

Waste-to-Performance Thermoplastic Composites through Valorization of Residual Hemp Noil via Micronization and Interfacial Engineering

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Research Article

Keywords: Hemp powder, Lignocellulosic composites, Melt torque rheology, Fiber–matrix interface, Waste valorization

Posted Date: September 7th, 2026

DOI: <https://doi.org/10.21203/rs.3.rs-10829413/v1>

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Additional Declarations: No competing interests reported.

Abstract

Valorization of residual hemp fiber (noil) offers a sustainable route for converting textile waste into value-added polymer composites. Here, acid-treated and untreated hemp residues were micronized and incorporated into polypropylene (PP), thermoplastic starch (TPS), polylactic acid (PLA), and PLA/poly(butylene adipate-co-terephthalate) (PBAT) systems through melt torque rheology using maleic anhydride, urea, and epoxidized soybean oil (ESO) as interfacial modifiers. Acid treatment reduced the average hemp-powder particle size from 63.14 to 19.67 μm , facilitating dispersion and fiber–matrix interactions. Mechanical testing revealed pronounced formulation-dependent behavior, with the PLA/MAH system reaching a maximum tensile stress of 11.23 MPa, while HP/TPS/MAH achieved a flexural stress of 30.28 MPa. Thermal stability was highest for HP/PP/MAH, exhibiting a maximum degradation temperature (T_{max}) of 470.5°C. FTIR and SEM further indicated treatment-dependent changes in surface chemistry and interfacial morphology. Collectively, these findings demonstrate the feasibility of converting residual hemp fibers into micronized reinforcements for sustainable thermoplastic composites.

Highlights

- Residual hemp noil was valorized as reinforcement for thermoplastic composites.
- Acid treatment reduced hemp particle size from 63.14 to 19.67 μm .
- Melt torque rheology enabled processing with PP, TPS, PLA, and PBAT matrices.
- PLA/MAH achieved a maximum tensile stress of 11.23 MPa.
- HP/PP/MAH exhibited the highest degradation temperature of 470.5°C.

1. Introduction

The growing demand for sustainable materials and circular manufacturing has intensified interest in converting lignocellulosic residues into value-added polymer composites. Industrial hemp (*Cannabis sativa* L.) is particularly attractive because of its renewability, biodegradability, low density, and favorable specific mechanical properties [1, 2]. Hemp fibers have consequently found applications in textiles, automotive components, construction materials, and polymer reinforcement [3–5]. However, hemp processing, particularly during yarn production, generates substantial quantities of short residual fibers or noil that are difficult to process using conventional textile technologies and are therefore frequently discarded or underutilized [6–8]. Valorizing these residues as reinforcing fillers provides an opportunity to reduce industrial waste while decreasing dependence on conventional synthetic reinforcements and supporting circular-material strategies [9–11].

Despite these advantages, direct incorporation of lignocellulosic hemp residues into thermoplastic matrices remains challenging. Hemp fibers contain cellulose together with hemicellulose, lignin, pectin, waxes, and other surface constituents [12, 13]. These non-cellulosic components can hinder wetting and

interfacial interactions between the hydrophilic reinforcement and comparatively hydrophobic polymer matrices, resulting in poor dispersion, interfacial voids, and inefficient stress transfer [14, 15]. Chemical surface treatment can partially remove these constituents, expose cellulose-rich surfaces, and increase surface roughness and accessible hydroxyl functionality, thereby promoting fiber–matrix interactions [16, 17]. Converting residual fibers into micron-sized powder may further facilitate homogeneous dispersion by increasing the effective interfacial area available for interaction with the surrounding matrix [18, 19]. Consequently, coupling chemical treatment with particle-size reduction represents a potentially effective strategy for transforming difficult-to-process hemp residues into functional composite reinforcements.

Matrix chemistry and compatibilization are equally important in determining composite performance. Polypropylene (PP) is widely employed in natural-fiber composites because of its low density, processability, and commercial availability, although its nonpolar nature limits adhesion to lignocellulosic surfaces [20, 21]. Maleic-anhydride-based coupling agents can improve compatibility by promoting interactions between polymer chains and cellulose hydroxyl groups. Previous studies on hemp/PP systems have demonstrated that surface treatment and maleated compatibilizers can enhance mechanical performance and reduce moisture sensitivity [22]. Nevertheless, most reported studies focus on conventional hemp fibers rather than micronized residual hemp noil, leaving the processing–structure–property relationships of this industrial by-product insufficiently understood.

Biodegradable and bio-derived matrices provide an additional route toward more sustainable hemp composites. Thermoplastic starch (TPS) is renewable, inexpensive, and biodegradable, but its relatively poor mechanical and barrier properties restrict broader structural applications. Polylactic acid (PLA) offers comparatively high strength and good processability but suffers from inherent brittleness and limited toughness [23]. Blending PLA with flexible poly(butylene adipate-co-terephthalate) (PBAT) can improve ductility; however, limited PLA/PBAT compatibility may constrain stress transfer and mechanical performance [24, 25]. Compatibilizers and reactive additives, including maleic anhydride (MAH), urea, and ESO[26], therefore provide potential routes to regulate interactions among hemp particles and these chemically distinct polymer matrices [27]. The supplementary analysis similarly emphasizes the central role of interfacial adhesion in PP-, TPS-, and PLA/PBAT-based hemp composites.

An additional challenge is understanding how highly filled hemp/polymer formulations behave during melt processing. Rheological behavior governs mixing, dispersion, wetting, and ultimately manufacturability by processes such as extrusion and injection molding [28, 29]. Although extensive research has examined natural-fiber composites, comparatively limited attention has been devoted to residual hemp noil converted into micron-sized powder and its melt-torque rheological behavior across different thermoplastic matrices [30, 31]. More importantly, a systematic comparison of acid-treated and untreated residual hemp powders in PP, TPS, PLA, and PLA/PBAT formulations under comparable melt-processing conditions remains lacking. This gap limits understanding of how physicochemical treatment, particle size, matrix selection, and compatibilization collectively govern processability, interfacial structure, and resulting composite properties.

Here, residual hemp fibers were valorized by physicochemical treatment and micronization, followed by incorporation into PP-, TPS-, PLA-, and PLA/PBAT-based thermoplastic systems using melt torque rheology. Acid treatment reduced the characteristic powder size from approximately 63.14 μm for untreated material to 19.67 μm for treated hemp, providing distinctly different reinforcement morphologies for comparative evaluation. The effects of matrix composition and compatibilizing additives, including MAH, urea, and ESO, were systematically investigated. Torque rheology was employed to elucidate melt-processing behavior, while mechanical testing, thermogravimetric analysis, Fourier-transform infrared spectroscopy, and scanning electron microscopy were used to establish relationships among processing, interfacial interactions, morphology, thermal stability, and mechanical performance. Through this integrated approach, the study seeks to establish a practical pathway for converting underutilized hemp-processing residues into value-added thermoplastic composites and to advance their potential use in sustainable automotive, construction, packaging, and related material applications.

