

# Synthesis and Properties of Novel Polyurethane-Urea Insulating Coatings from Hydroxyl-Terminated Prepolymers and Blocked Isocyanate Curing Agent

Hamid Reza Moeini

Research Center, Kimia Co., Bandar Imam, Mahshahr, Iran

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**ABSTRACT:** A novel series of one-pack solvent borne polyurethane-urea insulating coatings was prepared from hydroxyl-terminated prepolymers (HTP) and blocked isocyanate curing agent (BIC). HTP was prepared from poly(*z*(tetramethylene ether)glycol (PTMEG) with excess amount of toluene diisocyanate (TDI) and subsequent reaction of NCO-terminated polyurethane with tris(hydroxymethyl-aminomethane) (TRIS). BIC was prepared from the reaction of trimethylol propane, TDI, and *N*-methyl aniline. HTP, BIC, and final products were characterized by conventional methods, and the curing condition was optimized via gel content measurements. Crosslink density of samples was determined via equilibrium swelling method,

using Flory-Rehner equations. Thermal, mechanical, and electrical properties as well as the chemical resistance of prepared coatings were evaluated and compared with commercially available formulations. Effects of structural parameters on physical, electrical, mechanical, and dynamic mechanical (DMTA) properties of the polyurethane-urea coatings were investigated. The investigation of results showed superior electrical and mechanical properties of prepared green coating as a tailor-made electrical insulator for metals. © 2009 Wiley Periodicals, Inc. *J Appl Polym Sci* 112: 3714–3720, 2009

**Key words:** polyurethane; polyurea; insulating coating; poly(tetramethylene ether)glycol; blocked isocyanate

## INTRODUCTION

Polymer coatings on metal have been playing an important role in various technological applications including winding wires. These coatings are of various kinds of polyurethane, polyesterimide, polyamideimide, polyester, polyvinyl formal, and so on.<sup>1–12</sup> Because of these special properties, polyurethanes have found a wide range of applications as insulating materials in electrical industry. Coating of magnet wire with polyurethane varnishes is one of the most important uses of polyurethane-insulating materials. The property, which has led to the popularity of polyurethane-enameled magnet wire, is solderability at 360–420°C without the necessity of fluxing or stripping. Another feature of polyurethane-insulated wire is its superior *Q* value (reciprocal of dissipation factor).<sup>13–15</sup> Desired levels of electrical insulation, environmental stability, and chemical resistance are all a function of the raw material building blocks chosen to formulate the polyurethane. Although the choice of isocyanate used will have various effects on the final performance of

the polyurethane, but so far, the higher comes from the polyols. The polyol backbone can be completely hydrocarbon, polyether, polyester, or silicon in nature. Each has strong points, but rarely they can completely fulfill specific application requirements.<sup>16</sup>

PTMEG is one of the most important soft segments used for the preparation of different polyurethane products. Some applications of these polyols can be categorized as (1) thermoplastic and thermoset polyurethane elastomers,<sup>17–19</sup> (2) polyurethane adhesives,<sup>20</sup> (3) cast polyurethane elastomers,<sup>21</sup> and (4) polymer blends.<sup>22,23</sup> This polyol provides (a) excellent dynamic mechanical properties over a wide temperature range, (b) exceptional low temperature behavior, (c) superior hydrolytic stability, (d) excellent abrasion resistance, and (e) high resistance to microbes.

Considering these physical and chemical properties, PTMEG has more advantages over competitive polyols materials.

On the other hand, because of some advantages such as storage stability, nonsensitivity to moisture, ability to control cure rate and temperature, and different solubility behavior, blocked isocyanates have found various applications in coating industry. These applications include powder coatings, coil coatings, electrodeposition coatings, automotive coatings, insulating coating for wire, coatings for plastics, and so on.<sup>24,25</sup>

Correspondence to: H. R. Moeini (hamidmoeini@gmail.com).

TABLE I  
Different Formulations of PUUI-II

Sample code	PTMEG (g)	TDI (g)	TRIS (g)	Molar ratio	Av. OH eq. weight <sup>a</sup>	$M_n$ (Theo.)	$M_n$ (Exp.)
PUU-I	50	17.4	12.114	1 : 2 : 2	265.33	3905	4360
PUU-II	50	13.05	6.057	2 : 3 : 2	461.00	6231	6950

<sup>a</sup> Average OH eq. weight =  $(n_1M_1 + n_2M_2 + n_3M_3)/[2(n_1 - n_2 + 2n_3)]$ , where  $n_1$ ,  $n_2$ , and  $n_3$  and  $M_1$ ,  $M_2$ , and  $M_3$  are the molar ratio and the molecular weight of PTMEG1000, TDI, and TRIS.

