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The Flame Retardancy and Smoke Suppression Performance of Polyvinyl Chloride Composites with an Efficient Flame Retardant System

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Abstract: Polyvinyl chloride (PVC) is the most widely used general flame-retardant plastic worldwide; however, the large number of plasticizers added during processing significantly reduces its flame-retardant property. To prepare a new type of PVC material with highly efficient flame retardancy and smoke suppression, antimony trioxide (Sb_2O_3), talc powder, hydromagnesite, and zinc borate were added in different proportions to PVC to explore the flame-retardant properties, thermal weight, smoke density (Ds), and mechanical properties of the composite materials. Results showed that the limiting oxygen index value of each group was higher than 27% after adding talc powder, magnesite, and zinc borate to replace part of the Sb_2O_3. This value was within the refractory-grade level and indicated a good flame retardancy performance. The replacement effect was in line with the experimental expectation. The lowest Ds peak value was 656.4 when the flame retardants were added with 10 wt% Sb_2O_3, 50 wt% hydromagnesite, 20 wt% talc, and 20 wt% Zn_3BO_6. Compared with pure Sb_2O_3 as a flame retardant, the Ds peak value decreased by 46.7%. The thermogravimetric decomposition temperature of the composites in each group was generally higher than that of the group with pure Sb_2O_3 as a flame retardant, increasing by 45.3 °C. The thermal stability of the composites was improved, and the elongation at the break and tensile strength were 234.9% and 25.8 MPa, respectively, indicating good mechanical properties. The results showed that using compound flame retardants to replace most of the Sb_2O_3 is an effective technique for obtaining good flame retardancy and mechanical properties of PVC. This study, not only reduced the manufacturing cost of flame-retardant PVC, but also effectively reduced its smoke density and the time to reach the highest smoke density, which provided a research reference for the application and promotion of flame-retardant PVC.

Keywords: polyvinyl chloride; flame retardants; flame retardancy; mechanical properties

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1. Introduction

Polyvinyl chloride (PVC) is a white powder with an amorphous structure that is regarded as a high polymer. It exhibits good mechanical and flame-retardant properties and is widely used in construction, framing, and decoration [1,2]. PVC is a strong polar polymer, with a strong intermolecular force, and thus, the finished product lacks elasticity and flexibility. A plasticizer can weaken the intermolecular force of the PVC, reducing the softening temperature, increasing fluidity, and improving the processability and flexibility of PVC products [3]. Therefore, plasticizers, such as dioctyl phthalate (DOP) or trioctyl trimellitate (TOTM), should be added during processing to improve the tensile property of PVC [4]. However, the addition of a plasticizer will reduce the flame-retardant ability of PVC composite materials. At high temperatures or after a long period of burning, HCl and other harmful gases will be produced, and combustion will generate toxic black smoke and further automatic catalytic decomposition, causing a rapid decline in physical and chemical properties. Therefore, adding flame retardants to PVC composite materials is necessary to inhibit the production of smoke [5,6].

The flame retardancy and mechanical properties of PVC have been improved by adding Sb_2O_3 to raw materials. Sb_2O_3 is a highly efficient additive for flame retardants, and it exhibits a general effect when used independently. When it coexists with halogen-containing compounds, a synergistic effect is produced, considerably improving the flame-retardant effect [7]. However, Sb_2O_3 is highly toxic and seriously endangers human life and health. Hence, its use has been prohibited or restricted in many countries. Gao et al. used Sb_2O_3 and solid superacid to synergistically improve the flame retardancy and smoke suppression of PVC composites. The results showed that solid superacid and Sb_2O_3 could promote the decomposition, pyrolysis, and crosslinking of PVC to form a continuous carbon layer on the surface of the material, effectively preventing heat transfer and PVC combustion [8]. Previous studies have shown that adding a zinc ferrite flame retardant to PVC materials can effectively reduce the smoke amount of PVC. However, adding only zinc ferrite results in shortcomings, such as low flame-retardant efficiency and poor compatibility with the PVC matrix [9]. Li et al. studied the effects of chlorinated polyethylene and Sb_2O_3 on the flame retardancy and mechanical properties of the recycled polyvinyl chloride/acryl-butadiene-styrene blends. The results show that the mixture is further improved with the addition of Sb_2O_3 [7]. In the current experiment, to ensure a good flame-retardant effect, an environmentally protective flame-retardant agent was added to inhibit PVC combustion smoke and reduce toxic gas emissions. This agent is more conducive to inducing the flame-retardant and smoke suppression effects of double standards [10].

