

# Microscale combustion calorimetry assessment of green composites made with chicken feather-modified soy protein resins and jute fabric

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## ARTICLE INFO

### Keywords:

Soy protein-based resins  
Chicken feather  
Composites  
Thermal stability  
Agricultural waste

## ABSTRACT

Biodegradable, and sustainably produced, 'green' plastics are actively being researched to replace conventional, environmentally harmful petroleum-based plastics. However, before these green plastics get adopted, they must match the properties of their conventional counterparts. In many applications, fire-safety can be a key parameter where naturally derived green materials could potentially outcompete petroleum-based plastics. In the present research, green resins and composites were fabricated using soy protein isolate (SPI), waste chicken feather fibers (CFF), jute fabric (JF), and glutaraldehyde (GA), and evaluated for their critical fire-safety parameters through Microscale Cone Calorimetry (MCC) characterization. The loading of CFF from 0 to 30 wt% increased the specific peak heat release rate (pHRR) from 101 to 120 W/g for CFF/SPI resins without GA and from 94.5 to 114 W/g for GA crosslinked CFF/SPI resins. GA was thus shown to improve fire-safety for CFF/SPI resins. However, for JF/(CFF/SPI) composites, CFF did not show a proportional relationship with fire-safety. Rather, at 20 wt% CFF, the pHRR was minimized to 81.1 W/g for JF/(CFF/SPI) composites without GA and to 86.0 W/g for GA-crosslinked JF/(CFF/SPI) composites. This demonstrated that the addition of JF improved fire-safety despite its known combustibility, and even removed the need of the toxic crosslinker GA. Results also indicated that all variations of the fabricated CFF/SPI resins and JF/(CFF/SPI) composites had lower specific pHRR than typical petroleum-based plastics, clearly demonstrating the benefits of switching to SPI based green resins and composites. These green composites would be suitable for many applications including housing and transportation where fire-safety can be critical.

## 1. Introduction

### 1.1. Agricultural products and waste as an engineering resource

Petroleum-based plastics, used in many day-to-day applications, have been extremely difficult to replace in spite of their negative environmental impact [1]. Their low processing costs coupled with desirable mechanical and chemical properties have established their dominance in a diverse range of industries and applications, from the automotive industry to consumer electronics. Research into replacing petroleum-based plastics with naturally occurring 'green' materials that can be processed and recycled or composted in an environmentally favorable way has uncovered many novel sources with practical utility [2–5]. One such example is the sector of agricultural byproducts and wastes. Soy protein isolate, jute fabric, and chicken feather fibers are

three materials, among many, within this sector that can be used to develop composites suitable for applications in engineering and consumer products [6].

The poultry industry produces hundreds of millions of metric tons of chicken waste every year [7]. This waste is commonly landfilled or incinerated; both options having undesired negative environmental impacts [8]. However, engineering value can be found in this waste through the extraction of chicken feathers from the agglomerate mass containing blood, feces, and other components of the chicken waste. Chicken feathers are a hierarchical material composed of a main quill (chicken feather quill/CFQ) to which primary branches, called barbs (chicken feather barbs/CFB), are attached. CFQ and CFB share some similarities. For example, both are semi-crystalline, and hollow. However, CFQ is comprised almost entirely of  $\beta$ -sheet protein while CFB is primarily conformed in  $\alpha$ -helix [9]. This results in differences in their

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<https://doi.org/10.1016/j.jcomc.2023.100394>

mechanical, chemical, thermal, and other material properties [10,11]. The term ‘chicken feather fibers’ (CFF) has been used vaguely in literature, sometimes referring exclusively to either the CFQ [12] or CFB [13, 14], and other times referring to both [8]. This work used ‘CFF’ to refer to both parts, CFQ and CFB, and was procured as a mixture of both.

Soy protein isolate (SPI) is an agriculturally derived green material that has been extensively studied for its ability to crosslink and produce fully biodegradable green resins. It has shown great potential in replacing petroleum-based plastics [15–22]. Soy protein (SP) is not a singular protein but is composed of a number of various globulins [23]. These various proteins can form strong covalent disulfide crosslinks as well as alanine, lysinoalanine, lanthionine, and amide crosslinks, all of which allow for the synthesis of a cohesive SPI resin [18]. SPI is produced by refining the protein from soybeans to approximately 91% purity [24].

Both CFF and SPI are proteins made up of amino acids. They are chemically similar as evidenced by the Fourier Transform Infrared Spectroscopy (FTIR) data that show the two proteins have similar composition of amino acids, as well as similar bonding modes [17,25, 26]. The present work confirms that adding the two together can be accomplished easily without pretreating CFF to form CFF/SPI resins, because of their similar amino acid composition. Thus, there could be significant environmental benefits through incorporating CFF into SPI and consequently reducing the net amount of CF waste going into landfills or being incinerated. Furthermore, extensive research into investigating biomacromolecules as fire retardants has shown proteins and nucleic acids like DNA to be beneficial additives for fire-safety [27–30].

