Research Article

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Effect of cinnamon essential oil on morphological, flammability and thermal properties of nanocellulose fibre–reinforced starch biopolymer composites

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Abstract: The effect of different cinnamon essential oil (CEO) contents on flammability, thermal stability and morphological characteristics of nanocellulose fibre–reinforced starch biopolymer composites was studied. This sugar palm nanocellulose reinforced with sugar palm starch, containing 0–2% CEO, was prepared through solution casting technique. From scanning electron microscopy analysis, the cross-sections of the CEO-containing films showed appearance of micro-porous spots as micro-porous holes because of the occurrence of partial evaporation on the cryo-fractured surface as a result of the vacuum condition. Increment in CEO concentration resulted in increasing trend of the number and size of the micro-porous holes. Significant increase was observed in the thermal stability with the CEO loading when compared with neat composites. Besides that, increasing CEO loading also resulted in decrement of linear burning rate of the composites.

Keywords: morphological properties, flammability, sugar palm nanocellulose, sugar palm starch, essential oil

1 Introduction

Conventional packaging from petroleum-based plastics, such as polypropylene, polyethylene, polyvinyl chloride, polyamide, polyethylene terephthalate and high density polyethylene, is being extensively used in food and beverage industries considering their ease of processing, strength, cost effectiveness and durability [1,2]. Food packaging requires a considerable consumption of various materials, where utilization of plastics has exponentially increased over the last two decades, with approximately 5% annual growth. Plastics, after paper and cardboard, are also the second most regularly used materials in food packaging applications. According to Sanyang et al. [3], the lack of biodegradability of conventional packaging materials that are petroleum based is known to contribute to many environmental hazards, e.g. emitting dangerous toxins into the air and destroying ocean habitats from the coral reefs to thousands of other species. This will lead to loss of revenue for fishing and tourism industries. Moreover, restoring it is costly as the cities, states and country need to spend millions of their revenues to clear-out littered plastics as well as to build and sustain landfills.

In recent years, biodegradable polymers, particularly agro-based polymers, have been widely explored as alternatives to non-degradable polymers that are now primarily used in the production of food packaging films [4–7]. Starch is one of the widest packaging biopolymers available as it is easily available, biodegradable, renewable and of low cost [8,9]. Therefore, starch has drawn considerable interests as an alternative to non-biodegradable plastics, hence a promising new green material in the industry [10]. However, starch-based films for packaging applications have reportedly shown that they have poor antimicrobial properties [11,12]. These disadvantages limit their broad uses, particularly for the purposes of food packaging. Therefore, to cater this problem, materials scientists conducted several studies to enhance the antimicrobial properties without affecting their biodegradability.

Natural fibres such as cotton [13], rice husk [14,15], kenaf [16], water hyacinth [17], flax [18], jute [18], ginger

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[19,20] and sugar palm [21] from plants have attracted the attention of many researchers in the past, and they are used because of their important properties such as low cost of materials, ease of availability and biodegradability [22]. For example, cotton fibres were used by Boufi et al. [13] with plasmonic nanoparticles for the destruction of harmful molecules. Rice husk fibres were also used in the development of aluminium-based green metal composites to reduce soil pollution [14]. Jain et al. [15] used agricultural waste rice husk–reinforced epoxy for coating, electronic implements, aerospace and automotive structures. Besides that, many scientists have made an effort on cellulose fibre as conducting composites (i.e. activated carbon) using binder [22]. Thompson et al. [23] reported research on cellulose nanocrystals made from woods. The cellulose crystals were made into composites to produce transparent films, which have good mechanical properties. Bukit et al. [24] represented the work on nanoparticles made from oil palm boiler ash from a palm oil mill. The materials were characterized by X-ray diffraction (XRD) and Fourier transforms infrared (FTIR), and the materials showed great potential in nanocomposite industry. Similar work was represented by Chu et al. [25] on fly ashes and was also characterized using XRD, FTIR and scanning electron microscopy (SEM), which showed promising results. This shows that natural fibre had many advantages to be used in various applications. In the current research sugar palm fibres (SPMs) are used as reinforcement in starch composites in the form of nanocelluloses. One important advantage of using bio-nanocellulose in composites in food packaging is its use does not pose any health hazard unlike nanoparticles made from synthetic materials [26]. In food packaging, nanocellulose composite had better antibacterial activity than its individual constituent (starch and fibre) [27].

