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ADJUSTING THE PERFORMANCE PROPERTIES OF PRODUCTS OBTAINED BY INJECTION MOLDING FROM POLYAMIDE 12

***Serhii Prystynskyi*[1,✉]**

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Abstract. The influence of technological process parameters on the resistance to an aggressive environment (ethyl alcohol C_2H_5OH) of plastic parts obtained by injection molding from the polymer composite material polyamide 12 (PA12) was studied and shown.

Keywords: injection molding, process parameters, process capability, aggressive environment, chemical resistance, polar solvent, polyamide.

1. Introduction

In fact, the use of plastic products remains relevant and continues to improve in the world, due to its unique properties and the wide possibility of processing by choosing the necessary method. It is believed that polymers improve the world and lifestyle.[1] Baillie *et al.*[2] show that polymer composite materials have a unique interaction between the structure and properties of various components and conditions of use, which proves the popularity of the polymer products application. Nowadays metal products are replaced by products that are obtained from polymer composite materials. This is achieved by the unique properties of plastics, which are subject to regulation and are used in many areas of the production of the most modern high-tech equipment, in particular, solar-powered aircraft, "Falcon 9" spacecraft, modern cars such as"Tesla",[3] Mercedes-Benz, *etc.*

Despite the fact that technologies, including those for polymer processing, are constantly evolving, injection molding remains one of the main ways to produce polymer parts used in many industries.[4]

Depending on the application field, a number of different requirements can be applied for products obtained by the injection molding, including performance characteristics. It is worth noting that the parts used in the automotive industry are subject to stringent requirements, and the finished products are selectively tested for strength and reliability in the process of mass production by simulating operating conditions. One of these requirements is product resistance to an aggressive environment, in particular to creams, solvents, and other substances. This is due to the fact that the products are exposed to both the natural environment (climate) and the artificial one (detergents, creams, preservatives, etc.). That is why it is important to be able to manage such special properties and characteristics of products during their manufacturing by injection molding. The approach to defining and managing the properties of finished products can also affect the environment and ecological aspects. This is because many plastic products are disposed of already during their lifetime, potentially affecting the level of polymer waste that is difficult to recycle.

In view of this factor, great attention is paid to the quality of the parts produced, including the comparison of qualitative and quantitative indicators or characteristics, such as reliability, safety, and performance.[5]

The performance characteristics of the obtained components and the possibility of their control by process parameters or material modification are actively studied even nowadays, using relational analysis according to the Taguchi method.[6] Additionally, a two-stage optimization system is used to achieve ideal process settings,[7] including the cooling system for molding tools, which has a positive effect on product quality, thereby reducing the deformation of structural elements.[8] Recently, requirements have been put forward regarding the biodegradation of the finished parts at the end of their lifetime, but such components have low strength, which can be improved by the use of modifiers, such as reinforcing filler.[9] The focus is on achieving an ideal product with preserved or improved properties under optimal manufacturing process parameters without losing production cycle time. The biodegradation of polymers and the impact of its decomposition by-products on various ecosystems[10] and on the microbiology of the soil where cultivated plants are grown are also being actively investigated.[11]

Among the main external factors, affecting the product is the effect of various polar solvents, which can change the structure of the polymer due to intermolecular

[1] KOSTAL Ukraine TOV, 2 Pokrovska St., Pereiaslav 08400, Ukraine
✉ *s.prystynskyi@kostal.com*

interaction.[12] Scientists explain the dissolution effect as the interaction of energies between the solvent and the polymer,[13] as the transition from a polymer to a monomer. During the action of a polar solvent on polyamides, it is possible to observe swelling and deformation of the surface. This is due to the rearrangement of the distribution of plasticization hydrogen chains,[14] and the high hygroscopicity of polyamides and polar solvents, which can also affect hydrogen bonds.

Nowadays, a lot of attention is paid to the optimization of injection molding processes and the improvement of products obtained by this method. Recent studies demonstrate the possibility of reducing energy costs by avoiding overloading the drive of injection units by optimizing process conditions.[15] Scientists are also focusing on improving the products obtained by this method. In particular, they show the methods of deformation optimization even at the initial stage by modeling and integrating an artificial neural network and using integrated automatic optimization tools.[16] Optimization methods based on the Pv-T diagram to obtain better quality optical properties are also considered.[17] Research is also being conducted in the automotive industry to study the impact of certain process parameters on the performance of the finished product.[18] It is also worth noting that the parameters of the injection molding process affect the resistance of the resulting product to the action of chemical substances. In particular, scientific papers[19,20] have shown that certain process parameters, such as the injection speed and molding tool temperature affect morphology, mechanical properties, and especially chemical stability, although this phenomenon has not received sufficient attention.

