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COMBINED COMPOSITES BASED ON POLYLACTIDE 3D MATRICES AND MODIFIED EPOXY RESINS

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Abstract. Materials based on polylactide 3D matrices with their subsequent filling with modified epoxy resin were developed. Their elastoplastic, deformation, and strength characteristics were investigated. It was found that the filling plane of the studied products significantly affects the values of deformation and hardness. The introduction of epoxidized soybean oil into the composition improves the impact strength of the composites, increasing the flexibility of the material and increasing its ability to absorb and dissipate energy under impact loads. The combined composites are characterised by increased flexural strength, tensile strength, and flexural strain.

Keywords: epoxy resin, 3D printing, starch, epoxidized soybean oil, combined composites.

1. Introduction

Additive technology, also known as 3D printing, is an innovative manufacturing approach that has significantly changed traditional methods of manufacturing polymer products.[1-3] However, the mechanical characteristics of 3D printed product components are insufficient for practical applications due to their inherent anisotropy of properties, porosity, and relatively low strength. To overcome these shortcomings, a new method of strengthening 3D printed structures by filling their internal structure with reactive oligomers has been proposed.[4,5] Thus, the formed internal cavities inside the printed products are used as a mold to fill them with casting compounds based on reactive binders. After curing, the binder bonds with the printed substrate to form a composite structure characterized by increased stiffness, heat resistance, and mechanical strength.

This approach to manufacturing takes advantage of additive manufacturing by using the internal structure of the 3D printed component as a framework, which is formed by optimised settings of 3D printing parameters such as pattern and fill density, line thickness. Such hybrid manufacturing technologies have significant potential for products that require lightweight yet strong parts, including aerospace, automotive, and construction industries.[6-8]

At the same time, among the methods of improving the performance properties of reactive binders, in particular those based on epoxy resins, the method of modifying them with additives of various nature is considered promising. Considerable attention is paid to the development of structural composite materials containing natural fillers and plasticisers. This trend is driven by the abundance and renewability of natural resources, the low cost of raw materials, and the high environmental friendliness of natural components.[9,10] Among such applications, native starch and epoxied soybean oil are becoming increasingly common. Starch, as a natural polymer of plant origin, can be used as an effective filler due to its availability, environmental friendliness, and widespread use.[11] At the same time, due to its low cost, renewability, and biodegradability, starch can improve the stiffness and modify the structure of polylactide composites, as well as regulate the biodegradability and hydrolysis ability of polylactide. To increase the processability of composites during processing, in particular by 3D printing, and to regulate the properties of composites based on polylactide matrices and modified epoxy resins, it is advisable to introduce epoxidized soybean oil, which acts as both a plasticizer (reducing the yield point, increasing elasticity) and a compatibilizer (increasing process compatibility, forming more homogeneous structures, affecting intermolecular interactions between polylactide and starch macromolecules) in combined composites.

2. Experimental

2.1. Materials

A polylactide (PLA) filament with a diameter of 1.75 mm was used to form 3D matrices. The samples were formed in the form of rectangular bars using a 50% filling with a triangular geometry on a "Prusa i3 UA" 3D printer, with a wall thickness of 0.5 mm.

Epoxy resin (ER) of the ED-20 grade (DSTU 10587-87, CAS 25068-38-6) was used for the study. For its curing, a low-molecular-weight polyethylene polyamine (PEPA) hardener was chosen (TU 6-05-241-202-78).

To modify the epoxy resin, native potato starch ("VYMAL", Ukraine) and epoxied soybean oil (ESO) (AKESBO, Turkey) were used.

The choice of the component composition of epoxy resin-based compounds (starch content 10% by weight and ESO content 10% by weight) was based on preliminary experimental screening, which showed the effectiveness of the additives in the selected range. These concentrations allow avoiding interfacial separation of the system and ensuring effective curing of the epoxy resin.

2.2. Methods

Polymer compounds based on epoxy resins were formed by adding starch and ESO to the epoxy resin in an amount of 10% by weight, followed by thorough mixing for 5 min with an overhead mixer of the OS-60 type. The next step was to add PEPA hardener to the mixture in the appropriate ratio and mix for 10 min. The resulting composition was subjected to degassing in a vacuum and injected into the cells of the 3D molded polylactide-based product using a dispenser. The compositions thus obtained were cured at room temperature until the characteristic stickiness of the samples disappeared. The composites were heat-treated for 2 h at 353 K.