In the present study, hydromagnesite, talc, and zinc borate were used to replace a part of the Sb_2O_3 as flame retardants to conduct research on flame retarding and smoke suppression. Sb_2O_3 exhibits a good flame retardant effect, but it promotes the generation of toxic gases during combustion. The three environmentally friendly and green flame retardants that we used, namely, talc powder, hydromagnesite, and Zn_3BO_6, have many advantages, such as flame retardancy, smoke inhibition, carbon formation, and protective film formation. Experiments with different ratios of antimony trioxide, talc powder, hydromagnesite, and zinc borate were designed to determine the amount of flame retardant used in prior experiments. Therefore, a certain amount of Sb_2O_3 is retained in this experiment on the basis of adding the three flame retardants, while keeping the flame retardancy of the PVC composite material good in order to improve the smoke suppression effect [11]. To prepare a new type of PVC material with a high efficiency in flame retardancy and smoke suppression, Sb_2O_3, talc powder, hydromagnesite, and Zn_3BO_6 were added to PVC in different proportions to explore the flame-retardant properties, thermal weight, smoke density (Ds), and mechanical properties of the composites. Our research effectively reduced the amount of smoke generated during the combustion of the PVC composites. The flame-retardant properties of the PVC composites were improved after improving the flame-retardant system. This has reference value for the application and promotion of flame-retardant and cost-effective PVC.

2. Experiment

2.1. Experimental Drug

PVC(type S-70) was purchased from Formosa Plastics Corporation, Ningbo, China. Calcium zinc stabilizer was supplied by Foshan Xingzhibao New Material Co., Ltd., Foshan, China. Epoxidized soybean oil was from Yancheng Xinjinyuan Oil Technology Co., Ltd., Yancheng, China. Trioctyl trimellitate (TOTM), polyethylene wax, antimony trioxide (Sb_2O_3), talc powder, hydromagnesite, and zinc borate were provided by Sinopharm Chemical Reagent Co., Ltd., Shanghai, China.

2.2. Main Material

The composition of the experimental materials and formulations is as follows: 200 phr PVC, 70 phr TOTM, 15 phr Ca–Zn stabilizer, 10 phr soy oil, 0.4 phr wax, and 10 phr flame retardant. The detailed composition of the flame retardants is provided in Table 1 (detailed composition of flame retardants/phr).

Table 1. Detailed composition of flame retardants/phr.

No.	Sb_2O_3 /phr	Hydromagnesite /phr	Talc /phr	Zn_3BO_6 /phr
YN-0	10	0	0	0
YN-1	0	0	0	0
YN-2	0	5.0	2.5	2.5
YN-3	1.0	5.0	3.5	0.5
YN-4	1.0	5.0	3.0	1.0
YN-5	1.0	5.0	2.5	1.5
YN-6	1.0	5.0	2.0	2.0
YN-7	1.0	5.0	1.5	2.5
YN-8	1.0	5.0	1.0	3.0
YN-9	1.0	5.0	0.5	3.5

2.3. Experimental Method

Preparation of PVC Composites

Using a mixer, the raw materials were mixed and stirred evenly in accordance with the ratio given by the formula. Mixing was conducted under the following conditions: 180 °C temperature and 35 r/min rotor speed. After mixing for 5 min, the composite materials were taken out and transferred to a plate vulcanizing machine for hot-press forming. The processing temperature of the plate vulcanizing machine was 185 °C. The time involved was as follows: preheating 5 min + pre-pressure 2 min + pressure 5 min = 12 min. After taking out and cooling the specimens, a pneumatic slicer was used to cut out various test standard splines for later use.

2.4. Experimental Standard

In this study, the oxygen index was measured using a paramagnetic oxygen index tester (Motis Technology, Shanghai, China), and the total flow rate of oxygen and nitrogen was maintained between 10.55 L/min and 10.65 L/min. Spline size was $120 \times 7 \times 3\ mm^3$. Mechanical properties were tested using a universal testing machine (Shenzhen Sunstest Technology Co., Ltd., Guangzhou, China). The scale distance of the extensiometer was 25 mm, tensile rate was 50 mm/min, and each sample was repeated six times (in cavity). The size was $115 \times 6 \times 2.0\ mm^3$. The Ds of the PVC composite material was tested using a smoke density tester (Motis Technology, Shanghai, China). The test standard was implemented in accordance with ISO 5659, the measurement mode was set as 25 KW/m^2 (smoke mode), and size was $75 \times 75 \times 3\ mm^3$.

