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Influence of fiber filler on the structure and properties of polyvinyl chloride / acrylonitrile-butadiene-styrene blends

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Abstract: The paper deals with filling polyvinyl chloride (PVC) compositions modified with acrylonitrile-butadiene-styrene (ABS) in a wide concentration range (per hundredth of PVC resin (phr) 10–20 phr ABS) with short-fiber basalt fiber, intended for production of profile mounded products of various functional purposes. The introduction of ABS into PVC compositions makes it easier to process the material, and improves the impact toughness, which for rigid PVC products is an important factor since it expands the areas of their application. However, as a rule, polymer-polymer blends are characterized by low mechanical properties under compression, tension and bending, resulting from the formation of two-phase morphology, which is often heterogeneous. To increase stiffness and strength, 7 phr of short-fiber basalt fiber was introduced into their composition. Experimental samples were made by extrusion in the form of flat profiles. Filling with short-fiber basalt fiber resulted in an increase in the bending strength of PVC specimens over the entire concentration range with an optimum in the region of 10–20 phr ABS, as compared to unfilled blended polymer compositions. The method of energy dispersion analysis established that, depending on the ABS content in the mixture, the interaction of the polymer (PVC) with the filler (fiber) is observed either at the polymer-filler boundary or through the boundary layer of the elastomer.

Keywords: polyvinyl chloride; acrylonitrile butadiene styrene; PVC/ABS; basalt fiber; filler; polymer mixtures.

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Влияние волокнистого наполнителя на структуру и свойства смесей поливинилхлорид / акрилонитрил-бутадиен-стирол

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Аннотация: Работа посвящена наполнению поливинилхлорид (ПВХ) композиций, модифицированных акрилонитрил-бутадиен-стиролом (АБС) в широком интервале концентраций (10–40 масс. ч. на 100 масс. ч. ПВХ), коротковолокнистой базальтовой фиброй, предназначенных для производства профильно-погонажных изделий различного функционального назначения. Введение АБС в состав ПВХ-композиций позволяет облегчить перерабатываемость материала, а также улучшить ударную вязкость, что для жестких изделий из ПВХ является важным фактором для расширения областей их применения. Однако, как правило, полимер-полимерные смеси характеризуются низкими механическими свойствами при сжатии, растяжении и изгибе, что связано с формированием двухфазной морфологии, часто неоднородной. Для усиления жесткости и прочности в их состав введена коротковолокнистая базальтовая фибра в количестве 7 масс. ч. Экспериментальные образцы изготовлены методом экструзии в виде плоских профилей. Наполнение коротковолокнистой базальтовой фиброй привело к росту показателей прочности на изгиб ПВХ-образцов во всем интервале концентраций с оптимумом в области 10–20 масс. ч. АБС, по сравнению с ненаполненными смесевыми полимерными композициями. Методом энергодисперсионного анализа установлено, что в зависимости от содержания АБС в смеси взаимодействие полимера (ПВХ) с наполнителем (фибра) наблюдается либо на границе полимер-наполнитель, либо через граничный слой эластомера.

Ключевые слова: поливинилхлорид; акрилонитрил-бутадиен-стирол; ПВХ/АБС; базальтовая фибра; наполнение; смеси полимеров.

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1. Introduction

Polymeric materials are widely used in almost all industries [1, 2]. Being a large-tonnage material, polyvinyl chloride (PVC) occupies one of the leading places in the technology of polymeric and composite materials due to the presence of necessary for consumers technological and operational properties [3, 4]; this enables to process it into various profile products (sheets, window profiles, pipes, siding, etc.) [5, 6]. Increasing requirements for the operational and technological properties of PVC-based materials and products makes it necessary to introduce modifiers, technological additives, stabilizers, fillers, dyes, pigments, etc. into their composition [7]. Most often PVC compositions are modified by small doses of acrylic technological additives, a variety of which allows obtaining products with a different set of properties. Increasing the concentration of acrylic additives with PVC-based compositions can contribute to an increase in positive results, both in processing and operation [8].

