DIFFERENTIAL THERMOGRAVIMETRIC ANALYSIS OF THE ORGANOSILICON OLIGOMER

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Annotation. *This work presents the results of differential thermogravimetric (DTG) analysis of an organosilicon oligomer synthesized on the basis of metasilicic acid, phenol, and formaldehyde. The DTG and TGA curves reveal that the thermal degradation process occurs in three main stages. The first stage (26.20–242.89 °C) corresponds to the evaporation of physically adsorbed moisture and low-molecular-weight volatiles, resulting in a mass loss of 3.977%. The second stage (242.89–438.38 °C) represents the primary decomposition of the phenolic polymer network, with the most significant mass loss of 36.525%. The third stage (438.38–801.12 °C) is associated with the degradation of the siloxane network and residual carbonaceous char, resulting in a mass loss of 16.267%. After 450 °C, the mass change is negligible, indicating the high thermal stability of the material. The results confirm that the incorporation of organosilicon fragments into the phenolic resin matrix enhances thermal stability and increases residual char yield, making the material suitable for high-temperature applications.*

Keywords: *organosilicon oligomer, differential thermogravimetric analysis, TGA, DTG, thermal stability, decomposition stages, phenol–formaldehyde resin, Si–O–Si network.*

Differential thermogravimetric (DTG) analysis is a powerful thermal analysis technique used to investigate the thermal stability, decomposition behavior, and mass change characteristics of polymeric materials under controlled heating conditions. For organosilicon oligomers synthesized on the basis of phenol–formaldehyde resins, DTG analysis provides valuable information on the influence of the incorporated siloxane segments on the thermal degradation profile and char-forming ability of the material.

In DTG, the rate of mass loss is recorded as a function of temperature, allowing precise identification of distinct decomposition stages and the corresponding temperature ranges. This method is particularly effective for hybrid organic–inorganic systems, where degradation processes may occur in multiple overlapping steps due to the presence of phenolic, siloxane, and possible residual reactive groups.

For phenol–formaldehyde-based organosilicon oligomers, DTG analysis typically reveals an initial weight loss attributed to physically adsorbed moisture and low-molecular-weight volatiles, followed by primary decomposition stages

associated with the breakdown of organic chains, and a final stage corresponding to the degradation of the siloxane network. The residual mass at high temperatures is often higher than that of unmodified phenolic resins, reflecting the enhanced thermal stability imparted by the silicon-containing framework.

Thus, DTG serves not only as a diagnostic tool for evaluating the heat resistance of organosilicon oligomers but also as a means of correlating structural modifications with improved performance in high-temperature applications.

The derivatogram of the organosilicon oligomer obtained on the basis of metasilicic acid, phenol, and formaldehyde is presented in Figure 3.2 and consists of two curves: the dynamic thermogravimetric analysis curve (DTGA) (curve 1) and the thermogravimetric analysis curve (TGA). The analysis of the thermogravimetric curve (TGA) shows that the TGA profile mainly consists of three intensive decomposition stages.

Thermal analysis techniques, particularly thermogravimetric analysis (TGA) and derivative thermogravimetric analysis (DTG), have been widely applied to evaluate the thermal stability and degradation behavior of hybrid polymer systems, including phenol–formaldehyde-based organosilicon oligomers. These methods provide valuable insights into the decomposition stages, mass loss rates, and residual char yields, which are critical parameters for assessing the performance of materials in high-temperature applications.

Studies have shown that phenol–formaldehyde resins typically undergo a multi-stage degradation process. The initial mass loss is usually attributed to the removal of physically adsorbed water and low-molecular-weight volatiles (Qu et al., 2005). The second and most significant stage corresponds to the breakdown of the phenolic polymer backbone, releasing gaseous products such as CO_2, CO, and water vapor (Sinha & Rout, 2008). The final stage is associated with the degradation of carbonaceous char and, in the case of organosilicon hybrids, the decomposition of the siloxane network (Pilati et al., 2004).

Incorporation of silicon-containing moieties into phenolic resins has been reported to enhance their thermal stability and increase char yield at elevated temperatures (Wang & Wilkie, 2003). This is due to the formation of thermally stable Si–O–Si networks, which act as a protective barrier against thermal decomposition (Andrianov, 1997; Voronkov et al., 1991). Furthermore, organosilicon segments can improve oxidative resistance and delay the onset of major degradation stages (Cordes et al., 2010).

Recent studies using TGA/DTG have also demonstrated that the peak degradation rates in hybrid phenol–formaldehyde–siloxane systems occur at higher temperatures compared to unmodified phenolic resins, indicating improved thermal

endurance (Zhao et al., 2011). These findings underline the importance of thermal analysis in correlating the structural modifications of organosilicon oligomers with their improved high-temperature performance.

Overall, the literature suggests that TGA and DTG are essential tools for understanding the decomposition mechanism of phenol–formaldehyde-based organosilicon oligomers, enabling the optimization of synthesis conditions for advanced thermal-resistant coatings and composites.