Bast fibers such as jute, obtained from the phloem of the jute plant, have been commonly spun into yarns and woven into fabric (JF) for centuries. Jute fabrics offer good mechanical properties, low-cost, full biodegradability, and wide availability [2,31].

### 1.2. ‘Green’ resins and composites

A composite is a multiphase material consisting of a matrix or resin (continuous) phase that binds all parts together, a dispersed (discontinuous) phase made of different, usually reinforcing, materials and the interface, the two-dimensional surface between the dispersed and continuous phases [32]. Between the continuous and dispersed phases lies a region known as the interphase in which the properties of one phase transition into the other, and its size and chemical interactions are important considerations. A composite’s utility lies in its ability to optimize the differing properties of the matrix and dispersed phase/s [32]. Thus, an optimized interphase is critical as otherwise the phases would act separately, limiting the benefits of incorporating multiple phases.

The present work refers to ‘green’ resins and composites made wholly from naturally occurring materials, containing no petroleum products. CFF/SPI resins and JF/(CFF/SPI) composites produced in this study are fully green, while those crosslinked with GA may be considered partially green.

The motivation of this study is to upcycle CFF into green resins and composites and evaluate how MCC can characterize key fire-safety parameters to compare how these green resins and composites stack up to conventional petroleum-based plastics.

### 1.3. Flammability parameters

Flammability is a measure of how easily a material can undergo a chemical reaction that generates heat, i.e., combusts or burns. Naturally, limiting a product’s flammability is of great importance to ensure fire-safety.

Of all the flammability parameters, the heat release rate (HRR) and heat release capacity (HRC) are considered as the most crucial parameters to assess the fire-safety presented by a material [33,34]. As the

material combusts, heat is generated (released). This heat, in turn, promotes the burning of the remaining material at a faster rate. Thus, a high HRR aids in spreading fire by more readily aiding in the release of additional heat energy in a positive-feedback loop. Specific HRR refers to the rate at which combustion heat is released per unit initial mass of specimen during controlled thermal decomposition. Specific peak HRR (pHRR) is defined as the maximum specific HRR measured during testing. Thus, pHRR reveals the material’s peak potential to contribute to fire hazards. The time taken for a material to reach flaming combustion when subjected to a heat source is known as time to ignition.

HRC is defined as  $HRC = \frac{pHRR}{\text{heating rate}} \text{ J/g-K}$ , essentially normalizing the pHRR measurement to the heating rate, as was performed by Perroud et al. [35]. HRC can be thought of as the potential heat release per unit temperature change of the surface of a material undergoing pyrolysis. Since the heating rate is maintained at a constant 1 °C/s during the microscale combustion calorimetry (MCC) HRC and pHRR share the same value and, hence, trivial to include both. TpHRR is the temperature at which the pHRR occurs and is not indicative of a material’s flammability by itself.

Specific total heat release (THR) is the integral of the specific HRR vs temperature plot, and thus gives the total heat released through combustion of a specimen per unit initial mass. Char yield is the mass left after the fire tests and acts as a supplemental parameter for understanding THR. During pyrolysis, most crosslinked (thermoset) polymer specimens develop a char layer on the outer surface, which generally acts as heat insulation and decreases the mass flux of the specimen and ambient oxygen.

‘High-quality’ char offers better insulation that decreases or prevents further combustion than a lower quality char. A high-quality char is devoid of cracks, pores, or other similar defects that could facilitate smoldering by providing a constant supply of oxygen, albeit in low amounts, to the interior of the virgin specimen. Smoldering is a type of combustion that can be facilitated by poor char quality propagating pyrolysis deeper within the specimen by focusing the heat flow through radiation or convection through cracks or pores [36,37]. Additives can help to improve the char insulation quality. However, there is always an optimal ratio for additives, and incorporating an additive beyond this optimal ratio can result in a decrease in char quality and consequently a decrease in fire-safety. Additives, beyond their optimal levels, can even result in delamination of the char layer, which can result in the exposure of the non-pyrolyzed region, further decreasing the fire-safety aspect. Improving char quality is not the only route flame retardant additives can increase fire-safety. Other notable ways include improving thermal properties, altering pyrolysis pathways, diluting volatile gases, inhibiting chain reactions, and promoting high-quality char formation [38].

### 1.4. Fire-safety parameters of petroleum-based plastics found in literature

MCC is a well-established technique for assessing key fire-safety parameters such as pHRR, HRC, and THR [35,39–42]. There are two main methods: controlled thermal decomposition (Method A) in which a specimen undergoes controlled heating in an inert environment and controlled thermal oxidative decomposition (Method B) in which a specimen undergoes controlled heat in an oxidizing environment [41]. In both methods, the decomposed gases are swept from the specimen chamber into furnace then combusted, from which the pHRR, HRC, and THR parameters can be calculated using oxygen consumption theory [42]. As it only requires a few milligrams of a sample for testing, MCC has great utility for high throughput testing.

