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FUNCTIONALIZATION OF AMBERLITE XAD-2 WITH 5-SULFOSALICYLIC ACID

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Abstract:
The amberlite XAD-2 resin was functionalized by coupling through the azo spacer arm with 5-sulfosalicylic acid. Selective method diazo spacer technique (-N=N-) was used for the fictionalizations of amberlite XAD-2 and product was abbreviated as 5-SSA-N=N-AXAD-2. Intermediate products formed in reaction were determined by FTIR spectra. Peaks and their values obtained in each spectra are in good agreement with the standard values for particular functional groups.
Key words: Amberlite XAD-2; Resin; diazo spacer; Thermal degradation.

Introduction:

Use of polymeric resin in all spheres of life has been abundantly increased, because of its versatility. The synthesis of new copolymer resin attracted the attention of researcher [1]. Various modified phenol-formaldehyde resins have large number of practical application [2,3,4]. In recent years, synthesis and characterization of polymers has become a subject of interest. Now a day polymeric resins have been received much attention and importance due to their wide range of industrial application.

Employing chelating resin as adsorbent is an attractive analytical tool as the selectivity of the sorbent is greatly improved. The two means for attaching ligands to polymer matrix are impregnation and functionalization. Impregnation involved physical adsorption while functionalization involved a chemical bonding based on the covalent coupling of the ligand with polymer backbone through a diazo spacer arm (-N=N-)[5] or by SO_2 linkage or by CH_2 spacer technique [6]. Recently, amberlite XAD resins functionalized with pyrocathecole[7], organophosphrus extractants[8], Cyanex 272[9], 2-Mercaptobenzimidazole[10], Dihydroxypyridine [11], xylenol orange [7], 1-(2-pyridylazo)-2-napthol [12]etc.

Amberlite XAD series resin have efficient support for anchoring chelating legands due to their good porosity, uniform pore size distribution, high surface area, and excellent physical and chemical properties [6]. Amberlite XAD-2 and XAD-4 have been ideal for the functionalization based on their porosity and surface area. [13,14].

The present paper reports the synthesis of functionalized amberlite XAD-2 resin with 5-sulfosalicylic acid, its intermidate products were studied by FTIR spectra.

Experimental Section

Instrumentation:

Digital oil bath (Bio Technics India, Model BTI-38) with silicon oil was used for the synthesis. Infrared (IR) spectra (4000-400 cm^{-1}) were recorded on a Nicolet FT-IR spectrometer.

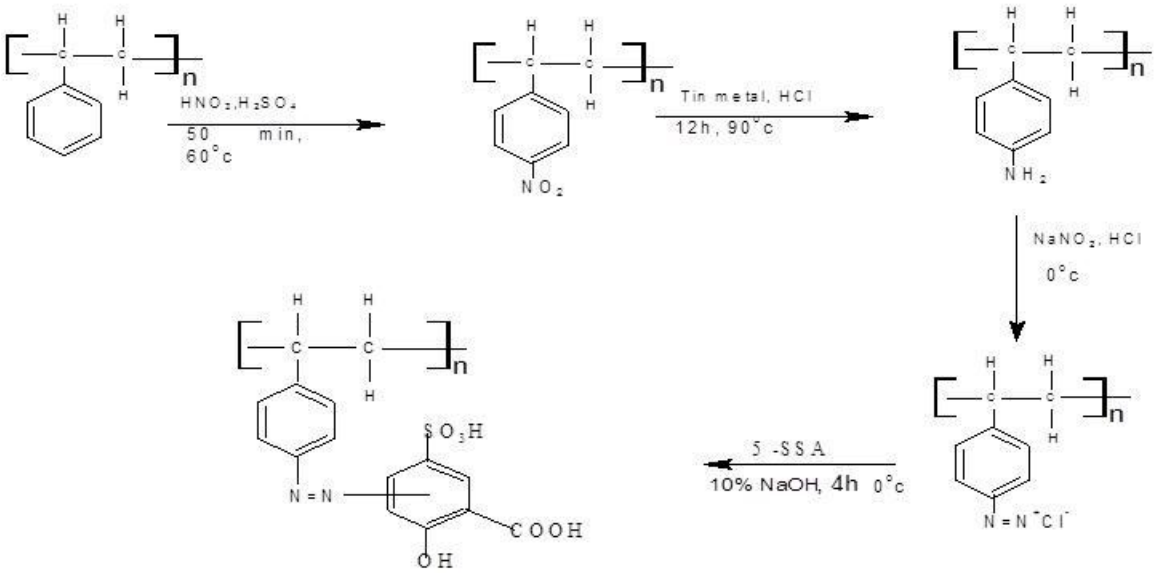
Reagents and solutions:

Chemicals used in the synthesis were pure analytical grade. Amberlite XAD-2 resin (surface area, 330 m^2 g^{-1}) pore diameter 9 nm, bead size 20-60 mesh was procured from Sigma-Aldrich (USA). 5-Sulfosalicylic acid (Sigma Aldrich), conc. HCl, conc. HNO_3 and conc. H_2SO_4 were procured from Merck, SD Fine Chemicals, India Ltd.

Synthesis of functionalized amberlite XAD-2 resin:

Amberlite XAD-2 beads (5 gm) was crushed and nitrated with 10 ml of concentrated HNO_3 and 25 ml of concentrated H_2SO_4 (nitrating mixture) for 30 min. at 50^0C. The reaction mixture was poured in ice cold water and nitrated resin (NO_2-AXAD-2) was collected by filtration. The intermediate product was repeatedly washed with distilled water until free from acid and dried. In second step nitrated resin was reduced by refluxing it for 12 hrs. with tin metal in conc. HCl (45 ml) and ethanol (50 ml). The modified aminated resin (NH_2-AXAD-2) was filtered and repeatedly washed with distilled water until free from acid. Aminated resin was treated with 100 ml of 2M HCl for 30 min. filter and wash with distilled water. It was then suspended in 200 ml of ice-cold water and then diazotized with 1M $NaNO_2$ and 1M HCl at 0 to -5^0C until the reaction mixture started to change the color of iodide paper to violet. The diazotized resin was

filtered, washed with ice cold water and reacted with 5-sulfosalicilyc acid (15 gm taken in 200 ml of 10% NaOH solution) the resulting product was filtered an wash with distilled water followed by dil. NaOH to remove unreacted 5-Sulfosalicylic acid then it wash with dil. HCl and finally again wash with distilled water. Final product was dried and stored in vacuum dessicator. The complete reaction scheme is shown below.



Scheme: Synthesis of 5-SSA-N_2-AXAD-2

Results and discussion

FTIR Spectra:

Infrared sectra of pure AXAD-2 polymer is shown in Fig.1, from the spectra it has been revealed that the polymer shows absorption band at 1400-1200 cm^{-1} suggest the preseance of Ar-CH2-Ar bridge[15]. A peak appired at 1603 cm^{-1} is due to aeromatic ring present in AXAD-2[1]. NO_2-AXAD-2 was confirmed by the prominent two peaks at 1525 and 1347 cm^{-1} which were attributed to N-O asymmetric and symmetric streaching vibration (Fig2) [6]. The NH_2-AXAD-2 was confirmed by IR absorption doublet at 3371 cm^{-1} shows N-H stretching of primary amine (Fig.3). Peak at 2124 cm^{-1} is due to –N=N- stretching (Fig. 4). A very broad band appeared in the region 3504 cm^{-1} may be assigned to the stretching vibration of the phenolic hydroxyl group exhibiting intermolecular hydrogen bonding[16], peak at 832 cm^{-1} shows tetra-substituted aromatic ring[16], peak at 795 and 763 cm^{-1} is due to -CH2- bending [1], peak at 2922 cm^{-1} was due to C-H stretching of aeromatics[16], absorption at 892 cm^{-1} suggest –CH2- wagging. (Fig. 5) [17].