In this work, novel kinds of modified polyether-based polyols were prepared via reaction of one equivalent of PTMEG with excess amounts of toluene diisocyanate (TDI) and subsequent capping of the intermediate NCO-terminated polyurethanes with tris(hydroxymethyl-aminomethane). In the second step, a blocked triisocyanate was prepared via reaction of TMP with TDI and capping with NMA. These compounds were used as starting materials for the preparation of one-pack polyurethane coatings (ASTM Type-III polyurethane coatings). The synthesis and characterization of raw materials and final polyurethane coatings as well as physical, chemical, mechanical, and electrical properties of products were investigated.

## EXPERIMENTAL

### Materials

Poly(tetramethylene ether)polyol, PTMEG (molecular weight 1000), and tris(hydroxymethyl-aminomethane), TRIS (Aldrich Company, St. Louis, MO), were dried under vacuum at 80°C for 24 h. TDI (mixture of 80% 2,4-isomer and 20% 2,6-isomer) from Merck was vacuum-distilled before use. Dimethyl formamide (DMF) was distilled under vacuum over CaH<sub>2</sub> and kept over 4-Å molecular sieves. Xylene was refluxed over sodium wire and then distilled and kept over 4-Å molecular sieves. Other materials and solvents were used as received.

### Characterization

FTIR spectra were obtained on a Bruker IF 548 instrument. <sup>1</sup>H-NMR spectra were recorded on a Bruker instrument, model AVANCE DR X500, in CDCl<sub>3</sub> as solvent. Thermal gravimetric analysis (TGA) was performed on a Stanton STA780 with heating rate of 10°C/min in air atmosphere. Tensile properties were determined from stress-strain curves with MTS tensile-tester model 10/M at a strain rate of 5 mm/min. The measurements were performed at 25°C with a film thickness of about 1 mm and stamped out with an ASTM D-638 Die. Dynamic mechanical testings (DMTA) were carried out on a UK polymer lab dynamic mechanical thermal analyzer model MKII over a temperature range

of -100 to 300°C at a heating rate of 10°C/min and frequency of 1 Hz. The dimensions of samples were 30 mm × 10 mm × 1 mm. Molecular weight measurements were performed on a gel permeation chromatography instrument (GPC) model Waters 150 C in tetrahydrofuran (THF) as solvent. Evaluation of breakdown voltage was performed on a high-voltage kite with 2 kV/S voltage increasing rate and a spherical electrode of 5 cm diameter and 50 Hz frequency. Dissipation factor and dielectric constant measurements were performed on Schering bridge device Tettexag 2801 ZQ. Samples were cut into circular-shape pieces of 5 cm diameter. The measurements were performed according to ASTM 149-94 and ASTM 150-94 under air atmosphere at 25°C. Hardness of samples was evaluated with a pendulum hardness tester using ERICHSEN instrument according to ASTM D-4366-94. Pencil hardness of samples was evaluated according to ASTM D-3363-92a. Adherence of coating to copper wire and solderability of coated magnet wire were evaluated according to CEI/IEC Standard, 2000, 60317-0-1 and CEI/IEC Standard, 2000, 60317-2, respectively. Density of samples was measured according to ASTM-D-1817.

### General procedure for the preparation of hydroxyl-terminated polyurethane prepolymer

A certain quantity of TDI was placed in a polymerization kettle equipped with a mechanical stirrer, a reflux condenser, and dry nitrogen inlet and outlet. A calculated amount of PTMEG with molecular weight of 1000 g/mol was added into the kettle. The mixture was kept at room temperature, and then gradually heated up to 85°C. The reaction was continued until the NCO content reached the theoretical value (as determined by dibutylamine titration). After cooling the reaction mixture to room temperature, TRIS was added portionwise. The reaction was continued until the NCO peak at 2270 cm<sup>-1</sup> in the FTIR spectra of sample taken from the reaction mixture disappeared.

Different formulations are collected in Table I.