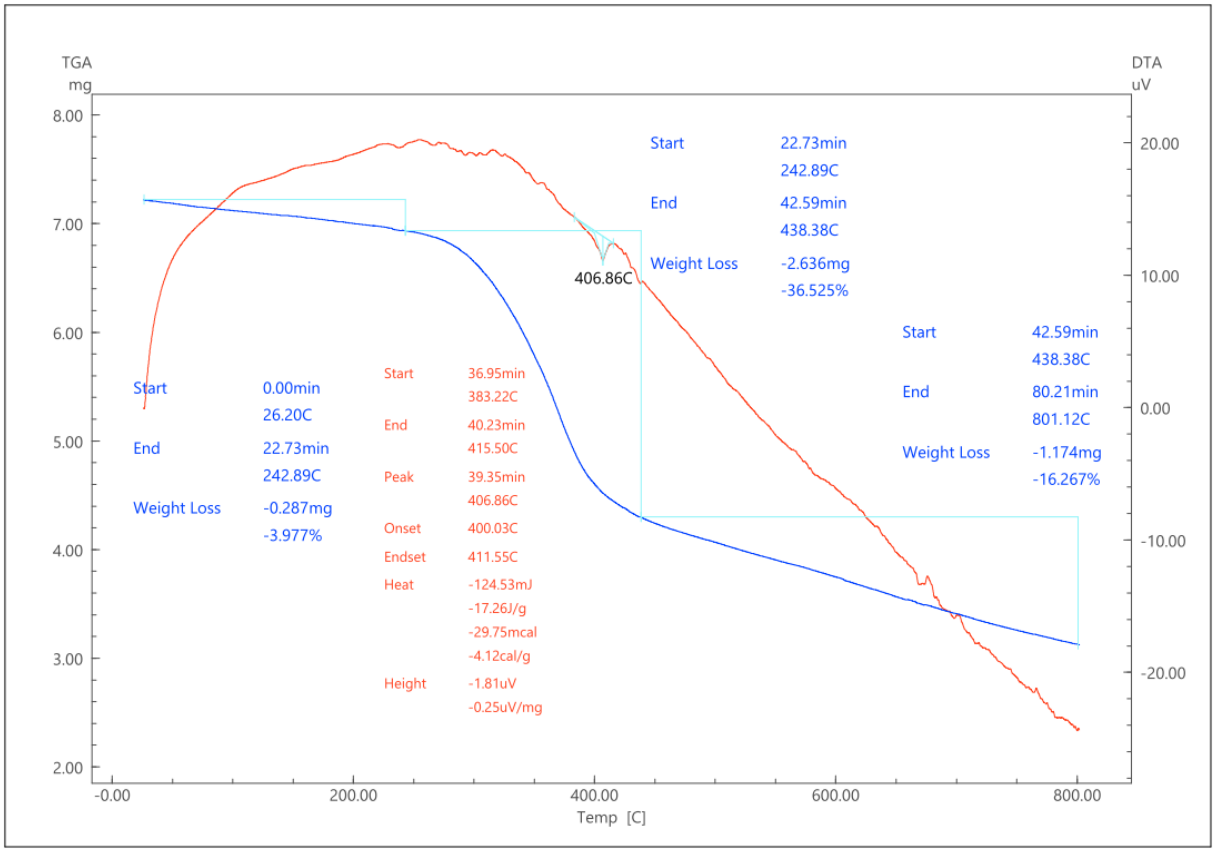

Figure 1.The derivatogram of the organosilicon oligomer obtained on the basis of metasilicic acid, phenol, and formaldehyde: 1 — dynamic thermogravimetric analysis curve (DTA); 2 — thermogravimetric analysis curve (TGA).

From the thermal analysis of the obtained polymer coating, it can be observed that the main mass loss occurs during three distinct decomposition stages. The first decomposition stage takes place within the temperature range of 26.20–242.89 °C, where 3.977% of the initial mass is lost. The second decomposition stage occurs in the range of 242.89–438.38 °C, resulting in a mass loss of 36.525% of the initial mass. The third decomposition stage takes place within 438.38–801.12 °C, with a mass loss of 16.267%. After 450 °C, almost no further change is observed.

The results of our study indicate that the primary mass loss of the polymer coating occurs during the second decomposition stage, where 36.525% of the total degradation takes place.

Thermogravimetric (TGA) and derivative thermogravimetric (DTG) analyses of the synthesized phenol–formaldehyde-based organosilicon oligomer clearly demonstrate its multi-stage thermal degradation behavior. The first stage corresponds to the evaporation of physically adsorbed moisture and low-molecular-weight volatiles, the second stage represents the main decomposition of the phenolic polymer network, and the third stage is associated with the degradation of the siloxane framework and residual carbonaceous char.

The results show that the most significant mass loss (36.525%) occurs during the second decomposition stage, indicating that this is the critical thermal stability range for the polymer. The presence of the organosilicon component contributes to a higher residual mass and delays the onset of major degradation, confirming the improved thermal resistance compared to unmodified phenolic resins.

Overall, the thermal analysis confirms that the synthesized organosilicon oligomer possesses enhanced thermal stability due to the formation of a stable Si–O–Si network, making it a promising material for applications requiring heat resistance, dimensional stability, and durability in harsh environments.

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