Effect of moisture on breakdown strength and glass transition temperature of bio-polyamide reinforced with glass or basalt fiber

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Abstract: In this study we investigate the effect of moisture content on the dielectric breakdown strength and glass transition temperature (T_g) of bio-polyamide 4.10 reinforced with glass or basalt fiber (15, 30, 50 wt%). Moisture absorption decreased with increasing fiber content, regardless of fiber type. The reduction in breakdown voltage (BDV) was more pronounced in the composites with higher moisture content reinforced with basalt fiber. This may be due to weaker interactions at the interface. The presence of moisture decreased the glass transition temperature of the composites. PA 4.10, especially reinforced with glass fiber, is characterized by better performance properties than PA 6.6 reinforced with 50 wt% glass fiber in applications requiring both stability of mechanical properties and dielectric properties.

Keywords: bio-polyamide, moisture absorption, glass transition temperature, dynamic mechanical analysis, breakdown strength.

Wpływ wilgoci na wytrzymałość na przebicie i temperaturę zeszklenia bio-poliamidu wzmocnionego włóknem szklanym lub bazaltowym

Streszczenie: W pracy zbadano wpływ zawartości wilgoci na wytrzymałość na przebicie i temperaturę zeszklenia (T_g) bio-poliamidu 4.10 wzmocnionego włóknem szklanym lub bazaltowym (15, 30, 50% mas.). Absorpcja wilgoci zmniejszała się wraz ze wzrostem zawartości włókna, niezależnie od jego rodzaju. Zmniejszenie napięcia przebicia (BDV) było bardziej widoczne w kompozytach o większej zawartości wilgoci wzmocnionych włóknem bazaltowym. Może to być spowodowane słabszymi oddziaływaniami na granicy faz. Obecność wilgoci obniżyła temperaturę zeszklenia kompozytów. PA 4.10, szczególnie wzmocniony włóknem szklanym, charakteryzuje się lepszymi właściwościami użytkowymi niż PA 6.6 wzmocniony 50% mas. włókna szklanego w zastosowaniach wymagających zarówno stabilności właściwości mechanicznych, jak i właściwości dielektrycznych.

Słowa kluczowe: bio-poliamid, chłonność wody, temperatura zeszklenia, dynamiczna analiza mechaniczna, wytrzymałość na przebicie.

Electrification, which is the process of supplying an area or facility with electrical energy or introducing electrical energy into a specific sector of the economy, is a crucial element of sustainable economic, social, and technological development. Products for the electrification sector encompass a wide range of devices and technologies that enable the generation, transmission, distribution, and utilization of electrical energy. The main categories of products related to electrification include generators; transformers; switchgears and power distribution systems; wires and cables; electric vehicle charging infrastructure; energy storage systems; protective and energy management devices/systems; and end-use devices.

Switchgears are responsible for controlling the flow of electricity and protecting the power grid by disconnecting and isolating its sections in case of failures. They are used in low voltage (LV; up to 1000 V), medium voltage (MV; above 1 kV up to 52 kV), and high voltage (HV; above 52 kV) networks. Medium voltage (MV) switchgears play a key role in the primary and secondary distribution of electrical energy [1], supplying power to public and industrial facilities, including very demanding applications such as power plants, oil refineries, gas industries, mining, maritime industries, and data centers.

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According to an analysis by Maximize Market Research [2], the global MV switchgear market was valued at USD 42.87 billion in 2023, and total MV switchgear revenues are expected to grow at an annual rate of 6.2% from 2024 to 2030, reaching USD 65.32 billion. The basic components of medium voltage switchgear include circuit breakers, switches, disconnectors, and earthing switches. Other significant elements are busbars, which conduct current within the switchgear, post insulators, and instrument transformers/sensors (current and voltage), which provide signals to measuring and protection devices.

Insulating materials are crucial for ensuring safe and reliable operation of MV switchgear. The choice of insulating material significantly impacts the environment, as each stage of the material's lifecycle – from raw material extraction, through production and use, to disposal – carries various ecological consequences. Thermosetting materials, such as epoxy resins, have been used for over 70 years [3-5], due to their excellent insulating, mechanical, and chemical properties. Despite many advantages, their use poses several environmental challenges. They are produced using petrochemical raw materials and chemical processes that can generate waste and harmful emissions. Additionally, due to highly crosslinked structure of material the epoxy resin products have limited recycling options (resulting in landfill disposal or incineration). Therefore, the thermosetting materials are increasingly being replaced by engineering thermoplastic composites. These materials offer benefits, such as low density, high rigidity and strength, good insulating properties, and corrosion resistance. Moreover, thermoplastic composites can be produced from renewable resources, processed in significantly shorter times, and can be recycled at the end of their lifecycle. This is highly significant for the environment and supports the goal of achieving a circular economy.

