Research Article

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Effects of cryogenic treatment and interface modifications of basalt fibre on the mechanical properties of hybrid fibre-reinforced composites

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Abstract: Although natural fibre-based thermoplastic composites (NFCs) have the advantages of environmental compatibility and low cost, their mechanical properties are still relatively poor. Hybridization with basalt fibres (BFs) can broaden the industrial applications of NFCs. Hybrid composites were manufactured by means of interlayer hybrid reinforcement; that is, the hybrid composites were prepared by the lamination moulding of BF prepregs and hemp/polylactide fibre (HF/PLA) felts. The effects of cryogenic treatment and interfacial modification in BF hybridization on the mechanical properties of HF/PLA composites were investigated. The study revealed that the hybridization of BFs with hemp fibres (HFs) significantly increased the mechanical properties of composites, and the cryogenic treatment and interface modification of BFs also improved the performance of hybrid composites. Compared with those of untreated BF-reinforced composites (UBF/HF/PLA), the tensile strength, flexural strength, and impact strength were increased by approximately 28.5% (120.82 MPa), 44.6% (90.29 MPa), and 192.1% (61.0 kJ/m²), respectively.

Keywords: basalt fibre, hybrid composites, cryogenic treatment, interface modification, mechanical properties

1 Introduction

At present, the demand for natural fibre-based thermoplastic composites (NFCs) is growing due to their low density and low cost (1). Especially in automotive components, NFCs have been used for door panels, seat backs, headliners, package trays, dashboards, and interior parts (2). Among various natural fibres, hemp fibre (HF) is a high-yield, cost-effective, functional, and environmentally friendly fibre, so it is regarded as an ideal reinforcement component in composites (3). However, the mechanical properties of NFCs are generally lower than those of their synthetic counterparts (4). Therefore, this study considered ways to improve the comprehensive performance of composites by adding other mineral fibre hybrids. The concept of hybridization involves embedding two or more types of fibres in a single matrix type to achieve a wider range of optimized properties (5). Hybridization is a common method to obtain properties that are between those of two original materials (6). The study has examined mixtures of natural and glass fibres (GFs) in reinforced composites (7). The results showed that basalt fibres (BFs) had superior mechanical properties compared to those of traditional high-performance fibres, especially GF (8). In addition to excellent mechanical properties, BFs have many other advantages: thermal resistance, water resistance, corrosion resistance, and chemical stability. These factors, coupled with the environmentally friendly characteristics of BF, mean that it has the potential to substitute or replace GF and serve as a new fibre in various applications (9–11). BFs provide better impact and environmental resistance than the corresponding hybrid GFs, and their cost is much lower than that of carbon fibre- and Kevlar fibre-reinforced composites (12). Therefore, hybrid composites, especially BF-reinforced hybrid composites, have been the focus of recent research.

In addition to the study of hybrids of traditional high-performance fibres, the most researched area of hybrid composites is the combination of traditional and hybrid

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vegetal fibres, and GF is particularly favoured by researchers. Fiorea *et al.* (13) studied the effect of external layers of GF fabric on the pinhole strength of flax/epoxy laminates. The results showed that the presence of GF fabric had a positive effect on the mechanical properties of mechanically fastened joints. Ahmed *et al.* (14) confirmed that the damage tolerance of jute-glass hybrid laminates was higher than that of jute laminates. Zhang *et al.* (15) studied the hybrid effects of flax/GF-reinforced hybrid composites. With increasing GF content, the tensile properties of the hybrid composites improved. Due to the excellent performance of BFs, researchers compared BFs with GFs and gradually changed their emphasis to BF hybrid composites. Wei *et al.* (7) used NaOH and HCL to etch BF and GF. The results show that BFs have better chemical stability and stronger acid resistance. Dhakal *et al.* (16) studied the effects of hybridization of BFs on the post-impact behaviour and damage tolerance capability of HF-reinforced composites. It was found that the addition of BFs to HF-reinforced composites significantly improved the residual performance and damage tolerance of the composites after impact.

