

## Effect of $\gamma$ -Irradiation on the Thermal Properties of Nuclear Grade Polystyrene Cation Exchange Resin—Indion-223

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For determination of optimum drying temperature of resin and to investigate its thermal characteristics, simultaneous TGA, DTA curves of resin sample have been recorded. For this unirradiated and irradiated resin samples in  $\text{Fe}^{3+}$ ,  $\text{Cr}^{3+}$  and  $\text{Li}^+$  ionic forms were used. It is seen that irradiation does not affect the overall decomposition for  $\text{Cr}^{3+}$  and  $\text{Fe}^{3+}$  forms of resin irradiated up to 5 MGy  $\gamma$ -dose. It is recorded that the ion exchangers in  $\text{Cr}^{3+}$  and  $\text{Fe}^{3+}$  formed subsequent to dehydration process, depolymerise exothermally over the temperature range 350-550°C.

### INTRODUCTION

Thermogravimetric and differential thermal analysis of nuclear grade air-dried ion exchange resin and investigation of its thermal characteristics was done.<sup>1</sup> Irradiation is an integral part of radiochemical processes and nuclear process industry. A number of uses of ion exchange materials, for example, hot eluting solutions of rare earths<sup>2</sup> and transplutonic elements need high temperature.<sup>3</sup> Hence it was thought worthwhile to investigate their effects on the thermal properties of ion exchange resin. This paper deals with thermogravimetric and differential thermal analysis of nuclear grade, strongly acidic, polystyrene cation exchange resin supplied by Ion Exchange (India) Ltd., Ambarnath (Bombay) under the brand name Indion-223 in various salt forms.

### EXPERIMENTAL

First the resin samples were prepared in  $\text{Fe}^{3+}$ ,  $\text{Cr}^{3+}$  and  $\text{Li}^+$  ionic forms. Irradiation of samples was carried out using a cavity type 2.5 Kci (nominal) cobalt-60, 400A-gamma chamber having a dose rate 0.25 Mrad/h. Simultaneous TGA and DTA of resin samples were carried out to determine optimum drying temperature for resin and to investigate their thermal characteristics. For this the unirradiated and irradiated resin samples of  $\text{Fe}^{3+}$ ,  $\text{Cr}^{3+}$  and  $\text{Li}^+$  ionic forms were heated thermogravimetrically in an inert atmosphere of nitrogen using 'Thermal Analyzer STA-780 Series'. The heating rate was maintained at 10°C/min and chart speed at 200 mm/h. The sample holder was a platinum crucible attached to Pt/Rh thermocouple which was coupled with CPC-706 temperature programmer.

## RESULTS AND DISCUSSION

Typical TGA, DTA curves for unirradiated and irradiated resin samples are shown in Fig. 1 and Fig. 2. Data obtained from such curves are presented in Table-1. It is seen that all the ion exchange resin samples, irrespective of their

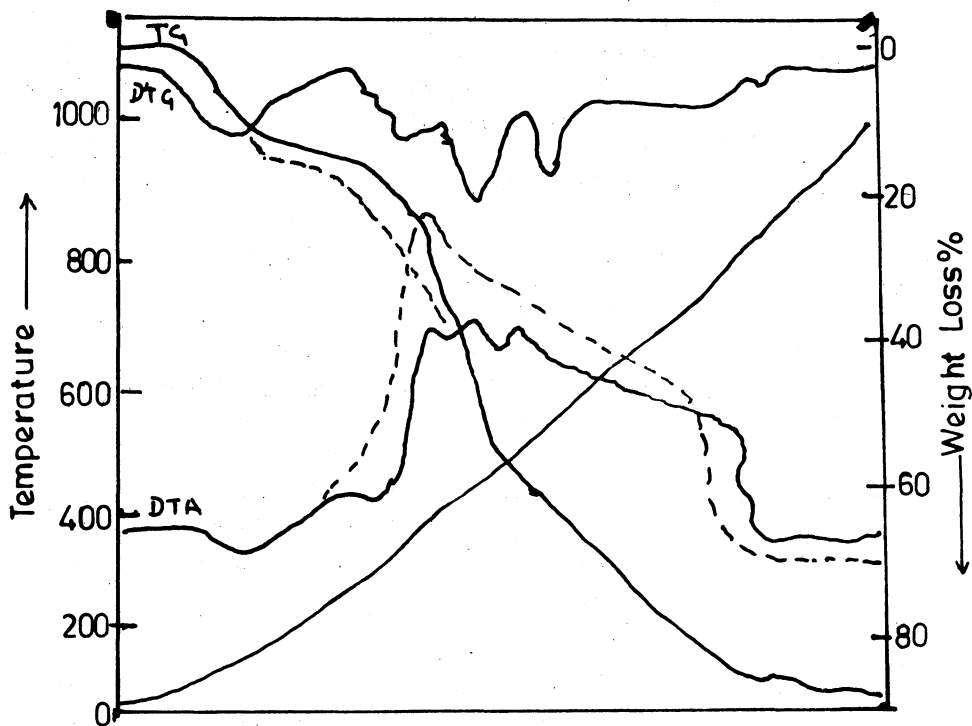


Fig. 1. Radiation effect on the DTA, DTG and TG for the  $\text{Fe}^3$  form of polystyrene nuclear grade resin, solid line for 5 MGy dose, weight 100 mg, rate  $10^\circ\text{C}/\text{min}$ .