At the same time, the effect of polyamide modification on their properties[21] and preliminary laser surface treatment, which positively affects adhesion,[22] is sufficiently studied. The influence of fillers on the physicochemical properties of polyamides is being actively investigated. The scientists are actively working on increasing the strength, particularly of polyamide 6 (PA6) strength,[23] which can also impact the process parameters and the properties of the finished product.

Despite the active studies on the injection molding process, it can be noted that the study of the influence of its main technological parameters on the chemical properties and resistance to polar solvents of products made from PA-based polymer compositions is insufficient. The research on the polyamide resistance to solvents is shown by Meng and Liu[24], who studied the resistance of the material to aggressive external factors, but a preference was given to physical and mechanical properties.

The study of performance characteristics is an important and necessary step in order to be able to improve finished products not only to maximize customer satisfaction but also to reduce waste generation both during production and use. Depending on the industry, products manufactured in developed countries are tested for resistance to aggressive environments, usually climatic tests and other "stress" tests aimed at evaluating the mechanical properties of products.

Therefore, it can be noted that there is limited data on the influence of the injection molding parameters on the performance of parts, in particular on the resistance to polar solvents.

However, when interfering with the validated serial parameters of the process, it is necessary to evaluate the stability (C_{mk}) of both the injection-molding machine and the injection molding process (C_{pk}). The acceptable level of C_{mk} in the automotive should exceed 1.67 ($C_{mk} \geq 1.67$). For C_{pk}, the level of more than 2.0 will be preserved ($C_{pk} > 2.0$).[25]

This paper presents a study on the influence of the main technological parameters on the properties of parts obtained by injection molding from PA12, in particular, on the resistance to the polar solvent ethanol C_2H_5OH, which is the purpose of the study.

2. Experimental

The parts for the experiment were produced on an ARBURG 270S injection molding machine using standard serial and adjusted process parameters from PA12 from the RTP Company of the RTP-299EX146375S-806906TRANSBLACK brand. The main characteristics of the used polymer composition are listed in Table 1 according to the specifications provided by the manufacturer.

Table 1. Properties and average values of injection molded specimens of PA12

Characteristic	SI Metric	Value
Density	g/cm^3	1,00
Impact Strength, Izod	kJ/m²	10
Tensile Strength	MPa	50
Tensile Modulus	MPa	1650
Injection Pressure	MPa	70-105
Melt Temperature	°C	270-300
Mold Temperature	°C	65-100
Drying Time	hrs	4
Drying Temperature	°C	80
Moisture Content	%	0.10
Dew Point	°C	0

The molded plastic part is used in the switch (Fig. 1), which performs the function of storing and recalling the memory of the driver and passenger seat settings in a modern car.

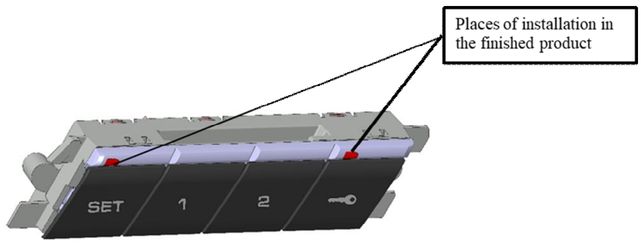

Fig. 1. 3D model of the finished product, in which an experimental plastic part was used

The part performs the function of light conduction of functional buttons, as well as being part of a decorative element. In the model of the finished product (Fig. 1), the studied polymer part is partially covered with a chrome element. The finished product in the car is exposed to various detergents and disinfectants, that contain various chemical components, in particular, ethyl alcohol, the use of which has been widespread during the COVID-19 pandemic.

The products obtained from PA12 (Fig. 2) with different process parameters were exposed to the action of the polar solvent ethyl alcohol C_2H_5OH (96.6 vol.%).

Fig. 2. General view of the parts obtained from PA12

We visually assessed warping and other geometric changes of the parts after exposure (time=10 min) in the solvent at an ambient temperature of 25 °C. The exposure time is based on the visual observation of the reaction, which was observed on average for 7-9 minutes. The overall dimensions of the part (Fig. 3) were measured using a Mitutoyo 293-821 digital micrometer, followed by processing the results and evaluating the process capability (C_{pk}) and injection molding machine capability (C_{mk}) and deviations from the nominal value.