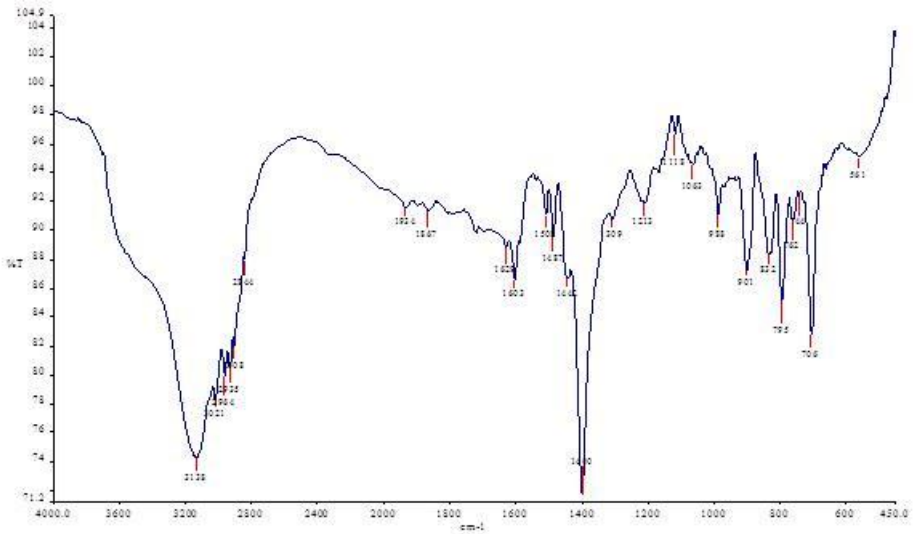


Fig. 1 FTIR Spectrum of pure-AXAD-2 resin

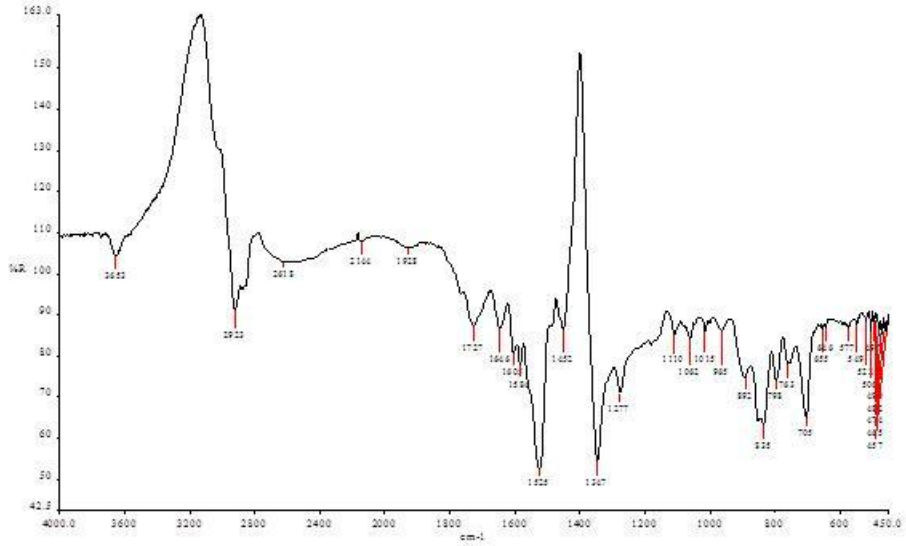


Fig. 2 FTIR Spectrum of NO_2-AXAD-2

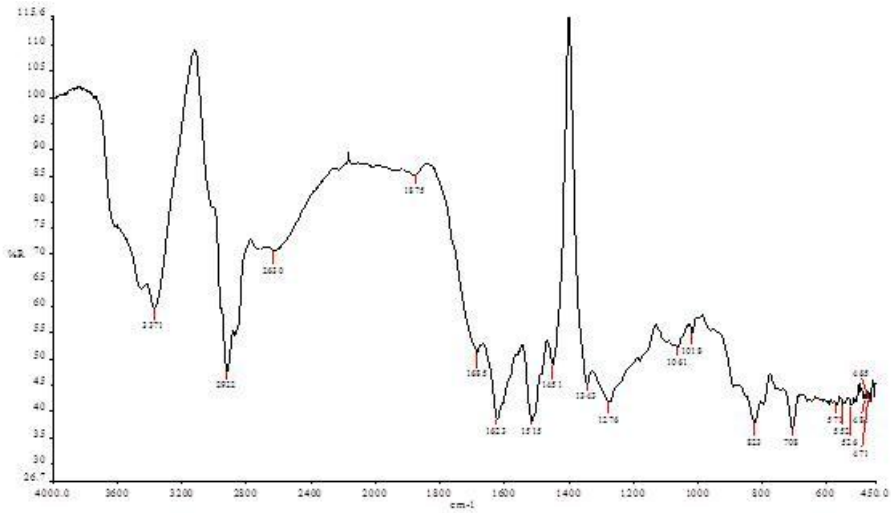


Fig. 3 FTIR Spectrum of NH_2-AXAD-2

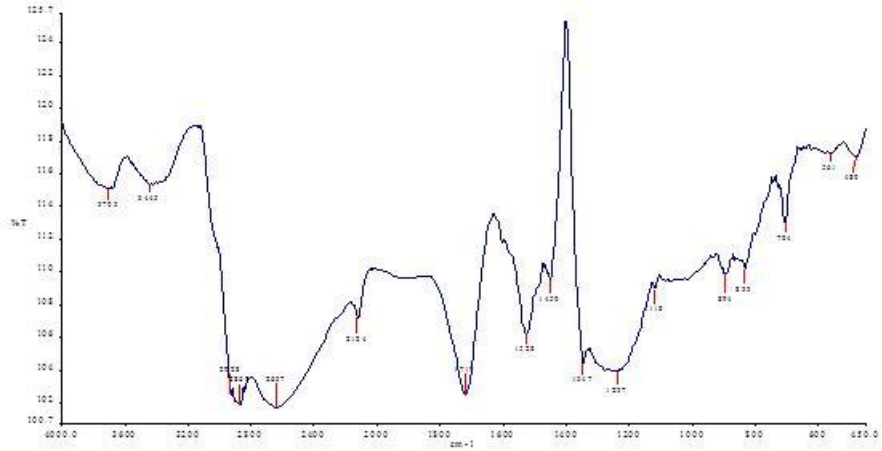


Fig. 4 FTIR Spectrum of Cl N_2-AXAD-2

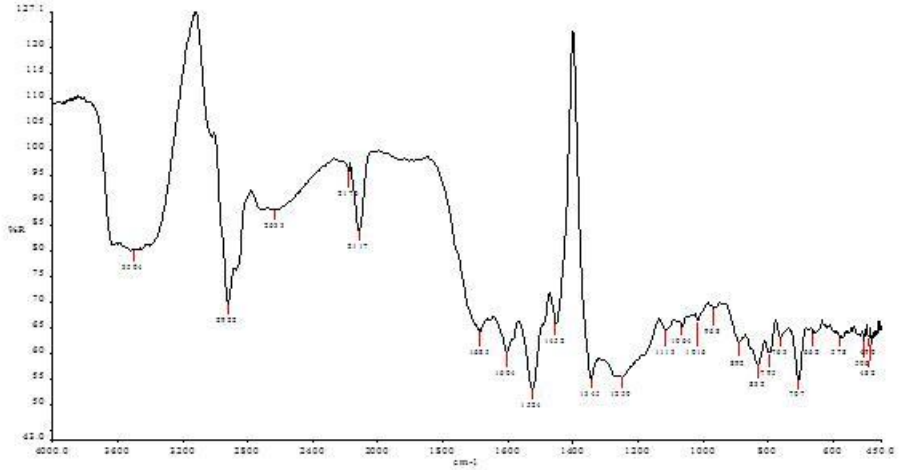


Fig. 5 FTIR Spectrum of 5-SSA-N_2-AXAD-2

Conclusion:

Modified amberlite XAD-2 product (5-SSA-N=N-AXAD2) is conformed by FTIR spectra. Peaks and their values obtained in each spectrum are in good agreement with the standard values for particular functional groups. Position of attachment of 5-SSA to the resin via diazo spacer is not clear as per fingerprint region of FTIR spectrum. Results from IR spectra were found to be in good agreement with given reaction scheme as shown above.

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