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## SYNTHESIS, CHARACTERIZATION AND THERMAL ANALYSIS OF RESINS FROM DIFFERENT CARDANOL BASED DYES

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### Abstract

Cardanol (Cashew phenol) is subjected to diazotisation with Aniline and m-Toluidine to get monomers like Cardanol based dye from Aniline (CBDFA) and Cardanol based dye from m-Toluidine (CBDFT). Such monomers have been condensed with formaldehyde in presence of acid catalyst to form resins. The resins have been characterized by FTIR spectra and their thermal behaviour have been studied.

**Key words:** Resin; Cardanol; Formaldehyde; Resin; Aniline; m-Toluidine.

### Introduction

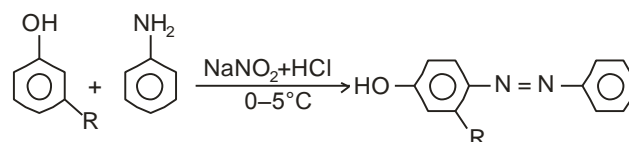
Renewable sources as a substitute to petrochemical derivatives have attracted the attention of many researchers for the synthesis of polymers. Cashew-nut shell liquid (CNSL) a byproduct of cashew industry is a unique natural source of unsaturated long chain phenol (Guo *et al.*, 2002; Petrovic *et al.*, 2005; Roloff *et al.*, 2005; Kong and Narine, 2007; Narine *et al.*, 2007). The phenolic nature of cardanol has promoted researchers to react with formaldehyde or with other aldehyde to produce numerous resinous materials (Phanikumar *et al.*, 2002; Oghome and Kehinde, 2004). In place of cardanol, many workers have synthesized and characterized a number of polymers from cardanol derivatives (Das *et al.*, 1998; Mohapatra *et al.*, 1994; Mishra D.K., Mishra *et al.*, 1996; Nayak *et al.*, 1999; Guru *et al.*, 1999). Cardanol based polymers have wide applications in composites (Tan, 1997; Sathiyalekshmi, 1993), polyurethanes (Sathiyalekshmi and Gopalakrishnan, 2000; Mythili *et al.*, 2004; Das and Lenka, 2011; Athawale and Shetty, 2010), surface coating (Santeusanio *et al.*, 2013) and few others (Unikrishnan and Thachil, 2006; Unikrishnan and Thachil, 2008; Devi and Srivastava, 2006; Devi and Srivastava, 2007). This communication reports on the synthesis, characterization and thermal analysis of resins obtained by condensation of cardanol-based dyes with formaldehyde.

### Experimental

#### Preparation of cardanol based dye from aniline (CBDFA)

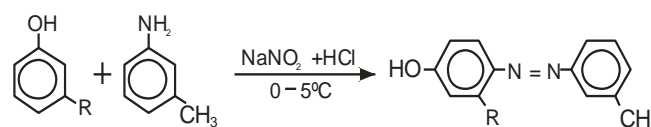
4.65gm of aniline was taken in 15ml of distilled water. 13.8ml of conc. HCl was added to it and cooled down to 0-

5 °C. A cold solution of 5gm of sodium nitrite in 10ml of water was added slowly with continuous stirring. To the whole solution a cold solution of 15gm of cardanol in 4ml of 5% NaOH solution was added. A brown red color dye was formed and separated by separating funnel.



#### Preparation of cardanol based dye from m-Toluidine (CBDFT)

5.35gm of m-Toluidine was taken in 15ml of distilled water. 13.8ml of conc. HCl was added to it and cooled down to 0-5 °C. A cold solution of 5gm of sodium nitrite in 10ml of water was added slowly with continuous stirring. To the whole solution a cold solution of 15gm of cardanol in 4ml of 5% NaOH solution was added. A brown red color dye was formed and separated by separating funnel.



#### Synthesis of Resin

The resin was synthesized by condensing cardanol based dyes (CBDFA/ CBDFT) (4 mmol) with formaldehyde (40 mmol) in presence of 1% hydroquinone and 2 ml of 6 N HCl at 90°C for 4 – 6 hours with periodical shaking. After completion of the reaction, the product was repeatedly

