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Studies on thermal behaviors for barium ion bound guar sample

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Abstract
Guargum is a water soluble natural polymer and it has great industrial use. Aqueous solution of guargum binds Y^{3+} and Cu^{2+} when pH is raised. Industrial guar sample can bind Ba^{2+} at higher pH. In this present investigation binding of Ba^{2+} ion by industrial guar sample has been understood by TGA, DTA and DSC analysis. These studies indicate, thermograms for industrial guar sample, guar - Ba^{2+} sample and barium hydroxide are different and hence guar - Ba^{2+} sample has polymeric part. DSC studies support complexation of guar sample involving Ba^{2+} ions in guar - Ba^{2+} sample.

Keywords: Guargum, barium ion binding, thermogravimetry, differential thermal analysis, differential scanning calorimetry, ceramic oxide

1. Introduction

Guargum and its derivatives are among the most important water soluble polymers. Structure of guargum (Fig.1) is well known. Guargum has great industrial use [1, 3]. Aqueous solution of guargum binds metal ions like Y^{3+}, Cu^{2+} etc. when pH is raised. To study involvement of guar sample in binding Ba^{2+}, IR spectral technique, SEM and EDS methods have been used earlier and reported by me [4]. In this present investigation, further study of binding Ba^{2+} by guar sample has been done by thermal analysis, especially to understand whether guar – Ba^{2+} sample has explosive nature or not. Earlier it has been found that yttrium ion bound guargum has some explosive tendency [5]. DTA, TGA and DSC thermograms for industrial guar sample, guar - Ba^{2+} sample and barium hydroxide, support presence of polymeric part in guar - Ba^{2+} sample and ion binding by guar sample. This work may help in understanding involvement of backbone in guargum-*graft*-acrylamide to bind Ba^{2+} ion.

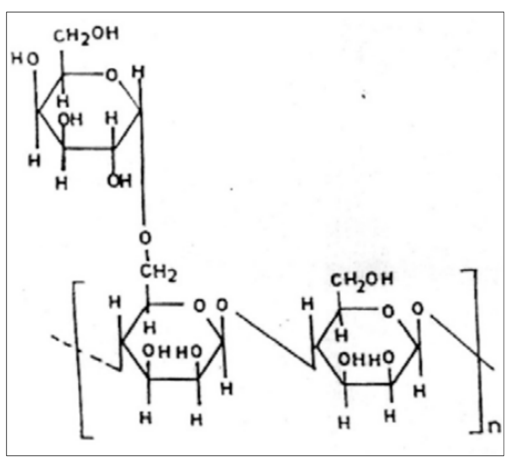

Fig 1: Structure of guargum.

2. Materials and Methods

2.1 Barium nitrate solution

Barium nitrate solution has been prepared by dissolving barium nitrate in distilled water to get a saturated solution.

2.2 Barium hydroxide
Approximately 10 ml barium nitrate solution is mixed with 10 ml 60% sodium hydroxide solution. Mixture is allowed to stand for 10 mins. Filtered, washed with little distilled water and dried in an oven.

2.3 Industrial guar sample
Guar sample used in this work is obtained from the Paper industry: ITC Limited, Paper Boards and Specially Papers Division, Unit: Tribeni, Hooghly, West Bengal, India. It was supplied from Sumita Hydro Colloid Limited, Jodhpur, India.

2.4 Guar-Ba^{2+} sample
50 ml approximately 1wt% guar sample is mixed with 5 ml barium nitrate [$Ba(NO_3)_2$] solution and stirred. Mixture becomes white. 10 ml 60% sodium hydroxide (NaOH) solution added and stirred. Mixture becomes light yellow. Mixture is allowed to stand overnight. 15 ml methyl alcohol (MeOH) added and allowed to stand for 10 mins. A tight mass separates which is collected by glass rod and kept in 5 ml methyl alcohol. After sometime, methyl alcohol is removed by decantation and washed the mass with 3 ml methyl alcohol for 3 times.
Then it is washed with 10 ml distilled water for five times. Then the mass is dried in an oven and collected for thermal analysis.

2.5 DTA and TGA
For simultaneous DTA and TG analysis, Pyris Diamond TG/DTA instrument has been used. Name of the company is Perkin Elmer. Simultaneous DTA and TGA have been carried out in presence of air upto 600 ^{0}C using samples as shown in Table-1.

Table 1: Sample size for simultaneous DTA and TGA

Sample	Approximate amount
Industrial guar sample	5.39 milligrams
Guar-Ba^{2+} sample	4.09 milligrams
Barium hydroxide	4.6 milligrams

Alumina crucible used for DTA and TGA. Heating rate used: 10 ^{0}C/min.

