

Thermal Degradation Studies of Some Strongly Acidic Cation Exchange Resins

Pravin U. Singare^{*1}, Ram S. Lokhande², Rupa S. Madyal³

¹Department of Chemistry, Bhavan's College, Andheri, Mumbai, India

²Department of Chemistry, University of Mumbai, Vidyanagari, Santacruz, Mumbai, India

³Department of Chemistry, South Indians' Welfare Society College, Sion, Mumbai, India

E-mail: pravinsingare@vsnl.net

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Abstract

The thermal degradation of some sulfonic cationites namely Amberlite IR-120, Indion-223 and Indion-225 was investigated using instrumental techniques like thermal analysis (TG) and Scanning Electron Microscopy (SEM). Fourier Transform Infrared Spectroscopy (FTIR) was used to characterize the resins degradation steps. The sulfonic cationites undergo degradation through dehydration, followed by decomposition of sulfonic acid functional groups liberating SO₂. The thermogravimetric analysis of above cationites at higher temperature up to 520°C, show mass loss of 61.61% and 25.43% respectively for Indion-223 and Indion-225, while Amberlite IR-120 cationite get burned off completely.

Keywords: Sulfonic Cationites, Thermal Degradation, FTIR, SEM, Nuclear Resin, Thermal Analysis

1. Introduction

Ion-exchange resins are produced and commercialized in a wide range of formulations with different characteristics, and have now a large practical applicability in various industrial processes, such as chemical, and nuclear industry for treatment of liquid waste [1-16]. For their versatile properties, the cationic resins are used both in the ion exchange area and in the heterogeneous catalysis field [17]. These resins exhibit a high exchange capacity and an excellent osmotic shock resistance. So, the cationic resins, produced with a high degree of purity, became important as catalysts in various food technologies [18] and for purification in heavy-water moderated nuclear reactors in nuclear industries [19-21]. In many cases their use is limited by the relatively low thermal stability [22]. Hence, knowledge of the thermal behavior of cation exchange resins is necessary. Abundant data exist on the thermal degradation of anion exchange resins [23-25] and on carboxylic cationites with low acidity [26-27]; literature seems to offer relatively poor information on polystyrene-divinylbenzene sulfonic cationites [28-33]. Therefore, in the present investigation thermal degradation of strongly acidic sulfonic cationites was performed to understand the degradation steps and to compare the relative thermal stability.

2. Experimental

2.1. Materials

The following commercial cationites were used:

- Strongly acidic gel-type resin with sulfonic acid functionality based on styrene-divinylbenzene matrix: Amberlite IR-120 (Rohm and Haas Co, USA).
- Nuclear grade strongly acidic gel-type resin with sulfonic acid functionality based on styrene-divinylbenzene matrix: Indion-223 (Ion Exchange India Ltd., Mumbai).
- Strongly acidic gel-type resin with sulfonic acid functionality based on styrene-divinylbenzene matrix: Indion-225 (Ion Exchange India Ltd., Mumbai).

The details regarding the physical properties of cationites used are given in **Table 1**.

The soluble impurities of the resins were removed by repeated soxhlet extraction using water and occasionally with distilled methanol to remove non polymerized impurities. The resins were then dried over P₂O₅ in desiccators at room temperature.

2.2. Thermal Analysis

The thermogravimetric experiments were performed on a

Table 1. The main characteristics of the investigated cationites.

Cationites	Exchange Capacity (mEq/mL)	Particle size	Moisture Content (%)	Maximum Temperature Stability °C
Amberlite IR-120	1.9	16 - 50 mesh	45	121
Indion-223	1.8	0.3 - 1.2 mm	53	120
Indion-225	2.0	0.3 - 1.2 mm	50	120

DTG-60H, (Shimadzu, Japan) thermal analysis system between 30°C - 550°C using aluminum cell (6 mm in diameter and 2.5 mm in depth). The measurements of resin samples were carried out in nitrogen flow (50 mL·min⁻¹) at heating rate ($\beta = 10$ K·min⁻¹). The mass of resin sample used was ~5 - 20 mg. In order to characterize the decomposition steps of the investigated ion-exchange resins, FTIR and Scanning Electron Microscopy (SEM) were used in addition to thermal analysis.

2.3. FTIR Spectra

FTIR spectra (in 4000 - 450 cm⁻¹ range) of thermal decomposed samples, up to the characteristics mass-loss steps temperatures, were recorded in KBr pellets (2 mg cationite/200 mg KBr) using a FTIR PerkinElmer 1750 spectrophotometer.

Since in the thermal analysis, the major weight loss was observed between 200°C - 400°C, the resin samples were heated in an oven for 3h at 10°C higher temperatures above the maximum operating temperature, and also at 200 and 400°C. The thermal degradation of resin was studied by comparing the spectra of fresh and heated resin samples.

2.4. Scanning Electron Microscopy (SEM)

The thermal degradation studies of ion exchange resins was also studied by examining the surface morphology of fresh resin samples and samples heated at 400°C using JSM-6380LA Scanning Electron Microscope (Jeol Ltd., Japan). The powders were precisely fixed on an aluminum stub using double-sided graphite tape and then were made electrically conductive by coating in a vacuum with a thin layer of carbon, for 30 seconds and at 30 W. The pictures were taken at an excitation voltage of 10 - 15 KV and a magnification of $\times 100$ and $\times 130$.

