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INFLUENCE OF MODIFIED THERMOREACTIVE FILLERS ON THE PROPERTIES OF HONEYCOMB 3D MATRICES

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The influence of epoxy and polyurethane fillers on the physical and mechanical properties of combined polylactide composites was investigated. An adapted method for determining the elastoplastic properties of the combined composites was proposed, based on identifying load–deformation relationships while considering the material's elastic response. The calculations revealed changes in material characteristics, particularly hardness and elastic modulus, depending on the nature of the filler, the composition of its modifiers, and the plane of load application. It was found that 3D polylactide matrices filled with epoxy resin exhibit high impact strength, highlighting their ability to effectively absorb and dissipate impact energy. Epoxidized soybean oil was shown to enhance the composite's impact strength by increasing its flexibility and improving its capacity to withstand dynamic loads. The introduction of modified epoxy resin contributed to increased flexural and tensile strength, thereby enhancing the material's resistance to applied forces. At the same time, composites filled with polyurethane demonstrated higher tear strength compared to epoxy-based systems; however, the addition of modifiers led to a slight reduction in this property. Variations in material properties with and without heat treatment indicate the potential for flexible adjustment of the resin composition to achieve the desired mechanical performance in the final products. The results confirm the feasibility of using FDM technology for manufacturing modified composites.

Keywords: polylactide, 3D printing, starch, epoxidized soybean oil, modification, honeycomb structure.

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Introduction

An effective way to improve the mechanical strength of products made using additive manufacturing (3D printing) is to infiltrate printed structures with modified polymer resins based on thermosetting binders to form polymer materials. This technology significantly reduces the internal voids inherent in most 3D printing methods. This improves the mechanical properties of finished products by increasing tensile strength and impact toughness while reducing brittleness. At the same time, the key advantages of 3D printing are preserved: high design flexibility; the ability to manufacture complex geometries; and rapid production scaling [1–4]. The proposed method does not require complex equipment and involves a series of simple post-processing operations, making it suitable for widespread implementation.

Of the polymer matrices used in the manufacture of such products, polylactide (PLA) is of particular interest. It is a biodegradable and biocompatible polymer derived from renewable sources [5]. Some of

the most versatile thermosetting materials used in various industries, including construction, transport engineering and aviation, are polyurethane, epoxy and polyester resins [6–8].

Polyurethanes are characterized by their high mechanical strength, wide elasticity range, resistance to aggressive chemical environments and good dielectric properties. Polyurethane resins consist of two main components, isocyanates and polyols, which form a solid, durable structure when mixed together. This allows the final properties of the material to be controlled, ranging from elastomers to hard and soft foams, adhesives, sealants, and protective coatings [3].

In addition to polyurethanes, epoxy resins, which are characterized by high adhesion, rigidity, and thermal stability, are promising materials for modifying 3D-printed products [9,10]. Their ability to fill the porous structure of printed products effectively and form a strong thermosetting matrix significantly increases mechanical strength and wear resistance. Epoxies are characterized by low shrinkage during polymerization, resistance to chemical reagents, and compatibility with functional fillers, expanding their application possibilities in composite systems. Due to these properties, epoxy resins are an effective alternative to or addition for polyurethanes in post-processing and strengthening technologies for additive products.

Despite their significant operational advantages, epoxy and polyurethane composites have several limitations. These include insufficient plasticity, susceptibility to internal stress accumulation, limited impact resistance and reduced chemical resistance of adhesive joints in aggressive environments. One of the key issues is their relatively low heat resistance and limited thermal stability, which significantly restricts their use at elevated temperatures [11].

In this regard, methods of physical and chemical modification are becoming increasingly relevant. In particular, the introduction of functional fillers, high-molecular compounds and plasticizers allows the operational characteristics of the material to be regulated [12]. Taking into account environmental friendliness, bioavailability, and the capacity to enhance mechanical and thermal properties, starch and epoxidized soybean oil (ESO) are ideal candidates for use as modifying additives.