The natural fibre–reinforced natural polymer had good advantages over synthetic fibre in terms of biodegradability, recyclability and low cost per weight to resist tensile load [28]. Therefore, selection of the right packaging technologies and materials is a vital aspect in preserving food freshness and quality as well as reducing environmental pollutants. One possibility that is being researched extensively is the inclusion of active substances, e.g. essential oil (EO) in the packaging contents as biopolymer starch-based films. [29]. To help improving and preserving the food safety, scientists have developed a novel packaging, known as active packaging (AP). It might also be a beneficial alternative for both conventional and modified packaging, in terms of preservatives' usage, because it provides microbial protection to the food by decreasing and inhibiting the growth of microorganisms, which subsequently extends the shelf-life of the packed food [30]. Natural antimicrobial ingredients have acquired rising demand as customers are more conscious of possible health hazards associated with the use of preservatives as well as aware of the effectiveness of AP [31–34]. Natural ingredients with antimicrobial activity include lemon oil [35], lemongrass [36,37], peppermint oil [37–39], cinnamon [29,37,39–42], lavender [43], Mexican oregano [44], neem [45], tea tree [39,46], *Lavandula angustifolia* [47], *Mentha pulegium* [47], turmeric [48], lime [49], *Origanum vulgare* L. [50], *Ziziphora clinopodioides* [51], grape seed [51] and *Zataria multiflora* Boiss [52].

Sugar palm starch (SPS) is being extensively used in the manufacturing of bio-based starch films and yielded promising results [3,21,61,53–60]. SPS films are defined as non-toxic, colourless, biodegradable, tasteless, odourless and isotropic. In a previous study performed by Ilyas et al. [21], SPS films that were plasticized with glycerol and sorbitol and reinforced with sugar palm nanocellulose (SPN) were developed [62–65]. The reported findings were significant as they provided information on an ideal formulation to manufacture composite film with enhanced mechanical, thermal and water barrier characteristics. Therefore, authors are currently attempting to incorporate the SPS films' formulation with antimicrobial agents as the carrier of natural additives that might in the foreseeable future be seen as new tendencies in the functional food packaging. AP provides the customer with microbial protection by reducing or inhibiting the growth of microorganisms, which could then prolong the shelf-life of the food.

A research conducted by Kechichian et al. [66] on cassava starch had incorporated clove and cinnamon essential oils (CEOs) and showed reduction in tensile properties, whereas water vapour permeability showed increment compared to control. These EOs were chosen as raw materials in the continuation of their research, which was performed by the same researchers as the present study. Besides that, other authors who presented antimicrobial effectiveness of cinnamon agents in literature include Souza et al. [29], Iamareerat et al. [40], Utami et al. [41], Liakos et al. [39], Rojas-Graü et al. [37] and Nazari et al. [42]. The main constituent of cinnamon oils is cinnamaldehyde, a well-known agent because of its antimicrobial activities.

EOs are plant based having volatile, natural and complex compounds. Besides having strong odour, they become interesting additives in food industry for having good medicinal and antimicrobial properties, as well as providing health benefits. EOs are dissolved with a surfactant because it is insoluble in water (hydrophobic). Tween 80 and Span 80 are two commonly used surfactants in hydrophile–lipophile balance [67]. Cinnamon was mainly used as spice for a long time, which contains main constituents like cinnamaldehyde and eugenol that are two important agents in antimicrobial activities [68]. Cinnamon EOs were reported to have good antimicrobial activity against meat-isolated *Pseudomonas putida* strain [30]. It was also reported that they had high activity on preventing the spoilage of fungi on bread. Oussalah et al. [69] revealed that cinnamon EOs were among the most active EOs, as screened for four pathogenic bacteria. In the current research, SPS films were developed via solution casting technique and were incorporated with cinnamon EOs.

None of the preceding studies, based on a literature survey, has discussed the effects of CEO on the flammability and thermal stability as well as morphology of biopolymer composites of SPN reinforced with SPS. It is well known in composites that the use of nanofillers such as nanocelluloses is important in influencing the composite properties such as thermal performances [70]. Lapčík et al. [70] reported that nanofillers are commonly used for structural and non-structural components, and packaging as well as advanced coating applications. Therefore, this study aimed to investigate the possible effects of using EO as fillers on the flammability, thermal stability and morphological properties of SPN/SPS biopolymer composites at different loadings.

2 Materials and methods

2.1 Materials

There are several places in Malaysia that are planted with sugar palm, hence becoming the sources for SPS and SPF. In this study, SPF and SPS were obtained from a village in Kuala Jempol, Negeri Sembilan, Malaysia. Chemicals used, e.g. sodium hydroxide (NaOH), ethanoic acid (CH_3COOH), Sodium chlorite ($NaClO_2$), sorbitol, glycerol and Tween 80, were purchased from Sue Evergreen Sdn. Bhd., Semenyih, Selangor, Malaysia.

