PERIÓDICO TCHÊ QUÍMICA
ARTIGO ORIGINAL

PROPRIEDADES FÍSICO-MECÂNICAS E TERMOFÍSICAS DE COMPOSITOS POLÍMEROS À BASE DE POLIPROPILENO SECUNDÁRIO PREENCHIDOS DE CASCA DE ARROZ

PHYSICAL AND MECHANICAL AND THERMOPHYSICAL PROPERTIES OF POLYMER COMPOSITES BASED ON RECYCLED POLYPROPYLENE FILLED WITH RICE HUSK

ФИЗИКО-МЕХАНИЧЕСКИЕ И ТЕПЛОФИЗИЧЕСКИЕ СВОЙСТВА ПОЛИМЕРНЫХ КОМПОЗИТОВ НА ОСНОВЕ ВТОРИЧНОГО ПОЛИПРОПИЛЕНА, НАПОЛНЕННОГО РИСОВОЙ ШЕЛУХОЙ

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RESUMO

A relevância do estudo se deve à deterioração da situação ambiental no mundo associada ao aumento da quantidade de resíduos plásticos, o que determina a viabilidade de desenvolvimento dos métodos para os envolver na reciclagem e produção futura de produtos plásticos. Este artigo tem como objetivo estudar os padrões de alterações nas propriedades físico-mecânicas e termofísicas de compósitos poliméricos à base de polipropileno secundário preenchido com casca de arroz na ausência de compatibilizadores. A principal abordagem para o estudo deste problema é a determinação do módulo de elasticidade na ruptura e flexão, resistência à tração na ruptura, deformação na ruptura e flexão, temperatura de flexão sob carga e temperatura de amolecimento Vicat, resistência de impacto de Charpy e Izod, parâmetros termofísicos dos compósitos poliméricos, o que possibilita considerar de forma abrangente as propriedades de compósitos biodegradáveis à base de polipropileno secundário na presença de casca de arroz. O artigo mostra que, com o aumento no teor de casca de arroz no compósito polimérico à base de polipropileno secundário, ocorre o aumento no módulo de elasticidade durante a ruptura e flexão. O enchimento de polipropileno com casca de arroz reduz um pouco a resistência à tração e reduz significativamente a elasticidade do polímero. O polipropileno secundário possui uma resistência ao impacto de Charpy superior à viscosidade de Izod. O enchimento do polímero com casca de arroz leva a diminuição da resistência ao impacto, de acordo com Charpy e com Izod. Além disso, com o conteúdo de carga superior a 5 partes de massa esses dois indicadores são quase idênticos. Nesse caso, há um ligeiro aumento na temperatura do início da decomposição térmica dos compósitos, o que determina sua estabilidade térmica durante o processamento. Foi revelado que compósitos poliméricos contendo 2-10 partes de massa de casca de arroz são caracterizados por um grau aumentado de cristalinidade da fase polimérica. Os materiais do artigo são de valor prático para o processamento de polímeros termoplásticos secundários, bem como para a criação de compósitos poliméricos biodegradáveis.

Palavras-chave: *força, elasticidade, resistência ao calor, termogravimetria, calorimetria diferencial de varredura.*

ABSTRACT

The relevance of the study is due to the deterioration of the environmental situation in the world associated with an increase of plastic waste, which determines the feasibility of developing methods for their involvement in recycling for the production of plastic products. Thereby, this article is aimed at studying the patterns of changes in the physical and mechanical and thermophysical properties of polymer composites based on recycled polypropylene filled with rice husk in the absence of compatibilizers. The leading approach to

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the study of this problem is the determination of the modulus of elasticity in flexure, tensile strength at break, strain-to-failure, and bending strain, bending temperature under load and Vicat softening temperature, Charpy V-notch impact energy and Izod impact strength, as well as the thermophysical parameters of polymer composites. The article shows that with an increase in the content of rice husk in a polymer composite based on recycled polypropylene, an increase in the modulus of elasticity in flexure occurs. Filling polypropylene with rice husk slightly reduces the tensile strength and significantly reduces the elasticity of the polymer. Recycled polypropylene has a higher Charpy V-notch impact energy than Izod impact strength. Filling the polymer with rice husk leads to a decrease in impact strength according to both Charpy and Izod, and with a compound content of more than 5 phr, both of these indicators are almost identical. In this case, there is a slight increase in the onset temperature of the composites, which determines their thermal stability during processing. It was revealed that polymer composites containing 2-10 mass parts of rice husk are characterized by an increased degree of crystallinity of the polymer phase. The materials of the article are of practical value for the processing of recycled thermoplastic polymers, as well as the creation of biodegradable polymer composites.