3. Results and Discussion

3.1. Flame Retardancy of PVC Composite Material

The ratio of Sb_2O_3 to magnesite was fixed, and the ratio of talc to zinc borate was changed by a gradient of 5%. Under the condition that the oxygen index of YN-0 was 31.9%, the limiting oxygen index (LOI) values of all the samples were analyzed and evaluated. As shown in Figure 1, compared with that of Groups 1 and 2 without flame retardant or Sb_2O_3, the oxygen index of Groups 3–9 was generally increased, and the value range was greater than 28%, indicating that the addition of Sb_2O_3 significantly improved the flame-retardant effect. The YN-9 group had the highest oxygen index (28.8%) of Groups 3–9, and zinc borate accounted for 35%. The zinc borate lost crystal water when it was higher than 300 °C, which could be attributed to endothermic cooling. During combustion, the zinc in the zinc borate could be partially converted into zinc oxide and other components into the gas phase, diluting combustible gas. The melting point of antimony trioxide is 656 °C, and the boiling point is 1425 °C. When antimony trioxide is used alone, the flame-retardant effect is not significant. Usually, antimony trioxide is used in combination with other flame retardants to achieve flame-retardant purposes due to its excellent synergistic effect. In the early stage of the combustion of the polymer, antimony trioxide forms a liquid protective film on the surface of the material to isolate air after melting. Antimony trioxide melts above 650 °C to absorb heat to reduce the temperature, making it difficult to fully ignite the material. At high temperatures above 1400 °C, antimony trioxide is vaporized and dispersed into the air, diluting the oxygen concentration, thereby acting as a flame retardant. Thus, the co-use of zinc borate and Sb_2O_3 could produce a synergistic effect, which could significantly improve the flame-retardant property of the material. When the content of zinc borate was higher, the flame-retardant property of the material was improved. After replacing part of the Sb_2O_3 with green flame retardants, the oxygen index of the material was greater than 28, which achieving flame retardant status. The replacement effect was in line with the expectation [12].

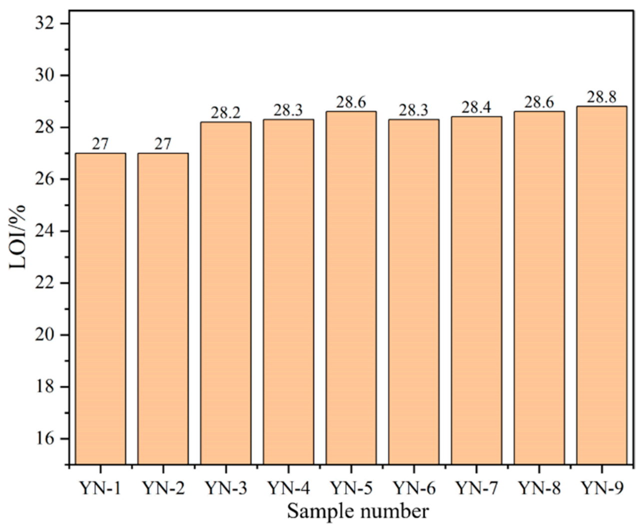

Figure 1. LOI histogram of different composites.

3.2. Smoke Density Test of PVC Composites

All the samples in the formula shown in Table 1 were used for follow-up experiments. The changes in transmittance and Ds of each group are shown in Figures 2 and 3. In the transmittance curve, Groups 1, 2, 3, 8, and 9 decreased rapidly, and the change curve nearly coincided. Groups 0, 4, 5, and 6 presented a slow decline, and the time for the transmittance to drop to the lowest was close to the longest group. In the Ds curve, the Ds value of the YN-0 group rose the fastest and had the highest peak value (about 1300). The Ds value of the group with a mass ratio of talc to zinc borate of 1:1 rose the slowest and the peak value was the lowest, i.e., approximately 650. The D peaks from high to low were 0, 1/2, 3, 4/7, and 8/9/5/6. At the end of the experiment, the D value was still the highest in the YN-0 group, i.e., about 1200. Group 6 was the lowest, at approximately 640. The results showed that the smoke suppression effect of the sixth group was the best, and the D value of the sixth group was decreased by 46.7% compared with that of the 100% Sb_2O_3 group, and the smoke suppression effect was evident after adding the new flame retardant [13]. The zinc borate exhibited a synergistic effect when combined with traditional flame retardants, which could significantly improve the flame retardancy and smoke suppression performance [14]. Therefore, after replacing most of the Sb_2O_3, the smoke suppression performance of the PVC composite was excellent. The smoke suppression effect was significantly stronger than that of 100% Sb_2O_3. When the proportion of talc and zinc borate was 20%, the smoke suppression performance of the PVC composite was the best.

