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Influence of chicken feather fibre processing technique on mechanical and fire performances of flame-retardant polypropylene composites

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A B S T R A C T

This study conducted a comparative analysis between chemical, and physical processing techniques to repurpose chicken feathers into flame-retardant chicken fibres (FRCF) as adjuvants to enhance the fire and mechanical performances of manufactured polypropylene (PP) composites. Repurposing chicken feathers can lower industrial waste that otherwise requires expensive waste management solutions. A suitable method of attaining FRCF was selected from the two processing techniques by comparing the processed fibres' fire reaction and mechanical outcomes. PP composites with 44 wt% FRCF achieved ~ 70% reduction in peak heat release rate compared to that of neat PP. The chemically-treated fibre composites achieved a V-0 rating in vertical burning tests, in contrast to the physically treated fibre (PT-FRCF) composites which achieved no rating. The fire and mechanical test results clearly demonstrated the impact of the fibre processing techniques on the manufactured composite's performance.

1. Introduction

The industrial processing of chickens to get rid of their feathers, which comprise almost 3 % of their body weights, contributes to many industrial landfills, requiring a costly waste management system [1]. The global chicken feather production was 102 million tonnes in 2021, with a steady growth of 2 % over the past ten years [2]. Solid wastes are substantial challenges that require special care in locations where chicken farming is regarded as a primary economic activity, mainly due to the adverse environmental effect.

Repurposing or reusing waste from any industry is critical in promoting circular economies. Natural fibres, specifically plant and animal fibres, which are carbon–neutral, biodegradable and capable of imparting both mechanical and flame-stalling properties to the composites, form a salient topic for current researchers.

Previous research has established that fibre surface modifications can enhance the natural fibre/matrix interfacial bonding [3], thus providing better mechanical properties. The fibres' surface morphology, chemical structure, and matrix polarity are primarily responsible for mechanical and chemical bonding at the interface [4]. In terms of specific mechanical properties, natural fibres show comparable or better results than those of many synthetic fibres (e.g. glass fibres) [3,5,6] due to their lower densities. Increased utilisation of naturally available materials is needed to reduce the environmental impact and the energy required to manufacture composites, which is high for synthetic fibres.

Natural fibres are amenable to modifications due to the presence of stabilised hydrogen bonds from keratin, cellulose and lignin, depending on their original sources. Processing fibres to activate hydrogen bonds at interaction sites can improve interfacial adhesion with polymer matrix. Chemical alterations, like chemical bleaching, can improve surface roughness, enhancing the possibility of mechanical interlocking at the interface. Generation of heat from chemical reactions enables sequential loading of materials onto the fibres, creating a modified fibre that has undergone structural or chemical modifications. Therefore, the fibre processing techniques are of significance for obtaining improved composite properties, as explained in a few studies [7–9].

Non-halogenated flame retardants (FRs) act as alternatives to halogenated FRs to reduce toxicity, and embrace eco-friendly materials and

Abbreviations: CF, Chicken feathers; FRCF, Flame retardant chicken feathers; CT-FRCF, Chemically treated chicken feathers; PT-FRCF, Physically treated chicken feathers; FRs, Flame retardants; IFR, Intumescent flame retardants; MP, Melamine Phosphate; APP, Ammonium polyphosphate; P-FRs, Phosphorous-based flame retardants; FRCF/PP, Flame retardant chicken feather and polypropylene.

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Table 1
Sample name and composition in wt.%.

Sample name	PP (wt.%)	MA-g-PP (wt.%)	FRCF				APP (wt.%)
			CF (wt.%)	PA (wt.%)	Melamine (wt.%)	Melamine Phosphate(wt.%)	
PP	100	_	_	_	_	_	_
CT-FRCF/PP	47	3	14.3	13	16.7	_	6
PT-FRCF/PP	47	3	14.3	_	_	29.7	6

process design [10–13]. Intumescent flame retardants (IFRs) are a type of non-halogenated FRs that form carbonaceous char barriers to protect the underlying polymer layer [14–18]. Moreover, IFRs that are mainly phosphorous-based have a lower impact on polymer composite's mechanical performance.

The performance of an intumescent flame retardant depends on the combination of its constituents, which are the carbonising agent or char former, the blowing agent and the acid source [16,17,19]. Its efficiency depends on the char forming rate and char strength, both of which happen in the condensed phase; therefore, research is usually focused on attaining the best combination for char formation. Commonly, 20–25 % of ammonium polyphosphate (APP) is used to obtain a V-0 rating for PP composites in a vertical burning test, but the associated drop in tensile strength by 25–30 % is of significant concern [6,10,20]. This happens due to the weak interfacial bonding between the hydrophilic APP and the hydrophobic PP matrix. In terms of adhesion between the fibres and matrix, the polarity of natural fibres (generally hydrophilic) is usually incompatible with the hydrophobic thermoplastic matrix, resulting in poor interfacial bonding. This problem can be partially or fully overcome by various fibre treatment methods and with the addition of compatibilisers [20].

Chicken feather (CF) is partially hydrophilic and partially hydrophobic due to polar and non-polar proteins, which is in a way helpful for improving the fibre/matrix interfacial bonding [1]. A few studies have shown that chicken feather fibres can help in arresting the drop in composite's strength by acting as reinforcement due to their moderate aspect ratios and better adhesion to polymeric matrix [21–23]. Therefore, using feather fibre as a carrier to introduce chemicals for improving the composite's property is a logical option. However, the influence of chemical processing techniques of chicken fibres on flame-retardant polymeric composites is not very well known and warrants a systematic study.

The current work investigated the repurposing of chicken feathers by turning them into flame retardant adjuvants to enhance flame retardancy, which can also lower industrial waste avoiding expensive waste management solutions. Meanwhile, lowering the amount of expensive phosphorous-based flame retardants (P-FRs) like APP in polypropylene composites, effectively reducing the cost of manufacturing flame-retardant PP composites. In this work, the effect of chemically etched melamine phosphate onto CF has been characterised using FTIR, scanning electron microscope, mechanical tests and cone calorimeter tests to investigate the impact of IFR treatment on the fire and mechanical performances of the modified CF reinforced composites. The chemical and physical mixing of melamine phosphate helped us realise the influence of fibre treatment techniques on the material properties of the manufactured composites. The flammability and thermal properties of flame-retardant chicken feather and polypropylene composites were evaluated by vertical burning (UL-94), cone calorimeter and thermogravimetric analysis (TGA), which gave conclusive evidence to decide which fibre processing technique was more effective.

2. Experimental methods

2.1. Materials

Raw chicken feathers were supplied by Wallace Group Ltd. (New Zealand). Without purification, phosphoric acid (PA, concentration: 85 wt% in H_2O) and melamine from Sigma-Aldrich were utilised. Clariant NZ Ltd provided PP (K515, MFI: 19) and Maleic anhydride grafted PP (MA-g-PP, Licocene PP MA 6452) as a polymer matrix and compatibiliser, respectively. Moreover, an injection moulding grade FR (Exolit AP 766, Clariant NZ Ltd) was selected as IFR.