Engineering thermoplastic composites firstly emerged in LV switchgears. Thangarajan *et al*. [6, 7] studied performance of polyamide PA 6.6 and PA 4.6 in application for Moulded Case Circuit Breaker (MCCB). Laboratory testing showed the thermoplastic insulation to have better arc quenching performance (increased arc voltage and lower fault clearing time) due to "off-gassing" property of thermoplastic insulation compared to thermosetting material (Dough molding compounds – DMC). Selected PA 4.6 reinforced with glass fibers also helped to reduce carbonization during arcing and resulting in longer lifetime of the switchgear. Materials based on renewable resources in electrical engineering, with particular focus on biodegradable polylactide acid (PLA), have been discussed in [8]. However, use of biodegradable materials is limited due to expected long lifetime of switchgears. Petersson *et al*. [9, 10] evaluated biobased polyamides PA 4.10 and PA 6.10 as insulation material for housing of Residual Current circuit Breaker with Overcurrent protection (RCBO, a LV DIN-Rail product). They showed that biobased compounds can deliver outstanding results for RCBO. The moisture absorption of biobased grades was at least 50% lower compared to referenced PA 6 and breaker prototypes passed required type testing, what proved that durable biobased polyamide can be successfully implemented in low voltage applications. Switching to a partially biobased polyamide, with reduced CO_2 equivalent by around 40% (3.4 CO_2 eq. (100 years)/kg of resin) vs. fossil-based polyamide, will cut the emission of CO_2 equivalents by 250 metric tons per year.

En early adoption of engineering thermoplastic composites in MV switchgear has been described by Fugel *et al*. [11]. Epoxy resin has been replaced by thermoplastic material for the embedded pole comprising the vacuum interrupter, a vital component of the MV circuit breaker. Use of the thermoplastic material reduced the weight of the complete pole by approximately 35% compared with thermosetting version, while improving mechanical strength and low temperature performance. Furthermore, the production of thermoplastic poles has reduced CO_2 emissions by more than 50% compared to their predecessors, equating to a reduction of approximately 3,000 metric tons of CO_2 per year. Shen [12] described the application of an engineering thermoplastic material in 12 kV withdrawable switchgear, used in circuit breaker, solid sealing pole, contact box, bus bushing and insulators. The study focused on: the selection of insulation material, the design of insulation components, as well as the key technical points of the engineering thermoplastics in 12 kV withdrawable switch. There are a number of research activities studying properties of engineering thermoplastics for MV applications [13-16], but focusing on fossil-based materials.

The goal of this research was to determine the properties of biobased polyamide modified with glass or basalt fibers, considering different degrees of filling, and compare them with the properties of fossil-based polyamide. The selection of reinforcing fibers was deliberate. Glass and basalt fibers share similarities in chemical composition and physical properties, as well as in their production processes. However, unlike most E-glass fibers, their production does not require additives or ingredients harmful to health and the environment, such as boron and fluorine oxides [17]. While glass fibers are a common choice for reinforcing polyamides, basalt fibers have proven to be a valuable alternative [18-20]. Chopped glass or basalt fibers were incorporated into biopolyamide matrix at concentrations of 15, 30, and 50 wt%. Polyamides, especially aliphatic ones (PA 6, PA 6.6), have a polar structure that readily absorbs water from the environment. Water molecules have a plasticizing effect by weakening the hydrogen bonds between adjacent polymer chains. Additionally, the incorporation of moisture into the polyamide matrix results in a remarkable decrease in the degree of crystallinity [21]. This significantly impacts the properties of polyamide materials, influencing their mechanical and morphological characteristics. Absorbed moisture, primarily by the amorphous phase [22], also

decreases the glass transition temperature of polyamides [25, 26]. The drop in T_g is a critical factor in mechanical performance, as it leads to a decrease in tensile modulus and tensile strength, making the material less stiff and strong.

