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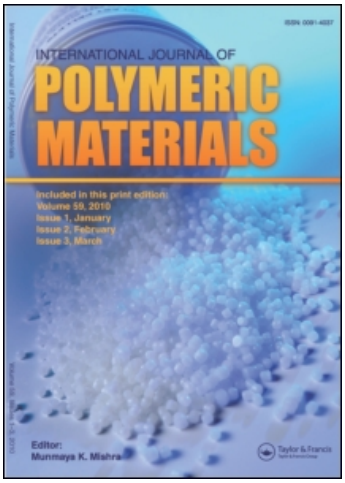

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Poly (Urethane-Urea)s: Part-1

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Poly (Urethane-Urea)s: Part-1

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Four various poly(urethane-urea)s (PUUs) were prepared by polycondensation of 4-aminophenol with various diisocyanates. The resultant poly (urethane-urea)s were characterized by elemental analyses, 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; p-aminophenol; molecular weight; electrical properties

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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 received no more attention yet. However only few poly (urethane-urea)s are reported from aminoglucose [2] or from mixture of diol, diamine and diisocyanate [3]. As it might give polymers having important properties, such type of polymer research has been adopted by condensation of monomer having both amino and hydroxy groups (e.g. 4-aminophenol and diisocyanate). Hence the present paper comprises synthesis and characterization of novel poly(urethane- urea)s by the synthetic route furnished in Scheme-1.

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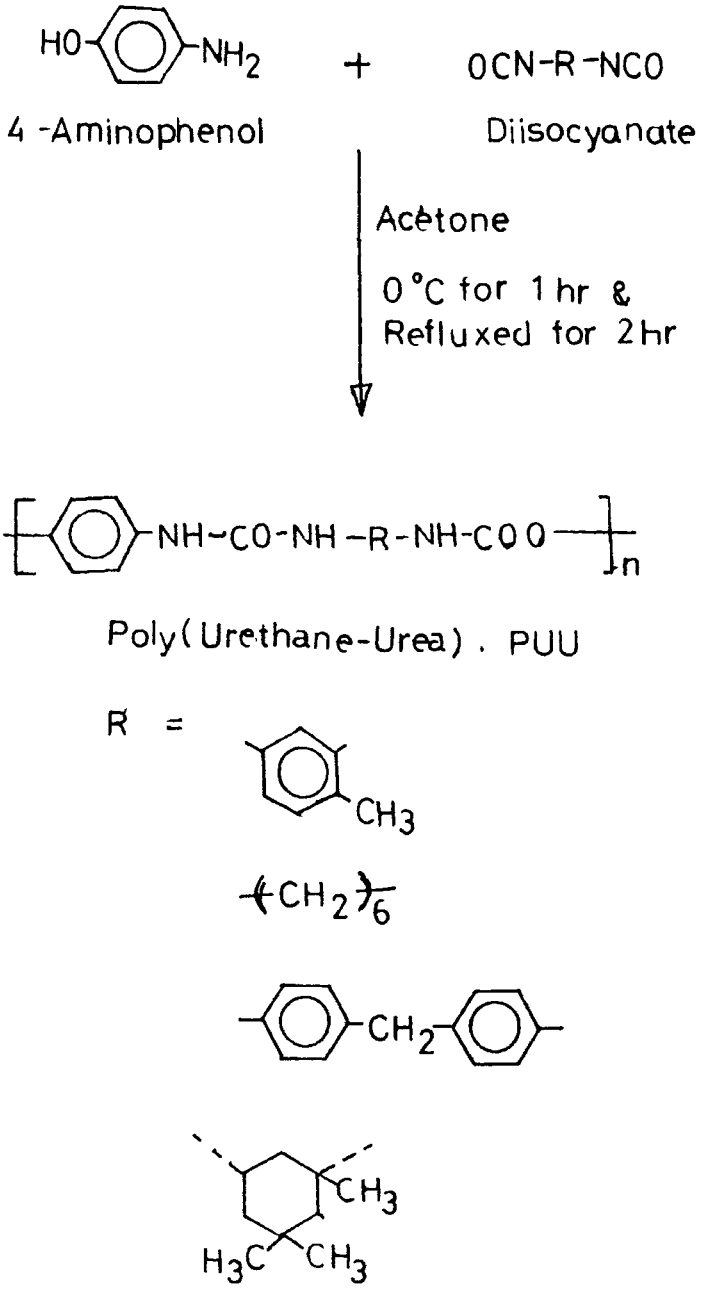

Scheme 1.