2.1.1. Preparation of melamine phosphate

Melamine (232.2 g) was mixed with water (1200 mL) in a 2000 mL beaker at 60 °C with a thermometer and a stirrer included in the setup. After the melamine particles stabilise in the water medium, phosphoric acid (85 wt%, 211.8 g) in water (100 mL) was added and thoroughly mixed using a magnetic stirrer. At 70 °C, the mixture was agitated for 30 min before cooling and crystallising. The melamine phosphate (MP) crystals were filtered out and dried for 18 h at 100 °C in an oven before powdering using a pestle.

2.1.2. Preparation of chemically treated flame-retardant chicken feather

Raw chicken feather fibres were washed with diluted hydrogen peroxide, washed with water and laundry detergent [24], and dried to remove excess moisture. A mechanical granulator (GR2020 granulator, MORETTO S.P.A, Italy) was used to chop chicken feathers (CF). Then it was treated with phosphoric acid (PA)/water solution at room temperature using a powder mixer. The PA-treated CFs were mixed with reactive amines (melamine) in a 1.1:1:1.2 ratio by wt.% (weight percentage) and were dried at 70 °C until reaching a constant weight to obtain chemically treated flame-retardant CFs (CT-FRCFs). The treatment method also draws similarity to the treatment method established in paper by Jung et.al [25].

2.1.3. Preparation of physically treated flame-retardant chicken feather

The chopped chicken feathers were mixed with melamine phosphate (MP) in a fixed ratio by wt.% at room temperature, Table 1. The physically treated fibres (PT-FRCF) were dried at 70 °C until a constant weight was reached.

2.1.4. Preparation of flame-retardant PP composite

Flame retardant chicken feathers (FRCFs), APP, MA-g-PP and PP were mixed using a powder mixer. Chemically treated and physically treated CFs made two separate batches of FRCFs. A co-rotating twin-screw scientific extruder (LTE 26–40, Lab Tech twin-screw extruder) was used to extrude FR CF/PP composite, the feeding zone and extruding zone set at 150 °C and 200 °C, respectively. The powder mixer of 44 % FRCF, 6 % APP, 47 % PP, and 3 % MA-g-PP by weight formed the base material for extrusion. The obtained extrudate was pelletised and then moulded into test samples for further characterisation.

2.2. Characterisation

2.2.1. Fourier transform infrared spectroscopy (FTIR)

The interactions between MP, CF and the surface functional groups of the modified CF were investigated by Fourier transform infrared using a NicoletiS50 spectrometer (Thermo Electron Corp., U.S.A).

2.2.2. Flammability tests

ASTM D 3801–10 was used to conduct vertical burning tests (equivalent to UL-94 standard). Samples with dimensions of length (L) = 125 mm, width (W) = 13 mm, and thickness (T) = 2.4 mm were

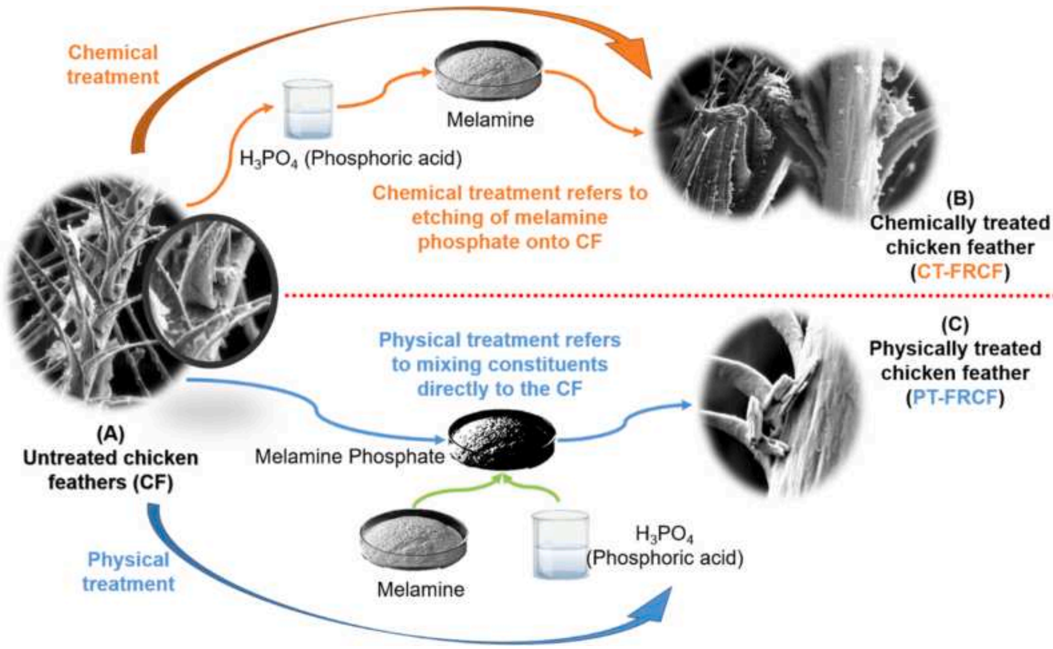

Fig. 1. Schematic diagram of fabrication of FR chicken feather: (A) untreated CF, (B) chemically treated chicken feather (CT-FRCF) and (C) physically treated chicken feather (PT-FRCF).

prepared and preconditioned for 48 h at 23 °C and 50 % relative humidity. The tests were repeated five times for each sample, and the average values have been reported. The test findings were divided into V-0, V-1, V-2, and no rating (NR). The quantitative information on flammability parameters was obtained using a cone calorimeter (Fire Test Technology, East Grinstead, UK) following ASTM E1354-11. The samples were tested horizontally with a 50 kW/m^2 external heat flux. Before the test, the samples were preconditioned for 48 h at 23 °C and 50 % relative humidity. Preconditioning of the samples is done according to ASTM E1354-11 to eliminate the possible discrepancies in results that might occur during heating of a hygroscopic material as a series of complex processes of dissociation, vaporisation, and moisture migration takes place during combustion.

2.2.3. Thermal stability

The thermal stability of MP, CF and FRCF was measured through a Thermogravimetric analyser (Q5000, TA Instruments, U.S.A) under a nitrogen atmosphere with a flow rate of 50 mL/min from room temperature to 700 ℃ at a heating rate of 10 ℃/min.

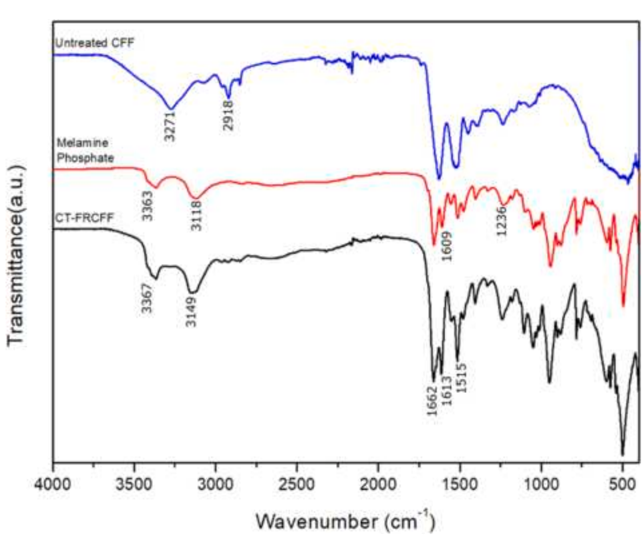

Fig. 2. FTIR curves of untreated CF, Melamine Phosphate and CT-FRCF in the range of (4000 cm^{-1}–400 cm^{-1}).