The analysis focused on determining the impact of moisture on dielectric breakdown strength and glass transition temperature using dynamic mechanical analysis (DMA) to assess the feasibility of replacing fossil-based polyamide composites with the studied composites based on polyamide from renewable resources. Compounded thermoplastic composites were characterized with regard to filler content, fiber fragmentation, mass flow rate, as well as the basic mechanical and low-cycle dynamic properties, and the findings were discussed in reference [25].

Therefore, in this work we investigated the effect of moisture content on the dielectric breakdown strength and glass transition temperature of biopolyamide reinforced with glass or basalt fibers. The properties of the obtained composites were compared with those of fossil-based polyamide. Partially made (68%) from renewable sources (castor oil) biobased polyamide 4.10 was selected as the matrix material.

T a b l e 1. Characteristic of polyamide 4.10 and polyamide 6.6

Property	Polyamide 4.10		Polyamide 6.6
Glass fiber content, wt%	–	30	50
Density, g/cm^3	1.16	1.40	1.56
MFR, g/10 min 275°C, 5 kg	91	45	30
Melting temperature, °C	250	250	260

T a b l e 1. Description of the manufactured composites and reference materials

Sample	Polyamide	Fiber content, wt%	
		Glass	Basalt
REF	PA 4.10	–	–
REF30		30	–
G15		15	–
G30		30	–
G50		50	–
B15		–	15
B30		–	30
B50			50
REF50	PA 6.6	50	–

EXPERIMENTAL PART

Materials

Bio-based polyamide 4.10 with the trade name EcoPaXX was supplied by Envalior, Düsseldorf, Germany (Table 1). PA 6.6 filled with 50 wt.% glass fiber with the trade name Ultramid was purchased from BASF, Ludwigshafen, Germany (Table 1). Glass fiber (Glass Chop Vantage HP 3610) with a diameter of 10 μm and nominal cut length of 4.5 mm, density of 2.54 g/cm^3, tensile strength of 2.4 GPa, and Young's modulus of 76 GPa was provided by Nippon Electric, Otsu, Japan. The basalt fiber (BSC13-3.2-KV02M) with a diameter of 13 μm and nominal cut length of 3.2 mm, density of 2.67 g/cm^3, tensile strength of 2.7 GPa and Young's modulus of 85 GPa was supplied by Basaltex, Wevelgem, Belgium.

Preparation of specimens

Composite pellets were obtained using a compounding line with a Steer Omega 20H (Bengaluru (Bengaluru), India) co-rotating twin-screw laboratory extruder (D = 20 mm, L/D = 44). The process was carried out at 260°C, and the screw speed was 90 rpm. The classic melt-compounding was used, where chopped fibers were introduced into the plasticized matrix using a side volumetric feeder. Biopolyamide was reinforced by introducing the fibers in amounts of 15, 30, and 50 wt%. Further details regarding the compounding process can be found in [25]. Table 1 presents a list of obtained composites and reference materials.

Polyamides were dried using vacuum technology with a Maguire LPD 100 (Aston, Pennsylvania, USA) at 80°C for 12 hours. Specimens were injection molded on a KraussMaffei KM 200 CX-380 injection molding machine (Munich, Germany) with a 30 mm diameter screw. The injection parameters were selected according to the manufacturer's recommendation and are summarized in Table 2. Two types of specimens were molded: flat plates with dimensions of 120 ´ 120 mm and a thickness of 1 mm for electrical breakdown strength measurements, and standard type ISO 527 1A specimens, from which specimens for DMA were cut to a length of 60 mm.

Molded specimens were dried in a vacuum oven, BMT Vacustation 111 (Brno, Czech Republic). At least 360 h ?? of drying was necessary to reach the constant weight of specimens at 80°C under vacuum (5 mbar). Conditioning was carried out in a climatic chamber (Angelantoni Test Technologies ACS DY200C, Massa Martana, Italy), and two levels of moisture were considered: conditioned, equilibrium moisture concentration in air with 50% relative

T a b l e 3. Injection molding parameters

Melt temperature, °C	Mold temperature, °C	Injection speed, cm^3/s	Packing pressure, bar	Packing time, s	Cooling time, s
290	80	90	500	10	20

humidity at 23°C; high humidity, equilibrium moisture concentration in air with 98% relative humidity at 85°C.