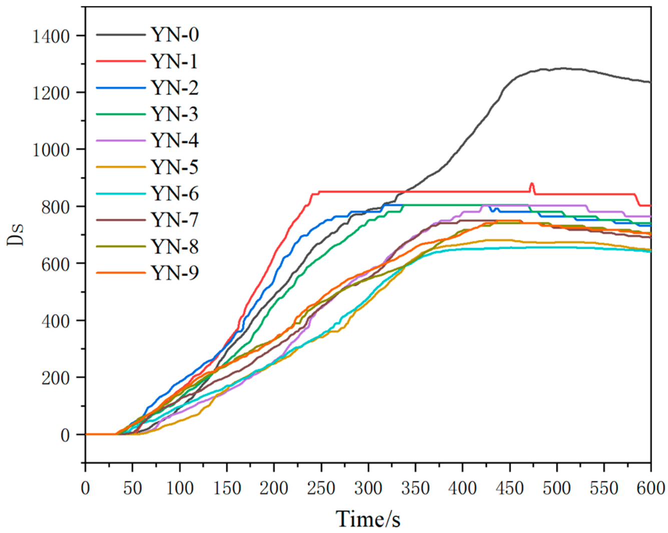

Figure 2. Changes in transmittance of different samples.

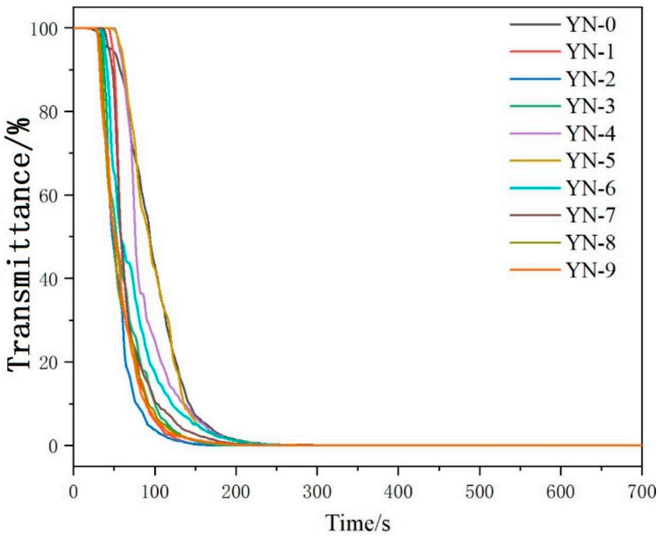

Figure 3. Smoke density of different samples.

3.3. Thermogravimetric Testing of PVC Composites

The results of the test of thermogravimetric changes of each group are shown in Figure 4. As shown in the figure, the materials in each group began to decompose when the temperature was around 250 °C, and the residual material decreased rapidly. Meanwhile, the decomposition rate slowed down gradually when the temperature was 300 °C. At about 450 °C, the decomposition rate of the material increased again and then gradually slowed down at 500 °C. The final material surplus became stable until the end of the test at 800 s. Before the second decomposition, the material reduction rate of each group was similar. When decomposition was gradually completed, the final material surplus of each group was significantly different, with the largest surplus of YN-9 and YN-2 being 19.34% and 18.92%, respectively. The smallest was that of YN-1, which was about 16.31%, and those of the other groups were about 18%. The thermogravimetric curves of different samples are highly coincident when the temperature is below 300 °C. Different thermal weight loss characteristics begin to appear when the temperature reaches above 300 °C, which is highly consistent with the thermal decomposition temperature of zinc borate. Zinc borate decomposes above 300 °C to form zinc oxide, boron trioxide, and water. Zinc oxide combines with chlorine atoms decomposed by PVC at a high temperature to form stable zinc chloride. With the decrease in the addition of talc powder, the residual residue of PVC hot air aging did not show obvious regular changes ranging from YN-3 to YN-9. In summary, it can be concluded that the difference in the residual residue of PVC has a strong positive correlation with the content of zinc borate in flame-retardant systems. As indicated in Table 2, the final residual amount of YN-0 was about 18%. Except for Groups 1 and 5, the residual amount of the other groups was generally higher than that of YN-0. After replacing Sb_2O_3, the initial thermal decomposition temperature of the PVC composite increased from 147.92 °C to 193.25 °C, and the thermal stability was improved to a certain extent [15].

