EFFECT OF RELATIVE HUMIDITY TO TRIBOLOGICAL AND MECHANICAL PROPERTIES OF POLYMER COMPOSITES

Lucia Boszorádová, Martin Baráth*, Martin Kotus, Rastislav Bernát, Zoltán Záležák

Institute of Design and Engineering Technologies, Faculty of Engineering,
Slovak University of Agriculture in Nitra, Tr. A. Hlinku 2, 949 76, Nitra, Slovakia

Abstract

Additive technologies are often used as an alternative to conventional methods for prototyping and one-off production of parts. When designing parts, it is necessary to take into account not only the properties of the materials declared by the manufacturer, but also the change in the properties of the materials due to the environment to which the finished part will be exposed. Therefore, the present paper deals with the effect of relative humidity of the environment on the tribological and mechanical properties of PA6 and its carbon and glass fibre reinforced composites. Using FDM technology, test specimens were fabricated from the materials under investigation. The specimens were exposed to environments with relative humidity levels ranging from 33% to 95%. Tribological and mechanical tests showed a significant effect on the properties of the materials. Increasing the relative humidity of the environment resulted in a decrease in wear resistance, the average weight loss increased at the highest relative humidity by 17 % - 24 %. For mechanical properties, a decrease in stresses of 15 % - 35 % was observed at the highest relative humidity.

Keywords: *additive technologies, abrasive wear, polymer material, relative humidity*

1. INTRODUCTION

Additive manufacturing processes can affect material properties such as strength, elasticity, heat resistance, etc. For example, different additive manufacturing technologies can have different effects on the microstructure of a material, which can affect its properties.

Additive technologies have become an alternative to traditional methods in prototyping and one-off parts production. When designing these parts, it is essential to consider not only the declared material properties by the manufacturers, but also the potential changes in material properties due to the environment to which the parts will be exposed (Ngo et al.,2018). This aspect is the focus of the present paper, which deals with the influence of the relative humidity of the environment to the tribological and mechanical properties of PA6 and its carbon and glass fiber reinforced composites.

A large amount of research in the field of polymer composite materials produced by additive technologies (3D printing) is primarily concerned with their mechanical properties. Research on mechanical properties allows the development of new polymer blends, the addition of different reinforcement materials and the optimization of printing processes to achieve the desired mechanical characteristics. However, as additive technologies gradually improve and become more widely adopted, researchers are increasingly focusing on other aspects of composite materials produced by additive technologies (Zaldua et al., 2019).

Zaghloul et al. (2023) in their research analyzed the tribological properties of Polyamide 6 (PA6) composites with emphasis on the PV factor, which is a consequence of velocity and pressure. They investigated the friction and wear of PA6 with the addition of glass fibers. The results showed that PA6 was advantageous up to intermediate PV values, but PA6 with 25% glass fibers (PA625GF) outperformed PA6 by a significantly better PVlimit of 1.04 MPa m/s. PA625GF demonstrated excellent tribological properties in the abrasive wear regime under extreme conditions with a low specific wear coefficient and a PVsafe value of 0.78 MPa.m/s. Overall, the glass fibers were beneficial for the wear resistance and performance of the composite under severe conditions.

In an experimental study by Kumar and Pannerselvam (2015), the mechanical and tribological properties of pure PA6 and glass fiber reinforced (GFR) PA6 composites were investigated as a possible alternative to metals. Their results showed that the coefficient of friction and specific wear rate of the PA6 composites decreased with increasing glass fiber content, with the optimum result achieved at 30% glass fiber content. It was also found that the coefficient of friction of GFR PA6 decreased with increasing load and sliding speed, while the specific wear rate increased with increasing load and friction speed. The applied load had a greater effect on the friction coefficient, while the friction speed was the key factor for the specific wear rate.

