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OPTIMIZING THE PARAMETERS OF THE GLASS FIBER IMPREGNATION PROCESS BY MEANS OF EXPERIMENT PLANNING METHOD.

The workpaper examines the structural features and selection of composite materials components, in particular the matrix and reinforcing elements, which together form a material with improved mechanical and operational properties. The classification of matrices by type (polymer, metal, ceramic) is described and the requirements for polymer resins most often used in the production of structural composites are given. Special attention is paid to the technology of filament winding, in particular the wet winding method, as the most common method of forming composite shafts from fiberglass and carbon fiber. Key factors affecting the quality of impregnation of reinforcing fibers with resin are identified, such as resin viscosity, fiber drawing speed, tension, bath geometry and properties of the resin system. It is emphasized that uniform and complete impregnation of fibers is critically important for ensuring the strength, stiffness and durability of the finished product.

The paper presents the results of studying the process of impregnation of fiberglass tows with resins applying the software module "Design Analysis of Experiments". Experiment planning methods were applied to determine the influence of the main technological parameters – winding speed, tow thickness and resin viscosity on the impregnation coefficient. A mathematical model and response surfaces were developed, which allow assessing the interaction of factors and establishing their optimal values. It was found that the maximum impregnation coefficient is achieved at a winding speed of 1136 RPM, a tow thickness of 81 micrometres, and a resin viscosity of 0.86 Pa·s. The results can be used to improve the efficiency of composite materials production processes.

Keywords: *composite, matrix, resin, fiberglass tow, impregnation, experimental design, viscosity, winding speed, impregnation coefficient, response surface*

ПОСОНСЬКИЙ СЕРГІЙ
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ОПТИМІЗАЦІЯ ПАРАМЕТРІВ ПРОЦЕСУ ПРОСОЧЕННЯ СКЛОВОЛОКНА МЕТОДОМ ПЛАНУВАННЯ ЕКСПЕРИМЕНТУ

Визначено ключові фактори, що впливають на якість просочення армуючих волокон смолою, такі як в'язкість смоли, швидкість протягування волокон, натяг, геометрія ванни та властивості смоляної системи. Підкреслено, що рівномірне та повне просочення волокон є критично важливим для забезпечення міцності, жорсткості та довговічності готового виробу.

У роботі представлено результати дослідження процесу просочення скловолоконних джгутів смолами з використанням програмного модуля «Design Analysis of Experiments». Застосовано методи планування експерименту для визначення впливу основних технологічних параметрів – швидкості намотування, товщини джгута та в'язкості смоли на коефіцієнт просочення. Побудовано математичну модель та поверхні відгуку, що дозволяють оцінити взаємодію факторів і встановити їх оптимальні значення. Отримано, що максимальний коефіцієнт просочення досягається при швидкості намотування 1136 об/хв, товщині джгута 81 мкм та в'язкості смоли 0,86 Па·с. Результати можуть бути використані для підвищення ефективності процесів виробництва композитних матеріалів.

Ключові слова: *композит, матриця, смола, скловолоконний джгут, просочення, планування експерименту, в'язкість, швидкість намотування, коефіцієнт просочення, поверхня відгуку.*

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Introduction

Composite materials are formed by combining two or more chemically dissimilar components that have a clear boundary between them. Unlike conventional mixtures, each component retains its own identity in composites, but together they form a material with improved characteristics. One of the components is the matrix - it forms a continuous phase; another component, which is responsible for strengthening the structure, is integrated into the phase. This reinforcing element is usually much stronger and stiffer than the matrix itself and plays the role of enhancing its mechanical properties.

Either thermosetting resins (e.g., epoxy, polyester, phenolic) or thermoplastics (e.g., polyethylene, polystyrene) can serve as a matrix in polymer composites. Metal matrices usually include aluminum, titanium, or magnesium, while ceramic matrices include materials based on aluminum oxide or calcium aluminosilicates [1,2].

