CARACTERÍSTICAS DE FORÇA DE COMPÓSITOS DE POLÍMERO BASEADOS EM POLIPROPILENO RECICLADO PREENCHIDO DE CASCA DE ARROZ NO PROCESSO DE ABSORÇÃO DE UMIDADE E ENVELHECIMENTO NATURAL**STRENGTH CHARACTERISTICS OF POLYMER COMPOSITES BASED ON RECYCLED POLYPROPYLENE FILLED WITH RICE HUSK DURING MOISTURE ABSORPTION AND NATURAL AGING****ПРОЧНОСТНЫЕ ХАРАКТЕРИСТИКИ ПОЛИМЕРНЫХ КОМПОЗИТОВ НА ОСНОВЕ ВТОРИЧНОГО ПОЛИПРОПИЛЕНА, НАПОЛНЕННОГО РИСОВОЙ ШЕЛУХОЙ, В ПРОЦЕССЕ ПОГЛОЩЕНИЯ ВЛАГИ И ЕСТЕСТВЕННОГО СТАРЕНИЯ**

SADRITDINOV, Aynur R.^{1*}; CHERNOVA, Valentina V.²; BAZUNOVA, Marina V.³; ZAKHAROVA, Elena M.⁴; ZAKHAROV, Vadim P.⁵;

^{1,2,3,5} Bashkir State University, Faculty of Chemistry, Department of Macromolecular Compounds and General Chemical Technology, 450076, 33 Zaki Validi Str., Ufa – Russian Federation

⁴ Ufa Federal Research Centre of the Russian Academy of Sciences, Department of Macromolecular Compounds, 450054, 71 October Ave., Ufa – Russian Federation

** Corresponding author
e-mail: aynur.sadritdinov@mail.ru*

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RESUMO

A relevância do problema em estudo deve-se ao aumento da carga ambiental sobre o meio ambiente devido ao contínuo crescimento dos resíduos plásticos, dos quais uma parcela significativa é de polipropileno. Uma das soluções para esse problema é o uso do polipropileno reciclado na reciclagem para obtenção de produtos plásticos à base de compósitos preenchidos com casca de arroz. Isso permite reduzir o custo original dos produtos acabados, regular suas propriedades físicas e mecânicas e reduzir o volume de polímero sintético de difícil decomposição que acaba em resíduos. A principal propriedade do polipropileno, que muda durante o seu enchimento com casca de arroz, é a absorção de umidade, que contribui para uma mudança nas características de resistência do compósito polimérico e acelera seu envelhecimento sob a influência de fatores ambientais. Nesse sentido, o objetivo deste artigo é identificar as regularidades da influência da umidade absorvida e do envelhecimento natural nas propriedades físicas e mecânicas de compósitos poliméricos à base de polipropileno reciclado preenchido com casca de arroz. A abordagem principal para o estudo deste problema são os testes de laboratório sobre a saturação de compósitos poliméricos com água com diferentes temperaturas e tempos de exposição, bem como os testes de materiais no processo de envelhecimento natural de longo prazo sob a influência de fatores ambientais. O fator chave que caracteriza a mudança nas propriedades físicas e mecânicas dos materiais, neste caso, é o módulo de elasticidade, alongamento e resistência à ruptura no processo de alongamento dos protótipos. Os materiais do artigo são de valor prático para o processamento de polímeros termoplásticos reciclados, bem como para a criação de compósitos poliméricos biodegradáveis.

Palavras-chave: *polipropileno reciclado, casca de arroz, absorção de umidade, envelhecimento natural, propriedades físicas e mecânicas.*

ABSTRACT

The relevance of the issue under investigation is conditioned by an increase of the ecological burden on the environment due to the continuous increase of plastic waste, a significant proportion of polypropylene. One solution to this problem is the involvement of recycled polypropylene to obtain plastic products based on composites filled with rice husk. It makes it possible to reduce the prime cost of finished products, regulate their physical and mechanical properties, and reduce the volume of difficult-to-decompose synthetic polymers that end up in waste. The key property of polypropylene, which changes during its filling with rice husk, is moisture absorption, which contributes to a change in the strength characteristics of the polymer composite and accelerates

its aging under the influence of environmental factors. In this regard, this research aims to identify the patterns of the influence of absorbed moisture and natural aging on the physical and mechanical properties of polymer composites based on recycled polypropylene filled with rice husk. The leading approach to the study of this issue is laboratory tests on the saturation of polymer composites with water of different temperatures and duration of exposure and testing materials during long-term natural aging under the influence of environmental factors. The key factor characterizing the change in the physical and mechanical properties of materials, in this case, is the elasticity modulus, elongation, and tensile stress at the break during stretching of the prototypes. The materials of the paper are of practical value for the processing of secondary thermoplastic polymers, as well as the development of biodegradable polymer composites.