Keywords: *failure resistance, elasticity, heat-distortion temperature, thermogravimetric analysis, differential scanning calorimetry.*

АННОТАЦИЯ

Актуальность исследования обусловлена ухудшением экологической ситуации в мире, связанной с ростом количества пластиковых отходов, что определяет целесообразность разработки способов их вовлечения в повторную переработку с целью производства пластмассовых изделий. В этой связи большие перспективы связаны с разработкой биоразлагаемых композитов, одним из направлений, создания которых является наполнение синтетических полимеров компонентами растительного происхождения. В связи с этим, данная статья направлена на изучение закономерностей изменения физико-механических и теплофизических свойств полимерных композитов на основе вторичного полипропилена, наполненного рисовой шелухой в отсутствии совместителей. Ведущим подходом к исследованию данной проблемы является определение модуля упругости при разрыве и изгибе, прочности при разрыве, деформации при разрыве и изгибе, температуры изгиба под нагрузкой и температуры размягчения по Вика, ударной вязкости по Шарпи и Изоду, теплофизических параметров полимерных композитов (температура начала разложения, тепловые эффекты плавления и кристаллизации), что позволяет комплексно рассмотреть свойства биоразлагаемых композитов на основе вторичного полипропилена в присутствии рисовой шелухи. В статье показано, что с увеличением содержания рисовой шелухи в полимерном композите на основе вторичного полипропилена происходит увеличение модуля упругости при разрыве и изгибе. Наполнение полипропилена рисовой шелухой незначительно снижает прочность при разрыве и существенно уменьшает эластичность полимера. Вторичный полипропилен имеет более высокое значение ударной вязкости по Шарпи по сравнению с вязкостью по Изоду. Наполнение полимера рисовой шелухой приводит к снижению ударной вязкости как по Шарпи, так и по Изоду, причем при содержании наполнителя более 5 м.ч. оба этих показатели практически совпадают. Наполненные рисовой шелухой полимерные композиты обладают большей теплостойкостью. При этом наблюдается небольшое повышение температуры начала термического разложения композитов, что определяет их термическую устойчивость в процессе переработки. Выявлено, что полимерные композиты, содержащие 2-10 массовые части рисовой шелухи, характеризуются повышенной степенью кристалличности полимерной фазы. Материалы статьи представляют практическую ценность для переработки вторичных термопластичных полимеров, а также создания биоразлагаемых полимерных композитов.

Ключевые слова: *прочность, эластичность, теплостойкость, термогравиметрия, дифференциальная сканирующая калориметрия.*

1. INTRODUCTION

Currently, there is not a single application area where materials based on synthetic polymers are not used. Along with their obvious advantage over other materials related to the possibility of obtaining products with a wide range of operational properties, some practically decompose under the influence of environmental factors when released into nature. With the increase in the production and consumption of synthetic polymers, the urgency of the problem of extracting these materials from waste and developing methods for their recycling increases. One of the most effective ways to solve this problem is to create polymer composites, the polymer matrix of which is recycled thermoplastic polymers in the presence of fillers, giving the material the required performance properties.

To date, a significant number of studies are devoted to the development of polymer composites based on thermoplastic polymers in the presence of vegetable compounds, which makes the material biodegradable (Yonghui *et al.*, 2015; Väisänen *et al.*, 2017; Yang, 2017; Erdogan and Huner, 2018; Orlov *et al.*, 2003; Krechetov *et al.*, 2018; Dipen *et al.*, 2019; Formalev *et al.,* 2018; Tarrés *et al.*, 2019). The use of a thermoplastic polymer makes it possible to repeatedly process such material by traditional methods (extrusion, injection molding), and a vegetable compound provides a reduction in the cost and weight of finished products, imparting the required performance characteristics and environmental friendliness of plastic products (Kasakova *et al.,* 2018; Usenbekov *et al.,* 2014).

