# Flammability and selected properties of PVC composites with boehmite (*Rapid communication*)

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**Abstract:** This work investigated the effects of mineral nonorganic fillers, i.e., two types of boehmite (Catapal B and Pural SB) flame retardant on selected properties of soft poly(vinyl chloride) (PVC) composites. The flammability, Shore hardness, and tensile properties of PVC composites have been tested. It was found that the type and amount of flame-retardant additive significantly influences the properties of the composites.

**Keywords:** PVC composites, mechanical properties, boehmite, flammability.

## Palność i wybrane właściwości kompozytów PVC z dodatkiem boehmitu (*Komunikat szybkiego druku*)

**Streszczenie:** Zbadano wpływ mineralnych napełniaczy nieorganicznych, tj. dwóch rodzajów boehmitu (Catapal B i Pural SB) na wybrane właściwości miękkich kompozytów PVC. Zbadano palność, twardość w skali Shore'a oraz właściwości przy rozciąganiu otrzymanych materiałów. Na podstawie uzyskanych wyników stwierdzono, że rodzaj i ilość dodatku uniepalniającego znacząco wpływają na właściwości otrzymanych kompozytów.

**Słowa kluczowe:** kompozyty PCW, właściwości mechaniczne, bemity, test palności.

Science and technology advancements in the 21st century have quickly replaced traditional materials, made polymer composites the primary choice for many industrial applications, and expanded their use to every aspect of daily life [1, 2]. Moreover, they find widespread applications in cable insulation and coatings. The CPR directive's classification of electric cables based on their fire response classes necessitates the constant search for new compounds to enhance their performance characteristics and increase their fire resistance, thereby curbing the spread of flames [3].

A composite material consists of an organic polymeric backbone reinforced with one or more external materials, such as glass, carbon, or natural fibers. The external reinforcement may be in the form of particles, fibers, or filaments, and its presence provides strength and durability to the material. These materials possess distinctive physical, thermal, chemical, and mechanical characteristics, including low density, high specific stiffness and strength, corrosion resistance, high fatigue strength, and low thermal expansion [4]. Conversely, the organic nature and properties of the polymer matrix and fibers result in composite materials exhibiting limited fire resistance. The fire resistance of the composite can be improved by adding flame retardant additives. Still, it is important to remember that each application needs a different composition made up of the right polymer, hardener, or flame retardants [5, 6]. The combustion of polymeric materials may occur in several different phases. For instance, in the gaseous phase, the flame retardant can impede the exothermic reaction by reducing the generation or binding of radicals formed by high temperatures. Additionally, the flame retardant can form a thermal barrier (e.g., charring) on the surface of the condensed phase, impeding the transfer of heat back to the burning polymer and reducing the release of flammable gaseous substances, thereby reducing the intensity of the flame [7]. Until

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recently, halogenated agents were the dominant flame retardants in polymer composites. One such agent was tetrabromobisphenol A (TBBPA), which was among the most widely used flame retardants. It produced volatile bromine radicals under temperature conditions, effectively interrupting the combustion cycle [8]. However, the implementation of new environmental standards, including REACH, WEEE, and RoHS, has led to a restriction on the use of halogenated substances as flame retardants. Consequently, an increasing number of laboratories and companies have begun incorporating alternative flame retardants into their product lines to comply with the new regulations and offer products that are less harmful to human health and the environment.

Nowadays, there is a growing group of flame-retardant alternatives to halogenated compounds that are available for use in electrical and electronic applications. The most popular of these are inorganic flame retardants and nitrogen- or phosphorus-based flame retardants. Metal hydroxides, such as aluminum hydroxide (ATH) and magnesium hydroxide (MDH), offer several advantages such as flame retardants, including commercial availability, low cost, non-toxicity, and environmental benefits [9, 10]. However, the composite must contain a significant amount of these agents (up to 70%) to achieve flame retardancy, which can often negatively impact the final product's properties and restrict their use [11]. Among the available inorganic ones, the mineral boehmite ($\gamma$-AlO(OH)) is a promising material that can impart excellent fire resistance to polymeric materials [10-13]. It has many unique properties, including a high melting point, low thermal conductivity, a large surface area, excellent chemical stability, and low cost.