2.2 SPS extraction and preparation

SPS was removed from inside the stem of a matured sugar palm tree using a chainsaw. The starch powder then underwent washing process by adding water to the mixture and followed by using a special designed extractor machine for sugar palm to extract the starch from the mixture. The mixture was then filtered using a sieve (<300 µm), where the fibre remained at the top of the sieve and starch granules flowed with the water into the container. The starch was separated from the water by pouring the water slowly until it reached the level of the starch as it is denser than water. Fibre residues that are by-products were isolated from wet starch. Then, the wet starch was sun dried for 30 min and oven dried at 120°C for 24 h [71].

2.3 SPF extraction and preparation

SPF is located on the stem of sugar palm tree as natural woven shape fibre. SPF wraps up the tree trunk and worker used an axe to cut and remove it from the tree. Then SPF was ground and filtered to 2 mm size.

2.4 Cellulose extraction

The two main processes carried out to extract the cellulose fibres from the SPF were delignification and mercerization. Lignin was removed from SPF to get the holocellulose through chlorination and bleaching processes according to ASTM D1104-56 (1978). According to ASTM D1103-60 (1977), α-cellulose is produced through further treatment of holocellulose [72].

2.5 Separation of sugar palm nanocrystalline celluloses

Acid hydrolysis method was used to prepare sugar palm nanocrystalline celluloses (SPNCCs). Cellulose was mixed with the aqueous H_2SO_4 (60 wt%) at 5:100 ratio (wt%) and was stirred at 45°C for 45 min at a rotation speed of 1,200 rpm using a mechanical stirrer. Then, washing process took place for the hydrolysed cellulose for four repetitions through centrifugation (6,000 rpm, 20 min and 20°C) to remove all the leftover H_2SO_4. Next, the cellulose was dialysed using distilled water until neutral pH (6.5–7) was reached. Sonication process was performed to the cellulose using a sonicator for 30 min. The final cellulose was freeze dried and kept in cool place before further analysis and application as starch film’s reinforcement.

2.6 Preparation of SPS/SPNCCs-incorporated CEO nanocomposite films

Composite films from SPNCCs were prepared by solution casting technique. Firstly, all materials and solutions such as sorbitol, glycerol, starch (10 g), SPNCCs (0.05 g), cinnamon EOs, tween 80 and distilled water (190 mL) were prepared. All the solutions were mixed together by simple stirring, and the mixture was put into the sonicator to produce a homogenous nanocomposite film. About 190 mL of distilled water was added to the prepared solution with different concentrations of cinnamon EOs, as presented in Table 1, and the solutions were sonicated for 30 min. After sonication process, 10 g of SPS solution was poured and stirred for 20 min at 1,000 rpm at 85°C in a disperser to gelatinize the starch. This process is vital to assure that homogenous dispersion happened simultaneously and the starch granules were uniformly degraded. The ratio of plasticizers used was 1:1, which was about 1.5 wt% of the plasticizers in this experiment. The film-forming process was performed under vacuum condition so that the air bubbles were removed. Then, the solution-casting process was performed by pouring 45 g of the suspension into a 15 cm diameter petri dish. The setup was then kept in an oven for overnight drying at 40°C. Next, the film was removed from the petri dish and kept in a controlled room at 23 ± 2°C and relative humidity of 53 ± 1% for 7 days.

2.7 Scanning electron microscopy

A field emission electron microscope (FEI NOVA NanoSEM 230, Czech Republic) was used to investigate the morphology of the films. All the samples were gold-coated by using a plasma argon (sputter coater K575X; Edwards Limited, Crawley, United Kingdom) to prevent unwanted charging. The SEM testing was conducted at a 3 kV acceleration voltage.

Table 1: Denotations of SPS/SPNCC-incorporated CEO nanocomposite films

Denotation of the films	Formulation		
	Sorbitol (wt%)	Glycerol (wt%)	CEO (wt%)
1	1.5	1.5	0
2	1.5	1.5	0.8
3	1.5	1.5	1.2
4	1.5	1.5	1.6
5	1.5	1.5	2.0

2.8 Thermogravimetric analysis

The thermal stability of the samples was investigated using a Q series thermal analysis machine (TA Instruments, New Castle, DE, USA). The process was conducted in an aluminium vessel in dynamic nitrogen environment. The heating rate was fixed at 10°C/min to heat the samples in a temperature range of 25–800°C. About 5–15 mg of the sample was put in the metallic vessel and was made ready for the heating process. Then, the temperature was increased for strong heating. The weight loss determination was analysed from the plot of per cent of mass loss against temperature (thermogravimetric analysis [TGA] curve).

