Influence of biochar and flame retardant on mechanical, thermal, and flammability properties of wheat gluten composites

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ARTICLE INFO

ABSTRACT

The use of environmentally friendly materials such as bio-sourced plastics is being driven by increased awareness of environmental issues caused by synthetic plastics. However, bio-sourced plastics have poor fire behaviour that limits their application. The addition of a flame retardant to these plastics is one effective way to increase the fire resistance property; however, the flame retardant should not interfere with the mechanical performance of the plastic. Most flame retardants act as stress concentration points, reducing tensile strength. Hence, to create a balance between tensile strength and fire resistance, biochar (to conserve strength) and lanosol (to improve fire resistance) were added to wheat gluten bioplastic in various ratios and the optimal ratio was identified. Wheat gluten composites were fabricated using compression moulding at four different concentrations of lanosol (2, 4, 6, and 8 wt.%) and biochar (2, 4, 6, and 8 wt.%). From the test results, the composite with 4 wt.% lanosol and 6 wt.% biochar exhibited a good balance between the mechanical and fire properties; it conserved the strength and improved the fire properties (39 % reduction in peak heat release rate).

1. Introduction

Plastics have a wide range of applications, ranging from industries to household products. Society has become overly reliant on the use of plastic due to its appealing properties such as low cost, availability, and ease of use, which has serious environmental consequences. Plastic litter and microplastics have accumulated in the global environment, causing serious pollution that affects groundwater and harms habitats. The growing environmental concern necessitates the use of materials that are both sustainable and environmentally friendly. The use of bio-sourced plastics instead of fossil-based polymeric materials could be an effective way to accomplish this. Bio-based plastics can be chemically synthesised from biological materials or biosynthesised by living organisms. It should be noted that bio-based plastics are derived from natural sources, making them renewable.

Wheat gluten (WG) is one such material that is a plant-based protein and a co-product of bioethanol and cereal processing industries, gaining much attention recently due to its utilisation as a bio-based plastic [1,2]. WG bio-based plastics are biodegradable, abundant, and relatively cheap hence, they could serve as a substitute for petroleum-based plastics [3–5]. It is necessary to improve the mechanical strength of WG plastics in order to increase their use. To increase the strength of WG, various researchers have used reinforcements. For instance, Wretfors et al [6] studied the effect of hemp fibre reinforcement in the WG matrix. The addition of 20 % hemp fibre increased the tensile strength and Young's modulus by 81 % and 858 %, respectively. Microfibers such

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as silica [7], hydroxyethyl cellulose [8], and methylcellulose [9], as well as other natural fibres such as wood, etc. and biochar [10–13] have also been used as reinforcements in WG plastics resulting in improvement in mechanical properties. Although the available literature suggested the use of fibre/particle reinforcement to increase mechanical strength, such reinforcements have only a minor effect on fire resistance, [14–19] and research into the fire properties of filler-added WG composites is rare. Flame retardants (FR) are generally used to increase the fire resistance of plastics, but the addition of FR to plastics shows a negative effect on the mechanical strength by developing stress concentration points on the resin matrix. Furthermore, most of the FR used are synthetic. This necessitates the development of a new plastic material system that is entirely biodegradable and has effective mechanical and fire resistance properties. In the present research, the synergistic effect of bio-based reinforcement in the form of biochar and bio-based FR in the form of lanosol was investigated on WG bio-based plastic.

Biochar is a co-product of the controlled pyrolysis of biomass [18,20, 21]. It can possess a high porosity, which gives it a low density and a high surface area. Furthermore, biochar is also capable of producing excellent bonding with polymer matrix through mechanical interlocking. The porous structure of biochar allows matrix infiltration resulting in effective bonding with the resin. The effects of biochar on polymer matrix and bio-based materials have been explicitly presented by researchers in the field [4,20,22,23]. It has been reported that biochar acts as an effective reinforcement in polymers such as polypropylene, PLA, epoxy, etc. [24–26]. In addition, the inclusion of 4-6 wt. % biochar in biodegradable materials like WG showed enhancements in mechanical strength [4,27]. In the case of WG, the addition of biochar made from gluten led to an increase in the indenter-modulus, enhancement of thermal stability, and a decrease in the water absorption of the composite [4].