3. Results and Discussion

3.1. Characterization and Thermal Degradation Study of Amberlite IR-120:

3.1.1. TGA Analysis

Figure 1 represents a dynamic weight loss profile of

Amberlite IR-120 from room temperature to 550°C. Thermogravimetric curve of Amberlite IR 120 shows 22% weight loss up to 200°C due to moisture content and the weight decreased gradually till 400°C. Above 400°C whole compound burned off completely and weight loss measurement was not possible.

3.1.2. FTIR Analysis

Figure 2 shows the IR spectrum of the fresh resin sample Amberlite IR-120. The bands at 2923 and 2876 cm⁻¹ are due to the aliphatic C-H stretching absorbance of methyl group in the main chain and in aromatic rings and of methylene group respectively. SO₂ asymmetric stretching at 1382 cm⁻¹. Strong band at 1652 cm⁻¹ indicates aromatic C=C bond. The four sharp peaks at 1009 cm⁻¹, 1037 cm⁻¹, 1126 cm⁻¹, 1186 cm⁻¹ are due to SO₃ symmetric stretching. The peaks at 1500 - 1600 cm⁻¹ are due to deformation and skeletal vibrations of C-H in DVB. Bands appear at 2366 cm⁻¹ which may be assigned to O-H stretching vibration originating from the polymer.

At 200°C, the 21% weight loss was observed without any loss of peak in the IR spectra. At 400°C IR investigation indicates that the peaks in the region 1500 - 1000 cm⁻¹ either shows a general broadening or no longer exists (**Figure 3**). Non-existence of the SO unit was confirmed by the absence of the peaks from 1500 - 1000cm⁻¹, with decomposition of the functional group i.e. sulfonic portions of the ring. But slight broadening of the band is observed at 1652 cm⁻¹ for aromatic C=C bond remained unchanged. This corresponds to the few chain scissions in the DVB matrix.

3.1.3. SEM Analysis

Figure 4, shows the surface morphology of Amberlite IR-120 resins at room temperature indicating plane spherical structure. At 400°C, resin show large cracks and thread like appearance on the surface (**Figure 5**).

3.2. Characterization and Thermal Degradation Study of Indion-223:

3.2.1. TGA Analysis

Thermogravimetric curve of Indion—223H⁺ shown in **Figure 6**. The 30% weight loss up to 200°C can be attributed to moisture content. The second major weight loss begins at 270°C and ends at 400°C, which might be due to slow degradation of side chain and loss of sulphonic functional group. The mass loss from 400°C to 521°C was gradual which might be due to degradation of styrene/DVB matrix.

3.2.2. FTIR Analysis

The IR spectrum of Indion-223 in the 3700 - 400 cm⁻¹

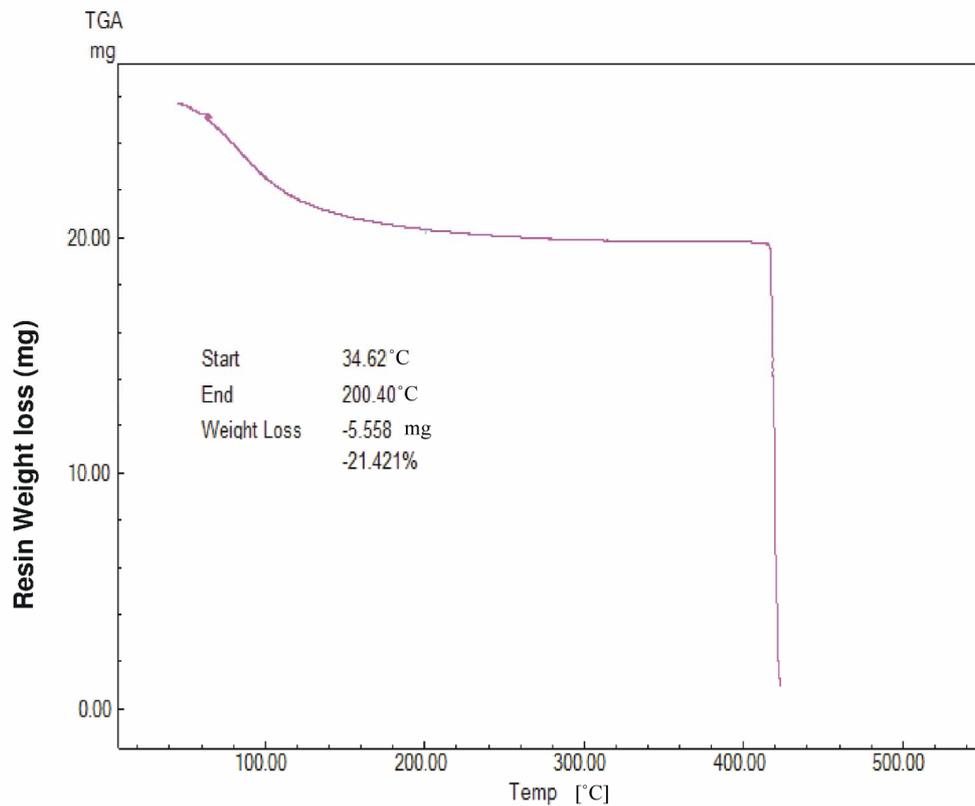


Figure 1. TG curve of Amberlite IR-120.

