



Hybrid Epoxy Composites Reinforced with Flax Fiber and Basalt Fiber

Danuta Matykiewicz^(✉) and Maciej Bogusławski

Polymer Processing Division, Faculty of Mechanical Engineering and Management, Institute of Materials Technology, Poznan University of Technology, Piotrowo 3, 61-138 Poznań, Poland
danuta.matykiewicz@put.poznan.pl

Abstract. The aim of this work was to evaluate the influence of basalt and flax fiber content on the thermo-mechanical and mechanical properties of epoxy composites. The mechanical properties were assessed by means of static tensile test and Charpy impact strength method. The thermo-mechanical properties of the composites were determined through dynamic mechanical thermal analysis (DMTA). Moreover, hardness was examined with Shore D durometer. The thermal stability was determined by thermogravimetric analyses (TGA) in inert atmospheres. The increase flax fiber content led to decrease the tensile strength value, elasticity modulus and storage modulus value of the epoxy composites. The most advantageous mechanical and thermomechanical properties were obtained for the composites containing six layer of the basalt fiber and then hybrid composites containing four layer basalt fiber and two layer flax fiber.

Keywords: Composites · Basalt fiber · Flax fiber · Thermomechanical properties

1 Introduction

Fiber reinforced composites have become very attractive engineering structures in recent years and have extensively replaced popular metallic and polymeric materials in many industrial application. The load transfer between fibers and polymer depend on chemical bonds, secondary interaction forces and mechanical interlocking [1]. The chemical structures of the fibers and matrix are different and many research activities are focus on improve the interfacial bonding properties of the constituents in the composites [2, 3]. Generally, fiber reinforced composites have specific strength and stiffness and improved thermomechanical properties in comparison to the unmodified polymer materials.

Epoxy resins, due to their good compatibility with different types of fibrous reinforcement, can be successfully used for production composite materials [4–6]. Popular fibers which are used as a reinforcement are glass, carbon, silicon, boron and synthetic fibers also different natural fibers [7]. Reinforced materials have a higher resistance to crack initiated failure than the neat resin, the crack being intercepted by the fiber reinforcement [8]. Therefore, the application of natural fibers and polymer materials in the automotive and building industry may yield economic, environmental and social

benefits [9, 10]. Furthermore, in order to expand the application of the thermoplastics and thermosets polymer different inorganic fillers such as: micro-/nano-SiO₂, carbon black, glass, Al₂O₃, Mg(OH)₂, CaCO₃, boron nitride and aluminum nitride are often introduced to the polymer composites [11–13]. Moreover, low-cost natural fillers extracted from stems, leaves, fruits, as well as parts of plants such as seeds, husks, hulls are used as a reinforcement of epoxy composites and bio based polymers [14–16].

Natural fiber reinforced polymer composites may be an alternative to composites based on petroleum-based materials and synthetic fibers [17]. Natural fibers can be divided into vegetable fibers, animal fibers, and mineral fibers. Flax, hemp, jute and sisal have been often used for engineering fiber composites. Flax fiber with specific mechanical properties and good vibration and sound absorbing properties is the one most often used lignocellulosic fibers [18, 19]. Moreover, the flax fibers are less toxic and cheaper than glass fibers as well as have high strength to weight ratio. Basalt fiber is manufactured during the melting process of the volcanic rocks without any additives and have better mechanical strength, thermal stability and chemical resistance than glass fiber [20]. Hybrid composites are manufactured from two or more different components for example fiber reinforcements with unique properties and origin [21, 22]. In literature is reported that the modified polymer materials and epoxy hybrid composites with glass, flax and basalt fibers and hybrid flax/basalt fiber reinforced green composites indicated good mechanical properties [21, 23–25].

Therefore, the aim of this work was to verify the influence of type of fibrous reinforcement such as flax and basalt fabrics on the epoxy composites thermomechanical properties. Mechanical and thermomechanical properties were evaluated by means of static tensile test, Charpy impact strength method, dynamic mechanical thermal analysis and thermogravimetric analysis. Moreover, hardness was assessed with Shore D durometer.