Specimens were weighed on a Radwag AS 310.X2 PLUS (Radom, Poland) scale before and after conditioning to determine the amount of moisture absorbed.

Methods

The dielectric strength was measured according to IEC 60243-1 standard using two cylindrical electrodes of different diameters with rounded edges having a radius of 3±0.2 mm. One of the electrodes had a diameter of 25±1 mm and a height of approximately 25 mm. The other electrode had a diameter of 75±1 mm and a height of approximately 15 mm. The electrodes were positioned axially with a tolerance of 2 mm. A short-term, rapid voltage rise test was selected, during which the AC voltage (50 Hz) is uniformly increased from zero until the sample breakdown. The voltage was increased at a rate of 2000 V/s, which is recommended by the standard for injection-molded samples. Measurements were conducted on plates with a thickness of 1±0.1 mm. Breakdown voltage was determined from the median of at least five test results. In case any test result deviated by more than 15% from the median, five additional tests were conducted, and breakdown voltage was determined from the median of the 10 results.

The entire assembly of electrodes and specimen was immersed in mineral oil to avoid partial discharges and surface flashovers during the test. The impact of moisture conditioning and elevated temperature on electrical breakdown strength was also evaluated.

Three-point flexural dynamic mechanical analysis (DMA) was conducted using a Netzsch Artemis DMA 242 E (Selb, Germany) to determine the glass transition temperature in dry and conditioned states. DMA experiments are run at very low strains to stay well within a polymer's linear region [27]. For each specimen, an initial temperature of 20°C was established. A thermal program was then followed, heating the materials at 3°C/min, to a final temperature of 100°C. The span length

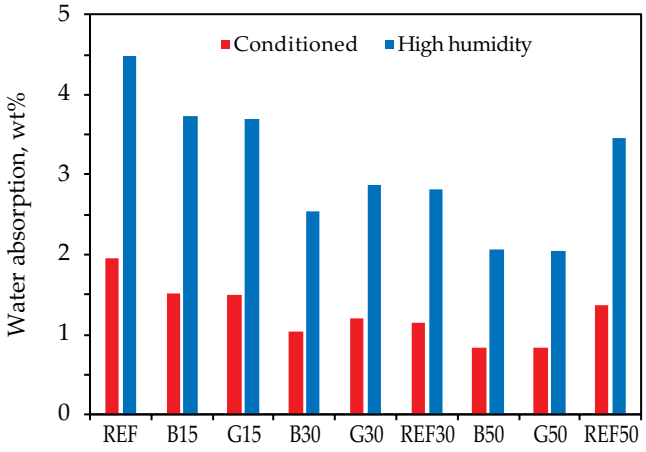

Fig. 1. Moisture absorption of the composites after conditioning

for this test was 40mm. An oscillating frequency sweep of 1 Hz, with an amplitude of 30 μm and 120 μm, for reinforced and unreinforced specimens, respectively, was employed. The glass transition temperature was taken from the maximum of the peak of the tan δ signal.

RESULTS AND DISCUSSION

A comparison of moisture absorption at two predefined conditions for tested composites and reference materials is presented in Fig. 1. Unreinforced PA 4.10 absorbed about 2% of moisture in the conditioned state, while this value more than doubled in the high humidity state, reaching 4.5%. With the increase in fiber content, the moisture absorption decreases as the volume fraction of the polymer decreases. This is independent of the fiber type, which is in line with the research results presented by Romańska *et al*. [20]. PA 4.10 reinforced with 50% glass or basalt fibers has significantly lower moisture content than PA 6.6 with the same fiber content (REF50). The difference is about 40% in both conditioning states. A similar observation was reported by Petersson *et al*. [10] for PA 4.10 reinforced with 30% glass fiber. The moisture absorption was 50% lower compared to fossil-based PA 6 in their study. Lower moisture absorption in the case of

