SYNTHESIS, IR-SPECTRAL AND THERMAL ANALYSIS OF A THERMOSTABLE SUBSTANCE BASED ON LIQUID GLASS AND SILICA SOL

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Abstract. *This paper summarizes the synthesis route, IR-spectral analysis, and thermogravimetric (TGA) and differential thermal (DTA) analysis of a thermostable substance synthesized from acrylic acid, ethylene glycol, colloidal silica sol, and liquid glass. The studies were carried out using modern physicochemical methods and methods consistent with national (GOST) and international (ASTM) standards.*

Keywords: *IR spectroscopy, thermogravimetric analysis, differential thermal analysis, liquid glass, silica sol, polymerization, thermal stability.*

INTRODUCTION

The demand for effective inorganic and organic flame retardants and intumescent fire-resistant polymer composite coatings is steadily growing in the chemical industry, construction, mechanical engineering, oil and gas, and other sectors. Ensuring the fire safety of buildings and structures and creating a new generation of protective agents that increase the fire resistance of combustible materials remain pressing tasks. The development of cost-effective and environmentally friendly technologies for producing thermostable compositions from local raw materials is of particular importance[1].

One promising approach to reducing the flammability and increasing the stability of polymer composite materials is the use of silicic-acid-based substances, in particular silica sol and liquid glass. SiO_2 nanoparticles interact with the polymer matrix to form a heat-resistant siloxane network[2]. Accordingly, the aim of this research was to synthesize a thermostable substance based on acrylic acid, ethylene glycol, and colloidal silica sol, to confirm its structure by IR spectroscopy, and to evaluate its thermal stability by TGA-DTA.

RESEARCH OBJECT AND METHODS

The research objects were the substance formed by modifying liquid glass and silica sol, as well as ethylene glycol samples. The synthesis was carried out in a laboratory reactor: acrylic acid and ethylene glycol were stirred at 80–90°C for 2–3 hours to obtain a complex polyester; after esterification, the mixture was cooled to 60–70°C, a polymerization initiator (potassium peroxodisulfate) and pre-mixed colloidal silica sol were added, and the mixture was heated at 70–80°C for 3–4 hours. During this process, the polyacrylate chains were crosslinked with the silica nanoparticles. The resulting nanocomposite was washed with methanol, filtered, and dried in a vacuum dryer at 50°C for 24 hours. IR-spectral analysis was performed on an IRAffinity-1S (SHIMADZU) FTIR spectrometer, and thermal analysis on a Shimadzu DTG-60 instrument in the range of 25–600°C.

RESEARCH RESULTS

The IR spectrum of the synthesized substance showed absorption bands corresponding to all theoretically expected functional groups (Figure 1, Table 1). The broad band at 3356 cm^{-1} corresponds to bound OH groups, the band at 2953 cm^{-1} to the CH_3 group, and the intense band at 1713 cm^{-1} to stretching vibrations of the carbonyl group in the –CO–CH_2–CH_2–CO– fragment[3].