2 Experimental

2.1 Materials

The following components were used in this investigation: epoxy resin Epodur CHS 574-0492, curing agent CHSE 574-0492 produced by SPOLCHEMIE, Czech Republic. Epoxy resin was mixed with curing agent at ratio 28 parts curing agent per 100 parts resin by weight. Basalt fiber woven fabric (B) type BAS 220.1270.P with weight of 220 g/m² and plain weave type (BASALTEX) and flax fiber woven fabric (F), with weight of 250 g/m² (Polski Len) were used as reinforcement.

2.2 Sample Preparation

The hybrid epoxy composites were produced in a mold using hand lay-up and then were pressed by vacuum bagging method, which ensures to obtain good quality composites by removing excess resin and air bubbles. Composites were cured at ambient temperature (20 °C) for 24 h and post-cured at 80 °C for 3 h. In all the cases 6 layers of fibers were used as reinforcement. First, the dry fabrics were impregnated by

resin using brush and roller in hand lay-up method. Then the prepared stack of wet fibers were placed in the mold and covered by vacuum bagging film. Next the vacuum contactor was mounted to the outer part of the vacuum foil and the whole system was connected to vacuum pump. The vacuum bag method was carried out with the constant pressure 0,8 bar during 1 h. The composites contained layers from basalt fibers, flax fibers and both flax and basalt fibers in different configuration. The samples were described as BBB; FFF; BFB; BF; FBF adequately to the type of the incorporated fibrous reinforcement. The arrangement of fibers in the composites is shown in Fig. 1. First type of sample described as BBB contain only basalt fiber fabric, while FFF sample contain only flax fiber fabric. Hybrid epoxy composites reinforced with both flax fiber and basalt fiber were de-signed as BFB and FBF were external laminate layers were made of 2 layers of basalt fabrics and 2 layers of flax fabrics, respectively. Sample BF contain equal amount of layers from flax and basalt.

3 Methods

3.1 Dynamic Mechanical Thermal Analysis (DMTA)

The dynamic-mechanical properties of the composites, were evaluated using DMTA methods (Anton Paar MCR 301, Austria) in a torsion mode, operating at frequency 1 Hz in the temperature range between 25 °C and 180 °C, and at the heating rate 2 °C/ min. The position of $\tan \delta$ at its maximum was taken as the glass transition temperature (T_g).

3.2 Tensile Mechanical Properties

Tensile mechanical properties were measured with INSTRON4481 universal testing machine in accordance with ISO 527-4. All tests were performed at ambient temperature (23 °C) with testing speed 1 mm/min and with a load cell of 50 kN.