The construction of normal deviation diagrams, calculations, and analysis of statistical data were carried out using MS Excel. The data were also evaluated using the 6SIGMA method. Limit tolerances of the overall dimensions were set at ±0.1 mm, which meets the specific requirements of the customer.

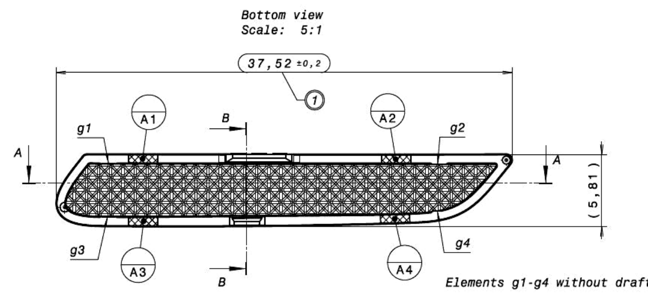

Fig. 3. The fragment of the 2D drawing of the parts made of PA12

The process parameters were adjusted gradually, in three stages, until the maximum possible effect of resistance to warping was achieved. The parts obtained from PA12 (Fig. 2) were subjected to visual inspection and measurements of the overall size of the width (Fig. 3). It was established that the change in the process parameters did not affect the visible surfaces of the parts and all quality criteria were met.

Adjustment of the main parameters of the injection molding process was carried out in several stages (Table 2).

At the first stage, only serial original parameters were used. In the following stages, the injection speed was increased by 31.4 %. To obtain better performance properties, the temperature of the cooling circuits was increased by 10 % and by 11.1 % compared to the original parameters of the injection molding process.

After each stage of adjusting the parameters, 30 units were obtained for further research in serial conditions.

All parts obtained from PA12 after proper relaxation were placed in a container with a polar solvent ethyl alcohol C_2H_5OH in a concentration of 96.6 vol.%. Under the influence of such an environment, the products were kept for 10 minutes, and then they were removed for a visual assessment of quality, assessment of overall dimensions, and chemical resistance.

Table 2. Comparison of main injection molding process parameters

Process parameters	Stage 1	Stage 2	Stage 3	Difference, %
Injection speed, mm/sec (m/sec)	48 (0.048)	70 (0.070)	70 (0.070)	31.4
Cooling circle №1, °C	90	90	100	10.0
Cooling circle №2, °C	80	80	90	11.1

3. Results and Discussion

3.1. Serial process parameters

Measurements of the critical overall size of the obtained parts were carried out. At the standard validated process parameters, the value of machine capability C_{mk} was 48.61, and the process capability C_{pk} was 23.39. The average size reaches its nominal value of 5.81 mm (confidence interval $p=0.00005<0.05$).

Fig. 4 shows that the process is stable, the spread of values is minimal, and mostly all the obtained parts have the nominal value of the overall size.

After relaxation, the products were exposed to ethyl alcohol with further evaluation of visual and geometric changes.

Fig. 5 shows the visually noticeable warping of the parts obtained from the PA12 polymer composition at standard process parameters.

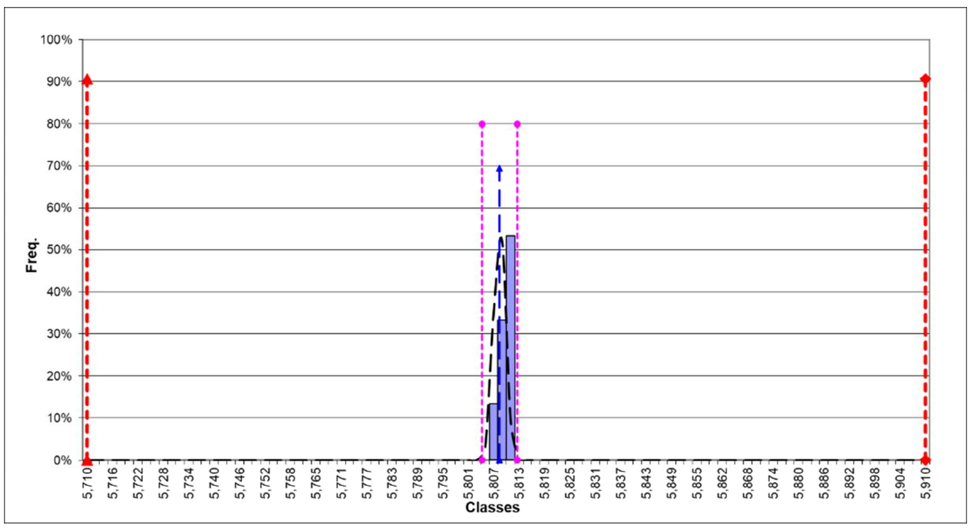

Fig. 4. Diagram of the normal deviation of the obtained results while measuring the overall dimensions of parts from PA12 on serial parameters