Table 1 presents mean HRC values of common petroleum-based plastics found from two earlier studies [43,44]. HRC is recorded in place of pHRR as both are equivalently important in assessing fire-safety of materials. HRC normalizes heating rate differences between studies and, is thus, considered a better metric for comparison. The mean values in Table 1 are written with the notation “x/y”, where “x” is the mean

**Table 1**  
HRC mean values found in literature.

Component	HRC (J/g·K)
Acrylonitrile butadiene styrene (ABS)	669/581
High density polyethylene (HDPE)	-/1490
Low density polyethylene (LDPE)	1680/-
Polyethylene terephthalate (PET)	332/357
Polymethyl methacrylate (PMMA)	514/475
Polypropylene (PP)	1570/1130
Polystyrene (PS)	927/859
Polyvinyl chloride (PVC)	138/129

value reported in the first study [43] and “y” is the mean value found in the second study [44].

While not completely comprehensive, these chosen plastics are widely considered to be the most frequently used petroleum-based plastics that end up as environmental waste [45]. Their values will be used as a reference to gauge the fire-safety benefits of the chicken feather-SPI based green plastics and composites produced in the current study.

## 2. Materials and methods

### 2.1. Materials

Chopped chicken feather fibers (CFF) were acquired from Prof. Justin Barone's Laboratory at Virginia Polytechnic Institute and State University (Virginia Tech). Soy protein isolate (SPI) powder (PRO FAM® 974) was obtained from Archer Daniels Midland Company (St. Louis, MO). High purity D-Sorbitol was procured from VWR Life Science (Radnor, Pennsylvania), and glutaraldehyde (GA) solution (Grade II, 25% in H<sub>2</sub>O) and sodium hydroxide (NaOH) pellets were purchased from Sigma-Aldrich (St. Louis, MO). Teflon® molds for resin casting were produced using 0.508 mm natural virgin polytetrafluoroethylene (PTFE) from ePlastics.com (San Diego, CA). Jute fabric (JF) was purchased from JOANN Fabrics (Ithaca, NY).

### 2.2. Preparation of CFF/SPI resins for MCC characterization

Pure SPI and CFF/SPI resins were prepared using the following procedure and denoted by their weight percentages, i.e., X/Y resin contains X% of CFF by weight and Y% of SPI by weight. The percentage of CFF was varied from 0% (i.e., pure SPI) to 30% by increments of 10%. The same process was repeated with the application of glutaraldehyde (GA) to create externally crosslinked (thermoset) resins. If a resin used GA, the term “wGA” was added after the X/Y ratio. For example, a 30% CFF GA crosslinked resin is denoted as 30/70 wGA. GA crosslinked resins are hereafter referred to as GA resins, while resins synthesized without the application of GA are referred to as GA-free resins. As a result of these combinations, eight (four GA-free and four with GA) compositions of CFF/SPI resins were prepared as listed in Table 2. Each composition was prepared at three different times for testing purposes and confirming the reproducibility of the results.

To prepare the resins, a water bath was placed on top of a stirring hot

**Table 2**  
CFF/SPI resin composition details.

CFF/SPI composition	CFF wt (%) / (g)	SPI wt (%) / (g)	GA mass (g)
0/100	0 / 0	100 / 15	0
10/90	10 / 1.5	90 / 13.5	0
20/80	20 / 3	80 / 12	0
30/70	30 / 4.5	70 / 10.5	0
0/100 wGA	0 / 0	100 / 15	1.5
10/90 wGA	10 / 1.5	90 / 13.5	1.5
20/80 wGA	20 / 3	80 / 12	1.5
30/70 wGA	30 / 4.5	70 / 10.5	1.5

plate set at 80 °C. While this was warming up, in a 600 mL beaker, 15 g of total protein (CFF + SPI) were weighed out in the desired proportions. For example, for the 30/70 resin, 4.5 g of CFF and 10.5 g of SPI were weighed. To this, 165 g of deionized (DI) water (1:11, SPI + CFF:DI water by wt.) was added and hand mixed using a spatula. 4.6 mL of 4 M NaOH was added to this mixture drop by drop, while stirring, until a pH of 11 was reached. After homogenizing the solution for 15 min, the beaker was transferred to a hot plate and a magnetic stirrer was placed in the beaker and stirred at 280 RPM at 80 °C for an additional 15 min. During this second 15 min period, 1.5 g of D-Sorbitol (10:1 SPI + CFF:D-sorbitol by wt.%), to be used as plasticizer, was measured out. If the resin was intended to be crosslinked, 1.5 g of GA (10:1 SPI + CFF:GA by wt.%) was weighed out during this time and added along with D-sorbitol at the end of the 2<sup>nd</sup> 15 min period. For the resins not crosslinked with GA, only the D-sorbitol was added.

