Cryogenically treated goat and human hair reinforced hybrid polymer composites

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Abstract: Composites of epoxy resin reinforced with goat and human hair were obtained and subjected to cryogenic processing for 24 hours at temperature 77 K. Morphological (SEM), mechanical and thermogravimetric analyses (TGA) were performed. The best properties were obtained by using a reinforcement consisting of 60 wt% human hair and 40 wt% goat hair. The SEM confirmed good interactions at the interface for this composite.

Keywords: goat hair, human hair, epoxy resin, composites, cryogenic treatment.

Hybrydowe kompozyty polimerowe wzmocnione włosem kozim i ludzkim poddane obróbce kriogenicznej

Streszczenie: Otrzymano kompozyty żywicy epoksydowej wzmocnionej kozim i ludzkim włosem, które poddano obróbce kriogenicznej przez 24 godziny w temperaturze 77 K. Zbadano strukturę (SEM), właściwości mechaniczne i termiczne (TGA). Najlepsze właściwości uzyskano stosując wzmocnienie składające się z 60% mas. włosa ludzkiego i 40% mas. włosa koziego. Metodą SEM potwierdzono dla tego kompozytu dobre oddziaływania na granicy faz.

Słowa kluczowe: futro kozie, włos ludzki, żywica epoksydowa, kompozyty, obróbka kriogeniczna.

Researchers and engineers who works in the field of composites employ natural fibers as a reinforcing material due to their extraordinary characteristics like economic viability, good weight-to-strength ratio, eco-friendly nature, and flexible fabrication methods. Composite materials with natural fibers have material properties that are suitable for various applications [1]. Due to the increasing demand for composite materials, the research community is searching for new fiber that is available easily and in abundance [2]. Animal fibers are studied, because they are environmentally friendly and have good properties due to their flexibility and stiffness. Also, they have excellent insulation properties. Animal fur, wool and silk are the major animal fibers that are commonly used for reinforcement in composites [3, 4].

Goat hair is one of the animal fibers that are available naturally in abundance as a waste by-product from the meat industry. The disposal and recycling of goat hair is considered a major problem. By utilizing goat hair as fiber for reinforcement, the recycling of this waste could be done successfully with minimal impact on the environment [5]. Human hair is a renewable resource, and it is a sustainable choice compared to other synthetic fibers. By using human hair as reinforcement, the environmental concern regarding the dumping of human hair as waste is reduced [6].

The composite materials utilized for applications in automobile and aerospace industries should have good mechanical properties. Enhancing the mechanical properties of fiber-reinforced composites could be achieved by cryogenic treatment. Cryogenically treated polymeric materials result in notable improvements in toughness, rigidity, and fracture toughness [7].

Himanshu Kumar Sinha *et al*. [8] fabricated hybrid composites by combining goat hair and coir and using epoxy as a matrix. Two sets of composites were fabricated, one only with hair fiber and another using coir along with goat hair fiber for reinforcement. The results showed that composites containing only goat hair had the best mechanical strength. In another work, Ankush Gupta [9] examined the mechanical properties of composites containing goat fur and banana fiber and found that the properties of the hybrid polymer improved with the increase in the fillers content. Everywhere on

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the globe, human hair is viewed as a waste product that slowly decomposes. The term "waste material" is now recognized as a viable material resource for a variety of uses. Including composites for superconducting systems, reinforced construction materials, and molded furniture and artefacts. Jayachandran *et al.* [10] studied the polymer composites reinforced with hair and coir. The results shows that more percentage of either hair or coir with epoxy resin improved impact and elastic characteristics but reduced point load bearing capability. When compared to coir reinforcement, the mechanical qualities of hair reinforcement are often better. Thomas *et al.* [11] analyzed the mechanical behavior of polymers reinforced by banana fiber-hair reinforcement. As a result, it was determined that the hybrid composite reinforced with banana and hair has more tensile strength than the composite reinforced only with banana fiber.