3.3 Charpy Impact Strength

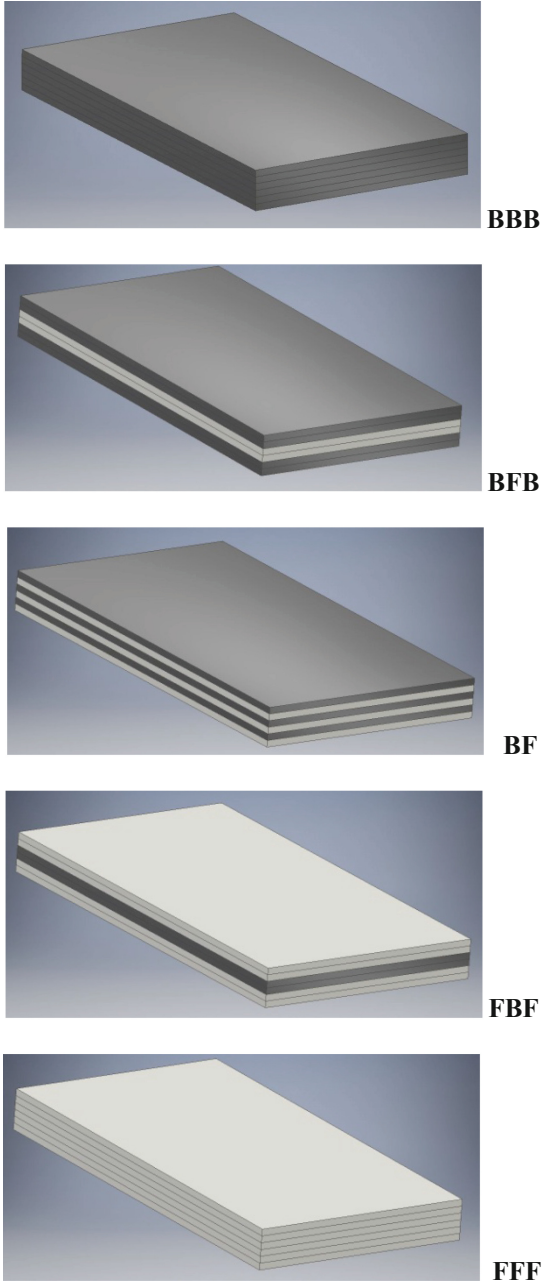
The impact strength of the unnotched samples was measured using the Charpy method (ISO 179) at room temperature. The INSTRON Woolpert PW9 impact tester with 25 J hammer was used.

3.4 Hardness

Shore D hardness was measured using a Sauter HBD 100-0 (Germany).

3.5 Thermogravimetry (TGA)

The thermal stability of composites was analyzed by thermogravimetric method (TGA) with temperatures ranging from 30 to 900 °C at the heating rate of 10 °C/min under nitrogen atmospheres using a TG 209 F1 Netzsch apparatus. Approximately 10 mg samples were placed in ceramic pans. The initial decomposition temperature



■ - layer of basalt fiber woven fabric; □ - layer of flax fiber woven fabric

Fig. 1. Schematic representation of the composites cross-sections.

T10% was determined as a 10% weight loss temperature. The residual mass ($\Delta W\%$) was defined at about 900° . Additionally, maximum intensity of thermal degradation temperatures (DTG) were evaluated.

4 Results and Discussion

4.1 Dynamic Mechanical Thermal Properties

DMTA method allows to assess the materials thermomechanical properties and determine parameters such as: storage modulus G' , loss modulus G'' and mechanical loss factor ($\tan \delta$) versus temperature T and glass transition temperature T_g . The plots of G' and $\tan \delta$ versus temperature for investigated composites are presented in Figs. 2 and 3. The values of composite storage modulus and glass transitions are collected in Table 1. In all cases the basalt fiber addition improved the value of G' in the range of temperature $25\text{--}70^\circ\text{C}$. The highest values of G' (6000 MPa) were noted for the sample containing only basalt fibers BBB. While, for the samples with the basalt fiber and flax fiber the placing of specific layers had a significant influence on their thermomechanical properties. For the hybrid epoxy composites the highest values of G' were indicated for sample BFB (4000 MPa). The sample containing only flax fibers FFF had the lowest G' values (1900 MPa). The glass transition temperature value for all the investigated composites was similar, at approx. 105°C . The $\tan \delta$ peak value presented in Fig. 3 is related to the damping properties of the materials and increases together with the increase of flax fiber content in the composites. This proves that the composites with flax fiber will be better suppress the vibrations than composites with basalt fiber.