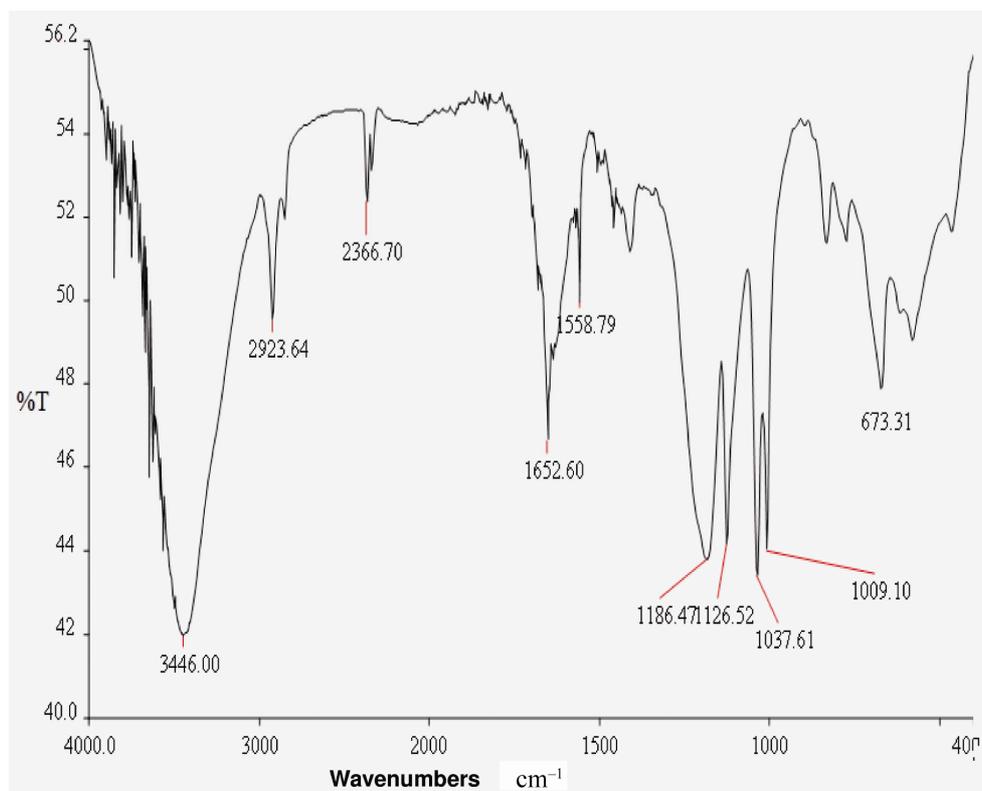


Figure 2. IR spectrum of Amberlite IR-120 at room temperature.

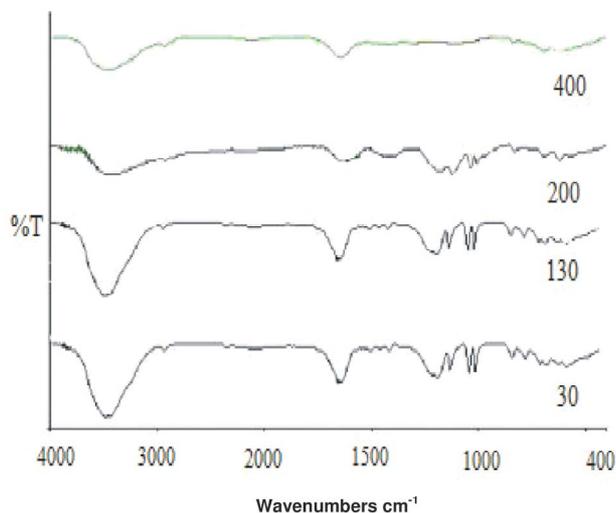


Figure 3. IR spectrum of Amberlite IR-120 at different temperature.

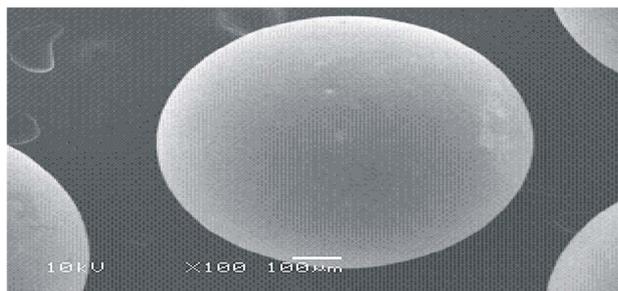


Figure 4. Scanning electron micrograph of the surface of the Amberlite IR-120 at room temperature.

