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STUDYING THE RHEOLOGICAL PROPERTIES OF A POLYLACTIDE MELT MIXED WITH WOOD FILLER

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Abstract. Composite materials based on wood filler are promising materials that are actively conquering the market. This is due to the advantages of using these materials in various fields: weather resistance and environmental compatibility, easy machining and possibility of recycling. Furthermore, it is sustainable use of wastes of timber sawing and furniture and woodworking industries, as well as low-grade wood. Wood powder is also known to be one of the components of consumables used in additive 3D printing technologies. Over the last decade, the commercial use of 3D printers has increased rapidly due to the fact that it allows creating prototype objects of complex shape based on a computer model. Experimental studies were carried out to determine the tensile strength and rheological properties of a composite made of polylactide 4043D, untreated wood powder brand 140 and wood powder thermally modified at 200 and 240 °C. The composite is intended for creation of three-dimensional objects by extrusion using a 3D printer. It was found that with an increase in the amount of filler in the composite, the tensile strength decreases. Also, samples with thermally modified filler show an increase in tensile strength in comparison with samples with untreated filler. Prototypes of 3D threads with different composition were obtained, during the study of which the melt flow index was examined. It was found that with increasing temperature of wood filler treatment the melt flow index increases. With a lower content of wood powder in the melt composition, there is a 2-fold increase in the melt flow index. The knowing of the rheological properties of the resulting compositions will allow achieving maximum performance and reduction of energy and production costs.

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Introduction

In recent decades, traditional hydrocarbon polymers have become the most commonly used materials in almost all areas of modern life owing to their high resistance to various external influences, low cost, reliability and a huge variety of properties. However, low degradability of polymers has become a real problem for such sectors as packaging, consumer goods, biomedicine, etc. For this reason, many countries have focused on products based on biodegradable materials like polylactic acid (PLA), polyhydroxybutyrate (PHB) and starch [10, 13, 15, 21].

Among biodegradable polymers, polylactide is the most widely used in the above areas. PLA is a partially crystalline biodegradable polymer with high mechanical properties, which is obtained from renewable sources, such as cane, corn and other plant materials. The decomposition time of PLA in natural environmental conditions is from 6 months to 2 years, as opposed to 500–1000 years for conventional synthetic polymers like polyethylene [1, 2, 7, 9].

PLA as a consumable is widely used in 3D printers that use Fused Deposition Modeling (FDM) technology. The thread is melted and fed through a special nozzle to the working surface of the 3D printer. As a result of building a 3D model with melted thermoplastics, a completely ready-to-use object that is not supposed to be used for a long time is created [3, 4].

However, due to the relatively high cost of equipment for the PLA production, the price for the biopolymer is sufficiently high. This is one of the reasons holding back the widespread introduction of polylactide as a replacement for conventional polymers. [14, 16].

The solution to this problem is the development and production of new composite mixtures and materials with the addition of filler, for instance, woodworking waste.

Due to the fact that global logging volumes have reached a value close to the growth rate of wood, the problem of maximum use of wood raw materials and above all processing wastes is also becoming more and more acute. Soft wastes in the form of sawdust and wood powder is processed in conditions of large-scale production with high energy intensity. Therefore, the development of inexpensive zero waste technology for wood waste recycling is an urgent issue [20].

Wood powder is a light organic filler of plant origin. As a material of local collection and production, wood powder is a cost-effective production waste. This is primarily due to the availability of sufficient amounts of both wood powder and wood waste of sawmilling and woodworking production. It is possible to obtain from them the necessary amount of powder with additional introduction of the grinding stage into the process. Its low cost and sufficiently high thixotropic properties are ideal for use as a filler for many materials [5, 8].

One of the ways to improve the properties of wood is its preliminary thermal treatment. This wood particle processing method involves the removal of hemicellulose from the material, which leads to the service life extension of products [12, 19].