Keywords: *recycled polypropylene, rice husk, moisture absorption, natural aging, physical and mechanical properties.*

АННОТАЦИЯ

Актуальность исследуемой проблемы обусловлена увеличением экологической нагрузки на окружающую среду за счет непрерывного роста пластиковых отходов, значительную долю которых составляет полипропилен. Одним из решений этой проблемы является вовлечение вторичного полипропилена в повторную переработку с получением пластмассовых изделий на основе композитов, наполненных рисовой шелухой. Это позволяет снизить себестоимость готовых изделий, регулировать их физико-механические свойства, снизить объем трудно разлагаемого синтетического полимера, попадающего в отходы. Ключевым свойством полипропилена, изменяющимся в процессе его наполнения рисовой шелухой, является поглощение влаги, что способствует изменению прочностных характеристик полимерного композита и ускорению его старения под действием факторов внешней среды. В связи с этим, данная статья направлена на выявление закономерностей влияния поглощенной влаги и естественного старения на физико-механические свойства полимерных композитов на основе вторичного полипропилена, наполненного рисовой шелухой. Ведущим подходом к исследованию данной проблемы являются лабораторные испытания по насыщению полимерных композитов водой с различной температурой и длительностью воздействия, а также проведение испытаний материалов в процессе длительного естественного старения под действием факторов внешней среды. Ключевым фактором, характеризующим изменение физико-механических свойств материалов в этом случае, является модуль упругости, удлинение и прочность при разрыве в процессе растяжения опытных образцов. Материалы статьи представляют практическую ценность для переработки вторичных термопластичных полимеров, а также создания биоразлагаемых полимерных композитов.

Ключевые слова: *вторичный полипропилен, рисовая шелуха, влагопоглощение, естественное старение, физико-механические свойства.*

1. INTRODUCTION:

Polypropylene is the most demanded thermoplastic for various polymeric products: medical materials, bottles, food containers, pipelines, chemical tanks. It is an affordable material that is easy to process and has good physical and mechanical properties. At the same time, out-of-service plastic products made of polypropylene, due to the long time required for their decomposition, cause serious harm to the environment (Asad *et al.*, 2011; Geyer *et al.*, 2017; Poletto, 2017; Herbort *et al.*, 2018; Salikhov *et al.*, 2018; Camargo and Saron, 2020). As a result, an urgent task is to develop plastic waste methods in recycling, including polymer composites based on multicomponent systems (Iwona *et al.*, 2017; Rahimi and García, 2017; Rhodes, 2018).

One of the ways to solve this problem is

to fill polypropylene with components of plant origin, which makes it possible to reduce the cost of the obtained polymer composites, improve the physical and mechanical properties of finished products and reduce the ecological burden by increasing the share of the biodegradable component in the plastic product and reducing the volume of plastic waste (Leja and Lewandowicz, 2010). For these purposes, the use of rice husks as a plant-based filler for recycled polypropylene has become widespread (Bondaletova and Bondaletov, 2013; Reza *et al.*, 2015; Aridi *et al.*, 2016; Chen *et al.*, 2016; Nwosu-Obieogu *et al.*, 2016; Arjmandi *et al.*, 2017; Bazunova *et al.*, 2018; Vasyukova and Bazunova, 2018; Oisik *et al.*, 2019).

One of the disadvantages of polymer composites based on a mixture of synthetic polymers and plant-based fillers is their weak resistance to water absorption (Ismail *et al.*, 2003; Espert *et al.*, 2004; Ares *et al.*, 2010; Yan

et al., 2016; Vaisanen *et al.*, 2017). Moisture absorption degrades the physical and mechanical properties of polymer composites and, consequently, affects the performance characteristics of plastic products. Also, moisture saturation of the plant component in the composition of the synthetic polymer matrix will affect the strength properties of the polymer composite during natural aging under the influence of environmental factors (Fujisawa *et al.*, 2019; Chiang *et al.*, 2020;).