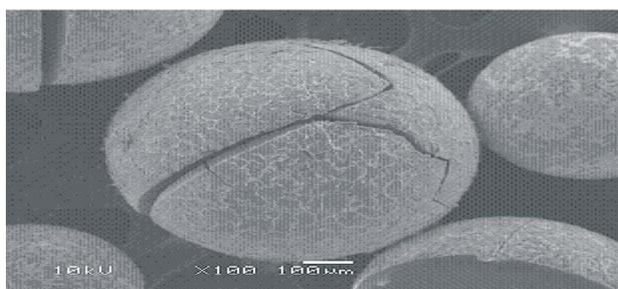


Figure 5. Scanning electron micrograph of the surface of the Amberlite IR-120 at 400°C.

region is shown in **Figure 7**. FTIR spectral analysis shows SO_3 sharp symmetric stretching band at $1005 - 1126 \text{ cm}^{-1}$. O-H stretching at 2364 cm^{-1} . S-O stretching at 668 cm^{-1} . SO_2 asymmetric stretching at 1382 cm^{-1} . C=C aromatic nucleus skeletal vibration band at $1500 - 1600 \text{ cm}^{-1}$ and OH hydrogen bonded broad stretching band at $3200 - 3500 \text{ cm}^{-1}$. The band at 2900 cm^{-1} attributed to C-H stretching vibrations in the main chain and in aromatic rings; the peaks at $1500 - 1600 \text{ cm}^{-1}$ are due to deformation and skeletal vibrations of C-H in Poly-

styrene/DVB.

FTIR investigation shows that that strong bands at 668 cm^{-1} , 1019 cm^{-1} and 1382 cm^{-1} which are related to SO and 2364 cm^{-1} stretching frequency of C-H units remained upto 200°C , but at 400°C both the groups disappeared (**Figure 8**). This indicates that at 200°C the 29.7% weight loss is due to moisture, whereas 31.8% loss is due to the functional group *i.e.* SO and breaking of C-H bond.

3.2.3. SEM Analysis

Surfaces of resins at room temperature (**Figure 9**) and at 400°C (**Figure 10**) were examined by a Jeol JSM-6380LA scanning electron microscope. It was found that at 400°C resin showed crack in the spherical structure which supports breaking of polymer matrix at that temperature.

3.3. Characterization and Thermal Degradation Study of Indion-225:

3.3.1. TGA Analysis

Thermogravimetric curve of Indion-225 H^+ is shown in **Figure 11**. Thermogravimetric curve shows $\sim 13\%$ weight loss up to 200°C , corresponding to the moisture content. Degradation of resin between $270^\circ\text{C} - 340^\circ\text{C}$ takes place sharply and further gradually up to 521°C which might be due to decomposition of the sulphonic functional group with rapid evolution of SO_3 or SO_2 showing mass loss of $\sim 12.5\%$.

3.3.2. FTIR Analysis

The IR spectrum of Indion-225 H^+ in the $3700 - 400 \text{ cm}^{-1}$ region is shown in **Figure 12**. FTIR spectral analysis shows four sharp peaks at 1009 cm^{-1} , 1037 cm^{-1} , 1126 cm^{-1} , 1186 cm^{-1} are due to SO_3 symmetric stretching. O-H stretching at $2900 - 2400 \text{ cm}^{-1}$. S-O stretching at 672 cm^{-1} . C=C aromatic nucleus skeletal vibration band at $1550 - 1670 \text{ cm}^{-1}$ and OH hydrogen bonded broad stretching band at $3200 - 3500 \text{ cm}^{-1}$. The sharp peak at 2900 cm^{-1} range attributed to C-H stretching skeletal vibrations of C-H in Polystyrene/DVB.

The degradation occurs in a single step and mass loss is 25% up to 521°C . SO_2 is the dominate product evolved at 190°C and 380°C while water is also present during the entire degradation pathway but in relatively small amount. Loss of SO unit can also be confirmed by IR investigation. The characteristic band for SO unit at 1009 cm^{-1} , 1037 cm^{-1} , 1126 cm^{-1} , 1186 cm^{-1} are no longer exist (**Figure 13**). The evolution of water occurs in the same temperature regime as SO_2 , between 185 and 400°C , as spectrum at 200°C shows broadened bands indicating slight decrease in SO content. This is likely due to the

