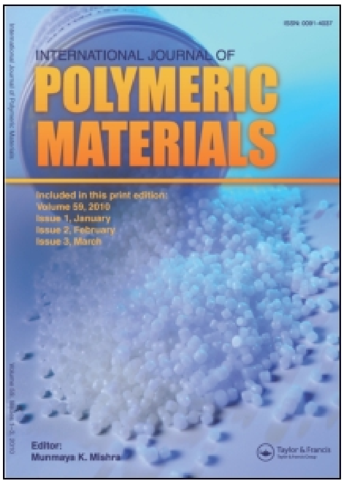


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# Synthesis and Characterization of Colored Polyureas

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# Synthesis and Characterization of Colored Polyureas

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*Various disperse dyes based on m-phenylenediamine have been prepared. These dyes were then polycondensed with Toluene 2, 4-diisocyanate. The resultant colored polyureas were characterized by elements analyses, IR spectral studies, $\overline{M}n$ estimation by monaqueous conductometric titration and thermogravimetry. All the polyureas were subjected to electrical conductivity determination at room temperature.*

**Keywords:** polyureas, IR spectral studies, number average molecular weight ($\overline{M}n$), thermogravimetry electrical conductivity

## INTRODUCTION

Polyureas may be classified as heterochain macromolecular compounds, which contain urea groups in their structure. Although the chemistry and technology of polyureas are of recent origin, the chemistry of ureas dates back over 100 years. Linear polyureas are thermoplastic polycondensation products with aliphatic or aromatic structures. Polyureas or copolyureas containing aliphatic structures exhibit a difference of

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50–100°C between their melting points and the beginning of decomposition; they are used for castings. Polyureas containing aromatic structures melt near their decomposition temperatures. They are soluble in some organic solvents and can be used for preparation of lacquers, varnishes, and coatings [1]. Melting points also depend on the molecular weight. Molecular weights are determined from the measurements of the solution viscosity. The inherent viscosity ranges from 0.5 to 2 dL/g. g. The relation between intrinsic viscosity and molecular weight has been investigated using the Mark–Houwink equation [2].

Polyureas were first prepared on a commercial scale at I.G. Farben, employing the reaction between diisocyanates and diamines. Mitsui Toatsu Chemical Ltd. Produces synthetic fibers from nonamethylenediamine and urea. Today, polyureas and copolyureas (especially polyurethane polyureas) have many practical applications as foams, elastomers, adhesives, fibers, and others [3].

The area of colored polyureas has not received attention academically. Thus, it was thought interesting to explore the field of colored polyureas. The present article comprises synthesis, characterization, and electrical conductivity of polyureas. The research work is scanned in Scheme 1.



## EXPERIMENTAL

### Materials

All the chemicals used were of analytical or laboratory grades.