This work continues the study of the effect of the addition of halogen-free flame retardants on the strength properties and flammability of soft PVC composites. In previous work [14], we described the effects of ATH and triphenyl phosphate (TPP) additives on the flammability and strength of PVC composites, while in this work we focused on examining the effects of two boehmite varieties on these properties.

## EXPERIMENTAL PART

### Materials

Experimental studies were carried out on homopolymer poly(vinyl chloride) (PVC) emulsion, marketed under the trade name Vinnollit E 68 SA produced by Westlake Vinnolit GmbH & Co. KG (Ismaning, Germany) classified as a low to medium viscosity resin and recommended to produce chemically blown foams. Plasticizers and stabilizers used in this investigation are: Ergoplast ES - (ESBO), epoxidized soya bean oil, is a homogeneous oily mixture of epoxidized glycerides of C16-C18 acids, Ergoplast FDO (bis(2-ethylhexyl) phthalate) Ergoterm SZ (zinc stearate), Ergoterm RZ (calcium stearate), (Boryszew ERG, Poland). The high-purity aluminum oxide hydroxide (boehmite) Catapal B (CA) and Pural SB (PU) were made available for our research courtesy of the company Sasol Germany GmbH.

**T a b l e 1. Characterization of Catapal B and Pural SB mineral fillers [15].**

| Filler | Particle size mm | Surface area BET m$^2$/g | Pore volume mL/g |
|---|---|---|---|
| Catapal B | 60 | 250 | 0.5 |
| Pural SB | 45 | 250 | 0.5 |

### Preparation of composites

The base mixture of each composite was prepared by an analogous procedure. In a 100 mL glass vessel, 10 g of PVC, 0.6 g of epoxidized soybean oil, 6 g of Ergoplast FDO plasticizers, 0.07 g of Ergoterm SZ, and 0.07 g of Ergoterm RC stabilizers were added. Corresponding weights of Catapal B (CA) or Pural SB (PU) flame retardants were added to each of the ingredient mixtures and thus prepared and mixed until the ingredients were completely combined. To obtain the final cured composites, the blended mixtures were filled between two thin rectangular glass panels, protected at the side and bottom edges by a dedicated fitting to ensure an even surface. The composites were then placed in an oven and heated to 135°C for 20 minutes. Following the specified period, the samples were removed and left to cool. Once the composites had reached ambient temperature, the samples of composites were carefully removed from between the glass plates. To assess the composites, they were cut into longitudinal, rectangular strips measuring approximately 8×1×0.2 cm each (Fig. 1).

### Methods

#### Tensile strength

Tensile testing was performed on a Zwick/Roell Z010 tensile-testing machine (Ulm, Germany) according to ISO 527-2 at the speed of 100 mm/min; the tensile test pieces were cut from the prepared sheets.

#### Hardness

The hardness of the composites was measured by the Shore A method on a Zwick 7206/H04 hardness tester (Ulm, Germany) according to EN ISO 868. Readings were taken after 15 s.

#### Flammability tests

The flammability of composites can be determined through the horizontal burning method of the sample, which is classified as Method A UL94HB under the