Experimental

To produce the composite materials, a two-component polyurethane resin (Axson F180, Axson Technologies, France) consisting of a white polyol and a yellow isocyanate, as well as an ED-20 epoxy resin (Ukrainian state standard DSTU 10587-87), were used. To cure the epoxy resin, a low-molecular-weight polyethylene polyamine (PEP) hardener (Ukrainian standard TU 6-01-33069) was selected.

To create the 3D matrices, 1.75 mm diameter polylactide filament from Monofilament (Ukraine) was used. Printing was performed on a Prusa i3 UA 3D printer. The digital model was sliced using PrusaSlicer software. The samples were formed as rectangular bars with 50% infill and a «triangle» geometry, with a wall thickness of 0.3 mm (Fig. 1).

Potato starch (PBP «VIMAL», Ukraine) and epoxidized soybean oil (AKESBO, Turkey) were used to modify polyurethane compositions.

Resin-based compositions were prepared in containers according to the established resin component ratio, after which the mixture was stirred for a further five minutes. The resulting composition was degassed in a vacuum before being introduced into the cells of the 3D-printed matrix via a dispenser. These compositions were then cured at room temperature until the samples were no longer sticky. Heat treatment was then carried out for two hours at 353 K.

The hardness of the samples was determined using the Brinell method in accordance with ISO 410:1982. This method involves pressing a steel ball into the surface of the sample at a perpendicular angle under a load for 30 seconds. After the load is removed, the diameter of the indentation is measured.

The impact strength of the samples was evaluated in accordance with the requirements of ISO 179-1, «Plastics – Determination of Charpy impact properties – Part 1: Non-instrumented impact test», using an impact load of 50 kgf.

The tensile strength and relative elongation at break, as well as the other physical and mechanical properties, of polylactide composites were determined

Fig. 1. 3D matrix obtained by FDM printing with thermosetting filler

in accordance with ISO 527-1 using a TIRA Test 2200 tensile testing machine.

The load-deformation curve was constructed using a Hepler consistometer, with the load on the sample gradually increased to create the curve. A conical indenter was used for the study and the maximum applied load was 300 N. Once the maximum load value had been reached, the device was gradually unloaded to construct the unloading curve. The depth of indentation was recorded using a calibrated scale installed on the device.

Results and discussion

Hardness is one of the key physical and mechanical characteristics of polymer materials. It determines their ability to withstand local mechanical stress, abrasion, and surface damage. For combined polymer composites, particularly those based on polylactide, epoxy and polyurethane resins, this indicator is important for ensuring operational reliability, durability, and the preservation of functional properties. It should be noted that the structure and properties of the surface layer of polymers can differ significantly from those of the internal volumes. This is due to the molecular structure, the thermodynamic conditions of formation, the duration of processing and the composition of the system's components. Insufficient surface hardness can result in the degradation of a material's protective and aesthetic properties during operation, while excessive hardness often leads to internal stresses and brittleness. The optimal combination of surface hardness, elasticity, impact resistance, and the ability to bond structural components together is a criterion for the durability and reliability of polymer composites.

In the process of 3D printing, it is known that products with different surface roughness are obtained depending on the printing orientation. In particular, the base plane that contacts the substrate has a smoother surface compared to the other planes, while the outer contour of the side exhibits the most pronounced «step effect» (layering effect).

Table 1 shows the hardness and impact strength values of composite products based on 3D-printed polylactide and polyurethane matrices, depending on the type and amount of modifier used in the composite filling matrix.

Adding starch significantly increases Brinell hardness. This is because the starch particles distribute the applied load more effectively, thereby increasing the material's resistance to indentation and deformation. The increased hardness indicates that starch contributes to creating a more rigid, strengthened epoxy matrix structure. Conversely, adding ESO to starch-modified epoxy resin leads to a noticeable decrease in hardness. ESO acts as a plasticizer, disrupting the density of polymer chains and increasing their mobility and flexibility.

Heat treatment further increases the Brinell hardness of all samples, regardless of their composition. This increase is probably due to the polymer matrix becoming more structured under the influence of temperature. Maintaining the temperature promotes better adhesion and bonding between the resin and fillers. Consequently, the material becomes less susceptible to deformation under load. Additionally, heat treatment can eliminate residual stresses and voids in the composite structure, creating a more homogeneous material.