Fig. 5. The general view of the product from PA12 using standard process parameters after aging in C_2H_5OH 96.6 vol.%

Significant changes are observed in the products, in particular, the deformation (Fig. 5), which occurred due to the interaction of the PA12 polymer composition and the polar solvent of ethyl alcohol. This can be explained by the fact that C_2H_5OH affected the crystal structure of the material, which apparently had weak intermolecular bonds.

After exposure to the solvent, the overall dimensions were measured again. The diagram (Fig. 6) shows significant changes in the distribution of the obtained values. At the same time, the process becomes unstable, given the fact that some samples had dimensions that exceeded the critical limits, and their average value was 5.87 mm, which indicates an increase in size by 0.06 mm compared to products that were not exposed to the solvent.

Evaluating the line diagrams (Fig. 7), it can be noted that after the parts were exposed to C_2H_5OH, there was a significant increase in overall dimensions compared to the parts that were not immersed in a polar solvent. It can also be visually seen that two dimensions went beyond the permissible limits.

3.2. Changes in injection speed of the injection molding process

During the experiment, significant changes were gradually made to almost all process parameters, including processing temperature and dosage. However, after repeated immersion of the obtained parts into C_2H_5OH under the same conditions, it was possible to observe a similar effect, which is shown in Fig. 5.

The next step was to study such a parameter as injection speed. The result was parts that were unchanged and met quality criteria. The increase in injection speed occurred until the process stabilization, which reached the C_{pk} value of 27.44 and C_{mk} value of 27.11. This meets the requirements and indicates a successful validation. It was found that the injection time was reduced by 38%. At the same time, the overall size of the products obtained from PA12 did not undergo significant changes in comparison with the serial parameters of the process and reached an average of 5.809 mm ($p=0.00004<0.05$), which is only 0.009 mm larger than the nominal value and is within the permissible limits.

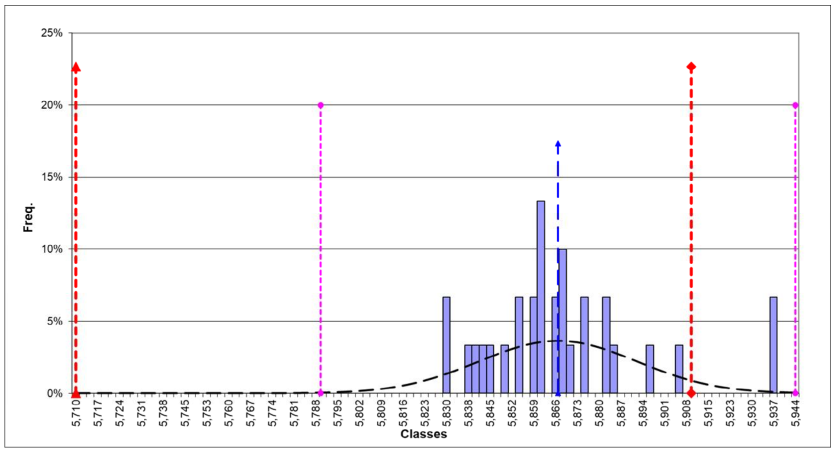

Fig. 6. Diagram of the normal deviation of the obtained results of measuring the overall dimensions of parts from PA12 on serial parameters after aging in C_2H_5OH 96.6 vol.%

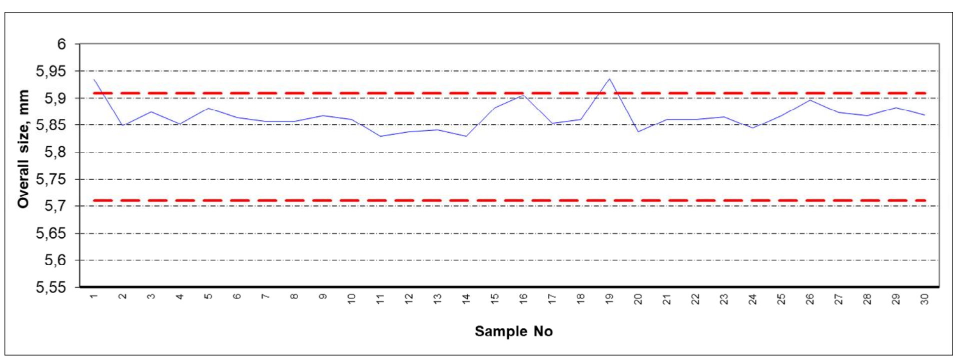

Fig. 7. Linear diagram of the obtained results of measuring the overall dimensions of parts from PA12 on serial parameters after aging in C_2H_5OH 96.6 vol.%