The Brinell hardness of the test samples was determined following ISO 410-82, which is based on pressing a steel ball into the sample under a force applied perpendicular to the sample surface for 30 seconds and measuring the diameter of the imprint after the load was removed.

The study of physical and mechanical characteristics was carried out based on load-strain curves.[12-13] The load-strain curves were obtained on a Hepler consistometer by gradually increasing the load on the sample and recording the corresponding strain. The maximum load applied was 300 N. A steel conical indenter with a sharpening angle of 58^0 08' was used for the study. After reaching the maximum load value, the device was gradually unloaded to obtain the unloading curve.

The impact strength of polylactide materials was determined following ISO 179-1:2010.

The surface hardness was also determined by the conical yield point method using a Hepler tester, using a steel conical indenter with a pointed angle at the apex of 58^0 08' under a load of 5.0 kg. The test duration was 60 seconds.

The physical and mechanical characteristics of polylactide composites, including tensile strength and relative elongation at break, were determined in accordance with ISO 527-1, using a TIRA Test 2200 tensile tester. Flexural strength and strain were determined in accordance with DSTU EN ISO 178:2019.

3. Results and Discussion

The developed method of producing combined composites based on polylactide 3D printed matrices and modified binders allows to obtain the advantages of both classical 3D printing and reactoplastic polymeric materials. This method involves reducing the printing time and the amount of material used to make the product. This method will also simplify the introduction of special-purpose fillers (conductive, magnetically sensitive) and modifiers into the composite without the need to create a filled filament for 3D printing.

To evaluate the physical and mechanical properties of the developed composites, a comprehensive study was conducted to determine the elastic-plastic, deformation, and strength characteristics. These properties were evaluated by constructing a load (P) - strain (h) curve. This method is an important tool for studying the mechanical properties of polymeric materials, especially polymer composites. This method determines the hardness and elastic modulus, which are obtained directly from the load-strain curve, taking into account the elastic deformation of the surface after the load is removed. The load-strain curve allows to take into account the highly elastic properties of materials.[14]

Fig. 1 shows a general view of a loading-unloading curve containing loading and unloading data for a typical viscoelastic-plastic material, such as polymers.

The data obtained from the unloading curve provides information on the elastic, viscoelastic, and plastic characteristics of the material. The value h_{max} represents the maximum strain corresponding to the maximum applied load P_{max} during compression, and h_p is the residual strain after the load is removed. The value h_c is the point of intersection of the tangent line drawn from the first part of the unloading curve, which describes the effects of elastic deformation, and the strain axis.

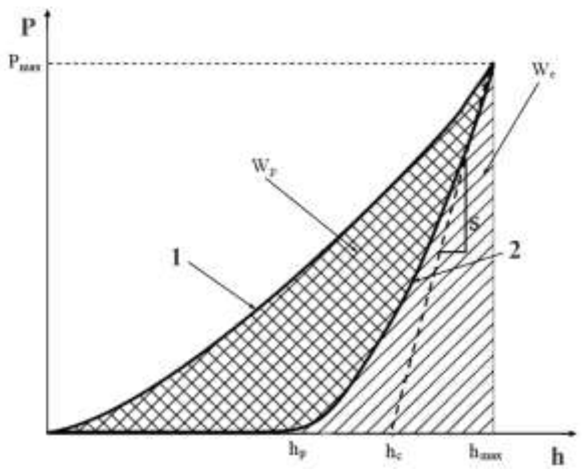


Fig. 1. General view of the loading-unloading curve for a highly elastic polymer material: 1 – loading; 2 – unloading