Lanosol is a naturally occurring substance extracted from red algae. From the research of Das et al [29], the addition of 4 wt.% to WG produced a significant improvement in the fire and thermal properties. The samples achieved a V-0 flammability rating in the Underwriter's Laboratory test and produced a high char residue i.e. 31 % increment compared to neat WG, in the thermogravimetric analysis test. In cone calorimeter tests, WG with 4 wt.% lanosol samples showed a 9 % increase in time to ignition and a 50 % reduction in peak heat release rate (PHRR) when compared to WG without lanosol.

Although lanosol has been shown to impart beneficial fire safety in WG, its inclusion is detrimental to mechanical properties, in particular, the tensile strength. Therefore, to create a balance between the mechanical and fire properties, the synergistic effect of biochar and FR (lanosol) should be explored. In this study, biochar and lanosol were added to WG, and the optimum concentrations for conserving the mechanical properties and enhancing the fire behaviour were investigated. A very important aspect of biocomposite manufacturing is determining the optimal concentrations of fillers or FRs that produce a balance in the fire and mechanical properties. From previous research, ca. 4-6 wt.% of biochar [4,27] and 4 wt.% of lanosol [28] in WG had the most desirable flame retardancy and strength, however, the effect of an increase or decrease in the loading amounts of these individual constituents on the bio-based plastics has not been reported in literature. Therefore, in this research, WG plastics were fabricated with varying proportions of lanosol and biochar to determine the best combination for the composite. The mechanical, thermal, and fire tests of the composites were conducted, and the results were compared to ascertain the effect of the loading amounts of fillers and FRs in composites.

2. Materials and methods

2.1. Materials

The main materials used in the manufacturing of the composites were wheat gluten, biochar, and lanosol. The wheat gluten powder was obtained from Lantmännen Reppe AB in Sweden. It had a 78 % protein content (using a 5.75 nitrogen conversion factor), 1.2 % lipids, 6 % starch, 7 % moisture, 0.9 % inorganic ash, and 7 % of additional carbohydrates. The WG powder was plasticised with glycerol purchased from PWG Produkter AB, Sweden. The biochar was produced by pyrolysing wood (90 % Spruce and 10 % other softwood) for one hour at 800 °C in an inert nitrogen gas environment using the Auger reactor. It had a specific surface area of 322 m^2/g and a particle size of 100 – 200 microns. The flame retardant, lanosol, was purchased from AApin Chemicals Limited, United Kingdom.

2.2. Bioplastic manufacturing

The WG biocomposites were fabricated using compression moulding method. The WG powder was plasticised by mixing with glycerol and kept under low humidity conditions to minimise water absorption as recommended in the previous research by Das et al [5]. The plasticised WG, biochar, and lanosol were mixed in a vortex mixer, distributed in moulds of size, $100 \times 100 \times 2$ mm^3, and placed in the press. The pressing was performed using a pressure of 5 Pa for 20 min at a temperature of 150 °C. The residence time and temperature for the composite manufacturing were obtained from literature [30,31]. The samples were then cooled inside the press without the release of pressure before they were removed. Table 1 lists the compositions of lanosol and biochar in the samples that were manufactured and their labels in the manuscript.

2.3. Characterisation methods

The samples were tested in four different experiments: fire/thermal tests including cone calorimeter, microscale combustion calorimetry (MCC), thermogravimetric analysis (TGA), and tensile testing. Scanning Electron Microscopy was used to examine the sample morphology. A brief description of the methods and the instruments used is presented in the next sections.

2.3.1. Thermogravimetric analysis tests

The TGA instrument was used to measure the mass loss of the wheat gluten composites as a function of temperature. A Mettler Toledo TGA/ DSC 1 STARe equipment was used for the test. The experiments were performed under nitrogen atmosphere using 5 - 10 mg samples, heated at 10 °C/min from room temperature to 900 °C. The flow rate of the nitrogen gas was 50 ml/min.

2.3.2. Microscale combustion calorimeter

Heat release parameters of the WG composites were measured using an MCC-3 equipment from Fire Testing Technology (FTT) at Nanjing University of Science and Technology, China. Approximately 3 mg of each sample was prepared and tested. The method A procedure, according to ASTM D7309-21, was adopted; samples were pyrolysed under inert conditions at about 75 – 600 °C and nitrogen gascarried the effluent to the combustor, operated at 900 °C [32,16]. The samples were tested at four different heating rates: 0.5, 1, 2, and 3 °C/s.

2.3.3. Cone calorimeter

The cone calorimeter experiment was conducted using a cone

Table 1
The different compositions of WG biocomposites.

