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*NOTE*

# Preparation and Application of a Novel Adhesive (Copolymer Compound)

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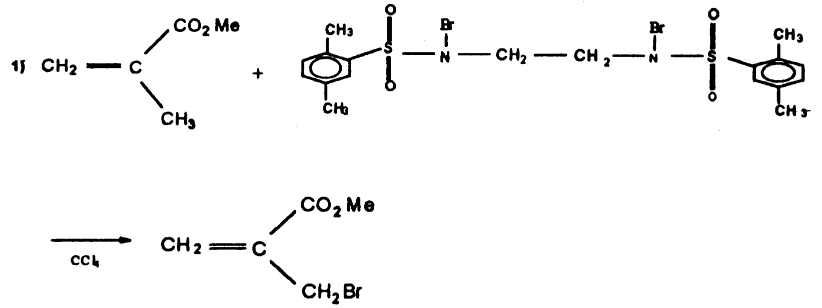
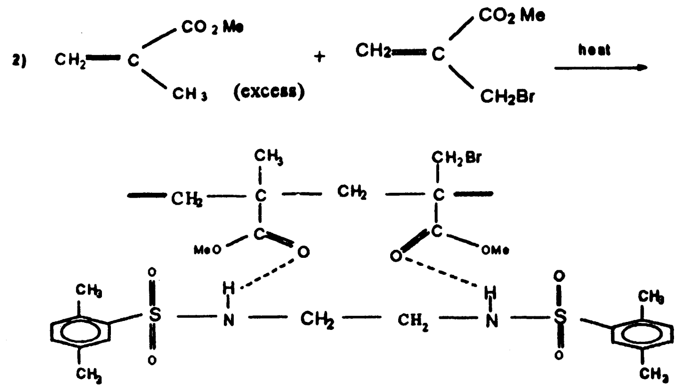
A novel adhesive compound was synthesised by reaction of bromosulfonamide with methylmethacrylate in ambient temperature. The novel adhesive is stable and not hygroscopic.

Adhesive materials are very important and have been synthesised by various methods. The largest quantities of UF and MF polymers are used for adhesives. Mostly, the products are applied as aqueous solutions of the resins, but powdered materials are also available. The advantage of the powdered products is due to their better chemical stability compared to the aqueous adhesive[1]. The combination of good processing characteristics and useful properties has led to the use of epoxy resins in many applications including adhesives[2]. Halogenated epoxy resins have been reported in literature[3–5]. The basic chemistry of polyurethane coatings and adhesives is similar to the elastomers and foams. Solvents may or may not be employed. Both aqueous and non-aqueous systems are used. The lower acrylated polymers have glass transition points below room temperature; they are typically soft and rubbery. They have been used in sensitive adhesive compounds. Amino resins, cyanoacrylate, epoxy, poly(vinylacetate), poly(vinylalcohols) are also used as adhesive compounds.

The main aim of this work was to synthesize a novel adhesive as a copolymer useful for sticking of various materials.

We combined methylmethacrylate and N,N′-dibromo-N,N′-ethylene-bis-(2,5-dimethyl benzenesulfonamide) for a period of 10 h at steam bath in redistilled carbon tetrachloride. The product was prepared according to experimental part with high yield. It was identified by $^{1}H$ $^{13}C$-NMR and IR spectroscopy. The mechanism of reactions is believed to be as given below:





According to this mechanism, first of all the methylmethacrylate was reacted with N,N′-dibromo-N,N′-1,2-ethylene-bis-(2,5-dimethyl benzenesulfonamide) in carbon tetrachloride; then the product was reacted with excess methylmethacrylate (unreacted reagent) and the obtained polymer contained functional groups of sulfonamides.

The synthesised polymer was tested with various solvents (Table-1).