Velmurugan *et al.* [12] used bamboo as fiber and olive tree leaf powder as additive for epoxy to examine influence of cryogenic treatment. The findings of this study indicate that 30 min. treatments produce the best results. It has an impact strength that is 26.5% higher, a flexural strength that is 21.8% higher, and a tensile strength that is 11.3% higher, compared to pure epoxy. The tensile and flexural modulus had values that are 27.1% and 48.6% greater, respectively, than the values for the treated composites. Shindo *et al.* [13] studied nonwoven polyester/epoxy composites mechanical response at cryogenic temperatures. Composite cylindrical components were evaluated for mechanical capabilities at ambient temperature and in the atmosphere with liquid nitrogen at 77 K using tension, compression, and flexure tests. The results show, that at 77K stiffness and strength increase, while exhibiting stiffness anisotropy. Yuanchen Li *et al.* [14] used cryogenic environment to study the nature of damage mechanism of e-glass epoxy composites in such environments. The obtained results revealed that the damage progression occurs at 293 K, 173 K, and 93 K.

Numerous examinations were done in the field of composites made of fly ash, plant and animal-based fibers, synthetic fillers, and many other materials that were used in variety of industries. The composite material incorporating human hair and goat hair has not yet been found in the literature. By creating a fiber-hybrid polymer composite reinforced with goat fur and human hair and obtaining improved mechanical properties from it, this research makes an effective effort to make use of goat hair and human hair waste. Also, keratin-based fibers possess good flexibility and insulation properties. The in-build properties of human and goat hair could be further improved by cryogenic treatment.

The aim of the work was to obtain composite materials from goat and human hair using epoxy resin as a matrix. The composites were cryogenically treated to improve mechanical properties. The basic justification for choosing this epoxy resin reinforcement was the more efficient use of waste as a potential resource. Moreover, due to

the excellent insulating properties of hair, the obtained composites can be used for applications in a cryogenic environment.

EXPERIMENTAL PART

Materials

The epoxy resin used was LY556 (bisphenol-A), and the hardener was HY951 (tri-ethylene-tetramine). Wax was used as a releasing agent for easy removal of composites after fabrication. Acetylene solution was used for cleaning all the utensils involved in fabrication. LY556 and HY951 used were from huntsman brand. All the chemicals and reagents were purchased from Pon Pure Chemicals, Perundurai, Erode, Tamil Nadu, India.

{P2} Fiber preparation

Goat fur was hand-picked from a goatskin obtained from a local meat shop, while human hair was collected from nearby barbershop. Both hairs were properly soaked and rinsed with warm water, then dried to remove the moisture. Before being utilized as reinforcement fibers in composite laminates, dust hairs and unknown particles were removed (Fig. 1).

The physical properties play a major role in contributing to the properties of reinforced composites. The physical properties of reinforcements are presented in Table 1.

T a b l e 1. Physical properties of human and goat hair [11]

Property	Human hair	Goat hair
Diameter, μm	100–120	80–110
Tensile strength (5 pieces together as a bunch), N	68	4–5
Density, g/cm³	1.4–1.6	1.0–1.2
Moisture absorption, %	9.8	7.8
Elongation at break, %	12.5–35.6	9.56–30.5

Composites preparation

Composites were obtained by the hand layup method. A mold with dimensions of 290 × 290 × 5 mm was used. Initially, wax was applied to the mold for easy removal of the composite. The epoxy resin and hardener were then mixed in a 10:1 (m/m) ratio and poured into the mold, leav-
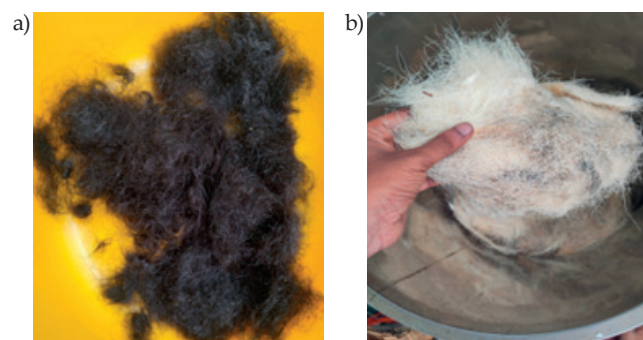
Fig. 1. Extracted human (a) and goat (b) hair