2.6 DSC
Differential scanning calorimetric analysis has been carried out using PYRIS Diamond DSC instrument. Name of the company is Perkin Elmer. DSC analysis has been carried out using sample size as shown in Table-2.

Table 2: Sample size for DSC analysis

Sample	Approximate amount
Industrial guar sample	3.64 milligrams
Guar-Ba^{2+} sample	1.52 milligrams
Barium hydroxide	3.79 milligrams

For DSC, sample is kept in aluminium crucible and a lid is fitted tightly applying pressure. Atmosphere is nitrogen atmosphere and experiment has been carried out upto 600 ^{0}C. Heating rate used: 10 ^{0}C/min.

3. Results and Discussion
3.1 DTA and TGA
DTA, TGA and DTG plots for industrial guar sample, guar –Ba^{2+} sample and for barium hydroxide are shown in Fig. 2-4 respectively. DTA plots for all the three cases are different. These indicate guar –Ba^{2+} sample is not barium hydroxide. It contains polymeric part. From TG plots, it is obvious that industrial guar sample and guar –Ba^{2+} sample have different thermal stability. % Residue values at 600 ^{0}C for these three cases are shown in Table-3.

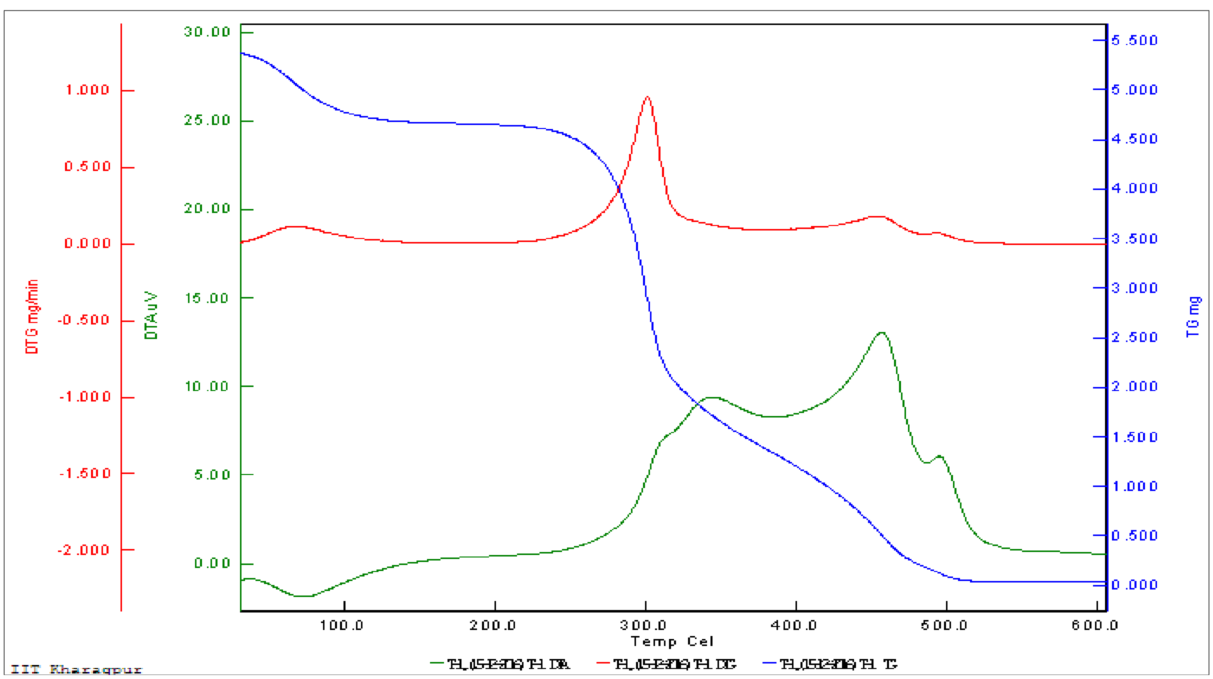

Fig 2: DTA, TGA and DTG plots for industrial guar sample.