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RESULTS AND DISCUSSION

The poly(urethane-urea) (PUU) formation is performed by facile reaction of NH_2 and OH with -NCO groups. The PUUs shown in reaction scheme are furnished in Table I. They are dark brown solid powders. They do not melt up to 250 °C and are insoluble in common organic solvents. Elemental contents (C,H,N) (Tab. I) of the polymers are consistent with the predicted structure (reaction scheme).

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 bends at 1700 cm^{-1}, 1270 cm^{-1} may be due to urethane linkage and the bands at 1640 cm^{-1}, 1255 cm^{-1} may be due to urea linkage [2]. The other IR spectral features are due to aromatic and aliphatic moieties present in monomers.

The end group analysis of all the polymers give the 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 [4, 5]. This might be due to depolymerization i.e. degradation. The urethane linkage convert into isocyanate on the loss at about 200 °C. On the bases of the structure of the polymer sample the possible %age 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-I. These results confirmed the view that the first step in the degradation of the polymer samples 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 6.3×10^{-13} to $3.8 \times 10^{-9}\,\Omega\,cm^{-1}$ depending upon the nature of the polymer. The examination of the results reveals that the produced PUUs can be ranked as semiconductors. The produced PUUs are insoluble and hence they cannot be processed. Hence its modifications in terms of application view the novel PUUs synthesis has been adopted. This is in progress.

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TABLE I Characterization of poly (urethane-urea) s (PUUs)

PUU Sample	*Mole formula of repeat unit*	*Mol. wt. of repeat unit*	*Elemental Analysis*						*Mn* *	*Elect. conductivity (σ) at 303 K ($\Omega . cm^{-1}$)*
			% C		*% H*		*% N*			
			Calcd.	*Found*	*Calcd.*	*Found*	*Calcd.*	*Found*		
PUU-1	$C_{15}H_{13}N_3O_3$	283	62.28	61.90	4.49	4.30	14.53	14.40	1800	7.9×10^{-10}
PUU-2	$C_{14}H_{19}N_3O_3$	277	60.64	60.23	6.85	6.73	15.16	15.8	2800	2.5×10^{-11}
PUU-3	$C_{21}H_{17}N_3O_3$	359	70.19	69.84	4.73	4.50	11.70	11.47	2900	3.8×10^{-9}
PUU-4	$C_{18}H_{23}N_3O_3$	329	65.85	65.43	7.00	6.76	12.76	12.48	3500	6.3×10^{-13}

*Estimated by non-aqueous conductometric titration.

TABLE II Thermogravimetric analysis of poly (urethane-urea) s (PUUs)

PUU Sample	*% age wt. loss at temp. C*						*Loss at 1 step (200–300 C)*	
	200	*300*	*400*	*500*	*600*	*700*	*Calcd.*	*Found*
PUU-1	5.0	16.0	40.0	55.0	78.0	92.0	16.60	16.00
PUU-2	2.5	11.2	35.0	50.0	73.0	90	10.00	11.20
PUU-3	1.5	13.0	36.0	48.0	72.0	90.0	12.50	13.00
PUU-4	1.5	9.5	32.0	46.0	70.0	90.0	9.10	9.50

EXPERIMENTAL

Materials

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

Procedure

To a ice cooled solution of 4-aminophenol (0.01 mole) in dry acetone (50 ml) a solution of diisocyanate in 50 ml dry acetone was added gradually with constant stirring. 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 air-dried (95% yield).

Measurements

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

References

[1] Encyclopedia of Polymer Science and Engg. **13**, John Wiley and Sons, N. Y. (1985). p 212, 243.
[2] Kurita, K., Hirakawa, N. and Iwakura, Y. (1979). *Makromol Chem.*, **178**, 2939 (1977). **180**, 2331.

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[3] Iwakura, Y., Hayashi, K. and Inagaki, K. (1967). *Makromol Chem.*, **104**, 56.
[4] Kurita, K., Imajo, H. and Iwakura, Y. (1979). *J. Polym. Sci. Poly. Chem.*, **17**, 1696.
[5] Saunders, J. H. and Frisch, K. C. (1963). Polyurethanes Chemistry and Technology. *Inter Sci. pulg. N.Y.*
[6] Patel, H. S., Patel, R. N. and Patel, S. R. (1981). *Angewt. Makromol. Chem.*, **99**, 125.
[7] Patel, H. S. and Brahmbhatt, D. I. (1992). *Phos. Sulfur. Silicon*, **73**, 57.

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