Polym. Bull. (2012) 68:1271–1285
DOI 10.1007/s00289-011-0603-z

ORIGINAL PAPER

Synthesis and characterization of linear and crosslinked poly(urethane urea) elastomers with triazine moieties in the main chain

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Received: 8 April 2010 / Revised: 12 August 2011 / Accepted: 13 August 2011 /

Abstract A series of new poly(urethane urea) is synthesized via a two-step polyaddition process from polyether, 1,6-hexamethylene diisocyanate, 2,4-diamino-6-phenyl-1,3,5-triazine and different crosslinkers: glycerin or castor oil. The hard to soft segment ratio ($OH_{polyol}/NCO/NH_{2chain\ extender}$) was varied systematically from 1/2/1 to 1/4/3. Poly(tetramethylene glycol) of molecular weight 1,400 was used as the soft segment. The structural behavioral characterization of these polymers was performed through FTIR spectroscopy, thermogravimetric analysis, dynamic mechanical and thermal analysis, stress–strain measurements, and water contact angle measurements. The resulting linear polyurethane urea elastomers exhibit good mechanical properties with breaking strains of 300–890% and tensile strengths of 8–13.5 MPa. Thermogravimetric analysis indicated that the thermal degradation of poly(urethane urea) started at about 280–300 °C, higher than the degradation temperature of conventional polyurethane. The improvement of properties was influenced by the hard segment content and the nature of the crosslinker, but most of all by the structure and amount of the urea introduced through 2,4-diamino-6-phenyl-1,3,5-triazine into the polymer backbone chain.

Keywords Polyurethane urea · Diamino triazine · Mechanical properties · Thermal behavior

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Introduction

Poly(urethane) and/or poly(urethane urea) are one of the most important groups of elastomeric materials in terms of research and practical applications [1–5]. Polyurethane elastomers can achieve a wide range of properties through changes in the proportion and structure of the soft segment (providing the basis for elasticity) or of the hard segment (providing the physical crosslinks) [6–10]. The poly(urethane urea)s are obtained in the same conditions as polyurethanes, with the difference that diamines are used as chain extenders instead of diols. Poly(urethane urea) elastomers possess improved mechanical and thermal properties compared with traditional polyurethane elastomers because the urea groups develop stronger, three-dimensional hydrogen bonding [11–14]. For long-term applications of poly(urethane urea)s, the hard domain morphology can be varied by changing the starting materials, hard segment content and degree of crosslinking [15, 16]. The nature of the polyurethane's hard segment domain influences the hydrogen bonding between the amine and carbonyl groups of the urethane or urea linkage [17].

To improve the thermo-mechanical properties of the polyurethane and poly(urethane urea), chemical crosslinks were introduced into the hard segment domains through various crosslinking agents [18–20]. The chemical modification of the hard segment through the introduction of heterocyclic structures like imide, oxazolidone, triazine, and phosphazene can improve thermal stability [21, 22] and such structures have been used for the formation of supramolecular structures through hydrogen bonds [23].

Benzoguanamine (2,4-diamino-6-phenyl-1,3,5-triazine) is currently used in increasing the thermoset properties of alkyd, acrylic and formaldehyde resins or as flame-retardant additives [24, 25]. Triazine moieties are found in a series of natural products and medicinal drugs. 2,4-Diamino-1,3,5-triazine derivatives possess a broad range of biological properties, such as antimicrobial, antifungal, or allergy inhibiting properties [26, 27].

Introducing 2,4-diamino-1,3,5-triazine derivatives into the polyurethane chains will increase the number of hydrogen bonds and it will also enhance the biological properties of the polymer. In the case of 2,4-diamino-6-phenyl-1,3,5-triazine, the phenyl groups may act as spacers between the polymer chains, thus possibly causing the modification of physical properties. The thermal stability and mechanical properties of the polyurethanes is mainly determined by the chemical structure of the backbone chain, containing various hard and soft segments.

In this study, 2,4-diamino-1,3,5-triazine derivatives moieties were introduced into the polyurethane backbone to improve its thermal stability and in order to enhance the relationships between the polymer hard segment structure and several properties such as mechanical and surface properties. For this purpose, 2,4-diamino-6-phenyl-1,3,5-triazine was used as a chain extender for NCO-terminated urethane prepolymers. The oligomers were prepared from poly(tetramethylene glycol) and aliphatic diisocyanate at various NCO/OH ratios. The linear poly(urethane urea) thus obtained were compared to glycerin- or castor oil-crosslinked polymers.