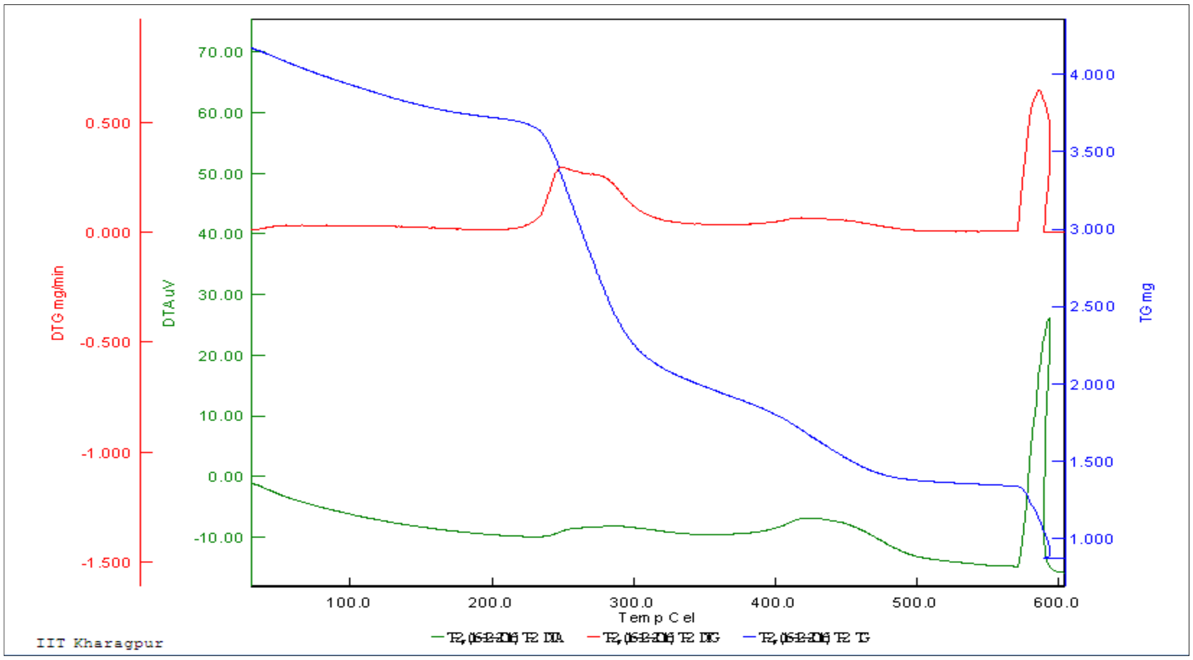

Fig 3: DTA, TGA and DTG plots for guar-Ba^{2+} sample.

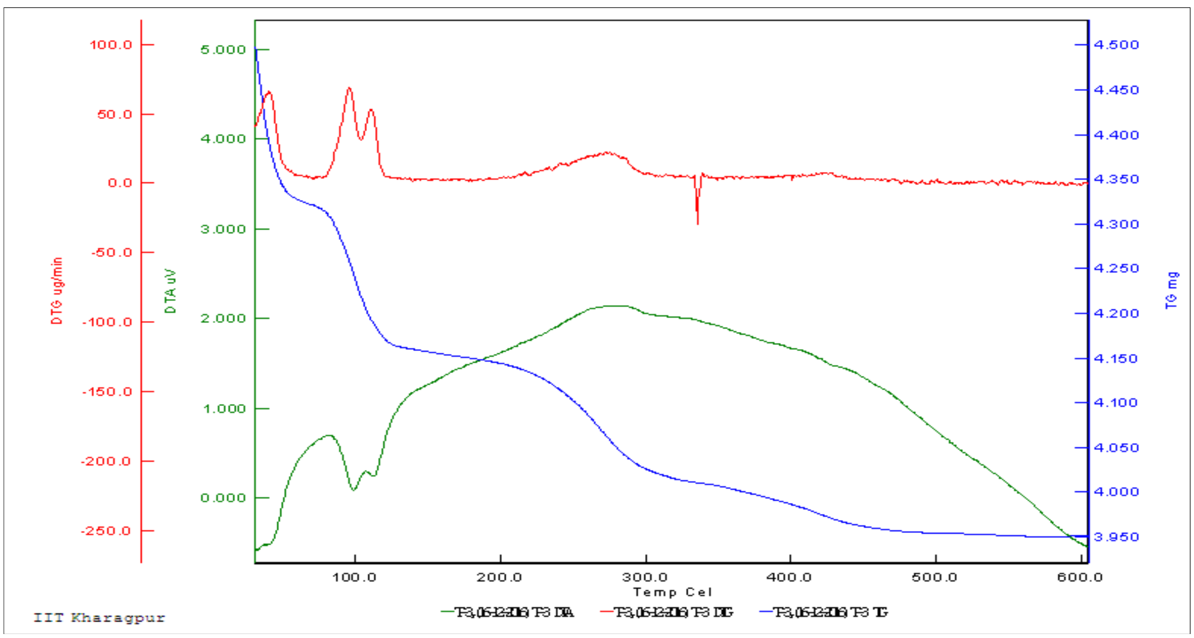

Fig 4: DTA, TGA and DTG plots for barium hydroxide.