When choosing a matrix material for a composite, a number of factors are taken into account, including thermal characteristics, availability, economic feasibility, environmental safety and health effects, as well as the possibility of technological processing. The matrix plays a key role as a binding phase, ensuring the transfer of mechanical load to the reinforcing fibers, maintaining their spatial orientation and protecting the fibers from damage and destruction.

When choosing a polymer resin, such properties as modulus of elasticity (stiffness), fluidity, tensile strength and general mechanical characteristics are important. In industry, in particular in the automotive industry, two types of polymers are most often used: thermosetting and thermoplastic. For structural composites with high requirements for mechanics, heat resistance and adhesion to reinforcement, thermosetting resins are most often used, since they have low viscosity and affordable cost, which facilitates the manufacturing process.

The resin selection process includes analysis of parameters such as viscosity, glass transition temperature, gelation and curing time, thermoforming ability, resistance to aggressive environments, and environmental safety indicators, including the level of emission of harmful substances during processing. Mechanical properties that are considered are interlayer viscosity, elastic modulus, strength in certain directions, and resistance to external influences.

Common thermosetting polymers used as matrices include epoxy, polyester, and vinyl ester resins. They are good at impregnating reinforcing fibers (e.g., glass fiber, carbon fiber, Kevlar) and provide a reliable bond during the composite formation process [3].

The matrix in the composite envelops the fibers, allowing them to withstand high loads even with lower stiffness and higher elongation. Therefore, the choice of an appropriate matrix material depends on the requirements for chemical, thermal, electrical stability, fire resistance, cost, environmental friendliness, operational reliability and manufacturability. The matrix determines not only the behavior of the material in operation, but also the processing parameters during production [2].

Reinforcement can have different shapes (most often fibers) and morphology, which directly affects the final properties of the composite. Most often, reinforcing elements in composites are fibers made of glass, carbon, aramid or boron. Usually their diameter is in the range from 5 up to 20 micrometers [3].

In addition to traditional fibers, other forms of reinforcement are also widely used in composites, including continuous carbon tow, glass roving, aramid yarn, and woven materials. Accordingly, the type, amount, and placement of the reinforcing phase determine the performance characteristics of the material.

Fiberglass and carbon fiber are the most common fibers for composite shafts. They are laid by means of using filament winding technology. This method involves laying a continuous roving of fiber onto a rotating mandrel according to a given pattern.

There are two main approaches to winding: wet and dry. In the wet winding process, the fiber is impregnated with a polymer resin before laying - this can be done either by passing the fiber through a resin bath or using a metered application system. In the dry method, the fiber is wound without prior impregnation, and impregnation and curing occur after laying several layers. After the workpiece is formed, it is removed from the mandrel.

The most wide spread method of impregnating fibers in industry is the resin bath method. This approach usually involves the use of thermosetting polymers. The term “resin system” in this case means a specially prepared mixture consisting of a base resin and a suitable hardener, which together provide the necessary properties for subsequent hardening. A schematic representation of the wet winding process is shown at the Picture 1.

The fiber bundles are passed through a bath filled with resin, then they are wound onto a mandrel that rotates at a given speed. The fiber is distributed along the length of the mandrel by a special feeding mechanism, which ensures uniform laying according to a predetermined pattern that corresponds to the configuration of the finished product. After applying the required number of layers, the material hardens directly on the mandrel. When the laminate reaches the required stiffness, the formed part is removed from the mandrel.

The degree of impregnation the fibers with resin directly affects the mechanical properties of the composite material, primarily its strength, stiffness and durability. Well-impregnated fibers provide effective adhesion between the matrix and the reinforcement, which allows to distribute the loading evenly. A composite with even impregnation absorbs impact energy better without sudden failure. Voids or “dry zones” act as stress concentration points.