2.2.4. Morphology

The morphology observation and elemental analysis of the treated feathers and reinforced composites were carried out by field emission environmental scanning electron microscopy (SEM Quanta200, FEI, USA) with energy-dispersive X-ray spectroscopy (EDS, EDAX Pegasus EDS detector, AMETEK.INC., USA).

2.2.5. Mechanical properties

According to ASTM D 638, the specimens' tensile characteristics were evaluated at room temperature at 50 mm/min speed on an Instron universal testing machine (UTM 5567, UK). An Instron universal testing machine (UTM 4463, UK) was used to evaluate the specimens' flexural qualities at room temperature following ASTM D 790. At least five specimens were examined for each sample to get the average tensile and flexural characteristic values.

3. Results and discussion

3.1. Fibre modification process

Morphological and structural changes of the fibres were investigated using SEM and FTIR. Improvement in physical and chemical adhesion from fibre modification processes may be followed from the schematics of Fig. 1. Chicken feather surface activation by treatment with phosphoric acid not only improved the acid dispersion through the composite material but also enhanced the effective matrix interaction, which had been established in previous research work [25]. The processing technique provided a suitable platform for effective melamine phosphate synthesis and intumescent flame-retardant dispersion avoiding agglomeration at specific sites as observed in the physical treatment process. The influence of processing techniques was observed by analysing the treated fibre and subsequently performing fire and mechanical characterisations to infer its overall effects on composite manufacturing.

Melamine phosphate manufacturing involved the mixing of

Table 2
FTIR peaks characterisation.

Before treatment (cm^{-1})	After treatment (cm^{-1})	Appearance	Group	Characteristics
3271	3149	Strong	O–H stretching	Intermolecular bonded
No Peak	3367	Medium	N–H stretching	Aliphatic Primary amine
1625	1662,1613	Strong	N–H bending	C=O amide I
1511	1515	Broad to Strong	N-O stretching	nitro compound

melamine with phosphoric acid in a 1.5:1 M ratio [26], which included continuous stirring under heated conditions for the onset of crystallisation. Later these crystals were pulverised into the standard commercial size of 5–15 μm. Considering the market selling price for individual chemicals and materials used in achieving the flame-retardant composites, a 16.8 % reduction in processing cost can be realised by incorporating melamine phosphate through the chemical etching technique. Utilising chicken feather (CF) as the phosphoric acid carrier with subsequent addition of melamine not only saved the cost of production of melamine phosphate but also improved its dispersion through the composite, which would be established later in this work. A comparison of intumescent flame-retardant treatments of CF will be elaborately analysed in the next sections.

3.2. Condensed phase investigation with FTIR

FTIR spectra of untreated chicken feather, melamine phosphate and chemically treated FRCF are shown in Fig. 2, Table 2. The characteristic FTIR peaks of chemically treated chicken feather shows the development of new peaks when MP is chemically etched over feather fibre. As shown in Fig. 3, the FTIR curve obtained by subtracting the CF curve from that of chemically treated FRCF was found to be in coherence with the curve of neat melamine phosphate. The result clearly establishes the presence of melamine phosphate on the etched fibres. The visualisation of new peaks around 3300 cm^{-1} leads us to believe that there is an addition of the N–H group after the treatment of chicken feathers. This chemical modification has contributed to improving the thermal stability and fire performance of chicken feather fibre composites. The extremes between 2900 and 2960 cm^{-1} confirmed the preservation of –CH_2 and –CH_3 functional groups in the chemically treated CF, similar to untreated CF. The peaks at 1625 cm^{-1} and 1511 cm^{-1} in the untreated

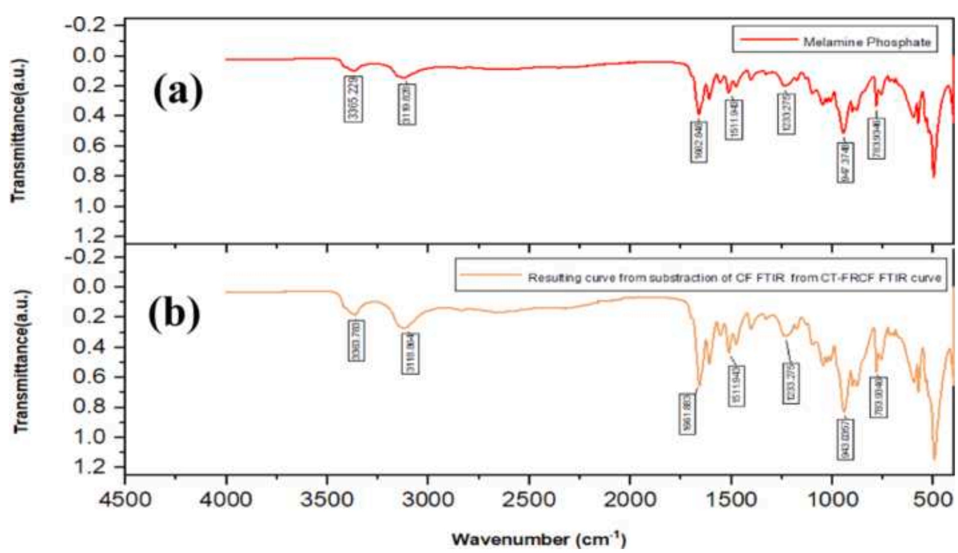

Fig. 3. (a) FTIR curve for melamine phosphate; (b) curve subtraction of untreated CF from CT-FRCF to see the remaining curve of chemically etched melamine phosphate.

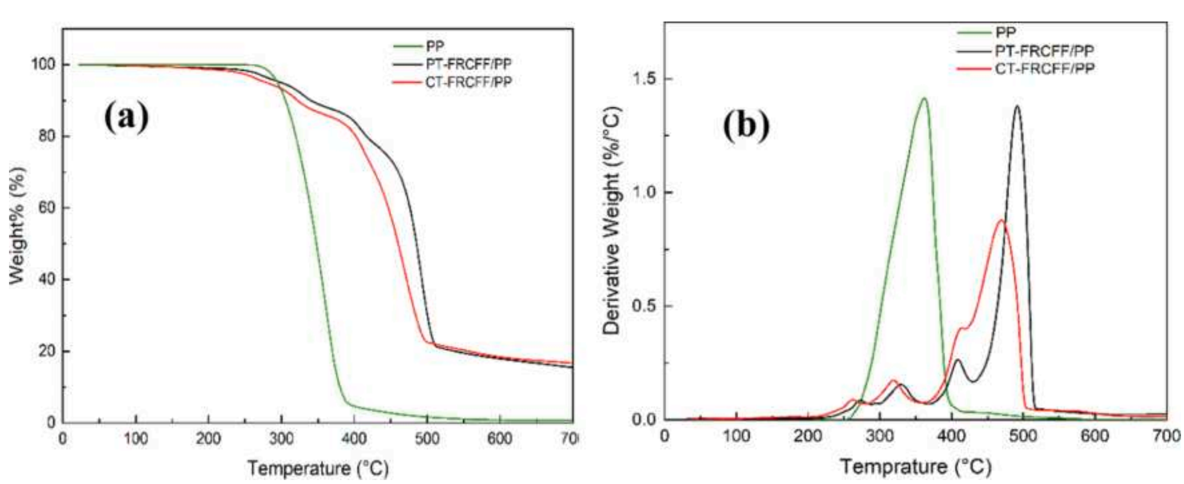

Fig. 4. (a) TG and (b) DTG curves of PP, physically treated (PT-FRCF/PP) composite, chemically treated (CT-FRCF/PP) composite.