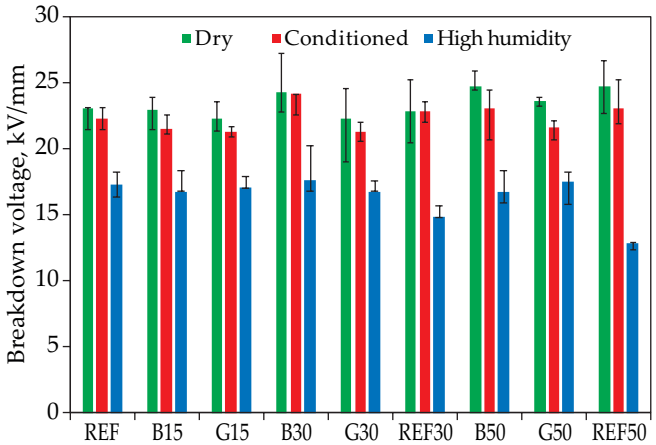

Fig. 2. Breakdown voltage of the composites at room temperature (23°C)

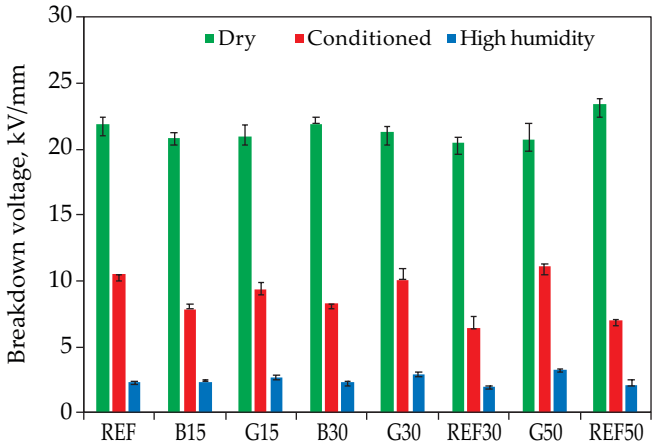

Fig. 3. Breakdown voltage of the composites at elevated temperature (85°C)