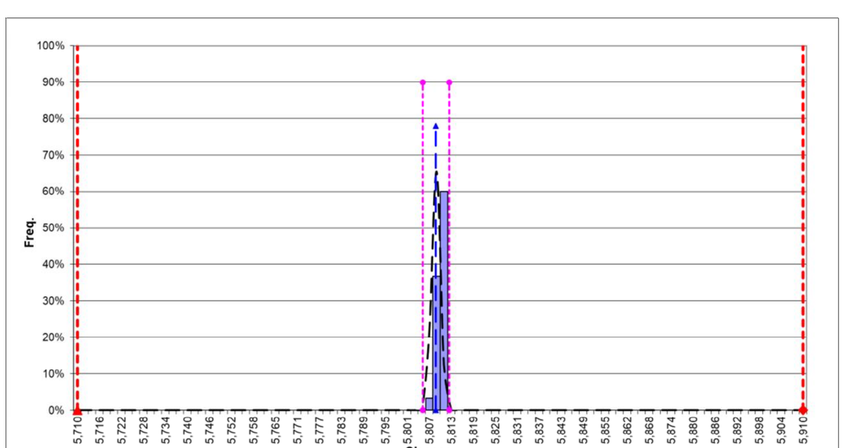

Fig. 8. Diagram of the normal deviation of the obtained results of measuring the overall dimensions of parts from PA12 with increased injection speed

Fig. 9. General appearance of the PA12 product with increased injection speed after aging in C_2H_5OH 96.6 vol.%

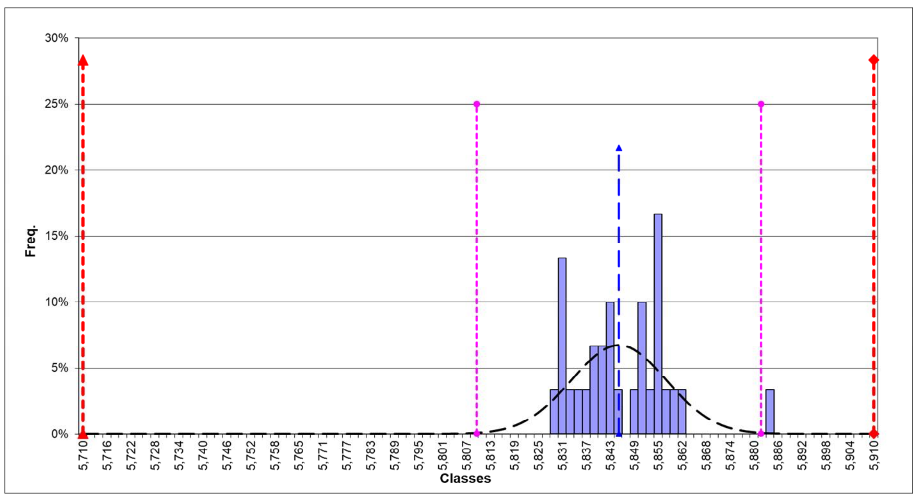

Fig. 10. Diagram of the normal deviation of the obtained results of measuring the overall dimensions of parts from PA12 with increased injection speed after exposure to C_2H_5OH 96.6 vol.%