Experimental

Materials

2,4-Diamino-6-phenyl-1,3,5-triazine, glycerin (Gly), castor oil (CO) were purchased from Aldrich and were used as received. 1,6-Hexamethylene diisocyanate (HDI) was purchased from Fluka and was used as received.

Poly(tetramethylene glycol) of molecular weight 1,400 (Terathane 1400) was purchased from Fluka and was degassed and dried under high vacuum (10 mmHg) at 120 °C for 2 h before being used.

Polymerization procedure

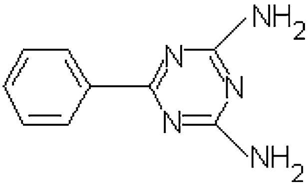
The poly(urethane urea)s were prepared as a melt in a two-step procedure and the molar amounts used for the preparations are shown in Table 1. The hard-segment composition was controlled by the molar ratios of poly(ether)-diol/HDI/diamine or triol used in the synthesis. The molar ratios of the formulations were 1/2/1, 1/3/2, and 1/4/3.

The synthesis of poly(urethane urea) was performed in a 250 mL glass reactor at normal pressure and under agitation. Polyether diol was reacted with diisocyanate at 80 °C for 2 h in order to yield a prepolymer. The polyurethane prepolymer was chain extended by adding a mixture of the 2,4-diamino-6-phenyl-1,3,5-triazine (Scheme 1), chain extender and crosslinker [if crosslinked poly(urethane urea) were to be obtained], the reaction taking place at 80 °C for the duration of an 2 h. The polymers were cast on a clear glass to obtain uniform sheets. After casting, these films were cured at 80 °C for 20 h. Poly(urethane urea) sheets thus prepared were used for the determination of morphological and thermo-mechanical properties.

Table 1 Molar ratios and chain extenders used in the preparation of the poly(urethane urea)s

Designation samples	Molar ratio polyether/HDI/chain extenders	Chain extenders The $NH_{2\text{chain extender}}/OH_{\text{crosslinker}}$ molar ratio was of 1/1 in each of the crosslinked polyurethane samples
PTA1	1:2:1	2,4-Diamino-6-phenyl-1,3,5-triazine
PTA2	1:3:2	2,4-Diamino-6-phenyl-1,3,5-triazine
PTA3	1:4:3	2,4-Diamino-6-phenyl-1,3,5-triazine
PTA4	1:2:1	Glycerin + 2,4-diamino-6-phenyl-1,3,5-triazine
PTA5	1:3:2	Glycerin + 2,4-diamino-6-phenyl-1,3,5-triazine
PTA6	1:4:3	Glycerin + 2,4-diamino-6-phenyl-1,3,5-triazine
PTA7	1:2:1	Castor oil + 2,4-diamino-6-phenyl-1,3,5-triazine
PTA8	1:3:2	Castor oil + 2,4-diamino-6-phenyl-1,3,5-triazine
PTA9	1:4:3	Castor oil + 2,4-diamino-6-phenyl-1,3,5-triazine

Scheme 1 Chain extender chemical structure



Measurements

The infrared (FTIR) spectra were recorded on a Bruker VERTEX 70 Instruments equipped with a Golden Gate single reflection ATR accessory. The spectra were recorded in the range of 600–4000 cm^{-1} with a nominal resolution of 4 cm^{-1}.

Thermogravimetric analyses of poly(urethane urea)s were made with a DERIVATOGRAF Q-1500 D apparatus (Hungary) in an air atmosphere at the heating rate of 10 °C/min. The initial weight of the samples was of about 50 mg and TG curves were recorded between 30 and 700 °C.

Dynamic mechanical analysis (DMA) was performed using a Perkin Elmer Diamond DMA, at a frequency of 1 Hz, to determine the storage modulus (E') and the loss factor (tan δ). All samples were scanned in a temperature range of -100 to 250 °C at a constant heating rate of 2 °C per min.

Stress–strain measurements were performed on dumbbell-shaped samples cut from the obtained poly(urethane urea) sheets. Testing was conducted at room temperature using a Shymadzu EZTest (Japan), equipped with a 5 kN load cell. The used cross-head speed was of 50 mm/min. At least five identical dumbbell-shaped specimens were evaluated for each polymer composition.

Contact angle measurements (CA) were performed using deionized water and ethylene glycol by a sessile drop method on a Dataphysics Contact Angle System KSV Instruments LTD, Finland. The contact angle was measured within 45–60 s of the addition of the liquid drop with an accuracy of ±2°.

Results and discussion

FTIR spectroscopy was used to investigate the formation of poly(urethane urea) and hydrogen bonds of various chemical groups of poly(urethane urea) with different hard segment structures. The FTIR spectra of the poly(urethane urea)s with different chain extender molar ratios are shown in Figs. 1, 2, and 3.