The application of the proposed method of using wood powder will provide an environmental effect, which includes the release of areas previously used for its storage and the exclusion of the powder dispersion in the areas adjacent to the enterprise, both industrial and residential. The use of thermal modification of powder will give it such properties as durability, increased hardness and absolute environmental safety.

The research purpose is to create the samples of 3D threads and study the tensile strength of composites and the rheological properties of melts.

Research objects and methods

Studies on the tensile strength determination were carried out in order to identify the optimal quantitative ratio of composite material (CM) components. Tensile strength is one of the most important characteristics of CM, which determines its maximum tensile strength at critical loads [17].

The test method was performed in accordance with GOST R 56785–2015 "Polymer Composites. Test Method for Tensile Properties of Flat Specimens" and involved stretching the CM sample with a constant rate of loading or deformation until the moment of rupture.

Five batches of samples were prepared. Samples contained polylactide 4043D and wood filler brand 140, thermally modified at 200 °C and 240 °C, as well as untreated filler, which was dried at a temperature of 130 °C. The moisture content of wood powder was less than 1 %, since the formation of steam caused the formation of microbubbles and voids in the material [18].

Samples in the form of a rectangular cross-section strip 250 mm long, 15 mm wide and 2 mm thick were used for testing. The JLTTC LDS-5L breaking machine was used as a testing machine.

The main technological characteristic of polymers is the melt flow index (MFI). Initially, samples of 3D threads were made using the Rondol Microlab Twin Screw extruder.

The obtained samples of 3D threads (fig. 1) were used to evaluate the rheological properties of melts of thermoplastic polymer materials.

1 *2* *3*

Fig. 1. Samples of 3D threads: *1* – untreated filler (dried at 130 °C); *2* – filler thermally modified at 200 °C; *3* – filler thermally modified at 240 °C

The determination of MFI of thermoplastics was carried out in accordance with GOST 11645–73 "Plastics. Determination of Flow Index of Thermoplastics Melt by Extrusion Plastometer" using the extrusion plastometer GT 7100 MIB.

The cut threads were placed in the device preheated to 190 °C for 15 min. After holding the samples under a pressure of 5 kg/s for 5 min, the material was allowed to flow. To measure MFI, pieces of extruded material were selected, sequentially cut off every 30 s. After cooling, each obtained segment was weighed with an error of not more than 0.001 g. The number of segments was at least three. The mass of the segment was determined as the arithmetic average of the weighing results of all segments.

MFI was calculated for each parallel measurement using the following equation:

$$\mathrm{MFI}_{(T,\,P)} = \frac{tm}{\tau},$$

where T – test temperature, K; P – load, N; t – standard time, s; m – average mass of the extruded segments, g; τ – time interval between 2 cut-off segments, s.

Research results and discussion

According to fig. 2, it can be concluded that the tensile strength of the composites gradually decreases with an increase in the mass content of the wood filler (WF), which is explained by the weakening of the bonds between wood particles and binder. However, the tensile strength of the composite with thermally modified filler was higher than that of the samples with untreated filler. Thus, based on the results presented in fig. 2, it can be concluded that the loosening of composites occurs due to the formation of voids, which subsequently reduce the tensile strength, since cracks can easily spread through the areas containing inter-fiber voids [6, 11].

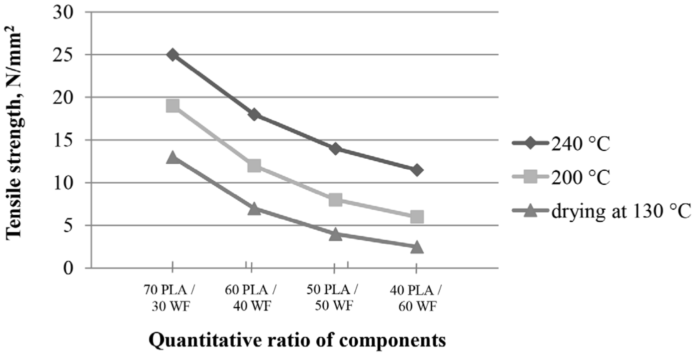

Fig. 2. Dependence of the tensile strength on the quantitative ratio of composite components