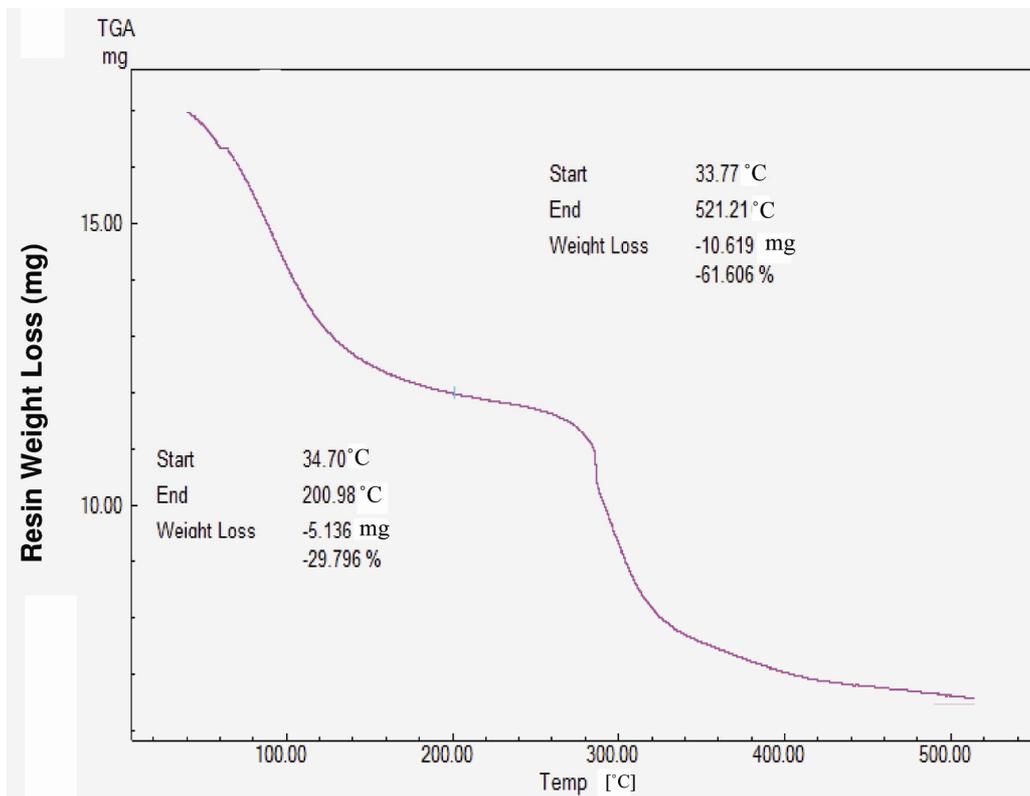


Figure 6. TG curve of Indion-223.

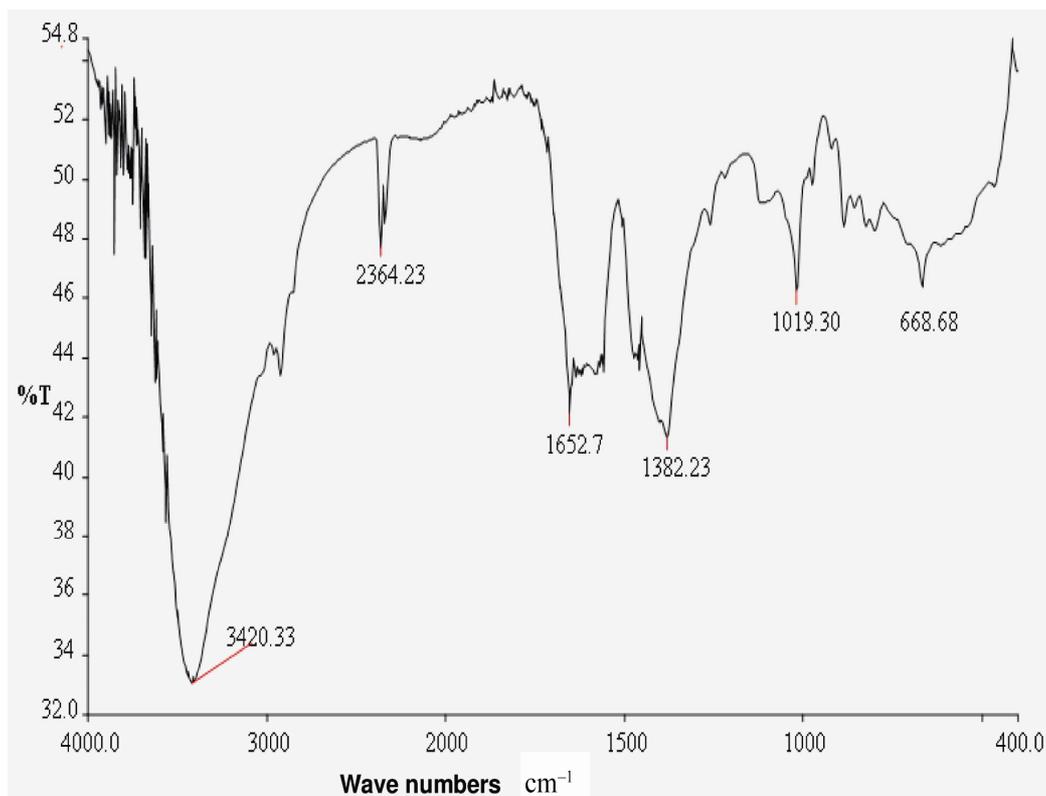


Figure 7. IR spectrum of Indion-2223 at room temperature.