Table 3: % Residue values at 600 ^{0}C

Sample	% residue value at 600 ^{0}C
Industrial guar sample	20.8
Guar-Ba^{2+} sample	0.7
Barium hydroxide	87.5

In DTA plot for guar –Ba^{2+} sample, near 600 ^{0}C there is a sharper peak (also in DTG plot) which is absent in DTA plots for industrial guar sample and for barium hydroxide. This is indicating slight exploding nature of guar –Ba^{2+} sample near 600 ^{0}C may be due to oxidative thermal degradation. % Residue value at 600 ^{0}C for guar –Ba^{2+} sample is also very very small but that for barium hydroxide is much higher and that for industrial guar sample is intermediate. This is supporting the difference in composition among industrial guar sample, guar –Ba^{2+} sample and barium hydroxide.

3.2 DSC

DSC plots for industrial guar sample, guar –Ba^{2+} sample and for barium hydroxide are shown in a single figure (Fig.5) for ease of comparison. In DSC plot for industrial guar sample at temperature near about 150 ^{0}C, there is rise in heat flow. This may be attributed to pyrolysis in absence of oxygen to form H_2O. Guargum contains –OH groups (Fig.1). Similar peak is present at comparatively lower temperature in the DSC plot for guar –Ba^{2+} sample. This is probably due to breaking of a complex involving Ba^{2+} in a closed system in absence of air in case of guar –Ba^{2+} sample. In case of barium hydroxide [$Ba(OH)_2$], within 100 ^{0}C to 150 ^{0}C, peaks are present in DSC thermogram which may be attributed to degradation in absence of air.

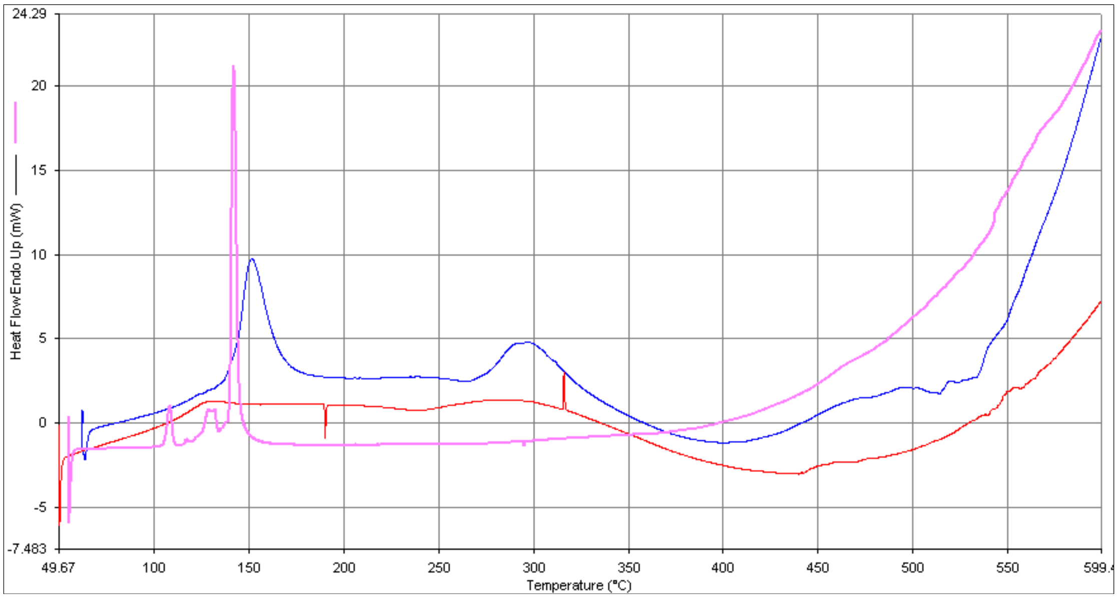

Fig 5: DSC plots for industrial guar sample (blue curve), guar-Ba^{2+} sample (red curve) and barium hydroxide (violet curve).

4. Conclusion

$Y_1Ba_2Cu_3O_{7-x}$ (ceramic oxide) superconductor can be prepared by polymeric precursor technique [6, 8]. So ion binding by polymer is an important study now-a-days. This work indicated guar sample can bind metal ion. It has been already found that guargum-*graft*-acrylamide (G-g-Am) can bind Y^{3+}, Ba^{2+} and Cu^{2+} at different pH ranges [7, 9, 11]. This work and earlier works [4] indicate guar sample itself can bind metal ion. So backbone in G-g-Am may also have role in binding Ba^{2+} by G-g-Am.

5. Acknowledgement

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