### Synthesis of BIC curing agent

In a four-necked glass reactor equipped with mechanical stirrer, reflux condenser, dropping funnel,

and N<sub>2</sub> inlet and outlet, 3 equiv of TDI (26.1 g, 0.15 mol) was placed. Then, a solution of 1 equiv of trimethylol propane (TMP, 6.74 g, 0.05 mol) dissolved in minimum amount of DMF was added dropwise to the reaction mixture. The reaction kettle was maintained at 45°C for 2 h and then cooled to room temperature. Three equivalents of *N*-methyl aniline (NMA, 16.4 g, 0.15 mol) was added to the reactor dropwise. The temperature was kept at 45°C, and the reaction was continued until all the NCO groups were reacted. The end of reaction was detected by disappearance of NCO peak at 2270 cm<sup>-1</sup> in the FTIR spectrum of reaction mixture. The content of reactor was poured into the excess amount of distilled water, and the precipitated white powder was filtered, washed with water, and dried in a vacuum oven at ambient temperature.

### Film casting

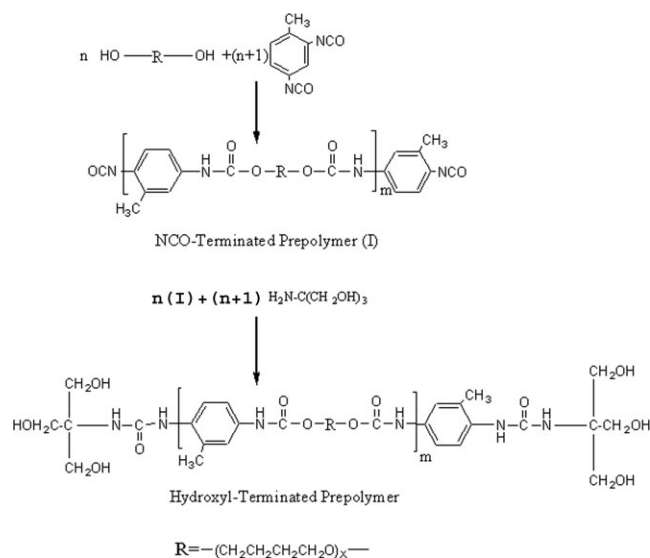
A 50% solid content solution of hydroxyl-terminated urethane prepolymer and *N*-methyl aniline-blocked triisocyanate in DMF/xylene (1/3 w/w) solvents mixture was prepared in equivalent ratio of NCO and OH groups. Thin layer of solution was spread on a Teflon mold and heated at 180°C for 30 min. The optimum temperature and time to reach maximum crosslinking was obtained via gel content measurements.

### Enamel wire coating

A 50% (w/w) solid content solution of PUU and blocked isocyanate curing agent (BIC) in DMF/xylene (1/3 w/w) solvents mixture was prepared, in such a way that equivalent ratio of NCO and OH groups was present in the mixture. Then, an annealed, degreased copper wire (0.5 mm in diameter and 20 cm long) was dipped into the varnish bath and pulled through a small die (0.58 mm in diameter) at room temperature. Then, coated wire was placed in an air-circulated oven and heated at proper temperature and time. This procedure was repeated four times with the die slightly enlarged each time (0.62, 0.63, 0.65, and 0.66 mm). The resulting polyurethane enamel wire had a diameter of 0.64 mm.

## RESULTS AND DISCUSSION

The preparation of polyurethane-urea was conducted in two stages. At the first stage, the prepolymers (hydroxyl-terminated prepolymers, HTP) were prepared. The synthetic route for the preparation of functional polyurethane polyols with average six-end hydroxyl groups is outlined in Scheme 1. The reaction of poly(tetramethylene ether)polyol with



**Scheme 1** Synthetic route used to prepare HTP.

excess amount of TDI leads to NCO-terminated polyurethane prepolymers, which subsequently reacted with TRIS to prepare hydroxyl-terminated polyurethane prepolymers at the second stage. The molecular weight of prepared HTP depends on PTMEG1000/TDI molar ratio. The molecular weight increased as this ratio approaches to unity. The amount of average OH equivalent weight of HTP was also depending on the whole molecular weight of prepolymers.

Poly(tetramethylene ether) represents a noncrosslinkable soft segment in HTP backbone, so increasing the molecular weight of HTP and subsequent increase of average OH equivalent weight, decreases the hydroxyl group density of prepolymers, which are main crosslinking sites. This variable (crosslink density) gave a versatile means for controlling the properties of final polyurethane coatings properties. Different formulations of HTP as well as their characteristic data are collected in Table I. There is good agreement between the experimental molecular weight of HTP obtained from GPC analysis with the theoretical value of molecular weight obtained from Carother's equation.<sup>26</sup>