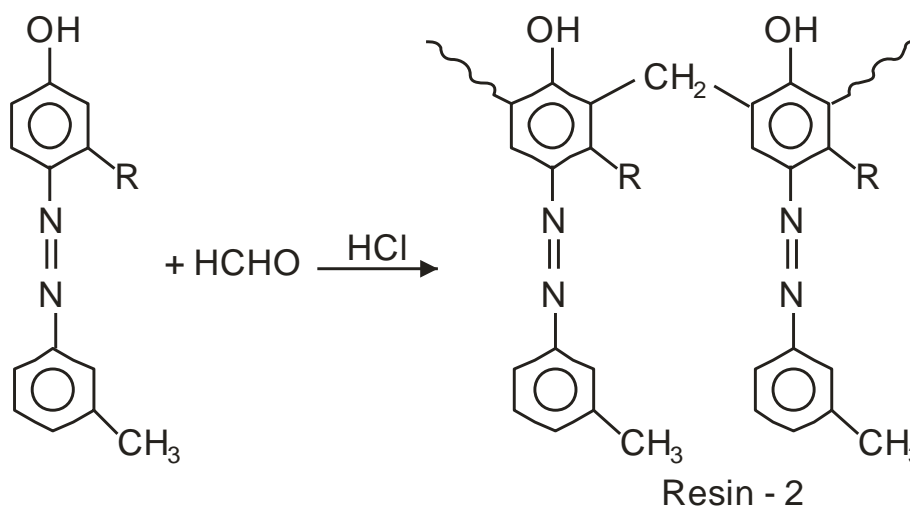
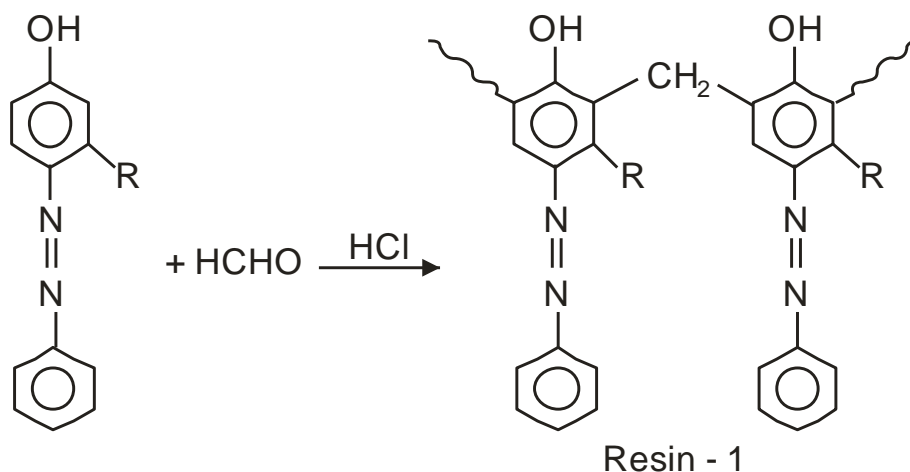
washed with hot water in order to drive out unreacted materials. The product was finally dried in vacuum at 60 °C.

The physico-chemical properties of the resins are furnished in Table 1.

**Table 1:** Physico – Chemical Properties of Resins

| Sample    | System              | Yield (%) | Colour        | Structure   | Solubility   |
|-----------|---------------------|-----------|---------------|-------------|--------------|
| Resin – 1 | CBDFA-FORMALDEHYDE  | 71        | Reddish Brown | Crystalline | Toluene, DMF |
| Resin – 2 | CBDFT -FORMALDEHYDE | 69        | Reddish Brown | Crystalline | Toluene, DMF |

### Reaction Scheme



**Table 2:** Thermal Decomposition of Resins

| Sample Code | Composition        | % Weight loss at various temperature |        |         |         |         |         |         |
|-------------|--------------------|--------------------------------------|--------|---------|---------|---------|---------|---------|
|             |                    | 100°C                                | 200°C  | 300°C   | 400°C   | 500°C   | 600°C   | 700°C   |
| Resin 1     | CBDFA-FORMALDEHYDE | 1.7482                               | 5.5944 | 20.6293 | 34.9656 | 64.3356 | 68.8808 | 76.9230 |
| Resin 2     | CBDFT-FORMALDEHYDE | 2.0979                               | 3.8461 | 19.9300 | 34.2657 | 65.7342 | 69.930  | 79.7202 |

## Results and Discussion

### FTIR Spectra

Fourier transform infrared (FTIR) spectra of Resin- 1 and Resin -2 are shown in Fig. 1(a) and 1(b) respectively.