2.9 Flammability test

Flammability test was carried out for all samples via horizontal burning test according to ASTM D635 with slight modification. Samples having dimensions of 120 mm × 10 mm × 0.2 mm were prepared and two lines at 25 and 100 mm from one end of the sample were drawn as the reference marks. Then, fire was ignited with natural gas on one end of the sample. The overall burnt length and the time taken for the flame to spread to the 25 and 100 mm reference marks were noted. The linear rate of the burning samples was calculated using equation (1).

$$V = 60L/t, \tag{1}$$

where V is the linear burning rate (mm/min), L is the burnt length (mm) and t is the time (minutes).

3 Results and discussion

3.1 Microscopy analysis

SPS-based films that are homogeneous, flexible and thin (0.25 mm) were obtained. After drying, they were easily peeled off the petri dish plates. From the observation, all films were yellowish in colour and slightly opaque (Figure 1). The microstructure test was performed to determine the appearance and presence of EO on the morphological surface of the biofilms. Microscopic film analysis is closely linked to the physical properties of the final materials of biofilms, including the visual, mechanical and barrier properties. Figure 2 displays the scanning electron microscopy (SEM) micrographs corresponding to the biofilm surfaces and also the SEM micrographs of active SPS films surface with remarkable changes. The control film surface without CEO exhibited a smooth and uniform texture without traces of starch granular or cracks (Figure 2a). The finding is similar to Ilyas et al. [21,53,60] who also reported the appearance of continuous and smooth microstructure for SPS film. Meanwhile, Acosta et al. [73], who studied the microstructure of starch–gelatin (SG) blend films, revealed that neat SG composite films (without EO) showed heterogeneous structures on their surface. Formation of circles was detected on the surface of the SG films because of the incomplete miscibility of starch and gelatin. It was also associated with the polymer separation phase that gave rise to starch-rich phase that was interpenetrated with a gelatin-rich phase. The incorporation of CEO in SPN/SPS biofilms affected the SPS matrix microstructure, as presented in Figure 2b–e. Adding 0.8, 1.2, 1.6% and 2.0% CEO to the SPN/SPS biofilm yielded uneven surface structures with the increasing surface coarseness with CEO concentration (Figure 2b–f), similar to the findings reported by Choi et al. [74]. Since the surface of the pure film had no micro-porous holes, these phenomena may be caused by the evaporation of essential oils after the drying process [29]. In Figure 2b–f, irregularities were exhibited in cross-section images of the control film that looked like a network of fibres. Homogenous and smooth surface was observed in Figure 2a, without the presence of micro-porous holes. The formation of this microstructure might be associated with the incomplete dissolution/gelatinization of starch granules that are held together by the solubilized-gelatinized starch fraction [29]. Similar results were obtained for different starch-based films as reported in ref. [4,29,43] and other authors. The crosssection of the films revealed the micro-porous spots that emerged as micro-porous holes under the micrographs (Figure 2b–e). The number and size of the microporous holes were found to increase with concentrations of CEO. The appearance of abundant holes corresponded to the position of the oil droplets, where these droplets could partially vaporize on the cryo-fractured surface during SEM analysis because of the high vacuum condition. They were elongated, which, as previously noted, could be explained by their distortion during drying of the film and consecutive packaging of the polymer chains [29]. Similar results were obtained by Peng and Li [75] and Sánchez-González et al. [76], who worked with lion oil and tea tree EO that were added to chitosan films and hydroxypropyl methylcellulose films, respectively.

Figure 1: Transparent film of CEO-reinforced SPN/SPS biocomposite film.

3.2 FTIR spectroscopy analysis

The FTIR spectra of the control SPS/SPNCC nanocomposite film and SPS/SPNCC nanocomposite films incorporated with various concentration of cinnamon EO are shown in Figure 3. The purpose of this analysis was to identify the changes occurred in the chemical structures of the films. Intermolecular rearrangement of polysaccharide chain orientation can be recognized by analysing the spectral differences between the films. The peak at 996 cm^{-1} was associated with C–O bond of C–O–C groups. The high peak that displayed at 2,926 cm^{-1} was assigned to C–H stretching, whereas the low peak at 1,644 cm^{-1} corresponded to C–O stretching. O–H group assigned at the broad peak of the film, which was observed from 3,000 to 3,700 cm^{-1}, is an indicator of hydrophilic properties because of the presence of hydroxyl group in nanofibre. These findings reflect those of Ilyas et al. [77]. The peak at 1,735 cm^{-1} was associated with aldehyde groups [78,79]. According to Salzer et al. [78] and Shankaranarayana et al. [79], CEO contains a high amount of aldehyde. An absorbance peak at 1,733 cm^{-1} appeared in cinnamon EO-incorporated films, and this peak shifted from 1,733 to 1,735 cm^{-1} as the concentration increased [79].