Myalski et al. (2020) investigated the effect of different types of carbon fiber fillers on the tribological and manufacturing characteristics of thermoplastic polyamide PA6. The results showed that the addition of graphene oxide to the PA6 matrix resulted in improved tribological properties, which was reflected in lower wear and friction coefficient. The graphene oxide-containing composite showed significantly better properties than the other variants, although it was more difficult to produce. Microstructural studies also indicated that the adhesion between the matrix and carbon fillers influenced the tribological properties, with graphene oxide and glassy carbon having a greater influence than bio-carbon.

2. MATERIALS AND METHODS

2.1. Materials

Several materials will be used in the experiments. The paper is concerned with investigating the effect of the concentration and type of reinforcing fiber on the changing relative humidity of the environment to which the materials are exposed. The specific materials investigated are PA6 and its glass and carbon fiber reinforced composites.

Spectrum PA6 Low Warp is an advanced material used in additive manufacturing, particularly in Fused Deposition Modeling (FDM) technology. It excels in its ability to minimize deformation during printing, thereby improving the accuracy and quality of the parts created.

BASF Ultrafuse PA6 GF30 is a high-performance material developed by BASF for additive manufacturing. It contains 30% glass fiber, which gives it excellent strength and stiffness, making it ideal for applications requiring durability and stability.

PA6 CF15 is a polymeric material based on polyamide 6, which is reinforced with 15 wt. % carbon fibers. These fibers give it excellent strength and lightness, making it ideal for applications where a combination of strength and light weight is key.

2.2. Methods

The test samples were produced using 3D printing technology. A Prusa Mk3S+ printer using FDM technology was used, in which the thermoplastic filament is heated and applied layer by layer to create the final 3D object. Table 1 shows parameters used for printing specimens.

	PA6	**PA6 CF15**	**PA6 GF30**
Nozzle temperature	260 °C	275 °C	270 °C
Bed temperature	90°C	unheated	100°C
Layer height	0,2mm	0,2mm	0,2mm
Nozzle diameter	0,4mm	0,4mm	0,4mm
Infill	solid	solid	solid
Number of perimeters	2	2	2

Table 1. Specimens printing parameters

The specimens for testing the tribological properties were in the shape of a cylinder with a diameter of 10 mm and a length of 60 mm. The orientation of the fiber was chosen to be perpendicular to the surface of the abrasive Fig 1.

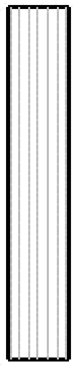

Fig. 1. Orientations of the layers used in the wear resistance tests (orientation perpendicular to the abrasive surface)

To investigate the dependence of individual material properties on the relative humidity of the environment to which the material was exposed, the samples were conditioned in desiccators. Environments with the desired relative humidity were created in the desiccators using saturated salt solutions. The following saturated salt solutions were used in the experiments:

- magnesium chloride solution $MgCl_2$ with 33% relative humidity ,
- ammonium nitrate solution NH_4NO_3 with 65% relative humidity,
- potassium chloride solution KCl with 85% relative humidity,
- potassium nitrate solution KNO_3 with 95 % relative humidity.

Every 24 hours the weight of the samples was checked, if it did not change its weight by more than 0.1% in 24 hours the samples were considered saturated.

The testing equipment (Fig.2) works with the Pin on Disc test geometry. As part of the test, the specimen is pressed by a normal force against a rotating disc on which a bonded abrasive is placed. Moving at a speed of 0.5 m/s, the disc has a diameter of 490 mm and the sample travels a path of 50 m. The machine feed ensures the movement of the sample from the center of the disc to its edge, the test being terminated when the sample has passed the defined path. The normal force is adjusted by means of interchangeable weights of a well-defined mass, the mass of the weights including the mass of the specimen holder. The abrasive material consists of Germaflex X abrasive cloth with a grit size of P120, containing corundum grain. The test rig is shown in Fig.2 and its main parts are the sample holder head, sample holder, weights, feed mechanism, and rotating disc with bonded abrasive, control. The main technical parameters of testing machine are given in Table 2.