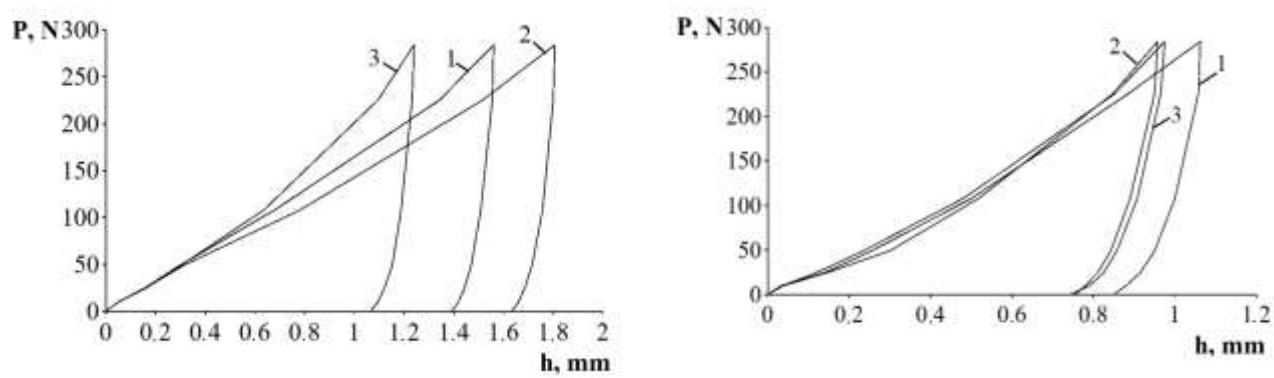
The slope of this line represents the estimated contact stiffness S at maximum deformation, and h_c is considered to be the actual value of the material deformation, which occurs mainly plastically. The value of work W_p corresponds to the area between the loading and unloading curves and determines the energy used for plastic deformation of the material, and W_e determines the elastic deformation energy.

The study of hardness determination using load-strain curves for combined PLA materials with epoxy resin composites was carried out (Fig. 2).

The research was conducted for three planes of the PLA die, which also allows us to assess the hardness characteristics of the material depending on the 3D printing surface. During 3D printing, depending on the orientation, products with different surface roughnesses are formed. In particular, the base plane of 3D printed products has a lower roughness compared to other planes; the 'step effect' (layering effect) is most pronounced for the outer contour plane.

It was found that the obtained load-strain curves are typical for viscoelastic materials and differ in the slope of the load curve. Since the measurements were carried out on the plane of the outer contour of the PLA matrix, an initial load of up to 50 N was applied to press the indenter directly into the printed polylactide material. The curves show the same slope for the samples filled with both modified and unmodified epoxy. With an increase in load above 50 N, a change in the slope of the curve is observed depending on the composition of the binder. The addition of 10% (w/w) starch to the epoxy resin significantly increases the deformation compared to the unmodified epoxy resin. At the same time, the addition of 10 wt.% of ESO reduces the strain at the same load values. The slope of the unloading curve for all samples indicates a slight elastic deformation.

The additional heat treatment significantly reduces the deformation for all samples. This is explained by a number of factors: additional structuring of the epoxy resin under the influence of temperature, physicochemical interactions between the matrix and the components of the casting compound. At the same time, the load-strain curves for the modified samples showed almost the same strain values, which were lower than those of the unmodified epoxy. The change in the slope of the unloading curve for the heat-treated samples compared to the untreated ones indicates an increase in elastic strain.



a b

Fig. 2. Load-strain curves for the outer contour of combined epoxy resin composites: a) no heat treatment, b) with heat treatment. Composition of the composites: 1– epoxy resin; 2 – ER : starch 90 : 10 wt.%; 3 – ER : starch : ESO – 80 : 10 : 10 wt.%

Based on the data obtained, the values of the strain parameters were calculated (Table 1): where h_{max} is the maximum strain, h_p is the residual strain after load removal, E is the elastic modulus, Ha is the hardness, ψ is the plasticity index, W_e is the recovered elastic strain energy, and W_p is the dissipated strain energy.

There is a significant difference in the strain and hardness values depending on the studied plane of the product. The filling plane of the specimens was used to pour epoxy thermosetting oligomers, so the place of contact between the indenter and the composite surface plays a significant role.[15] The highest hardness values are observed for the filling plane, since the modified epoxy resin has the largest area of direct contact with the indenter. This is obviously due to the significant difference in hardness between the epoxy and the polylactide 3D printed matrix.

Table 1. Calculated values of the parameters of the load-strain curve for the developed combined composites

No.	Parameter	Composition		
		Epoxy resin	ER : starch 90:10(wt.)	ER : starch : ESO 80:10:10 (wt.)
1	Maximum deformation h_{max}, mm	0.96*/1.56*	0.93 / 1.81	1.12 / 1.24
2	Elastic deformation h_p, mm	0.72 / 1.39	0.74 / 1.63	0.92 / 1.07
3	Modulus of elasticity E, MPa	591.14 / 521.53	611.61 / 448.42	735.22 / 661.36
4	Hardness H_a, GPa	14.63 / 5.06	15.61 / 3.74	10.02 / 8.14
5	Plasticity index, ψ	0.80 / 0.93	0.84 / 0.94	0.87 / 0.91
6	Restored elastic strain energy W_e, kJ	21.94 / 13.61	17.72 / 13.85	17.41 / 13.92
7	Dissipated strain energy W_p, kJ	88.72 / 189.49	93.0 / 227.19	118.8 / 133.1

* – plane on the filling side; ** – outer contour plane.