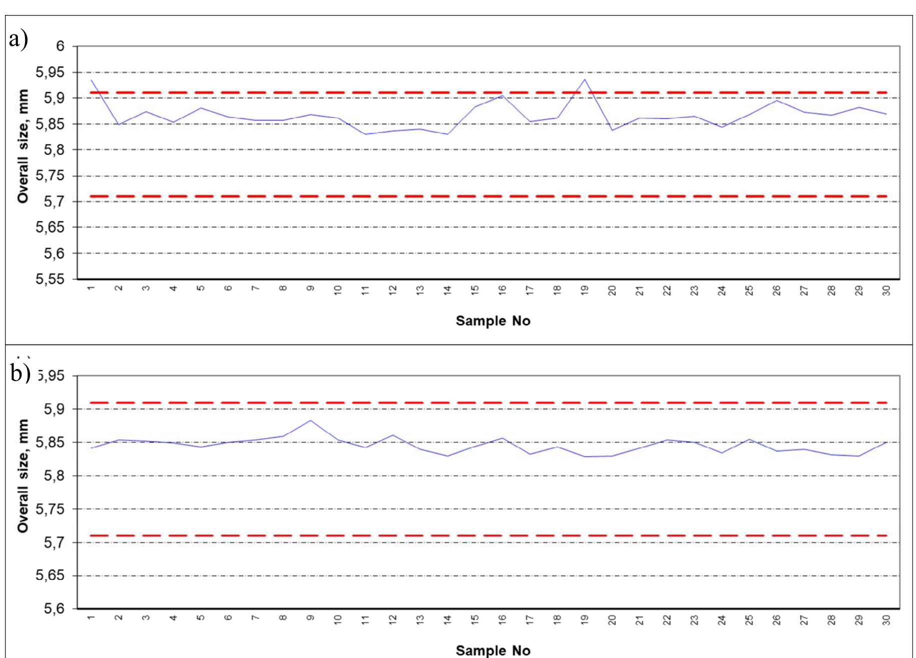

Fig. 11. Linear diagram of the obtained results of measuring the overall dimensions of parts from PA12 after aging in C_2H_5OH 96.6 vol.%: a) serial parameters: b) with changed injection speed

The normal deviation diagram (Fig. 8) shows a high level of process capability despite the changes made, where most of the estimated dimensions are close to the nominal value.

After keeping the parts under normal climatic conditions, they were exposed to a polar solvent for 10 min. It is possible to observe in Fig. 9 the warping of the components, and therefore, by changing only the injection parameters, it is impossible to achieve the required level of resistance to the polar solvent C_2H_5OH.

Fig. 10 shows the effect of the solvent on the overall dimensions of the products obtained by injection molding with a changed injection speed. It can be seen that there is no deviation of dimensions from the permissible limits, taking into account that the deviation has decreased by an average of 0.021 mm compared to the standard process parameters.Comparing the diagrams (Fig. 11), it can be observed that the overall dimensions of products obtained by the injection molding with increased injection speed and aged in C_2H_5OH are within acceptable limits (Fig. 11b) in comparison with serial parameters (Fig. 11a), where deviations of dimensions beyond the maximum acceptable limits are recorded.

3.3. Changes in the injection speed and temperature of the cooling circuits of the injection molding tool of the injection molding process

Taking into account the fact that the temperature of the cooling circles of the injection molding tool has an effect on the relaxation of the parts, it was decided to slightly increase the temperature parameters of the cooling system while maintaining the increased injection speed. After changing the temperature in the cooling circuits of the injection molding tool, the resulting parts have not changed in terms of quality criteria and are identical to those obtained at the serial parameters of the injection molding process (Fig. 3). Molded from PA12 products were exposed to the same aggressive environment in order to evaluate chemical resistance.

Therefore, as shown in Fig. 12, with optimally selected process parameters, the warping of parts is almost invisible visually. This is probably due to the fact that at a higher injection speed and at a higher temperature of the cooling system of the injection molding tool, strong chain intermolecular bonds can be achieved, which show better resistance to aggressive chemical environments, as shown by the example of the polar solvent ethyl alcohol.

Fig. 12. The general view of the parts from PA12 with increased injection speed and changed cooling temperature of the injection molding tool after aging in C_2H_5OH 96.6 vol.%