Fig. 2. Testing machine

Average speed	**0.5 m.s^{-1}**
Feed per revolution	3 mm
Diameter of the friction disc	480 mm
Specific pressure	0.51 N. mm $^{-2}$
Length of the test specimen	60 mm
Diameter of the test specimen	10 mm
Grit size of the abrasive disc	P120

Table 2. Technical parameters of the testing machine

The evaluation of the wear resistance of the material was carried out by detecting the weight loss of the test specimen during the test. The weight loss was calculated according to the following relation:

$$\Delta m = m_0 - m_1 \ (g) \qquad (1)$$

Where: Δm – weight loss (g),

m_0 – the weight of the sample before the test (g),

m_1 – the weight of the sample after the test (g).

In order to eliminate random errors, each experiment was performed three times in succession and then the average weight loss of the test samples was calculated using Eq:

$$\Delta m_p = \frac{\sum_1^n \Delta m}{n} \qquad (2)$$

where: Δm_p – average weight loss of samples (g),

Δm – weight loss of samples (g),

n – number of repetitions of the test.

We use regression analysis as a statistical method to examine the relationship between one or more independent variables and one dependent variable.

3. RESULTS

The specific materials investigated are PA6 and its glass and carbon fiber reinforced composites. In Fig.3 we can see the dependence of the average mass loss on the relative humidity of the environment for the different materials investigated. These dependencies are described by regression functions whose coefficients are given in Table 2. A polynomial regression function was used to construct these dependencies:

$$Y = B_0 + B_1X + B_2X^2 \qquad (3)$$

Where: Y – dependent variable (average weight loss),

X – independent variable (relative humidity),

B_0 – regression coefficient,

B_1 – regression coefficient,

B_2 – regression coefficient.

The wear resistance of PA6, PA6 GF30 (glass fiber-reinforced), and PA6 CF15 (carbon fiber-reinforced) was assessed through controlled experiments at different levels of relative humidity (33%, 65%, 85%, and 95%). The results showed a clear trend where increasing relative humidity led to higher weight loss in all materials tested. The pure PA6 samples showed an average weight loss that increased steadily with humidity. At 33% relative humidity, the weight loss was around 1.2 grams, while at 95% relative humidity, it increased to approximately 1.5 grams. This indicates a significant susceptibility to moisture-induced degradation. The glass fiber-reinforced PA6 exhibited higher weight loss compared to pure PA6, particularly at higher humidity levels. Starting at about 1.1 grams at 33% humidity, the weight loss rose to around 1.6 grams at 95% humidity. The increase in weight loss with humidity suggests that the glass fibers, while improving mechanical strength, might contribute to greater material degradation under humid conditions. The carbon fiber-reinforced PA6 showed the lowest weight loss across all humidity levels, indicating superior wear resistance. At 33% humidity, the weight loss was around 0.9 grams, increasing to about 1.2 grams at 95% humidity. The smaller increase compared to PA6 and PA6 GF30 suggests that carbon fibers provide better stability and resistance to wear in humid environments. These findings are illustrated in the provided graph, where the trend lines clearly depict the increasing weight loss with higher humidity for each material. The regression analysis results, detailed in Table 3, show that the polynomial regression functions fit the data well, with R^2 values indicating a strong correlation between humidity and weight loss for each material.