The diversity of reinforcement fibres and matrices is behind the development trends in diversification and functionalization of hybrid fibre-reinforced composites and improves the freedom to design composites. In this study, a BF hybrid HF-reinforced polylactide composite laminate was prepared to obtain better mechanical properties at a lower cost and with significant environmental benefits. The effects of HF content, cryogenic treatment, and interface modification of BFs on the mechanical properties of composites were investigated.

2 Experimental methods

2.1 Materials

The experimental raw materials and reagents are shown in Table 1.

2.2 Fibre treatment

2.2.1 Cryogenic treatment

Before cryogenic treatment, BF surfaces were pre-treated with acetone to remove impurities. The SLX cryogenic

Table 1: The raw materials and reagents

No.	Raw materials and reagents	Manufacturer
1	Hemp stems	Heilongjiang Province, China
2	PLA fibres	Dongguan Mingxian Plastic Co., Ltd
3	Continuous basalt fibre	Sichuan Aerospace Tuoxin Basalt Industry Co., Ltd
4	Acetone	Beijing chemical plant
5	Absolute ethanol	
6	Dichloromethane	
7	Deionized water	Self-made
8	Silane coupling agent KH550	Kangjin New Material Technology Co., Ltd
9	PLA particles	Zhonglian Plastic Technology Co., Ltd

Figure 1: Programme chart of cryogenic temperature control of BF.

treatment system (SLX-30, Institute of Physics and Chemistry, Chinese Academy of Sciences, China) was used to make the BFs undergo uniform cryogenic treatment. For BF, its thermal conductivity was not as good as that of metal, and its structure was relatively stable. Therefore, a short-term cryogenic treatment may not achieve the desired effect. Considering comprehensively, drawing on the literature (17), a programme was diagrammed for the cryogenic temperature control of BFs, as shown in Figure 1. The cryogenic treatment process was a cooling rate of $2^{\circ}\text{C}/\text{min}$, holding for 1 h at -80°C , holding for 1 h at -120°C , holding at -196°C for 12 h, and finally, returning to ambient temperature without control.

2.2.2 Interface modification

To select the best modified concentration of a silane coupling agent, KH550, for BFs, KH550 solutions with concentrations of 0.5%, 1.0%, 1.5%, and 2.0% were prepared, and the cryogenically treated BFs were modified. Table 2 shows the abbreviations for different treatments of BF.

Table 2: Abbreviations for different treatments of BFs

No.	Basalt fibre	Abbreviation
1	Untreated basalt fibre	U-BF
2	Cryogenically treated basalt fibre	C-BF
3	0.5% KH550 modified basalt fibre after cryogenic treatment	K0.5-BF
4	1.0% KH550 modified basalt fibre after cryogenic treatment	K1.0-BF
5	1.5% KH550 modified basalt fibre after cryogenic treatment	K1.5-BF
6	2.0% KH550 modified basalt fibre after cryogenic treatment	K2.0-BF

2.3 Prepreg preparation

The solution immersion method was used, the solvent was dichloromethane, and the solution preparation method was as follows: the PLA particles were dried in an oven at 80°C for 4 h, and then a PLA solution with a mass fraction of 6% was prepared to fully dissolve the particles. BFs were placed in a mould (the mould size of tensile test pieces of prepreg was 230 mm × 200 mm × 1 mm, and the other size was 200 mm × 140 mm × 1 mm, which was the same size as the compression mould), the prepared PLA solution was poured into the mould; after 1 h, the impregnated fibres were dried in an oven at 80°C for 4 h to constant weight.

2.4 Preparation of composites

HF/PLA felts were prepared according to the method in ref. (18); the HF content was 30, 40, and 50 wt%. The release agent was sprayed evenly on the mould, and BF prepreps, HF/PLA felts, and BF prepreps were put into the compression mould (homemade mould: 200 mm × 140 mm × 2 mm) in order. Finally, a composite sheet was made on a press vulcanizer (BD-8820-BE-50T, Baoding Precision Instrument Co., Ltd., China) (185°C, 9 MPa, 6 min). The manufacturing process of the composites is shown in Figures 2 and 3.