FTIR spectra of PUUI-II compounds showed a broad peak at ca. 3420 cm<sup>-1</sup> relating to urethane N-H stretching. The peak at ca. 3290 related to O-H stretching. Symmetrical stretching vibration of methylene groups in the backbone of polyols appeared at 2857 cm<sup>-1</sup>, and the unsymmetrical stretching vibration of these groups appeared at about 2940 cm<sup>-1</sup>. NCO groups have strong peak at ca. 2272 cm<sup>-1</sup>. Aromatic C-C has a peak at ca. 1594 cm<sup>-1</sup>. C-N stretching and NH bending are at ca. 1542 cm<sup>-1</sup>. The strong peak at 1731 cm<sup>-1</sup> is because of the combination of urethane NH-CO. The urea

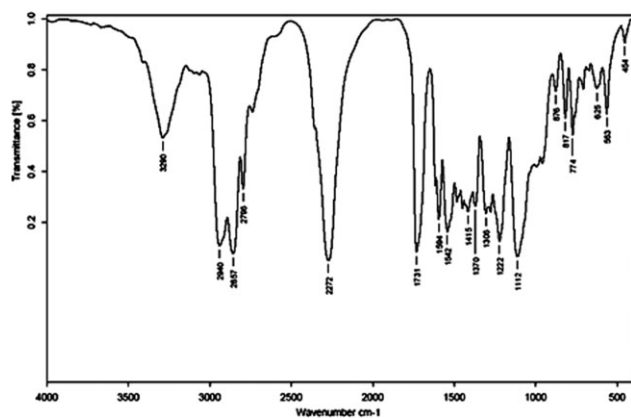


Figure 1 FTIR spectrum of PUU-I.

peak appeared at ca.  $1668\text{ cm}^{-1}$ . Esteric C=O stretching appeared at  $1222\text{ cm}^{-1}$ , and stretching vibration of polyol ether bonds appeared at ca.  $1112\text{ cm}^{-1}$ . Representative FTIR spectrum of PUU-I is shown in Figure 1.

$^1\text{H-NMR}$  spectra of PUUI-II polyols show 1.49–1.78 (internal methylene groups of PTMEG), the methyl groups from TDI showed a singlet at 2.10 ppm, 2.35 (methylene groups of  $-\text{CH}_2\text{CO}-\text{O}-$ ), 3.35 (methylene groups of  $-\text{CH}_2-\text{OH}$ ), 4.05–4.15 (methylene groups of  $-\text{CH}_2\text{O}-\text{CONH}-$ ), 6.87–7.13 (aromatic C–H), 7.25 (urethane N–H). Representative  $^1\text{H-NMR}$  spectrum of PUU-I is shown in Figure 2.

The synthetic route for the preparation of BIC is outlined in Scheme 2. The reaction of 1 equiv TMP with 3 equiv TDI leads to an intermediate compound (PI), which subsequently reacted with 3 equiv of NMA to give BIC. The use of unsymmetrical diisocyanate molecules containing isocyanate groups with different reactivity such as TDI help to prepare PI intermediate without the risk of gel formation or molecular weight build up.

FTIR spectrum of BIC showed a peak at  $1729\text{ cm}^{-1}$  related to urethane carbonyl (NHCOO) and an

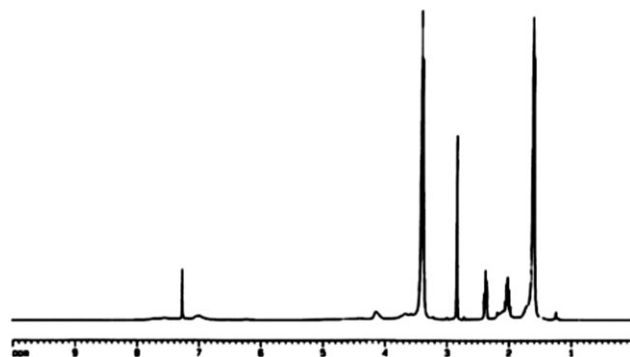
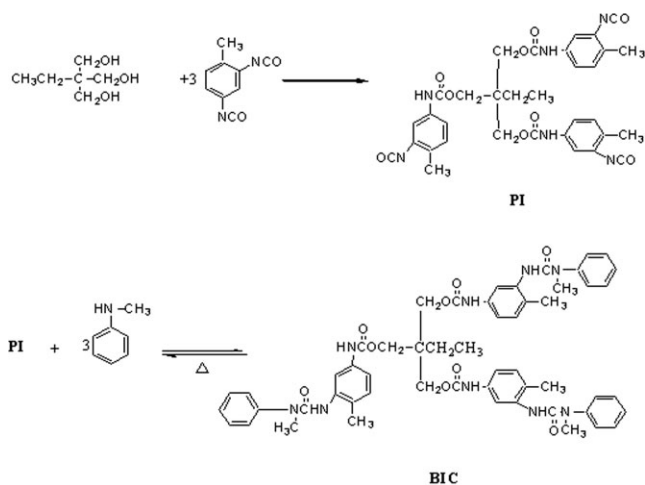


Figure 2  $^1\text{H-NMR}$  spectrum of PUU-I.