In such materials, the decisive processes that determine their morphology and properties are the transition layer located at the interface. In these layers, complex interfacial intermolecular interactions occur, leading to the formation of supramolecular structures of various types [9] and giving the materials properties that none of the components had separately [10].

Introduction of other polymers into PVC causes a number of problems related to the uniform distribution of the modifier in the composition [11, 12], which is primarily due to the different rheological and thermal characteristics of PVC and the polymer modifier.

The most interesting is the consideration of acrylonitrile-butadiene-styrene (ABS) copolymer. In 1948, the first steps were taken to obtain useful thermoplastics of the ABS type by mixing poly(styrene-acrylonitrile) with “Buna-N” (butadiene-acrylonitrile copolymer) or “Buna-S” (butadiene-styrene copolymer) [13]. However, the industrial introduction was not successful and was accompanied by insufficient low-temperature impact properties. Later it was found that polybutadiene remained elastic at temperatures lower than other copolymers, but polybutadiene and poly(styrene-acrylonitrile) were incompatible [14]. The solution to the problem was preliminary preparation of polybutadiene by emulsion polymerization with the subsequent use of this latex as a medium for emulsion polymerization of styrene and acrylonitrile. In 1959, the American company Borg Warner patented ABS [15], obtained by grafting styrene and acrylonitrile to polybutadiene, and in 1960 it was actively used in polymer compositions based on PVC [16]. Thus, by 1960 ABS had become a two-phase system in the form of butadiene rubber, which has a hardening effect, and the rigid phase of styrene-acrylonitrile copolymer (SAN), which increases the compatibility of PVC-based mixtures and enhances interfacial adhesion due to its polarity [17, 18].

At the moment, heterophase blends of polymers do not cause any concern regarding deterioration of properties. Since the 1970s the output of blended materials has begun to noticeably exceed the rate of production [19].

In PVC/ABS blends, the interaction between PVC and SAN phases is an important factor in obtaining optimum compatibility of the blend components. This interaction is influenced by the acrylonitrile content of the SAN copolymer. Observations have shown that PVC/ABS miscibility occurs at acrylonitrile content of 12–26 %, otherwise the mixture may become immiscible [20, 21].

Most studies of PVC/ABS blends are focused on blending recycled components, which are known to be inferior in the main indicators to the initially obtained PVC and ABS [22].

At the same time, the main direction of the study of such mixtures is the consideration of formulations with a matrix layer of ABS modified by the dispersed phase of PVC [23]. However, the area of application of mixtures based on ABS modified by PVC is limited to the manufacture of products for the automotive industry, containers, crates, garbage cans, etc. [24, 25], not considering the production of products for the construction industry. Since PVC is the main “construction” polymer, PVC-based mixtures modified by ABS can be recommended for the production of construction products for various functional purposes [26].

When combining the components of PVC/ABS blends, preference is given to manufacturing products by pressing [27], which does not allow for full reliance and comparison of test results obtained by extrusion method. There are also a number of interesting studies on the extrusion technology of PVC/ABS blend products. For example, in extrusion production of wood-filled polymer pipes an attempt was made to replace chlorinated polyethylene and recyclability modifier with ABS. It is shown that without deterioration of physical and mechanical properties of wood-filled composites the use of ABS gives an opportunity to exclude both impact strength modifier and recyclability modifier from the formulation [28].

Fillers are one of the most common components in polymer-based composite materials. Fillers are a special kind of modifiers, since they serve not only to reduce the cost of the final product by reducing the polymer consumption, but often to give the material special properties [29].

Powdered disperse fillers and fiber fillers are widely used.

Fiber fillers are inferior to disperse fillers in terms of variety of assortment, but they can be more effective in terms of improving physical and mechanical properties. Basalt fiber fillers are the most promising for polymers. An important factor influencing the increasing use of basalt fibers as a reinforcing component is the absence of a full-fledged alternative [30]. For example, in the USA, 1 kg of basalt fiber is 3 times cheaper than that of glass fiber, and 15 times cheaper than that of carbon fiber [31]. At the same time, the application area of basalt fibers is close to glass fibers [30].