It was also found that the introduction of starch into the epoxy resin leads to a significant increase in deformation and a concomitant decrease in hardness, both on the outer contour and at the base of the composite structure. At the same time, the addition of ESO cancels out the negative effect of starch, which is probably due to the increased adhesion between the PLA matrix, epoxy resin, and ESO. In particular, it is possible to form a transition layer of PLA-ESO-epoxy compound with the migration of ESO molecules into the PLA matrix and the formation of a strengthened combined structure.[16,17] By calculating the value of the consolidated modulus based on Poisson's ratio, it is possible to calculate the elastic modulus, which is an important characteristic that determines how resistant the material is to deformation. It was found that the introduction of starch into the epoxy resin leads to a noticeable decrease in the elastic modulus, in particular in the outer contour and base planes. However, after adding 10 wt.% ESO, a significant increase in the elastic modulus was observed for all measurement planes.

The strain energy in polymers, as in other materials, is closely related to the processes of restructuring and destruction of the fluctuating network during deformation. When an external mechanical load is applied to a polymeric material, stresses are distributed within the material, which leads to an increase in its internal energy.[18] According to the results obtained, 80-90% of the strain energy transmitted in the system of the developed composites is dissipated under the influence of the load, which corresponds to irreversible internal damage in the material and plastic deformation. In turn, 10-20% of the energy remains as elastic strain energy, which is reversible and bidirectional and reflects the internal properties of the material, primarily the elastic modulus and Poisson's ratio.

To confirm the targeted adjustment of the properties of the developed composites, their impact strength was studied. The impact strength of polymers and composites is a parameter that determines their ability to absorb energy under impact loads.[19] This property is important for predicting the behavior of a material under sudden dynamic loads, such as impacts or falls. In the case of engineered composites, impact strength reflects their resistance to crack initiation and propagation under dynamic mechanical forces. The impact strength values of the composites are shown in Fig. 3.

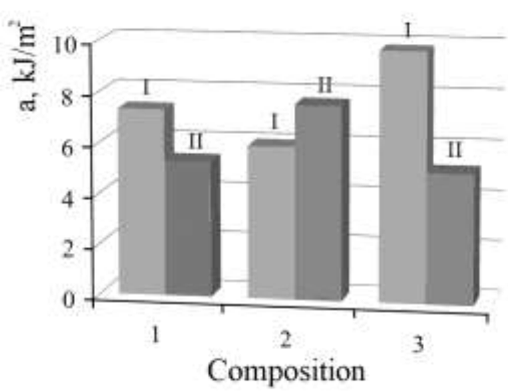
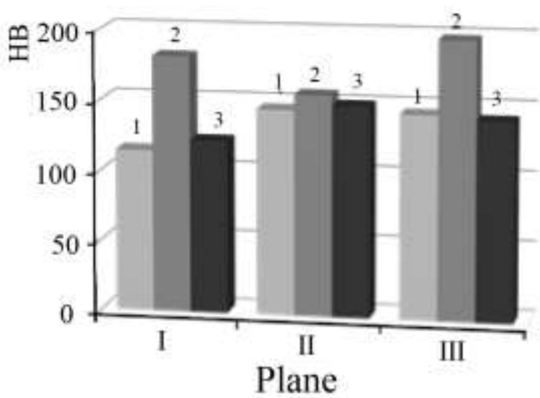


Fig. 3. Impact strength before and after heat treatment of composites based on epoxy resins: 1 – epoxy resin; 2 – ER : starch 90 : 10 (wt.%); 3 – ER : starch : ESO 80 : 10 : 10 (wt.%). I – without heat treatment; II – heat treatment at 353 K for 120 min