For the characterization of the hydrogen-bonding state of linear poly(urethane urea), two main vibration regions were generally used: the N–H stretching vibration (3150–3500 cm^{-1}) and the carbonyl C=O stretching vibration in the amide I region (1500–1650 cm^{-1}).

In the case of linear poly(urethane urea)s (Fig. 1), the sharp peak at 1617 cm^{-1} was attributed to the hydrogen-bonded urea carbonyl groups. The small peak that appears at 1590 cm^{-1} was related to the hydrogen-bonded urethane carbonyl groups. The peak for the hydrogen-bonded urea carbonyl is relatively more intense

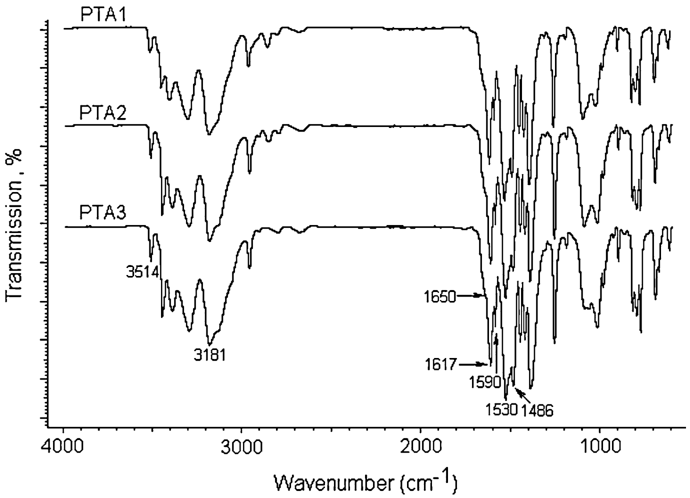


Fig. 1 FTIR spectra of films of poly(urethane urea)s obtained with 2,4-diamino-6-phenyl-1,3,5-triazine as chain extender

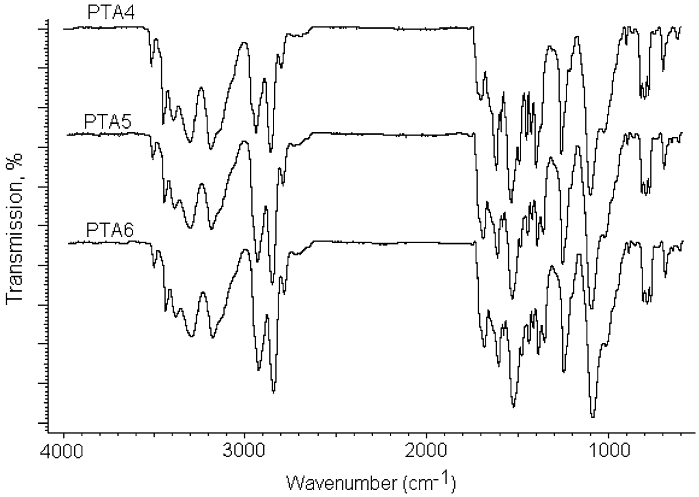


Fig. 2 FTIR spectra of films of poly(urethane urea)s obtained with 2,4-diamino-6-phenyl-1,3,5-triazine and glycerin as chain extenders

than the peak corresponding to the hydrogen-bonded urethane carbonyl group. This higher degree of urea–hydrogen bonding results in an increased virtually crosslinked structure [28, 29].

The strong absorption peaks for the stretching vibration associated to the (C=N) group from the triazine ring clearly appear at the frequencies of 1530 and 1486 cm^{-1}.

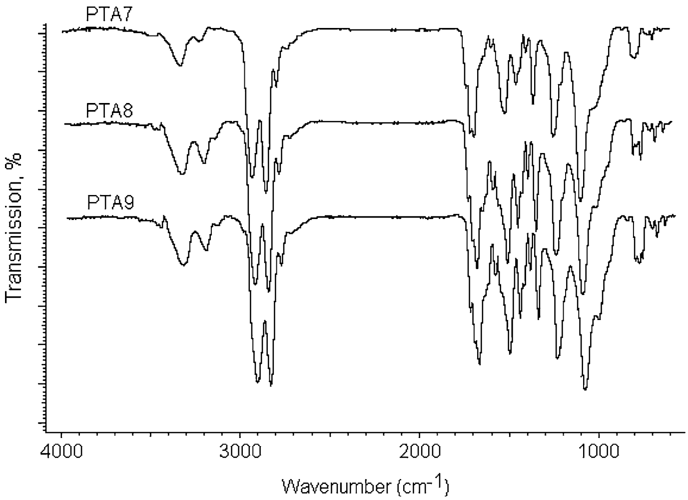


Fig. 3 FTIR spectra of films of poly(urethane urea)s obtained with 2,4-diamino-6-phenyl-1,3,5-triazine and castor oil as chain extenders