T a b l e 2. Designation of composites and reinforcement composition

Composite	Goat fur, wt%	Human hair, wt%
C0	0	0
C1	0	100
C2	80	20
C3	40	60
C4	50	50
C5	75	25
C6	60	40
C7	100	0

ing enough space for the hair and the rest of the epoxy resin. In the next stage, goat fur and human hair were evenly distributed in the mixture of resin and hardener in the required proportion (by weight). Then, resin and hardener were poured onto the fiber layer to appropriately strengthen the fibers in the matrix. Excess resin from the top of the mold was removed with a roller. Finally, the mold was closed and pressed using a hydraulic press. Then the sample was cured for 24 hours. Seven sets of specimens of different proportions of goat and human hair were obtained, as presented in Table 2 with their corresponding designations.

Cryogenic treatments

A chamber filled with nitrogen liquid at 77 K is used for cryogenic treatment of fiber reinforced polymer composites. The specimens were dipped in the liquid nitrogen and kept for 24 hours. This treatment is known as deep cryogenic treatment.

Methods

Mechanical properties

Tensile, flexural, and compressive properties were examined according to the ASTM 3039, ASTM D790 and ASTM D 695-02 standard, respectively. The crosshead speed was 2 mm/min. All tests were performed using universal testing machine (Daksh UTE 40, Indore, India). Izod impact strength was determined according to the ASTM D256-05 standard. The hardness was evaluated according to ASTM D2240 standard.

Thermal analysis

Thermal stability was determined by thermogravimetric analysis (TGA) using a TG/DTA-EXSTAR/6300 thermogravimetric analyzer (Seiko Instrument Inc., Chiba, Japan) in nitrogen atmosphere at heating rate of 10°C/min, from 30 to 700°C. The temperature of the beginning of thermal decomposition (T_{onset}) and the temperature of the maximum decomposition rate (T_{max}) were determined. The minimum visible on DTG curve correspond to T_{max}.

Morphological analysis

The microstructure of cryogenic treated composites was studied using a Zeiss Sigma 300-VP scanning electron microscope (Germany) operating at 5 kV. The impact fractured surface of the samples was coated with a thin gold layer to avoid charging and increase image contrast.

RESULTS AND DISCUSSIONS

Mechanical properties

Mechanical properties of the composites are presented in Table 3. The composites have much better mechanical properties than epoxy resin, as evidenced by greater flexural, tensile, and compressive strength, as well as greater impact strength and hardness.

The flexural strength of composites reinforced with goat hair or human hair is based on several parameters, including the type and length of the hair, the matrix used, the fiber content, and the fabrication process. It could be noted that there is an increase in several properties of the manufactured composites when human hair is introduced. Like the increase in tensile strength, there is an upward trend in the flexural characteristics when the human hair fiber content is higher than that of goat fiber. The highest flexural strength was recorded when the human hair fiber content was 60 wt%, and the goat fiber content was 40 wt%, and the lowest without human hair fiber. Flexural strength decreased due to the increase in goat hair content. When the goat fiber was held at a higher concentration, stress transfer was reduced, leading to a loss of flexural strength. The lack of sufficient bonding between the epoxy and hair led to a reduction in flexural strength. Cryogenic treatment effectively glues the polymer matrix and fibers together. An effective connection ensures effective stress distribution between the phases. In this way, the stress concentration is reduced, and the flexural strength is increased [16].