One of the promising vegetable compounds for creating polymer composites based on thermoplastic polymers is rice husk (Arjmandi *et al.*, 2015; Chen *et al.*, 2016; Kenechi *et al.*, 2016; Arjmandi *et al.*, 2017; Dasa *et al.*, 2019). Prospects for the use of rice husk to obtain polymer composites are due to its low water absorption (Chen *et al.*, 2016; Lomakin *et al.,* 2018) and increased thermal stability (Hidalgo-Salazar and Salinas, 2019) due to the high content of silicon oxide in the compound (Cardona-Uribe *et al.*, 2018). Due to the thermodynamic incompatibility of rice husks and polymer matrices, various compatibilizers are traditionally used to create polymer composites with the required physical and mechanical properties (Arjmandi *et al.*, 2015; Majeed *et al.*, 2017; Formalev *et al.,* 2016; Lomakin *et al.*, 2017; Raghu *et al.*, 2018; Orji and McDonald, 2020), which increases the cost of finished plastic products. The purpose of this work was to study the patterns of change in the physical and mechanical and thermophysical properties of polymer composites based on recycled polypropylene filled with rice husk in the absence of compatibilizers.

2. MATERIALS AND METHODS

Recycled polypropylene was used in the work as a polymeric matrix in the form of crushed material from substandard plastic products produced by injection molding. Rice husk was used to fill the recycled polypropylene (composition: cellulose 40-45%, lignin 20-25%, hemicellulose 15-17%, mineral substances 18-22%) with a particle diameter of not more than 0.4 mm. The ratio of parts by mass (phr) of the compound was calculated on 100 mass parts of the polymer (Binoj *et al.*, 2016; Danilova-Tretiak *et al.*, 2017). The test samples were obtained by injection molding on a Babyplast 6/10P injection-molding machine at a temperature in the zones of 225 °C, 235 °C, 220 °C, an injection pressure of 65 bar, an injection speed of 30%, and a clamping force of 35 bar. The temperature of the supply of cooling water to the mold was 12 °C, and the dwelling time was 10 s.

The physical and mechanical properties of the test samples at the break and cross breaking were determined according to GOST 11262-2017 and GOST 4648-2014, respectively, on a Shimadzu AGS-X universal testing machine at a temperature of 23 °C, a moving, gripping speed of 1 mm/min when determining the module of elasticity and 5 mm/min when determining other characteristics (GOST 11262-2017; GOST 4648-2014). Uniaxial tensile mechanical tests were performed on prototypes with dimensions according to GOST 11262-2017 (type 5). The tests were carried out on a universal testing machine AGS-X10kN (Shimadzu, Japan) at a temperature of 23±2 °C and a moving capture speed of 1 mm/min to determine the modulus of elasticity (GOST 9550-81) and a speed of 5 mm/min to determine other characteristics. Mechanical tests for static bending were performed on samples with sizes recommended according to GOST 4648-2014. The tests were carried out on a universal testing machine AGS-X10kN (Shimadzu, Japan) at a temperature of 23±2 °C and a traverse speed of 1 mm/min to determine the modulus of elasticity (GOST 9550-81) and a speed of 5 mm/min to determine other characteristics. Charpy V-notch impact energy and Izod impact strength (notched and unnotched) were determined according to GOST 4647-2015 and GOST 19019-2017, respectively, on a GT-7045-HMH pendulum (GOST 4647-2015; GOST 19019-2017). The Charpy impact toughness (with notch and without notch) was determined according to GOST 4647-2015 on the pendulum testing machine GT-7045-HMH (Taiwan, Gotech Testing Machines). The summary of test method in which a sample lying on two supports is subjected to a pendulum impact at a constant speed (when struck "flat" or "with edge"), the impact line being located in the middle between the supports and directly opposite the notch of the notched prototypes. The impact is applied to the surface of the prototype opposite the notch.