The degree of impregnation of fibers in the resin bath depends on several key factors that affect the quality of the composite material and the efficiency of load transfer between the matrix and the reinforcement. The main ones are: resin viscosity, fiber pulling speed through the bath or impregnation time, fiber type and thickness, fiber tension during passage through the bath, bath geometry and depth, and resin system composition. For example, carbon fibers have worse wettability than glass fibers and may require additional treatments. Optimal tension promotes the opening of the fiber bundle and facilitates resin penetration. Additives, fillers, or accelerators can change the rheological properties of the mixture, affecting the impregnation process.

The quality of impregnation is critical for achieving maximum strength and reliability of a composite product. That is why impregnation control is a key stage in composite manufacturing technology.

Forming the article’s objectives

The workpapaer objective is determining the optimal parameters of the glass fiber impregnation process when forming a composite shaft using the wet filament winding method.

Analysis of research and publications

In [5], an improved version of the wet filament winding technology is described. In particular, the traditional resin bath is replaced by an injection system that provides impregnation of the fibers before they are laid on a rotating mandrel. The E-glass fiber bundles, that were used in the workpaper, were EC 15 1200 TEX (PPG Industries, United Kingdom) with a density of 2550 $kg \cdot m^3$ and a filament diameter of 17 micrometres, and the number of filaments in the fiber bundle was approximately 2000. The width of the fiber bundle (tow) was 8 mm, Picture 2, while the tow thickness H varied from 54 micrometres to 162 micrometres at a tension of 10 N and a pressure in the resin layer between the fibers of 83.3 kPa. The resin and hardener were stored separately and fed by precisely tuned gear pumps that directed both components to a static mixer. The already mixed resin system was fed to a specially designed fiber impregnation device.

The article also presents the theoretical principles of designing such an impregnation system and the results of modeling the main parameters (resin viscosity, winding speed, impregnation time, fiber bundle thickness, fiber tension, fiber linear density, and resin injection pressure) that affect the resin infiltration rate and the efficiency of impregnation of the reinforcing material. Fiber impregnation in a resin injection system is carried out in four stages. The first stage involves forcing the resin into the fiber bundle through the applicator zone. In the second stage, the resin is injected directly into the bundle through the nozzle. The third stage includes the accumulation of resin in a local reservoir under the nozzle with subsequent capillary penetration. The fourth stage provides additional impregnation using a scraper located after the injector.

The authors [5] found out that the highest impregnation efficiency is observed when using stages I and IV. This is due to the fact that in the zone between the fiber bundle and the surface of the pin, where a wedge-shaped layer of resin is formed, increased pressure occurs. Such pressure promotes active penetration of the resin through the fiber structure. In impregnation systems based on the use of a pin, the main part of the impregnation occurs only in a limited area of the pin - relatively short in relation to the entire length of contact. This segment was called the "impregnation zone" h_{imp}, Picture 2, since it is there that the fibers are intensively saturated with resin [5].

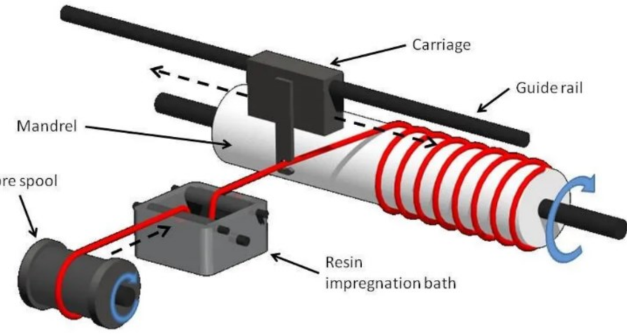

Fig.1. Schematic representation of the wet winding process [4].

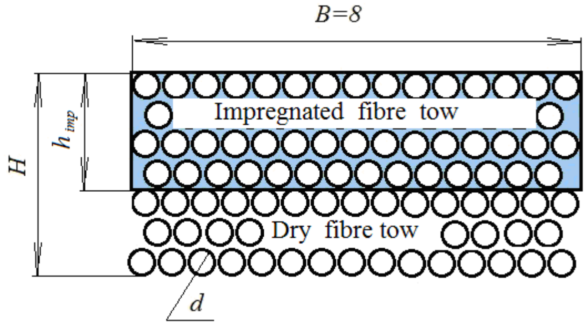

Fig.2. Schematic representation of the cross-section of the harness during resin impregnation.