3 Test methods

3.1 Surface morphology analysis

3.1.1 Scanning electron microscopy (SEM)

The morphology of the surface of the BFs and the fracture surfaces of the composites were observed with a scanning

Figure 2: The manufacturing process of composites (conceptual diagram).

electron microscope (XL-30 ESEM FEG, FEI, United States). The samples were coated with gold, and the accelerating voltage was 20 kV.

3.1.2 Atomic force microscopy (AFM)

The morphology of the surfaces of the BFs was observed with an atomic force microscope (Multimode 8, Brooke Company, United States). The surface roughness parameters of the fibres were recorded and analysed.

3.2 Tensile test of prepreg

Based on GB/T 3354-2014 (19), a universal testing machine (WSM-5KN, Changchun Intelligent Instrument Equipment Corp., China) was used to measure the tensile properties of BF prepreps. The size of the tested sample was 230 mm × 12.5 mm. The two ends added reinforcements of the same

Figure 3: The manufacturing process of composites.

material, the size of which was $50 \text{ mm} \times 12.5 \text{ mm}$, and there are four pieces in total. The tensile rate of the test was 2 mm/min , and the test environment conditions were 25°C and a relative humidity of 65%. There were six groups of samples; the number of valid test samples in each group was five, and the average value was evaluated and calculated.

3.3 Microstructural test

3.3.1 Fourier transform infrared (FTIR) spectroscopy

The infrared spectrum of BFs was recorded with an FTIR spectrometer (FTIR-4100, JASCO, Japan). Approximately 20 mg KBr and 2 mg fibres were mixed, ground, and then pressed into small discs.

3.3.2 X-ray diffraction (XRD)

X-ray diffraction (D/Max-2550PC, Rigaku, Japan) was used to analyse the changes in the molecular structure

of the BFs. The scanning parameters were as follows: CuK α radiation (40 kV, 40 mA), scanning range of $10\text{--}60^\circ$, and scanning speed of $2^\circ/\text{min}$.

3.4 Mechanical testing of composites

Based on ASTM D638-03 (20), the tensile properties of the sample were measured. The shape of the sample is dog bone with the size of $165 \text{ mm} \times 19 \text{ mm}$, the clamp spacing of 115 mm, and the tensile rate of 2 mm/min . Our sample condition meets Type I in ASTM D638-03. Based on ASTM D790-17 (21), the flexural samples properties of the sample were measured. The sample size was $80 \text{ mm} \times 10 \text{ mm}$, the span was 32 mm, and the indenter reduction rate was 2 mm/min . Based on GB/T1843-2008 (22), a Charpy test device (JJ-20B, Changchun Intelligent Instrument Equipment Corp., China) was used to measure the impact properties of the samples. The sample size was $80 \text{ mm} \times 10 \text{ mm}$. The test environment conditions were 25°C and a relative humidity of 65%. There were fifteen groups of samples, the number of valid test samples in each group was five, and the average value was evaluated and calculated.

4 Results and discussion

4.1 Effect of treatment of BFs

4.1.1 Fibre surface morphology

Figure 4 shows SEM micrographs of the surfaces of BFs. In Figure 4a and b, the surface of the untreated BFs was smoother, and the surfaces of the BFs after cryogenic treatment were rough, and some bulges were observed. On the one hand, the specific surface areas of the BFs were increased, and the contact surface with the resin matrix was increased, thereby improving the dispersion of the fibres in the matrix. The friction on the surfaces of the fibres was increased, and the bonding strength of the interface between the fibres and the matrix was enhanced.

The reason for the differences in the surface of BFs before and after cryogenic treatment was considered. From a macro perspective, BFs undergo thermal expansion and contraction, and the temperature is too low, resulting in permanent and irreversible changes. In addition, according to the analysis of the structure of BFs by researchers (23), BFs are an intermediate substance with amorphous components as the main component, and they are relatively ordered in the lengthwise direction

Figure 5: Short-range structural sketch of BF.

of the fibre. The amorphous microstructure was connected to an irregular three-dimensional grid in close range. A partial short-range sketch of the BF is shown in Figure 5. Therefore, from a microscopic perspective, BFs may undergo internal during the treatment process (17).