The introduction of modifiers, particularly starch and ESO, leads to a noticeable decrease in Brinell hardness for polyurethane resin-based composites. This is primarily due to the formation of a transition layer between the polyurethane matrix and the starch particles. The presence of starch disrupts the polymer network's homogeneity, thereby weakening the composite structure and making it more susceptible to local deformation under load.

The impact strength of polymers and composites determines their ability to absorb energy during impact loading. This property is important for predicting how a material will behave under sudden dynamic loads, such as impacts or falls. In the case of the developed

Table 1

Properties of poly(lactic acid) (PLA) composites based on epoxy and polyurethane resins

Composition, wt.%				Properties	
ED	PU	ESO	starch	Hardness according to Brinnell, HB	Impact strength, kJ/m^2
100	–	–	–	114.5	7.3/5.4*
90	–	–	10	181.8	6.0/7.7*
80	–	10	10	122.5	9.8/5.2*
–	100	–	–	93.0	5.8
–	90	–	10	81.5	4.7
–	80	10	10	77.3	4.0

Note: * – heat-treated composites.

composites, impact strength reflects their resistance to crack initiation and propagation under dynamic mechanical forces. The impact strength values are given in Table 1.

The high impact strength of PLA 3D matrices filled with epoxy resin demonstrates their effectiveness in absorbing and dissipating impact energy. This makes them less susceptible to damage under such conditions.

Adding starch decreases the impact strength compared to pure epoxy resin. This indicates an increase in the brittleness of the composite, which can be attributed to the reduced compatibility of its components. Additionally, the decrease in impact strength may be attributed to the suboptimal interfacial adhesion between the starch and the epoxy resin. As starch is hydrophilic, it may struggle to bind with the more hydrophobic epoxy resin, resulting in cracks forming and spreading more easily under dynamic loading [13].

It has been found that ESO improves the impact strength of the composite. ESO apparently acts as a plasticizer, increasing the material's flexibility and its ability to absorb and dissipate energy under impact loads [14]. The addition of ESO probably neutralizes the brittleness caused by the starch content, ensuring a more even distribution of stresses and thus increasing the composite's strength.

It has been established that the effect of additional heat treatment varies depending on the composition of the samples. Heat treatment resulted in a decrease in impact strength for unmodified epoxy resin and a composite containing both starch and ESO, probably due to an increase in PLA crystallinity or changes in the polymer network, reducing the composite's ability to absorb impact energy. However, for a composite containing 10% starch by mass, heat treatment had the opposite effect, increasing the impact strength. This can be explained by improved structuring and increased adhesion between the starch and the resin after heating.

Variations in impact strength in different resin compositions, both with and without heat treatment, demonstrate the potential to adapt the composition to achieve the desired mechanical properties of finished products. Adjusting the ratio of additives, such as starch and ESO, and applying additional heat treatment ensures the composite's impact resistance meets the requirements of its application.

At the same time, the initial polyurethane resin samples demonstrate higher impact strength than other polyurethane composites, although this is still lower than epoxy resin-based composites. Adding starch and ESO reduces strength further, probably due to complex interactions between the components of the polyurethane resin, modifier and plasticizer system.

In order to determine the potential applications of the composite materials developed, it is important to evaluate their properties under various load conditions, particularly during bending and stretching.

Figure 2 shows the results of a study investigating the impact of modified epoxy and polyurethane resins on the bending strength and deformation of polylactic acid (PLA) matrices during bending.

An unfilled polyactide matrix has the lowest flexural strength value. Introducing epoxy resin increases flexural strength, improving the material's ability to withstand applied forces. Additionally, this modification significantly increases the deformation of the sample, indicating the material's enhanced ability to bend under load and improving its overall flexibility and strength.