Scheme 2 Synthetic route used to prepare BIC.

intensive peak at  $1671\text{ cm}^{-1}$  related to urea carbonyl (NHCONH) groups, respectively. A peak at  $3272\text{ cm}^{-1}$  was related to hydrogen-bonded urea/urethane N–H bonds, and another one at  $3436\text{ cm}^{-1}$  was related to nonhydrogen-bonded urea/urethane N–H bonds.

$^1\text{H-NMR}$  spectrum of BIC showed two distinct triplet and quartet peaks at 0.91–0.94 ppm and 1.49–1.52 ppm due to ethyl group protons of TMP part. The methyl groups from TDI showed a singlet at 2.10 ppm and methyl groups from NMA showed a singlet at 2.40 ppm. The methylene groups attached to urethane linkage showed a singlet at 4.15 ppm. The aromatic C–H bond appeared as a multiplet at 6.76–7.80 ppm, and urea and urethane protons appeared at 6.1 and 8.02 ppm, respectively.

In blocked isocyanates chemistry, the charge difference between carbonyl carbon and blocking agent is of great importance. The greater the charge difference, the greater will be the strength of the bond, and if the blocking agent is less nucleophilic, the negative charge density at the blocking agent will be less, which makes the bond more liable. Such a charge separation is apparently more in the NMA-blocked system in comparison with phenol-blocked system; the methyl group intensifies the negative charge.<sup>27,28</sup> Hence, deblocking is expected to be difficult in NMA-blocked system. However, it was found that NMA-blocked isocyanate was deblocked at much lower time and temperature. A possible explanation to this phenomenon is that the tertiary nitrogen present in the NMA-blocked isocyanate autocatalyzes the deblocking through the formation of an intermolecular four-centered complex, and hence, reduces the reaction temperature and time. Another feature of NMA-blocked polyisocyanate is that released amine will catalyze urethane formation in the subsequent reactions.

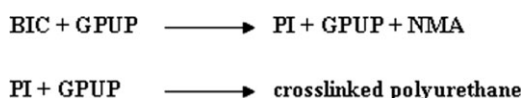
TABLE II  
Composition of Final Formulations for the Preparation of Cured Samples

Sample code	Polyol	NCO/OH	Polyol (g)	Curing agent (g)	Xylene (g)	DMF (g)
XPUU-1	PUU-I	1/1	1	1.227	1.670	0.557
XPUU-2	PUU-I	1.1/1	1	1.349	1.761	0.587
XPUU-3	PUU-II	1/1	1	0.706	1.279	0.427
XPUU-4	PUU-II	1.1/1	1	0.777	1.333	0.444

The BIC was used as curing agent for HTP (Table II). Upon heating, deblocking of isocyanate groups of BIC occurred and PI with free isocyanate groups regenerated. Subsequent reaction of isocyanate groups of PI with free hydroxyl groups of HTP led to crosslinked polyurethanes (Scheme 3). FTIR spectra of cured samples showed characteristic peak of urethane carbonyl at  $1705\text{ cm}^{-1}$ , which was mixed with etheric carbonyl of PTMEG polyol. Urethane N—H stretching bond appeared at  $3325\text{ cm}^{-1}$ . Combination of C—N stretching and N—H out-of-plane bending appeared as a peak at  $1526\text{ cm}^{-1}$ . Esteric C—O groups showed a stretching peak as a doublet at  $1053$  and  $1209\text{ cm}^{-1}$ . There were no sign of free isocyanate peak at  $2270\text{ cm}^{-1}$ , which confirmed the complete conversion of curing agent.

The optimum temperature and time to reach maximum crosslinking was obtained via gel content measurements.<sup>29</sup> Gel content measurements were performed in a Soxhelt extractor using acetone as solvent. The measurements were conducted for polyurethane products obtained from different heating times (20, 40, 50, and 60 min) at different temperatures (150, 180, and  $210^\circ\text{C}$ ). The investigation of results showed that at  $150^\circ\text{C}$ , insufficient degree of crosslinking was obtained. At  $210^\circ\text{C}$ , although high degree of crosslinking was achieved at lower time, however, after passing through definite time, degradation of samples occurred. Also, all the samples obtained at  $210^\circ\text{C}$  showed some degree of discoloration. The best curing condition (higher gel content at minimum possible time without deterioration of samples) was obtained at  $180^\circ\text{C}$  for 30 min heating.