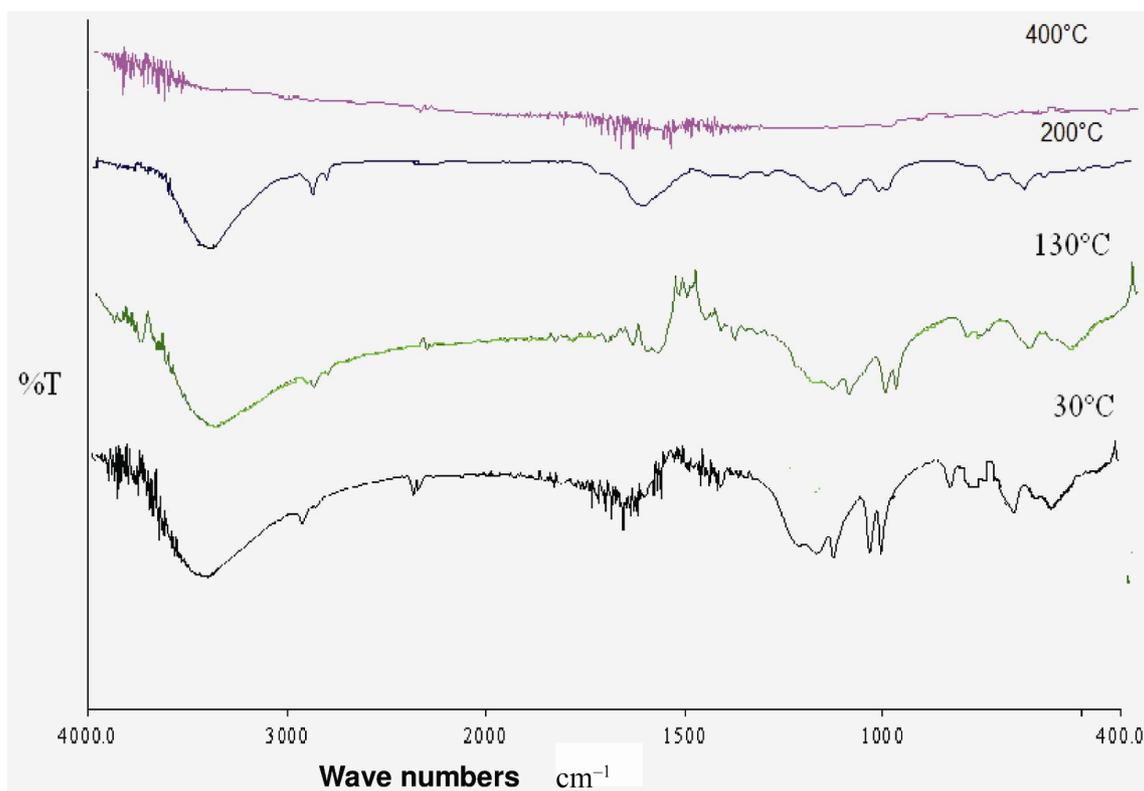


Figure 8. IR spectrum of Indion-2223 at different temperature.

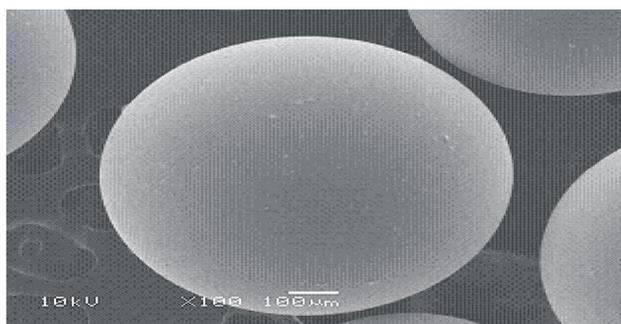


Figure 9. Scanning electron micrograph of the surface of the Indion-223 at room temperature.

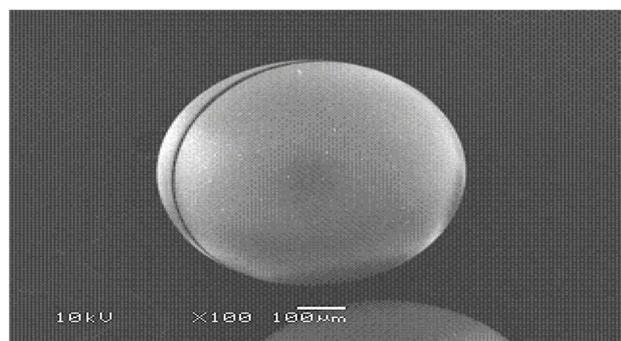


Figure 10. Scanning electron micrograph of the surface of the Indion-223 at 400°C.

formation of sulfurous acid by the process noted above. Water is also evolved early in the degradation and this is likely due to the loss of physically combined water as has been observed for the other compounds [34].

3.3.3. SEM Analysis

Figure 14 shows the surface morphology of the Indion-225 H⁺ at room temperature indicating plane spherical structure. Similar to Indion-223 H⁺; Indion-225 H⁺ also shows crack on the spherical surface when heated at 400°C (Figure 15)

4. Conclusions

From the FTIR analysis of three sulfonic acid cationites, it was observed that the degradation takes place through dehydration, followed by decomposition of sulfonic acid functional groups. The thermal analysis shows that up to 200°C, Indion-225 cationite shows mass loss of only 13%, as against mass loss of 21% and 30% shown by Amberlite IR-120 and Indion-223 respectively. The thermal analysis at a higher temperature up to 520°C, Amberlite IR-120 cationite gets completely burned, while Indion-225 and Indion-223 shows total mass loss of 25% and 62% respectively. Hence the thermal stability of three cationites increases in the order of Amberlite