The tensile strength of human hair was found to be efficient. Tensile strength increases when there is more human hair in the composite but decreases when there is more goat hair. The hairs have properties that widen the stress distribution at the interfaces, facilitate the good linkage and bonding of the fibers with the epoxy resin [15]. The reduction in tensile strength is due to the fragile character of the goat hair, in turn human hair is more rigid and stiff. The C3 composite, with 60 wt% human hair and 40 wt% goat hair, is characterized by the highest tensile strength. Cryogenic treatment increases the tensile strength of the material. This is since there will be improvements in crystal structure and the defects at the micro level will be rectified by cryogenic treatment. The internal stresses are also relieved, which in turn enhances the tensile load-bearing capability of the fabricated hybrid-reinforced polymer composite.

T a b l e 3. Mechanical and thermal properties of the composites

Sample	Flexural strength MPa	Tensile strength MPa	Compressive strength MPa	Izod impact strength J	Shore D hardness ShD	T_{onset} °C	T_{max} °C
C0	7	34	32	2.2	47	140	164
C1	42	15	41	5	62	236	420
C2	45	17	44	7	64	232	410
C3	59	29	58	9	78	286	457
C4	53	21	52	6	72	253	433
C5	48	16	45	5	66	241	420
C6	51	19	51	7	73	254	435
C7	39	12	38	4.5	61	216	380

The proportion of human hair in appropriate amount improved the compressive properties [17]. The highest compressive strength is 58 MPa (C3), and the lowest is 38 MPa (C7). There was an improvement in the fiber properties because the cryogenic treatment made the fiber stronger, which allow for better alignment of the fiber with epoxy during compressive stress. Adhesion between the matrix and the fibers also improved due to the cryogenic treatment. Cryogenic treatment reduced the formation of microcracks during the application of the load. It also reduced the formation of microcracks by healing the existing microcracks.

The cryogenic treatment plays a major role in enhancing the impact strength. Under impact loading, microcracks tend to expand. Cryogenic treatment reduces the number of microcracks, which makes the material stronger under impact loading. It can also make it easier for the fibers and material of the composite to stick together. This helps keep the fibers from coming apart from the matrix when they are subjected to impact load, this improves the impact strength of the fiber reinforced polymer composite [18]. The human-goat hair ratio also has a significant effect on the impact strength of the composites (Tab. 3). The highest impact strength was obtained for the C3 (9 J) and the lowest for the C7 (4.5 J) composite. It can be concluded that the cryogenic treatment is beneficial for improving the impact strength by improving the adhesive characteristics of the epoxy and fiber in the composite material.

The Shore hardness of the composites is listed in Table 3. The composites are characterized by 13–30 ShD higher hardness compared to epoxy resin. Like other mechanical properties, the C3 composite had the highest hardness and the C7 composite had the lowest hardness.

Thermogravimetric analysis

Thermogravimetric analysis was used to explain how the hair fibers interact with each other and the matrix under the influence of temperature changes. The thermal decomposition of epoxy resin and the composites is summarized in Table 3 and shown in Figure 2. The addition of goat and human hair significantly improved thermal stability of epoxy resin, due to 76–146°C higher T_{onset} and
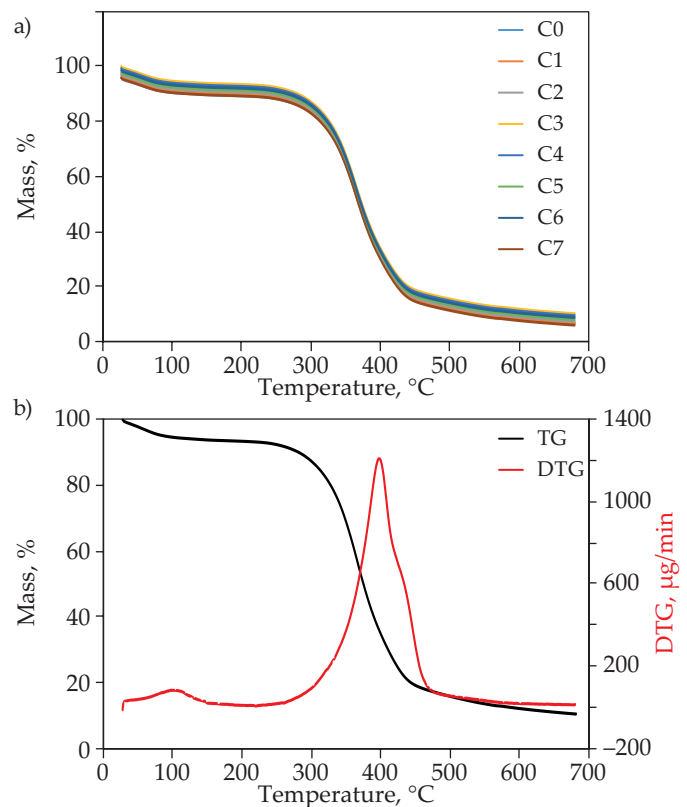
Fig. 2. TGA curves: a) C0–C7 composites, b) C3 composite