BIC was used as the curing agent for PUU (Table II). Upon heating, deblocking of isocyanate groups of BIC occurred and PI with free isocyanate groups was regenerated. Subsequent reaction of isocyanate groups of PI with free hydroxyl groups of PUU led to crosslinked polyurethanes (Scheme 3).



Scheme 3 Curing reaction lead to crosslinked polyurethane product.

For increasing the gel content, excess amount of curing agent was used. This phenomenon is a result of extra allophanate bond formation via reaction of isocyanate groups of curing agent with urethane N—H groups of HTP. These samples (XPUU-2 and XPUU-4) showed decreased flexibility.

Determination of crosslink density was performed via equilibrium-swelling method according to the Flory-Rehner equation.<sup>30–33</sup> These results are shown in Table III. Investigation of these data showed that increasing of molecular weight of HTP, resulted in the reduction of hydroxyl value of HTP and consequently decreasing amount of available hydroxyl groups in a definite mass of HTP. The amount of curing agent is dependent on the chain length of HTP. The larger the chain length (larger the value for average OH equivalent weight of HTP), the less curing agent is needed. This is due to the fact that the terminal OH groups are the main sites of crosslinking. Therefore crosslink density of samples decreases with the increase of hydroxyl value. Also,  $M_c$ , which is a measure of average molecular weight between crosslinked points, increased with the decrease of hydroxyl value of HTP. The measurements showed that the use of excess amount of isocyanate curing agent results in the increase of crosslink density in comparison with the samples obtained via equivalent ratio of polyol and curing agent.

Mechanical property of the prepared films (XPUU) was determined by the analysis of stress–

TABLE III  
Crosslink Density and Molecular Weight Between Crosslinked Point Data Obtained from the Reaction of Equivalent or Excess Amount of Isocyanate Curing Agent with HTP

Sample ID	$V_p$	$d_p$ ( $\text{g}/\text{cm}^3$ )	$M_c$ (g/mol)	$v_c$ ( $\times 10^3\text{ mol}/\text{m}^3$ )
XPUU-1	0.5012	1.605	616.06	2.605
XPUU-2	0.6145	1.607	516.09	3.114
XPUU-3	0.3324	1.593	1113.405	1.431
XPUU-4	0.3516	1.598	1021.578	1.564

$V_p$ , volume fraction of polymer in the swollen state;  $d_p$ , density of polymer;  $M_c$ , average molecular weight between two crosslinks;  $v_c$ , crosslink density.

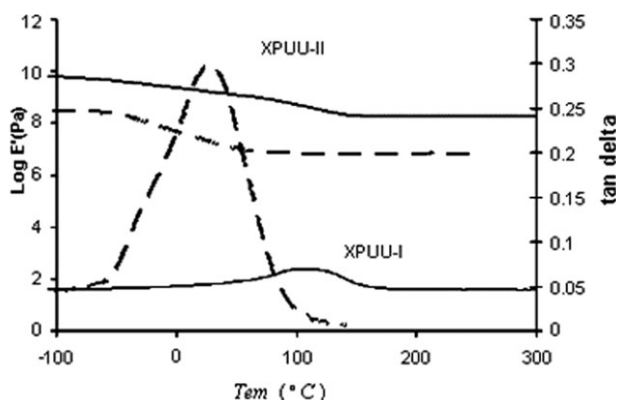
**TABLE IV**  
**Mechanical and Thermal Properties of Polyurethane Samples Obtained from the Reaction of Equivalent Amount of PUU and Isocyanate Curing Agent**

Sample ID	Tensile strength (MPa)	Elongation at break (%)	Modulus (MPa)	Pendulum hardness (s)	Pencil hardness	$T_{10\%}$ (°C)	$T_{50\%}$ (°C)	Char yield %
XPUU-1	10.92	34.13	302.616	85 ± 2	HB	271.5	450	7.33
XPUU-3	5.96	45.97	42.67	35 ± 2	B	250.6	448	5.33

$T_{10\%}$ : temperature of 10% weight loss;  $T_{50\%}$ : temperature of 50% weight loss; Char yield %: weight percent of remained polymer at 600°C.

strain curves, and tests data are collected in Table IV. According to these results, with increasing crosslink density, decreasing of the PTMEG molar ratio, tensile strength of XPUUs and modulus of samples were increased, and in turn with increasing the molar ratio of PTMEG, elongation at break of samples was increased. The increase of soft segment content renders the crosslinked films easy to elongate, and therefore, elongations at break increased. Sample XPUU-1 showed the behavior of a tough elastomers with yield point, followed with necking and drawing, and XPUU-3 displayed a smooth transition in the stress-strain behavior from the elastic to plastic deformation regions similarly to lightly crosslinked weak rubbers. In comparison with other polyurethanes with similar structure,<sup>34-36</sup> decrease in elongation at break was observed. These decreases were attributed to the presence of etheric bonds, which are weakest bonds of the prepared samples.