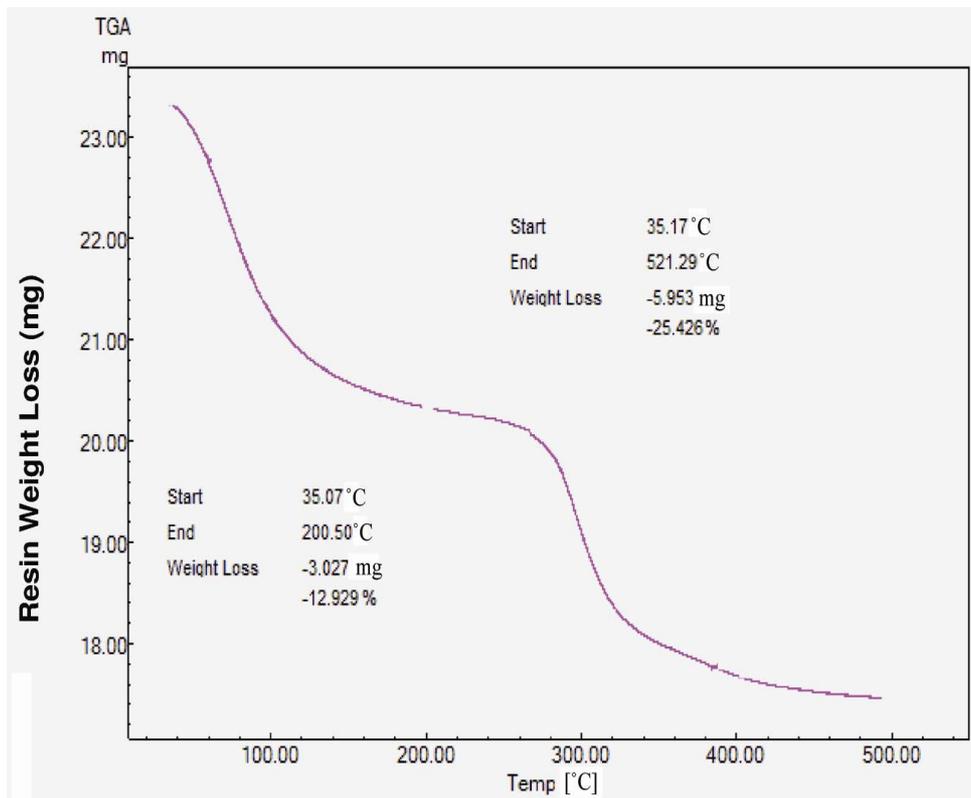


Figure 11. TG curve of Indion-225.

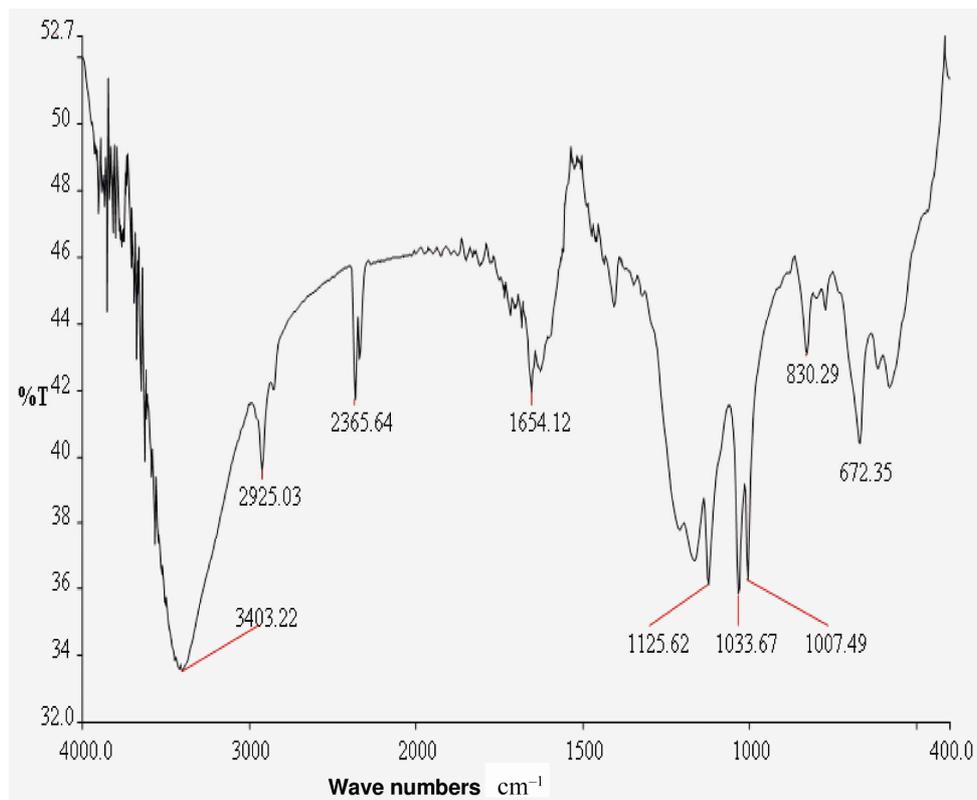


Figure 12. IR spectrum of Indion-2223 at room temperature.