The amine-aldehyde Maillard reaction was allowed to proceed for 30 min, after which the resin in the beaker was transferred into a 12.7 cm × 12.7 cm Teflon® mold [46]. The mold with the resin was placed into a Thermo Fisher Scientific Precision (Waltham, MA) convection oven set at 49 °C until the resin weighed roughly 21 g. Resin weight was measured to roughly estimate the amount of moisture left in the sample post drying in the oven, with the understanding that 15 g of protein, 1.5 g of plasticizer, and – if used – 1.5 g of GA would amount to 16.5 g, or 18 g when GA was added. Resin weight of 21 g was chosen as the benchmark as any further drying warped the sample such that hot pressing caused fracture. On the other hand, any less drying with resin weight greater than 21 g caused the sample to warp post hot pressing as it cooled. This was true regardless of whether GA was added or not. Achieving a weight of 21 g took approximately 22 ½ h at 49 °C.

Once removed from the oven, the resin sheets were sandwiched between Teflon® sheets and left for 10 min to reach 120 °C on a Carver hydraulic hot press (Carver, 3891-4PROA00, Wabash, IN). After that they were hot pressed (or cured if GA was present) using 1.59 mm spacers between the platens at 120 °C, 9072 kg, for 13 min to obtain 1.59 mm thick resin sheets for flammability characterization. Pieces weighing 30–50 mg from each resin sheet were taken out for MCC characterization.

### 2.3. Fabrication of JF/(CFF/SPI) hybrid composites for flammability characterization

Jute fabric was cut using a shear cutter into three 12.7 cm × 12.7 cm pieces. The aggregate weight of the three mats was 10 g. Using the SPI resin preparation technique described in the previous section, 15 g of resin was prepared and transferred into the molds. The resin was then evenly applied to both sides of each jute fabric piece. After the resin application, three pieces were stacked on top of one another, placed in a 12.7 cm × 12.7 cm Teflon® mold, and allowed to dry for 22.5 h, the same time as for drying the resin sheets, for parity.

After drying, the stacked resin impregnated jute fabric pieces were hot pressed using the procedure detailed in the previous section. Using this process, eight (four GA free, four with GA) compositions of JF/(CFF/SPI) composites were fabricated as listed in Table 3. Each composite

**Table 3**  
JF/(CFF/SPI) hybrid composite composition details.

JF/(CFF/SPI) composition	JF wt (g)	CFF wt (%) / (g)	SPI wt (%) / (g)	GA wt (g)
JF/(0/100)	10	0 / 0	100 / 15	0
JF/(10/90)	10	10 / 1.5	90 / 13.5	0
JF/(20/80)	10	20 / 3	80 / 12	0
JF/(30/70)	10	30 / 4.5	70 / 10.5	0
JF/(0/100) wGA	10	0 / 0	100 / 15	1.5
JF/(10/90) wGA	10	10 / 1.5	90 / 13.5	1.5
JF/(20/80) wGA	10	20 / 3	80 / 12	1.5
JF/(30/70) wGA	10	30 / 4.5	70 / 10.5	1.5

composition was prepared at three separate times to confirm the reproducibility of the results.

All composites contained 60% resin (SPI or CFF/SPI) and 40% fabric (jute), by weight. As mentioned earlier, the CFF component of the composite was counted towards the resin and not the fiber weight fraction. The JF/(CFF/SPI) composites were broken into 30–50 mg pieces for MCC characterization.

#### 2.4. MCC characterization of CFF/SPI resins and JF/(CFF/SPI) hybrid composites

Four pieces, weighing between 30 and 50 mg, were obtained for each resin/composite composition for MCC characterization. These tests were carried out at Nanjing University of Science and Technology, China.

Following ASTM D7309, the eight resin compositions and eight composite compositions were tested to find their pHRR, temperature at peak heat release rate, THR, and char yield [41].

The specimens were placed in the sample chamber of a Deatak™ MCC-3 (St. McHenry, IL), and heated at a rate of 1 °C/s to a temperature of 703 °C. A combustor temperature of 900 °C was chosen for complete oxidation. Flow rates of 80 cc/min and 20 cc/min for N<sub>2</sub> and O<sub>2</sub> respectively were used during the pyrolysis and combustion processes. Ultimately, each composition had three sets of data to obtain average values and standard deviations.

#### 2.5. Statistical analysis

Descriptive statistics employed were means and standard deviation (SD). To compare the effects of the additives between two groups, *t*-test with unequal variance was used. No adjustments due to multiple comparisons were made because of small sample sizes. Significance was determined at the 5% level. All statistical analyses were performed in Stata 17 (StataCorp LC, College Station, TX).

### 3. Results and discussion

#### 3.1. MCC results of CFF/SPI resins

Table 4 presents the average MCC results for CFF/SPI resins with and without GA. *P*-values and box plots can be found in the Supplementary Information that allow better visualization of the trends in variation.

Data in Table 4 indicate that for GA-free CFF/SPI resins, the mean pHRR increased from 101 to 120 W/g as CFF% was increased from 0 to 30. Average TpHRR, however, stayed close to 331 °C with increase in CFF% from 0 to 30. Average THR initially decreased from 14 to 9 kJ/g as CFF% increased from 0 to 10%, and then increased to 15 kJ/g at 30% CFF.