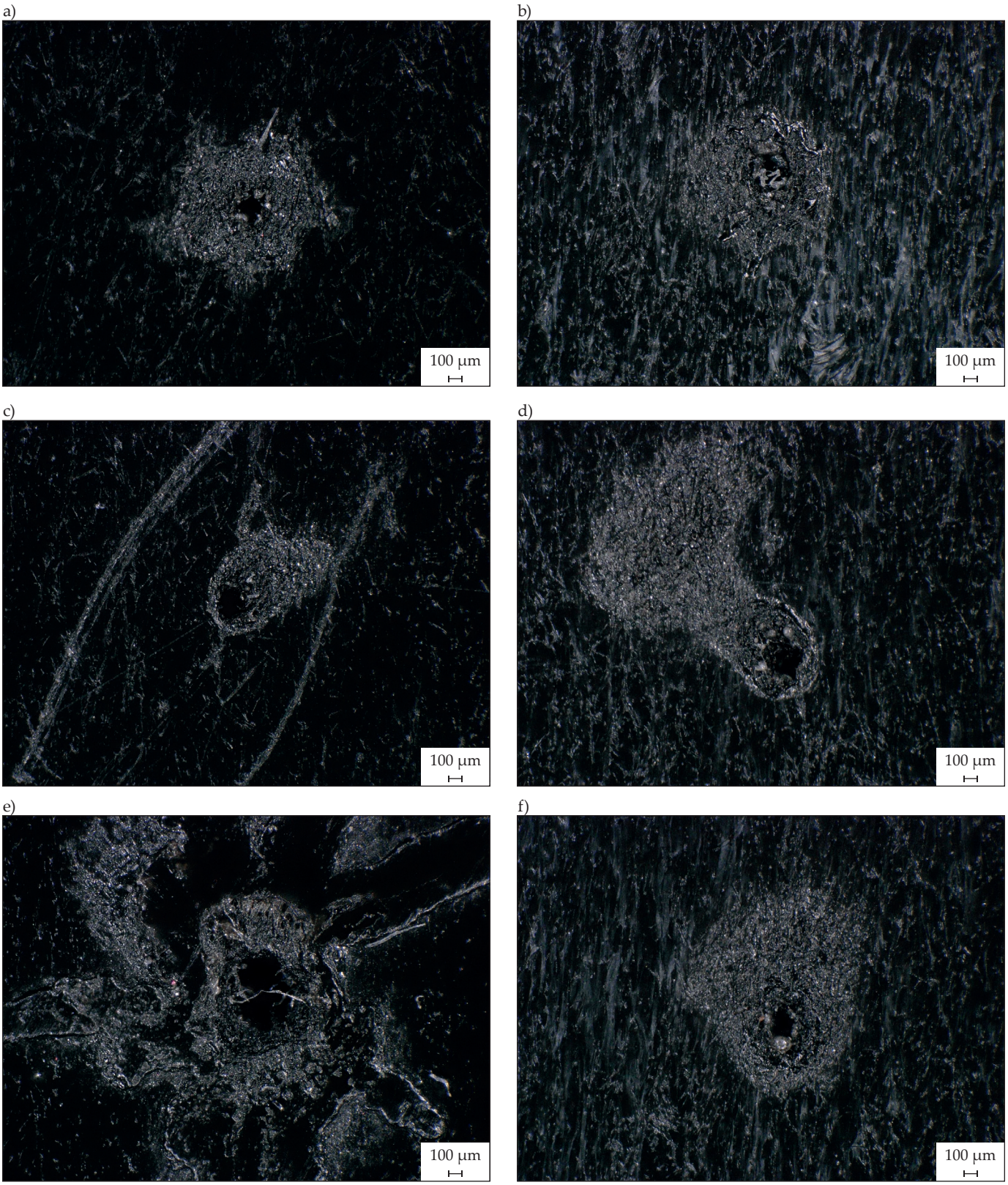

Fig. 4. Optical microscope images of electrical insulation breakdown (23°C): a) dry PA 6.6, b) dry PA 4.10, c) conditioned PA 6.6, d) conditioned PA 4.10, e) PA 6.6 with high humidity, f) PA 4.10 with high humidity; magnification 100×

PA 4.10 yields better dimensional stability in changing environmental conditions.

The median electrical breakdown voltage (BDV) of tested composites and reference materials at room temperature is presented in Fig. 2. The performance of all tested materials in dry and conditioned states at room temperature is similar, with median BDV within the range of 21–25 kV/mm. High moisture content, in the case of high humidity conditioning, noticeably decreases BDV by approximately 5 kV/mm for PA 4.10 and its composites.