This research aimed to study the effect of absorbed moisture and natural aging on the physical and mechanical properties of polymer composites based on recycled polypropylene filled with rice husk (Rogovina, 2016; Garcia and Robertson, 2017).

The paper indicates that filling hydrophobic secondary polypropylene with rice husk leads to an increase in the hydrophilicity of the polymer composite surface and an increase in the amount of absorbed moisture. Polymer composites achieve a high degree of water saturation in hot water (100 °C) in no more than 30 minutes, while the elasticity modulus and strength at break decrease and the change in these characteristics are determined not by the duration of exposure but by the amount of absorbed moisture (Wang *et al.*, 2020). In the process of natural aging under the influence of environmental factors for composites containing up to 15 parts per hundred rice husk, there is an increase in the elasticity modulus at the break, which decreases for recycled polypropylene filled with 30 parts per hundred of rice husk with a natural aging duration of more than 90 days. A method is proposed for predicting the effect on the strength characteristics of polymer composites based on recycled polypropylene filled with rice husks during long-term natural aging due to short-term (no more than 30 minutes) exposure to hot water (Zheng *et al.*, 2017; Zakharov *et al.*, 2018). This will make it possible to estimate the lower limit of the decrease in the elasticity modulus and tensile stress under the influence of environmental factors (Taynton *et al.*, 2016; Fakhretdinov *et al.*, 2017; Turku *et al.*, 2017; Yang *et al.*, 2017).

2. MATERIALS AND METHODS:

The leading approach to the study of this problem is laboratory tests on the saturation of polymer composites with water of different temperatures and duration of exposure and testing materials in the process of long-term

natural aging under the influence of environmental factors. The key factor characterizing the change in the physical and mechanical properties of materials, in this case, is the elasticity modulus, elongation, and strength at the break during stretching of the prototypes.

It was used recycled polypropylene in the study by crushing substandard plastic products produced in industrial conditions. Rice husks (cellulose 40-45%, lignin 20-25%, hemicellulose 15-17%, mineral substances 18-22%) were used as a plant-based filler, the fraction of which contained particles with a diameter of no more than 0.6 mm. The relation of the mass fraction (pph) of rice husks was calculated per 100 fractions of recycled polypropylene.

Homogenization of the initial components was carried out in a mixing cylinder of a "Plastograph EC" (Brabender) plastograph at a temperature of 180 °C, a screw speed of 30 rpm for 15 min at a load of 200 N. The prototypes for testing were prepared by injection molding using an automatic molding machine "Babyplast" horizontal type with injection volume up to 15 cm³. The dimensions of the test samples correspond to GOST 11262-2017 (Appendix B. Small test samples. Type 5), where the sample length is 80 mm, the width of the test sample is 5 ± 0.2 mm, the thickness of the test sample is 1-4 mm (in this study – 2 mm). The initial weight of the test samples was 1094 ± 0.3 mg. The surface finishing of the samples was not performed. The moisture content of rice husks was determined on a moisture analyzer MOC63u (Shimadzu, Japan) in a standard mode and was 6.8% (GOST 11262-2017..., 2018).

Testing of polymer composites for water absorption was carried out following GOST 4650-2014. Following GOST 4650-2014, two methods were applied. Method 1: the samples were dried for testing in an oven at a temperature of 50 ± 2 °C for at least 24 hours, then cooled to ambient temperature in a desiccator and weighed. Further, the weighing results were recorded in milligrams accurate to the first decimal place. This process is repeated until the mass of the samples is constant, i.e., two consecutive weighings will differ by no more than ± 0.1 mg. The test samples were then placed in a thermostat filled with distilled water at a temperature of 23.0 ± 1.0 °C. After holding in water for 24 ± 1 h, the test samples were removed from the water, the water was removed from the surface of the samples with a clean, dry cloth or filter paper, and within no more than 1

min after removing the samples from the water, they were weighed to the nearest 0.1 mg. Following method 2 pursuant to GOST 4650-2014, the test samples were dried in a drying oven at a temperature of 50.0 ± 2.0 °C for at least 24 hours. Then the samples were cooled to ambient temperature in a desiccator and weighed to the nearest 0.1 mg. This process is repeated until the mass of the samples is constant. Then the test samples were placed in a thermostat with boiling distilled water so that they rest on one edge and were completely immersed in water. (30 ± 2) min later, the test samples are removed from the boiling water and cooled in distilled water to ambient temperature. After cooling the samples for (15 ± 1) min, they are removed from the water one by one. Water is removed from the surface of the samples with a dry cloth, and the samples are immediately weighed to the nearest 0.1 mg (GOST 4650-2014..., 2015).