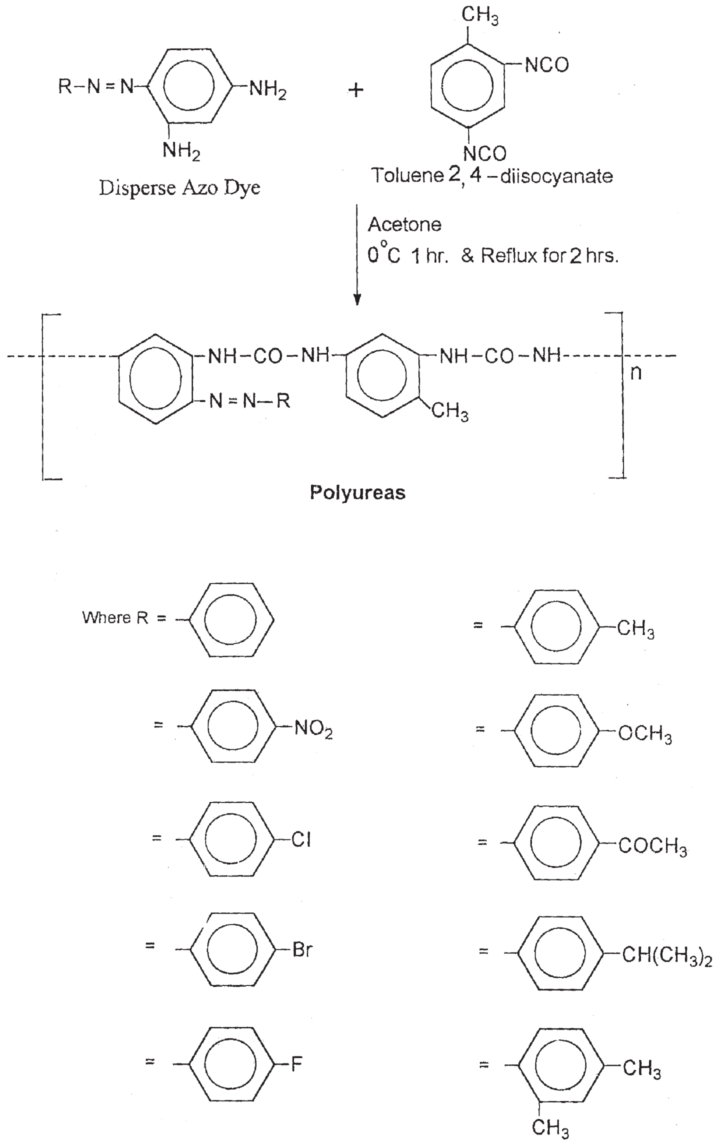
### Synthesis of Azo Disperse Dyes

The azo disperse dyes having structures shown in Scheme 1 where prepared by the methods reported in literature [4].

### Synthesis of Colored Polyureas

All the polyureas (PURs) based on azo disperse dyes where prepared in a similar manner. The general process is as follows.

To an ice-cooled solution of azo disperse dye (0.01 moles) in dry acetone (50 ml) a solution of Toluene 2, 4-diisocyanate, (0.01 mole) in 50 ml dry acetone was added gradually with constant stirring. A colloidal suspension was formed immediately, which was then stirred at room temperature for an hour. The resultant suspension was refluxed ($\sim 60°C$) for 2 h on a water bath. The resulting solid product was then filtered off and air-dried (95% yields).




**SCHEME 1** Synthesis and aromatic polyurea products.

## MEASUREMENTS

C,H,N contents of all polyureas samples were estimated by Carlo Erba, Italy, C, H, N, O, and S elemental analyzer. The IR spectra of oligomers were scanned in KBr pellets on Perkin Elemer 257 spectrophotometer, Number average molecular weights ($\overline{M}n$) of PURs were estimated by nonaqueous conductometric titration. The titration was carried out in formic acid against perchloric acid as titrant. A digital conductometer, Toshniwal, India was used for this purpose. The values of number average molecular weight ($\overline{M}n$) of all oligomer samples were calculated following the method reported by one of the authors [HSP] [5]. Thermo gravimetric analyses for oligomers were carried out on a Du Pont thermobalance in air at a heating rate of $10^{\circ}K\,min^{-1}$. The electrical conductivity of each PUR sample was measured on pellets (1 cm diameter, 0.45 cm thickness) at room temperature ($30 \pm 1^{\circ}C$) using a Million Megohmmeter RM 160 MK IIA BPL, India. The preparations of the pellets of all the PUR samples and other details have been described in an earlier communication [6].



## RESULT AND DISCUSSION

The polyureas (PURs) formation is performed by a facile reaction of $-NH_2$ groups dye moiety with $-NCO$ groups. All PURs shown in scheme-1 are furnished in Table 1. They are found to be colored solid powders. They do not melt up to 250°C and are insoluble in common organic solvents. Elemental contents (C, H, N) (Table 1) of each of the PURs are consistent with the corresponding predicted structure (reaction scheme).

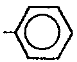
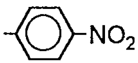
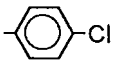
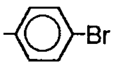
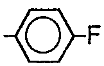
IR spectra (not shown) of all the PURs are identical in almost all aspects and inspection of all the spectra reveals that all the spectra comprise important IR spectral features of urea linkages. The IR bands at 1620–1680 $cm^{-1}$, 1240–1250 $cm^{-1}$ may both be due to urea linkage. The other IR spectra features, due to aromatic and aliphatic segments of monomers, appear at their respective positions.

As the produced oligomers are insoluble in the organic solvents the authors have used, the colligative properties (i.e., viscosity, osmometry) have not been studied. Therefore, the number average molecular weight ($\overline{M}n$) of all the oligomer sample has been measured by end group $-NH_2$ by nonaqueous conductometric titration. The results of $\overline{M}n$ values are furnished in Table 1.

The TG thermograms (not shown) of all the PURs are identical in nature. Inspection of the TG thermograms reveals that all the PURs decompose in one step. They start their degradation at about 200°C, and lose their weight rapidly between 200 and 600°C.