Composition	Label
80 wt.% WG + 20 wt.% glycerol	WG
70 wt.% WG + 20 wt.% glycerol + 8 wt.% lanosol + 2 wt.% biochar	8L2B
70 wt.% WG + 20 wt.% glycerol + 6 wt.% lanosol + 4 wt.% biochar	6L4B
70 wt.% WG + 20 wt.% glycerol + 4 wt.% lanosol + 6 wt.% biochar	4L6B
70 wt.% WG + 20 wt.% glycerol + 2 wt.% lanosol + 8 wt.% biochar	2L8B

calorimeter from FTT Limited, East Grinstead, UK. Three replicates from each sample were tested under irradiation levels of 50 kW/m^2 according to the standards in ISO 5660.

2.3.4. Tensile test and scanning electron microscope (SEM)

The tensile properties of the samples were measured according to ASTM D638 protocol. The instrument used was the Intron 5567 Universal Testing Machine having a 30 kN load cell and a crosshead speed of 5 mm/min. Five samples each of the different blends were subjected to tensile force to analyse the tensile properties. Microscopic images of the fractured samples were taken with a Hitachi TM 1000 (Japan). The accelerating voltage of the equipment was 10 kV with a working distance of 6 mm.

2.3.5. FTIR analysis

A Fourier Transform Infrared Spectroscopy equipment (FT-IR) was used to analyse the chemical properties of the composites. A Perkin Elmer Spectrum 100 instrument was used for the analysis. The wavelength range investigated was from 600 to 4000 cm^{-1}.

3. Results and discussion

3.1. Thermal properties of neat WG and WG composites

The results from the TGA tests in Fig. 1(a) show that all the samples have fairly equal onset decomposition temperatures, ca. 200 °C, and mass loss. However, considering the mass loss within the temperature range, neat WG lost the highest mass (82 % of the initial mass) during the experiment, compared to the samples containing lanosol and biochar. Biochar is a porous carbon skeleton with high thermal stability. Hence, combining biochar with a flame retardant (lanosol) makes it more thermally stable. This explains the difference in thermal stability between the neat WG and the composites as confirmed by Das et al [29]. Furthermore, this was also evident in the char residue formation shown in the subsequent sections. The 6 % lanosol + 4 % biochar added biocomposite had the highest char residue of 28 %. However, there were no significant differences between the residues of the remaining samples, 8L2B, 4L6B, and 2L8B, which had 26 %, 25 %, and 25 % char, respectively. Fig. 1(b) shows the decomposition rate of the samples, where the ones with low lanosol content (4L6B, 2L8B, WG) had fast decomposition. It can be observed that neat WG exhibited the highest decomposition rate in comparison with the composites. Collectively, the results show that the addition of lanosol and biochar increased the char residue of gluten over the entire temperature range. In our previous research, it was found that the residual mass after 750 °C was ca. 34 % higher for lanosol/WG composites compared to neat WG and for the biochar/WG composite, the same was 11 % [27].

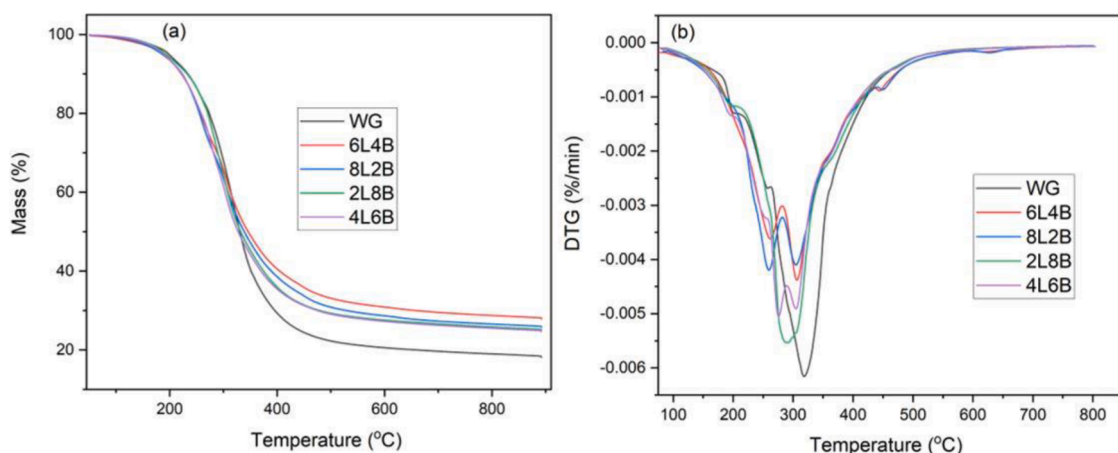### 3.2. Combustion characteristics from MCC tests