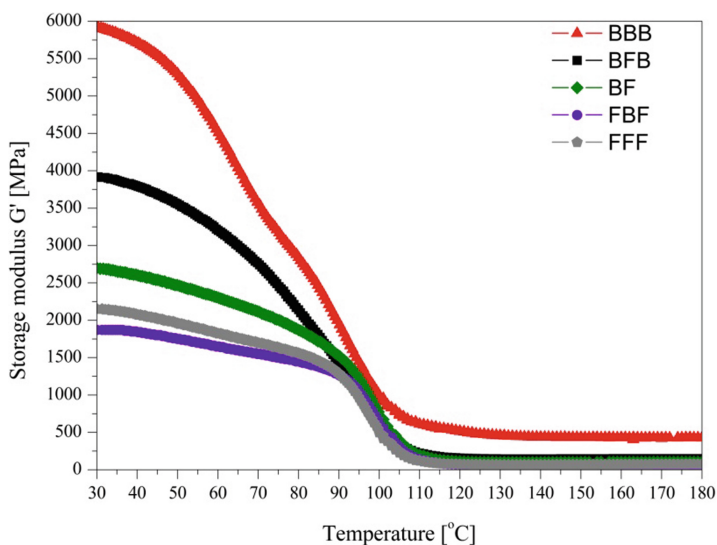


Fig. 2. The DMTA curves of the composites of storage modulus G' vs. temperature.

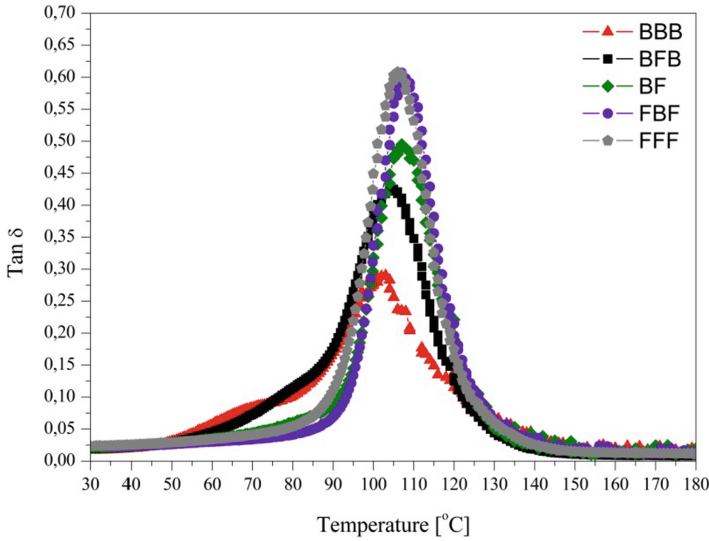


Fig. 3. The DMTA curves of the composites of $Tan \delta$ vs. temperature.

Table 1. The values of composite storage modulus and glass transitions.

Name	G' [MPa]at 30 °C	G' [MPa]at 115 °C	T_g [°C]	$Tan \delta$
BBB	5950	548	102	0,31
BFB	3920	177	105	0,42
BF	2700	132	108	0,50
FBF	1870	86	107	0,61
FFF	2150	84	106	0,61