Further modification of epoxy resin using starch and ESO increases its flexural strength. This can be attributed to the strengthening effect of the starch

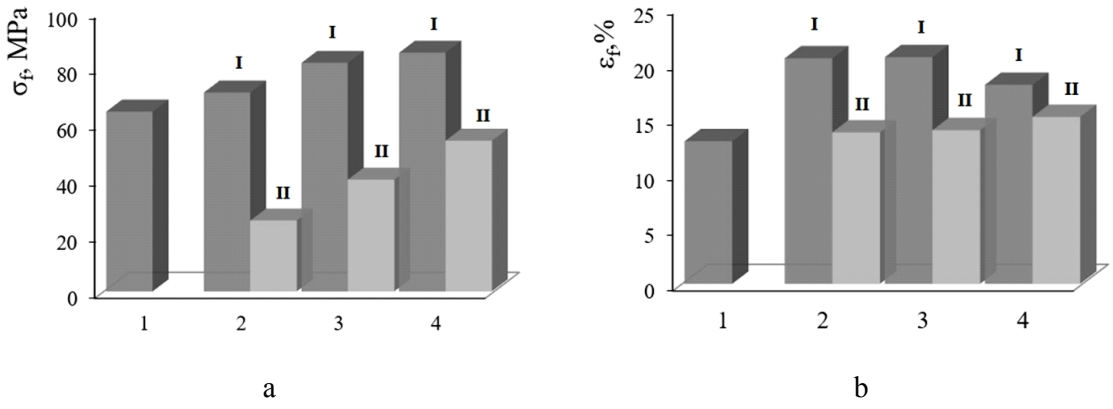

Fig. 2. Flexural strength (a) and flexural strain (b) of composite materials. Composition of composites, wt.%: 1 – PLA matrix without filler; 2 – ED/PU=100; 3 – ED/PU:starch=90:10; 4 – ED/PU:starch:ESO=80:10:1. Resin: I – epoxy; II – polyurethane

particles, which contribute to a more rigid composite structure. The presence of ESO also affects the material's mechanical properties, acting as a plasticizer. It contributes to the composite's greater flexibility and plasticity, enabling it to absorb and dissipate energy effectively during deformation. The increased deformation values observed with the addition of ESO suggest that the material becomes more elastic.

Table 2 shows the effect of modified epoxy and polyurethane resins on tensile strength and relative elongation.

A significant increase in flexural strength is observed in 3D-printed PLA matrices filled with unmodified epoxy resin after additional heat treatment. The curing process is accelerated by elevated temperatures during heat treatment, promoting the formation of a more robust internal structure.

Similar patterns were observed when the tensile strength of samples was tested. After heat treatment, tensile strength increased by 200%. However, the addition of starch alone had no effect on the tensile strength values. This suggests that, while starch improves resistance to deformation during bending by strengthening the composite, its structural characteristics limit its ability to improve tensile properties.

Adding starch and ESO decreases the tensile strength to 11 MPa, with ESO's plasticizing effect having a predominant influence on this indicator. While ESO increases the flexibility of the composite, it also reduces its tensile strength by destroying the rigid structure formed by the epoxy resin and starch. Relative elongation at tensile strength is lowest among all samples, indicating that adding ESO makes the material more elastic and less plastic. This reduction in plasticity is due to the combination of starch and ESO having a reinforcing effect, which limits the material's ability to deform under tensile load. Meanwhile, the separated ESO phases contribute to increased brittleness.

At the same time, the bending strength of composites with polyurethane resin is significantly lower than that of epoxy composites, which increases by 100% with the addition of modifiers. Meanwhile, tensile strength is slightly higher than in epoxy systems, though it decreases slightly after the addition of modifiers.

As with hardness measurements taken using the load-deformation curve method, measurements were taken for the filling planes of the samples under investigation.

Figure 3 shows the load-deformation curves of a PLA composite made from epoxy and polyurethane resins.

It has been established that the load-deformation curves obtained for the developed composites are characteristic of elastic-plastic materials and differ in slope. The introduction of 10% starch by mass had practically no effect on the deformation values (Fig. 3, curves 2 and 3), regardless of the resin type. However, a slight decrease in deformation values was observed for epoxy resin samples that had been heat-treated. Epoxy resin-based materials have the highest resistance to deformation when subjected to additional heat treatment, regardless of the nature of the additives, which is explained by the additional structuring of the epoxy resin at elevated temperatures. An increase in load leads to a change in the slope of the curve, the value of which depends on the modifier content. At the same time, the plasticizing effect of EPO is clearly visible for all materials (Fig. 3, curve 4). Clearly, EPO in such systems can reduce the total number of cross-links and act as an interphase plasticizer, which decreases the material's ability to resist deformation. Polyurethane composites also have significantly lower deformation resistance values than epoxy composites. Nevertheless, the slope of the unloading curve is similar for all samples, indicating insignificant elastic deformation in the developed composites.