The averages of the measurements from MCC, PHRR, heat release capacity (HRC), total heat released (THR), temperature (pTemp), and time to PHRR (pTime) obtained at different heating rates (0.5, 1, 2, and 3 K/s) are shown in Table 2. The heat release rate curves at 1 K/s for each of the samples are shown in Fig. 2.

According to Fig. 2 and Table 2, the 2L8B sample had the lowest PHRR and HRC (a decrease of 33 % and 34 %, respectively compared to neat WG) while 8L2B had the lowest THR (32 % decrement from WG value). Overall, lanosol and biochar significantly enhanced the fire resistance of WG. It can be clearly seen from the MCC results that the loading amounts of 2 % lanosol and 8 % biochar had the best influence on the fire resistance of WG. In an actual sense, high percentages of lanosol should exhibit the best fire resistance but in this case, the sample with the lowest concentration of lanosol outperformed the other compositions. To make a tangible analysis of the fire properties of the composites, results from the cone calorimeter will be required, which is provided subsequently.

3.3. Cone calorimeter results

Fig. 3 shows plots of heat release rate as a function of time for all the samples fabricated. For each composition, the curve with the PHRR closest to the mean value from the triplicate experiments is shown. Table 3 shows the averages of the values for PHRR, TTI, fire performance index (FPI), and THR for three replicates.

Contrary to the MCC results, the sample with the highest lanosol content had the best fire resistance, i.e. it had the lowest PHRR (a decrement of 43 % compared to neat WG), highest FPI value (0.05 m^{2}s/ kW), and the longest time to ignition, 20 s (an increment of 13 % compared to neat WG plastic). This is acceptable, because increasing the quantity of flame retardant in a material to a certain weight percent can increase the fire resistance. The dispersion of lanosol illustrated in the SEM images explained in Section 3.5 further affirms the high fire resistance of the 8L2B sample. The disparities in the cone and MCC test

Table 2
Fire properties of neat WG and WG blends.

Sample	PHRR [W/g]	HRC [J/gK]	THR [kJ/g]	pTemp [°C]	pTime [s]
WG	206 ± 132	128 ± 17	17 ± 10	357 ± 27	259 ± 168
8L2B	162 ± 127	91 ± 19	11 ± 4	326 ± 18	238 ± 158
6L4B	165± 122	97± 12	13 ± 4	327 ± 16	236 ± 158
4L6B	188 ± 84	146 ± 84	16 ± 8	346.± 26	272 ± 222
2L8B	138 ± 113	84 ± 19	12 ± 4	332 ± 23	238 ± 155

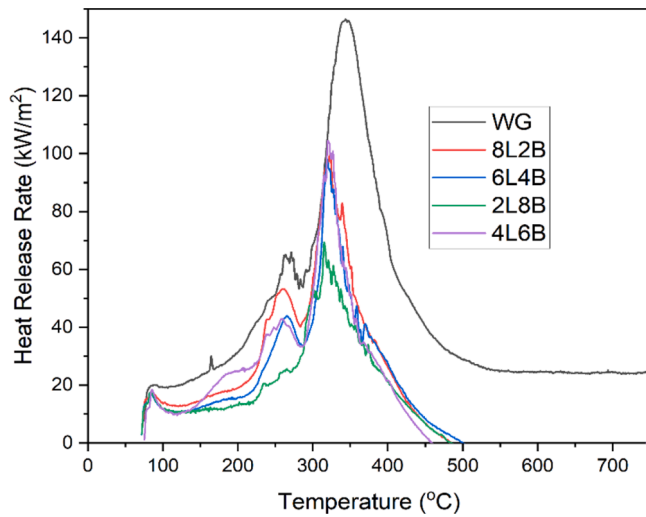**Fig. 1.** TGA curves of the samples (a) Mass versus temperature (b) DTG curves.