The broad peaks at 3,000–3,500 cm^{-1} were because of the relative peak strength of stretching vibrations for O–H groups in the films. Films incorporated with cinnamon EO had higher amplitude of peaks near 3,266 cm^{-1}. This indicates the presence of EO that contains hydrocarbons in the matrix. As the EO concentrations in the films increased, the peak at wavenumber 3,266 cm^{-1} shifted to 3,282 cm^{-1}. The peak shifting can be attributed to the fact that hydrogen bonding between molecules was partially destroyed [62].

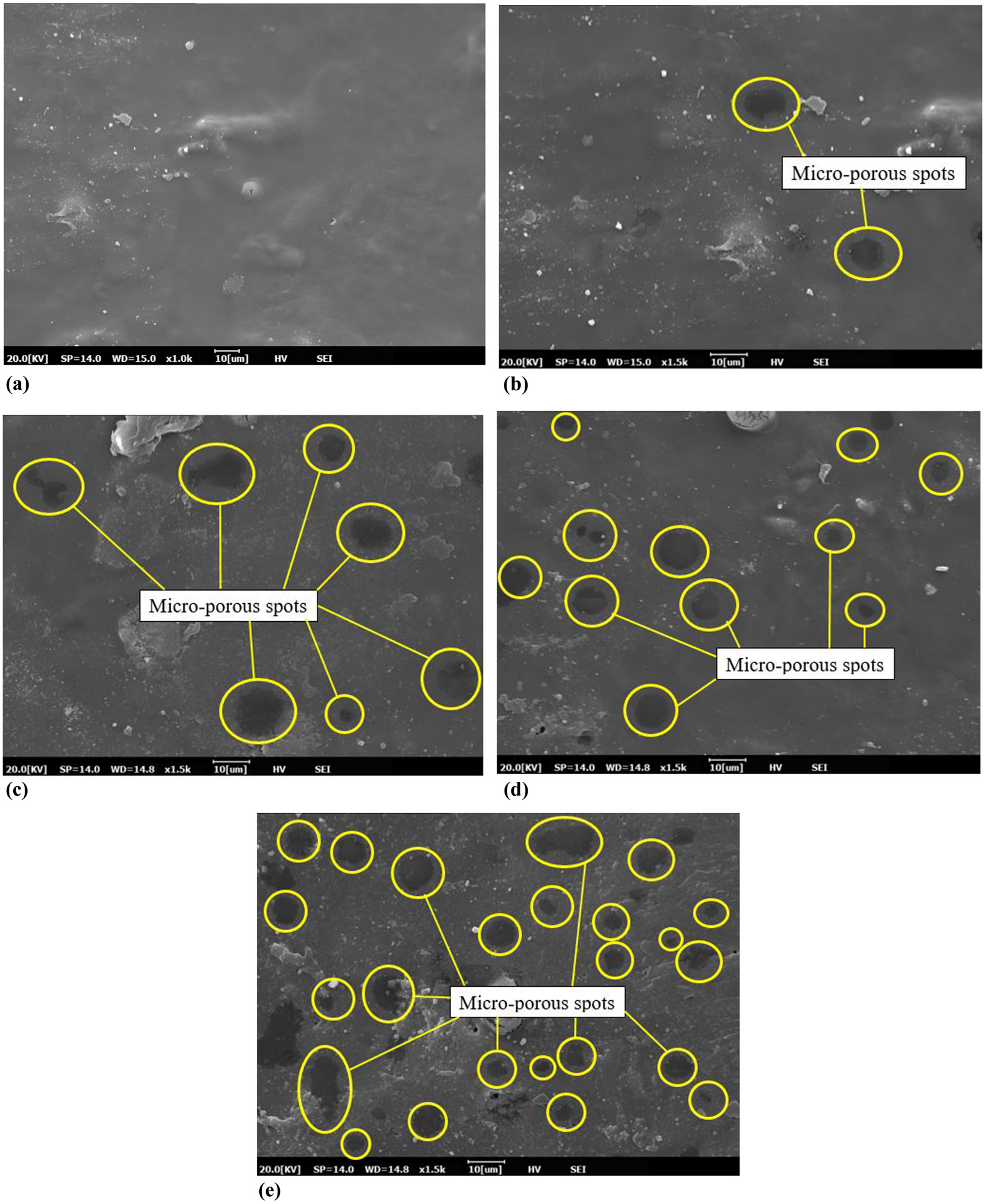

Figure 2: SPN/SPS biocomposite film incorporated with (a) 0%, (b) 0.8%, (c) 1.2%, (d) 1.6 and (e) 2.0% of CEO.

