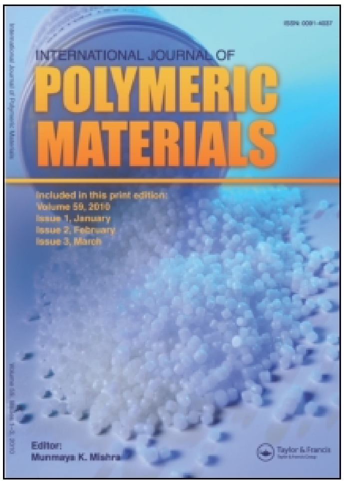

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K. B. Patel[a][b]; K. R. Desai[a]; H. S. Patel[c]
[a] Department of Chemistry, South Gujarat University, Surat, Gujarat, India [b] Gujarat Narmada Valley Fertilizers Co. Ltd., Dist. Bharuch, Gujarat, India [c] Department of Chemistry, Sardar Patel University, Vallabh Vidyanagar, Gujarat, India

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Poly(Urethane-Urea)s-Part-5

K. B. PATEL[a,*], K. R. DESAI[a,†] and H. S. PATEL[b]

[a] *Department of Chemistry, South Gujarat University, Surat, Gujarat, India;* [b] *Department of Chemistry, Sardar Patel University, Vallabh Vidyanagar, Gujarat, India*

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Four Different poly(urethane-urea)s (PUUs) were prepared by polycondensation of DMU(Dimethylol urea) with various diisocyanates. The resultant poly(urethane-urea)s were characterized by elemental analysis, IR spectral studies, number average molecular weight ($\overline{Mn}$) estimated by non-aqueous conductometric titration and thermogravimetry. The electrical properties of these polymers have also been measured at room temperature.

Keywords: Poly(urethane-urea)s; Diisocyanates; DMU; Molecular weight; Electrical properties

INTRODUCTION

Each of polyureas and polyurethanes are well known candidates for industrial polymer applications [1]. The introduction of both the groups *viz.* urea and urethane into one polymer chain has not yet received much attention. However only few poly(urethane-urea)s are reported from aminoglucose [2] or from mixture of diol, diamine and diisocyanate [3, 4]. As it might give polymers having important properties, such type of polymer research has been pursued by condensation of monomer having both urea and hydroxy groups (*e.g.*, DMU) [5]. Though DMU is well established product for various

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[*] Present address: Gujarat Narmada Valley Fertilizers Co. Ltd., Narmadanagar-392 015, Dist. Bharuch. Gujarat, India.

[†] Corresponding author.

application [1]. Hence the present paper comprises synthesis and characterization of novel poly(urethane–urea)s by the synthetic route furnished in Scheme 1.

EXPERIMENTAL

Materials

All the chemicals used were of analytical grade or laboratory grade.

Synthesis of Poly(Urethane–Urea)s

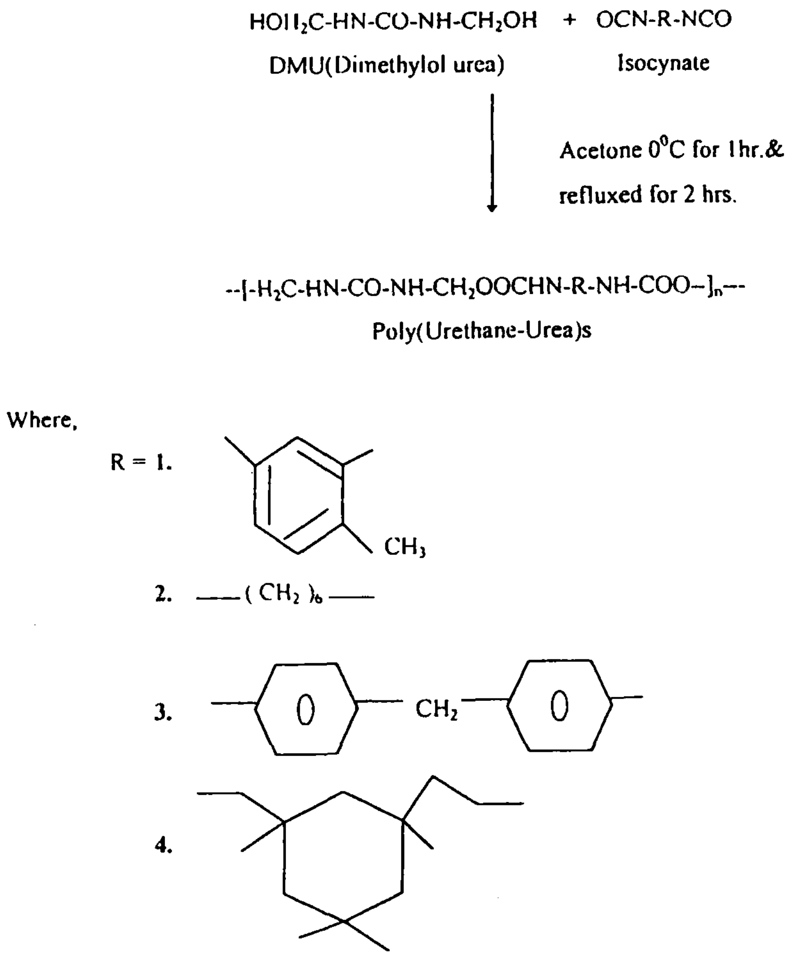
To an ice cooled solution of DMU(0.1 M) in dry acetone(50 ml) a solution of diisocyanate(0.1 M) in 50 ml dry acetone was added gradually with constant stirring and the stirring was continued at room temperature for 1 hour. Then it was refluxed for 2 hours on a water bath. The resulting solid product was then filtered off and washed with acetone and air-dried(95% yield).