The Izod impact toughness of prototypes (with and without notches) was determined according to GOST 19109-2017 on the GT-7045-HMH pendulum testing machine (Taiwan, Gotech Testing Machines). The summary of test method consists in destruction of the cantilevered sample by the impact of the pendulum at a certain distance from the fixing point, in the case of a notched sample, from the midline of the notch. The bending temperature under load (1.8 MPa) and the Vicat softening temperature (50 N) were determined according to GOST 32657-2014 and GOST 15088-2014, respectively, on an HV-3000-P3 device. Bending temperature under load. The prototype is subjected to three-point bending under the action of a constant load from the side of the main (preferably) or lateral plane of the prototype to obtain bending stress. The temperature is raised at a uniform speed and its value is measured at which a standard deflection occurs, corresponding to the established increase in stress during bending.

The softening temperature of thermoplastics according to Vicat. The summary of the method is to determine the temperature at which a standard indenter with a flat bottom surface under the influence of load penetrates into the test prototype, heated at a constant speed, to a depth of 1 mm. Indenter is located perpendicularly to the surface of the prototype (GOST 32657-2014; GOST 15088-2014).

The methods of thermogravimetric analysis (TGA) and differential scanning calorimetry were used (DSC) in order to analyze the thermophysical parameters of polymer composites. Studies of polymer samples were carried out under the following conditions: TGA – temperature range 25-600 °C, dynamic mode – the heating rate of 5 deg/min, medium – air, TGA-DSC device; DSC – temperature range – 40-200 °C, dynamic mode – heating/cooling rate of 10 deg/min, medium – air, DSC-1 device. These methods allow to determine the following thermophysical indicators: T_c is the onset temperature; T_1, T_5 is the temperature corresponding to a decrease in the mass of the sample by 1 and 5%, respectively; T_{max} is the temperature corresponding to the maximum rate of mass loss at TGA; T_{ml}, T_{cr} is the melting and crystallization temperature; ΔH_{ml}, ΔH_{cr} is the thermal effect of melting and crystallization.

3. RESULTS AND DISCUSSION:

The results showed that the modulus of elasticity both at the break and cross breaking increases as the content of the vegetable compound in the polymer composite increases (Table 1). The modulus of elasticity at the break for a recycled polypropylene filled with rice husk in a volume of 30 phr increases by 1.36 times, and the modulus of elasticity in flexure – by 1.47 times in comparison with the initial polymer. In light of the fact that the modulus of elasticity correlates with the ability of a polymer sample to deform elastically under the influence of an external load, the filling of polypropylene with rice husks determines the possibility of obtaining more rigid plastic products (Das *et al.*, 2018; Rutkowska *et al.*, 2018).

It should be noted that plastic products made from polymer composites in the presence of a vegetable compound are more resistant to elastic deflections. This is confirmed by minor changes in the breaking elongation for polymers with varying degrees of filling of rice husk (Table 1). Filling recycled polypropylene with even a small amount of rice husk leads to a significant decrease in the tensile elasticity of the material. Addition 2 phr of rice husk reduces the strain at break from 588% for recycled polypropylene without compound to 26% for the composite (Table 1).

A further increase in the content of rice husk successively reduces the breaking elongation to 5% for a composite with 30 phr of the compound (Saigal and Pochanard, 2019; Stolin *et al.*, 2020). Compared to recycled polypropylene, a polymer composite has a lower tensile strength at break. With an increase in the content of rice husk up to 15 phr, the strength decreases almost linearly from 25.5 MPa to 21.3 MPa, and a further two-fold increase in the compound concentration determines a decrease in tensile strength by only 2.3% (Table 1).

Along with a decrease in the cross-breaking strength for filled composites, the resistance of plastics to dynamic impact worsens, which manifests itself in a corresponding change in impact value (Figure 1). Recycled polypropylene has a relatively high Charpy V-notch impact energy (rib impact) of 40.7 kJ/m². The Izod impact strength for this polymer is 26.1 kJ/m². Filling the polymer with rice husk leads to a decrease in impact strength according to both Charpy and Izod, and with a compound content of more than 5 phr, both of these indicators are almost identical (Bisht and Gope, 2015; Sutar *et al.*, 2018). Applying a notch simulating the presence of a mechanical defect in a plastic product significantly reduces the impact strength, according to both Charpy and Izod (Figure 1). For notched specimens, Izod impact strength is higher than Charpy V-notch impact energy for all considered polymer composites. Compared with recycled polypropylene, an increase in impact value is observed with the addition of 2 phr of rice husks, which can be used to obtain plastic products more resistant to external dynamic effects (Figure 1).