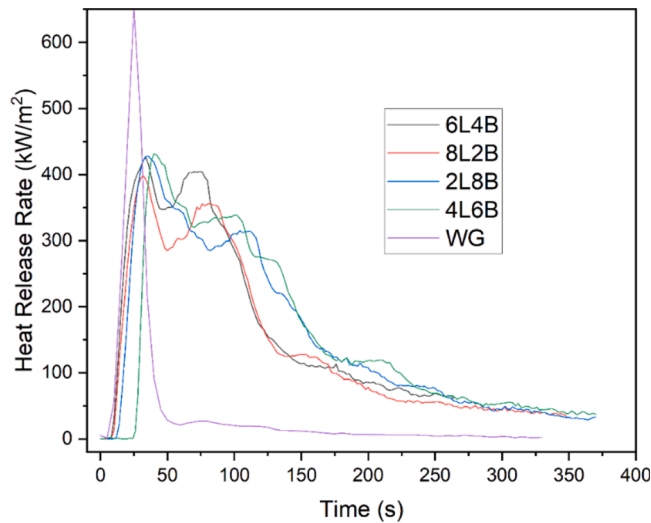**Fig. 2.** Heat release rate versus temperature curves of neat WG and composites.

Fig. 3. Heat release rate versus time curves of neat WG and composites from the cone calorimeter test at 50 KW/m².

Table 3
Reaction-to-fire properties at incident radiation of 50 kW/m².

Samples	PHRR [kW/m²]	TTI [s]	FPI [m²s/kW]	THR [MJ/m²]
WG	709 ± 80	17 ± 8	0.02 ± 0.01	44 ± 44
8L2B	402 ± 7	20 ± 8	0.05 ± 0.02	52 ± 3
6L4B	446 ± 28	11 ± 0.6	0.03 ± 0.0004	56 ± 2
4L6B	433 ± 36	19 ± 9	0.04 ± 0.02	54 ± 2
2L8B	419 ± 21	15 ± 3	0.04 ± 0.01	55 ± 5

*Peak heat release rate (PHRR), time to ignition (TTI), fire performance index (FPI), total heat release (THR)

could be attributed to the sample size required for the experiments, the oxygen concentration during combustion in both cases, and the influence of material heterogeneity on MCC measurements especially with flame retardant plastics. These inconsistencies have been explained in the research of Xu et al [33] and also affirm the assertions made by Mensah et al [32] that fire tests are not standalone and must be combined for a reliable flammability assessment.

Additionally, both the MCC and the cone calorimeter test results of the 6L2B and 8L2B show curves having two distinct peaks signifying the

formation of compact char after ignition and a drastic reduction in PHRR. The second peaks in 2L8B and 4L6B curves are not well defined compared to the samples with higher concentrations of lanosol, which could be attributed to the formation of a thin char that cracked after a short exposure to heat flux.

Between 6L4B and 4L6B, the flammability results were not dependent on the lanosol content. The 4L6B sample had a longer ignition time ca. 19 s, 72 % higher than that of 6L4B and higher FPI (0.04 m²s/kW). The PHRR of 6L4B increased by ca. 3 % compared to that of the 4L6B. The THR value of 6L4B was ca. 4 % higher than 4L6B. Compared to the PHRRs of just biochar and WG (694 kW/m²) and just lanosol and WG (645 kW/m²) composites, the values reported in the current investigation for the composites, were, on average, ca. 37 % lower (comparison made by averaging the values of 694 and 645 kW/m²) [27]. However, the PHRR of just lanosol and WG composite without any glycerol plasticiser was 17 % lower than the ones with glycerol from this current investigation [28], which highlights that the plasticiser is detrimental for fire performance. It was also observed that the THR of the composites was higher than WG showing that the composites burnt for a longer period.

During the experiment, all the composites expanded before charring. The char served as an insulating layer preventing the transfer of heat and O_2 to the un-pyrolysed material. The burning samples went through a char cracking phase, which allowed oxygen to diffuse into the un-burnt material. The heat flux and the volatile gases sustained the fire until the entire sample was consumed. Visually, in Fig. 4, the chars from the samples with a larger proportion of biochar look approximately the same. Overall, the char from the 8L2B sample was more compact with fewer pores compared to the other samples.