2. Experimental

2.1 Materials

Two types of hemp fiber were obtained from Wuhan Hemp Biological Technology Co., Ltd. (Wuhan, China), while residual hemp stalks were collected from the Wuhan Textile University laboratory (Wuhan, China). Sulfuric acid (H_2SO_4), used for fiber hydrolysis, was supplied by Wuhan Jiangbei Chemical Reagent Co., Ltd. (Wuhan, China), and JFC was purchased from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). For composite fabrication, polylactic acid (PLA) was obtained from Anhui BBKA Biochemical and Futero-Pla Co., Ltd. (Anhui, China), polypropylene (PP) from Sinopec Beihai Refining and Chemical Co., Ltd. (Shanghai, China), poly(butylene adipate-co-terephthalate) (PBAT) from Zhejiang Huaon Environment Protection Materials Co., Ltd. (Zhejiang, China), and thermoplastic starch (TPS) from Shandong Runquan Biotechnology Co., Ltd. (Shandong, China). Maleic anhydride (MAH), used as an interfacial compatibilizing agent, was supplied by Tianjin Damao Chemical Reagent Co., Ltd. (Tianjin, China). Analytical-grade urea (AR) was obtained from Tianjin Cameo Chemical Reagent Co., Ltd., and epoxidized soybean oil (ESO) was supplied by Hairma (Nantong) Technology Co., Ltd. (Nantong, China). All fibers, polymers, chemicals, and reagents were used as received without further purification.

2.2 Preparation of Hemp Powder and Composite Samples

Residual hemp fibers were cut into approximately 2–3 mm pieces prior to physicochemical treatment. For the acid treatment, 10 g of hemp fiber was immersed in 200 mL of deionized water containing sulfuric acid (2 g L^{-1}) and JFC (2 g L^{-1}), and the mixture was maintained in a water bath at 100°C for 1 h. After treatment, the fibers were thoroughly washed with deionized water 7–8 times until a neutral pH (~ 7) was reached. Excess water was removed using a hand roller, and the treated fibers were subsequently oven-dried at 200°C for 1 h. For comparison, untreated residual hemp material was cut into approximately 2–3 mm pieces and subjected to the same drying procedure without chemical treatment.

The dried treated and untreated hemp fibers were subsequently pulverized using an XL-06B high-speed crusher (Aashia Company, China; 1100 W, 220 V, 25,000 rpm). Approximately 1 kg of material was processed per batch. This grinding process produced micron-sized hemp powders, with average particle sizes of approximately 19.67 μm for the acid-treated fibers and 63.14 μm for the untreated fibers, as determined in this study. The powder-preparation process is illustrated in **Figure S3**.

The resulting hemp powders were combined with the respective polymer matrices and additives according to the formulations listed in Table 1. Before melt processing, hemp powder and the corresponding polymer/additive components were premixed in the crusher for 1–2 min to promote homogeneous distribution. Melt compounding was then performed using an RM-200C torque rheometer (Harpo Electrical Technology Co., Ltd., Harbin, China). The mixing chamber was preheated to the required processing temperature and filled to approximately 95% of its capacity. Processing temperatures of 120°C and 160°C were employed depending on the polymer formulation, with TPS-containing formulations processed at 120°C. The rotor speed was maintained at 50 rpm, and the samples were processed for 30 min. Torque and melt-temperature profiles were continuously recorded as functions of processing time using the instrument control and data-acquisition software. Following melt compounding, the resulting materials were compression-molded at 160°C for 5 min to obtain composite specimens for subsequent characterization. The probable chemical interactions during the fiber treatment and composition are schematically presented in Fig. 1.

(a)

Table 1
Formulations and processing conditions of composites prepared using (a) acid-treated and (b) untreated hemp powder.

(a)	Sample	HP (g)	PP (g)	TPS (g)	PLA (g)	PBAT (g)	MAH (g)	Urea (g)	ESO (mL)	Temperature (°C)	Time (min)
	S-1	15	12	–	–	–	3	–	–	160	30
	S-2	16	–	16	–	–	8	2	–	120	30
	S-3	16	–	–	16	–	8	–	–	160	30
	S-4	15	–	–	15	5	4	–	1.60	160	30
(b)	S-1	16	–	–	16	–	4	–	1.50	160	30
	S-2	16	2	–	–	–	4	–	–	160	30
	S-3	16	–	16	–	–	–	4	–	120	30

2.3 Characterizations and Measurements

The mechanical properties of the composites were evaluated using a universal testing machine (Shimadzu Enterprise Management Co., Ltd., Shenyang, China). Tensile testing was performed at a crosshead speed of 2 mm / min⁻¹, while three-point bending tests were conducted using a 10 kN load

cell, a support span of 30 mm, and a crosshead speed of 9 mm/ min^{-1} . All measurements were carried out at $23 \pm 5^\circ\text{C}$ and $50 \pm 5\%$ relative humidity. Seven specimens were tested for each formulation, and the average values were determined. The maximum load, maximum stress, displacement, and strain were recorded.

The surface morphology of the hemp powders and the fracture morphology of the composite specimens were examined by scanning electron microscopy (SEM; SU8600, Hitachi). Prior to imaging, the samples were sputter-coated with gold and examined at an accelerating voltage of 3 kV. Fourier-transform infrared (FTIR) spectroscopy was employed to investigate the chemical characteristics of the hemp powders and corresponding composites using an IRTracer-100 spectrometer (Shimadzu Corporation, Kyoto, Japan) in attenuated total reflectance (ATR) mode. Spectra were acquired at a resolution of 4 cm^{-1} with 40 scans per sample.

Thermal stability and degradation behavior were investigated by thermogravimetric analysis (TGA) using an STA 300 thermobalance (Hitachi High-Tech Science Corporation, Tokyo, Japan). Samples weighing approximately 5–9 mg were heated from 30 to 700°C at a heating rate of $10^\circ\text{C min}^{-1}$ under a continuous nitrogen flow of 66 mL min^{-1} .

3. Results and Discussion

3.1. Torque Rheological Behavior

The melt-processing behavior of the seven composite formulations was evaluated by torque rheometry to assess the effects of hemp-fiber treatment, polymer matrix, and formulation on flow behavior during compounding. The acid-treated and untreated hemp powders had average particle sizes of approximately 19.67 and $63.14 \mu\text{m}$, respectively, and were incorporated into different PP, TPS, PLA, and PLA/PBAT-based formulations Table 1. Because torque reflects the resistance of the material to rotor motion, changes in the torque–time profile provide an indication of the evolution of melt resistance during mixing and the establishment of a relatively stable compounded state.

As shown in Fig. 2a, the acid-treated formulations exhibited distinct torque–time profiles depending on the polymer/additive composition. A relatively high torque was observed during the initial stage of mixing, followed by a progressive decrease for most formulations. This behavior can be attributed to the initial resistance of the solid components to rotor motion, followed by polymer softening/melting and progressive dispersion of the hemp powder under shear. As mixing proceeded, the reduced melt resistance resulted in lower torque and a tendency toward a more stable regime. Such torque relaxation during melt processing is consistent with increased molecular mobility and homogenization of the polymer–fiber mixture [32]. Marked formulation-dependent differences were nevertheless observed. For the acid-treated composites, S-1 (HP/PP/MAH) and S-3 (HP/PLA/MAH) showed a more pronounced decrease in torque with processing time, whereas S-2 (HP/TPS/MAH/urea) and particularly S-4 (HP/PLA/PBAT/MAH/ESO) displayed different torque evolution Fig. 2a. The comparatively high and

sustained torque of S-4 indicates greater resistance to melt deformation during the early-to-intermediate stages of processing. This behavior is reasonably associated with its multicomponent PLA/PBAT/hemp formulation and the presence of MAH and ESO, which collectively alter the melt response compared with the simpler PP- and PLA-based systems. However, because the formulations differ simultaneously in polymer type, additive composition, and component proportions, the torque differences cannot be attributed exclusively to hemp-particle size or acid treatment.