Hardness of samples was evaluated by two methods: (a) pencil hardness and (b) pendulum hardness. The pendulum-damping test has been found to have good sensitivity in detecting differences in coating hardness, where hardness is defined as the resistance to deformation. It can be concluded that the increase of crosslink density results in the increase of samples hardness, Table IV.



**Figure 3** DMTA curves of prepared cured films.

The  $T_{10\%}$  as a criterion of thermal stability was increased as the crosslink density decreased. This is due to the decrease in the concentration of weaker urethane bonds with the decrease of crosslink density. The study of thermal stability of prepared coatings by TGA method (Table IV) showed that regardless of the type of HTP, all samples undergo spontaneous decomposition at ca. 220°C. The initial decomposition may correspond to aliphatic and etheric bonds, which are the weakest bonds of the prepared samples. The urethane linkage decomposes at ca. 275°C leading to the formation of carbon dioxide, alcohols, and carbon monoxides.

Examination of DMTA (Fig. 3) curves of prepared samples showed that modulus of the plateau region increased with increasing crosslink density. Also, as crosslink density increased,  $\alpha$ -type transition peak at  $\tan \delta$  curves shifted to higher temperature with the decrease of HTP molecular weight. This is attributed to the restriction in the segmental mobility of the polymer backbone. Sharpness of  $\tan \delta$  peaks, which is a criterion of phase separation, decreased with decreasing HTP molecular weight (i.e., increasing of crosslink density).<sup>37</sup>

Results of evaluation of electrical properties are collected in Table V. In all electrical applications, it is desirable to keep the electrical losses at a minimum value. Electrical losses indicate the inefficiency of an insulator. The dissipation factor (DF) is a measure of such electrical inefficiency of insulating material. Investigation of data indicated that the DF value of prepared polymers were in the range of ( $5.84 \times 10^{-5}$ – $12.25 \times 10^{-5}$ ), and by increasing the crosslink density, the value of DF decreased. This is due to the fact that reactive polar groups are either

**TABLE V**  
**Electrical Properties of Samples**

Sample	Thickness (mm)	Dissipation factor (DF) $\times 10^{-5}$	Dielectric constant (60 Hz)	Dielectric strength (V/cm)
XPUU-1	0.65	5.96	2.64	29.38
XPUU-3	0.71	12.25	5.93	26.06

neutralized or hindered by crosslinking.<sup>38,39</sup> Dielectric constant indicates the ability of an insulator to store electrical energy. So, it is desirable to have the capacitance of the insulating material as minimum as possible. The dielectric constant of prepared samples was in the range of 2.64–5.93, which is superior to common polyurethane insulator. Dielectric strength indicates electrical strength of a material as an insulator. The higher the dielectric strength, the better is the quality of an insulator. The measured value of ca. 29 V/cm indicated the excellent insulating power of prepared polyurethane.

One desirable characteristic of polyurethane wire enamels is the adherence of coating to magnet wire. The samples showed good adherence to copper wire. On the other hand, flaking of samples upon stretching was not observed.

Another important characteristic of polyurethane wire enamel is solderability without stripping. Solderability is a critical issue in automated processes. Measuring this factor for prepared samples indicated that samples exhibited acceptable level of solderability at 385°C for 2 s, which was examined via visual inspection of wire. The accepted specimens showed a continuous film of solder completely wetting the wire.

Investigation of chemical resistance by normal vision inspection of any change in the appearance or loss of film integrity in different media confirmed that the samples showed excellent chemical resistance in acidic (H<sub>2</sub>SO<sub>4</sub>, 10%) and basic (NaOH, 1%) media. However, the samples underwent some degree of swelling in toluene and DMF media.<sup>40</sup> Samples with higher crosslinking density showed better electrical properties.