216–293°C T_{max}. The mass loss up to about 286°C is attributed to the removal of the solvent. Final degradation occurs at 380–457°C, which is due to the degradation of the epoxy resin and fibers. The heat resistance of the C3 composite is much higher than the others, which can be explained by the higher fiber degradation temperature due to the breaking of C-O bonds and other types of hydrogen bonds that occur in human hair and resin. The increase in thermal resistance was also influenced by cryogenic treatment of fiber-reinforced polymers [20]. Depolymerization and degradation have been reduced by cryogenic treatment.

SEM analysis

Scanning electron microscopy (SEM) was used to assess the microstructure of the obtained composites.
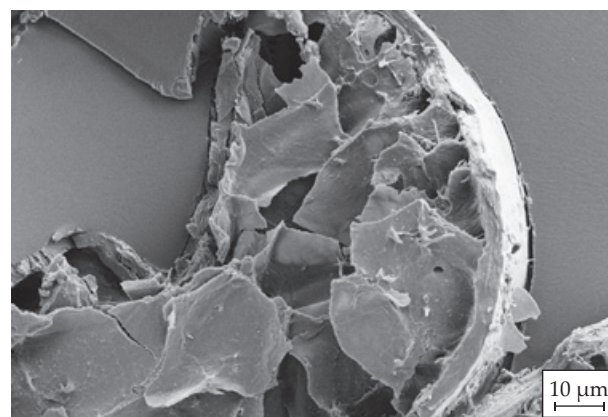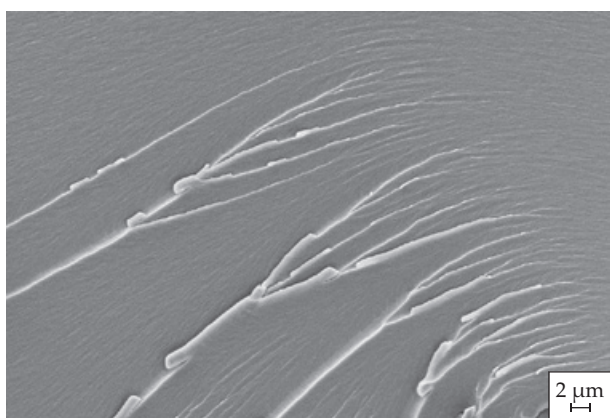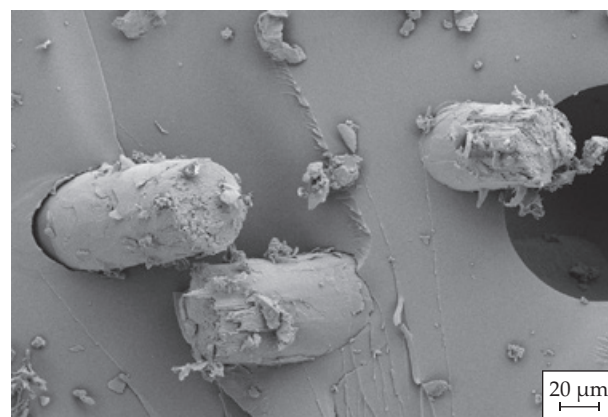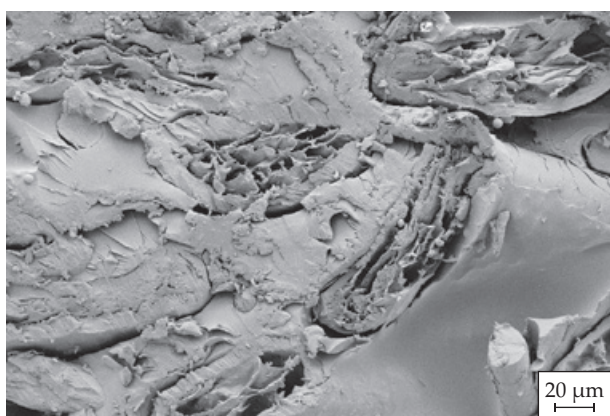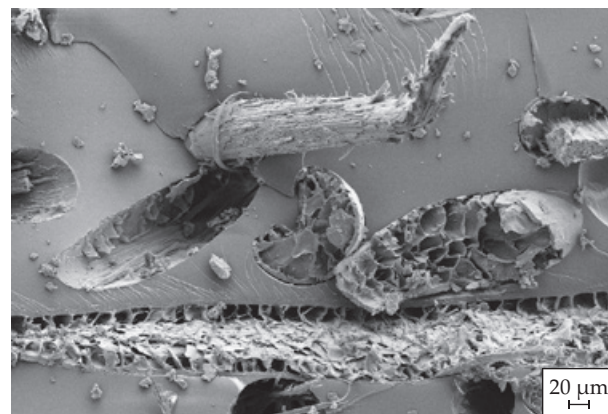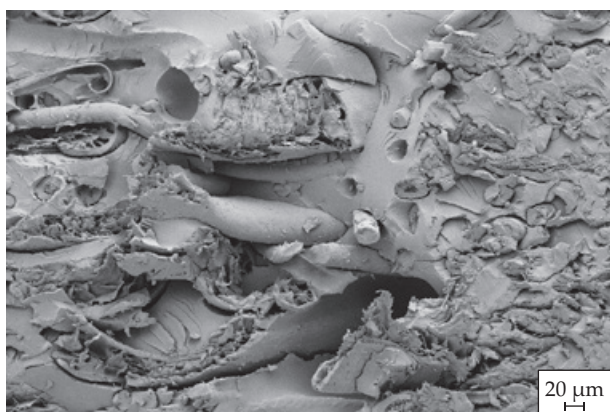
Fig. 3. SEM images of C3 composite at various magnification