The untreated-hemp formulations also exhibited composition-dependent rheological behavior Fig. 2b. S-1 (WHP/PLA/MAH/ESO) and S-2 (WHP/PP/MAH) showed an initial increase followed by a substantial reduction in torque, indicating decreasing resistance as the polymer phases softened and the components became progressively mixed. In contrast, S-3 (WHP/TPS/urea), processed at 120°C, maintained a substantially higher torque and showed a gradual increase at longer processing times. The higher melt resistance of this formulation is consistent with its different TPS-based composition and lower processing temperature. Reduced temperature generally restricts polymer-chain mobility and consequently increases resistance to flow, although the contribution of urea and the specific TPS/hemp interactions cannot be separated from the temperature effect using the present dataset [33].

3.2. Mechanical Properties

The mechanical performance of the composites was evaluated by tensile and three-point flexural testing to elucidate the effects of hemp treatment, polymer matrix, and compatibilizing additives. The fracture images of samples after tensile experiments are shown in Fig. 2c-d. The acid-treated formulations exhibited pronounced differences in tensile behavior Table 2a. S-1 (HP/PP/MAH) showed the lowest measurable tensile performance, with a maximum load and stress of 3.72 N and 0.162 MPa, respectively. Incorporation of TPS in S-2 (HP/TPS/MAH) increased these values to 15.50 N and 0.674 MPa [34], although the maximum strain remained comparable to S-1 (2.81 vs. 2.84%). Thus, changing the matrix from PP to TPS under the investigated formulation conditions substantially increased the load-bearing capacity but produced little change in deformation at maximum load [35]. A substantially greater tensile response was obtained for the PLA-containing formulations [36]. S-3 (HP/PLA/MAH) exhibited the highest maximum load (258.20 N) and stress (11.23 MPa), representing approximately 69- and 69-fold increases, respectively, relative to S-1. This pronounced improvement indicates that the PLA-based formulation provided substantially greater resistance to tensile loading under the investigated conditions. The favorable performance is consistent with the inherently good mechanical properties of PLA and the role of MAH-containing compatibilization strategies in promoting interfacial interactions [37]. S-4 (HP/PLA/PBAT/MAH/ESO) exhibited a slightly lower maximum load (245.94 N) and stress (10.69 MPa) than S-3, corresponding to decreases of approximately 4.8%. In contrast, its maximum displacement and strain increased from 1.15 mm and 3.85% for S-3 to 1.26 mm and 4.20%, respectively. This combination of slightly reduced strength and increased deformation suggests a more compliant mechanical response of the multicomponent PLA/PBAT formulation. However, because S-3 and S-4 differ simultaneously in hemp content, PBAT, and ESO, the increased deformation cannot be assigned to an individual component without additional controls[38]. The untreated-hemp formulations showed

generally lower tensile performance than the strongest acid-treated formulations Table 2b. S-1 (WHP/PLA/MAH/ESO) exhibited a maximum load of only 6.11 N and maximum stress of 0.266 MPa, whereas S-3 (WHP/TPS/MAH/urea) reached 82.63 N and 3.59 MPa. S-2 (WHP/PP/MAH) was reported as too weak to provide reliable tensile measurements. These results demonstrate a strong dependence of tensile behavior on formulation. The comparatively high performance of the urea-containing TPS formulation suggests favorable interactions within this system; however, the present experimental design does not isolate the effect of urea, and therefore a direct causal relationship should not be claimed.

Table 2
Tensile properties of composite formulations prepared with (a) acid-treated and (b) untreated hemp powder

(a)	Sample	Maximum load (N)	Maximum stress (MPa)	Maximum displacement (mm)	Maximum strain (%)
	S-1 (HP/PP/MAH)	3.720	0.162	0.851	2.838
	S-2 (HP/TPS/MAH)	15.500	0.674	0.844	2.815
	S-3 (HP/PLA/MAH)	258.204	11.226	1.154	3.848
	S-4 (HP/PLA/PBAT/MAH/ESO)	245.938	10.693	1.260	4.201
(b)	S-1 (WHP/PLA/MAH/ESO)	6.112	0.266	0.714	2.379
	S-2 (WHP/PP/MAH)	–	–	–	–
	S-3 (WHP/TPS/MAH/Urea)	82.633	3.593	0.807	2.689
	– Not measurable				

The flexural response also varied markedly among formulations Table 3a. Among the acid-treated systems, S-2 (HP/TPS/MAH) exhibited the highest maximum flexural load and stress, reaching 15.25 N and 30.28 MPa, respectively. These values were substantially higher than those of S-1 (3.12 N, 6.26 MPa), S-3 (5.04 N, 10.01 MPa), and S-4 (4.37 N, 8.67 MPa). The superior flexural resistance of S-2 indicates that the HP/TPS/MAH formulation was particularly effective in resisting bending deformation under the investigated conditions. Interestingly, the formulation ranking differed between tensile and flexural loading. Although S-3 and S-4 showed the greatest tensile loads and stresses, their flexural strengths were substantially lower than that of S-2. Conversely, S-3 and S-4 exhibited much greater displacement at maximum flexural load (4.95 and 4.90 mm, respectively) than S-1 and S-2 (0.10 and 0.69 mm). This contrasting behavior demonstrates that the formulations optimized for tensile load bearing were not necessarily those providing the greatest resistance to bending. The relatively large flexural displacement of the PLA-containing formulations indicates greater deformation before reaching maximum bending load, whereas S-2 combined comparatively high flexural stress with lower displacement. For the untreated-hemp composites (Table 3b), S-3 (WHP/TPS/MAH/urea) again

exhibited the strongest flexural response, with a maximum load of 16.45 N and maximum stress of 32.66 MPa. These values exceeded those of S-1 (1.80 N, 3.57 MPa) and S-2 (3.66 N, 7.26 MPa). Notably, S-3 also slightly exceeded the maximum load and stress of the corresponding acid-treated HP/TPS/MAH formulation (15.25 N and 30.28 MPa). Therefore, the available flexural data do not support a general conclusion that acid treatment universally improves mechanical performance. Instead, the results indicate that matrix/additive selection has a major influence on the mechanical response and can outweigh the effect of fiber pretreatment for specific formulations. The particularly strong tensile and flexural performance of the WHP/TPS/MAH/urea formulation suggests that urea-containing TPS systems merit further investigation. This improvement is attributed to enhanced fiber–matrix interactions; however, because no otherwise identical TPS formulation with and without urea was tested, the independent contribution of urea cannot be quantitatively established from the present dataset. Accordingly, enhancement should be discussed as a formulation-dependent effect rather than definitive evidence of urea-induced interfacial strengthening.