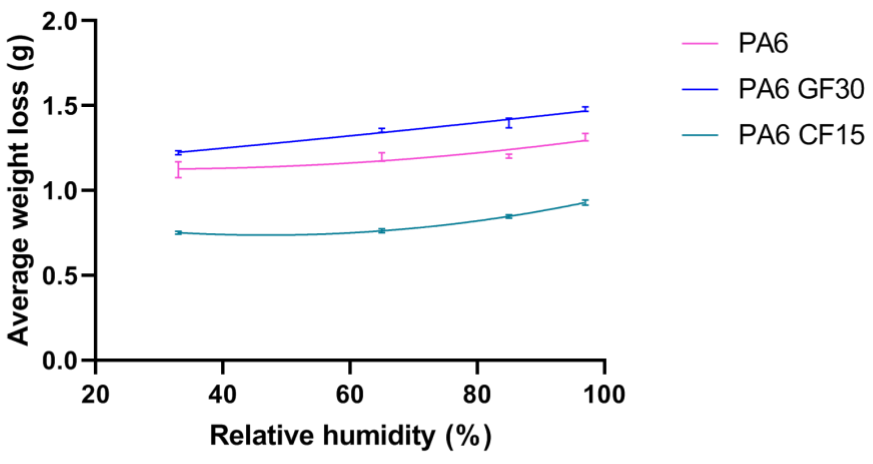

Fig. 3. Dependence of the average mass loss of samples from selected materials on the relative humidity of the environment

Materials	Regression coefficients			R^2(-)
	B_0	B_1	B_2	
PA6	1.1550	-0.002014	$3.562 \cdot 10^{-5}$	0.7779
PA6 GF 30	1.1160	0.003108	$5.337 \cdot 10^{-6}$	0.9550
PA6 CF 15	0.9043	-0.007158	$7.644 \cdot 10^{-5}$	0.9822

Table 3. Regression function coefficients and detrending coefficients of selected materials

The study also evaluated the mechanical properties, specifically the ultimate tensile stress, of the materials at different humidity levels. The results showed (Table 4) a marked decrease in tensile strength with increasing humidity, reflecting the negative impact of moisture on material integrity. The tensile strength of pure PA6 decreased significantly with increasing humidity. At 33% relative humidity, the ultimate tensile stress was 72.816 MPa. This value dropped to 54.549 MPa at 95% humidity, indicating a 25% reduction. This substantial decrease underscores the vulnerability of pure PA6 to moisture-induced weakening. The glass fiber-reinforced composite demonstrated the highest tensile strength among the materials tested. Starting at 102.866 MPa at 33% humidity, the tensile stress decreased to 87.63 MPa at 95% humidity, representing a 15% reduction. Despite the reduction, PA6 GF30 maintained higher tensile strength than the other materials across all humidity levels, highlighting the reinforcing effect of glass fibers. The carbon fiber-reinforced composite showed the highest decrease in tensile strength with increasing humidity. From 54.651 MPa at 33% humidity, the tensile stress reduced to 35.296 MPa at 95% humidity, a 35% reduction. Although the absolute values were lower than those for PA6 GF30 and pure PA6. PA6 GF30 maintained better mechanical performance than pure PA6 and PA6 CF15 under high humidity conditions.

Relative humidity	Ultimate tensile stress (MPa)		
	PA6	PA6 GF30	PA6 CF15
33%	72.816	102.866	54.651
65%	72.215	95.421	35.099
85%	63.348	106.437	37.528
97%	54.549	87.63	35.296

Table 4. Average measured stress values at the highest strength of specimens from selected materials

4. DISCUSSION

In most cases, the dependence of concentration effects has not been examined on a broader scale. None of the authors have investigated these dependencies at multiple environmental relative humidity. For example, the authors Kumar and Panneerselvam (2016) were concerned with evaluating the wear resistance of PA6 and PA6 glass fiber reinforced materials. Laboratory wear resistance tests were carried out using a bonded abrasive in the form of abrasive cloth, with a grit size of P320. Their results showed a decrease in the mass loss of the specimens with an increase in the content of glass fiber reinforcement in the material. By increasing the glass fiber content from 0% to 30%, they achieved a reduction in the mass loss of the samples of approximately 49%. Preliminary results from our experiment indicate that the presence of glass fibers in the material increased the average mass loss of the material by approximately 14%. This increase in average mass loss may be due to the different abrasive grain size. When we used a coarser abrasive with P120 grit, it could have caused the reinforcing fibers to delaminate and reduce the contact area with the abrasive. As also the results of the research by Zsidai and Katai (2016), which dealt with the comparison of wear resistance of PA6 and PEEK composites with different compositions. In their experiments, they used an abrasive with a grain size of P100. Their results show significantly higher wear resistance of the material containing glass fiber versus the PA6 material without reinforcing fibers.