3.4. Prediction of cone calorimeter test results with MCC parameters

Predicting the materials' flammability parameters has been a long-term goal in fire research. Mensah et al [16,34] have used machine learning methods to estimate MCC results. Here, an attempt was made to predict the peak heat release rate from flaming combustion (cone calorimetry) with the results from a non-flaming test (MCC) using a simple empirical equation (see Eq. (1)) derived by Das et al [35].

$$PHRR = \left(\frac{\chi \cdot \eta_c \cdot PTemp}{h_g \cdot n} \right) q''_{net} \tag{1}$$

where *PHRR* is the peak heat release rate from MCC, *PTemp* is the temperature at peak heat release, q''_{net} is the net radiative heat flux on the sample, and h_g is the heat of gasification of the samples. For this study h_g of wheat gluten was assumed to be 1 kJ/kg, and q''_{net} was 50 kW/m². *n* is an arbitrary integer, which varies from 3 to 12 depending on the polymer type. χ indicates the combustion efficiency, for a well-ventilated flaming combustion is within 0.5–0.9. The combustion efficiency of 0.9 was used in the calculation. η_c is the heat release capacity, which can be calculated by dividing the PHRR measured from MCC by the heating rate.

For the prediction of wheat gluten composites, the n value used was 3. The PHRR measured at four different heating rates, 0.5, 1, 2, and 3 K/s were used for the estimation and the average of the corresponding values was recorded in Table 4.

From the results, it can be observed that the difference between experimental and predicted PHRR ranges from 25 kW/m² to 50 kW/m², which is quite acceptable except for 4L6B, which had a deviation due to the inconsistencies in the sample tested at 0.5 K/s.

3.5. Tensile and microscopic properties

The stress versus strain curves of the samples is portrayed in Fig. 5. Table 5 shows the mechanical properties of the samples, i.e. the tensile strength, and the modulus of elasticity.
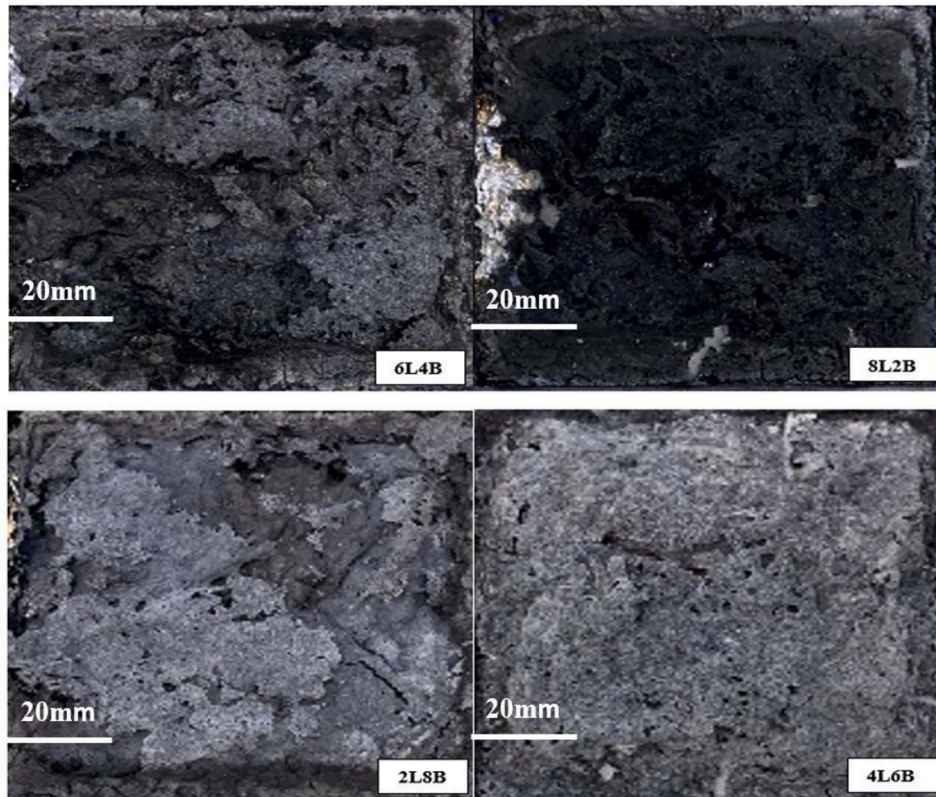
Fig. 4. Pictures of sample chars from the cone calorimeter tests.

Table 4
Comparison of experimental and predicted PHRR.

Sample	Experimental PHRR kW/m^2	Predicted PHRR kW/m^2
WG	652.9	682.3
8L2B	398.2	449.2
6L4B	426.1	476.6
4L6B	431.2	739.6
2L8B	427.8	456.7

Table 5
The maximum tensile strength and modulus of elasticity of the samples.