The use of rice husks can increase the heat-distortion temperature of plastic products based on recycled polypropylene (Figure 2). Introduction to the polymer 2 phr of compound leads to a slight decrease in bending temperature under load, the value of which with a further increase in the degree of filling of the composite increases from 57.4 °C for recycled polypropylene to 65.5 °C for a polymeric composition filled with 30 phr of rice husk. The Vicat softening temperature, in this case, rises from 75 °C for polypropylene to 84.6 °C for a composite with 30 phr of rice husk (Figure 2).

Rice husk used as a compound has a low value for the onset temperature T_H: a decrease in the mass of the product begins even at temperatures above 26 °C. A noticeable (by ~ 4%) decrease in the mass of the sample upon heating to 100 °C indicates the presence in the rice husk of a sufficiently large number of volatile components (probably moisture or solvent).

A further increase in temperature is accompanied by a gradual decrease in the mass of the product. The decomposition of rice husk on the differential thermal analysis (DTA) curve corresponds to two main temperature ranges (Table 2). At the stage of the temperature range of 170-358 °C with a maximum $T_{max,1}$ at 288°C, a 55% decrease in product mass is observed. At the stage of the temperature range of 358-470 °C with a maximum $T_{max,2}$=403°C, there is a decrease in the mass of the product by 23%, which, apparently, corresponds to the burnup of the resulting coke residue (Table 2).

It should be noted that the temperatures ($T_{max,1}$, and $T_{max,2}$) on the DTA curve, corresponding to the maximum decomposition rate of rice husk, are shifted to the lower values relative to similar parameters for recycled polypropylene (Table 2). The value of the residue (27.9%) after heating the rice husk to a temperature of 400 °C is noticeably larger compared to the same parameter for recycled polypropylene (Gowda *et al.*, 2018; Mohapatra, 2018). This indicates that the decomposition of rice husk, the main component of which is cellulose, in this temperature range occurs to a lesser extent (with a lower rate), compared with polypropylene. A relatively large amount of residue (17.2%) after heating the rice husk to 600 °C indicates the presence of thermally stable silicon oxide in the product (Cardona-Uribe *et al.*, 2018; Dorca *et al.*, 2019). Composites based on recycled polypropylene filled with various amounts of rice husk have rather close values of the onset temperature T_c 218-231°C (Table 2). It is noteworthy that the indicated T_c values are higher than that of rice husks and recycled polypropylene (Table 2).

An increase in the content of the vegetable compound from 2 to 30 phr does not lead to a significant change in the parameter T_c of the polymer composite, the values of parameters T_1 (242-252 °C) and T_5 (262-267 °C) are also in a rather narrow range (Table 2). It is seen that thermal-oxidative breakdown of polymer composites proceeds in two stages: the main decomposition (by 85-94%) occurs in the temperature range from T_c to 380-400°C; the temperature range of 400-600 °C corresponds to a decrease in the mass of the sample by ~ 3-7% (Table 2). With an increase in the content of vegetable compound in the polymer composite from 5 to 30%, the maximum temperature on the DTA curve shifts to a lower range: $T_{max,1}$ from 356 to 327 °C and $T_{max,2}$ from 421 to 417 °C. The observed changes are due to the influence of rice husk, whose decomposition corresponds to lower temperatures, compared with the decomposition of polypropylene (Gadzama *et al.*, 2020; Ghosh and Dwivedi, 2020). With an increase in the filler content in the compound from 2 to 30 phr the proportion of the product remaining after heating the sample to 400 °C (from 6.5 to 11.4%) and 600 °C (3.2% to 5.9%) increases, and the dependence of the residue on the compound content is linear (Nyior and Mgbeahuru, 2018; Pandey, 2020).