Mechanical uniaxial tensile tests were performed following GOST 11262-2017 on an AGS-X10kN universal testing machine (Shimadzu, Japan) at a temperature of 23 ± 2 °C and a moving gripper speed of 1 mm/min to determine the elasticity modulus and a speed of 5 mm/min to determine other characteristics (GOST 11262-2017..., 2018).

To analyze the wetting properties of the test samples surface, the contact angle of wetting was determined from the projection of a drop of distilled water applied to the surface of the material. The contact angle of wetting was calculated according to Equation 1:

$$\cos \theta = \frac{(d/2)^2 - h^2}{(d/2)^2 + h^2} \quad (\text{Eq. 1})$$

Where d – water drop diameter; h – drop height.

In order to study the patterns of changes in the physical and mechanical properties of polymer composites under the influence of environmental factors, the prepared samples were subjected to natural aging from September to February (including all months) following GOST 9.708-83 "Unified system of protection against corrosion and aging. Plastics aging test methods under the influence of natural and artificial climatic factors". The essence of the method lies in the fact that the samples are exposed to natural climatic factors (in this study, UV irradiation is at room temperature) for a given

test duration, and the resistance to the specified effect is determined by changing one or several property indicators (in this study, physical and mechanical) (GOST 9.708-83..., 1985).

3. RESULTS AND DISCUSSION:

Filling the hydrophobic polypropylene with rice husk with a hydrophilic surface leads to an improvement in the surface wetting properties of the polymer composite (Table 1), which is confirmed by a corresponding change in the contact angle of wetting. Recycled polypropylene is characterized by low values of the surface hydrophilicity (contact angle of wetting of 72 degrees), which is characteristic of polyolefins. The introduction of a hydrophilic filler consistently increases the surface wettability with water and a rice husk content of 30. the contact angle of wetting is reduced to 54 degrees. An increase in the hydrophilicity of the surface of the polymer composite when it is filled with rice husk will increase the moisture absorption of the plastic product and accelerate the biodegradation of the plant-based component in the polymer matrix under the influence of microorganisms.

Analysis of the data obtained on the effect of moisture on polymer composites during their exposure to hot water at 100 °C indicates that the maximum value of water absorption is achieved when the exposure time of the sample is no more than 30 min (Figure 1).

This is caused by the fact that the kinetic dependencies of the amount of absorbed moisture on holding the sample in hot water have the form of curves that reach a plateau upon water absorption for 30 min. Further exposure to hot water has practically no effect on the increase in the mass of the prototype. With an increase in rice husk content, the polymer composite increase in the degree of water absorption occurs. So, when the sample is kept in hot water for 3 hours, the moisture absorption of the composite containing 2 pph rice husks is 0.9% and increases 6 times with an increase in the degree of filling of polypropylene with plant filler by 15. A lower increase in the amount of absorbed water than an increase in the content of rice husks in the polymer sample indicates that water absorption mainly occurs in the outer layers of composite materials. This determines an insignificant increase in the mass of absorbed water when polymer samples are kept in hot water for more than 30 minutes.

A decrease in the temperature of polymer

composites in water to 23 °C does not fundamentally affect the form of water absorption curves (Figure 2). The moisture content also increases with an increase in the degree of filling of recycled polypropylene with rice husk. Simultaneously, a decrease in temperature leads to a decrease in the rate of saturation of the polymer sample with water. Thus, when immersed in water with a temperature of 23 °C, the composites, on average, only on the second day, reach the same values of the mass content of absorbed water as when the experiment is carried out in the water with a temperature of 100 °C for 30 minutes.