Table 3
Peak temperatures of PP, PT-FRCF/PP, CT-FRCF/PP.

Sample	T5% (°C)	Tmax1 (°C)	Tmax2 (°C)	Tmax3 (°C)	Tmax4 (°C)	Residue at 700 °C (wt.%)
PP	294.6	363.3				0.73
PT-FRCF/PP	299	273	330	408	491.86	15.28
CT-FRCF/PP	279.6	262.7	321	413.5	472.76	16.72

Table 4
UL-94 test results for PT-FRCF/PP and CT-FRCF/PP composites.

Sample	Total self-extinction time after first 10 s flame application (s)	Total self-extinction time after second 10 s flame application (s)	Dripping igniting cotton	UL-94 rating
PT-FRCF/PP composite	57	Flame reaches clamp	Yes	No rating (NR)
CT-FRCF/PP composite	1.5	1	No	V-0

feathers were attributed to amide I and amide II groups, respectively, which later mimicked the bands of melamine phosphate after chemical treatment [27].

4. Thermal stability

Drawing a comparison between the flame-retardant mechanisms of physically treated and chemically treated CF, thermogravimetric (TG) and derivative thermogravimetric (DTG) measurements for their respective composites have been adopted in Fig. 4 and summarised in Table 3. PP shows a single rapid decomposition stage at 363.6 °C with nearly 97 % mass loss for dehydration and scission reactions. The TG curves show that treated chicken feather reinforced composite has a lower onset temperature than that of neat PP although the decomposition rate is significantly lower, highlighting the improvement in thermal stability with the addition of flame-retardant chicken feathers (FRCF).

In the main decomposition temperature (250 °C to 550 °C), the curve for fibre-reinforced composite shows lowered decomposition rate with nearly 77–80 % mass loss for the catalytic degradation of the released acid source and polyphosphate compounds from MP in the FRCF system [28]. As APP and MP create a system of intumescent flame retardancy, the released acid esterifies the carbon source. The ester decomposes via dehydration, yielding a carbonaceous residue. The liberation of an acidic source below the decomposition temperature of the carbonising agent is preferable, with polymeric decomposition occurring around the dehydration temperature [29]. The chicken feathers contain 17.7 wt% of the cysteine amino group as its principal hydrophobic constituent. Under heating, it could combine with phosphoric acid liberated from MP, catalysing the phosphorylation of hydroxyl groups in CF [30]. MP experiences the condensation reaction, chain scission and crosslinking reactions at the temperature below 300 °C, producing polyphosphate compounds that could react with the PP matrix to enhance the formation of char layers. This results in an increased char residue after subsequent pyrolysis. The acidic catalyst promotes dehydration and char formation, resulting in rigid char around the edges of CF-PP composites, which hinders the flame propagation into the sample. These enhanced char layers may act as protective layers on the composite's surface. The MP content produces polyphosphate compounds resulting in an earlier decomposition of composite and higher carbon residues after burning. Though physically treated FRCF/PP composite has a lower rate of decomposition in temperatures below 450 °C, the rate of decomposition remains lower for chemically-treated FRCF/PP after 450 °C, which shows improved quality of formed char, thus inhibiting the decomposition of the underlying composite. Heavier char formation for chemically treated FRCF/PP composite is evident from the ~ 2 % increment in the residue weight in comparison to physically treated FRCF/PP composite and 16 % when compared with residue weight of neat PP.

Fig. 5. Residual specimens after vertical burn test: (a) PT-FRCF/PP; (b) CT-FRCF/PP.

4.1. Reaction to small flame test

The flammability performances of PP, PT-FRCF/PP and CT-FRCF/PP were further analysed using UL-94 vertical burn tests by applying flame directly to the samples placed vertically as per ASTM D3801 standard, summarised in Table 4. The neat PP showed continuous flame drips with igniting of the cotton, and the flames reached the holding clamp resulting in the apparent no rating (NR). However, for the PT-FRCF/PP composite, it started to burn slowly at first, only to ignite and extinguish after > 50 s for the first flame application. During this time, a char layer was formed around the specimen's bottom end, which prevented the flame spread upward by acting as a barrier. Eventually, it dropped off, detaching the source of fire from the specimen. The second flame application to the partially charred PT-FRCF/PP composite led to continuous dripping. The drips ignited the cotton with flames reaching the holder clamp showing similarity to the burning of PP that suggested an ineffective intumescent char barrier. There was some char formation around the edges of the composite, which hindered the continuous burning of the sample, but it was not adequate to stop the flames from reaching the clamp, Fig. 5(a). Hence PT-FRCF/PP composites were labelled with no rating (NR), while CT-FRCF/PP composites had self-extinguishing characteristics.

The char formed for CT-FRCF/PP composites, Fig. 5 (b), effectively created a barrier between the composite and the atmosphere, leading to flame-out within seconds after both flame applications, hence a V-0 rating could be achieved. This V-0 rating could be attributed to the chemical treatment of melamine phosphate onto chicken feathers as every other parameter in composition and manufacturing of the physically treated (PT-FRCF/PP) and chemically treated (CT-FRCF/PP) composites remained very similar.

It is evident from Fig. 5 that physically treated FRCF/PP ignites while trying to form a solid char to promote intumescence but failing to do so. It may be attributed to the large melamine phosphate particle size of 18.39 ± 8.8 μm as compared 0.78 ± 0.3 μm particle size of the chemical treated CF, Fig. 6. Better dispersion of melamine phosphate along the