Therefore, the inclusion of wood filler in the composite by more than 50 % of the composite weight significantly reduces the tensile strength of the polymer matrix. However, to reduce the cost of the composite material, it is necessary to reduce the

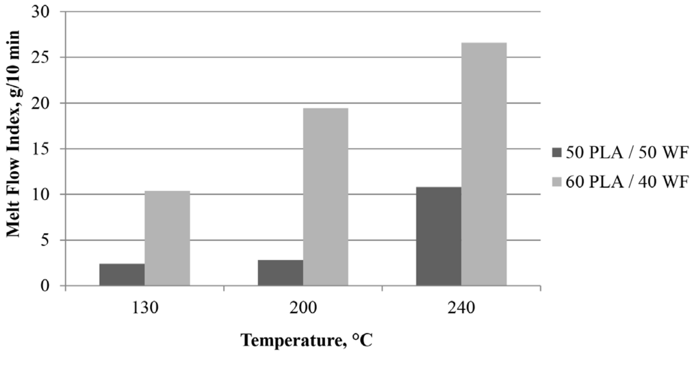

Fig. 3. Dependence of MFI on the temperature of the filler treatment

amount of polymer used. For this purpose, samples with 50 and 60 % PLA content were considered in further studies.

The results allow concluding that with an increase in the temperature of wood filler treatment, MFI increases (fig. 3). Moreover, the content of wood powder in the composition of the composite material affects the MFI value: an increase in MFI is observed with a decrease in the filler amount.

Conclusion

In the course of the study, we found the patterns presented below.

Tensile strength depends on the mass content of wood filler. With an increase in the amount of wood powder, the tensile strength of composites decreases. Since the preliminary thermal modification gives the wood such a property as increased strength, it was found that an increase in the tensile strength of the samples was observed with an increase in the temperature of the filler processing.

As the polymer content and the processing temperature of the wood filler increased, the melt flow rate increased. Thus, the extrusion rate of the melt from the extruder nozzle increased, which increased the productivity of the equipment.

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ИССЛЕДОВАНИЕ РЕОЛОГИЧЕСКИХ СВОЙСТВ РАСПЛАВА ПОЛИЛАКТИДА В СМЕСИ С ДРЕВЕСНЫМ НАПОЛНИТЕЛЕМ

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Аннотация. Композиционные материалы на основе древесного наполнителя, являясь перспективными, активно завоевывают рынок. Это связано с преимуществами применения данных материалов в различных областях: стойкость к атмосферным воздействиям и экологичность, легкость механической обработки и возможность утилизации отходов. Кроме того, это и рациональное использование низкосортной древесины, а также отходов лесопиления, мебельной и деревообрабатывающей промышленности. Известно применение древесной муки в качестве одного из компонентов в аддитивных технологиях 3D-печати. За последнее десятилетие коммерческое использование 3D-принтеров возросло благодаря тому, что они позволяют создавать объекты-прототипы сложной формы на основе компьютерной модели. Проведены экспериментальные исследования по определению предела прочности на разрыв и реологических свойств композита из полилактида 4043D и термически обработанной при температуре 200 и 240 °С и необработанной древесной муки марки 140, предназначенного для создания трехмерных объектов методом экструзии с помощью 3D-принтера. Установлено, что с увеличением количества наполнителя в композите уменьшается предел прочности при растяжении, а также что по сравнению с образцами из необработанного наполнителя у образцов из термически модифицированного наполнителя наблюдается возрастание предела прочности. Получены опытные образцы различных по составу 3D-нитей, для которых был исследован показатель текучести расплава. Показано, что с ростом температуры обработки древесного наполнителя этот показатель повышается. При меньшем содержании древесной муки в составе расплава отмечается его увеличение в 2 раза. Знание реологических свойств получаемых композиций позволит достигнуть максимальной производительности, снизить энергозатраты и себестоимость готовой продукции.
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