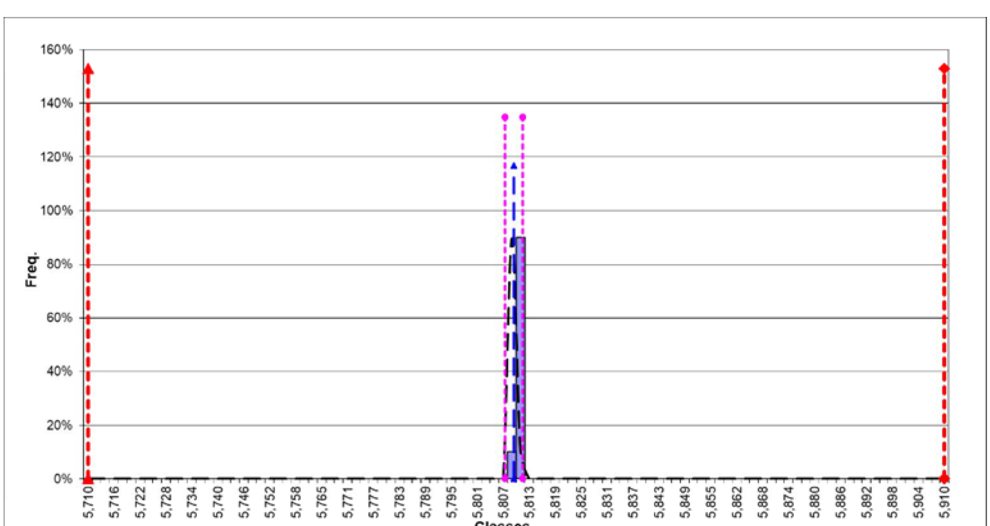

Fig. 13. Diagram of the normal deviation of the obtained results of measuring the overall dimensions of parts from PA12 with increased injection speed and increased cooling temperature of the injection molding tool

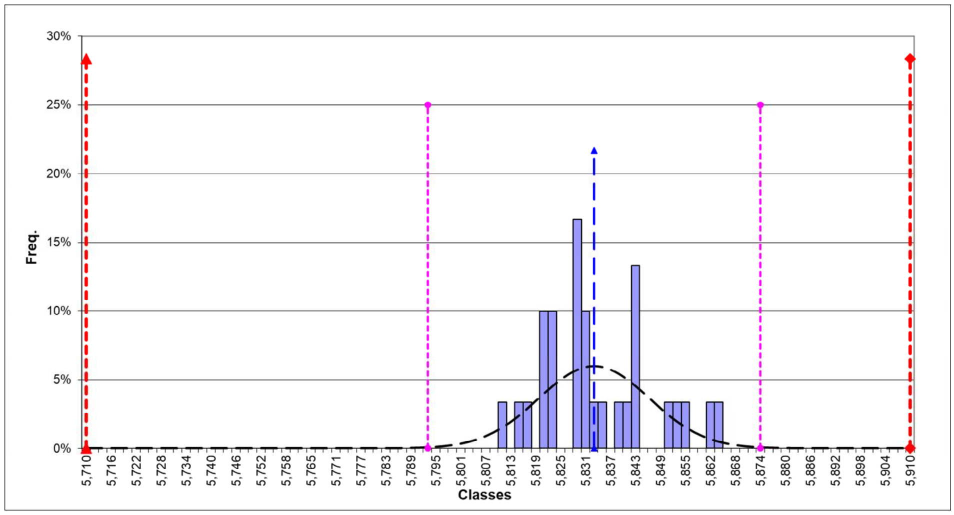

Fig. 14. Diagram of the normal deviation of the obtained results of measuring the overall dimensions of parts from PA12 with increased injection speed and increased temperature of the cooling system of the injection molding tool after exposure to C_2H_5OH 96.6 vol.%