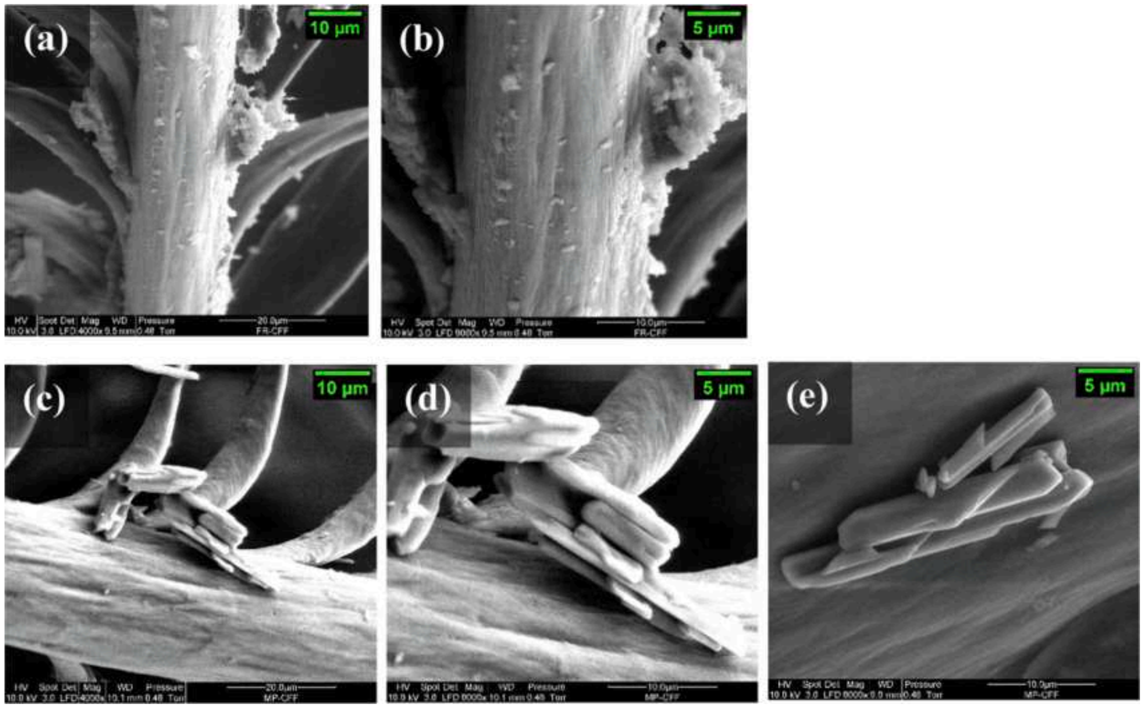

Fig. 6. SEM images of (a and b) CT-FRCF and (c–e) PT-FRCF distinctly show the melamine phosphate particles.

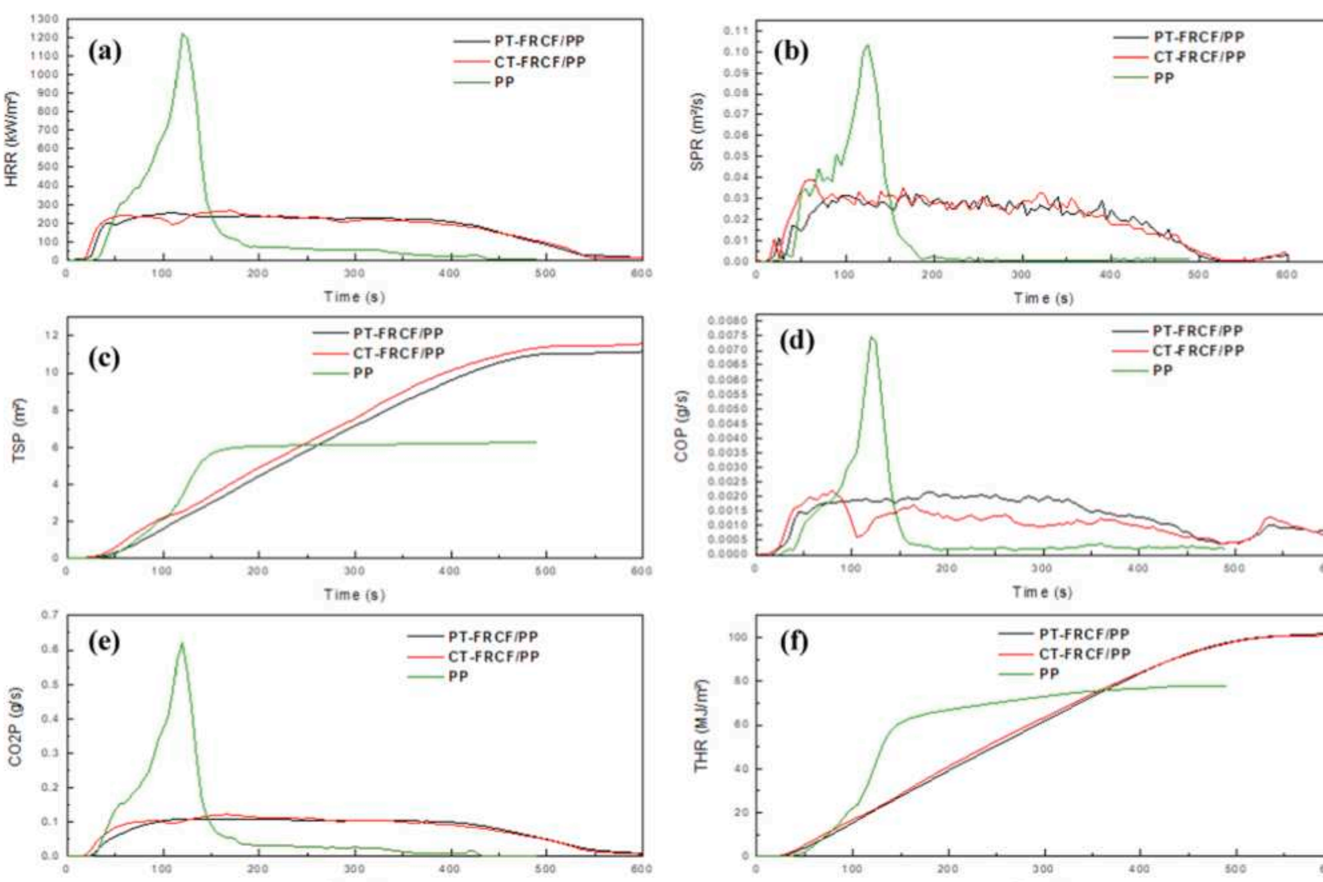

Fig. 7. (a–f) Cone calorimeter test results of PP, PT-FRCF/PP and CT-FRCF/PP.

Table 5
Detailed Cone calorimeter test results of PP, PT-FRCF/PP and CT-FRCF/PP.

Sample	T_{ig} (s)	PHRR (kW/m^2)	T_{PHRR} (s)	FGR	THR (MJ/m^2)	EHC (MJ/kg)	MLR (g/s*m^2)	MARHE (kW/m^2)	SPR (m^2/s)	TSP (m^2)	Av-COY (10^2 kg kg^{-1})	FRI	TOC
PP	34.25 ± 2.22	1106.69 ± 148.11	126.25 ± 11.09	8.76	83.59 ± 4.13	40.46 ± 4.49	4.83 ± 0.82	423.215 ± 26.45	0.0153	6.56 ± 0.72	0.025 ± 0.0028	1	48.66 ± 4.33
PT-FRCF/PP	22 ± 1	254.76 ± 4.54	115 ± 13.23	2.21	100.80 ± 1.01	30.07 ± 0.326	5.5866 ± 0.19	214.71 ± 4.89	0.019067	11.41 ± 0.28	0.0318 ± 0.00217	3.281	55.989 ± 0.538
CT-FRCF/PP	21.67 ± 1.52	276.707 ± 20.85	158.33 ± 16.07	1.75	98.55 ± 2.47	30.61 ± 0.56	6.047 ± 0.02	205.70 ± 8.49	0.02047	10.86 ± 0.52	0.0245 ± 0.0015	4.254	54.719 ± 1.69

T_{ig}: Time to ignition; PHRR: Peak heat release rate; T_{PHRR}: Time to peak heat release rate; FGR: Fire growth rate; THR: Total heat release; EHC: effective heat of combustion; MLR: Mass loss rate; MARHE: maximum average rate of heat emission; SPR: Smoke production rate; TSP: Total smoke production; Av-COY: Average Carbon Monoxide yield; FRI: Fire retardancy index; TOC: Total oxygen consumption.