5. CONCLUSIONS

The study investigated the effect of relative humidity on the tribological and mechanical properties of PA6 and its composites reinforced with glass and carbon fibers. The results demonstrated that environmental relative humidity significantly impacts the wear resistance and mechanical strength of these materials. Increasing relative humidity led to a notable increase in average weight loss for all tested materials, indicating reduced wear resistance. PA6 CF15, the composite with carbon fibers, exhibited the lowest weight loss, demonstrating superior wear resistance compared to both pure PA6 and PA6 GF30, the composite with glass fibers. At the highest relative humidity level (95%), the average weight loss increased by 17% to 24%, highlighting the detrimental effect of high humidity on the wear properties of these materials. A decline in ultimate tensile stress was observed for all materials as relative humidity increased. This decline was most pronounced in the PA6 CF15, followed by pure PA6, and least in PA6 GF30. PA6 GF30 consistently exhibited the highest ultimate tensile stress across all humidity levels, underscoring the reinforcing effect of glass fibers on mechanical strength. The reduction in mechanical strength ranged from 15% to 35% at the highest humidity level, emphasizing the significant impact of moisture on the structural integrity of these composites. The inclusion of reinforcing fibers markedly influenced the properties of PA6. Carbon fiber reinforcement (PA6 CF15) proved more effective in enhancing wear resistance, whereas glass fiber reinforcement (PA6 GF30) significantly improved mechanical strength. Regression analysis revealed the reinforcing fibers substantial role in determining the material properties, with carbon fibers providing better wear resistance and glass fibers offering higher tensile strength. The results indicate that the choice of reinforcement (carbon vs. glass fibers) should be tailored to the specific performance requirements of the application, whether it is wear resistance or mechanical strength. The study emphasizes the importance of considering environmental conditions, particularly relative humidity, when designing and selecting materials for additive manufacturing applications.

ACKNOWLEDGMENTS

This work was supported by VEGA, grant number 1/0691/23.

REFERENCES

1. Ngo, T, Kashani, A, Imbalzano, G, Nguyen, T.Q, Hui, D 2018, "Additive manufacturing (3D printing): A review of materials, methods, applications and challenges", *Composites Part B*, vol. 143, pp. 172-196.
2. Zaldua, N, Maiz, J, Calle, A, García-Arrieta, S, Elizetxea, C, Harismendy, I, Tercjak, A, Müller, A 2019, "Nucleation and Crystallization of PA6 Composites Prepared by T-RTM: Effects of Carbon and Glass Fiber Loading", *Polymers 11,* no. 10: 1680.
3. Kumar, S, Panneerselvam, K 2015, "Research on Tribological Behaviors of Pure and Glass Fiber Reinforced Nylon 6 Composites against Polymer Disc", *Journal of Material Science and Mechanical Engineering,* vol.2, no. 6, pp. 24-28.
4. Zaghloul M, Steel, K,Veidt, M, Heitzmann, M 2023, " Assessment of the tribological performance of glass fibre reinforced polyamide 6 under harsh abrasive environments", *Tribology International,* vol. 191.
5. Myalski, J, Godzierz, M, Olesik, P 2020, "Effect of Carbon Fillers on the Wear Resistance of PA6 Thermoplastic Composites",*Polymers* no.10, 2264.

6. Kumar, S, Panneerselvam, K 2016, "Two-body Abrasive Wear Behavior of Nylon 6 and Glass Fiber Reinforced (GFR) Nylon 6 Composite", *Procedia Technolog*, pp 1129-1136.
7. Zsidai, L; Kátai, L 2016, " Abrasive wear and abrasion testing of PA 6 and PEEK composites in small-scale model system ", *Acta Polytechnica Humgarica*, pp 197-214.