Table 3

Flexural properties of composite formulations prepared with (a) acid-treated and (b) untreated hemp powder

(a)	Sample	Maximum load (N)	Maximum stress (MPa)	Maximum displacement (mm)	Maximum strain (%)
	S-1 (HP/PP/MAH)	3.115	6.258	0.102	0.029
	S-2 (HP/TPS/MAH)	15.255	30.279	0.691	0.195
	S-3 (HP/PLA/MAH)	5.042	10.007	4.946	1.393
	S-4 (HP/PLA/PBAT/MAH/ESO)	4.367	8.669	4.897	1.379
(b)	S-1 (WHP/PLA/MAH/ESO)	1.798	3.569	0.196	0.055
	S-2 (WHP/PP/MAH)	3.659	7.262	0.631	0.161
	S-3 (WHP/TPS/MAH/Urea)	16.453	32.657	0.572	0.161

3.3. Morphological Characterization

The acid-treated composites exhibited distinctly different fracture morphologies depending on formulation (Fig. 3a-d). S-1 (HP/PP/MAH) showed a relatively rough and heterogeneous fracture surface containing numerous cavities and evidence of particle/fiber pull-out. Such voids and pull-out features are characteristic of localized interfacial debonding, where the reinforcing phase is detached from the matrix during fracture rather than remaining firmly embedded. These defects can interrupt stress transfer across the fiber–matrix interface and are consistent with the comparatively low tensile and flexural performance of S-1. In comparison, S-2 (HP/TPS/MAH) displayed a less rough and more compact fracture surface, with fewer prominent defects than S-1. The improved morphological integrity is consistent with its substantially higher flexural stress (30.28 MPa) relative to S-1 (6.26 MPa). A more

continuous interface with fewer pull-out cavities can facilitate stress transfer between the dispersed hemp phase and polymer matrix, thereby improving resistance to bending [39]. Nevertheless, the tensile performance of S-2 remained relatively low, indicating that fracture morphology alone does not govern the overall mechanical response. S-3 (HP/PLA/MAH) exhibited a comparatively rough fracture morphology with dispersed domains/particles. This formulation also showed the highest tensile stress among the acid-treated group (11.23 MPa), indicating that the observed heterogeneous fracture surface did not necessarily correspond to poor mechanical integrity. Rather, the rougher fracture path may indicate greater resistance to crack propagation and energy dissipation during tensile failure. However, the small domains within the PLA matrix are attributed to phase separation; this interpretation should be treated cautiously because SEM morphology alone cannot conclusively establish phase separation. S-4 (HP/PLA/PBAT/MAH/ESO) also exhibited a heterogeneous and relatively rough surface with visible cavities. Despite these defects, S-4 retained a high tensile stress (10.69 MPa) and exhibited the highest tensile strain among the acid-treated formulations (4.20%). The combination of PLA/PBAT and ESO may contribute to the more deformable response observed mechanically [40]; however, the independent contributions of PBAT and ESO cannot be distinguished because these variables were changed simultaneously. Overall, the SEM observations indicate that interfacial defects, including void formation, debonding, and pull-out, varied considerably among the acid-treated formulations and broadly corresponded to differences in their mechanical response.

(a ₁)	(b ₁)	(c ₁)	(d ₁)
(a ₂)	(b ₂)	(c ₂)	(d ₂)
(e ₁)	(f ₁)	(g ₁)	
(e ₂)	(f ₂)	(g ₂)	

Figure 3. SEM images of the fracture surfaces after tensile testing. The acid-treated composite: (a₁) S-1 HP/PP/MAH, (b₁) S-2 HP/TPS/MAH, (c₁) S-3 HP/PLA/MAH, and (d₁) S-4 HP/PLA/PBAT/MAH/ESO. The untreated hemp composites: (e₁) S-1 WHP/PLA/MAH/ESO, (f₁) S-2 WHP/PP/MAH, and (g₂) S-3 WHP/TPS/MAH/urea. (a₂–g₂) Their corresponding higher-magnification images.

The untreated hemp composites also displayed formulation-dependent fracture characteristics **Fig. 3e–g**. S-1 (WHP/PLA/MAH/epoxidized soybean oil) exhibited a comparatively smoother surface with fewer visible cavities. The observation associates this morphology with plastic deformation of the thermoplastic matrix and the presence of ESO. However, despite the relatively compact morphology, S-1 exhibited low tensile and flexural stresses (0.266 and 3.57 MPa, respectively), demonstrating that a smoother fracture surface should not necessarily be interpreted as stronger interfacial adhesion or

superior mechanical performance. In contrast, S-2 (WHP/PP/MAH) displayed greater surface roughness and a higher density of cavities than S-1 and S-3. These morphological defects are indicative of heterogeneous fracture and possible interfacial debonding. Untreated lignocellulosic fillers retain cellulose, hemicellulose, lignin, and pectin and consequently contain abundant polar hydroxyl functionalities [41]. Their compatibility with comparatively hydrophobic polymer matrices can therefore be limited, potentially promoting interfacial defects and filler pull-out. This interpretation is consistent with the weak tensile behavior reported for the untreated (WHP/PP/MAH) formulation. Notably, S-3 (WHP/TPS/MAH/urea) exhibited a comparatively compact fracture morphology with lower apparent roughness and fewer pronounced defects. This formulation simultaneously showed the highest tensile stress (3.59 MPa) and flexural stress (32.66 MPa) among the untreated composites. The correspondence between the more continuous morphology and improved mechanical performance suggests more effective stress transfer within this formulation [42]. The chemical similarity and potential interactions between the hydrophilic hemp constituents and TPS may favor compatibility compared with more hydrophobic matrices, while urea may additionally influence TPS processing and intermolecular interactions [43]. Nevertheless, the specific contribution of urea cannot be isolated from the present formulation design.

Taken together, SEM analysis demonstrates a strong relationship between composite formulation, fracture morphology, and mechanical response. Surfaces containing extensive cavities and pull-out features generally indicate interfacial debonding and inefficient stress transfer, whereas more continuous morphologies with fewer interfacial defects are associated with improved mechanical integrity. However, the SEM results also demonstrate that surface roughness alone cannot be used as a direct measure of interfacial adhesion; matrix ductility, fracture mechanism, filler dispersion, and additive composition must also be considered. Consequently, the morphological observations are most appropriately interpreted together with the tensile and flexural results rather than as independent evidence of fiber–matrix bonding.

3.4. Thermal Properties of the Composites

The acid-treated formulations exhibited composition-dependent multistage thermal degradation (Fig. 4a). For comparative analysis, the mass losses were divided into three temperature regions: 100–250, 250–400, and 400–600°C (Table 4). In the first region, S-1 (HP/PP/MAH), S-2 (HP/TPS/MAH), S-3 (HP/PLA/MAH), and S-4 (HP/PLA/PBAT/MAH/ESO) showed mass losses of 7.91, 11.69, 14.33, and 9.23%, respectively. This low-temperature mass loss can be associated with the removal of absorbed moisture and volatile species and, depending on formulation, the initial decomposition of thermally labile constituents. Therefore, attributing the entire 100–250°C mass loss exclusively to moisture, as in the original text, would likely overestimate the moisture contribution. The major decomposition occurred predominantly between 250 and 400°C, with mass losses of 36.7, 49.7, 82.8, and 72.5% for S-1–S-4, respectively.

For hemp-containing systems (Fig. 4c), this region encompasses degradation of thermally labile lignocellulosic constituents, particularly hemicellulose and cellulose, together with decomposition of the

corresponding polymer matrices. Importantly, the large mass losses of S-3 and S-4 cannot be assigned solely to hemicellulose because these formulations contain PLA or PLA/PBAT, whose matrix degradation contributes substantially within this temperature range. Thus, the observed profiles represent overlapping degradation processes of the polymeric and lignocellulosic components rather than independent degradation of hemp constituents. Above 400°C, the mass losses decreased to 13.60, 6.32, and 7.93% for S-2, S-3, and S-4, respectively, whereas S-1 retained a substantially larger mass loss of 43.28%. The broad high-temperature decomposition of lignocellulosic material can partly arise from the progressive degradation of lignin and carbonaceous residues [44]. However, the pronounced high-temperature mass loss of S-1 is primarily consistent with the PP-rich formulation, as PP undergoes major thermal decomposition at considerably higher temperatures than the PLA-containing systems.