The stretching vibration of N–H bonds was identified through five peaks placed in between 3181 and 3514 cm^{-1}. The non-hydrogen-bonded N–H stretching peak appears at about 3514 cm^{-1} and only increases slightly with the increase of the triazine amount. This correlates with the fact that the free hydrogen-bonded carbonyl group appears as a shoulder at 1650 cm^{-1}. This indicated that many of the N–H groups were in hydrogen-bonded state. This is supported by the fact that the peak assigned to the hydrogen-bonded urea groups and centered around 3181 cm^{-1} is very strong.

For poly(urethane urea) with glycerin in their hard segments, the stretching vibration of N–H bonds is somewhat preserved, but differences appear for the carbonyl C=O stretching vibration (Fig. 2). In the case of all poly(urethane urea)s obtained with glycerin, the peak for urea carbonyl (–NH–CO–NH–) appeared around 1580–1620 cm^{1}, while the one for urethane carbonyl (–O–CO–NH–) appeared around 1690–1710 cm^{-1}.

In the case of the –NH– stretching band of the poly(urethane urea)s obtained with castor oil, a reduction of the obtained peaks becomes noticeable. This indicates that the strength and number of hydrogen bonds decreases (Fig. 3). This lower amount of hydrogen bonds in the poly(urethane urea) is due to a steric hindrance effect caused by the large amount of dangling chains generated by the usage of castor oil, which blocks the formation of hydrogen bonds [30]. This also leads to a decrease in mechanical properties.

Thermogravimetric analysis

The thermal stability of poly(urethane urea) is influenced by the chemical structure and the ratio of hard to soft segments. The TG data of the poly(urethane urea) are presented in Figs. 4, 5, and 6.

Fig. 4 TG thermograms of poly(urethane urea)s obtained with 2,4-diamino-6-phenyl-1,3,5-triazine as chain extender

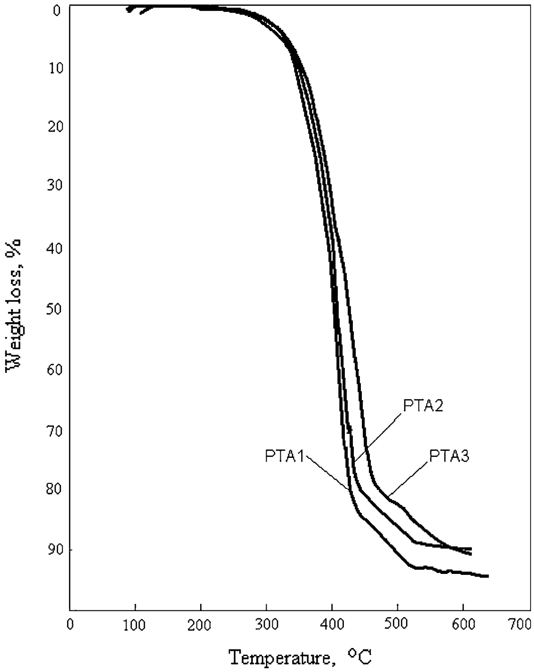


Fig. 5 TG thermograms of poly(urethane urea)s obtained with 2,4-diamino-6-phenyl-1,3,5-triazine and glycerin as chain extenders