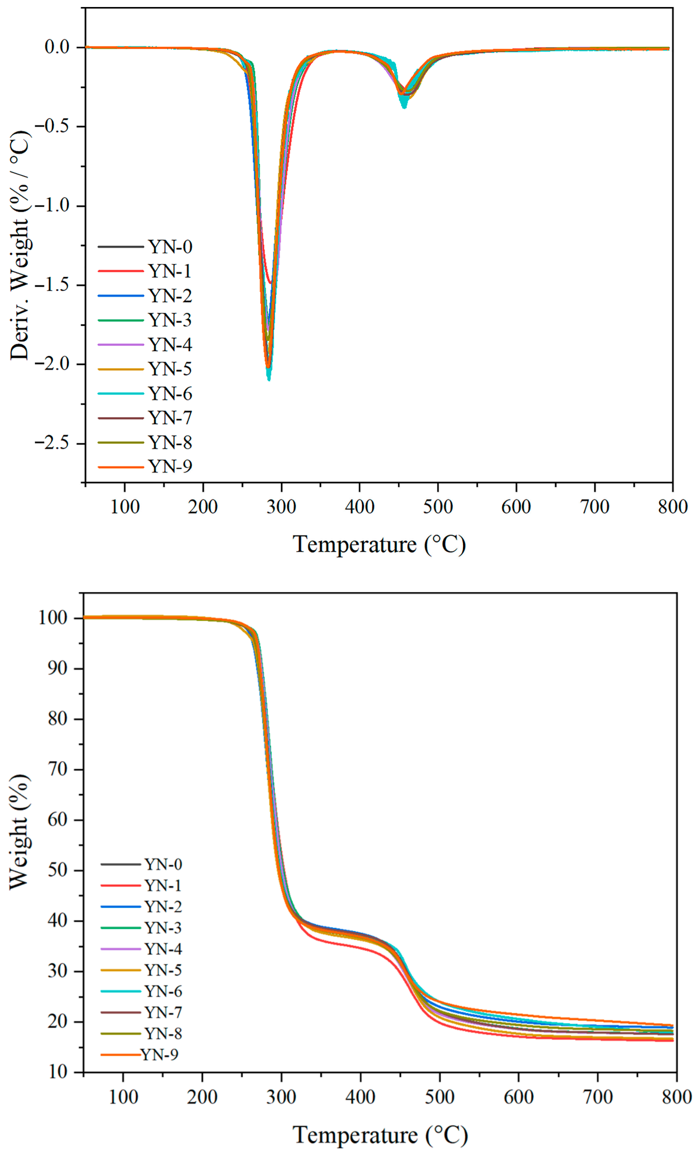

Figure 4. Thermogravimetric test of different samples.

Table 2. Thermogravimetric process thermal decomposition temperature, fastest decomposition temperature, and residual mass after thermal decomposition of different samples.

Thermal Decomposition	Temperature Range (°C)	T_{max} (°C)	800 °C Residual Mass (°C)
YN-0	147.92–611.65	282.74	15.60
YN-1	162.30–612.18	289.67	16.31
YN-2	164.99–628.16	281.10	18.92
YN-3	120.42–628.28	283.11	17.66
YN-4	153.33–624.87	284.68	17.64
YN-5	162.94–634.49	285.35	16.55
YN-6	178.45–643.19	285.65	17.87
YN-7	157.06–625.16	282.93	17.59
YN-8	152.19–633.21	282.63	18.29
YN-9	193.25–649.79	282.49	19.34