Sample	Maximum tensile strength [MPa]	Modulus of elasticity [MPa]
WG	7 ± 0.7	209 ± 33
6L4B	6 ± 1	175 ± 37
8L2B	5 ± 1	132 ± 51
2L8B	8 ± 0.8	174 ± 45
4L6B	5 ± 1	150 ± 59

From the tensile test results in Table 5 and Fig. 5, 2L8B is the combination with the highest tensile strength (8 MPa), which is linked to the high amount of biochar in the sample. This can be explained by the fact that due to the high amount of biochar present, more pores were available that created a mechanical interlocking with gluten during processing [36]. When compared to just lanosol and WG composite, the tensile strength of the composites from the current investigation containing both biochar and lanosol were higher, although not by much. However, when comparing with just biochar and WG composites, the tensile strengths of the composites in these experiments were lower. Hence, it was seen that the addition of both lanosol and biochar endows tensile strength values that are between the values of the two aforementioned composites (i.e., just lanosol/WG and biochar/WG) [27]. The pores in the biochar can be seen in Fig. 6(c) and the mechanical interlocking developed can be seen in Fig. 6(d). In contrast to the high biochar-added composite (2L8B), the high lanosol-added composite (8L2B) had a low tensile strength. 8L2B has the lowest tensile strength, 5.17 MPa. At high lanosol addition, the lanosol blocks the pores in the biochar, reducing mechanical interlocking. Further high concentrations resulted in lanosol agglomeration in the matrix region, creating stress concentration points in the interface region. During load, cracks initiate in this region, leading to sudden failure. This analysis is supported by the
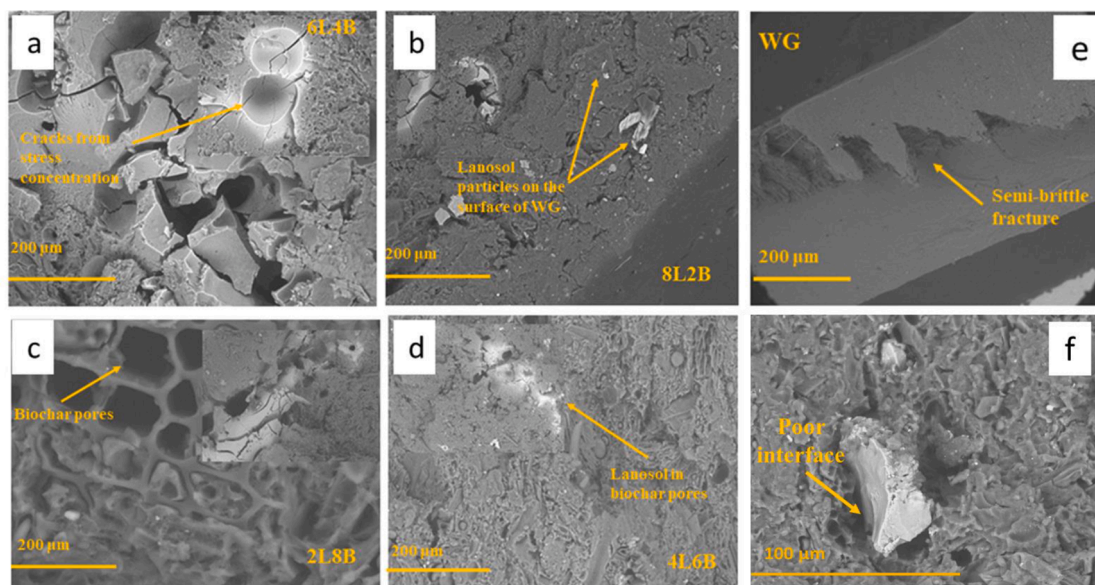

Fig. 5. Stress-strain curve of the biochar and lanosol added samples.

Fig. 6. Micrographs of tensile fractured surfaces.

SEM images (Fig. 6a and 6b), which show cracks have formed at locations with lanosol particles. The tensile strengths of the samples are low compared to other polymeric composites, but this reduction happened due to the application of plasticiser (glycerol), which was necessary to make a rigid plastic like gluten processable. With this tensile strength, these materials can be used in packaging applications and the results were also comparable to packaging bioplastic developed by Marichelvam et al [38].