The DTG-derived thermal parameters further demonstrate the strong influence of matrix composition (Fig. 4b, d). S-1 exhibited the highest thermal stability, with $T_{\min} = 348.4 \pm 1.3^{\circ}\text{C}$ and $T_{\max} = 470.5 \pm 1.5^{\circ}\text{C}$. The high $T_{\max}$ is consistent with the characteristic high-temperature decomposition of the PP-rich matrix [45]. S-2 showed slightly lower $T_{\min}$ ($346.2 \pm 2.2^{\circ}\text{C}$) and $T_{\max}$ ($395.9 \pm 9.8^{\circ}\text{C}$), indicating that incorporation of TPS shifted the principal degradation process toward lower temperature. This behavior can be attributed to the comparatively lower thermal resistance of the polysaccharide-based TPS phase and the presence of thermally labile constituents within the multicomponent system. A substantially different degradation response was observed for S-3 and S-4. S-3 exhibited $T_{\min} = 171.35 \pm 5^{\circ}\text{C}$ and $T_{\max} = 352.7 \pm 75^{\circ}\text{C}$, whereas S-4 showed $T_{\min} = 184.05 \pm 0.5^{\circ}\text{C}$ and $T_{\max} = 346.44 \pm 4.4^{\circ}\text{C}$. Their lower $T_{\max}$ values relative to S-1 and S-2 indicate that the PLA-containing systems undergo their principal mass-loss event at lower temperatures. For S-4, the presence of PBAT, hemp powder, and ESO further broadens the chemical complexity of the system and can generate overlapping degradation events. Consequently, differences in $T_{\min}$ and $T_{\max}$ among the formulations should primarily be interpreted in terms of overall composition and overlapping degradation mechanisms, rather than being attributed solely to hemp loading or acid treatment. Overall, the thermal-stability sequence based on $T_{\max}$ was S-1 (470.5°C) > S-2 (395.9°C) > S-3 (352.7°C) > S-4 (346.44°C). These results demonstrate that matrix chemistry is a dominant determinant of the thermal response of the acid-treated composites, while hemp powder and computerizing additives modulate the onset, breadth, and distribution of the degradation processes.

Table 4

Thermal degradation parameters and mass loss of composite formulations prepared with (a) acid-treated and (b) untreated hemp powder

(a)	Samples	T _{min} (°C)	T _{max} (°C)	Weight loss, 100– 250°C (%)	Weight loss, 250– 400°C (%)	Weight loss, 400– 600°C (%)
	S-1 (HP/PP/MAH)	348.4 ± 1.3	470.5 ± 1.5	7.906	36.7	43.28
	S-2 (HP/TPS/MAH)	346.2 ± 2.2	395.9 ± 9.8	11.69	49.7	13.60
	S-3 (HP/PLA/MAH)	171.35 ± 5.0	352.7 ± 75	14.33	82.8	6.32
	S-4 (HP/PLA/PBAT/MAH/ESO)	184.05 ± 0.5	346.44 ± 4.4	9.23	72.5	7.93
(b)	S-1 (WHP/PLA/MAH/ESO)	161 ± 8.18	322.43 ± 4.8	9.005	74.89	5.202
	S-2 (WHP/PP/MAH)	339.8 ± 3.8	465.7 ± 7.8	8.545	25.635	55.558
	S-3 (WHP/TPS/MAH/Urea)	298.36 ± 3.6	332.35 ± 3.5	10.7304	51.50	10.3425

The untreated hemp-powder composites also exhibited formulation-dependent multistage thermal degradation (Fig. 4c-d and Table 4). Between 100 and 250°C, S-1 (WHP/PLA/MAH/ESO), S-2 (WHP/PP/MAH), and S-3 (WHP/TPS/MAH/urea) exhibited mass losses of 9.01, 8.55, and 10.73%, respectively. These initial losses can be associated with desorption of moisture and volatile components, together with the onset of degradation of low-thermal-stability constituents. The slightly greater loss of S-3 may be related to the hydrophilic nature of the TPS-containing formulation and the presence of additional thermally labile components. The principal differences among the formulations became evident at higher temperatures. Between 250 and 400°C, S-1 exhibited the largest mass loss (74.89%), followed by S-3 (51.50%) and S-2 (25.64%). The dominant mass loss of S-1 in this region is consistent with decomposition of its PLA-rich matrix together with degradation of the lignocellulosic fraction. S-3 similarly underwent substantial degradation within this interval because the TPS and hemp components contain polysaccharide-rich structures that decompose predominantly within the intermediate-temperature region. In contrast, only 25.64% of S-2 was lost between 250 and 400°C because a substantial fraction of its PP-rich matrix remained stable until higher temperatures. This distinction was particularly evident between 400 and 600°C. S-2 exhibited a mass loss of 55.56%, markedly higher than S-1 (5.20%) and S-3 (10.34%). The shift of the dominant degradation of S-2 toward this higher-temperature region is consistent with the greater intrinsic thermal stability of PP relative to PLA- and TPS-containing matrices. Residual lignin-derived structures may also contribute to broad high-temperature degradation because lignin decomposes over a wider temperature interval than cellulose

and hemicellulose [46]. However, the magnitude of the high-temperature mass loss in S-2 indicates that PP decomposition is the predominant contribution rather than lignin degradation alone. The $T_{\min}/T_{\max}$ values further support these differences. S-1 showed $T_{\min} = 161 \pm 8.18^{\circ}\text{C}$ and $T_{\max} = 322.43 \pm 4.8^{\circ}\text{C}$, while S-3 exhibited $T_{\min} = 298.36 \pm 3.6^{\circ}\text{C}$ and $T_{\max} = 332.35 \pm 3.5^{\circ}\text{C}$. By contrast, S-2 displayed markedly higher $T_{\min}$ and $T_{\max}$ values of 339.8 ± 3.8 and $465.7 \pm 7.8^{\circ}\text{C}$, respectively, confirming its superior resistance to thermal degradation. Accordingly, the thermal-stability sequence based on $T_{\max}$ was S-2 (465.7°C) > S-3 (332.35°C) > S-1 (322.43°C).

Comparison of the treated and untreated series further shows that the thermal response cannot be attributed to acid treatment alone because the formulations are not compositionally identical across all samples. For example, the matrices, compatibilizers, and additives vary among PP, TPS, PLA, and PLA/PBAT-based systems. The data therefore show most clearly that polymer-matrix chemistry governs the principal degradation temperature, while hemp treatment and additives influence lower-temperature degradation processes and the overall shape of the TGA/DTG profiles. In particular, PP-based composites displayed the highest $T_{\max}$ values in both series, whereas PLA- and TPS-containing formulations underwent their major degradation at lower temperatures.

3.6. Chemical Structure of Hemp-Fiber Composites

The chemical structures and potential interactions within the acid-treated and untreated hemp composites were investigated by FTIR spectroscopy (Fig. 4e-f). The spectra exhibited characteristic absorption bands associated with the lignocellulosic hemp phase, polymer matrices, and MAH-containing formulations, while changes in band position and intensity provided evidence of compositional and interfacial differences among the composites.