The differences in absorption peaks for films incorporated with cinnamon EO indicated that the molecular structure had been altered [80]. It is probably because of the formation of hydrogen bonding between hydroxyl group and cinnamon EO compounds. The peak shifting from lower to higher wavenumber was largely attributed to

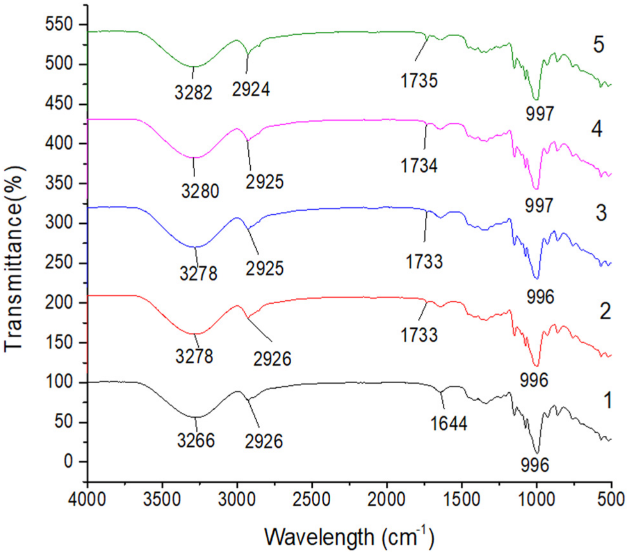

Figure 3: FTIR spectra of cinnamon EO-incorporated SPS/SPNCC nanocomposite films.

different conformations of molecular structures induced by the addition of cinnamon EO.

Furthermore, the peak at 996 cm^{-1} was shifted to 997 cm^{-1} probably because of new interaction between C–O stretching vibrations for EOs and the starch molecules. The changes in wavenumber were probably caused by the electron jumps between orbitals with well-defined energy differences, and the bonds in the given molecules may bend, rotate or stretch with certain frequencies [81]. The result also showed a similar IR spectrum compared to control film. The increase in EO concentration caused the inter/intra-molecular interaction existed between the starch and the EO via hydrogen bonding or the van der walls force [77]. The band greater than 996 cm^{-1} was attributed to C–O stretching vibrations of polysaccharide compound of starch and glycerol [55]. The results suggested that the addition of cinnamon EO influenced the molecular interaction of polymer chain in the film matrix [43]. As the substances were mixed, physical blends against chemical reactions caused changes in the spectral peak [82]. It is generally known that hydrogen bonding will increase the wavenumber of bending vibrations but decrease the wavenumber of stretching vibrations [81]. The FTIR results indicate the existence of the interaction between SPS and cinnamon EO.

3.3 Thermogravimetric analysis

The effect of the CEO on the thermal stability of the composites was examined by TGA and derivative thermogravimetry (DTG) curves, and the results are presented in Figure 4. Thermal analysis test allows food manufacturers to optimize production, storage, transportation, cooking and consumption quality of the food. TGA techniques continually examined the mass of a sample as it is heated or cooled at a regulated rate or is held at a selected temperature for a set length of time. TGA is useful for tracking processes that involve a shift in the mass of a food packaging materials, and applications typically involve evaporation, desorption and vaporization behaviour, as well as thermal stability, decomposition and compositional investigation. Besides that, the TGA indicates maximum temperature for the food packaging to package food. From the thermograms, it can be observed that both the SPN/SPS and the CEO/SPN/SPS biocomposite films degraded in similar steps, as shown in Figure 4. In the TGA and DTG curves of control SPS, multi-step thermal decomposition events have been observed. Similar results with two-step thermal decompositions in majority of starch-based films were obtained and reported in the literatures [21,54]. Apart from that, the mass loss of the sample was decreased with the increment of CEO loading, as stated in Table 2. The first degradation step represented by small peak DTG curve occurred below 100°C, approximately at $T_{\max} \approx 69$–90°C. This was because of the evaporation of the broken inter- and intra-molecular hydrogen bonds followed by loss of water. Besides that, mass loss at this temperature range might also be because of the removal of water or evaporation of low molecular weight compounds and loosely bonded water in the sample. As more CEO added in the film solution, the amount of water decreased. The same phenomenon was observed to occur for the other film samples, including control SPS, as can be clearly seen in their DTG curves.