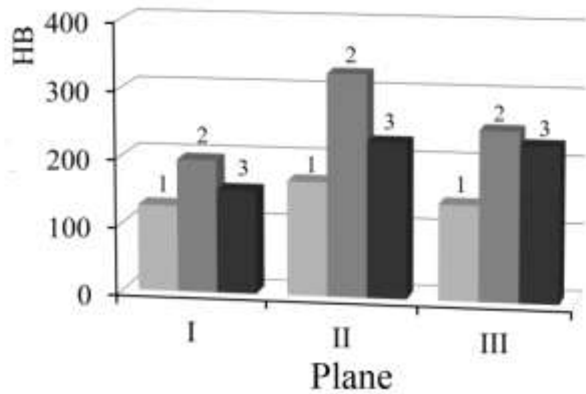
The high impact strength observed for epoxy-filled 3D matrices highlights their ability to effectively absorb and dissipate impact energy, making them less susceptible to fracture mechanisms in service. The addition of starch results in a lower impact strength compared to pure epoxy resin. The reduction in impact strength may be due to poor interfacial adhesion between starch and epoxy. Starch, being hydrophilic, may not bind well to the more hydrophobic epoxy, resulting in weaker adhesion, making cracks easier to form and propagate faster under dynamic loading.

At the same time, the introduction of ESO improves the impact strength of the composite. Apparently, the ESO acts as a plasticiser, improving the flexibility of the material and increasing its ability to absorb and dissipate energy during impact loading. The addition of ESO is likely to neutralise the starch-induced brittleness by reducing the shrinkage effect of starch macromolecules and providing a more uniform stress distribution in the composite.

The Brinell hardness values, which were investigated using a spherical indenter with a diameter of 5 mm, at a load of 100 kg and a holding time of 30 s, are shown in Fig. 4.



a



b

Fig. 4. Brinell hardness before (a) and after (b) heat treatment at 353 K for 120 min. for PLA matrices filled with epoxy composites. I — plane on the filling side; II - outer contour plane; III - base plane. Composition, wt.% : 1 – epoxy resin; 2 – ER : starch 90 : 10 (wt.%); 3 – ER : starch : ESO 80 : 10 : 10 (wt.%)

For all measurement planes, a similar effect of resin modification on the hardness value was observed. The addition of starch significantly increases the Brinell hardness. This is because starch particles distribute the applied load more efficiently and increase the material's resistance to indentation and deformation. The increased hardness indicates that the introduction of starch contributes to the creation of a more rigid and strengthened structure of the epoxy matrix. At the same time, the addition of ESO to starch-modified epoxy resin results in a noticeable decrease in hardness. Epoxied soybean oil acts as a plasticiser that breaks the density of polymer chains, increasing their mobility and flexibility.

The use of heat treatment further increases the Brinell hardness for all samples, regardless of their composition. This enhancement is likely due to the structuring within the polymer matrix caused by the temperature. Temperature curing promotes better adhesion and bonding between the resin and the fillers. As a result, the material becomes less susceptible to deformation under stress. In addition, heat treatment can eliminate any residual stresses and voids in the composite structure, creating a more homogeneous material.

Surface hardness values determined by different methods correlate with each other.

Fig. 5 shows the surface hardness of epoxy resin composites depending on the nature and content of the modifier and additional heat treatment determined by the conical yield point method.

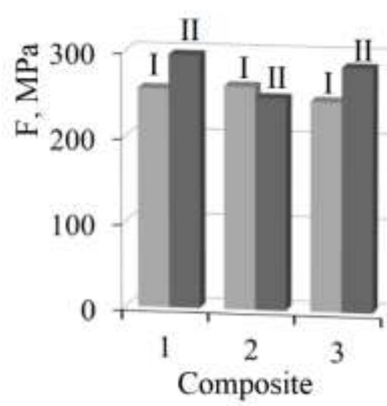


Fig. 5. Surface hardness before (I) and after (II) heat treatment of epoxy resin-based compounds, wt.%: 1– epoxy resin; 2 – ER : starch 90 : 10 wt.%; 3 – ER : starch : ESO – 80 : 10 : 10 wt.%

The surface hardness value for an unmodified epoxy resin indicates a high mechanical resistance to external forces. Heat treatment affects the structuring within the epoxy matrix and also increases the surface hardness. This indicates the importance of additional processing to improve the structural integrity of unmodified epoxy.