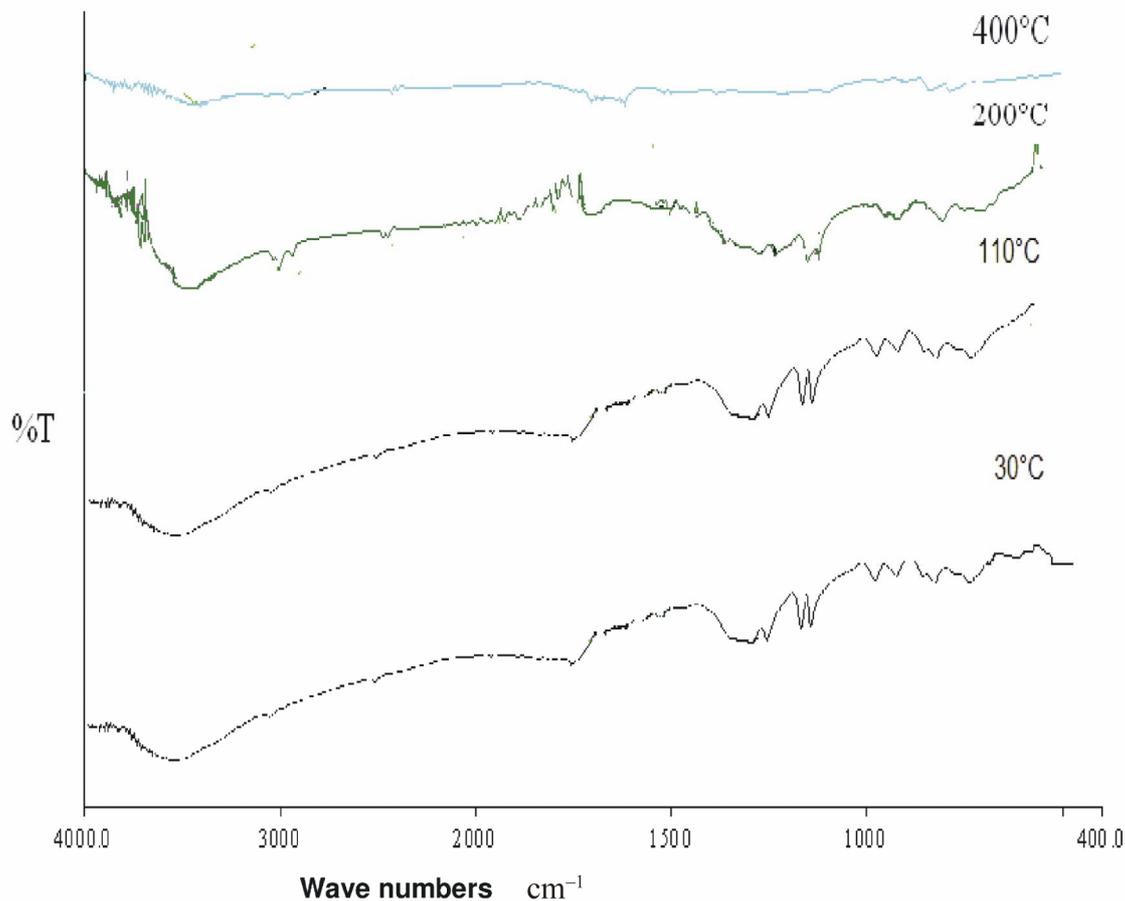


Figure 13. IR spectrum of Indion-2223 at different temperature.

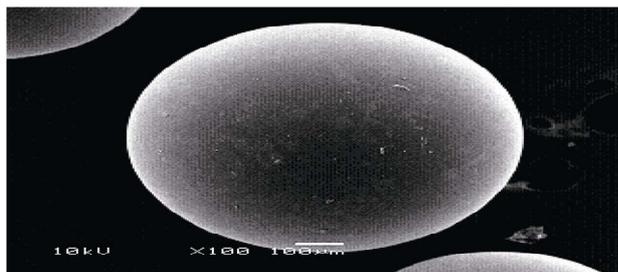


Figure 14. Scanning electron micrograph of the surface of the Indion-225 at room temperature.

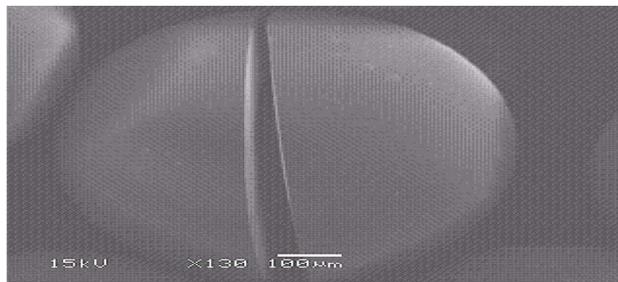


Figure 15. Scanning electron micrograph of the surface of the Indion-225 at 400°C.

IR-120 < Indion-223 < Indion-225.

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