When conducting DSC analysis, it was found that filling the recycled polypropylene with rice husk in the range of 2-30 phr does not lead to a noticeable change in the melting (T_{ml}=166.2-166.7°C) and crystallization (T_{cr}=116.2-117.8°C) temperatures of composites whose values are close to the corresponding indicators of recycled polypropylene (Table 3). At the same time, the compound has a significant effect on the enthalpy (thermal effect) of the melting and crystallization of the polymer phase. The introduction of already a small amount (2 phr) of rice husk into the polymer leads to a noticeable (12 J/g) increase in the melting enthalpy of the composite compared to an unfilled polymer, and the crystallization enthalpy also increases, but to a lesser extent (Table 3). It should be noted that

ΔH_{ml} and ΔH_{cr} of composites containing 2-10 phr of rice husk is higher than that of the initial polypropylene. It can be assumed that the compound particles can play the role of crystallization nuclei, which contributes to an increase in the degree of crystallinity of the polymer phase and an increase in the heat of fusion and crystallization. With a further increase in the compound content in the polymer composite, the values of the enthalpy of melting and crystallization naturally decrease. When changing the content of rice husks in the composite from 2 to 30 phr the values of ΔH_{ml} and ΔH_{cr} decrease, respectively, by 1.47 and 1.36 times (Table 3), which, obviously, is associated with a decrease in the amount of the polymer phase in the composite.

4. CONCLUSIONS:

With an increase in the content of rice husk in a polymer composite based on recycled polypropylene, an increase in the modulus of elasticity in flexure occurs, and plastic products made from polymer composites with the compound are more resistant to flexural deflections. Filling polypropylene with rice husk slightly reduces the tensile strength at break and significantly reduces the elasticity of the polymer. Recycled polypropylene has a higher Charpy V-notch impact energy than Izod impact strength. Filling the polymer with rice husk leads to a decrease in impact strength according to both Charpy and Izod, and with a compound content of more than 5 phr, both of these indicators are almost identical. Applying a notch simulating the presence of a mechanical defect in a plastic product significantly reduces the impact strength, according to both Charpy and Izod.

For notched specimens, Izod impact strength is higher than Charpy V-notch impact energy for all considered polymer composites. Polymer composites filled with rice husks have a greater heat-distortion temperature, characterized by bending temperature under load and Vicat softening temperature. In this case, there is a slight increase in the onset temperature of the composites, which determines their thermal stability during processing. It was revealed that polymer composites containing 2-10 phr of rice husks are characterized by an increased degree of crystallinity of the polymer phase.

Compared with pure polypropylene, rice husk based composites have a higher modulus of elasticity at break and bending. Polymer composites are more resistant to high temperature, which is manifested in high values of bending temperature under load, Vicat softening and the beginning of decomposition. Filling polypropylene with rice husk leads to a decrease in strength and breaking elongation, decrase of Charpy and Izod impact toughness.

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Table 1. *Physical and mechanical properties of polymer composites*

Content of rice husk, phr	Modulus of elasticity at the break, MPa	Tensile strength at break, MPa	Breaking elongation, %	Modulus of elasticity in flexure, MPa	Relative flexural strain, %
0	1132.1	25.5	588.4	878.2	19.1
2	1154.2	24.7	26.4	981.0	19.2
5	1161.1	23.9	15.6	1019.4	19.9
10	1373.5	22.9	11.6	1104.1	20.4
15	1453.9	21.3	5.6	1228.2	20.1
30	1537.5	20.8	5.2	1288.9	18.8

Table 2. *The results of the thermogravimetric analysis of polymer composites*

Content of rice husk, phr	$T_{H.}$,°C	T_1, °C	T_5, °C	Residue, %		T_{max} , °C	
				at 400°C	at 600°C	1	2
0	211	243	262	5.4	2.4	349	443
2	226	252	267	6.5	3.2	332	467
5	218	242	262	6.4	2.8	356	421
10	229	250	266	8.5	4.3	329	445
15	231	249	266	10.7	5.5	326	417
30	231	247	265	11.4	5.9	327	417
100	172	193	208	27.9	17.2	288	403

Table 3. *Parameters of melting and crystallization of polymer composites*

Content of rice husk, phr	T_{ml}, °C	ΔH_{cr}, J/g	T_{cr}, ºC	ΔH_{cr}, J/g
0	166.0	-80.4	118.2	102.1
2	166.3	-92.6	117.2	104.1
5	166.2	-86.7	116.2	103.5
10	166.3	-84.5	116.3	100.7
15	166.2	-71.3	117.2	86.3
30	165.5	-63.0	117.8	76.3