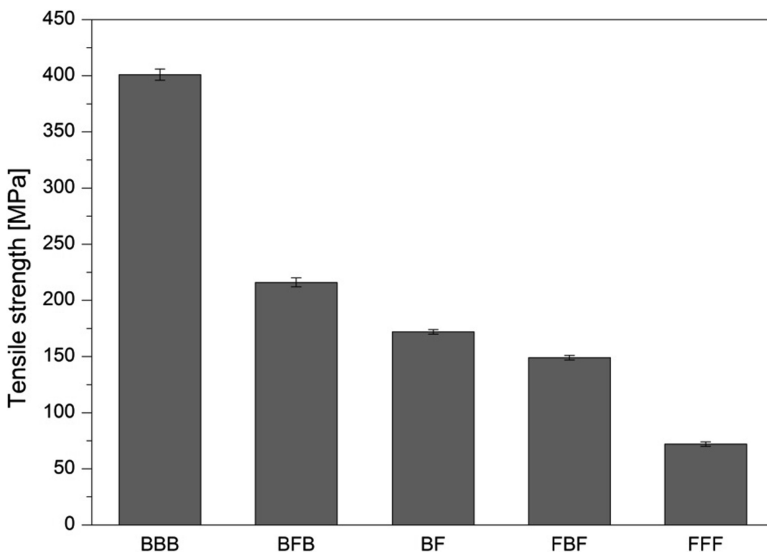
4.2 Mechanical Properties

The mechanical properties of the fiber reinforced composites may be result of the strength of fibers, as well as their interfacial bonding properties [26]. The comparison of the values of tensile strength, elasticity modulus, elongation at break and impact strength of the investigated materials are collected in Table 2. Additionally, the values of tensile strength and impact strength of the composites are shown in Figs. 4 and 5.

The value of tensile strength of the BBB sample was approx. 400 MPa, while for the FFF sample it reached 72 MPa. Moreover, based on our previous work [27] it may be stated that epoxy/basalt composites produced in the hand lay-up method have inferior the strength properties than epoxy/basalt composites produced by the vacuum bag method. This effects is due to the removal of excess air and resin in the manufacturing process. For the hybrid composites with both basalt fiber and flax fiber the tensile strength value increases proportionally with the increasing the number of basalt

Table 2. Mechanical properties of the composites.

Name	Tensile strength [MPa]	Elasticity modulus [MPa]	Elongation at break [%]	Impact strength [kJ/m ²]
BBB	401 ± 5	8531 ± 60	5,1 ± 0,07	125 ± 8
BFB	216 ± 4	5310 ± 250	4,4 ± 0,2	105 ± 6
BF	172 ± 2	4120 ± 100	4,8 ± 0,06	110 ± 5
FBF	149 ± 2	3643 ± 72	5,2 ± 0,04	60 ± 4
FFF	72 ± 2	3050 ± 290	5,5 ± 0,2	35 ± 5

**Fig. 4.** Tensile strength of the composites.

layers. It should be emphasized, that the addition of 2 layers of rigid basalt fibers to the flax fibre reinforced composite will increase its strength by about 50% (from 72 MPa for FFF to 149 MPa for FBF) [28]. Likewise, in all cases basalt fiber introduction led to an increase of the elasticity modulus of the composites. The highest values of the elasticity modulus were noted for the BBB sample (8531 MPa) and then for BFB sample (5310 MPa). The elongation at break values for all investigated samples were comparable (approx. 5%).

The flax fiber presence led to a decrease in the impact strength values of the hybrid epoxy composites. This is due to the basalt fiber being more rigid than flax fiber, as well as have better crack resistance. The Shore D hardness values were dependent on the structure of the composites. Placed of stiff basalt fabrics in the outer layers slightly improved the hybrid composites hardness. The relationship between the Shore D hardness and the basalt fiber content in epoxy materials is shown in Fig. 6.

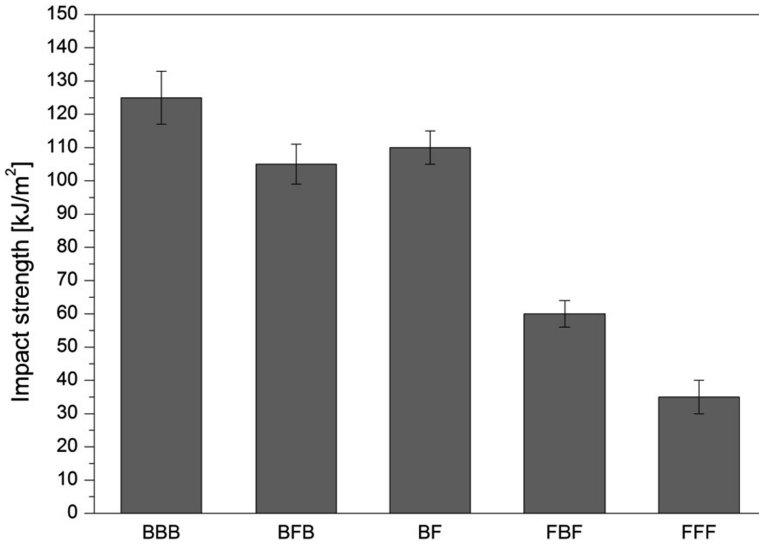


Fig. 5. Impact strength of the composites.