Measurements

C, H, N contents of polymers were estimated using C, H, N, O elemental analyser, (Carlo Erba, Italy). The IR spectra of polymers were scanned in KBr pellets on Perkin Elmer 257 spectrophotometer, Number average molecular weights ($\overline{Mn}$) of polymers were estimated by non-aqueous conductometric titration. It was carried out respectively in formic acid (for NH_2 end group) against standard sodium methanolate. Digital conductometer, Toshniwal, India was used for this purpose. The value of molecular weight ($\overline{Mn}$) of all polymer samples were calculated by the reported method [6]. Thermogravimetric analysis for polymers were carried out on Du Pont thermobalace in air at a heating rate of $10°K\,min^{-1}$. The electrical conductivity of each of PUU samples was measured on pellets(1 cm diameter, 0.45 cm thickness) at room temperature (30 ± 1°C) using a Million Megohmeter RM160 MK IIA BPL, India. The preparation of the pellets of all the PUU samples and other details have been conducted as described in [7].

RESULTS AND DISCUSSION

The poly(urethane-urea)s (PUU) formations performed by facile reaction of DMU and diisocyanate. The PUUs shown in the reaction scheme are furnished in Table I. They are dark brown solid powders. They do not melt up to 250°C and insoluble in common organic solvents. Elemental contents C, H, N (Tab. I) of the polymers are consistent with the predicted structure (reaction scheme).



SCHEME 1

TABLE I Elemental analysis of PUUs (1a–d) based on DMU

PUU sample	Mole. formula of repeating unit	Mol. wt of repeating unit	Elemental analysis						Mole wt Mn*	Electrical Conducitivity (σ) at 303 ($\Omega \cdot cm^{-1}$)
			%C		%H		%N			
			Cald	Found	Cald	Found	Cald	Found		
1a	$C_{11}H_{20}N_4O_5$	288	45.83	45.70	6.94	6.80	19.44	19.20	1850	6.9×10^{-12}
1b	$C_{15}H_{20}N_4O_5$	336	53.57	53.20	5.95	5.70	16.67	16.40	2800	8.4×10^{-9}
1c	$C_{12}H_{14}N_4O_5$	294	48.98	48.70	4.76	4.60	19.00	18.80	3000	9.4×10^{-9}
1d	$C_{18}H_{18}N_4O_5$	370	58.38	58.10	4.86	4.60	15.14	15.00	3550	7.8×10^{-12}

*Estimated by non aqeous conductometric titration.

TABLE II Elemental analysis of PUUs (1a–d) based on DMU

PUU	*% wt loss at temp°C*						*% wt loss at 1st step (200–300°C)*	
sample	200	300	400	500	600	700	*Cald*	*Found*
1a	3.00	15.00	37.00	53.00	75.00	92.00	15.30	15.10
1b	2.00	13.00	34.00	52.00	75.00	92.00	13.10	13.40
1c	4.00	15.00	37.00	54.00	75.00	92.00	14.97	15.10
1d	3.00	12.00	32.00	50.00	72.00	90.00	11.90	11.40

IR spectra (not shown) of all the PUUs are identical in nature. They comprise important IR spectral features of urea and urethane linkages. The IR bands at 1700 cm^{-1}, 1270 cm^{-1} may be due to urethane linkages and the bands at 1640 cm^{-1}, 1255 cm^{-1} may be due to urea linkages [2]. The other IR spectral features are due to aromatic and aliphatic moieties in monomers. The end group analysis of all the polymers give a low molecular ($\overline{Mn}$) weight (Tab. I). As the produced polymers are insoluble in organic solvents, the colligative properties (*i.e.*, viscosity, osmometry) have not been studied. The thermogravimetric analysis of all the PUUs reveals that they decompose into two stage. The first stage starts from 200°C and the second stage starts from 380°C. The degradation starts at 200°C is indication of urethane groups. It is reported that polyurethanes start their degradation at this temperature range [8]. This might be due to depolymerization *i.e.*, degradation. The urethane linkage convert into isocyanate on the loss at about 200°C. On the basis of the polymer sample the possible % wt loss due to this depolymerization during the 1st step of degradation is calculated.

The values for the first step in the degradation of the polymer sample are furnished in Table II. These results confirmed the view that the first step in the degradation of the polymer sample is due to depolymerization of all the 'urethane units' of the polymer chains.

The electrical conductivity measured at room temperature of all four PUU samples are shown in Table I and they are in the range of 7.8×10^{-12} to $8.4 \times 10^{-9}\,\Omega \cdot cm^{-1}$ depending upon the nature of the polymer. The examination of the result reveals that the produced PUUs can be ranked as semiconductors. The produced PUUs are insoluble and hence they can not be processed.

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