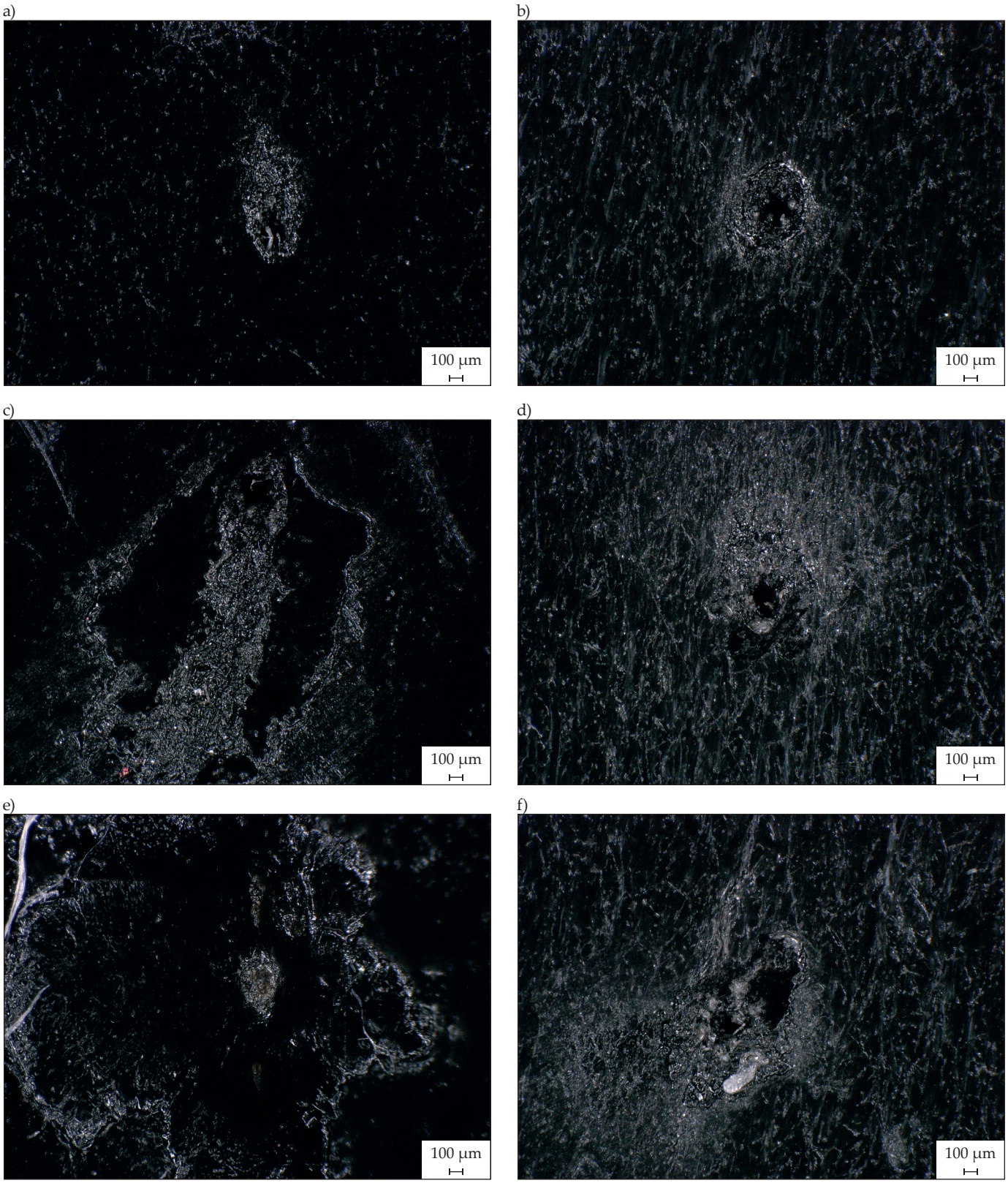

Fig. 5. Optical microscope images of electrical insulation puncture (85°C): a) dry PA 6.6, b) dry PA 4.10, c) conditioned PA 6.6, d) conditioned PA 4.10, e) high humidity PA 6.6, f) high humidity PA 4.10; magnification 100×

PA 6.6 with 50% GF (REF50) is more affected, as its BDV decreased by about 50%, to the lowest recorded value of 12.8 kV/mm.

In Fig. 3 the median electrical breakdown voltage (BDV) of tested composites and reference materials at a temperature of 85°C is presented. BDV values for specimens in the dry state were only slightly decreased compared to their performance at room temperature and remain above 20 kV/mm. The impact of moisture is significant. In the conditioned state, BDV decreased in most cases by

more than 50% for PA 4.10 and its composites. The highest BDV was achieved for PA 4.10 reinforced with 50 wt% of glass fibers, and BDV slightly decreased as the fiber content decreased. The lowest values were measured for reference materials reinforced with glass fibers (REF30 and REF50). In the case of specimens with the highest moisture content, BDV decreased up to 10 times compared to the dry state. The highest BDV was again recorded for PA 4.10 reinforced with 50 wt% of glass fibers, and BDV slightly decreased as the fiber content decreased.

BDV at elevated temperature for moisture-saturated composites reinforced with basalt fiber was lower than for corresponding composites reinforced with glass fiber. This might be caused by different fiber pretreatment and weaker performance of the fiber-matrix interface, but the root cause requires further investigation.

BDV reduction for moisture-saturated polyamides was also confirmed by Douar *et al.* [27]. They evaluated the impact of thermo-hydro aging on BDV of different polymers. In the case of PA 6 and PA 6.6, both reinforced with 50 wt% of glass fibers, the BDV after aging decreased by a factor 10. However, they have attributed this significant decrease to the high degree of hydrolysis, which should not be the case for the results presented in this study. The mechanism of decreased BDV strength for polymers exposed to a humid environment was also discussed by Bottge *et al.* [28]. In their research, the polymer matrix of PA composite showed several microstructural defects: gaps at the interface between glass fibers and the polymer matrix, and voids. These microstructural defects enable the infiltration of PA with moisture and the storage of water clusters. The absorbed water dissolves ionic components of the thermoplastic matrix. Additionally, a portion of the moisture is absorbed by the hydrophilic glass fibers. Both effects increase ion conductivity, leading to dielectric losses that promote thermal-type electric breakdown within thermoplastic materials. In Fig. 4 and Fig. 5, the insulation puncture because of electrical breakdown for PA 6.6 and PA 4.10, both reinforced with 50 wt% of glass fibers, at room temperature and at 85°C, respectively, are presented.