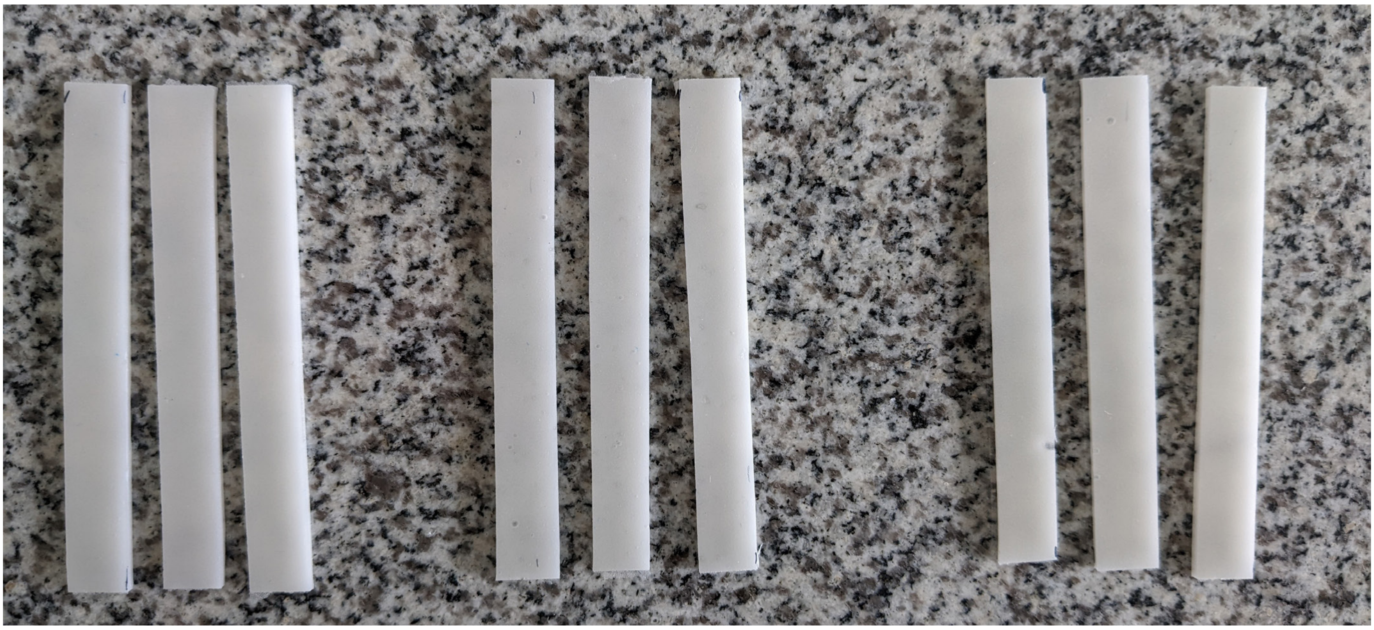

**Fig. 1. Appearance of selected PVC composite strips**

PN-EN 60695-11-10:2014-02. The test sample is positioned horizontally within a metal paw mounted on a tripod. A 50W burner is positioned at an angle of 45° and the flame is applied to the sample from the free end of the strip for 30 seconds. Subsequently, the time until the sample is freely extinguished is measured. Alternatively, the sample can be extinguished after 15 seconds of free burning and the length of the burnt sample, and the rate of combustion can be measured. The linear burning rate ($v$, mm/min) is calculated using Equation 1:

$$v = 60 \cdot L/t \tag{1}$$

where: $L$ – the length of the burnt sample (mm); $t$ – the burning time (s).

## RESULTS AND DISCUSSION

After removing PVC composites from the dryer, we observed differences between samples containing different amounts of each flame retardant after the heating stage. The overall trend, evident in all samples, was an increase in structural inhomogeneity as the flame-retardant content in the composition increased. The blank sample exhibited a uniform appearance without air bubbles in its structure.

It might be helpful to make a quick comparison of the flame retardants in the lab to see if they are good enough for more research into a mixture that will be used in the extrusion of cable insulation and coatings. To develop the formulation, subsequent stages will necessitate the execution of extrusion tests on the composition. These tests will subject the composites to temperature in addition to pressure and mechanical forces, which will enhance the degree of plasticization and homogeneity. Moreover, these tests will enable the determination of the parameters and properties of the product with greater accuracy.

### Hardness

The Shore hardness of the obtained compositions was measured in accordance with method A, and the results are presented in Table 2.

**T a b l e 2. Hardness of examined PVC/flame retardant composites**

| Flame retardant | Amount of flame retardant wt% | Hardness, Sh A | | | | | |
|---|---|---|---|---|---|---|---|
| | | 1 | 2 | 3 | 4 | 5 | Average |
| Reference | 0 | 69 | 68 | 68 | 69 | 69 | 68.6 |
| CA | 10 | 61 | 62 | 60 | 62 | 63 | 61.6 |
| | 20 | 68 | 70 | 68 | 69 | 70 | 69.0 |
| | 30 | 70 | 74 | 73 | 74 | 75 | 73.2 |
| | 40 | 79 | 80 | 77 | 77 | 76 | 77.8 |
| | 50 | 80 | 81 | 82 | 80 | 79 | 80.4 |
| PU | 10 | 66 | 63 | 65 | 65 | 66 | 65.0 |
| | 20 | 71 | 73 | 72 | 73 | 71 | 72.0 |
| | 30 | 77 | 76 | 78 | 76 | 76 | 76.6 |
| | 40 | 81 | 80 | 79 | 79 | 80 | 79.8 |
| | 50 | 83 | 85 | 83 | 81 | 84 | 83.2 |

The average hardness of the molded sample without flame retardant is 68.6 Sh on the A scale in both series of obtained composites. The hardness of the composites increased with increasing filler content. The differences in the hardness of analogous composites in both series can be explained by better dispersion of PU filler in the polymer matrix, which has a smaller particle size. Materials with 50 wt.% PU show the highest hardness compared to other composites.