It is noted that the differences between mean parameters were rarely statistically significant. Even from the most conservative perspective, this is a positive observation as it indicates that the CFF did not significantly decrease the fire-safety aspect, evidencing its ability to be

**Table 4**  
MCC results of CFF/SPI resins.

Sample (CFF/SPI)	pHRR <sup>1</sup> (W/g)	TpHRR <sup>2</sup> (°C)	THR <sup>3</sup> (kJ/g)
0/100	101 ± 14	331 ± 6	14 ± 8
10/90	112 ± 7	331 ± 6	9 ± 1
20/80	118 ± 19	330 ± 9	10 ± 1
30/70	120 ± 14	331 ± 3	15 ± 8
0/100 wGA	95 ± 5	336 ± 9	12 ± 1
10/90 wGA	107 ± 13	333 ± 5	11 ± 1
20/80 wGA	114 ± 7	334 ± 5	14 ± 5
30/70 wGA	114 ± 14	323 ± 5	11 ± 1

<sup>1</sup> Peak heat release rate

<sup>2</sup> Temperature at peak heat release rate

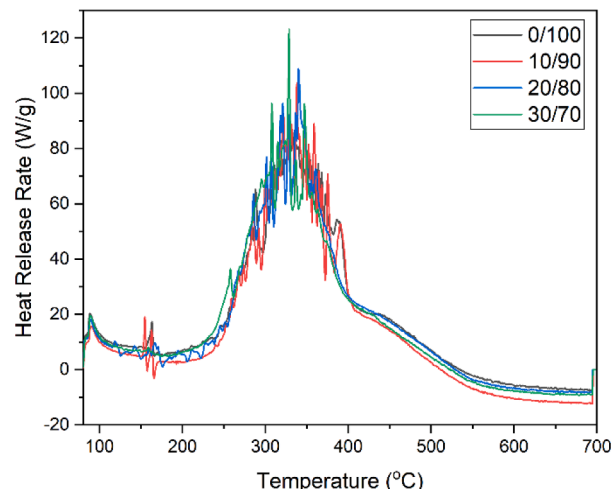
<sup>3</sup> Total heat release

recycled for engineering applications. However, using the boxplots provided in the Supplementary Information to interpret trends and variation, accepting that the small sample size affected the statistical analysis, and delving into the materials science behind the calorimetry experiment, an argument can be made that CFF does improve the fire-safety performance when added to SPI resins.

Data in Table 4 also indicate that for GA-free CFF/SPI resins, adding CFF increased the mean pHRR. However, this does not necessarily mean that CFF is not a beneficial flame-resistant additive; rather, this could simply be a constraint of the MCC technique when used on an additive with a higher heat release rate than the material it is added to. Zhuge et al. experimentally investigated the application and limitation of MCC characterization by testing the flammability of PC-siloxane copolymer, and how it changed when diphosphate was added to it [40]. Diphosphate was found to have a higher HRR than the copolymer, and the MCC characterization indicated that as diphosphate was increased from 0 to 100%, pHRR and THR increased from 159 and 10 to 400 W/g and 25 kJ/g, respectively. Yet, the cone calorimetry characterization revealed that adding diphosphate significantly reduced the THR and decreased the pHRR after adding 3.5% diphosphate by wt. [40]. The authors of the study, Zhuge et al., hypothesized that the reason for this discrepancy was because the additive (diphosphate) most likely improved the char quality of the specimen, which was difficult to observe in the MCC characterization due to the high HRR of the additive [40]. This improvement in char quality was quantified by the decrease in THR, as an improved char layer would better insulate, and thus reduce overall heat release [40].

The “false” pHRR increase described in this earlier study by Zhuge et al. parallels the results observed by adding CFF to SPI in the present work [40]. CFF reduced THR up until a certain ratio after which the pHRR and THR increased dramatically. This is likely due to better char quality developed during pyrolysis. Following the logic outlined by Zhuge et al., the increase in pHRR observed in this work when CFF is added to the SPI resins is attributed to the potentially higher HRR of CFF given that THR decreased as CFF was added. Furthermore, since the pHRR does not decrease with an increase in THR (a warning sign for a false positive correlation), CFF can be conservatively considered to be an additive with some net flammability benefit when added to SPI.

Moisture content of additives can also influence MCC parameters. Water acts as a heat sink during pyrolysis, diverting the heat energy to evaporate itself [47]. This decreases the energy available for pyrolysis and is indicated by a decrease in pHRR values. However, SPI used in this work is reported to have a moisture content of 4.8% [48]. In contrast, the various fractions that compose CFF have moisture contents that vary from 8.8 to 12.3% [8]. Thus, the moisture content and absorbance of



**Fig. 1.** Typical HRR vs temperature plots for GA-free CFF/SPI resins.



CFF is not attributed to the increase in pHRR. Fig. 1 shows the HRR vs temperature plots of GA-free CFF/SPI resins.