A prolonged pyrolysis process took place at 200–370°C and reached maximum mass loss at 296.25°C. From Figure 4, it was obvious that control film was less stable compared to other films, having the lowest decomposition temperature of 289°C compared to CEO-reinforced SPN/SPS biocomposites. The weight loss might be associated with the vaporization and decomposition (oxidative and thermal) of bio-polymers and elimination of glycerol and volatile products [54,83]. In the TGA thermogram of CEO/SPN/SPS, the weight loss was initiated at around 40°C, showing an onset point at around 80°C, indicating that CEO has volatile nature. The flash point of cinnamon oils is between 38 and 60°C.

The thermal stability of polymer networks was shown to be improved by the CEO, as control films displayed lower weight loss temperature ($T_{\max} \approx 289.18$°C) than CEO films. The thermal stability of the films increased with the loading of CEO. The lowest loading (CEO sample 1) resulted in lower thermal stability and was increased with increased

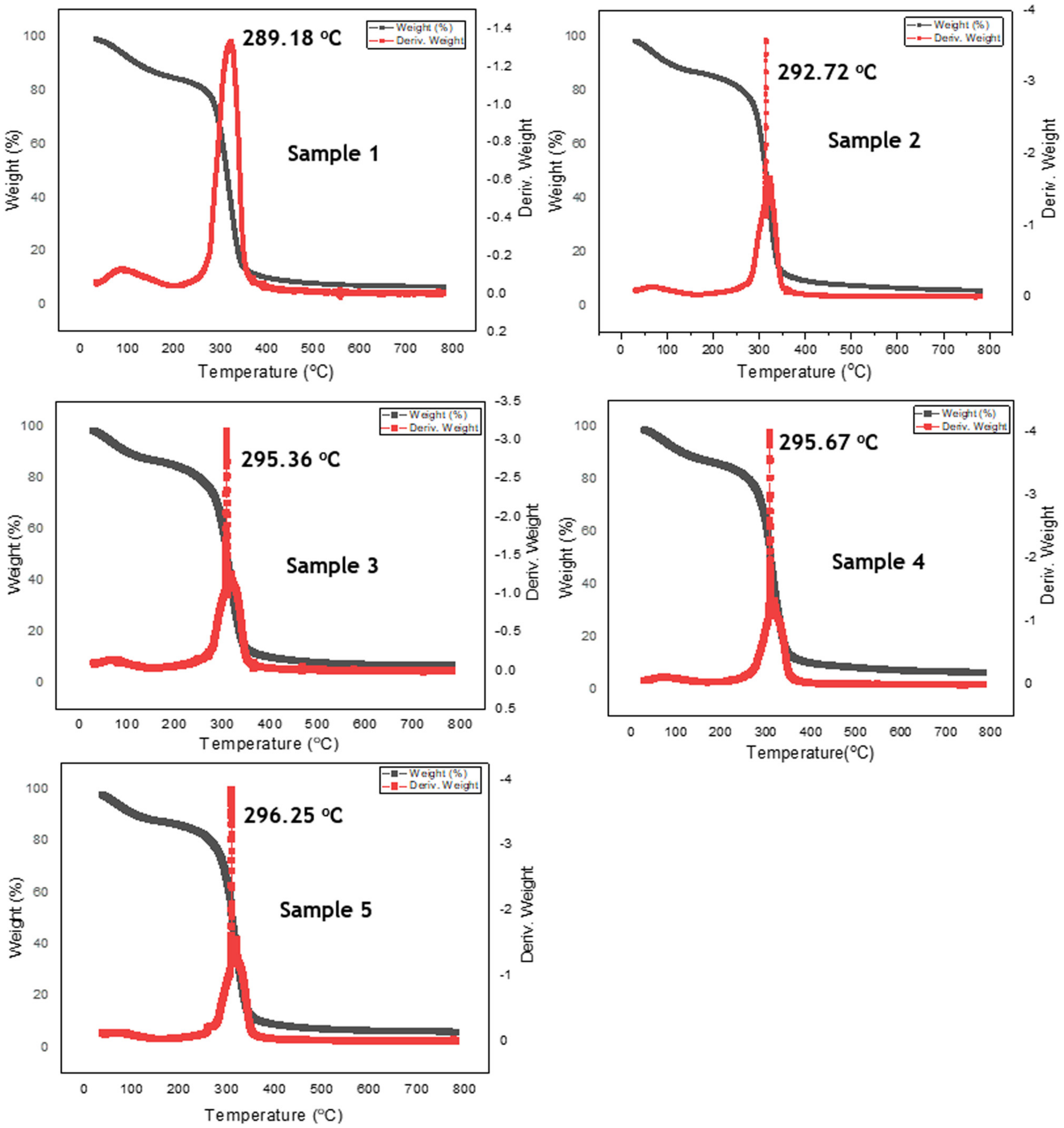

Figure 4: TGA and DTG curves of SPN/SPS and CEO/SPN/SPS biocomposite films in different concentrations.