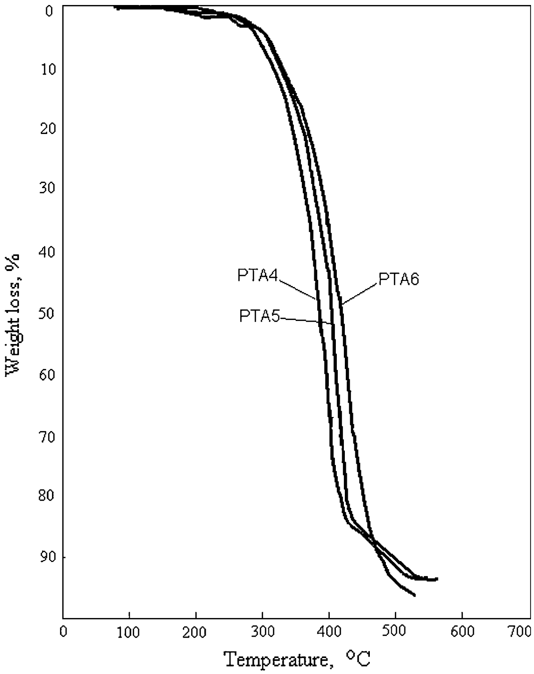
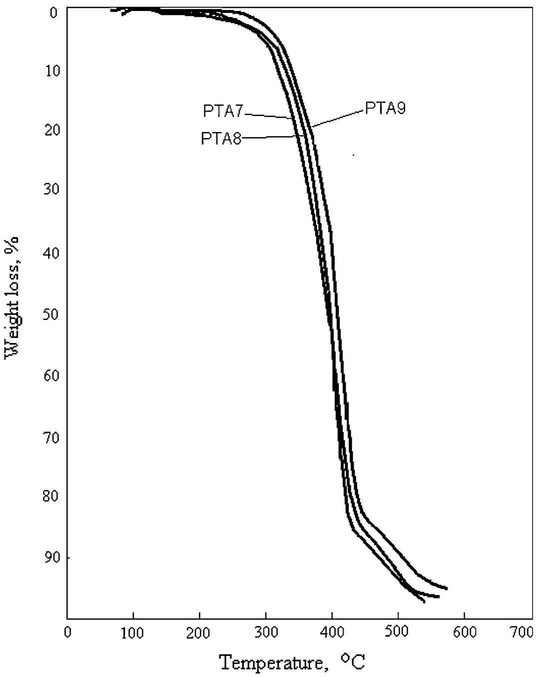


Fig. 6 TG thermograms of poly(urethane urea)s obtained with 2,4-diamino-6-phenyl-1,3,5-triazine and castor oil as chain extenders



The linear poly(urethane urea)s (PTA1–PTA3) start to lose weight at about 290 °C, indicating a good thermo-stability of these polymers compared with the poly(ether urethanes) which were previously studied [18, 31]. The maximum decomposition temperature of linear polymers was observed in the range of 418–430 °C (Fig. 4). Greater difference in weight loss occurs in the final stage of degradation, in which the decomposition temperature increases with increasing the amount of triazine moiety. An increase in the triazine content could increase the thermal stability of these poly(urethane urea). This final stage corresponds to the degradation of the triazine rings from the hard segments. This enhanced stability was attributed to the higher H-bonding capacity of urea groups present in the hard segment of linear poly(urethane urea).

The crosslinked poly(urethane urea)s (PTA4–PTA9) present a similar thermal decomposition behavior. The TG traces of poly(urethane urea) obtained with triazine and glycerin are shown in Fig. 5. The thermal stability of these polymers is slightly lower than that of linear poly(urethane urea). This can be explained by the fact that the crosslinked chains restrict the freedom of movement, thus causing a decrease in the number of hydrogen bonds. The same trend is observed in the case of polymers obtained with castor oil, due to the steric hindrance effect of the dangling chains of the castor oil.

Dynamic mechanical analysis

Figures 7, 8, and 9 show the temperature dependencies of the storage modulus (E') and loss tan δ for the poly(urethane urea).

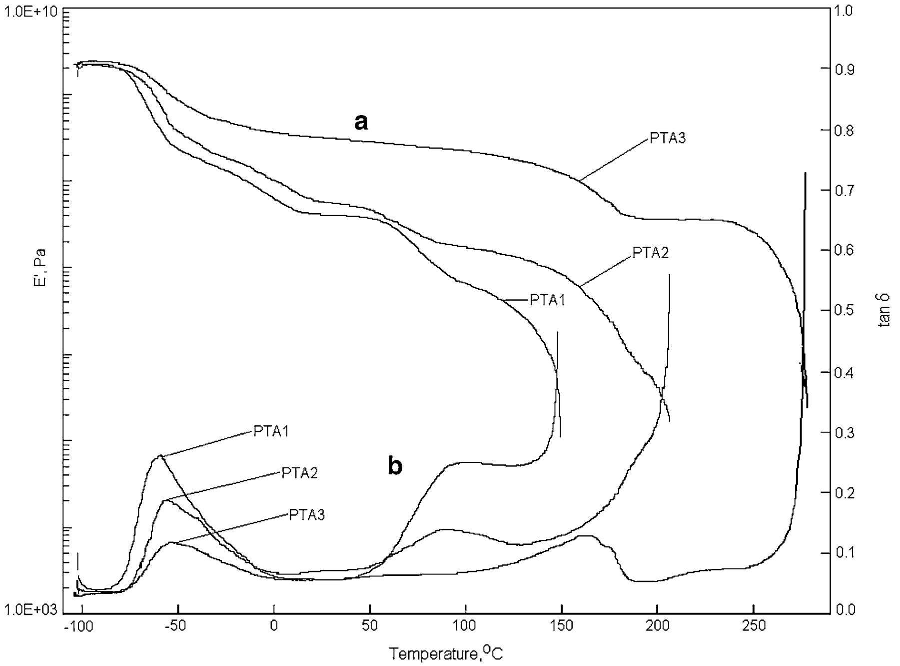


Fig. 7 Storage (E') modulus (*a*) and tan δ peaks (*b*) of the linear poly(urethane urea)s having various hard segment content

Upon increasing the triazine content, the E' of linear poly(urethane urea) increases, due to the higher amount of hydrogen bonds (coming from urea groups) between molecular chains.