For the acid-treated composites (Fig. 4e), a broad absorption band at approximately $3200\text{--}3400\text{ cm}^{-1}$ was particularly evident in the TPS-containing formulation and is assigned primarily to O–H stretching of hydroxyl-rich starch and cellulose. The similarity between the spectral features of TPS and cellulose arises from their polysaccharide structures and abundance of hydroxyl functionalities [47]. Changes in the intensity and shape of this band may reflect alterations in the hydrogen-bonding environment during melt processing and interaction between the hemp-derived cellulose and TPS matrix. Carbonyl-related bands provided further information on the MAH-containing systems. Absorption in the approximately $1785\text{--}1790\text{ cm}^{-1}$ region was assigned to carbonyl vibrations associated with anhydride functionalities. For S-3 (HP/PLA/MAH), bands at approximately 1862 and 1784 cm^{-1} were attributed to anhydride-related carbonyl vibrations, while the band at 1462 cm^{-1} was assigned to C–H bending [48]. These features confirm the presence of MAH-derived functionalities in the formulation; however, FTIR alone does not establish the extent of grafting or covalent coupling. For S-4 (HP/PLA/PBAT/MAH/ESO), prominent bands in the $1033\text{--}1128\text{ cm}^{-1}$ region were associated with C–O stretching, while the band near 1457 cm^{-1} was assigned to C–H deformation. The strong carbonyl absorption around 1733 cm^{-1} is characteristic of ester-containing polymer components, including PLA. A shoulder near 1751 cm^{-1} was

additionally observed and was attributed to ester carbonyl species potentially associated with MAH-mediated interfacial reactions. Rather than considering this feature definitive proof of MAH grafting, its appearance is more appropriately interpreted as consistent with changes in the carbonyl environment and possible esterification/interfacial interactions among MAH, the polyester matrix, and hydroxyl-containing hemp components.

Comparison with the untreated systems Fig. 4f revealed additional spectral changes associated with sulfuric-acid treatment. The untreated material exhibited a band around 1250 cm^{-1} that was substantially diminished or absent after treatment; this feature was attributed to C–O-related vibrations of lignin-associated structures. Likewise, attenuation of bands in the $1600\text{--}1650\text{ cm}^{-1}$ and $1740\text{--}1750\text{ cm}^{-1}$ regions after treatment was associated with changes in non-cellulosic constituents, including hemicellulose-, lignin-, pectin-, and wax-associated functionalities. These spectral changes suggest partial removal or chemical modification of non-cellulosic surface components during H_2SO_4 treatment rather than complete removal of lignin or hemicellulose. Differences were also reported around 1100 and 2850 cm^{-1} following acid treatment. The former lies within the C–O/C–O–C vibration region of polysaccharide structures, whereas the latter is associated with aliphatic C–H stretching. Their attenuation therefore indicates modification of the chemical environment of the lignocellulosic surface during acid treatment. In the untreated composites, the carbonyl band around 1744 cm^{-1} remained comparatively prominent and was attributed to ester-containing structures and potential interactions involving hydroxyl-containing hemp constituents and maleic functionalities.

Overall, the FTIR results demonstrate that sulfuric-acid treatment altered the surface chemistry of the hemp powder, particularly the spectral contributions associated with non-cellulosic constituents. The persistence of characteristic polymer bands in the composites confirms incorporation of the respective PP, TPS, PLA, and PLA/PBAT matrices, while changes in the O–H and carbonyl regions indicate modified intermolecular environments in the MAH-containing systems. These chemical changes, together with the SEM observations, are consistent with formulation-dependent differences in fiber–matrix interactions and consequently contribute to the distinct mechanical and thermal responses observed among the composites.

4. Conclusion

This study establishes a melt-processing strategy for valorizing residual hemp fibers as micron-scale reinforcement in thermoplastic composites. Physicochemical treatment decreased the hemp-powder particle size from 63.14 to $19.67\text{ }\mu\text{m}$, providing a finer reinforcement and altering fiber–matrix interactions. Among the acid-treated formulations, HP/PLA/MAH exhibited the highest tensile performance, reaching a maximum load and stress of 258.20 N and 11.23 MPa , respectively, whereas HP/TPS/MAH showed a flexural stress of 30.28 MPa . The corresponding untreated WHP/TPS/MAH/urea formulation reached 32.66 MPa in flexural stress, demonstrating that performance depended strongly on matrix chemistry and compatibilization rather than treatment alone. Thermal

behavior was similarly formulation-dependent; HP/PP/MAH displayed the highest T_{max} (470.5°C), comparable to 465.7°C for untreated WHP/PP/MAH. Together with FTIR and SEM observations, these results highlight the coupled roles of particle treatment, polymer selection, and interfacial modification in controlling composite performance and demonstrate a viable pathway for converting hemp-processing residues into value-added sustainable materials.

Declarations

Disclosure and Declaration of AI Use.

Generative AI tools were used in the writing process. All content was subsequently reviewed and edited by the authors, who take full responsibility for the final version.

Author Contribution

sarker Md shamim : conceptualization, methodology, original draft writing –review and editing. Mayakrishnan Gopiraman: , review and editing. Yonghe Huan: visualization and software. Xuedi Zhang: investigation and supporting . Osama R. M. Metawea: visualization and software ,review and editing. Dejian Ma: visualization and software. Shangyong Zhang: conceptualization, funding acquisition, writing –review and editing. Ick Soo Kim: review and editing-Sakil Mahmud: conceptualization, methodology, original draft writing –review and editing, funding acquisition.

Acknowledgement

This research was funded by Enterprise, Wuhan Hemp Biotechnology Co., Ltd., China, Project Name: Green Preparation and Functional Product Development of High-quality Hemp Fiber, Project No.: 202210181. Dr. Mahmud was involved in this grant.

Data Availability.

All data are included in this article. Any additional information will be provided by the authors upon reasonable request.

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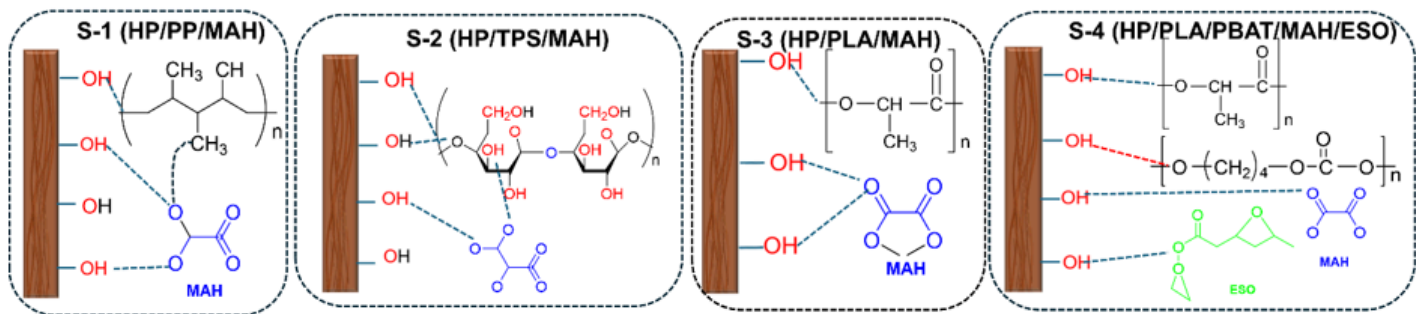
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Figures