3.4. Mechanical Properties of PVC Composites

The mechanical properties observed in this experiment were mostly elongation at the break and tensile strength. The ratio of Sb_2O_3 to magnesite was still fixed, and the ratio of talc to zinc borate was changed by a gradient of 5%. The changes in tensile strength and elongation at the break of the groups (YN-1 toYN-9) are shown in Figure 5. When the talc content was reduced, the tensile strength and elongation at the break of the composites were improved, with Group 6 demonstrating the best improvement effect. The main reason is that the addition of too much talc powder increases the possibility of agglomeration of talc powder during processing, which leads to an increase in interface defects. The contact area between the flaky talc powder and PVC matrix is small, and the force between the two phases is weak. The interface is easy to de-bond during the force process, resulting in a decrease in mechanical properties [16,17]. Compared with YN-8, the tensile strength and elongation at the break of YN-9 suddenly increased because the zinc borate surface was covered with a layer of coupling agent. When the content of zinc borate was high, the interaction between the PVC and zinc borate particles was enhanced, and the tensile strength of the system was improved [18]. The experimental results showed that the tensile strength of the PVC composites was generally higher than 20 MPa, the elongation at the break was mostly higher than 200%, and the mechanical properties were the best when the mass ratio of talc powder to zinc borate was 1:1. As can be seen from Table 1, the YN-0 sample used Sb_2O_3 as a flame retardant. In Figure 5, compared with YN-0, hydromagnesite, talc, and zinc borate were used as flame retardants instead of Sb_2O_3 in YN-2. In YN-3 to YN-9, 90% of the Sb_2O_3 was replaced with hydromagnesite, talc, and zinc borate. Compared with YN-2 to YN-9, the tensile strength of YN-0 is lower, only 18.88 MPa, and the elongation at the break is higher, 258.29%. After a partial replacement of Sb_2O_3, the mechanical properties of the material remained good.

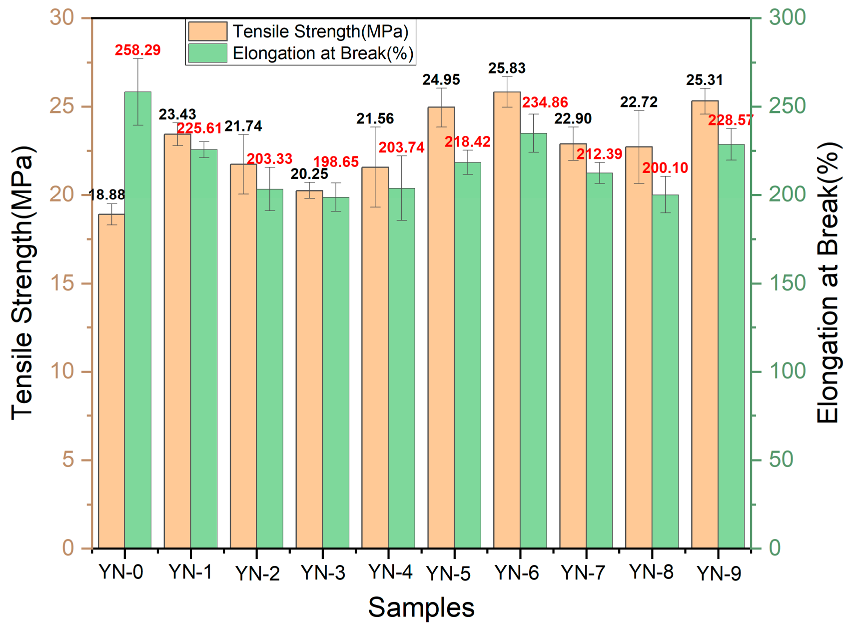

Figure 5. Histogram of tensile strength and elongation at break of different composites.

4. Conclusions

To prepare a new type of PVC material with highly efficient flame retardancy and smoke suppression, Sb_2O_3, talc powder, hydromagnesite, and zinc borate were added in different proportions to PVC to explore the flame-retardant properties, thermal weight, Ds, and mechanical properties of the composite materials. The results showed that the limiting oxygen index value of each group was higher than 27% after adding talc powder, magnesite, and zinc borate to replace part of the Sb_2O_3. Such a value is within the range of refractory-grade materials and indicated a good flame retardancy performance. The replacement effect was in line with the experimental expectation. The lowest D peak value was 656.4 when the flame retardants were added as 10 wt% Sb_2O_3, 50 wt% hydromagnesite, 20 wt% talc, and 20 wt% Sb_2O_3. Compared with pure Sb_2O_3 as a flame retardant, the D peak value decreased by 46.7%. The thermogravimetric decomposition temperature of the composites in each group was generally higher than that of the group with pure Sb_2O_3 as a flame retardant, increasing by 45.3 °C. The thermal stability of the composites was improved, and the elongation at the break and tensile strength were 234.9% and 25.8 MPa, respectively, indicating good mechanical properties. The results showed that using compound flame retardants to replace most of the Sb_2O_3 is an effective technique for obtaining good flame retardancy and mechanical properties of a PVC. This experiment mainly explores and improves the flame-retardant properties of PVC composites. The improved PVC composites can also be used in building plates, decoration, and so on.

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