Fig. 2 presents the HRR vs temperature plots for CFF/SPI resins with GA. For these GA crosslinked resins, the mean pHRR increased from 95 to 114 W/g as CFF% increased from 0 to 30. Average TpHRR, however, decreased from 336 to 327 °C as CFF% increased from 0 to 30. Average THR initially decreased from 12 kJ/g to its minimum of 11 kJ/g as CFF% increased from 0 to 10, and then increased to its maximum of 14 kJ/g with 20% CFF, before decreasing once more to 11 kJ/g at 30% CFF.

As expected, CFF/SPI resins wGA show lower mean pHRR values, compared to respective GA-free CFF/SPI resins. Synergistic crosslinking has been demonstrated in some earlier studies to increase fire-safety through the formation of compact char layers that inhibit further combustion and by increasing the complex viscosity of the specimens during heating [49,50]. Thus, in the present case, GA seems to help improve fire-safety through mechanisms attributed to synergistic crosslinking. This presents itself in the reduced variation in THR values. However, the incorporation of CFF seems to decrease the advantage of GA crosslinking. As the CFF percentage increased, the difference between pHRR and THR values between resins with and without GA at the same CFF percentage diminished. The influence of CFF on the flammability of CFF/SPI resins with GA also parallels that of GA-free resins. CFF was seen to increase mean pHRR, but lower mean THR up till 10% CFF incorporation. As in the case with CFF/SPI resins without GA, 10% CFF was seen as the optimal ratio for CFF/SPI resins with GA. Inspection of the THR, which is tied to char quality, shows that CFF and GA individually improve char quality. This hypothesis is further confirmed by the MCC results for the JF/(CFF/SPI) composites, which clearly demonstrate the benefits of incorporating CFF.

### 3.2. MCC results of JF/(CFF/SPI) hybrid composites

Table 5 reports the summary of statistics for JF/(CFF/SPI) hybrid composites. P-values and box plots can be found in the Supplementary Information to better visualize the trends in variation.

For GA-free JF/(CFF/SPI) composites, mean pHRR increased from 83 to 88 W/g with the addition of 10% CFF. The pHRR minimized to 81 W/g with 20% CFF and maximized to 94 W/g with 30% CFF. Average TpHRR decreased from 336 to 329 °C as CFF increased from 0 to 30%. Mean THR increased from 9 to 11 kJ/g with the addition of 10% CFF, was minimized to 8 kJ/g with 20% CFF and maximized to 18 kJ/g with 30% CFF. Fig. 3 shows the HRR vs Temperature plots for GA-free JF/(CFF/SPI) composites.

CFF, in JF/(CFF/SPI) composites, optimized mean flammability properties (lowest mean pHRR and THR, and smallest variation) at the

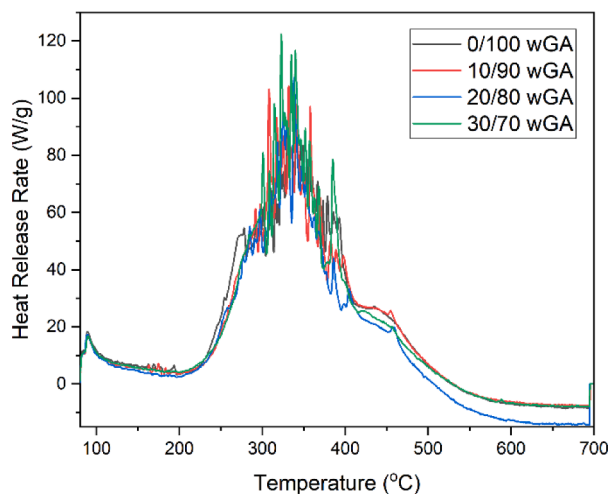


Fig. 2. Typical HRR vs temperature plots for CFF/SPI resins with GA.

Table 5

MCC results of JF/(CFF/SPI) hybrid composites.

Sample (CFF/SPI)	pHRR <sup>1</sup> (W/g)	TpHRR <sup>2</sup> (°C)	THR <sup>3</sup> (kJ/g)
JF/(0/100)	83 ± 1	336 ± 1	9 ± 1
JF/(10/90)	88 ± 5	336 ± 1	11 ± 2
JF/(20/80)	81 ± 2	331 ± 4	8 ± 1
JF/(30/70)	94 ± 10	329 ± 4	18 ± 9
JF/(0/100) wGA	93 ± 6	332 ± 6	10 ± 1
JF/(10/90) wGA	97 ± 20	336 ± 3	16 ± 10
JF/(20/80) wGA	86 ± 8	332 ± 4	8 ± 1
JF/(30/70) wGA	99 ± 2	332 ± 5	10 ± 1

