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Spectral Analysis of the γ-Irradiated Nuclear Grade Resin—Indion-223

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The infrared spectroscopic measurements have been carried out to follow the course of the radiolytic degradation and bond formation processes occurring in the γ-treated resin samples. IR spectra of untreated resins, spectra in different cationic forms and spectra of γ-irradiated resins are studied.

INTRODUCTION

Cathers[1] was the first to publish data in the year 1965 concerning radiation damages to ion exchangers. Different types of sources like ^{60}Co facilities,[2] electron accelerators[3] and reactors[4] have been employed for exposing ion exchange materials to ionizing radiation. Variety of irradiation conditions have been used in experimental studies which includes ion exchangers exposed to radiation while immersed in water at different pH[5] or in HNO_3 of different concentrations or in acetic acid.[7] Therefore the resistance to radiation of these materials are studied along with the spectral analysis of the γ-irradiated nuclear grade resin.

EXPERIMENTAL

For the present work nuclear grade polystretin-based cation exchange resin supplied by Ion Exchange (India) Ltd., Ambernath, under the brand name Indion-223 is used. All the chemicals used during the course of study were of analytical reagent grade. γ-treatment was accomplished by 2.5 KCi ^{60}Co source. The resin was converted into different ionic forms like Li^+, Cr^{3+} and Fe^{3+}.

Normal and irradiated ion exchange resins and KBr powder used in the preparation of pallet were dried in a desiccator over P_2O_5 till constant weight. The resin was ground in an agate mortar and about 1.5% by weight of it was used for pelletization. Disc of *ca.* 1 mm thickness was made using an evacuated die by applying 10 ton/inch pressure. Fourier transform infrared spectra of these samples were scanned in the region 4000–400 cm^{-1} on computerised FTIR spectrophotometer model FTIR-4200 manufactured by Shimadzu Corporation, Japan. Before use, the instrument was standardized with the aid of polystyrene sample film.

RESULT AND DISCUSSION

The infrared spectrophotometric measurements have been carried out to follow the course of the radiolytic degradation and bond formation processes occurring in the γ-treated resin samples. For comparison, IR spectra of untreated resins are also obtained. It is observed that spectra of polystyrene resin in different cationic forms are practically identical with that of H^+ form. IR spectrum obtained for the untreated H^+ form of the resin is presented in Fig. 1. According to the known data[8] the absorption peak at 675 cm^{-1} corresponds to —C—S bond, 1200–1000 cm^{-1} corresponds to the aromatic mone substitution and absorption at 1420 cm^{-1} is due to the polymeric backbone. Radiation brings dramatical changes in IR spectra of the resins and influences intensity of all these bands. In Table-1 all the observed frequencies and their assignments are given. Typical changes that occur because of γ-irradiation are summarized in Table-2.

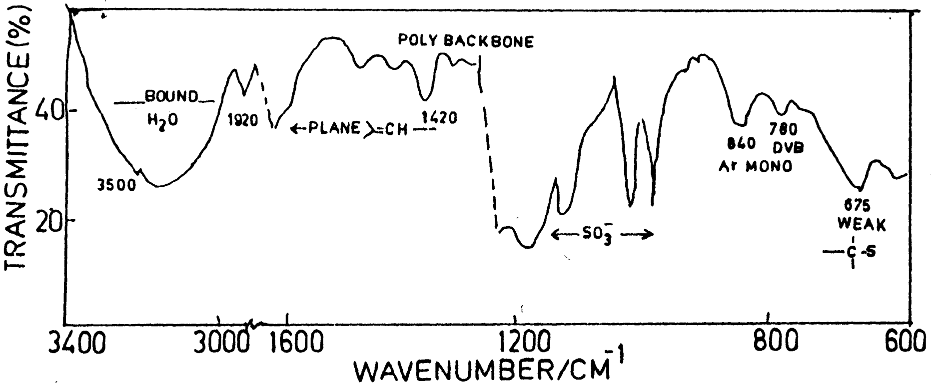


Fig. 1 Infrared spectrum of unipradiated polystyrene nuclear grade cation changer in H^+ form.