TABLE-1
SOLUBILITY OF ADHESIVE AT ROOM TEMPERATURE

| Solvent | Soluble per cent (%) |
|---|---|
| $CCl_4$ | 100 |
| $CHCl_3$ | 100 |
| DMF | 100 |
| DMSO | 100 |
| $CH_3COCH_3$ | 100 |
| $CH_3OH$ | Partial |
| $C_2H_5OH$ | Partial |
| $H_2O$ | None |

The novel adhesive was tested with different materials shown to have the following properties (Table-2):

TABLE-2

| Glass | Wood | Leather | Metal | Carpet |
|---|---|---|---|---|
| Excellent | Partial | Excellent | Partial | Excellent |

### Preparation of N,N′-1,2-ethylene-bis-(2,5 -dimethyl benzenesulfonamide)

65 g (0.3186 mol) of *p*-xylene sulfonyl chloride was placed in a beaker. The beaker was heated on a water bath (80°C) until the compound changed to a liquid. 10.62 mL (0.1593 mol) of ethylene diamine was added dropwise to the reactant and the mixture was stirred with a glass rod. The mixture was heated (80°C) and stirred for 0.5 h. The mixture was cooled and 100 mL water was added. The product was collected on a buchner funnel and washed with a little cold water.

The product was recrystallised from ethanol. The yield of pure N,N′-1,2-ethylene-bis (2,5-dimethylbenzenesulfonamide) (m.p. 143–144°C) was 56.5 g (89.5%). IR spectrum (KBr pellets): 3300, 1460–1370, 1310 and 1140 $cm^{-1}$. $^1H$ NMR spectrum ($CD_3COCD_3$, TMS): δ 2.549 (s, 3H), 2.357 (s, 3H), 2.990 (s, 2H), 6.568 pm (br, 1H), 7.275–7.703 (m, 3H).

### Preparation of N,N′-dibromo-N, N′-1,2-ethylene-bis-(2,5-dimethyl benzenesulfonamide)

10 g (0.02525 mol) N,N′-1,2-ethylene-bis-(2,5-dimethyl benzenesulfonamide) was dissolved in a slight molar excess of chilled sodium hydroxide solution (of approximately 3 M strength) at room temperature and transferred the solution to a beaker. 3 mL (0.0584 mol) of bromine dissolved in 15 mL carbon tetrachloride was added to the solution with vigorous stirring and immediately yellow precipitate was formed. The product (yellow precipitate) was collected on a buchner funnel and it was washed with 30 mL distilled cold water and then dried in a vacuum desiccator at room temperature for 6 h. The yield of pure N,N′-dibromo-N,N′-1,2-ethylene-bis-(2,5-dimethylbenzenesulfonamide), m.p. 96–98°C, was 11.3 g (81%). The product was stable at standard condition and was not sensitive to air. The reagent was identified by NMR, IR spectroscopies and elemental analysis. IR spectrum (KBr pellets): 3050, 2980, 1490, 1320 and 1165 $cm^{-1}$. $^1H$ NMR spectrum ($CD_3COCD_3$, TMS): δ 2.553 (s, 3H), 2.347 (s, 3H), 2.998 (s, 2H), 7.258–7.695 (m, 3H).

### Preparation of a Novel Adhesive (Copolymer Compound)

5.41 g (0.0098 mol) of N-bromosulfonamide, 1.96 g (0.0196 mol) of methylmethacrylate and 10 mL of dry, redistilled carbon tetrachloride were poured in 100 mL round-bottomed flask. Refluxed on a water bath for 10 h. By this time all the solid phase (sulfonamide) stayed at the top surface of the liquid.

Sufonamide was filtred off by reduced pressure. It was washed with 10 mL dry carbon tetrachloride. The solvent of filterated solution was evaporated by steam bath. Residue (1.7 g, 87%) was characterized by NMR and IR spectroscopy and showed to be a novel adhesive compound. IR spectrum: 3300, 3100, 2900, 1680, 1650, 1460–1370, 1310 and 1140 $cm^{-1}$. $^1H$ NMR spectrum ($CCl_4$, TMS): δ 2.024 (s, 3H), 2.366 (s, 3H), 2.582 (s, 3H), 3.037 (s, 2H), 3.616 (t, 2H), 3.816 (s, 3H), 7.275–7.703 (m, 3H), $^{13}C$ spectrum: 14.106, 17.494, 18.948, 20.055 ($—CH_3$, $—CH_2$, $—CH_2$, $—CH_2$), 25.706 ($—CH_2Br$), 120–140 ($C_6H_3$), 166.341, 168.006 ($—COCCH_2Br$, $—COCCH_3$).

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