It was found that the addition of 10% w/w starch has almost no effect on the surface hardness. However, additional heat treatment slightly reduces its value. This is obviously due to the partial destruction of starch under the influence of temperature. The addition of 10 wt.% of ESO leads to a decrease in hardness, which is explained by the plasticiser's effect on increasing the flexibility and strength of the material, but is accompanied by a decrease in the hardness of its surface. Heat treatment further strengthens the structure.

To substantiate the potential applications of the developed composite materials, it is important to evaluate their properties under different loading conditions, in particular, under dynamic bending and tensile loads. Therefore, we studied the effect of the modified epoxy resin on the values of flexural and tensile strength. The results are shown in Table 2.

The lowest flexural strength is characterised by an unfilled polylactide matrix. The introduction of epoxy resin improves the flexural strength, increasing the ability of the material to withstand the applied load. In addition, this modification leads to a significant increase in the strain of the specimen, indicating a greater ability of the material to bend under load, thereby improving its overall flexibility and strength.[16] Further modification of the epoxy with starch and ESO significantly increases the flexural strength. This can be attributed to the strengthening effect of starch particles, which contribute to a stiffer composite structure.[18] The presence of ESO also significantly affects the mechanical properties of the material, acting as a plasticiser. The reduced strain values observed with the addition of ESO indicate that the material becomes more flexible but loses some of its stiffness.

Similar patterns were observed when the tensile strength of the samples was tested. The addition of starch did not affect the tensile strength values. This indicates that although starch contributes to the improvement of the resistance to deformation during bending by strengthening the composite, it plays a more limited role in improving the tensile properties due to its structure. At the same time, the combined addition of starch and ESO leads to a decrease in tensile strength of up to 11 MPa, with the predominant influence of the plasticising effect of ESO on this indicator. While ESO provides flexibility to the composite, it also reduces its tensile strength by breaking down the rigid structure formed by the epoxy and starch.

Table 2. Physical-mechanical properties of combined composites

Filling	Bending strength, MPa	Deformation during bending, %	Tensile strength, σ_p, MPa	Relative elongation, ε_r, %
Output PLA matrix (without filling)	64.2	12.9	-	-
Unmodified epoxy resin	71.0	20.4	16.1	7.9
ER 90 wt.% starch 10 wt.%	81.6	20.4	16.0	6.1
ER 80 wt.%, starch 10 wt.%, ESO 10 wt.%	85.2	18.0	11.1	5.0

4. Conclusions

The peculiarities of obtaining combined composites based on polylactide 3D printed matrices and modified epoxy resin have been investigated. It was found that the obtained load-strain curves are typical for viscoelastic materials and differ in the slope of the loading curve.

The introduction of starch leads to a decrease in impact strength compared to pure epoxy resin, and ESO improves the impact strength of the composite by increasing the flexibility of the material and increasing its ability to absorb and dissipate energy under impact loads. It was found that the introduction of 10 wt.%. ESO to starch-modified epoxy resin increases the elastic modulus of composites.

It was found that the filling plane of the studied products significantly affects the strain and hardness values. It was found that the introduction of starch leads to an increase in bending strain by 15-16% and a concomitant decrease in hardness. At the same time, the additional introduction of epoxied soybean oil into the starch-modified resin contributes to an increase in the elastic modulus by 20-25% for all planes of load application.

Based on the study of the effect of modified epoxy resin on flexural and tensile strength, it was found that the combined addition of starch and ESO leads to a significant increase in flexural strength and a decrease in tensile strength up to 11 MPa.

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КОМБІНОВАНІ КОМПОЗИТИ НА ОСНОВІ ПОЛІЛАКТИДНИХ 3D МАТРИЦЬ І МОДИФІКОВАНИХ ЕПОКСИДНИХ СМОЛ

Анотація. *Розроблено матеріали на основі полілактидних 3D матриць із подальшим заповненням їх модифікованою епоксидною смолою. Досліджено їхні пружно-пластичні, деформаційні і міцнісні характеристики. Виявлено, що площина заповнення досліджуваних виробів істотно впливає на значення деформації і твердості. Введення в композицію епоксидованої соєвої олії покращує ударну в'язкість композитів, підвищуючи гнучкість матеріалу, та збільшує його здатність поглинати та розсіювати енергію під час ударних навантажень. Комбіновані композити відзначаються підвищеними значеннями міцності на згин, міцності під час розривання та деформації під час згинання.*

Ключові слова: *епоксидна смола, 3D друк, крохмаль, епоксидована соєва олія, комбіновані композити.*