The linear poly(urethane urea) had two tan δ peaks, one near −60 °C attributed to the glass transition of soft segments and another at 80 °C which is assigned to the hard segment glass-transition temperature. In the case of the poly(urethane urea) with the largest amount of triazine (PTA3), the second peak is shifted to a higher temperature, 165 °C, respectively. This difference can be attributed to the extension of the phase separation caused by the restriction of chain mobility. With the increase in triazine content, the T_g of the soft segment also shifted from −62 to −58 °C, fact which can be explained by the increased virtual-crosslinking density of the urea groups and decreased mobility of the hard segments. Also, the intensity of tan δ decreases with the increasing amount of triazine, indicating a decrease in amorphous region.

The peak intensity of the poly(urethane urea) obtained with glycerin and castor oil as crosslinkers was higher than that of the linear poly(urethane urea)s because the additional chemical crosslinks increase the rigidity of the poly(urethane urea) hard segments.

Tensile properties

The mechanical properties are a result of the different molecular compositions of the poly(urethane urea). Tensile measurements of all polymers were performed and the results in the form of stress–strain curves were shown in Figs. 10, 11, and 12.

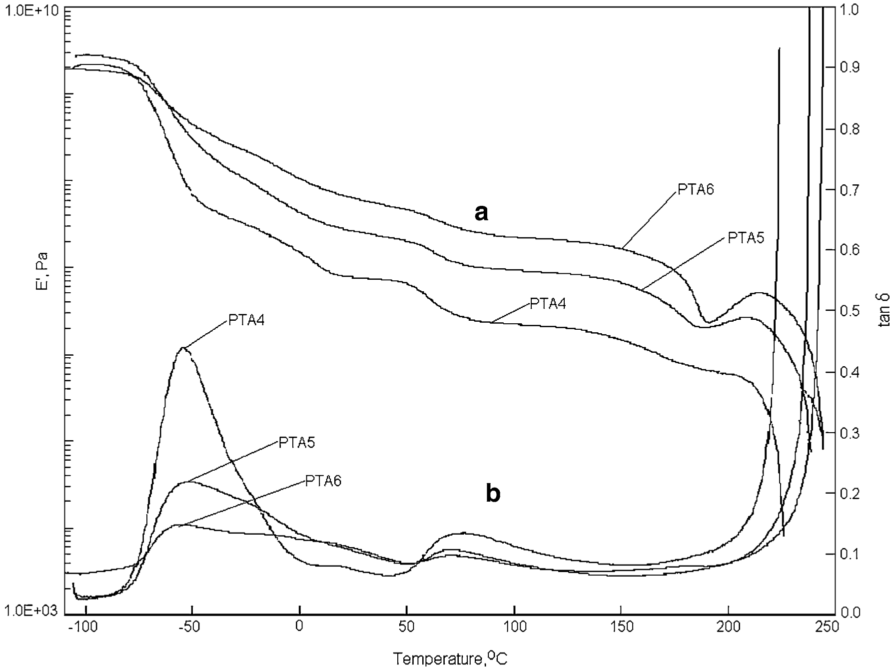


Fig. 8 Storage (E') modulus (*a*) and tan δ peaks (*b*) of the poly(urethane urea)s crosslinked with glycerin having various hard segment content

The tensile strength increased and elongation at break decreased with the increase in triazine content in the poly(urethane urea) formulations. It looks like the heterocyclic triazine groups have a great influence on the tensile strength and elongation at break.

Thus, the tensile strength increased from 8 to 13.5 MPa, while the elongation at break decreased from 890 to 280% with the increase of the molar ratio of polyether/ HDI/ triazine from 1/2/1 to 1/4/3. These results are explained by the powerful increase of intermolecular interactions and hydrogen bonding with the increase of the triazine moiety content in the hard segment of the molecular chains. The improvement of the tensile strength is given by the heterocyclic triazine groups, which are rigid.