Basalt fiber is characterized by increased strength and the possibility of application in a wider range of negative and positive temperatures. Due to their strength, environmental friendliness, heat resistance, and durability, polymer products filled with basalt fiber are very widely used in the construction industry [32]. Review of works on use of basalt fibers has shown that the main investigations concern filling of polypropylene [33], polyolefin [34], epoxy [35], polyethylene [36] polymer composite materials. However, little attention has been paid to basalt fiber filling of PVC, one of the largest-tonnage polymers.

The aim of the paper is to study the effect of basalt fiber on polyvinyl chloride compositions modified with acrylonitrile butadiene styrene copolymer. The objectives of the study are to evaluate the strength properties and analyze the microstructure of basic PVC/ABS polymer mixtures as well as mixtures containing 7 phr basalt fiber.

Table 1. Extrusion temperature regime

Cylinder zones ……...	1	2	3	4	5	6	7	8	9	10
Temperature, °C …………	190	193	195	195	197	197	197	200	200	200

2. Materials and methods

2.1. Methods for obtaining compositions

Recipes of investigated compositions were calculated for per hundredth of suspension PVC resin (phr) of C-6359-M brandand included 5 phr of bi-basic lead stearate (complex stabilizer), 3 phr of calcium stearate (stabilizer-lubricant) and from 0 to 40 phr ABS-20P (impact strength and recyclability modifier). ABS-20P has a softening point of about 100 °C and melt flow properties at 220 °C and 10 kgf at least 5–12 g / 10 min. The short-fiber modified basalt fiber from CEMMIX Company oiled with water-soluble alkaline K-B42 with an average fiber diameter of 8–10 μm and an average length of 100–500 μm was used as filler for the compositions in question. Basalt fiber meets the requirements of TU 20.59.59-001-90557835-2017.

Pre-mixing of the powdered components was carried out on a laboratory mixer LDU-3 MPR with a propeller nozzle for 4 min at 700 rpm.

The LabTechScientific LTE 16-40 laboratory extruder was used to produce flat profiles of 2×22 mm or larger (depending on the swelling coefficient of the melt) and 20–25 cm in length. The temperature mode was adjusted over the ten cylinder zones (Table 1) with screw speeds of 16, 20, and 25 rpm; the optimal motor load was 30–50 % of the maximum.

2.2. Methods for determining properties

Tensile and static bending tests were carried out on the tensile testing machine PM-250 according to Russian Standard 11262-2017 (ISO 527-2:2012) “Plastics. Tensile test method” and Russian Standard 4648-2014 (ISO 1782010) “Plastics. Method of static bending test”, respectively. To determine the tensile strength, the traverse speed was 100 mm/min, for the static bending strength – 50 mm·min^{-1}. Tests were performed on five specimens at room temperature. The error of load readings on the scales within the working range of measurements is up to 2 %.

Table 2. Mechanical properties of PVC/ABS compositions

Properties	ABS concentration, phr				
	0	10	20	30	40
Tensile strength, MPa	52/44	50/45	49/45	49/44	49/42
Bending strength, MPa	65/83	51/81	50/75	52/73	53/69

Note: The numerator gives data for the properties of compositions without fiber, but the denominator for those with 7 phr of basalt fiber.

Electron micrographs were taken on a CarlZeiss Merlin high-resolution scanning electron microscope at a primary electron accelerating voltage of 5 kV and a probe current of 300 pA for minimal impact on the study object. Samples were placed in liquid nitrogen, followed by chipping. The chips were made in the direction of the extrusion axis. The chipped samples were fixed on a holder and placed in a chamber of the Quorum Q 150TES vacuum unit. The conductive layer was applied by cathodic spraying with Au/Pd alloy in an 80/20 ratio. The thickness of the deposited layer was 15 nm. The elemental composition of the samples in different parts of the composite with fixation of nitrogen as a reference element in the ABS composition and chlorine in the PVC composition was studied using energy dispersive analysis.

3. Results and Discussion

Table 2 shows the mechanical properties of unfilled and basalt fiber-filled PVC compositions with different contents of ABS for comparison.