(a)



(b)

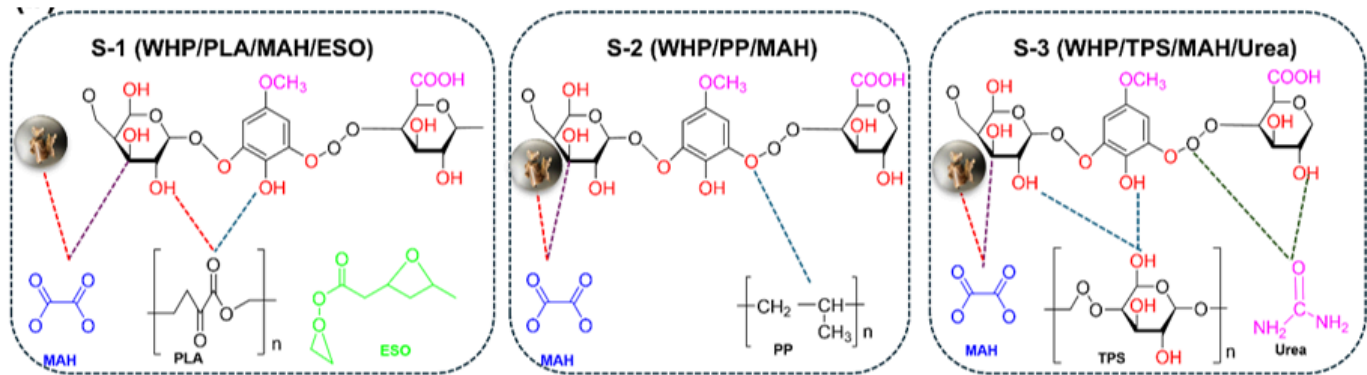


Figure 1

Schematic illustration of the synthesis route from leftover hemp fiber to micron-sized hemp powder for reinforcement in composite materials: (a) with acid-treated and (b) without acid-treated composites.

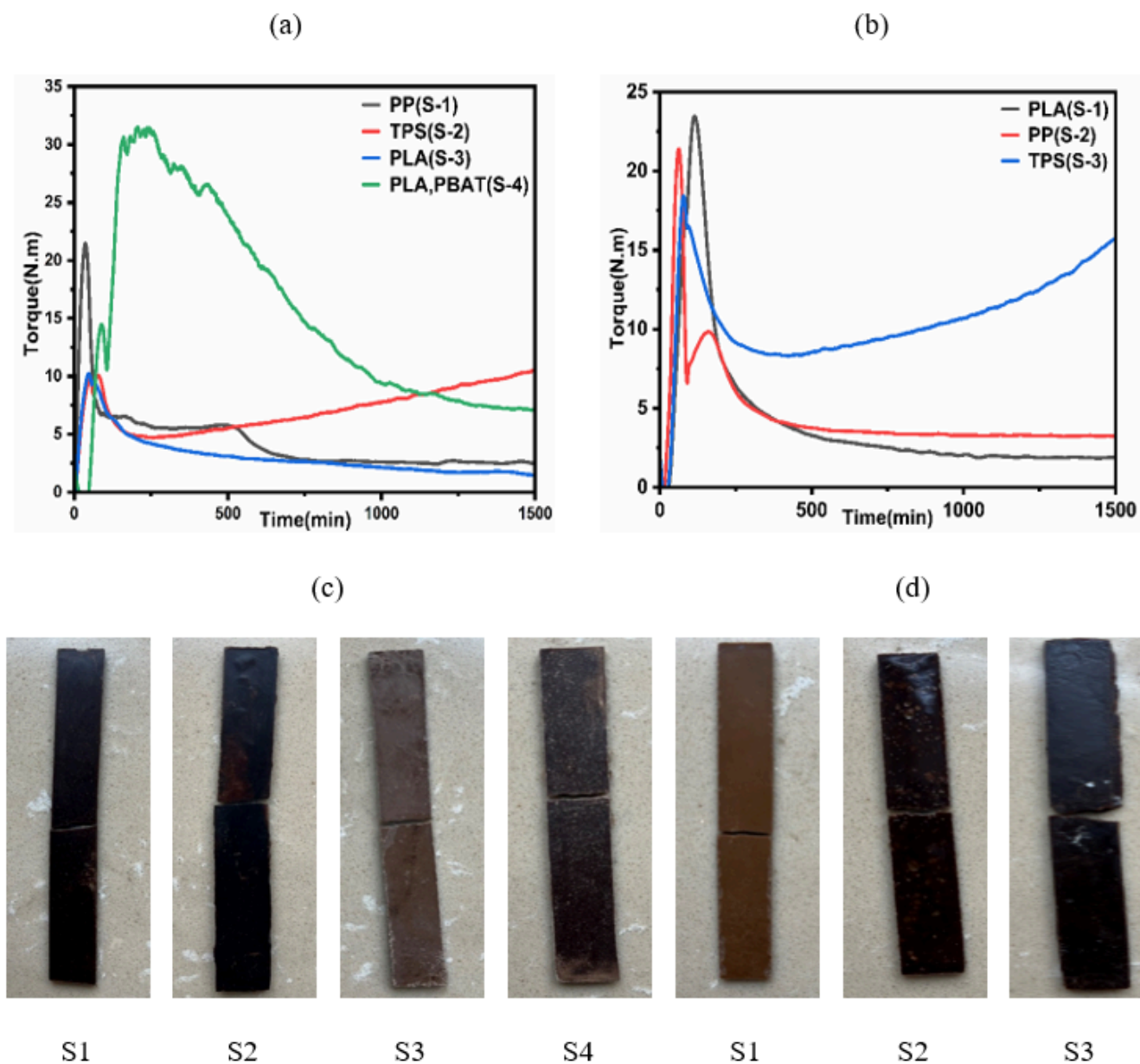


Figure 2

Torque–time profiles of composites prepared with (a) acid-treated and (b) untreated hemp powder under the specified melt-processing conditions. Images of the fracture sample after tensile test: (c) acid-treated and (d) untreated hemp-reinforced composites.