The workpaper [6] analyzes the influence of epoxy resin viscosity on the physical and mechanical characteristics of carbon fiber shells formed by filament winding on a polyurethane foam mandrel. The cylindrical samples were exposed to resins with different viscosity levels, which allowed us to trace their influence on the properties of the finished product. The results showed that with a decrease in resin viscosity, the key characteristics of the composite shell improve. In particular, with a decrease in viscosity within 0.6–0.76 Pa·s, a decrease in resin absorption by fibers by 4.5% is observed, as well as a decrease in the mass and thickness of the shell by 3% and 5.4%, respectively, compared with the cases of using resins with a viscosity of 1.38–2.08 Pa·s. In addition, with a lower viscosity, the volume fraction of fibers and the density of the material increase by 6.3% and 2.8%, respectively. These indicators stabilize or change slightly after further thermal exposure, which is accompanied by a decrease in viscosity.

The workpaper [7], shows the analisys of how the diameter of individual fibers and the number of fiber bundles affect the impregnation process under liquid-phase molding conditions. The simulation results indicate that both the diameter of the fibers and the number of fibers in the bundle have a significant impact on the duration of the infiltration process. For example, the time of complete impregnation for a bundle of 60,000 carbon fibers turned out to be ten times longer than for a bundle of 6,000. In addition, there is an inversely proportional relationship between the diameter of the fibers and the infiltration time: when the diameter of the fibers decreases from 3.5 mm to 35 nm (by a factor of 100), the impregnation time increases by a factor of 100 for a bundle of the same diameter.

It was also found that the volume fraction of fibers in the tow has a significant effect on the uniformity of impregnation. Any spatial variations in the distribution of fibers can cause the formation of dry zones due to uneven infiltration rates. According to the model, the accumulation of a critical volume of air inside the tow can lead to an increased level of porosity, which is undesirable in the finished composite.

The authors of [8] tested cylindrical samples of composite materials based on polypropylene (GF/PP) and polyphenylene sulfide (GF/PPS), reinforced with glass fiber, using filament winding technology. The main attention was focused on the selection of optimal technological parameters, such as heating temperature, pressure of pressure rollers and winding speed, in order to determine their influence on the compressive strength of the samples.

According to the results of the experiments, the optimal processing modes for GF/PPS and GF/PP materials were established: heater temperature 575 °C and 275 °C, respectively, roller pressure 4 bar (corresponding to a compaction force of 321.70 N), as well as a winding speed of 1600 mm/min for GF/PPS and 1200 mm/min for GF/PP. The influence of these parameters was considered in the context of the use of glass fiber reinforcement, which plays a key role in shaping the mechanical properties of the composite.

Presentation of the main material

The research was focused on optimizing key parameters of the epoxy resin impregnation process of glass fiber during the formation of a composite shaft by wet winding, including tow thickness, resin viscosity, and winding speed. All other parameters were taken according to [5]. The impregnation coefficient was defined as the ratio of the impregnated cross-sectional area of the tow to its total area.

The problem of experiment planning can be presented in mathematical form as the need to obtain an idea of the response surface, which is described in the form of a functional dependence or analytical model [9]:

$$Y = f(x_1, x_2, \dots, x_n), \quad (1)$$

where Y – (optimization criterion), and $(x_1, x_2, \ldots, x_n)$ –factor variables that affect the outcome of an experiment and can change during the research process. Thus, the task is to establish a functional relationship between the mathematical expectation of the process output and the values of the controlled parameters.