The data presented in Table 2 show that with the introduction of ABS tensile strength remains at the level of unmodified PVC. With the increase of ABS content there is a decrease in the index of bending strength, which is associated with the release in the PVC matrix of ABS particles in the form of dispersed phase of different sizes [26]. The use of short-fiber fiber caused an increase in the bending strength. The observed insignificant decrease in tensile strength (in 1.08–1.18 times compared to unfilled compositions with appropriate ABS content) with the introduction of fiber is within the limits of acceptable values of indicators for this type of rigid PVC products.

The use of basalt fiber with an increase of ABS content in the composition led to a decrease in the absolute values of the bending strength. However, as it follows from the data in Fig. 1, despite the reduction in the absolute values of the bending strength in the presence of ABS, the degree of increase in the index was higher compared to unfilled PVC/ABS. At the same time, the maximum increase in flexural strength in the presence of fiber was observed in the range of 10–20 phr ABS in the mixed composition.

To explain the results of the study through the energy dispersive method the elemental composition by percentage mass was analyzed for unfilled and filled with basalt fiber PVC compositions. For example, a microphotograph with element analysis points for PVC compositions filled with basalt fiber and modified with 40 phr ABS (Fig. 2).

Figure 3 shows the examples of spectra of samples containing 40 phr ABS to calculate the number of reference elements on the fiber surface and in the polymer matrix. As it follows from them, the ABS made joint structure with PVC matrix, i.e. it was present only in dispersion medium. On the surface of basalt fibers (Fig. 3*a*) the presence of reference element of nitrogen was not revealed, which testifies to predominance of interphase interaction on the border of basalt filler with PVC rather than with ABS.

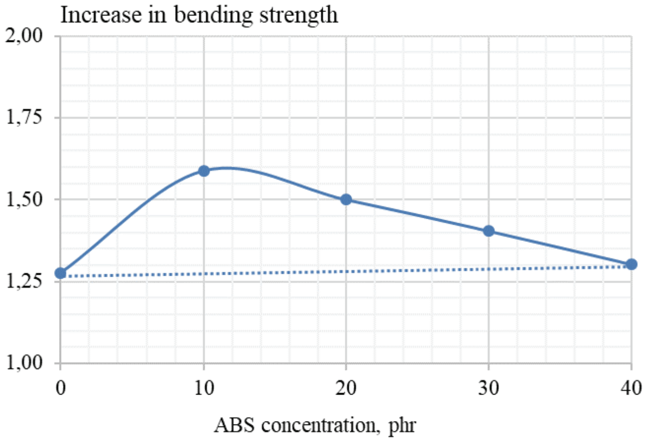


Fig. 1. Degree of increase in bending strength of PVC/ABS samples (fiber content 7 phr)

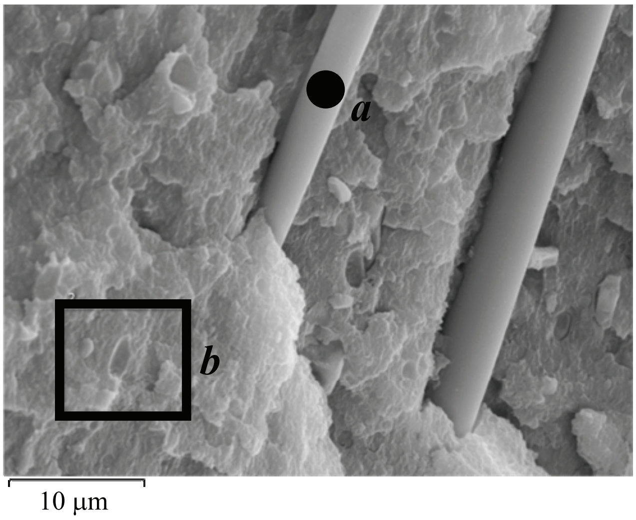


Fig. 2. Example microphotograph of a PVC composition filled with basalt fiber and modified with 40 phr ABS, with element analysis points:
a – in the dispersed phase on the surface of the basalt fiber;
b – in the dispersion medium of the PVC/ABS matrix