In Figure 4c, when the KH550 content was 0.5%, the fibre surfaces did not improve significantly, and there were fewer protrusions. Compared with the C-BF shown in Figure 4b, this was not a significant improvement. On the one hand, the reason may be that the modified concentration of 0.5% KH550 was not high, and it had no obvious substantial effect on BFs. On the other hand, there may have been some improvement in the fibre, but no obvious change was observed. As shown Figure 4d, when the content of KH550 was 1.0%, the fibre surface had a greater improvement compared with that of K0.5-BF, and the fibre surface convexity increased, but the convex shape was smaller. As shown in Figure 4e, when the content of KH550 was 1.5%, the convexity of the fibre surface increased and was distributed in strips. As shown in Figure 4f, when the content of KH550 was 2.0%, the fibre was similar to KH1.5-BF, showing strip-like protrusions.

The protrusions on the fibre surface helped the fibre bond with the matrix. Fibre modification can improve the interfacial performance of the fibre. KH550 was used for surface modification to enhance the performance of BFs; at the same time, it formed a layer of silane film on the surface of the fibre, which not only protected the fibres from oxidation and corrosion, but also improved the surface activity of the BFs.

Figure 6 shows AFM micrographs of the surface of BFs. The surfaces of the untreated BFs were smooth, and some protrusions appeared on the surface after cryogenic treatment. The protrusions on the modified BFs gradually increased, increasing its surface area and improving the bonding ability with the matrix. This is consistent with what is observed in the SEM image.

Figure 4: SEM micrographs of (a) U-BF, (b) C-BF, (c) K0.5-BF, (d) K1.0-BF, (e) K1.5-BF, and (f) K2.0-BF.

Figure 6: AFM micrograph of (a) U-BF, (b) C-BF, (c) K0.5-BF, (d) K1.0-BF, (e) K1.5-BF, and (f) K2.0-BF.

Table 3: Surface roughness parameters of BFs

	RMS (nm)	Ra (nm)	Rmax (nm)	SA (μm^2)
U-BF	223	162	812	20.6
C-BF	291	219	1,117	23.3
K0.5-BF	352	246	1,179	28.8
K1.0-BF	359	277	1,333	30.8
K1.5-BF	415	321	1,726	31.7
K2.0-BF	531	417	2,200	32.6

In addition, the surface roughness parameters of BFs are shown in Table 3, which proves that the surface roughness of BFs gradually increased with the different surface treatment methods.

4.1.2 Tensile properties of prepregs

Figure 7 shows that compared with that of U-BF, the tensile strength of other BF prepregs was greatly improved. The tensile strength of C-BFs increased by 10%, and that of K1.0-BF increased by more than 20%. When the concentration of KH550 was 1.0%, the modification effect was the best, and the maximum tensile strength reached 334 MPa. It has also been observed that even when the concentration of KH550 was between 1.5% and 2.0%, the tensile strength of BF prepregs was lower than that of K1.0-BF. However, compared with that of U-BF and C-BF, the tensile strength still improved. Cryogenic treatment can improve the tensile properties of BFs to a certain extent, and proper modification can also improve the tensile properties of BFs.

Figure 7: Tensile strength of BF prepreps.

The analysis showed that the modified concentration of coupling agent had an optimal value. When it was lower than the optimal value, the interface bonding ability was insufficient, making its performance poor. When it was higher than the optimal value, although the surface roughness was greater, the BF was damaged, so the performance was not as good as that under the optimal modified concentration.

4.1.3 FTIR analysis

According to the comprehensive analysis in Sections 4.1.1 and 4.1.2, U-BF, C-BF, and K1.0-BF were selected as the research objects for FTIR analysis. Figure 8 shows the FTIR spectra of BFs. The curves of the three spectra were basically the same, which indicated that the chemical properties of the BFs were not affected by the treatment. The spectrum had a wide band in the range of

Figure 8: FTIR spectra of (a) U-BF, (b) C-BF, and (c) K1.0-BF.