loading (CEO samples 2 and 3). This phenomenon could be associated with polymer structure rearrangement following the addition of higher CEO concentrations. It was observed that the polymer structure changed with the smallest quantity of CEO, but upon adding more CEO, the thermal stability increased. This was related to the rise in crystallinity indexes when the CEO concentration increased, as described by Noshirvani et al. [84]. Ma et al. [85], who observed the same pattern, concluded that the use of olive oil in the film matrix has led to an increased gelatine transition helix–coil temperature. They decided that olive oil created non-miscible emulsified phases in the films. The thermal stability is also parallel to the degree of crystallinity. Higher degree of crystallinity resulted in higher thermal stability [21,54,86]. This was ascribed to lower neat SPS values similar to the findings also observed in other works [87,88].

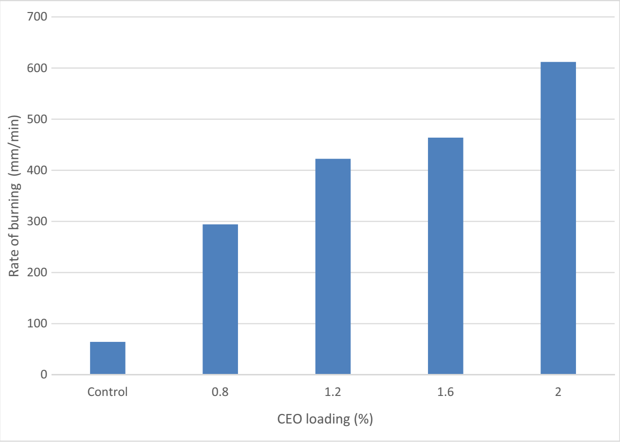

Figure 5: The effect of different CEO loadings on the flammability of SPN/SPS biocomposite film.

3.4 Flammability analysis

Plastics have become one of the most useful materials known to humankind. Because of their chemical composition, plastics can easily ignite when exposed to sufficient heat in the presence of oxygen. Because of the rate of burning for plastics, considerable work has been directed to study and minimize the flammability issues of these materials, like the addition of flame retardant chemicals to prevent or minimize the combustion of these materials. This test is done to classify and measure burning characteristics of plastics. Figure 5 shows the effect of varying CEO loadings on the flammability of SPN/SPS composites, as indicated by the linear burning rate. The burning rate was increased with CEO concentration. With 2% CEO, the SPN/SPS biocomposite displayed the shortest burnout time compared to the unfilled composites and then had the highest linear burning rate. Most EOs, including CEO, are extremely flammable [89–91]. CEO possesses complex mixture comprising over 300 different compounds [92,93]. CEO consists of volatile organic compounds, typically having molecular weight below 300 [94,95]. The volatile compounds are from different chemical classes, such as ethers and oxide, ketones, alcohols, amines, aldehydes, phenols, esters and amides. The flash point for CEO is 71°C, which explained the flammability characteristic of the film composites. During the burning process, the CEO formed a non-protective oil layer on the surface of the matrix, serving as an oxygen conductor and permitting heat to penetrate the matrix [96,97]. Therefore, the quantity of decomposed volatiles that escaped the interior polymer matrix was increased, resulting in a shorter burning time and thus increasing the linear burning rate.

4 Conclusions

The thermal stability of the CEO/SPN/SPS biopolymer composites was remarkably improved with increasing CEO loadings (sample 5, $T_{\max}$ = 296.25°C) compared to the thermal stability of the unfilled compound (sample 1, $T_{\max}$ = 289.18°C). The control film surface without CEO displayed a uniform and smooth texture with no traces of starch granular or crack. The presence of the CEO in the SPN/SPS biofilms affected the microstructures of the SPS matrix, resulting in rough surface structures and increased surface coarseness with CEO concentrations. The appearance of numerous holes was consistent with the position of oil droplets, where the number of holes rose as EO loading increased. Because of the high vacuum situation, this can partially evaporate on the cryo-fractured surface during SEM study. The linear burning rate was increased with the CEO concentration. The SPN/SPS biocomposite with 2% CEO showed the shortest burnout time compared to the unfilled composites and hence had the fastest linear burning rate.

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