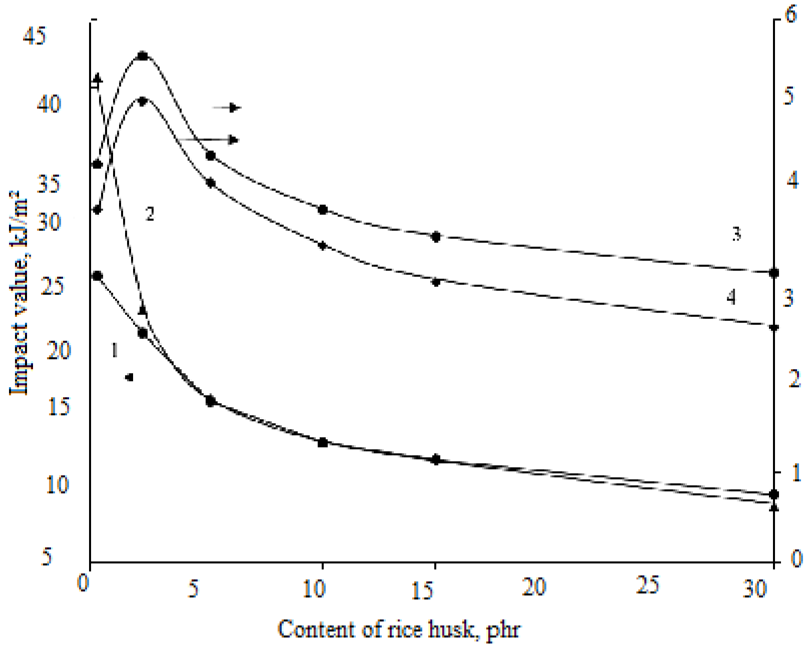

Figure 1. *Dependence of Izod impact strength (1.3) and Charpy V-notch impact energy (rib impact) (2.4) on the content of rice husk in a polymer composite. Unnotched samples (1.2), notched samples (3.4)*

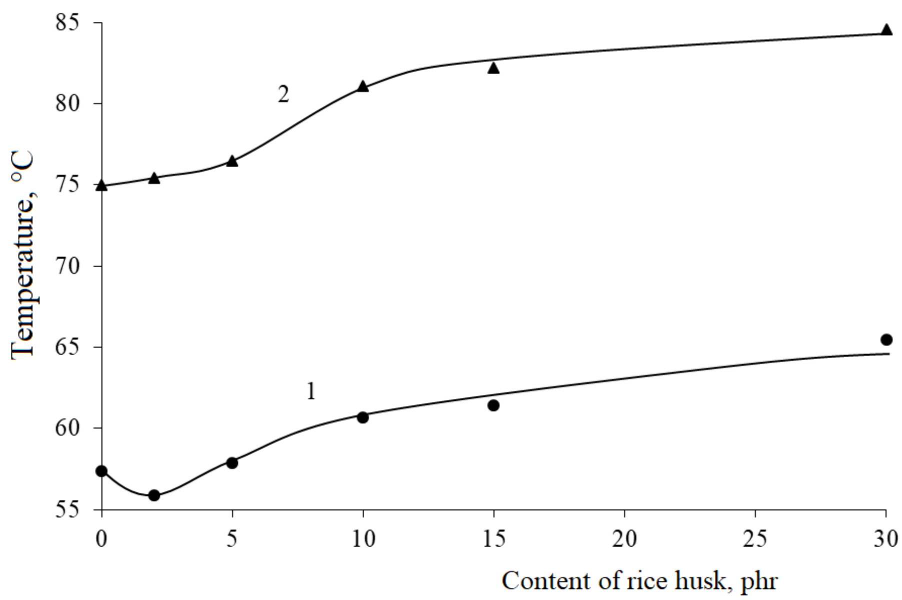

Figure 2. *Dependence of bending temperature under load (1.8 MPa) (1) and Vicat softening temperature (50 N) (2) on the content of rice husk in a polymer composite*