3,600–3,200/cm, which was mainly attributed to the stretching vibration of the OH groups, which may be caused by residual water or silanol groups ($-\text{Si}-\text{OH}$) adsorbed on the fibre surface (24,25). In the range of 3,000–2,800/cm, characteristic CH stretch bands derived from methylene (CH_2) and methyl (CH_3) groups can be observed. This ensured the organic properties of the fibre, which is a typical feature of the most common sizing agents used in the production of GFs and BFs (26). The $-\text{CH}_2-$ and $-\text{CH}-$ groups were located at peaks near 2,925 and 2,852/cm, respectively. In the low-energy region of the spectrum, there was an obvious carbonyl ($\text{C}=\text{O}$) absorption peak at 1,650/cm. The typical symmetric tensile vibration and asymmetric tensile vibration absorption peaks of $\text{Si}-\text{O}-\text{Si}$ were observed at approximately 1,010 and 705/cm, respectively.

As shown in Figure 8b, there was no obvious difference in the infrared spectrum of the fibre before and after cryogenic treatment, but the intensity of the absorption peak changed. This may be due to the change in the absorption peak caused by the rupture of the intermolecular hydrogen bonds in the structure of BFs, which made the structure more compact but did not produce chemical effects. As shown in Figure 8c, the spectrum of the modified BFs showed a significant decrease in OH groups and an increase in $\text{Si}-\text{O}$ groups. The $-\text{OH}$ on the surface of the modified BFs was hydrolysed. The Si in the KH550 solution adhered to the surface of BFs and formed $\text{Si}-\text{O}$ groups, which eventually led to an increase in the polar $\text{Si}-\text{O}$ bonds and improved the surface of the fibre.

4.1.4 XRD analysis

According to the comprehensive analysis in Sections 4.1.1 and 4.1.2, U-BF, C-BF, and K1.0-BF were selected as the research objects for XRD analysis. Figure 9 shows the XRD patterns of BFs. Amorphous silicate glass was arranged in a short-range order, which was suitable for the nearest neighbour atoms, and there was no periodicity at longer distances. In Figure 9, the three curves were similar, with diffuse diffraction peaks between 20° and 30° . BFs maintained a short-range order and long-range disordered glass structure before and after cryogenic treatment and modification treatment (27).

4.2 Effects of composites on mechanical properties

Figure 10 shows the mechanical properties of composites with different HF contents and BFs. Figure 10 shows that

Figure 9: XRD patterns of (a) U-BF, (b) C-BF, and (c) K1.0-BF.

the mechanical properties of the BF-reinforced composites were 2 to 11 times higher than those of the composites without BFs. With an increasing HF content, the mechanical properties of the composites first increased and then decreased, reaching a maximum at an HF content of 40 wt%, which indicated that the fibre content had an optimal ratio, and it was located at 40–50 wt%. Cryogenically treated BF-reinforced composites (CBF/HF/PLA) were compared with UBF/HF/PLA: tensile strength increased by 10.1–13.9%, up to 105.61 MPa, flexural strength increased by 19.1–31.1%, up to 79.52 MPa, and impact strength increased by 17.4–53.1%, up to 33.2 MPa. This showed that cryogenic treatment modified the BFs, which improved the interfacial performance of the BFs and the mechanical properties of the composites. Modified BF-reinforced composites (KBF/HF/PLA) were compared with UBF/HF/PLA: tensile strength increased by 14.7–28.4%, up to 120.82 MPa, flexural strength increased by 35.4–44.6%, up to 90.29 MPa, and impact strength increased by 148.1–192.1%, up to 61.0 MPa. This showed that the silane coupling agent improved the interfacial performance of BFs and the mechanical properties of the composites.