TABLE-1  
EFFECT OF GAMMA-RADIATION ON THERMAL PROPERTIES OF AIR-DRIED  
POLYSTYRENE NUCLEAR GRADE CATION EXCHANGER IN DIFFERENT  
IONIC FORMS

Ionic form	Unirradiated		Dose/MGv	Irradiated	
	Endotherm/ $^\circ\text{C}$	Exotherm/ $^\circ\text{C}$		Endotherm/ $^\circ\text{C}$	Exotherm/ $^\circ\text{C}$
$\text{Li}^+$	121	400-450	2.5	121	400-450
	25% loss		5.0	121	400-440
$\text{Cr}^{3+}$	120-310	475	2.5	120	420-450
	20% loss		5.0	120	New Product
$\text{Fe}^{3+}$	120-310	400-450	2.5	120.300	390-440
	25% loss		5.0	26% loss	
				28% loss	

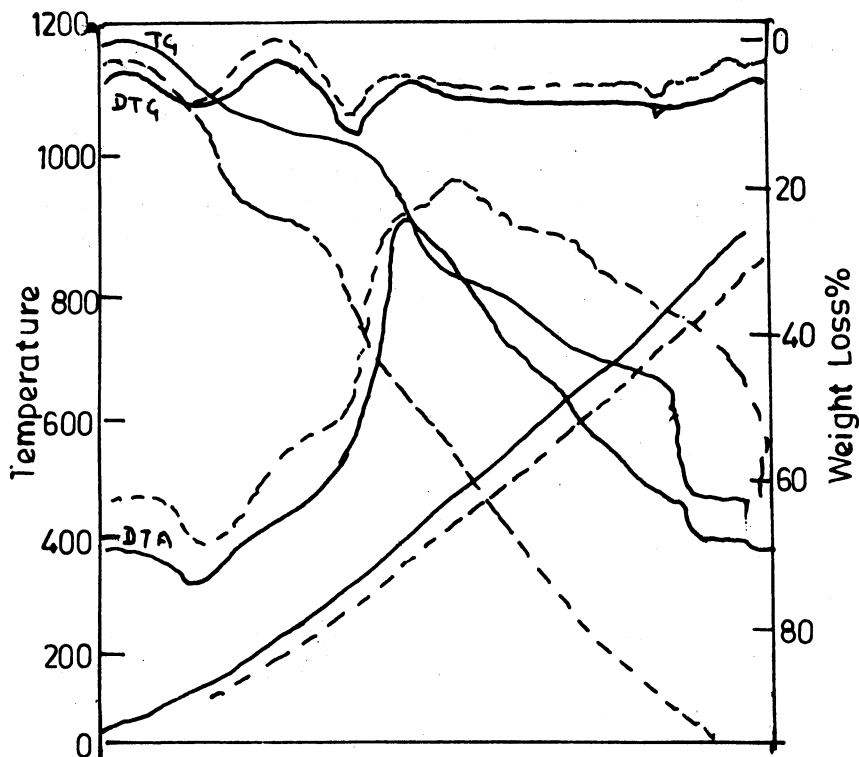


Fig. 2. Radiation effect on the DTA, DTG and TG for the  $\text{Cr}^{3+}$  form of polystyrene nuclear grade resin, solid line for 2.5 MGy dose, weight 100 mg, rate  $10^\circ\text{C}/\text{min}$ .

ionic form, show an endotherm at  $120^\circ\text{C}$ . This indicates loss of bound water from resin matrix.  $\text{Li}^+$  and  $\text{Fe}^{3+}$  forms of the resin have relatively larger water content of ca. 20% in the  $\text{Cr}^{3+}$  form. This varying trend in the moisture content is because of the hydration shell of the ion in the resin matrix. A second endotherm arises due to the elimination of  $\text{H}-\text{SO}_3$  groups observed at  $300-330^\circ\text{C}$ .

Again it is observed that the irradiation does not affect largely the overall decomposition of  $\text{Cr}^{3+}$  and  $\text{Fe}^{3+}$  forms of resin irradiated upto 5 MGy. It undergoes 4% weight loss during the endothermic reaction occurring at  $120^\circ\text{C}$ , where a radiation induced sulphonyl cross-linkage is possible. The second endotherm has actually been observed in both the cases. Subsequent to the dehydration process, the ion exchangers depolymerise exothermally over the temperature range  $350-550^\circ\text{C}$ . This is in good agreement with the results reported on Tulsion-T-42-P ion exchanger resin by Dedgaonkar *et al.*<sup>4</sup> in the  $\text{Ba}^{2+}$ ,  $\text{Sr}^{2+}$  and  $\text{Fe}^{3+}$  forms.

The exothermal peak at ca.  $450^\circ\text{C}$  in the  $\text{Fe}^{3+}$  form of resin may be attributed to the decomposition of  $\text{Fe}_2(\text{SO}_4)_3$  likely to be formed during desulphonation. This decomposition temperature was lowered by  $10^\circ\text{C}$  in the gamma treated samples at doses 2.5 MGy and 5.00 MGy indicating desulphonation and the subsequent  $\text{Fe}_2(\text{SO}_4)_3$  formed itself undergoes radiolytic decomposition beyond a certain dose. In the same way the thermally decomposed end products of lithium

and chromium forms of the resin could be likely assumed to their respective sulphates. However, it is very difficult to understand the changes in the exothermic decomposition of these resins upon irradiation without a proper interpretation of their unirradiated decomposition characteristics.

### REFERENCES

1. A.A. Vasilev, *Zhur. Priki Khim.*, **30**, 1022 (1957).
2. L. Wo and J.J. Massone, *Prakt. Chem.*, **5**, 21 (1957).
3. Preobranzhenskii and A.V. Kalyamin, *Zhur. Neorg. Khim.*, **2**, 1164 (1957).
4. V.G. Dedgaonkar, Sunil Waghmare, *Proc. Symp. Radiochem. and Radiation Chem.*, RES-8, Kanpur (1985).

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