Fig. 4. SEM images of C7 composite at various magnification

SEM analysis was performed for two composites with the best (C3) and worst (C7) mechanical and thermal properties. It was observed that cryogenic treatment affects the structure of the composites. SEM images of the C3 composite (Fig. 3) show good interactions between the polymer matrix and the reinforcement, which results in its good properties. The high micro-cracking resistance of this composite can be explained by the appropriate amount of human hair [21] and cryogenic processing. In Fig. 4, the presence of pores resulting from fiber disruption is clearly visible. In the case of the C3 composite, the interfacial interactions are the best compared to other composites due to the greater infiltration of liquid nitrogen caused by the lowest content of goat hair.

CONCLUSIONS

The study examined the effect of human and goat hair on the structure, mechanical and thermal properties of epoxy resin. The composites were cryogenically treated to improve mechanical properties. Flexural, tensile, and compressive strength, impact strength and hardness were determined. The composites were also subjected to thermogravimetric analysis. The composite with a reinforcement of 40 wt% goat hair and 60 wt% human hair showed the best mechanical and thermal properties. Moreover, the composite reinforced with only goat hair was characterized by the lowest mechanical properties and thermal stability. SEM confirmed good interactions at the phase boundary. This work can be extended by

manufacturing composites with different reinforcement orientations to further improve properties.