## CONCLUSION

Novel kind of one-component polyurethane insulating coating was prepared via the reaction of polyurethane polyols with blocked isocyanate curing agent. The evaluation of properties of final coatings showed that the prepared samples exhibited the combination of excellent electrical insulation characteristics, chemical resistance, and crosslink density, which resulted in a less crosslinked structures and consequently higher  $M_c$  and lower  $v_c$  values and hardness properties in comparison with other polyurethanes with similar structures such as that prepared from poly(propylene glycol) and TDI<sup>11,12</sup> and commercially available samples.<sup>41</sup>

## References

- Honma, H.; Kogo, Y. Jpn Pat. JP 07,165,873 (1995).
- Wen-Yen, C.; Wen-Chang, C. J Appl Polym Sci 1988, 35, 1433.
- Chiang, W. Y.; Chiang, W. C. J Appl Polym Sci 1988, 35, 1421.
- Shen, D. C. U.S. Pat. 4,362,861 (1982).
- Cicero, R.; Wasaki, T. I. U.S. Pat. 4,997,891 (1991).
- Harber, J. J. U.S. Pat. 4,808,477 (1989).
- Lee, W. F.; Chen, I.; Chang, C. H. J Appl Polym Sci 1993, 50, 259.
- Biorcio, L.; Mensi, C. U.S. Pat. 4,526,912 (1985).
- Osada, Y. Jpn Pat. JP 02,024,908 (1990).
- Braun Stein, R.; Schmitt, F. U.S. Pat. 5,756,634 (1998).
- Yang, C. P.; Lee, L. T. J Appl Polym Sci 1988, 35, 449.
- Yang, C. P.; Lee, L. T. J Coat Technol 1987, 59, 61.
- Shugg, T. Handbook of Electrical and Electronic Insulating Materials; Van Nostrand Reinhold Company: New York, 1986; p 179.
- Payette, L. J. IEEE Electr Insul Mag 1990, 6, 8.
- Winkeler, M. IEEE Electr Insul Mag 1991, 7, 61.
- Hegemann, G. Polyurethanes as Insulating Materials in Electrical Applications, Polyurethane World Congress, September 24–26, 1991.
- Huang, Y. J.; Lee, S. C.; Dong, J. P. J Appl Polym Sci 2000, 78, 558.
- Liaw, D. J. J Appl Polym Sci 1997, 66, 1251.
- Sanchez-Adsuar, M. S.; Papon, E.; Villenave, J. J. J Appl Polym Sci 2000, 76, 1590.
- Sanchez-Adsuar, M. S.; Pastor-Blas, M. M.; Torregrosa-Macia, R.; Martin-Martinez, J. M. Int J Adhes Adhesives 1994, 14, 193.
- House, D. W.; Banmann, W. M.; Scott, R. V. J Elast Plast 1987, 19, 252.
- Chen, H. L.; Wang, S. F.; Lin, T. L. Macromolecules 1998, 31, 8924.
- Chen, H. L.; Liaw, D. J.; Liaw, B. Y.; Shin, C. L. Polym J 1998, 30, 874.
- Wicks, D. A.; Wicks, Z. W. Prog Org Coat 2001, 41, 1.
- Wicks, D. A.; Wicks, Z. W. Prog Org Coat 1999, 36, 148.
- Odian, G. Principle of Polymerization; Wiley: Hoboken, NJ, 2004.
- Sultan Nasar, A. J Polym Sci Part A: Polym Chem 1999, 37, 1815.
- Gnanarajan, P. J Polym Sci Part A: Polym Chem 2000, 8, 4032.
- Lee, W. F. J Appl Polym Sci 1993, 50, 59.
- Downey, W. J.; Brauer, M.; Chao, J. C. U.S. Pat. 466,968 (1987).
- Semsarzadeh, M. A.; Navarchian, A. H. J Appl Polym Sci 2003, 90, 963.
- Bharadwaj, V. J Macromol Sci Pure Appl Chem 2002, 39, 115.
- Ebdon, J. R. Polymer 1984, 25, 1633.
- Yeganeh, H.; Shamekhi, M. A. Polym Int 2005, 54, 754.
- Yeganeh, H.; Moeini, H. R. High Perform Polym 2007, 19, 113.
- Moeini, H. R. J Appl Polym Sci 2007, 106, 1853.
- Ng, H. N.; Allegrezza, A. E.; Seymour, R. W.; Cooper, S. L. Polymer 1973, 14, 255.
- Blythe, A. R. Electrical Properties of Polymers; Cambridge University Press: Cambridge, 1979.
- Klug, R. S. IEEE Electr Insul Mag 1988, 4, 24.
- JIS-Standard. JIS-C-2358. 1969.
- Licari, J. J.; Hughes, L. A. Handbook of Polymer Coatings for Electronics Applications, Chemistry, Technology and Applications; Noyes Publications: NJ, 1990.