As mentioned above, the winding speed is taken as a factor V, RPM; thickness of the harness H, micrometres; and resin viscosity μ, Pa·s. The impregnation coefficient K_{imp}, % was taken as the optimization criterion. Thus, model (1) will have the following form:

$$K_{imp} = f(V, H, \mu). \quad (2)$$

To determine the optimal parameters that ensure the maximum degree of impregnation of the fiber bundle with epoxy resin, the method of mathematical experimental design was applied. In particular, a central compositional plan of the uniform-rotatable type was used to build a regression model and calculate its coefficients.

As the basic levels of factors, values, close to those actually used in wet winding conditions, were chosen. The limits of factors' variation were determined in accordance with the permissible technological ranges. In addition to the basic levels, star points with values of +1.68 and –1.68 were also included in the planning matrix (Table 1). The complete experimental matrix is given in Table 2.

Table 1

Factor levels and variation intervals

Factors	Levels				
	1,68	+1	0	– 1	– 1,68
V – winding speed, rpm	1472	1200	800	400	128
H – bundle thickness, micrometres	126,36	108	81	54	35,64
μ – resin viscosity, Pa·s	3,38	2,7	1,7	0,7	0,02

Results processing and search for optimal parameters were carried out by means of Statistica software. The coded regression equation, which reflects the dependence of the impregnation coefficient on the process variables, was constructed on the basis of the obtained experimental data and has the following form:

$$K_{imp} = b_0 + b_1V + b_2V^2 + b_3H + b_4H^2 + b_5\mu + b_6\mu2 \quad + b_7VH + b_8xV\mu + b_9H\mu. \quad (3)$$

As a result of the modeling, the values of the regression coefficients were determined (Picture 3), and a Pareto diagram was built (Picture 4), which ranks the standardized effects from the most influential to the least significant. In addition, the diagram contains a critical line, which reflects the level of statistical significance of the effects, which depends on the adopted level α . Analyzing the graph, we can make a conclusion about the dominant influence of such factors as the winding speed with the coefficient $b_1 = 0{,}0672$, resin viscosity with coefficient $b_5 = -17{,}5557$ and the thickness of the harness with the coefficient $b_4 = -0{,}0121$ on the effective sign. All coefficients that fall within the reference line are considered insignificant, namely: $b_2, b_3, b_6, b_7, b_8, b_9$.

Table 2

Experimental design matrix in real values and test results

№	V, rpm	H, micrometres	μ, Pa·s	K_{imp}, %
1	400	54	0,7	98
2	400	54	2,7	58
3	400	108	0,7	63
4	400	108	2,7	52
5	1200	54	0,7	98
6	1200	54	2,7	92
7	1200	108	0,7	98
8	1200	108	2,7	75
9	128	81	1,7	45
10	1472	81	1,7	90
11	800	35,64	1,7	65
12	800	126,36	1,7	60
13	800	81	0,02	98
14	800	81	3,38	75
15	800	81	1,7	93
16	800	81	1,7	93

Then the final equation for the impregnation coefficient will be:

$$K_{imp} = 23{,}1945 + 0{,}06721V - 0{,}0121H^2 - 17{,}5557\mu. \quad (4)$$

Factor	Regr. Coefficients; Var.:Impregnation coefficient, %; R-sqr=,8785; 3 factors, 1 Blocks, 16 Runs; MS Residual=107,1113 DV: Impregnation coefficient, % Regressn Coeff.	Std.Err.	t(6)	p	-95,% Cnf.Limt	+95,% Cnf.Limt
Mean/Interc.	23,1945	50,22171	0,46184	0,660466	-99,6936	146,0826
(1)Winding speed, rpm(L)	0,0672	0,04694	1,43149	0,202250	-0,0477	0,1820
Winding speed, rpm(Q)	-0,0000	0,00002	-2,07580	0,083216	-0,0001	0,0000
(2)Bundle thickness, micrometres(L)	1,4672	0,84241	1,74166	0,132206	-0,5941	3,5285
Bundle thickness, micrometres(Q)	-0,0121	0,00467	-2,59617	0,040872	-0,0236	-0,0007
(3)Resin viscosity, Pa·s(L)	-17,5557	17,77321	-0,98776	0,361412	-61,0452	25,9338
Resin viscosity, Pa·s(Q)	-0,3350	3,40442	-0,09842	0,924808	-8,6654	7,9953
1L by 2L	0,0003	0,00034	0,81988	0,443629	-0,0006	0,0011
1L by 3L	0,0069	0,00915	0,75155	0,480749	-0,0155	0,0293
2L by 3L	0,0556	0,13552	0,40994	0,696086	-0,2761	0,3872