TABLE-1

INFRARED SPECTRAL DATA (cm^{-1}) ON THE UNIRRADIATED POLYSTYRENE NUCLEAR GRADE RESIN

Frequency	Characteristics
675	Weak —C—S
840–780	Aromatic mono substitution
1200–1000 (Single band)	—503⁻
1420	Polymeric back bone
1600–1400	def —C—H of benzene
2900	st —C—H of benzene
3500	st —O—H (H_2O) (bound water)

def = Deformation St = Stretching

H^+ form of the resin showed significant instability towards radiation as compared to other forms.[9] In our case Li^+ form is less stable towards radiation as compared to Cr^{3+} and Fe^{3+} salt form. Fe^{3+} form showed high stability even at

TABLE-2
FTIR SPECTRAL ANALYSIS OF γ-IRRADIATED NUCLEAR GRADE POLYSTYRENE RESIN SAMPLES

Dose/MGy	Ionic form	Sample	Observation	Characteristics
6.0	Cr^{3+} and Fe^{3+}	Fine particles in the aqueous affluent	a, b, c	—
6.0	Cr^{3+}	Intact irradiation	a, b, c	—
4.0	Cr^{3+} and Fe^{3+}	Intact irradiated resin particles	No change	—
6.0	Fe^{3+}	Intact irradiated resin particles	No change	—
6.0	Cr^{3+}	Intact irradiated resin particles	a, b, c, d, i	non-substituted benzene
12.0	Cr^{3+}	Intact irradiated resin particles	a, b, c, d, i	Formation
12.0	Fe^{3+}	Intact irradiated resin particles	k no change	ketone
2.0	Li^{+}	Fine particles in the effluent	a	Complete loss of weak —C—S
	Cr^{3+} and Fe^{3+}	Fine particles in the effluent	No change	—
6.0	Li^{+}	Fine particles in the aqueous effluent	b	Complete loss of weak —C—S
			c	Breakage of polymeric backbone
			d	—SO_3— breakage
			e	Generation of double bond weak —SO_4
			g	Loss of aromatic mono substitution
12.0	Li^{+}	Intact irradiated resin particles	a, b, c, d, e, f, g	
			h	Weak —SO_4^{2-} double band generation
			i	ion deformation

a—Disappearance of band at 680 cm^{-1}, b—Disappearance of band at 680 cm^{-1}, c—Disappearance of band at 1420 cm^{-1}, d—Broadening of bands over 1300–1000 cm^{-1}, e—New band at 980 cm^{-1}, f—New band at 570 cm^{-1}, g—Disappearance of band at 840 and 780 cm^{-1}, h—New peak at 600 cm^{-1}, i—New peak at 1380 cm^{-1}, j—New peak at 1580 cm^{-1}, k—New peak at 1710 cm^{-1}

12 MGy dose. IR spectra of the resin in two different states after irradiation namely (i) intact particles and (ii) fine particles observed in the effluent showed the following common features. Selected IR spectra are given in Figs. 2–4.

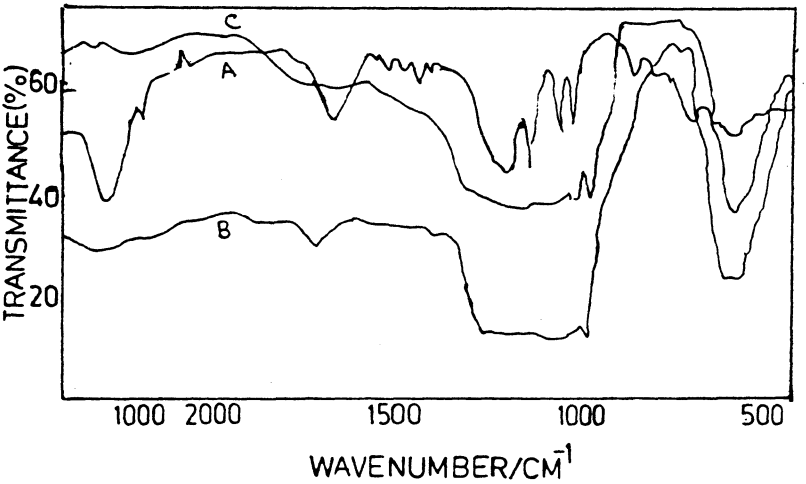


Fig. 2 Infrared spectra of pure Li^+ form of polisterene nuclear grade resin (A) and of intact irradiated resin after 12 mgy dose (B) and it's effulent fine practicals wllected after 6 mgy dose (C)