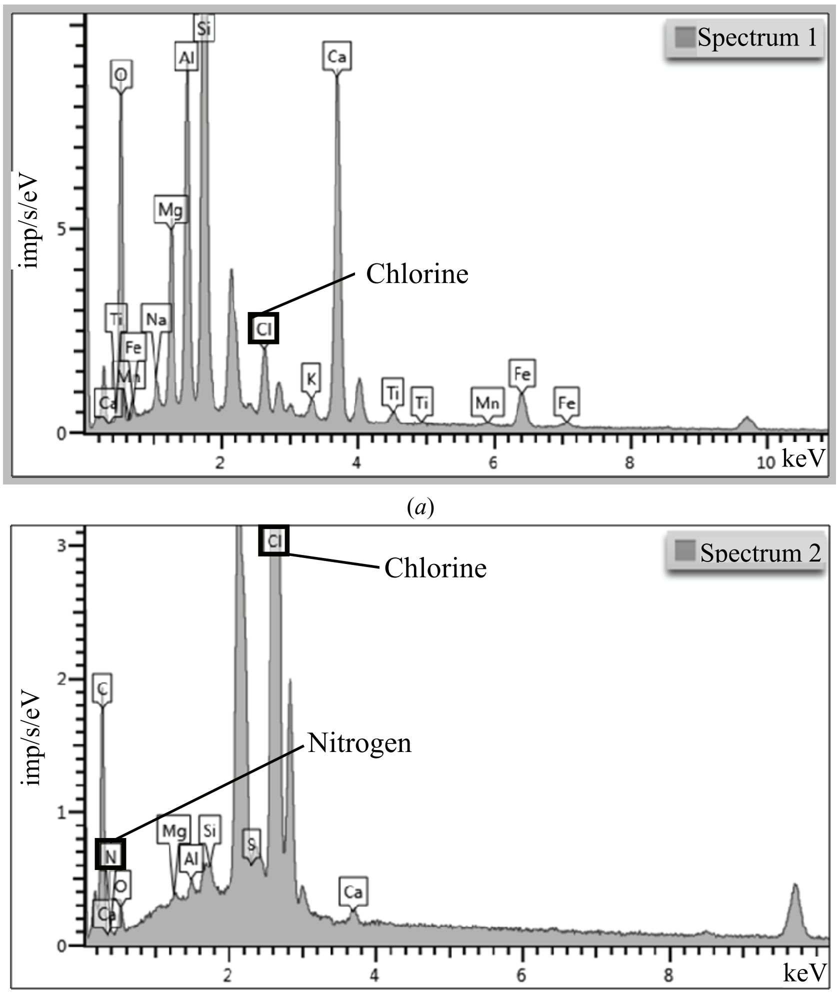
The comparison of the data in Figs 4 and 5 reflects the effect of basalt fiber on the distribution of ABS in the PVC matrix. In contrast to unfilled compositions (Fig. 4), the ABS in the presence of basalt fiber formed joint structures with PVC at concentrations greater than 10 phr (Fig. 5 shows the element nitrogen in the dispersed phase of PVC in the range from 2.5 to 4 %).

The character of changes in the PVC content in the dispersion medium of the matrix is practically similar both in unfilled compositions and in the presence of basalt fiber (curves *a* in Figs 4 and 5), differing only in absolute values.

The processed data in Figs 4 and 5 are presented in Table 3 as a change in the ratio of PVC and ABS in the dispersion medium.

The analysis of the results enables to draw the following conclusions.

As already noted, in PVC/ABS compositions only at the content of 40 phr ABS joint dispersion medium structure is formed.



(*a*)

(*b*)

Fig. 3. Examples of energy dispersion spectrum of PVC composition filled with basalt fiber and modified with 40 phr ABS, with the analysis points of the elements:
a – in the dispersed phase on the surface of the basalt fiber; *b* – in the dispersion medium of the PVC/ABS matrix