### Tensile properties

The effect of boehmite addition on static tensile properties was also determined, and the results are included in Table 3.

**T a b l e 3. Tensile properties of PVC/flame retardant composites**

| Flame retardant | Flame retardant wt% | Elongation at break % | Tensile strength MPa |
|---|---|---|---|
| Reference | 0 | 100 | 1.58 |
| CA | 10 | 140 | 0.43 |
| | 20 | 180 | 0.88 |
| | 30 | 227 | 3.54 |
| | 40 | 217 | 3.57 |
| | 50 | 207 | 4.49 |
| PU | 10 | 345 | 6.09 |
| | 20 | 210 | 4.71 |
| | 30 | 27.5 | 2.10 |
| | 40 | 25.7 | 1.90 |
| | 50 | 19.7 | 1.90 |

An apparent increase in strength was only observed with CA. Analyzing the flame retardant's effect on elongation reveals that the amount of CA in the sample led to a proportional increase in elongation. In contrast, in the PU series, increasing the amount of flame retardant by more than 20 wt% resulted in a drastic decrease in strength, which is unexpected considering the similar physical and chemical properties of the flame retardants. Differences in changes in strength properties between analogous compositions in the two series may be due (as well as hardness) to the particle size of the boehmite used. It seems that the finer the particle size, the easier it is to distribute in the polymer matrix and, at the same time, the interactions between polymer and filler are stronger, making the strength higher. However, after a certain percentage of filling is reached, the strength may decrease, and this may be since the interactions between the polymer matrix and the filler are reduced, and the interactions between the filler particles are increased. In the case of the compositions tested, the finer the grains and the more grains there are, the interactions between the polymer matrix and the filler disappear, and the interactions between the filler particles become more important. In contrast, in the series with CA (filler with larger particles), the key role on the strength properties is determined by polymer-filler interactions. Other factors that have a significant impact on the properties, including tensile stress, of filled systems are boundary layers formed during the adsorption of polymer macromolecules on the filler surface. Therefore, the properties of filled composites change significantly compared to the properties of the original polymer because of molecular interaction and structure transformation at the polymer - filler interface.

### Flammability

The effect of boehmite addition on the susceptibility of the obtained compositions to burning has been analyzed. Table 4 shows data from the flammability tests, while Figures 2 and 3 present the appearance of selected samples during and after the flammability test.

The study confirmed that depending on the mass content in the composite, boehmite, when added as a flame retardant to PVC, exhibits good flame-retardant properties. CA demonstrates increasingly better flame-retardant properties with the increase in its content in the sample. The optimal content of CA in the material appears to

**T a b l e 4. Flammability of PVC/flame retardant composites**

| Flame retardant | Amount of flame retardant, wt% | *L*, cm | *V*, mm/min | *t*, s | Flammability class |
|---|---|---|---|---|---|
| Reference | 0 | 2.3 | – | – | HB75 |
| CA | 10 | – | – | 57 | HB75 |
| | 20 | 4.5 | 128 | 21 | HB |
| | 30 | 1.8 | 73 | 14.8 | HB |
| | 40 | 1.5 | 77 | 11.7 | HB |
| | 50 | 0.6 | 120 | 3 | HB |
| PU | 10 | 3.0 | 37 | 48 | HB |
| | 20 | 2.0 | 27 | 45 | HB |
| | 30 | 1.7 | 32 | 32 | HB |
| | 40 | 0.7 | 13 | 32 | HB |
| | 50 | 0.5 | 17 | 18 | HB |


**Fig. 2. Combustion test of reference sample**

be 20–30 wt%, as evidenced by the unchanged results in length, which burned at higher contents and showed a minimally shorter burning time after the fire source was extinguished. Moreover, the composite with even a large addition of CA exhibits good mechanical properties, which may allow for its wide application in the industry as a universal flame-retardant additive in PVC-based composites. The addition of PU proved to be a more effective flame retardant among those tested, considering both flame-retardant and mechanical properties.