Table 6
PHRR of composites with and without treated chicken feathers.

Sample	PP	PP/APP	PP/MP/APP	PT-FRCF/PP	CT-FRCF/PP
PHRR (kW/m^2)	1106.69 ± 148.11	586.47 ± 18.15	338.76 ± 29.37	254.76 ± 4.54	276.707 ± 20.85

chicken feather surface enhances the char forming ability under exposure to combustion. However, the physically treated FRCF/PP composites could not achieve V-0 due to the continuous burning after second flame application. The uneven dispersion of melamine phosphate particles in the composite results in the inability to create an effective char barrier against the flame. Furthermore, the inconsistent carbonisation degree on the surface and the poor shielding from heat source can create regions with inadequate fire inhibition capabilities that could not prevent flames from infiltrating into the material.

4.2. Forced flame combustion tests

PP and PP composites' forced flaming reactions with CF were analysed using a cone calorimeter. Fire reaction properties, such as heat release rate (HRR) and total heat release (THR), which help us analyse the heat and gas production capabilities of PP, PT-FRCF/PP and CT-FRCF/PP, have been illustrated in Fig. 7 with more detailed results mentioned in Table 5. The composite samples contain keratinous natural filler (CF), making the material partially hygroscopic [31]. The heat transfer rate in a hygroscopic material is influenced significantly by the release of the entrapped moisture within the samples.

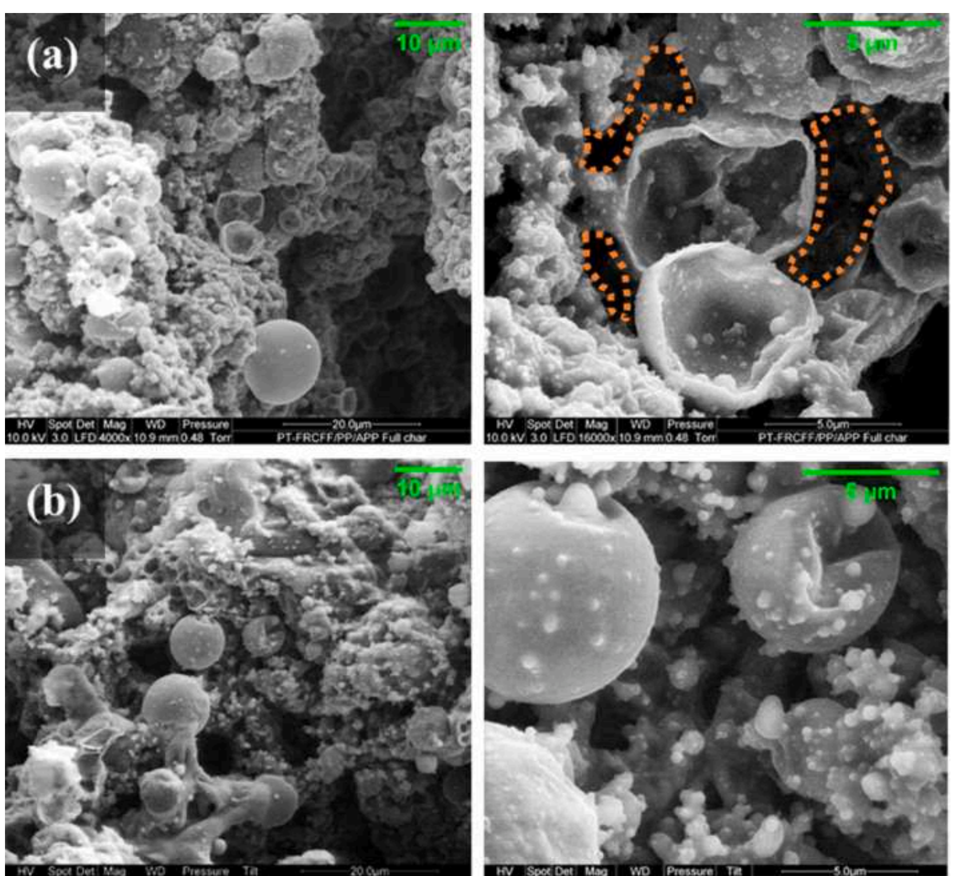

Fig. 8. SEM images of (a) PT-FRCF/PP and (b) CT-FRCF/PP.

Fig. 9. Images of char residue obtained after cone calorimeter: a) PT-FRCF/PP; b) CT-FRCF/PP.

The addition of the IFR system does contribute to the reduction of PHRR and THR. It is observed that the composite containing melamine phosphate, 6% APP and PP though performed better than PP/ 6% APP composite - the PHRR was higher than those of both PT-FRCRFF/PP and CT-FRCFF/PP, suggesting the beneficial role played by chicken feather fibre (Table 6).

The decrease in PHRR is explained by the formation of a char barrier, which helps retain combustible fuel in the condensed phase. The addition of IFRs lowers the effective heat of combustion (EHC) in comparison to one of pure PP, which implies a gas phase result that is caused by the dilution of combustible fuel from the release of ammonium, as well as radical scavenging by PO and PO_2 from APP for oxygen free radicals leading to inhibition of chain reactions during combustion, which is a definite indicator of the mode of action of phosphorous [32]. Moreover, micro-images of the char after the cone calorimeter measurements, Fig. 8, clearly show different structures between the two composites. The blowing agents' action is well sustained by the interface of the CT-FRCF/PP with less void enabling it to form a stronger char barrier than its counterpart (Fig. 8) [33].

The HRR curve of PT-FRCF/PP shows that char was developed in 40 s. However, it is followed by a gradual rise in HRR, which implies the breaking of char to expose underlying material and release volatile gases for combustion. While CT-FRCF/PP sample has a steady HRR after the first char formation at about 55 s, the char formed by CT-FRCF/PP is stronger than those produced by PT-FRCF/PP, which can be visually confirmed by the char images, shown in Fig. 9. The smoke production rate (SPR) helps confirm that the char produced by CT-FRCF/PP is more robust, which highlights the strength of char formed by the co-synergistic actions of APP and chemically-etched melamine phosphate [34] and the reduced exposure of the underlying material to heat source. The quick rise in SPR for CT-FRCF/PP followed by a reduction after 55 s helps to manifest the effectiveness of char formed.

On the contrary, the gradual rise in SPR for PT-FRCF/PP suggests the inability to form a solid char to hinder the flame propagation onto the underlying polymer layer. Though the weight of char produced by both the composites is very similar, the char formed for CT-FRCF/PP looks dense and steady, as the HRR data suggests. The continuous rise and drop of SPR for PT-FRCF/PP show the char layer's continuous making and breaking, resulting in a weak intumescent system.

