Thermal analysis of tri-ethylene glycol di-methacrylate

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Thermal characterization of triethylene glycol di-methacrylate was carried out in view of its vast applications. The main objective of this paper is to investigate the thermal behaviour of the impurities present in the compound tri-ethylene glycol di-methacrylate and its effects during applications of the compound. Thermal characterisation was carried out under two different experimental conditions. One at low heat flux rate of 10°C/min, and another at higher heat flux rate of 100°C/min. At low heat flux rate, the ethers present as impurities in the compound tri-ethylene glycol di-methacrylate are stable below the temperature of 150°C. This is desirable for applications in anaerobic adhesive and optical articles where exposure to temperature beyond 150°C is not required. At higher heating rate of 100°C/min, the endothermic decomposition of the impurities as ethers present in the compound tri-ethylene glycol di-methacrylate is observed, and the same is desired from the application point of view of using tri-ethylene glycol di-methacrylate as a moderant in single base propellants.

The tri-ethylene glycol di-methacrylate is used as moderant in propellant industry1. In addition to this the above compound has vast industrial applications2–9. Methacrylate esters of glycol finds applications in, anaerobic adhesive having good bond strength, formulation of adhesives for Acryonitrile Butadiene Styrene (ABS) plastics, manufacture of polymers with high refractive index, optical articles, manufacture of plastic lenses, and formulation of composition used for dental application. During synthesis of tri-ethylene glycol di-methacrylate, due to side reactions ethers are produced to small extent and it is rather difficult to remove ethers from the product. The purity of the compound tri-ethylene glycol di-methacrylate during its synthesis depends on the catalyst used and the concentration of catalyst.

The commercially available compound is 90 to 95% pure and contains about 5 to 10% of ether as impurities. The present study was undertaken to investigate the thermal behaviour of the compound tri-ethylene glycol di-methacrylate and the impurities present in this compound.

Results and Discussion

The thermal characterization of tri-ethylene glycol di-methacrylate sample having purity of 94.1% was carried out under the following experimental conditions:

- Purge gas: Nitrogen
- Gas flow rate: 100 mL/min
- Rate of heating: 10°C/min
- Temperature range: 30°C to 900°C

The thermogram of this experiment is shown in Fig. 1.

Under the low heat flux rate condition, it can be seen from Thermogravimetry (TG) curve, that a weight loss of about 5.9% is observed at 181.5°C. This is attributed to loss of ethers present in tri-ethylene glycol di-methacrylate and the impurities present in this compound.

Fig. 1—DTA and TG curves for tri-ethylene glycol dimethacrylate under low heat rate
Applications such as anaerobic adhesives, optical articles, etc. do not require exposure to temperature beyond 150°C. The study at controlled heating rate of 10°C/min confirms this aspect. A process of a large weight loss starts at about 272.2°C and there is almost total weight loss (~97%) at 464.6°C. This is attributed to decomposition of tri-ethylene glycol dimethacrylate.

In differential thermal analysis (DTA) two peaks are observed. One exothermic peak at 169.3°C which is due to oxidation of ether as the purge gas, contains about 8% oxygen. The second peak at 301.4°C is an endothermic peak due to decomposition of tri-ethylene glycol di-methacrylate. A second experiment was carried out under the following experimental conditions.

Purge gas: Nitrogen
Gas flow rate: 100 mL/min
Rate of heating: 100°C/min
Temperature range: 30-530°C

The thermogram of this experiment is given in Fig. 2. Under the high heat flux rate condition, the thermogravimetry curve (TG curve) shows just one step loss of nearly 100%. This is due to the fact that the ethers undergo endothermic decomposition and decomposition of tri-ethylene glycol di-methacrylate starts immediately. The important observation of this experiment is the DTA curve which shows two broad endothermic peaks. The difference in DTA of two experiments is due to the fact that in second experiment, the process is so fast that only the endothermic reactions, that is decomposition of ethers and decomposition of tri-ethylene glycol di-methacrylate can take place due to shorter contact time and availability of smaller amount of oxygen.

In Fig. 3, the DTA curve of the first experiment is superimposed on the DTA curve of the second experiment.

From the study it can be concluded that the purity of the compound tri-ethylene glycol di-methacrylate as specified in Fluka Grade Chemical is around 90 to 95% approximately and remaining impurities 5 to 10% are ethers formed as by-products during synthesis of tri-ethylene glycol di-methacrylate. The purity of the investigated sample with regard to contents of tri-ethylene glycol di-methacrylate is around 94.1% and the impurities present to the extent of 5.9% are ethers. From the study at controlled heating rate of 10°C/min it is concluded that the compound tri-ethylene glycol di-methacrylate and the impurities present in the compound are stable below temperature of 150°C which is desired for its application in anaerobic adhesives and optical articles, where exposure to temperature beyond 150°C is not required. At higher heat flux rate of 100°C/min, the impurities present in the sample in DTA analysis has shown endothermic peak which is favourable from application point of view of this compound as a moderant in propellant industry. As the burning of single base propellant in the gun is milli-second phenomenon therefore at such high rate of heat flux, the impurities as ethers present in the compound tri-ethylene glycol di-methacrylate will certainly undergo endothermic decomposition which is favourable from the point of view of the application of the compound tri-ethylene glycol di-ethacrylate as moderant in propellant industry.
References
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