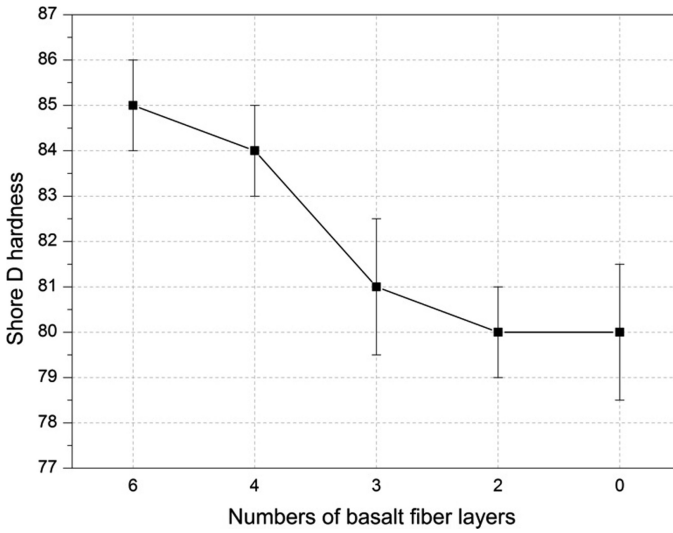


Fig. 6. Plot of the hardness value versus the number of layers the basalt fiber sheets.

Results of mechanical analysis revealed that the hybrid epoxy composites containing basalt fiber reinforcement showed higher strength and elastic modulus than composites reinforced just with flax fiber. Therefore, it should be underlined, that the preparation of the hybrid composites containing both flax and basalt fiber is a favorable method leading to improving the mechanical properties of flax composites.

4.3 Thermal Stability of the Composites

Thermogravimetric analysis (TGA) is an analytical method applied to evaluate the thermal stability and the thermal performance of the inorganic and organic materials. Specific data on the composites thermal properties, such as: (T10%) and (T50%), maximum intensity of thermal degradation temperatures (DTG), and residual mass, are collected in Table 3. Plots of weight loss vs. temperature (TG), together with their derivatives (DTG) recorded in nitrogen atmospheres, are presented in Figs. 7 and 8. A one-step degradation, which may be assigned to the pyrolysis process, for all investigated materials was observed [29]. The highest T10% value was noted for BBB sample. while the lowest T10% value was recorded for FFF sample. The DTG peak values average at 367 °C could result from the resin decomposition process as well as flax fibers. It was noted that flax fiber presence led to a increase in rate of degradation values of the hybrid epoxy composites. On the other hand, introduction basalt fiber into the flax/epoxy composites improves their thermal properties. This effect results from the much higher thermal stability of basalt fibers compared to flax fibers. Additionally, high amount of the residual mass for composites with high amount of the basalt fiber was observed.

Table 3. TGA data of the composites investigated under nitrogen Atmospheres.

Name	T10% [°C]	Residual mass [%]	DTG
BBB	357,6	64,56	367.8 °C; -4.98%/min
BFB	336,8	37,92	367.4 °C; -7.64%/min
BF	331,8	31,16	360.9 °C; -8.13%/min
FBF	331,0	20,70	369.6 °C; -10.38%/min
FFF	324,9	10,70	367.6 °C; -11.61%/min

