m_n$ — mass of the component present in PU; $f_{n_1}, f_{n_2}, f_{n_3} \ldots$ — functionality of the component (polyol, isocyanate, crosslinker etc.) present in PU; $M_1, M_2, M_3 \ldots M_n$ — molar mass of components present in PU [12].

To verify the theoretical value of M_c we carried out DMA tests and calculated actual M_c value based on the ideal rubber theory equation [13]:

$$E' = \frac{3\rho R T_{abs}}{M_c} \tag{6}$$

where: E' — plateau modulus, ρ = 1.2 g/cm^3 — density, R — gas constant, T_{abs} — absolute temperature.

The actual M_c value for PU synthesized from RO/TEA polyol and a polymeric MDI Lupranate M20 was 752 g/mol. The theoretical M_c value calculated using eq. (5) was 735 g/mol. Experimental M_c values calculated using E' values are close to theoretical values of M_c for PUs from RO polyols.

The molecular structure of our synthesized PU networks varied from rigid highly crosslinked to poorly crosslinked structure containing more flexible chains.

Mechanical properties

The mechanical properties of PUs are dependent on the extent of crosslinking and thus M_c value in polymer chain. To synthesize PU networks with desired properties, it is crucially important to understand the structure-property relationships of PU network.

Crosslinking is important for elastomers since it controls tensile strength and elasticity. PU networks were prepared by the reaction of given polyol systems (Table 2) with different mixtures of MDI, a polymeric MDI Suprasec 2651 and a polymeric MDI Lupranate M20. The isocyanate index was 110. The extent of crosslinking and thus M_c was regulated by the functionality of the isocyanate mixture. We worked with seven different isocyanate components (pure isocyanate or isocyanate mixture) with f_n in the range between 2.0 and 2.7.

Experiments were carried out so we could determine the impact of M_c on the properties of resulting PU networks. The results of PU mechanical testing, modulus of elasticity (E), tensile strength (σ) and elongation at break (ε) depending on M_c are presented in Fig. 2 and 3.

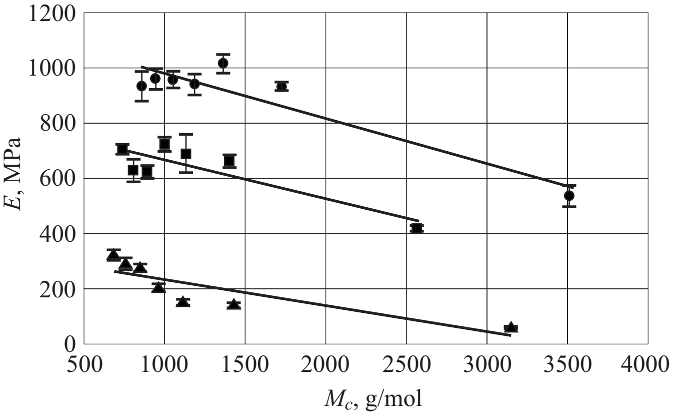


Fig. 2. Modulus of elasticity (*E*) of PU networks

As we can see from curves in Fig. 2, the highest E is shown by PU network from RO/TEA/TEG with urethane group concentration of 4.08 mol/1000 g, but the lowest E is shown by PU network from RO/TEA/CL with urethane group concentration 3.08 mol/1000 g. Previously discussed theoretical assumption that the highest modulus of elasticity will be shown by PU network with the highest urethane group concentration and thus *CED*, respectively, is experimentally confirmed.

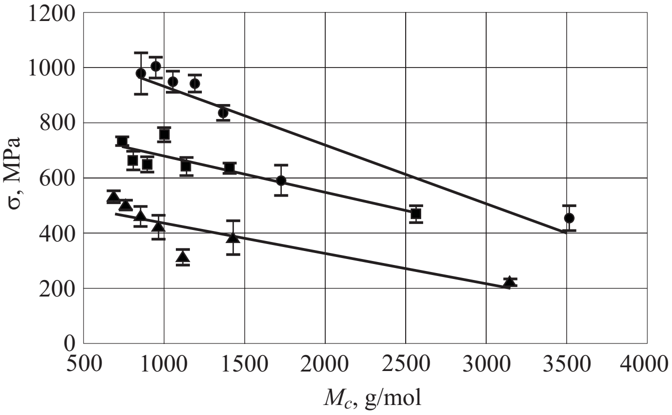


Fig. 3. Tensile strength (σ) of PU networks

In all PU networks, E increases with decreasing M_c (increasing crosslinking). It is due to the decreased macromolecule chain flexibility which leads to higher modulus of elasticity. As a result we obtain more rigid and stiffer material.

The same trend is observed for σ of PU networks (Fig. 3). σ decreases with increasing M_c, as well as higher σ is observed for PU network with higher urethane group concentration.

Usually crosslinks are introduced in less perfect manner than desired. We can conclude from the results of mechanical testing of PU networks (Fig. 2 and 3), that the crosslinks were not introduced ideally. There is a noticeable dispersion of the results. Imperfections in real PU network can be caused by the highly complex structure of PU macromolecule containing long dangling chains. Dangling chains can lead to impaired hydrogen bonding. Imperfections can also be introduced by unreacted functionalities. The results for ε showed that with increasing M_c and thus decreasing crosslinking, ε increases for all PU networks. That is due to the fact that the main polymer chain between urethane groups is still free to move. The highest elongation at break showed PU network from RO/TEA/CL reaching 95.9 ± 6.5 % at the highest M_c of 3147 g/mol and 48.6 ± 4.9 at the lowest M_c of 685 g/mol. PU networks from RO/TEA and RO/TEA/TEG showed considerably lower elongation at break.

CONCLUSIONS

Bio-based natural oil polyol was synthesized from RO using transesterification with triethanolamine. RO polyol systems were formulated to obtain PU networks with different properties.

PU networks with higher urethane group concentration (thus *CED*) showed the highest E and σ. Although there were imperfections in crosslink introduction into PU networks, E and σ is observed with decreasing molecular weight between crosslinks.

At the same time PU networks with decreasing urethane group concentration and increasing molecular weight between crosslinks showed higher elongation at break.

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Received 30 IV 2013.

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