In Fig. 5, it can be seen that the stress-strain curves for 8L2B and 4L6B follow a fairly similar shape, with a high ductility showing an increment of 37 % and 28 %, respectively compared to the WG before a fracture occurs. This can be attributed to the manufacturing of the composites. When neat gluten was processed in the hot press under the actual heat and pressure, the crosslinking/polymerisation increased, which resulted in the gluten polymer becoming quite rigid with a high modulus of elasticity. It is possible that when manufacturing 8L2B and 4L6B, not all materials could crosslink, and this is due to the heat shielding effect from biochar. This could explain the reason for the higher ductility and lower tensile strength in the WG samples with lanosol and biochar compared to the values for neat gluten. The semi brittle failure in the WG can also be understood through the Figure 6(e). The development of poor interface between lanosol particulates and WG can be seen in Figure 6(f). When particles are added to polymers, the molecular chain mobility reduces making it stiff. However, if a void forms around a particle due to poor interfacial bonding, the modulus might get reduced. This aforementioned phenomenon is attributed to the lower modulus of WG composites as observed in the current investigation.

3.6. FT-IR analysis

Fig. 7 depicts the FT-IR spectra of the composites and the neat WG. There were no significant differences in WG composites with different biochar and lanosol contents. The peaks at ca. 1587-1750 cm^{-1} and ca. 1490-1585 cm^{-1} correspond to the vibrational stretching of amide I (C=O) and the vibration frequency of amide II (N-H), respectively. The presence of amide III is confirmed by the medium intensity peaks at ca. 1430-1490 cm^{-1}. Peaks at ca. 3070-3600 cm^{-1} correspond to the broad hydroxyl (O-H) band. The variation in the peak intensity for the β-sheet region in the wavenumber range ca. 1590-1730 cm^{-1} was observed for the composites. This was due to the presence of biochar particles. The peak at 1038 cm^{-1} indicates the presence of glycerol in the WG and

composites. Overall, it is clear that the addition of biochar and lanosol in varying proportions resulted in no significant changes in the chemical structure of WG composites when compared to neat WG.(Fig. 7)

4. Conclusions

In this study, the synergistic effect of lanosol and biochar on the mechanical and fire properties of wheat gluten was investigated. The optimal concentration of lanosol and biochar required to produce a balance between mechanical and fire resistance was identified. Four concentrations of biochar/ lanosol/ WG composites were prepared; 2L8B, 8L2B, 4L6B, and 6L4B, and the results were compared to plasticised WG.

- The best fire resistance was displayed by the 8L2B sample, which had the highest lanosol content, among the tested samples.
- According to the mechanical test results, almost all the composite samples had comparable tensile strengths to the neat WG with
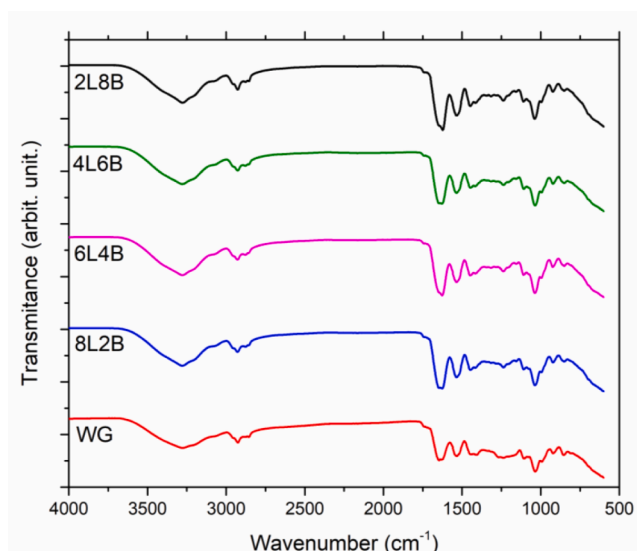

Fig. 7. FT-IR spectra of WG and WG composites.

samples 6L4B and 2L8B having the closest values to the control sample.

Overall, with a PHRR of 433 kW/m^2, TTI of 19 s, tensile strength of 5 MPa, and an elastic modulus of 150 MPa, 4L6B presented a balance between the mechanical and fire-resistant properties of WG. It conserved the mechanical properties of WG and significantly improved the fire resistance. Therefore, ca. 4 wt.% lanosol and ca. 6 wt.% biochar are proposed as the optimum concentrations for the production of WG/biochar/lanosol composites.

Declaration of Competing 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.

Data availability

Data will be made available on request.

Acknowledgements

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