4.3 Analysis of the micromorphology of composites

4.3.1 Influence of HF content on the micromorphology of composites

As shown in Figure 11, the micromorphology of composites of HF/PLA felt with HF and the bonding of HF in

Figure 10: Mechanical properties of composites: (a) tensile strength, (b) flexural strength, and (c) impact strength.

the polylactide matrix were studied. It can be seen from Figure 11a and d that when the content of HF was low (30 wt%), the combination of fibre and polylactide was more uniform at the fracture surfaces of composites. The fractures were relatively regular, and there were many voids left after the fibres were pulled out of the matrix. It can be seen from Figure 11b and e that as the HF content increased (40 wt%), the area of fibres at the cross-section also increased, and the fibres and voids that were

Figure 11: SEM micrographs of composites: (a) 0-30, (b) 0-40, (c) 0-50, (d) U-30, (e) U-40, and (f) U-50.

Figure 12: SEM micrographs of composites: (a) UBF/HF/PLA, (b) CBF/HF/PLA, and (c) KBF/HF/PLA.

pulled out also increased. Compared with the 30 wt% composites, the 40 wt% composites had more fractured fibres and better interface bonding, which were consistent with the mechanical property test results. Figure 11c and f show that when the HF content reached 50 wt%, there was too much HF, it was not possible to completely cover the polylactide matrix, more fibres were exposed in the cross-section, the arrangement was disordered, and more holes were observed. This further proved that too many fibres reduced the mechanical properties of the composites.

4.3.2 Influence of BF treatment on the micromorphology of composites

Figure 12 shows cross-sections of untreated BFs (UBF/HF/PLA), cryogenically treated BFs (CBF/HF/PLA), and KH550-treated BFs (KBF/HF/PLA) combined with 40 wt % HF/PLA felt when subjected to external forces. Figure 12a shows that the gaps between U-BFs and HF/PLA felts were large, the surfaces of BFs were smooth, and the fibre fractures at the section were neat, which indicated that the interface performance of untreated BFs was low.

When composites were subjected to external forces, it was easier to peel the fibres from the HF/PLA felt and to pull the BFs out of the impregnating material and the matrix. Figure 12b shows that the gap between C-BFs and HF/PLA felts was smaller than that for UBF/HF/PLA. It was obvious that part of the matrix adhered to the surface of the BFs, and the fibre fracture was uneven. This showed that the adhesive force of the cryogenically treated BF increased. When composites were subjected to an external force, the fibres still peeled off from HF/PLA felts, but the cryogenic treatment modified the fibres to a certain extent. The material had a certain bearing capacity, which proved that the mechanical properties of CBF/HF/PLA were better than those of UBF/HF/PLA. Figure 12c shows that BFs treated with the silane coupling agent, KH550, were more closely bonded to the impregnating material and the matrix, a large amount of matrix was attached to the surface of the fibres, and the fibres at the cross-section were messier. This showed that the KH550 modification treatment improved the interface bonding properties of the fibres, which proved that the mechanical properties of KBF/HF/PLA were better than those of UBF/HF/PLA.

5 Conclusion

In this study, a BF hybrid HF-reinforced polylactide composite laminate was prepared to obtain better mechanical properties at a lower cost and with significant environmental benefits. The effects of HF content, cryogenic treatment, and interface modification of BFs on the mechanical properties of composites were discussed. After BFs underwent cryogenic treatment, the surfaces of the fibres were obviously improved, and there were some bulges. Different concentrations of KH550 silane coupling agent had different effects on fibre surface modification. As the concentration of KH550 solution increased, the BF surface bulges gradually increased, and the surface roughness also increased. Compared with that of U-BF, the tensile strength of C-BF was increased by 10%, and the tensile strength of K1.0-BF was increased by more than 20%. The mechanical properties of BF hybrid composites were greatly improved. As the content of HF increased, the mechanical properties first increased and then decreased, reaching a maximum at 40 wt% of HF. Compared with UBF/HF/PLA, CBF/HF/PLA (95.17, 77.04 MPa, 23.79 kJ/m²) and KBF/HF/PLA (107.25, 86.67 MPa, 50.78 kJ/m²) had better mechanical properties. Therefore, the properties of NFCs can be improved by means of hybrid fibres.

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