The results obtained indicate that an increase in the time of exposure of polymer samples, even with a decrease in water temperature from 100 °C to 23 °C determines the possibility of increasing the moisture content in the sample. In particular, a sample of recycled polypropylene filled with 30 pph rice husks, when exposed to hot water with a temperature of 100 °C, absorbs 5.7% moisture within 3 hours. A sample of the same composition at 23 °C for 8 days is saturated with 8% water. Moisture saturation of the polymer composite will contribute to a change in its deformation and strength characteristics. As a result, the study of the physical and mechanical properties of materials makes it possible not only to analyze their behavior during operation but also to predict the ability of materials to biodegrade after they are out of service. In this case, the determining factors of the bioavailability of composite materials are the structure of the composites (particularly their density), the hydrophilicity of the surface, and the ability to absorb water.

To simulate the effect of moisture absorption processes on the deformation and strength characteristics of polymer composites, for filled polymer samples exposed to hot water, their physical and mechanical properties were determined. As the studies have indicated, water absorption in hot water significantly affects the deformation and strength characteristics of polymer composites (Table 2).

Filling of recycled polypropylene with rice husk in an amount of 5 pph leads to an increase in the elasticity modulus from 1910 MPa to 1926 MPa. With a further increase in the content of rice husks, a decrease in the elasticity modulus is observed, down to 1489 MPa, when the polymer is filled with rice husks in an amount of 30 pph. Holding the polymer composite in hot water for 30 min leads to a decrease in the elasticity modulus. The minimum decrease in which is at

the level of 1% compared with the sample not exposed to moisture, is achieved for a composite containing 10 pph rice husk. Further increase in the content of rice husks to 15 and 30 pph leads to a decrease in the elasticity modulus by 9 and 36%, respectively, after 30 minutes of moisture absorption in hot water. It should be noted that a further increase of exposure time of the polymer compound to hot water to 60 and 90 min leads to a slight decrease in the elasticity modulus. This is because the change in the physical and mechanical parameters of polymer composites is determined not by the duration of exposure to hot water but by absorbed moisture content.

The tensile stress of recycled polypropylene increases from 25.4 MPa to 25.9 MPa with the addition of 5 pph rice husk. A further increase in the content of the plant-based filler leads to a decrease in the tensile strength, the value of which reaches 14.7 MPa for a composite with 30 pph rice husks. Exposure to hot water for 30 min of the sample with 2 pph rice husk reduces the tensile strength by 2.7%, and for polymer composites with 5 pph and 30 pph this decline is 12% and 36%, respectively. For the elasticity modulus, an increase in exposure to hot water for more than 30 min has practically no effect on the value of the stress at break of the polymer composite. In the process of water absorption by the polymer sample, the elongation at break decreases, while the maximum decrease in this indicator occurs for the sample containing 10 pph rice husks.

The absorption of water by polymer composites filled with plant-based components will affect the physical and mechanical properties of plastic products due to the destruction of the cellulose-containing filler under the action of microorganisms, as well as in the process of exposure to the polymer matrix from the water-saturated areas in the process of multiple freeze-thaw cycles. In order to study the effect of these factors on the physicochemical properties of polymer composites, the prototypes were exposed to the external environment, which makes it possible to take into account the influence of cyclic temperature changes and, to a lesser extent, biodegradation under the action of microorganisms on the water-saturated areas of the samples of recycled polypropylene filled with rice husk.

In the process of natural aging under the influence of environmental factors during the entire test period (up to 180 days) for polymer composites containing up to 15 pph rice husks, there is a higher elasticity modulus compared to

the original sample (Figure 3). The greatest increase in the elasticity modulus occurs when the samples are kept during the first 30 days. Further exposure to the external environment for 90 days and 180 days consistently reduces the elasticity modulus. A significant decrease in the elasticity modulus under the influence of environmental factors occurs for samples containing 30 pph rice husks. When holding such samples for 90 days and 180 days the elasticity modulus decreases to 1050 MPa and 926 MPa, respectively. It should be noted that for composites containing up to 10 pph the elasticity modulus of samples not subjected to natural aging differs slightly from the value achieved after exposure to hot water. Simultaneously, when filling recycled polypropylene with 30 pph rice husks, the elasticity modulus after treatment with hot water decreases to a value reached after natural aging for 180 days.