Fig.3. Fragment of the Statistica program window for determining regression coefficients.

Applying the built-in module "Design Analysis of Experiments" in the software environment allows us to investigate the degree of influence of each of the technological parameters on the efficiency of impregnation, as well as to determine the such values of factors when the maximum impregnation coefficient of the fiberglass tow is achieved. Based on the analysis of the modeling results (Picture 5), the optimal process parameters were established at which the highest level of impregnation is ensured: the winding speed is 1136 rpm, the tow thickness is 81 micrometres, and the resin viscosity is 0.86 Pa·s.

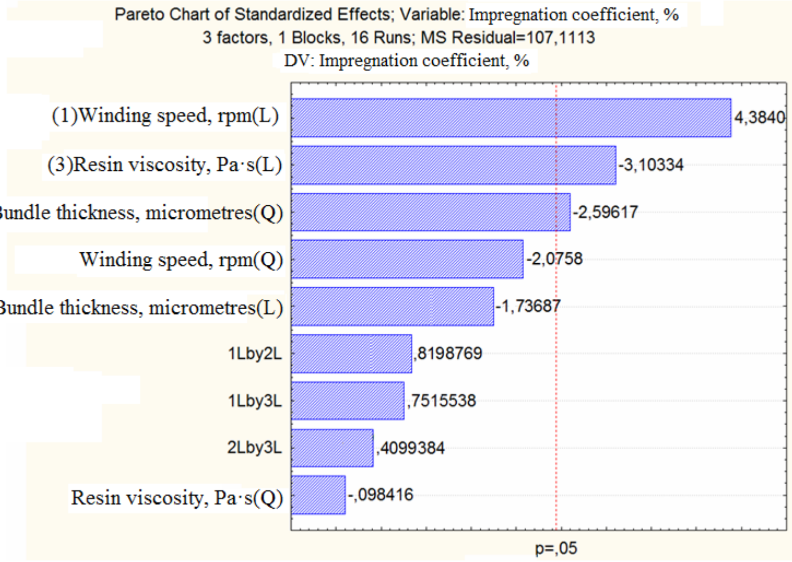

Fig.4. Significance diagram of regression coefficients.

Using the obtained model (4), which describes the dependence of the impregnation degree on the parameters of the technological process, the corresponding response surfaces were constructed. Picture impregnation 6 shows graphical interpretations of these dependencies with gradual fixing (blocking) of each of the optimized factors.