The crosslinked poly(urethane urea) showed low elongation at break which varied in the range of 450–300% for poly(urethane urea) obtained with glycerin and between 560 and 200% for poly(urethane urea) obtained with castor oil. Also, tensile strength decreases from 7–9 MPa for poly(urethane urea) with glycerin to 4.8–6 MPa for poly(urethane urea) with castor oil. With the increase of the molar ratio, the degree of chemical crosslinks increases and the intermolecular interactions—including the hydrogen bonding were decreased. For the poly(urethane urea) obtained with castor oil, there is also a decrease in mechanical properties as a result of the large amount of dangling chains from the castor oil, which appear as imperfections in the final product.

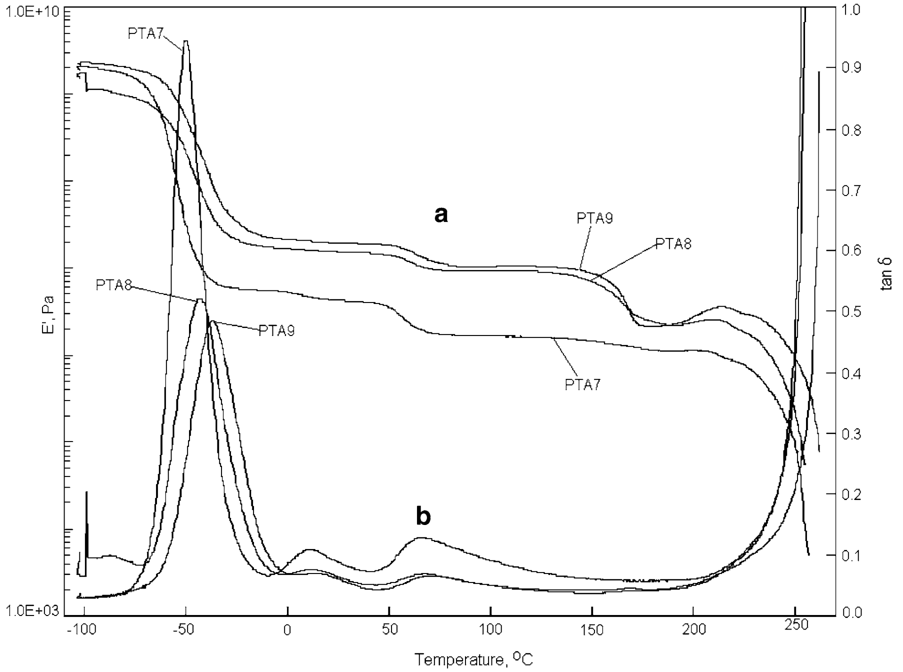
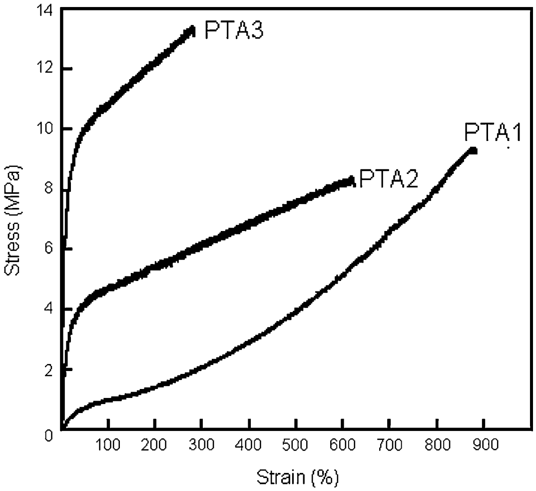


Fig. 9 Storage (E') modulus (*a*) and tan δ peaks (*b*) of the poly(urethane urea)s crosslinked with castor oil having various hard segment content

Fig. 10 Stress–strain curves of linear poly(urethane urea) films with different triazine content



Contact angle analysis

Contact angle is used to determine the hydrophilicity of the film surfaces of the obtained poly(urethane urea) and the results are shown in Table 2.

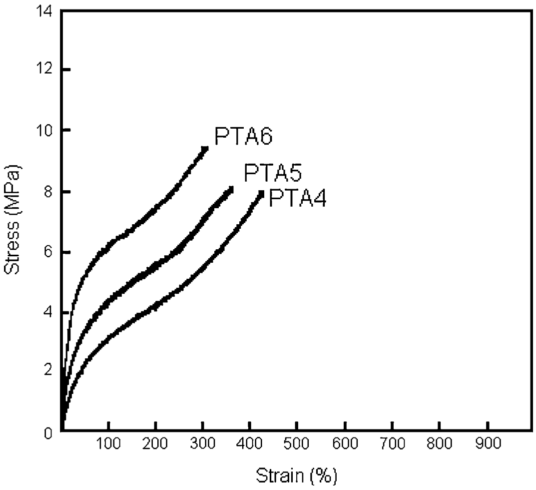


Fig. 11 Stress–strain curves of poly(urethane urea) films obtained with glycerin and different triazine content