To further strengthen our findings, corelating the gathered data with theoretical analogies was imperative. THR is proportional to the combustion efficiency (χ), the heat of combustion of volatiles (h_c^0), the mass of specimen (m_0), and the char yield (μ), as shown in the equations (1–3). [35,36].

$$EHC \sim \chi . h_c^0 \tag{1}$$

$$HRR \sim \chi .(1-\mu).h_c^0 \tag{2}$$

$$THR \sim \chi .(1-\mu).h_c^0 .m_0 \tag{3}$$

Since the fuel load decrease was more dramatic for CT-FRCF/PP, which achieved greater residue yields, the effective heat of combustion ($\chi\ h_c^0$) was reduced. The reductions in EHC for CT-FRCF/PP and PT-FRCFF/PP compared to 20APP/PP (4% and 5.6% respectively) and PP (24.3 and 25.6 % respectively) are explained by the reductions in combustion efficiency. The dilution of fuel also affects the overall heat of combustion. Reduction in combustion efficiency leads to incomplete combustion, increasing smoke production and CO yield. For CT-FRCF/PP and PT-FRCFF/PP, the total smoke production (TSP) increased to 1.7 and 1.6 times compared to neat PP, respectively. This phenomenon is also linked with the average carbon monoxide yield increase during burning (av-COY) due to incomplete combustion.

Based on the heat release rate (HRR) curves, the CT-FRCF/PP shows a 21 % reduction in fire growth rate (FGR) compared to PT-FRCF/PP, which helps assess the fire hazard status of the composite according to the equation suggested by Shao et al. that is as follows [14]:

$FGR = PHRR/T_{PHRR}$ (4)

The FGR suggests the time to flashover, which indicates the time within which a person in distress can be evacuated, or the fire extinguisher can arrive, lower the FGR better is the material in terms of commercial applicability, which is evident from the delay in time to reach the PHRR.

The flame retardancy index (FRI), a ratio of the product of the total heat release and fire growth rate of the polymer and its composites, can also be used to determine the flame retardancy of composites. PT-FRCF/PP and CT-FRCFF/PP both show value (>1), which suggests that the additive did enhance the flame retardancy of PP, as shown in Table 5. The composite samples contain keratinous natural filler (CF), making the material partially hygroscopic[31]. The heat transfer rate in a hygroscopic material is influenced significantly by the release of the entrapped moisture within the samples. Moreover, CT-FRCFF/PP showed a 29.6 % increment in its FRI than those of PT-FRCFF/PP, strengthening the point that chemical treatment is better than the physical treatment of melamine phosphate for improved flame retardancy capabilities in CF.

4.3. Mechanical properties

The Young's modulus, tensile strength, elongation at break and flexural characteristics of the investigated PP composites with treated chicken feather reinforcement are summarised in Table 7. The CT-FRCF/PP composite exhibits a Young's modulus 1.87 times higher than those of the pure PP with an 8 % reduction in tensile strength. However, the PT-FRCF/PP composite has 1.47 times higher Young's modulus compared to those of PP with a 23 % reduction in tensile strength. At 44 % FRCF loadings, the FRCF composites behaved disparate: CT-FRCF

Table 7
Tensile and flexural properties of PP and FRCF/PP composites.

Sample	Tensile strength (MPa)	Young's modulus (GPa)	Elongation at break (mm)	Flexural strength (MPa)	Flexural modulus (GPa)
PP	27.1 ± 0.103	1.27 ± 0.066	71.8 ± 8.4	37.9 ± 0.62	1.21 ± 0.046
PT-FRCF/PP	20.9 ± 0.279	1.87 ± 0.092	4.56 ± 0.076	33.6 ± 0.13	1.4 ± 0.045
CT-FRCF/PP	25.2 ± 0.133	2.38 ± 0.079	3.56 ± 0.084	58.1 ± 1.56	2.99 ± 0.07

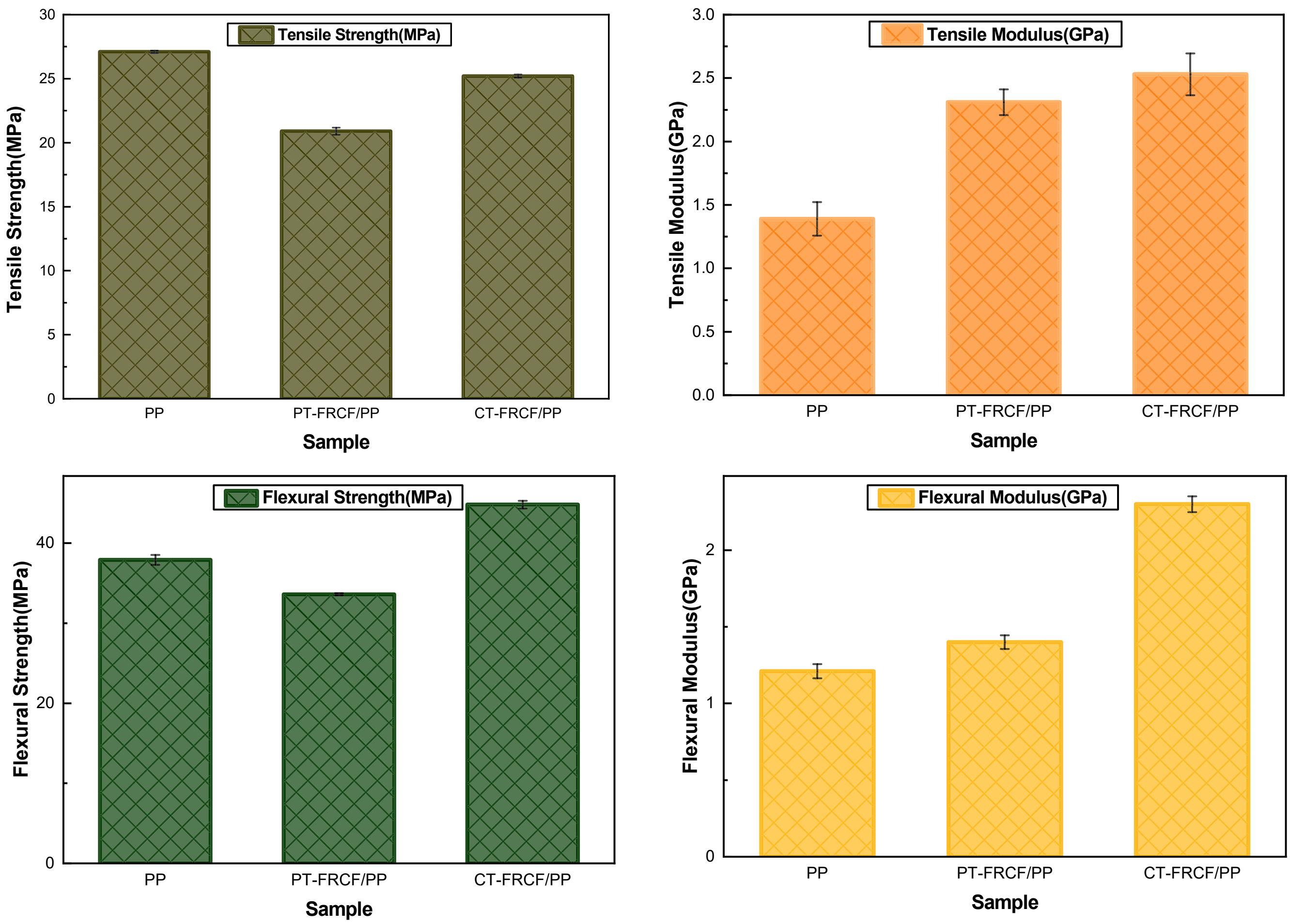

Fig. 10. Mechanical properties of FRCF-PP composites with different treatment methods.