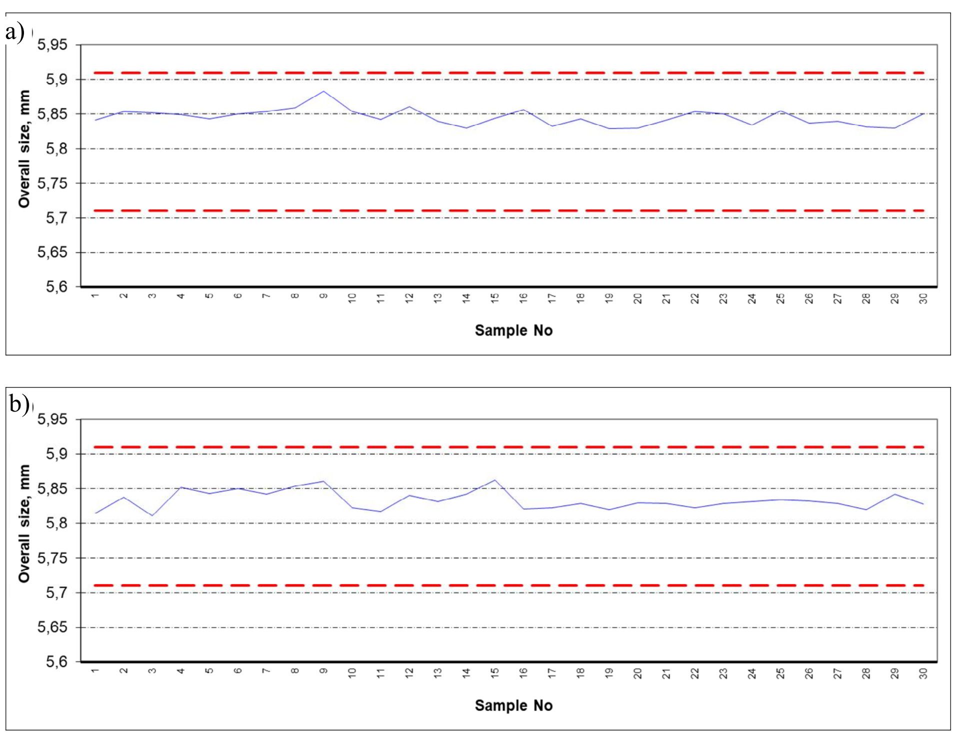

Fig. 15. Linear diagram of the obtained results of measuring the overall dimensions of parts from PA12 after exposure to C_2H_5OH 96.6 vol.%: a) with changed injection speed; b) with changed injection speed and cooling temperature of the injection-molding tool

After measuring the resulting parts and processing the data, it was established that the capability of the process C_{pk} reached its maximum of 48.61 and is equal to the capability of the machine C_{mk}, as shown in the diagram in Fig. 13. This indicates that comprehensive changes in injection molding parameters have improved stability.

To objectively confirm the visual observations of improved chemical resistance of parts made from PA12, we also measured parts aged in C_2H_5OH under the same conditions.

The standard deviation diagram (Fig. 14) shows that there are no values of the overall dimensions of the parts that are not only within the limits but also within the range of ±3σ, which in turn indicates a low probability of falling outside the permissible values. This is an important characteristic of operational properties, as it will not affect the final finished product for the direct consumer. Under these conditions, the average size was 5.833 mm ($p=0.00003<0.05$) and a deviation is smaller by 0.012 mm than that under the parameters without adjusting the temperature of the cooling circuits of the injection molding tool.

It should be noted that the response of PA12 parts obtained by injection molding to changes in the parameters of injection speed and cooling temperature of the injection molding tool (Fig. 15b) does not differ significantly from the response to parameters with changes in injection alone (Fig. 15a), but demonstrates higher performance and a smaller spread of values.

4. Conclusions

The experiment has established the influence of technological parameters on the capabilities of products obtained by injection molding from the polymer composite material PA12. It has been experimentally proven that the injection speed and cooling temperature of the injection molding tool affect the resistance of plastic parts to aggressive environments using ethyl alcohol C_2H_5OH as an example. Thus, with an increase in the injection speed by 31.4% and an increase in the temperature of the injection molding tool by an average of 10.6%, a visually lower level of part warping was recorded in two cooling circuits.

The study revealed the possibility of significant improvement in the performance characteristics of PA12 products produced by injection molding. It is proved that an increase in the injection speed by 31.4% and an increase in the cooling temperature of the injection tool in both circuits by an average of 10.6% have a positive effect not only on the chemical stability of PA12 parts but also double (207.8%) the stability of the process; C_{pk} increased from 23.39 to 48.61. It is shown that the parts obtained with optimized injection molding process parameters are almost not subject to warping under the influence of the polar solvent C_2H_5OH. It should be noted that the effect was achieved without losing the efficiency of the technological process, which in turn has a positive effect on economic indicators along with a higher level of quality and improved performance of finished products where plastic parts are used. This also has a significant impact on environmental aspects, given that material that has not been destroyed during prolonged use can be recycled. Thus, during product and process validation, by assessing the possibilities for improving the characteristics of process parameters, it is possible to ensure a preventive reduction in the waste of non-recyclable polymer composite materials.

However, it is worth noting that the data on such experiments are limited, and the issue of the influence of process parameters on chemical resistance requires further research.

The results obtained can significantly improve the level of quality, efficiency, and performance characteristics of injection molded parts in mass production at minimal cost. This makes it possible to proactively positively influence environmental aspects by reducing rejects by including such tests in validation plans at the initial stages of the product life cycle and revalidating existing serial processes where such characteristics are important to the consumer.

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РЕГУЛЮВАННЯ ЕКСПЛУАТАЦІЙНИХ ВЛАСТИВОСТЕЙ ВИРОБІВ ОТРИМАНИХ МЕТОДОМ ЛИТТЯ ПЛАСТМАС ПІД ТИСКОМ З ПОЛІАМІДУ 12

Анотація. *Досліджено та показано вплив параметрів технологічного процесу на стійкість до агресивного середовища (етилового спирту* C_2H_5OH*) пластикових деталей, які були отримані методом лиття пластмас під тиском з полімерного композиційного матеріалу поліаміду 12 (PA12).*

Ключові слова: *лиття пластмас під тиском, параметри процесу, стабільність процесу, агресивне середовище, хімічна стійкість, полярний розчинник, поліамід.*