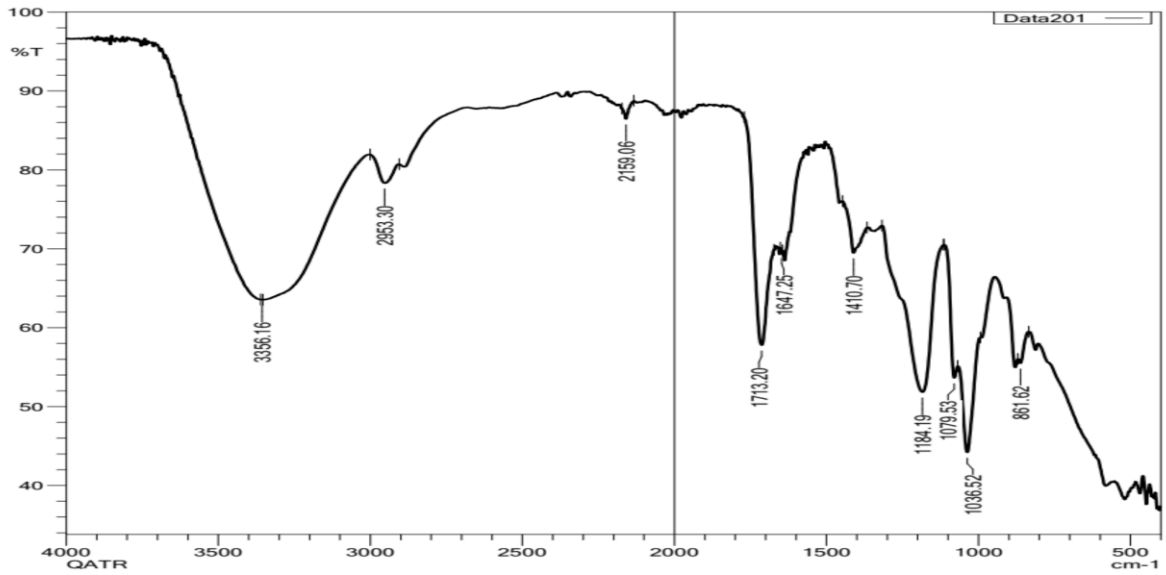


Figure 1. IR spectrum of the synthesized substance (IRAffinity-1S, SHIMADZU).

Absorption band, cm^{-1}	Functional group	Vibration type	Structural fragment
3356	O–H (bound)	stretching	hydroxyl, silanol groups
2953	CH_3	stretching	organic chain
1713	–CO–CH_2–CH_2–CO–	C=O stretching	polyester (acrylate–ethylene glycol)
1647	RR’C=CH_2	C=C stretching	acrylate fragment
1410.7	C–H	bending	organic chain
1036.52	Si–O–Si	stretching	siloxane network

Table 1. Main absorption bands in the IR spectrum of the synthesized substance (IRAffinity-1S).

The band at 1647 cm^{-1} is attributed to the RR’C=CH_2 group, and the band at 1410.7 cm^{-1} to C–H bending vibrations. In particular, the intense Si–O–Si band at 1036.52 cm^{-1} confirms that the polyacrylate chains are chemically bonded to the silica sol particles and that a three-dimensional siloxane network has formed in the substance. This network is the main structural element responsible for the thermal stability of the substance.

The thermal stability of the substance was studied by TGA-DTA (Table 2). According to the TGA curve, the sample loses significant mass in the range of 25–200°C, with the main part of the loss occurring around 90°C, which is attributed to the evaporation of bound water (moisture). In the range of 200–400°C the mass remains relatively stable, indicating that the sample reaches a stable state after dehydration.

In the range of 400–500°C (around 431°C), an additional mass loss is observed, associated with the thermal decomposition of organic components. The DTA curve shows one endothermic and two exothermic effects[4].

Temperature, °C	DTA effect	Process
90.63	strong endothermic	evaporation of bound water
217.17	sharp exothermic	polymerization, crystallization or oxidation
431.17	small exothermic	thermal decomposition step
450–600	minor peaks	later stages of oxidation

Table 2. Main thermal effects recorded in the TGA-DTA analysis of the synthesized substance (DTG-60, 25–600°C).

As Table 2 shows, the strong endothermic effect at 90.63°C and the corresponding mass loss are associated with the release of bound water, while the sharp exothermic peak at 217.17°C is related to the thermal transformation of organic or crystalline phases in the sample (polymerization, crystallization, or oxidation). The small exothermic effect at 431.17°C corresponds to a high-temperature thermal decomposition step. Comparison of the results with literature data showed that the properties of the synthesized substance are close to those of flame retardants exhibiting fire resistance[5].

CONCLUSION

A thermostable substance was synthesized from acrylic acid, ethylene glycol, colloidal silica sol, and liquid glass in the presence of a potassium peroxodisulfate initiator. IR-spectral analysis confirmed the presence of OH (3356 cm^{-1}), CH_3 (2953 cm^{-1}), C=O (1713 cm^{-1}), RR’C=CH_2 (1647 cm^{-1}), C–H (1410.7 cm^{-1}), and Si–O–Si (1036.52 cm^{-1}) groups, i.e., the formation of polyacrylate chains and a siloxane network. TGA-DTA analysis in the range of 25–600°C revealed the release of bound water (90.63°C), a phase transformation (217.17°C), and high-temperature decomposition (431.17°C), while the substance remains thermally stable between 200 and 400°C. The synthesized substance exhibits properties characteristic of flame retardants and can be used to create fire- and heat-resistant coatings for wooden and inorganic building structures, as well as a raw material for various branches of the chemical industry.

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