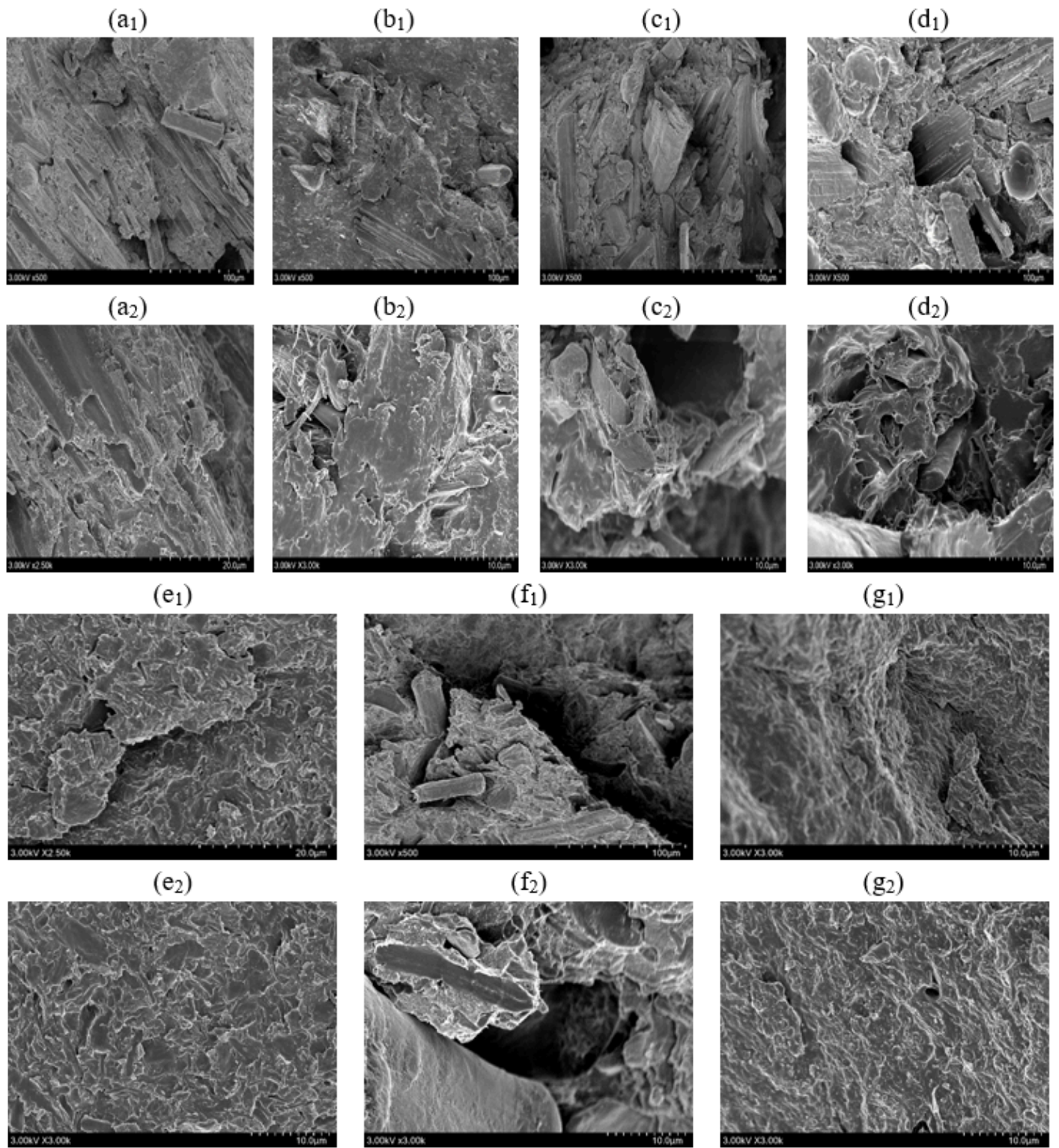


Figure 3

SEM images of the fracture surfaces after tensile testing. The acid-treated composite: (a₁) S-1 HP/PP/MAH, (b₁) S-2 HP/TPS/MAH, (c₁) S-3 HP/PLA/MAH, and (d₁) S-4 HP/PLA/PBAT/MAH/ESO. The untreated hemp composites: (e₁) S-1 WHP/PLA/MAH/ESO, (f₁) S-2 WHP/PP/MAH, and (g₂) S-3 WHP/TPS/MAH/urea. (a₂–g₂) Their corresponding higher-magnification images.

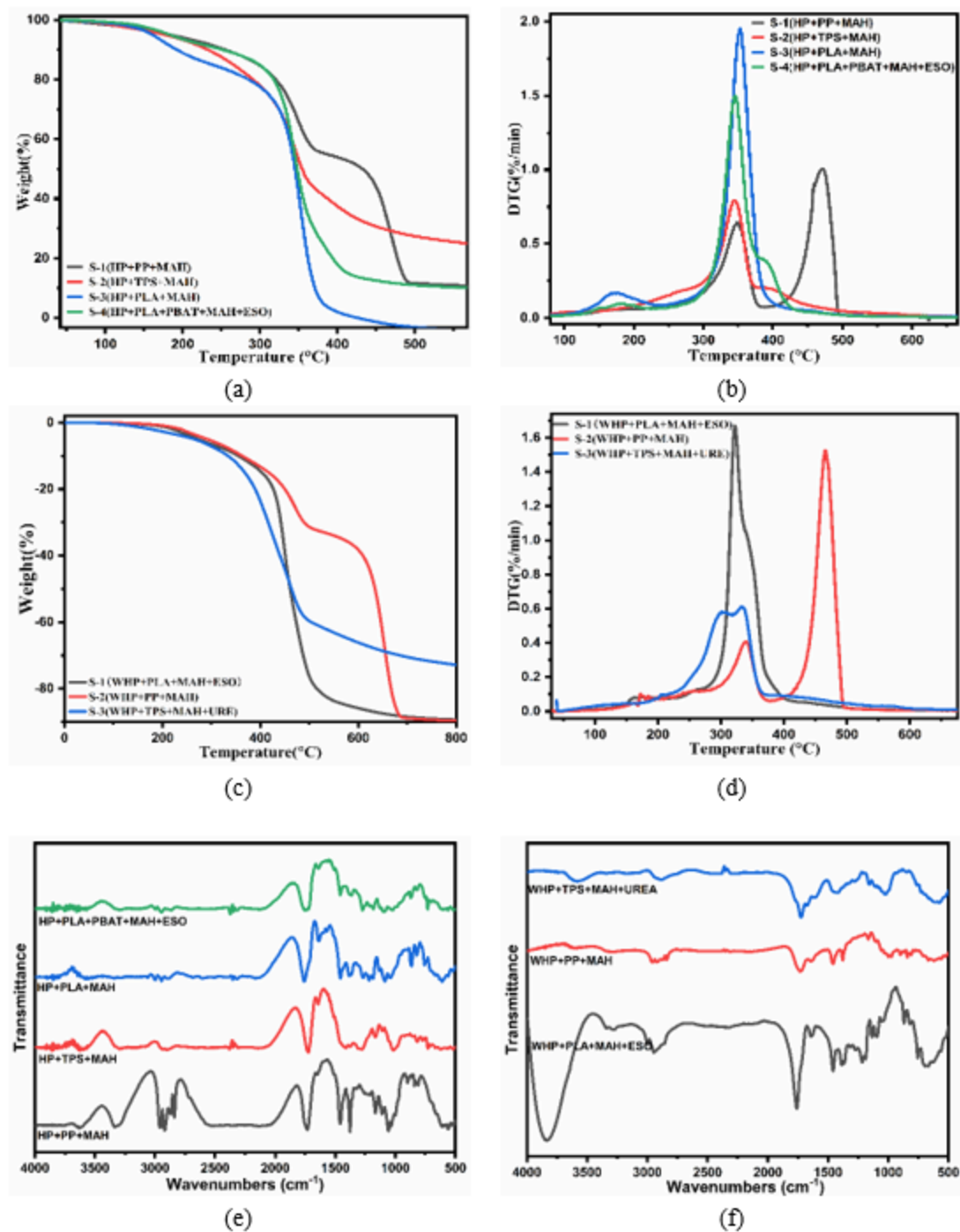


Figure 4

The (a, c) TGA, (b, d) DTG curves, and (e, f) FTIR spectra of (a, b, e) acid-treated and (c, d, f) untreated hemp composite.

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