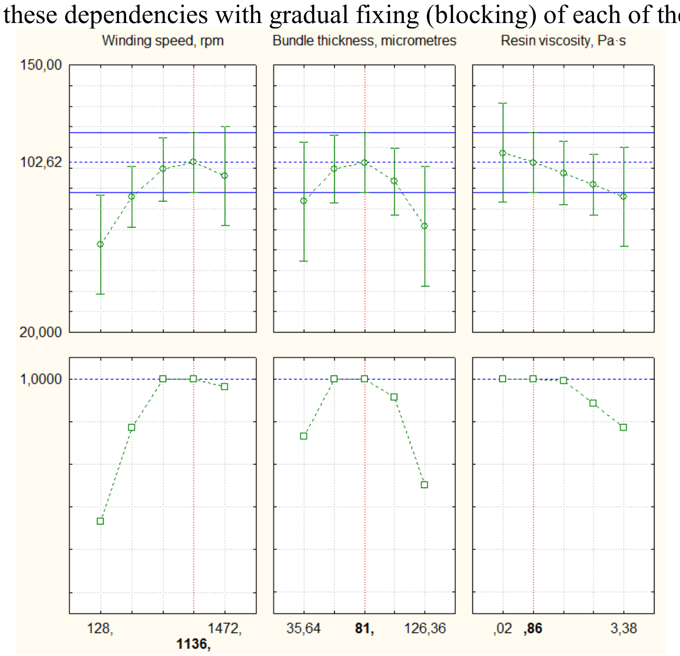

Fig.5. Searching for the optimal parameters of the impregnation process.

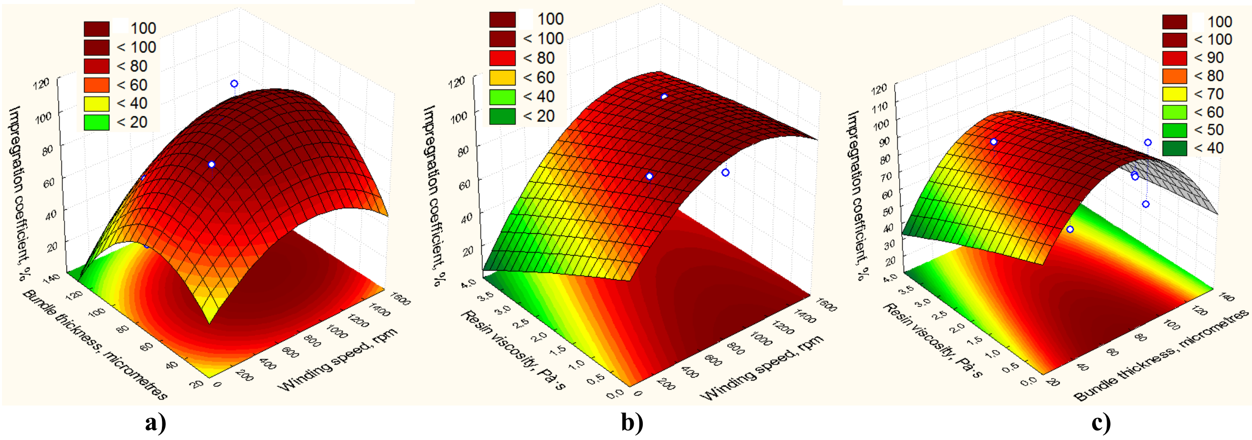

Fig.6. Dependence of the impregnation coefficient on the parameters when blocking the next optimal factor: a) resin viscosity 0,86 Pa·s; b) tow thickness 81 micrometres; c) winding speed 1136 rpm.

Reducing the thickness of the fiber bundle helps to reduce the time for transverse resin impregnation. At practical sight, it is important to find an optimal compromise between the level of fiber separation to improve impregnation and avoiding excessive mechanical stress on them. Excessive fiber damage can deteriorate the strength characteristics of the composite material, and too much segmentation can cause air entrapment, which in turn leads to the formation of voids in the composite structure.

Increasing the viscosity of the resin system leads to an increase in the time required for complete saturation of the fibers. In the case of using a resin bath, the viscosity may change over time, which is connected with the course of chemical reactions or instability of the temperature regime. The problem is complicated if stagnation zones form in the bath, where the resin mixture is not updated properly, which contributes to local thickening and violation of the uniformity of impregnation.

Conclusions

The influence of the main technological factors on the impregnation process of fiberglass tows was determined as the result of the research, applying experimental design method. The optimal values of the parameters were obtained; due to themthe maximum impregnation coefficient is achieved: tow winding speed – 1136 rpm, tow thickness – 81 micrometres, resin viscosity – 0.86 Pa·s.

Developed response surfaces and results' analysis confirmed that the application of the experimental design method allows to determine and optimize the technological process modes, increasing the quality and reliability of the obtained composite materials.

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