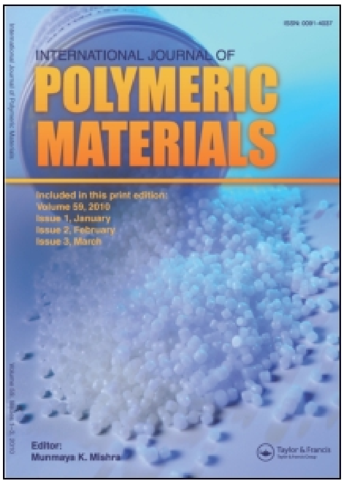

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Polyurethanes Based on Resacetophenone

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Polyurethanes Based on Resacetophenone

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Polyurethanes were prepared by condensation of resacetophenone (RAP) with various diisocyanates (DICs). The resultant polyurethanes (PUs) were characterised by elemental analysis, IR spectral study, molecular weight by nonaqueous conductometric titration and thermogravimetric analysis (TGA). The electrical conductivity of PUs were also measured at room temperature.

Keywords: 2,4-Dihydroxyacetophenone [Trivial name: Resacetophenone (RAP)]; Polyurethanes (PUs); IR spectroscopy; TGA

INTRODUCTION

The Polyurethanes (PUs) are well known candidates for industrial applications [1]. Most of the PUs were obtained from aliphatic diols and aro- matic diols [1]. One of the aromatic diol 2,4-dihydroxyacetophenone [*i.e.*, Resacetophenone (RAP)] has reactive –OH groups and it may afford PUs with keto groups. This may find important applications. Hence it was thought interesting to synthesize and characterized PUs based on RAP.

EXPERIMENTAL

Materials

2,4-Dihydroxyacetophenone [Trivial name: Resacetophenone (RAP)] mp 144°C, was prepared by method reported in literature [2].

Diisocyanates were obtained from Merck, Germany. All other chemicals used were of Analar grade.

Synthesis of PUs

To a well stirred solution of RAP (0.01 mole) in tetrahydrofuran (THF) 50 ml; diisocyanate (0.1 mole) in 50 ml THF was added at 0–5°C. Then the reaction mixture was refluxed for 2 hrs. The polymer products was filtered off, washed and air drying. The details of all PUs are furnished in Table I.

Measurements

The C, H, N, contents of all the samples were obtained from Carlo Erba C, H, N, S Elemental Analyser, Italy. IR spectra of all the samples were scanned in KBr pellets on a Perkin Elmer 983 Spectrophotometer. Molecular weight estimated by a non-aqueous conductometric titration method reported earlier [3, 4]. The electrical conductivity measurements were carried out over a wide temperature range in air using Hewlett-Packard high resistance meter. Thermogravimetric analysis (TGA) were performed on a Du Pont 951 thermal analyser.

RESULTS AND DISCUSSION

Polycondensation between RAP and DICs is performed at 60–70°C for 5 hrs. The obtained PUs were in form of pale yellow amorphous powders. The C, H, N contents of all PUs (Tab. I) are consist with the

TABLE I Analytical and thermal data of polyurethanes

PU sample	*Yield %*	*Elemental analysis*							*%wt. Loss at °C from TGA*				
		%C		*%H*		*%N*		$\overline{Mn}$*					
		Calcd.	*Found*	*Calcd.*	*Found*	*Calcd.*	*Found*		220	300	400	500	600
PU-1	70	62.57	62.08	4.29	4.25	8.58	8.55	2000	2.0	36	56	80	90
PU-2	80	64.17	64.12	6.95	6.92	7.48	7.46	2300	2.9	40	60	82	93
PU-3	85	60.00	59.97	6.25	6.22	8.75	8.73	3000	3.0	38	62	80	94
PU-4	90	68.65	68.80	4.47	4.45	6.96	6.94	2700	2.8	40	60	80	92

* Estimated by non-aqueous conductometric titration.

predicted structure (Scheme I). They are softned in the range of 220–240°C depending upon the nature of the polymer.

The IR spectra of all PUs comprises the important spectral band due to urethane linkages. They are $1700\,\mathrm{cm}^{-1}$ and $1270\,\mathrm{cm}^{-1}$ present in all the IR spectra. The other bands due to either aromatic ring or aliphatic moity are also observed from the respective monomer segments.

Number average molecular weight of all the polymers were estimated by non-aqueous conductometric titration. The values of $\overline{M}n$ are in the range of 2000–3000 depending upon the nature of polymer. As little solubility of these polymer in common organic solvents, the viscosity study of this polymer has not been attempted.

The TGA data of all the samples are furnished in Table I. The examination of TGA thermograms (not shown) reveals that all PUs start their degradation around 220°C. Their decomposition exhibit rapid in the range of 220–450°C and loss completely around 600°C. The degradation may be either formation of isocyanate or other macrosegments. The calculated value for isocyanate degradation is shown in Table I. The observed value is almost nearer to calculated value. This suggests that major portion during degradation would be isocyanate.

The present polycondensation in higher ratio of isocyanate would yield oligomer containing RAP moity, which would act as UV absorber. So novel paint formulation study of oligomeric RAP containing PU and other additives is under progress.

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