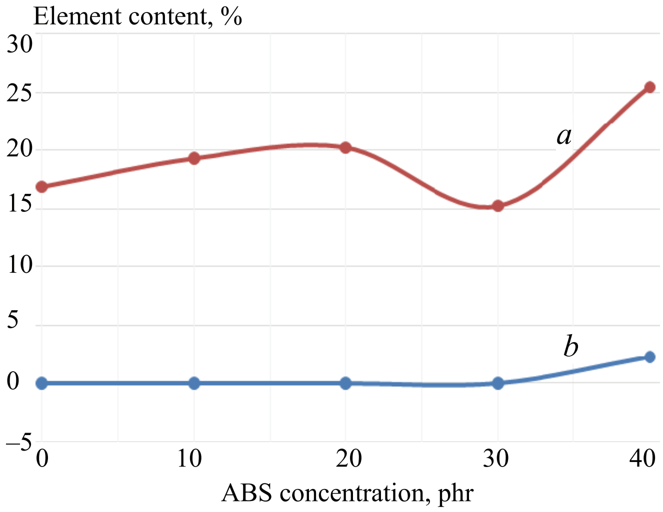


Fig. 4. Elemental composition of unfilled compositions in PVC dispersion medium:
a – chlorine; *b* – nitrogen

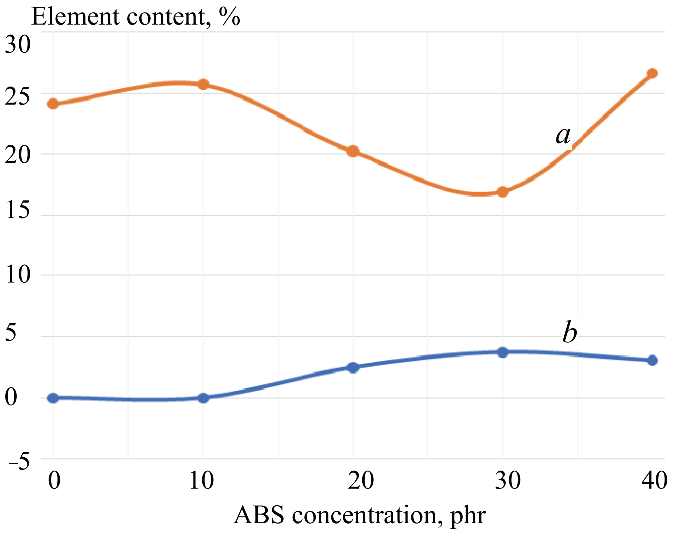


Fig. 5. Elemental composition of compositions filled with basalt fiber in PVC dispersion medium:
a – chlorine; *b* – nitrogen

Table 3. Component content change (%) in the dispersion medium of PVC/ABS composite

Compositions	ABS concentration, phr				
	0	10	20	30	40
PVC/ABS in unfilled composite	100/0	100/0	100/0	100/0	92/8
PVC/ABS in the presence of fiber	100/0	100/0	89/11	82/18	90/10

Note: The numerator shows the chlorine content (%), the denominator shows the nitrogen content (%).

In fiber-filled compositions without ABS modifier and with 10 phr ABS, the dispersion medium is completely formed by PVC. At concentrations of 20, 30 and 40 phr ABS content of PVC in dispersion medium is in the limit of 82–90 %, i.e. ABS is in the composite structure in two different states – as part of the dispersion medium of PVC and as a dispersed phase. It is obvious that the developing in general heterogeneity of microheterogeneous structure explains the changes in the bending strength when the PVC/ABS mixture is filled with fiber.

The results of dispersion analysis revealed that at 10 % ABS – part of ABS is found on the surface of the fiber (in the amount of 5.02 % of nitrogen by mass element composition). The PVC content on the surface of the basalt fiber varies in the range 0.37–2.49 %, ambiguously depending on the ABS content.

4. Conclusions

Thus, the presence of ABS in PVC contributes to an increase in the bending strength, the maximum of which is manifested in the range of 10–30 phr ABS. Despite the reduction of absolute values of the bending strength, the use of basalt fiber with increased ABS content in the composition causes a greater degree of increase in the index compared to unfilled PVC/ ABS compositions.

The significance of the research is justified by the development of effective compositions for the production of profile-shaped molded products for construction purposes.

In the future, the features of filling PVC/ABS mixtures with basalt fiber in a wider concentration range with the possibility of phase inversion in the dispersion medium of the polymer mixture will be explored.

5. Funding

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6. Conflict of interests

The authors declare no conflict of interest.

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