Figure 1. SEM-EDX spectrum (**A**) and SEM image (**B**); XRD patterns (I–illite; Q–quartz; K–kaolinite; H–halite) (**C**); FTIR spectrum; (**D**) curves of the studied raw diatomite samples.

The results of thermogravimetric analysis (TGA/DSC) (Figure 2) show a profound weight loss (9%) when natural diatomite was heated to 1000 °C. Other mass losses (5.1%) were detected within the range of 30–300 °C (DTG peak at 160 °C) and are due to loss of physically bound water (dehydration) from the diatomite surface [48]. The mass loss of 2.5% in the 290–500 temperature range (DTG peak at 350 °C) is due to the release of bound silanol groups (dehydroxylation) from the diatomite structure [49]. The obtained TGA/DSC results indicate that the observed peaks in the diatomite derivatogram correspond mainly to losses of water molecules of different chemical nature, starting from those adsorbed on the surface and those included in the internal structure as binding agents in the form of hydroxyl groups.



Figure 2. TGA curves of the studied raw diatomite sample.

3.2. Characterization of the Synthesized (AgCl,Ag)NPs/Diatomite Composite

3.2.1. Energy-Dispersive X-ray Spectroscopy (EDX) Studies

The results of SEM-EDX spectral analysis of natural diatomite and synthesized (AgCl, Ag)NPs/diatomite composite are shown in Figure 3A and Table 1. The results show a marked quantitative decrease in diatom of exchange cations such as Na, Mg, Al, K, Ca and Fe. The respective cations react with the anion, followed by the formation of soluble nitrate salts. The silver content in the prepared hybrid nanocomposites was found to be 0.71, 4.65 and 7.21% when using silver nitrate solution with initial silver concentrations of 100, 500 and 1000 mg/L, respectively (Table 1).

Table 1. Elemental composition of natural diatomite and (AgCl, Ag)NPs/diatomite composite.

	С	0	Na	Mg	Al	Si	S	Cl	К	Ca	Ti	Fe	Ag
Natural diatomite	0.48	45.99	1.27	1.09	7.09	38.37	-	0.59	1.17	0.30	0.52	3.14	-
0.71% Ag/diatomite	0.80	50.31	0.72	0.87	5.64	35.76	0.43	0.23	1.18	0.30	0.40	2.65	0.71
4.65% Ag/diatomite	0.67	48.77	0.75	1.08	6.50	31.93	0.26	1.08	1.12	-	0.43	2.75	4.65
7.21%Ag/diatomite	0.66	49.37	0.73	0.94	5.75	30.05	0.22	1.21	1.04	-	0.49	2.34	7.21

The molar and mass correlations of silver and chlorine in the synthesized composites are presented in Table 2. These calculations show that the resulting silver-containing composites contain a mixture of AgCl-NPs and AgNPs and also indicate their ratios in the composites.