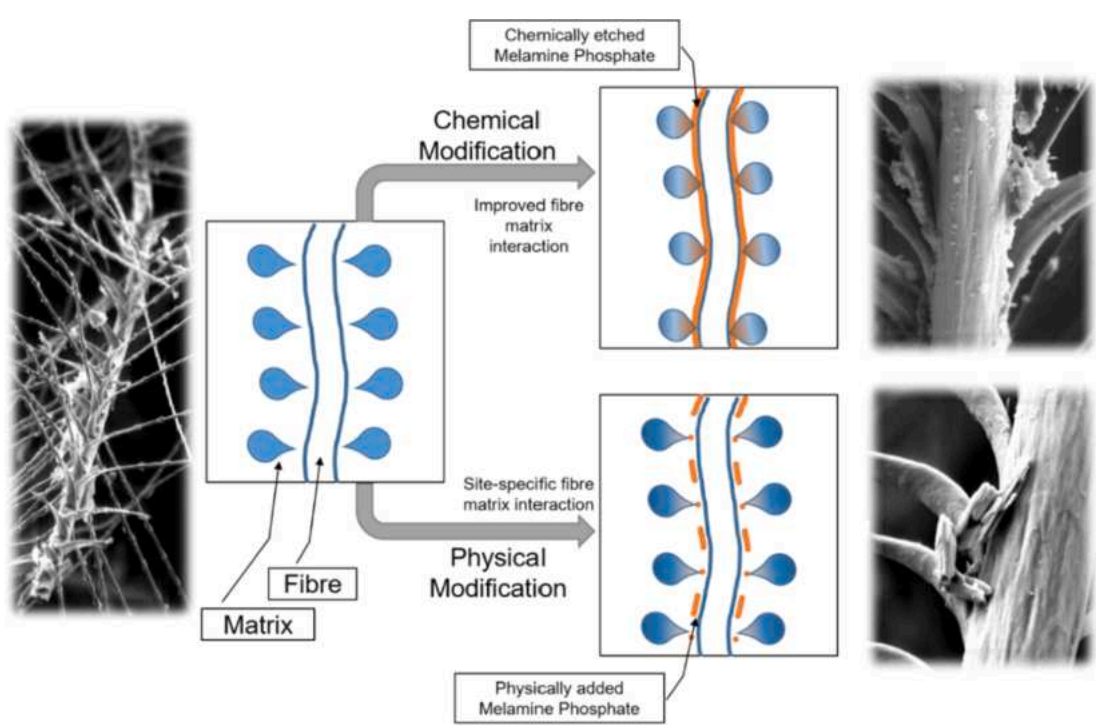

Fig. 11. Schematic model for fibre modification process.

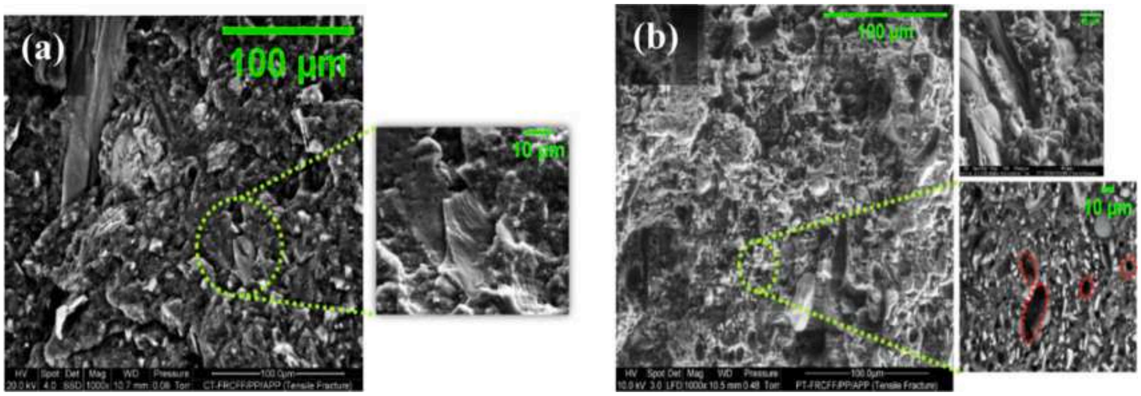

Fig. 12. SEM image of fracture surface after tensile test: (a) CT-FRCF/PP, (b) PT-FRCF/PP.

composite showed a relatively high Young's modulus (1.87 times higher) than those of PT-FRCF composites (1.47 times higher) in comparison to neat PP. The overall effect of flame-retardant addition to PP led to decreased elongations at the break with higher elastic modulus, caused by increased material's brittleness. The nonuniform structures of the CF gets compensated effectively by FRCF, which has been established by Jung et al. [25]. However, the addition of FRCF helps improve the tensile properties of PP composites compared to PP-APP composite, also reducing the APP loading to achieve similar flame retardancy as established by comparing data from other publications [6,20,25].

The excellent distribution of micron-sized MP for CT-FRCF and agglomerated MP for PT-FRCF relates well with the schematics of fibre modification process (Fig. 11) with supporting visuals from Fig. 6 and Fig. 12. Fig. 12 clearly shows better bonding between the FRCF in the CT-FRCF/PP composite. In comparison, heterogeneity is observed among the PT-FRCF/PP composite constituents, with pockets highlighted in red showing debonding of FRCF from the PP matrix under tensile loading, referring to weak interfacial bonding.

As observed from Table 7 and Fig. 10, the flexural strength for CT-FRCF/PP was about 18% higher than that of neat PP, while PT-FRCF/PP was about 12% lower. Overall mechanical properties for CT-FRCF/PP were better in comparison to PT-FRCF/PP and similar to PP for tensile characteristics while showing higher flexural strength than those of PP and PT-FRCF/PP. These investigations pointed out that the mechanical properties of CT-FRCF/PP and PT-FRCF/PP were comparable while being superior in some scenarios to PP, establishing the ability of CT-FRCF to act as a reinforcing filler for PP composites.

5. Conclusions

Investigating the influence of chicken feather fibre processing technique as flame retarding and reinforcing filler adjuvants for polypropylene composites has highlighted the practical use of waste materials for an economically viable waste management option. Chemical etching of MP onto CF helped improve the FRCF/PP composites mechanically while exhibiting acceptable flame retardancy characteristics compared to its commercial counterpart. The improved cohesiveness between PP, chemically treated-FRCF and low loading of APP could be reasoned to the increased availability of phosphorylated polar groups and crosslinked via P/P-N/P-O bonds. The development of tenacious polyaromatic intumescent char improved the flame retardancy of CT-FRCF over PT-FRCF, increased fuel retention, and provided efficient protective layer effects. The impact of melamine as a blowing agent in physically treated-FRCFF was not as effective as in chemically treated-FRCFF, clearly evidenced by the voluminous char formed. An economical alternative to expensive phosphorous-FR additives like APP makes using CF from poultry waste as flame retardant additives in polymeric composites meaningful. Furthermore, the transformation of the chicken feather into a high-value product in the form of flame-retardant bio filler will help avoid costly waste management solutions.

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

No data was used for the research described in the article.

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