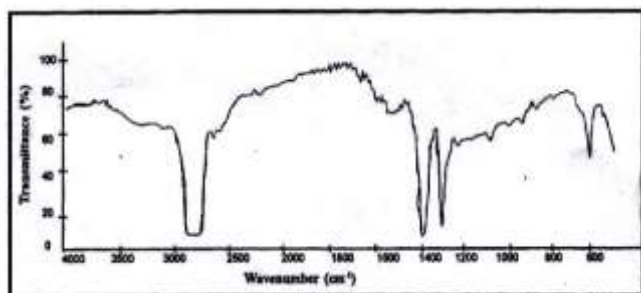


Fig. 1a: Fourier transform infrared (FTIR) spectra of Resin 1.

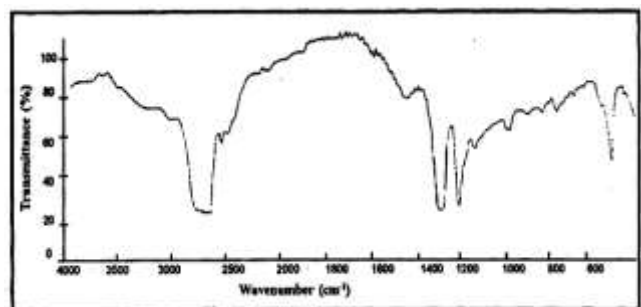


Fig. 1b: Fourier transform infrared (FTIR) spectra of Resin 2.

The interaction between chemical groups on either the same or different molecules can cause a shift of the IR peak positions of the participating groups, which is commonly observed in macromolecular system due to the existence of hydrogen bonds (Lu and Zhang, 2002). The peaks such as  $3500 - 3300 \text{ cm}^{-1}$  (O - H hydrogen bonded),  $3100 - 3000 \text{ cm}^{-1}$  (aromatic C - H stretching),  $2924 - 2855 \text{ cm}^{-1}$  (Symmetric C - H stretching),  $1590 \text{ cm}^{-1}$  (C = C stretching of aromatic ring) and a peak at  $1428 \text{ cm}^{-1}$  is due to -N=N- stretching of azo group.

### Thermo gravimetric analysis of the Resins

The thermo gravimetric analysis in Table 2 reveals that the resins decomposed in three distinct steps. In the first step of thermal degradation 100 - 200°C weight loss up to 5% is observed in Resin 1 & 2 (Fig. 2a and 2b). This may be attributed to the removal of moisture retained in the resin. A gradual weight loss occur in the temperature ranges 200-400°C may be caused by thermal degradation of the small fragments like  $\text{CH}_3$ , OH and the side chain. The resins are thermally more stable up to 400°C. The weight loss around 80% in the last stage of thermal degradation may be due to the depolymerisation and the cleavage of the aromatic ring in an oxydegradative manner.

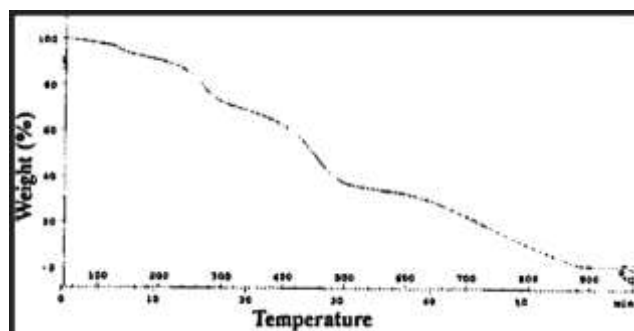


Fig. 2a: Thermal degradation of weight in Resin 1

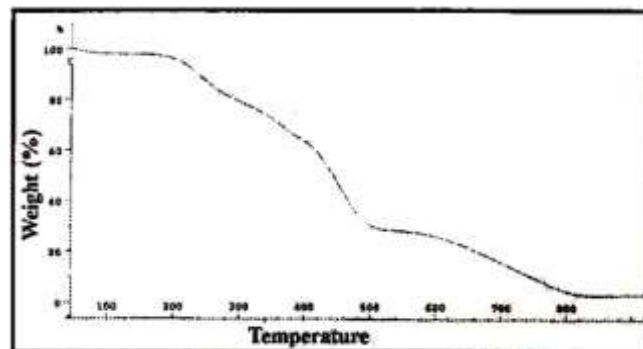


Fig. 2b: Thermal degradation of weight in Resin 2.

## Conclusion

The structure of the resin is established by FTIR study. The kinetic study shows that due to presence of azo group in the resins, the decomposition temperature is enhanced. The resins show good thermal stability up to 400 °C. The resin of cardanol based dye from m-toluidine has comparatively greater percentage of weight loss due to low degree of polymerization.

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