<sup>1</sup> Peak heat release rate

<sup>2</sup> Temperature at peak heat release rate

<sup>3</sup> Total heat released

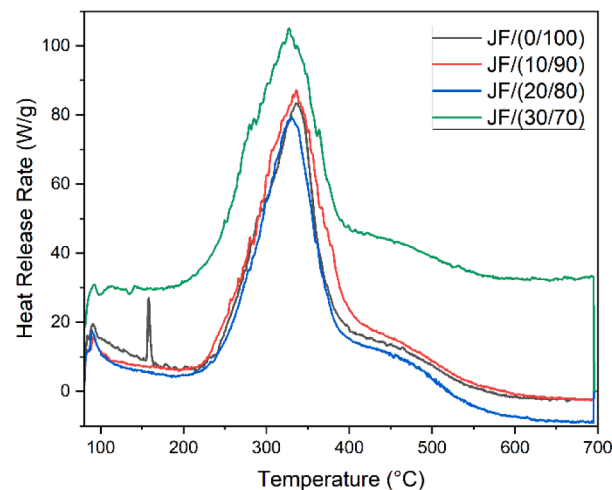


Fig. 3. Typical HRR vs temperature plots for GA-free JF/(CFF/SPI) composites.

ratio of 20/80. The mechanism by which it decreases these properties is most likely through the creation of a high-quality char layer with the help of JF. This is likely the case as above 20% CFF content, the mean pHRR and THR, and variation increase which is typical of the lower char layer quality as mentioned in the previous section.

Compared to the GA-free CFF/SPI resin specimens, the GA-free JF/(CFF/SPI) composites had significantly lower mean pHRR values across respective CFF percentages. Since the composites were prepared with identical resin synthesis, pre-curing, and hot-pressing conditions with respect to the resin specimens, the thermal properties of the JF and/or the chemical interactions between the JF, CFF, and SPI created during curing are attributed to the difference between the fire behavior of the resins and their respective composites.

The moisture content of JF is not a factor for the lower pHRR values reported in the composite specimens since JF only contains about 10% water (of total mass) in the conditioned state [51]. This is higher than the moisture content of SPI but within the range of CFF content. Yet, GA-free JF/(0/100) composites reported an average pHRR of 83 W/g and THR of 9 kJ/g which are, respectively, 18 W/g and a 5 kJ/g lower than the average pHRR (101 W/g) and THR (14 kJ/g) of GA-free 0/100 resins. Furthermore, as CFF is introduced, the composites still maintain lower pHRR values. Thus, either the lignin content of JF, which is known to increase thermal stability [52], or an interaction with CFF and SPI during curing helped increase the overall fire-safety of the composite.

For JF/(CFF/SPI) composites with GA, pHRR increased from 93 to 97 W/g with the addition of 10% CFF, whereas the same was minimized to 86 W/g with 20% CFF and maximized to 99 W/g with 30% CFF. TpHRR increased slightly from 332 to 336 °C with the addition of 10% CFF but decreased back to 332 °C as CFF content was increased to 30%. THR increased from 10 to its maximum of 16 kJ/g with the addition of

10% CFF, was minimized to 8 kJ/g for 20% CFF content but increased again to 10 kJ/g for 30% content. The effect of GA is shown by the HRR vs temperature plots for JF/(CFF/SPI) composites with GA in Fig. 4.

Comparing the HRR vs temperature plots of the JF/(CFF/SPI) composites, with and without GA, shown in Figs. 1 and 2 to those of the CFF/SPI resins, with and without GA, found in Figs. 3 and 4, it is clear that the addition of JF reduced the HRR regardless of the presence of GA. Interestingly, JF/(CFF/SPI) composites with GA had worse fire-safety properties (higher mean pHRR) than GA-free JF/(CFF/SPI) composites. This is somewhat counterintuitive, as the results in the previous section indicated that crosslinking with GA improved fire-safety properties of the CFF/SPI resins, so GA could be expected to improve the fire-safety of the composites as well. While no literature nor this study has investigated the effect of GA on the fire-safety of jute, the most probable answer is that the GA modified JF may have produced a lower quality char compared to the GA-free JF composites. This decrease in char quality compared to GA-free JF likely resulted in composites with GA to have slightly inferior fire-safety properties.

Table 6 adds the mean HRC values for the CFF/SPI resins and JF/(CFF/SPI) hybrid composites produced and tested in this work to HRC values for the synthetic polymers presented in Table 1 for easy visualization of the superior flammability properties of the green resins and composites based on soy protein and chicken feather fibers. Note that the prefix 'greener' is used for GA crosslinked resins and composites since GA itself is not an environmentally friendly chemical. A comparison of the HRC flammability parameter between groups makes it abundantly clear that the green and greener resins and composites fabricated in this study have lower HRC values compared to the synthetic polymers prevalently in use today. Table 6 also demonstrates that the toxic crosslinker GA is not necessary to create naturally derived resins and composites that retain low HRC values.

#### 4. Conclusions

This study demonstrated that CFF can be successfully incorporated into SPI and JF to produce green CFF/SPI resins and JF/(CFF/SPI) composites, offering an environmentally beneficial pathway to upcycle CFF waste. The MCC results of this study indicate that for SPI based resins, addition of CFF increased pHRR from 101 to 120 W/g for CFF/SPI resins without GA and from 94.5 to 114 W/g for GA-crosslinked CFF/SPI resins as CFF loading increased from 0 to 30%. While inclusion of CFF worsened pHRR, other flammability metrics like THR improved. The net effect of CFF addition on SPI's flammability was thus difficult to ascertain. GA was found to improve the SPI resins' fire-safety as CFF/SPI resin with GA had lower pHRR at all loading amounts of CFF from 0 to 30% compared to CFF/SPI resins without GA.