Conflict of interest
The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

REFERENCES

[1] Ali H., Rohit K., Dixit S.: *Journal of Natural Fibers* **2023**, *20(1)*, 2181268.
https://doi.org/10.1080/15440478.2023.2181268
[2] Waghmare S., Shelare S., Aglawe K. *et al.*: *Materials Today: Proceedings* **2022**, *54(3)*, 682.
https://doi.org/10.1016/j.matpr.2021.10.379
[3] Manivannan J., Rajesh S., Mayandi K. *et al.*: *Journal of Natural Fibers* **2022**, *19(11)*, 4007.
https://doi.org/10.1080/15440478.2020.1848743
[4] Neto J., Queiroz H., Aguiar R. *et al.*: *Journal of Renewable Materials* **2022**, *10(3)*, 561.
https://doi.org/10.32604/jrm.2022.017434
[5] Jayaseelan J., Vijayakumar K.R., Ethiraj N. *et al.*: *IOP Conference Series: Materials Science and Engineering* **2017**, *282*, 012018.
https://doi.org/10.1088/1757-899X/282/1/012018
[6] Verma A., Singh V. K., Verma S.K. *et al.*: *International Journal of Waste Resources* **2016**, *6*, 1000206.
http://dx.doi.org/10.4172/2252-5211.1000206
[7] Lakshmaiya N., Kaliappan S., Patil P.P. *et al.*: *Coatings* **2022**, *12(11)*, 1675.
https://doi.org/10.3390/coatings12111675
[8] Kumar Sinha H., Thakur N.: *International Journal of Aerospace, Mechanical, Structural and Mechatronic Engineering* **2015**, *1*, 2454.
[9] Gupta A.: *Journal of Waste Management* **2014**, 498018.
https://doi.org/10.1155/2014/498018
[10] Jayachandran A.H., Mercy J.L: *International Journal of Chemical Technology Research* **2016**, *9(3)*, 57.
[11] Mathew T., VS A.K., Nr S. *et al.*: *International Journal of Engineering and Computer Science* **2017**, *4*, 6667.
[12] Velmurugan G., Natrayan L., Chohan J.S. *et al.*: *Biomass Conversion and Biorefinery* **2023**.
https://doi.org/10.1007/s13399-023-04591-1
[13] Shindo Y., Takeda T., Narita F.: *Cryogenics* **2012**, *52(10)*, 564.
https://doi.org/10.1016/j.cryogenics.2012.07.008
[14] Li Y., Wei Y., Meng J. *et al.*: *Composites Communications* **2022**, *35*, 101326.
https://doi.org/10.1016/j.coco.2022.101326
[15] Rahman F., Wahid-Saruar M., Shefa H.K. *et al.*: *Journal of Natural Fibers* **2023**, *20(1)*, 2168820.
https://doi.org/10.1080/15440478.2023.2168820
[16] Vinod B., Sudev L.J.: *Materials Today: Proceedings* **2022**, *64(1)*, 330.
https://doi.org/10.1016/j.matpr.2022.04.692
[17] Kumar T.N., Goutami K., Aditya J. *et al.*: *IOSR Journal of Mechanical and Civil Engineering* **2015**, *12(4)*, 65.
http://doi.org/10.9790/1684-12466575
[18] Sreenivasa C.G., Joshi A.G.: "Influence of Cryogenic Treatment on Mechanical Behavior of Glass Fiber-Reinforced Plastic Composite Laminate" in "Polymers at Cryogenic Temperatures" (Editors: Kalia. S., Fu S.-Y.), Springer Berlin, Heidelberg 2013, p. 181.
[19] Zhang M., Pan R., Liu B. *et al.*: *Materials* **2023**, *16(1)*, 396.
https://doi.org/10.3390/ma16010396
[20] Shao Y., Xu F., Liu W. et al.: Composites Part B: Engineering 2017, 125, 195.
https://doi.org/10.1016/j.compositesb.2017.05.077
[21] Nanda B.P., Satapathy A.: *IOP Conference Series: Materials Science and Engineering* **2017**, *178*, 012012.
http://doi.org/10.1088/1757-899X/178/1/012012