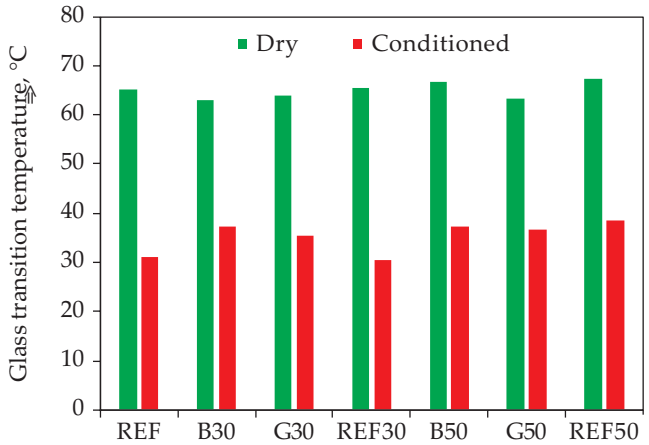

Fig. 6. Glass transition temperature obtained from the tan δ curves from DMA testing for the composites and reference materials in dry and conditioned state

All specimens failed with a described thermal-type electric breakdown. However, in the specimens for which the BDV was above approximately 20 kV/mm (i.e. dry and conditioned samples tested at room temperature, and dry samples tested at elevated temperature), high partial discharges were observed at the edge of the smaller electrode (triple point with the highest electrical field – electrode, thermoplastic composite, and oil) leading to local heating at the specimen surface followed by breakdown, which was localized close to the edge of the smaller electrode. For all the other specimens, insulation puncture was located within the flat area of the smaller electrode, and a thermal-type electric breakdown mechanism was indicated by local material swelling. This is clearly visible in the case of PA 6.6 specimens in Fig. 4e, 5c, and 5e. Material swelling is less pronounced for PA 4.10, and this is also reflected in higher BDV values.

The glass transition temperature of all the composites and reference materials was taken from the maximum of the peak of the tan δ signal and is shown in Fig. 6. The measured value of T_g for dry PA 4.10 is in accordance with existing results [29] and similar values were observed for its composites with glass or basalt fibers, as well as for reference PA 6.6 with 50% GF (REF50). Conditioning of the specimens significantly decreases T_g for all tested materials – it is in the range of 31–38°C. A significant shift of T_g for polyamides was also reported by Guttmann *et al.* [30] and Randhawa e*t al.* [31]. Measurement at high humidity state was not possible as T_g drops below the operating temperature range of 20°C [32, 33].

CONCLUSIONS

The research on the biobased thermoplastic compounds revealed the significant findings regarding the moisture absorption and its impact on the electrical breakdown voltage (BDV), and glass transition temperature. Unreinforced PA 4.10 exhibited the substantial moisture absorption, increasing from approximately 2% in the conditioned state to 4.5% in the high humidity state. However, the increased fiber content in the composites reduced moisture absorption, independent of fiber type, glass, or basalt. PA 4.10 reinforced with 50 wt% of fibers has significantly lower the moisture content than the referenced PA 6.6 with the same fiber content. The difference is about 40% in both conditioning states. At room temperature, the BDV values for all the materials in dry and conditioned states ranged from 21–25 kV/mm, but high moisture content significantly reduced BDV, especially for PA 6.6, which experienced up to a 50% reduction. At 85°C, dry state BDV values slightly decreased but remained above 20 kV/mm, while moisture caused more than a 50% decrease in BDV for the PA 4.10 composites. BDV reduction was more pronounced in the moisture-saturated composites reinforced with basalt fibers compared to glass fibers. This might be caused by the different fiber pretreatment and weaker performance of the fiber-matrix

interface, but the root cause requires further investigation. Moisture saturation decreased the glass transition temperature of all the tested materials. PA 4.10 composites, particularly those with glass fibers, may offer better performance than the referenced PA 6.6 reinforced with 50 wt% of glass fibers in the applications requiring both mechanical stability and electrical insulation.

Authors contribution

D.B. – conceptualization, methodology, validation, investigation, writing-original draft, writing-review and editing, visualization; A.K. – methodology, validation, writing-review and editing; S.K. – conceptualization, supervision.

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Conflict of interest

The authors declare no conflict of interest.

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