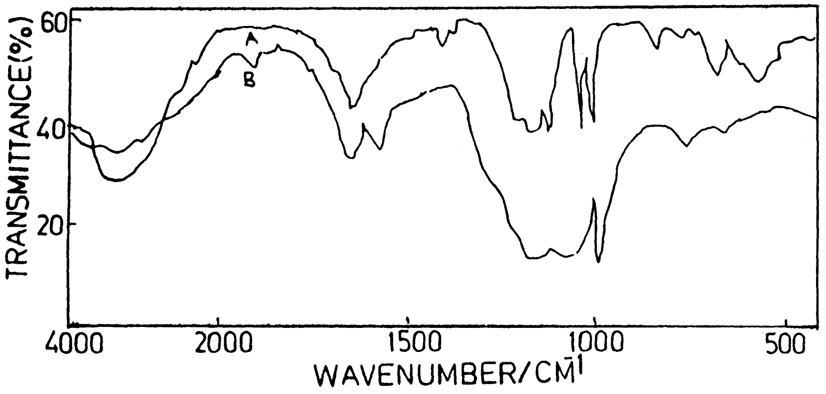


Fig. 3 Infrared spectra of Cr^{3+} form of polisterene nuclear grade resin pure sample (A) after 12 mgy dose (B).

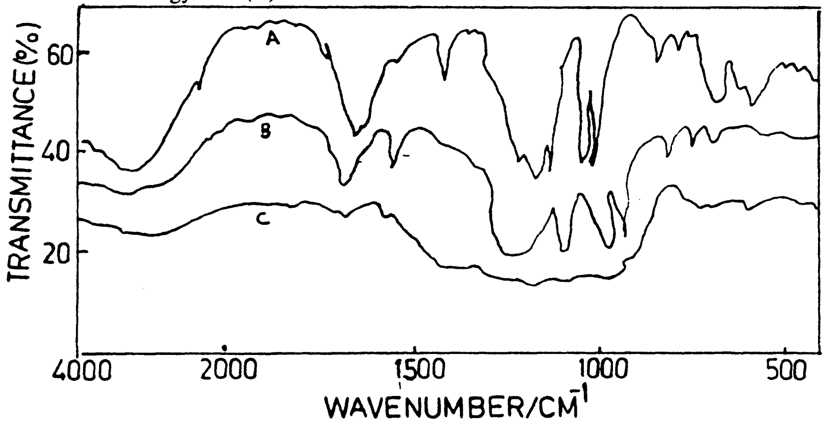
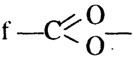


Fig. 4 Infrared spectra of nuclear grade resin in Fe^{3+} form pure sample (A) after 6 mgy dose (B) and of intact irradiate resin agter absorbed dose of 12 mgy (C)

The most common and the significant observation is the broadening of bands over the range 1200–1000 cm^{-1}; it has been attributed to the scission of —C—SO_3— bond. Second observation is the disappearance of band at 1420 cm^{-1} which indicates the breakage of the polymeric backbone (cross-links). Simultaneously, formation of a new peak at 990–980 cm^{-1} is observed which indicates the generation fo RCH= CH_2 groups.

A new peak appeared at 780 cm^{-1} after an absorbed dose of 6 MGy and at 1350 cm^{-1} after the absorption of 12 MGy dose by the Cr^{3+} form of polystyrene resin. These peaks appear very slight for Fe^{3+} form of resin at still higher dose namely 12 MGy. In the case of intact irradiated particles of Li^+ form fo resin, a new peak appeared at 980 cm^{-1}. Here the particles were collected from its effluent after dose 6 MGy which is attributed to the formation of  $-C{\scriptstyle\begin{matrix}\nearrow O\\ \searrow O\end{matrix}}-$ group. This peak appears for Cr^{3+} and Fe^{3+} forms of resin at higher radiation doses. Li^+ form also indicates large broadening of —SO_3^- bands (1200–1000 cm^{-1}) as compared to Cr^{3+} and Fe^{3+} form showing the tremendous radiation damage to the exchange sites. Recent studies by Prini *et al*[10, 11] on hydrothermal decomposition on polystyrene resin at 250°C in the Li^+ and H^+ forms found similar changes in our case of radiation decomposition. Resins in the salt form were observed to be more stable towards hydrothermal degradation. Their results differed with regards to oxidation of polymeric backbone. No bands due to —C—O—C or C=O groups were observed even upon heating up to seven days. Dedgaonkar *et al.*[9] reported similar results on resin in Sr^{2+}, Ba^{2+}, Zn^{2+}, Cd^{2+}, Al^{3+} and Fe^{3+} form. In IR spectrum concurrently, formation of broad band at 600 cm^{-1} appears which is characteristic formation of free —SO_4^{2-}.

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