Long-term polymer composites exposure to environmental factors leads to a decrease in tensile stress (Figure 4). Before exposure to the external environment, the polymer composite has a maximum strength value at 5 pph rice husk content. In the process of natural aging for 30 days, 90 days, and 180 days tensile stress decreases by 8.2%, 13.6%, and 26.5%, respectively, compared to the sample not subjected to aging. It should be noted that, in comparison with natural aging, the effect of hot water on polymer composites containing rice husks of more than 10 pph has a greater effect on the decrease in strength characteristics. In the process of natural aging, a sequential decrease in tensile stress occurs, while the lower limit of this decrease can be modeled in the process of a short-term (no more than 30 minutes) exposure to hot water. Consequently, it is possible to propose a method for predicting the effect on the strength characteristics of polymer composites based on recycled polypropylene filled with rice husks during long-term natural aging due to short-term (no more than 30 minutes) exposure to hot water.

4. CONCLUSIONS:

Thus, filling hydrophobic recycled polypropylene (contact angle of wetting is 72 degrees) with rice husk in the amount of 30 pph allows increasing the hydrophilicity of the polymer composite surface (the contact angle of wetting is reduced to 54 degrees). This determines an increase in the amount of absorbed moisture with an increase in the

degree of filling the recycled polypropylene with rice husks. Highly filled (more than 10 pph) rice husk samples of recycled polypropylene achieve a high degree of moisture absorption in hot water in less than 30 minutes. A decrease in the water temperature from 100 °C to 23 °C reduces the rate of moisture absorption by almost 100 times, while the long-term presence of the sample in water with a temperature of 23 °C determines the possibility of achieving a higher degree of water saturation. Under the influence of hot water on polymer composites, the elasticity modulus and stress at break decrease, while the change in these characteristics is determined not by the duration of exposure but by the amount of absorbed moisture.

In the process of natural aging under the influence of environmental factors for composites containing up to 15 pph rice husks, there is an increase in the rigidity of materials expressed by an increase in the elasticity modulus at the break. A significant decrease in the elasticity modulus occurs for recycled polypropylene filled with 30 pph rice husks, with a natural aging duration of more than 90 days. It should be noted that the rigidity of the polymer composite under the influence of environmental factors for 180 days reaches the same value as when exposed to hot water. In the process of natural aging, a sequential decrease in tensile stress occurs, while the lower limit of this decrease can be modeled in the process of a short-term (no more than 30 minutes) exposure to the material with hot water.

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Table 1. Contact angle of wetting Θ of polymer composites at 23 °C

Rice husk content, pph	Θ , °C
0	72
2	68
5	63
10	62
15	57
30	54

Table 2. Deformation and strength characteristics of polymer composites after absorption of hot water

Water absorption time, h.	Rice husk content, pph	Elasticity modulus, MPa	Tensile stress at break, MPa	Elongation at break, %
0	2	1910	25.4	12.5
	5	1926	25.9	13.4
	10	1872	22.2	11.7
	15	1788	19.0	7.2
	30	1489	14.7	3.8
0.5	2	1876	23.1	11.2
	5	1907	22.8	10.8
	10	1855	18.9	8.1
	15	1625	14.3	5.9
	30	956	7.7	3.4
1	2	1861	23.0	11.1
	5	1900	22.6	10.7
	10	1847	18.3	8.0
	15	1611	14.0	5.6
	30	952	7.6	3.3
1.5	2	1855	23.1	10.9
	5	1882	22.4	10.4
	10	1827	18.1	7.8
	15	1599	14.1	5.4
	30	952	7.6	3.2

Figure 1. Water absorption of polymer composite samples at a temperature of 100 °C. Rice husk content 2 (1), 5 (2), 10 (3), 15 (4) and 30 (5) parts per hundred.

Figure 2. Water absorption of polymer composite samples at a temperature of 23 °C. Rice husk content 2 (1), 5 (2), 10 (3), 15 (4) and 30 (5) parts per hundred.

Figure 3. Dependence of the elasticity modulus of polymer composite on the content of rice husks. Original sample (1), natural aging for 30 (2), 90 (3), 180 (4) days.

Figure 4. Dependence of tensile stress at break of a polymer composite versus rice husk content. Original sample (1), natural aging for 30 (2), 90 (3), 180 (4) days.