Conclusions

The influence of modified thermosetting fillers (epoxy and polyurethane resins) on the properties of polylactide 3D matrices was revealed based on the experimental data obtained.

Table 2

Physical and mechanical properties of composite materials

Composition, wt.%				Properties	
ED	PU	ESO	Starch	Tensile strength, σ_t, MPa	Relative elongation, ε_{re}, %
100	–	–	–	16.1	7.9
90	–	–	10	16.0	6.1
80	–	10	10	11.1	5.0
–	100	–	–	21.6	4.7
–	90	–	10	19.7	4.4
–	80	10	10	16.1	4.8

It was found that the introduction of starch increases the Brinell hardness of composite products based on 3D-printed matrices. The addition of ESO to starch-modified epoxy resin results in a noticeable decrease in hardness, attributed to the plasticizing effect of epoxidized soybean oil. Heat treatment further enhances the Brinell hardness of all samples, regardless of their composition.

PLA-based 3D-printed matrices filled with epoxy resin demonstrated high impact strength. The incorporation of starch led to a reduction in impact strength compared to the pure epoxy resin, while the addition of ESO slightly improved it.

For PLA matrices filled with unmodified epoxy resin, a significant increase in flexural strength was observed after additional heat treatment. Modified epoxy resin also contributed to improvements in both flexural and tensile strength, enhancing the material's resistance to applied forces. Based on the analysis of load-strain patterns, the plasticizing effect of ESO was evident across all combined PLA composites.

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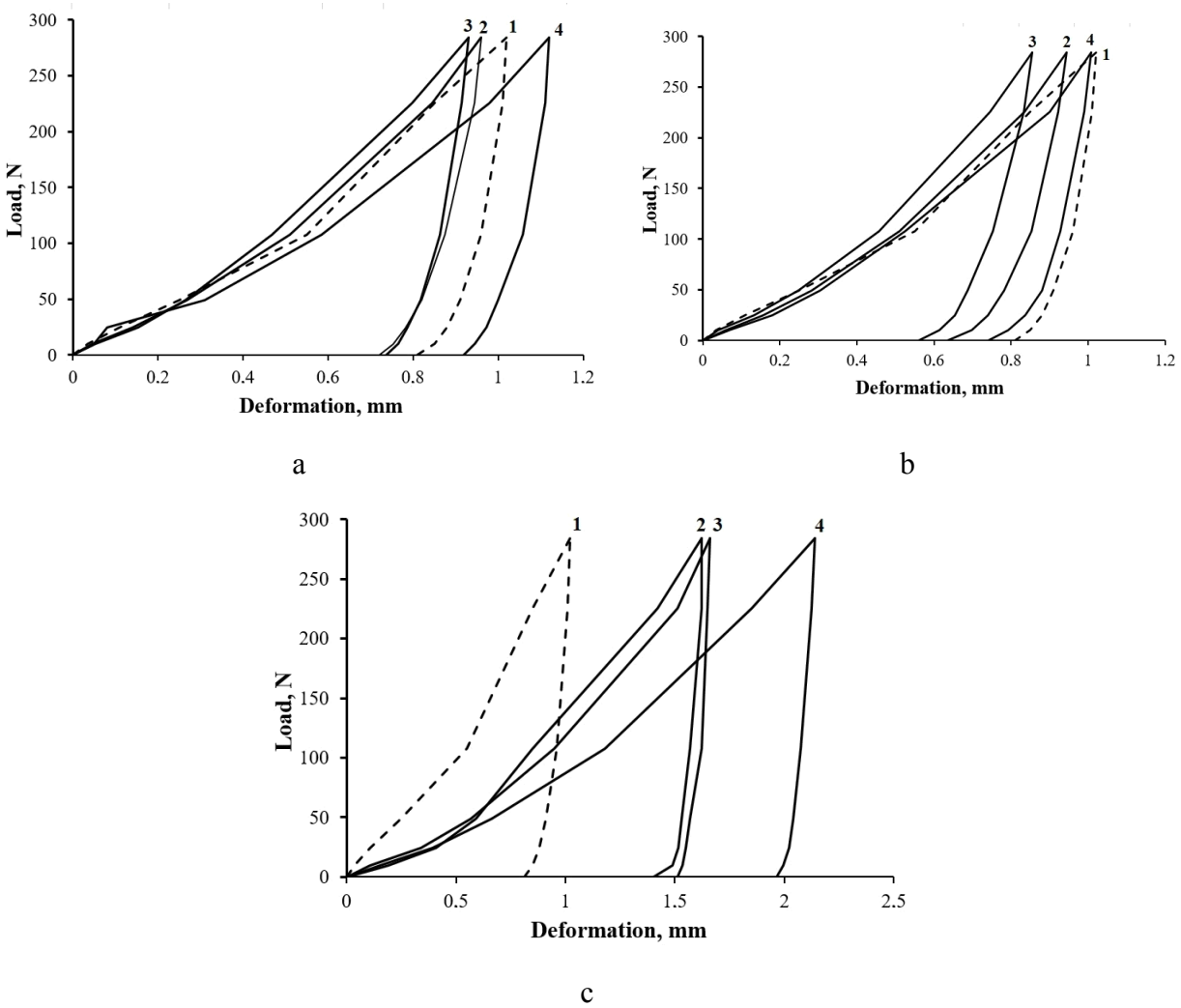