The electrical conductivities, measured at room temperatures, of all PUR samples are shown in Table 2. They are in the range of $8.5 \times 10^{-10}$ to $1.3 \times 10^{-7}\,\Omega cm^{-1}$ depending on the nature of oligomer.

**TABLE 1** Characterization of Polyureas (PURs)

| PUU sample | R = | Color of sample | Mole formula of repeating unit | Mol. wt. of repeating unit | $\overline{M}n$ | Elemental analysis | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | % C | | % H | | % N | |
| | | | | | | Calcd. | Found | Calcd. | Found | Calcd. | Found |
| PUR-1 |  | Reddish yellow | $C_{21}H_{18}O_2N_6$ | 386 | 3450 | 65.28 | 65.10 | 4.66 | 4.50 | 21.76 | 21.50 |
| PUR-2 |  | Brown | $C_{21}H_{17}O_4N_7$ | 431 | 3030 | 58.46 | 58.20 | 3.94 | 3.70 | 22.73 | 22.50 |
| PUR-3 |  | Red | $C_{21}H_{17}O_2N_6Cl$ | 420 | 3330 | 60.00 | 59.50 | 4.04 | 3.80 | 20.00 | 19.90 |
| PUR-4 |  | Red | $C_{21}H_{17}O_2N_6Br$ | 465 | 3700 | 54.19 | 54.00 | 3.65 | 3.50 | 18.06 | 17.80 |
| PUR-5 |  | Dark brown | $C_{21}H_{17}O_2N_6F$ | 404 | 3230 | 62.37 | 62.10 | 4.20 | 4.00 | 20.79 | 20.50 |

(*Continued*)

**TABLE 1** Continued

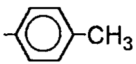
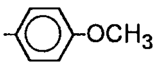
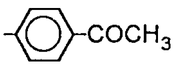
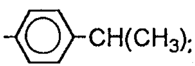
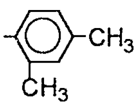
| PUU sample | R = | Color of sample | Mole formula of repeating unit | Mol. wt. of repeating unit | $\overline{M}n$ | Elemental analysis | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | % C | | % H | | % N | |
| | | | | | | Calcd. | Found | Calcd. | Found | Calcd. | Found |
| PUR-6 |  | Reddish brown | $C_{22}H_{20}O_2N_6$ | 400 | 4000 | 66.00 | 65.80 | 5.00 | 4.80 | 21.00 | 20.90 |
| PUR-7 |  | Dark brown | $C_{23}H_{20}O_3N_6$ | 416 | 4170 | 63.46 | 63.20 | 4.80 | 4.50 | 20.19 | 20.00 |
| PUR-8 |  | Brown | $C_{23}H_{20}O_3N_6$ | 428 | 4350 | 64.48 | 64.20 | 4.67 | 4.30 | 19.62 | 19.40 |
| PUR-9 |  | Dark brown | $C_{24}H_{24}O_2N_6$ | 428 | 4760 | 67.28 | 67.10 | 5.60 | 5.40 | 19.62 | 19.50 |
| PUR-10 |  | Red | $C_{23}H_{23}O_2N_6$ | 415 | 4160 | 66.50 | 66.20 | 5.54 | 5.40 | 20.24 | 20.00 |

**TABLE 2** Electrical Conductivity of PURs

| PUR samples | Electrical conductivity ($\sigma$) at 303°K ($\Omega \cdot cm^{-1}$) |
|---|---|
| PUR-1 | $3.2 \times 10^{-8}$ |
| PUR-2 | $8.5 \times 10^{-10}$ |
| PUR-3 | $2.1 \times 10^{-9}$ |
| PUR-4 | $7.6 \times 10^{-9}$ |
| PUR-5 | $9.8 \times 10^{-9}$ |
| PUR-6 | $8.2 \times 10^{-7}$ |
| PUR-7 | $5.4 \times 10^{-7}$ |
| PUR-8 | $3.8 \times 10^{-7}$ |
| PUR-9 | $8.2 \times 10^{-7}$ |
| PUR-10 | $1.3 \times 10^{-7}$ |

An examination of the results reveals that the produced PURs can be ranked as poor insulators. The application of PURs is under investigation.



## CONCLUSION

The present work deals with colored polyureas. The nature of these oligomers is amorphous, colored powder. They have low thermal stability. Having urea groups they can be compatible with thermoplastics and can easily form colored articles even when processed at high temperature. The colored polyureas cannot bloom or bleed out of the articles.

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