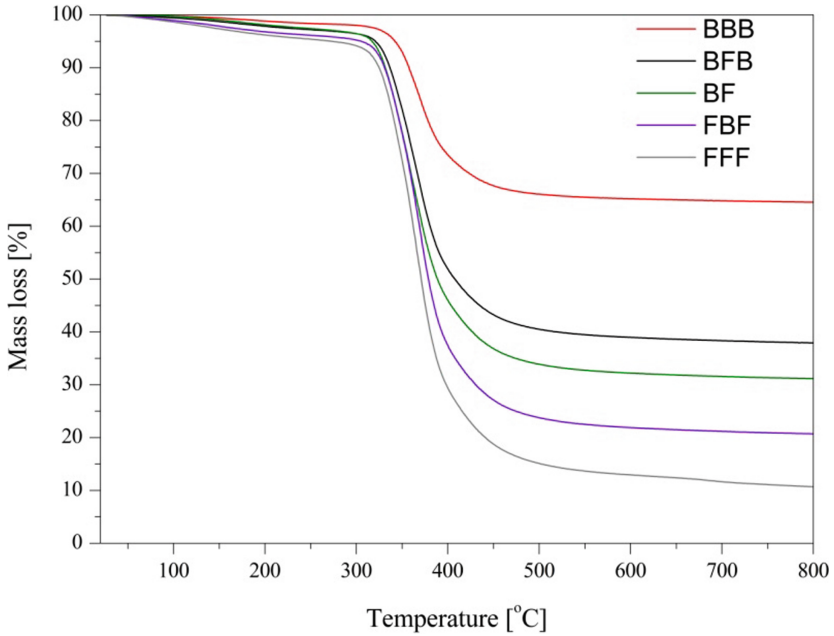


Fig. 7. TG curves of investigated composites under nitrogen atmosphere.

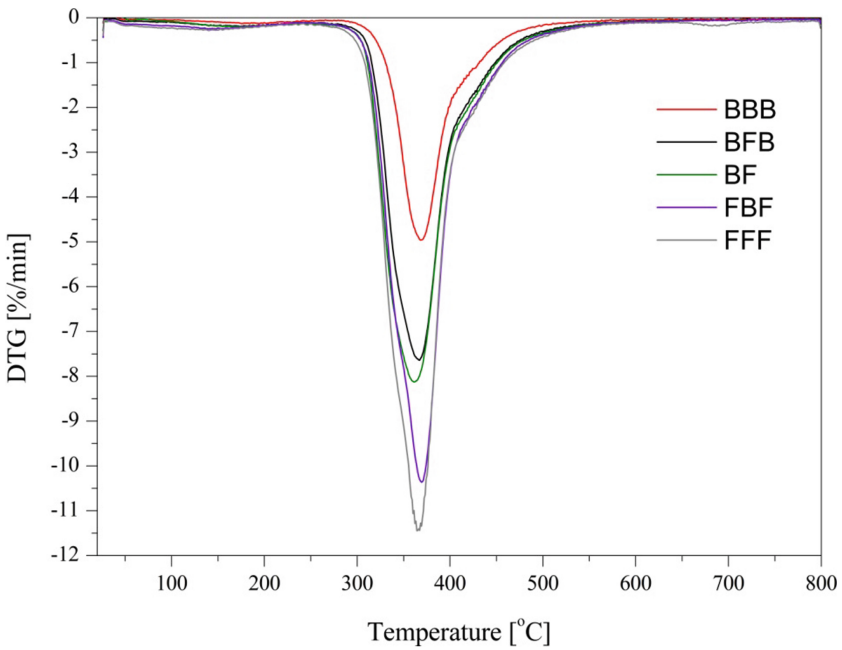


Fig. 8. DTG curves of investigated composites under nitrogen atmosphere.

5 Conclusions

Vacuum bagging method was used to production hybrid epoxy composites reinforced with flax fiber and basalt fiber. The best mechanical and thermomechanical properties indicated composites reinforced with only one type of fiber in the form of basalt fibers. For the hybrid composites with both basalt fiber and flax fiber the mechanical properties such as tensile strength, elasticity modulus and impact strength increases proportionally with the increasing the number of basalt layers. It should be stressed that the presence of basalt fiber into the epoxy composites with flax fiber significantly improve their thermomechanical properties.

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