Fig. 3. Load-deformation curves for the filling plane for composite materials: a – epoxy resin without heat treatment; b – epoxy resin with heat treatment; c – polyurethane resin. Composite composition, wt.%:
1 – PLA matrix without filler; 2 – ED/PU=100; 3 – ED/PU:starch=90:10; 4 – ED/PU:starch:ESO=80:10:1

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ВПЛИВ МОДИФІКОВАНИХ ТЕРМОРЕАКТИВНИХ ЗАПОВНЮВАЧІВ НА ВЛАСТИВОСТІ СТІЛЬНИКОВИХ 3Д МАТРИЦЬ

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Досліджено вплив епоксидного та поліуретанового заповнювача на фізико-механічні властивості комбінованих полілактидних композитів. Запропоновано адаптований метод визначення пружно-пластичних властивостей комбінованих композитів, що базується на встановленні закономірностей навантаження-деформації з урахуванням пружної реакції матеріалу. На підставі проведеного розрахунку відзначено зміну характеристик матеріалу, зокрема твердості та модуля пружності, залежно від природи заповнювача, вмісту модифікаторів та площини прикладання навантаження. Встановлено, що полілактиді 3Д матриці, заповнені епоксидною смолою відзначаються високою ударною в'язкістю, що підкреслює їх здатність ефективно поглинати та розсіювати енергію удару. Виявлено, що епоксидована соєва олива покращує ударну міцність композиту, підвищуючи його еластичність і здатність протидіяти динамічним навантаженням. Введення модифікованої епоксидної смоли сприяє підвищенню міцності на згин і розтяг підвищуючи здатність матеріалу протистояти прикладеним зусиллям. Водночас композити з поліуретановими заповнювачами мають вищу міцність під час розривання у порівнянні з епоксидними системами, однак введення модифікаторів частково знижує цей показник. Варіації у властивостях матеріалів, зокрема з та без термічної обробки, свідчать про можливість гнучкого регулювання складу смоли для досягнення необхідних механічних характеристик готових виробів. Отримані результати підтверджують доцільність використання технології FDM у виробництві модифікованих композитів.

Ключові слова: полілактид, 3Д друк, крохмаль, епоксидована соєва олива, модифікування, стільникова структура**.**

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Keywords: polylactide; 3D printing; starch; epoxidized soybean oil; modification; honeycomb structure.

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