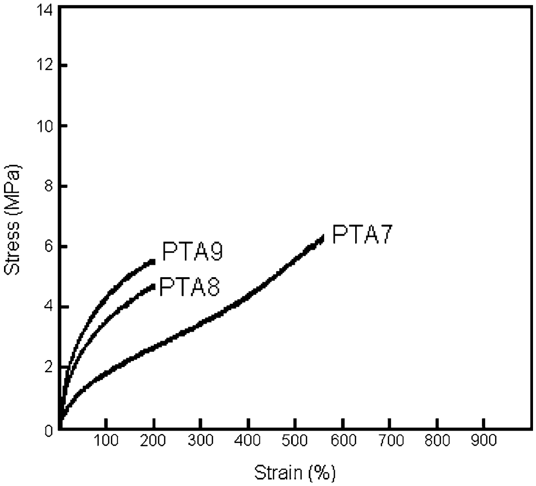


Fig. 12 Stress–strain curves of poly(urethane urea) films obtained with castor oil and different triazine content

The water contact angle of all of the poly(urethane urea) films shows that they are hydrophobic in nature. Contact angle increases with increasing the amount of triazine moiety in the hard segments. Also, contact angle increases with the use of crosslinkers and a maximal value is obtained when using castor oil as a crosslinker. The increase in contact angle of crosslinked poly(urethane urea) indicates that the surface polarity decreases through the formation of a crosslinked structure. For castor oil-crosslinked poly(urethane urea)s, this increase in contact angle must be due to the migration of low-energy dangling chains from the castor oil to the polymer–air interface, thus creating a hydrophobic surface.

The interfacial tensions, as calculated from contact angle data, are presented in Table 3.

Interfacial tensions increase by using crosslinkers and especially by using castor oil. This behavior was probably due to the mobility of the dangling chain from the

Table 2 Contact angle (θ) and work of adhesion (W_a) values of the poly(urethane urea) film surfaces

Sample	Water		Ethylene glycol	
	θ (°)	W_a (mN/m)	θ (°)	W_a (mN/m)
PTA1	90.5	72.2	72.2	62.7
PTA2	91.4	71	79.5	56.7
PTA3	93.6	68.2	81.4	55.1
PTA4	96.3	64.7	77.3	58.5
PTA5	101.9	57.8	78.3	57.7
PTA6	104.7	54.3	84.1	52.9
PTA7	101.8	57.8	79.6	56.7
PTA8	105.8	52.9	82.5	54.3
PTA9	106	52.7	85.9	51.4

Table 3 Interfacial tensions for a solid–liquid system (γ_{sl}) of the poly(urethane urea) films

Sample	λ_{sv}^{p} (mN/m)	λ_{sv}^{d} (mN/m)	γ_{sl} (mN/m)	
			Water	Ethylene glycol
PTA1	7	13.5	21.2	5.8
PTA2	10.5	6.9	19.3	8.8
PTA3	9.2	7.1	20.9	9.1
PTA4	4.3	14	26.4	7.8
PTA5	1.3	19.6	35.9	11.2
PTA6	1.5	15.2	35.3	11.8
PTA7	1.7	17.7	34.3	10.7
PTA8	0.7	18.7	39.3	13.2
PTA9	1.4	14.4	36	12.5

p polar, *d* disperse

films' surface. Poly(urethane urea) surfaces are dominated by hydrophobic hard segments. Changes in the chemical structure of the hard segments lead to changes of the poly(urethane urea) films' surfaces.

Conclusions

A series of new poly(urethane urea) were synthesized via the reaction of isocyanate-terminated polyurethane prepolymer with 2,4-diamino-6-phenyl-1,3,5-triazine as chain extender and glycerin or castor oil as crosslinkers.

Introducing 2,4-diamino-triazine derivatives in the backbone chain improves the thermal and mechanical properties because of the higher degree of inter-chain hydrogen bonds. The tensile strength increased by increasing the content of triazine moiety, but elongation at break decreased greatly.

The surface films properties of the elastomer are strongly dependent on the relative composition of the main chain with triazine moieties.

Due to their good properties and due to the specific characteristics of the triazine moiety, these poly(urethane urea) can be useful in many potential applications.

Acknowledgment The author gratefully acknowledges Dr. Mariana Cristea for her assistance with the DMA measurements.

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