For JF/(CFF/SPI) composites with and without GA, 20% CFF loading resulted in the lowest pHRR at 81.1 W/g for JF/(CFF/SPI) composites without GA and 86.0 for GA-crosslinked JF/(CFF/SPI) composites. Unlike the CFF/SPI resins, adding GA increased the pHRR parameter for JF/(CFF/SPI) composites. JF/(CFF/SPI) composites with and without GA showed superior fire-safety properties compared to both CFF/SPI resins, with or without GA, indicating that JF aids in reducing fire hazard regardless of the inclusion of GA. Furthermore, of all the formulations, the JF/(20/80) composition was shown to have the lowest pHRR at 81.1 W/g, evidencing that this study produced a fully green composite with the best fire-safety parameter amongst the tested combinations.

Future work will supplement the MCC results with cone calorimetry and thermogravimetry to more fully evaluate the fire-safety parameters and study the mechanical behavior of the green composites as well.

Data also indicated that the HRC of all resins and composites produced in this work were far lower than the traditional petroleum-based plastics. Thus, fully green CFF/SPI resins and JF/(CFF/SPI) composites have a clear advantage over some commonly used petroleum-based plastics, providing a good reason to switch from synthetic plastics to

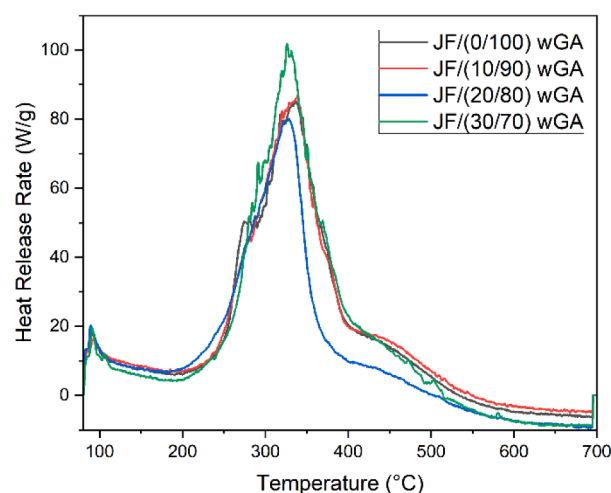


Fig. 4. Typical HRR vs temperature plots for JF/(CFF/SPI) composites with GA.

Table 6  
HRC mean values.

Component	Classification	HRC (J/g·K)
ABS	Synthetic Polymer	669/581
HDPE	Synthetic Polymer	-/1490
LPDE	Synthetic Polymer	1680/-
PET	Synthetic Polymer	332/357
PMMA	Synthetic Polymer	514/475
PP	Synthetic Polymer	1570/1130
PS	Synthetic Polymer	927/859
PVC	Synthetic Polymer	138/129
CFF/SPI 0/100	Green Resin	101
CFF/SPI 10/90	Green Resin	112
CFF/SPI 20/80	Green Resin	118
CFF/SPI 30/70	Green Resin	120
CFF/SPI 0/100 wGA	Greener Resin	94.5
CFF/SPI 10/90 wGA	Greener Resin	107
CFF/SPI 20/80 wGA	Greener Resin	114
CFF/SPI 30/70 wGA	Greener Resin	114
JF/(CFF/SPI) (0/100)	Green Composite	83.1
JF/(CFF/SPI) (10/90)	Green Composite	87.7
JF/(CFF/SPI) (20/80)	Green Composite	81.1
JF/(CFF/SPI) (30/70)	Green Composite	94.4
JF/(CFF/SPI) (0/100) wGA	Greener Composite	92.8
JF/(CFF/SPI) (10/90) wGA	Greener Composite	97.4
JF/(CFF/SPI) (20/80) wGA	Greener Composite	86.0
JF/(CFF/SPI) (30/70) wGA	Greener Composite	99.3

green alternatives.

#### Declaration of Competing Interest

The authors declare that they have no competing financial interests or personal relationships that could influence work reported in this paper.

#### Data availability

Data will be made available on request.

#### Acknowledgments

Authors express thanks to Professor Justin Barone for providing the chicken feather fiber used in this work. Authors also acknowledge the partial financial support provided by Tata-Cornell Institute for materials and Archer-Daniels-Midland Company (Chicago, IL) for providing soy protein isolate. The use of the Cornell Center for Materials Research

Shared Facilities, which are supported through the NSF MRSEC program (DMR-1719875), is also acknowledged.

## Supplementary materials

Supplementary material associated with this article can be found, in the online version, at [doi:10.1016/j.jcomc.2023.100394](https://doi.org/10.1016/j.jcomc.2023.100394).

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