The trend holds in the case of PU: the higher the flame-retardant content, the lower the losses and the shorter the burning time, but it does not increase as rapidly as in the case of CA. However, it was added to a higher amount of 30-40 wt%. Only a content of 40-50 wt% in the PVC composite exhibits the best flame-retardant properties, but it unfortunately shows worse mechanical properties compared to CA.

## CONCLUSIONS

The use of the proposed raw materials allowed the preparation of homogeneous soft PVC composites with inorganic fillers, i.e., boehmites. Flammability tests showed that the fillers used reduced PVC flammability, so they acted as flame retardants. Regardless of the type of filler, with the increase in its content, the burning rate, and the burning time after the removal of the fire source were reduced. The addition of 20 wt.% boehmite increased the hardness of PVC, while the materials based on Pural SB showed greater hardness. This may be since this filler was characterized by smaller grain sizes compared to the Catapal B filler, thanks to which finer fractions were better dispersed in the PVC matrix. Tensile tests showed that in the series of composites with CA, the tensile strength increased with an increase in the filler content, while in the series with PU the trend was the opposite.

In summary, the use of an inorganic filler such as boehmite allows for obtaining flame-retardant PVC composites as an alternative to insulating materials based on halogenated flame retardants.

*Authors contribution*

K.D. – conceptualization, methodology, writing-original draft, writing-review and editing, project administration, funding acquisition; A.G. – conceptualization, methodology, validation, project administration, funding acquisition; K.D. – conceptualization, methodology, validation, investigation, writing-original draft, writing-review and editing; A.P. – methodology, validation, formal analysis, writing-original draft, writing-review and editing, visualization.

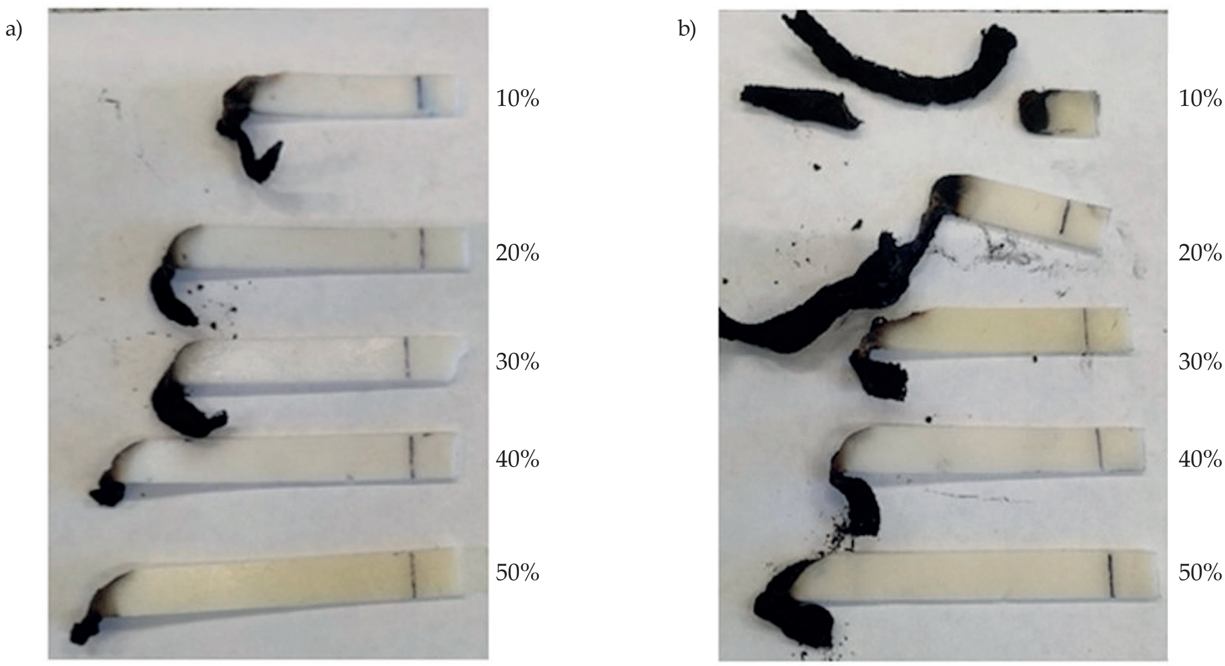


**Fig. 3. Appearance of composite strips after flammability tests**

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***Conflict of interest***
The authors declare no conflict of interest.



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