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Research article

Effect of corn husk fibre loading on thermal and biodegradable properties of kenaf/cornhusk fibre reinforced corn starch-based hybrid composites

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ABSTRACT

This paper documents the thermal and biodegradation behaviour of kenaf/cornhusk fiber reinforced corn starch-based hybrid composites film (CS/K–CH) produced by solution casting method. To develop both components as biodegradable hybrid composite, this research used corn starch as matrix, kenaf fiber and cornhusk fibre as a filler. Changes in physical structure and weight from the soil burial test were measured using Mettler Toledo digital balance ME. Films produced from physically blended corn starch reinforced kenaf biocomposites films (CS/K) biocomposite film had faster biodegradation and lost 96.18% of weight within 10 days compared with corn starch hybrid composites that only lost 83.82% of total weight. It was observed that the control film, CS/K biocomposite film was completely degraded after 10 days, meanwhile it took 12 days for hybrid composite films to be fully degrade. The thermal properties such as TGA and DTG were also measured. Addition of corn husk fiber significantly improve the film's thermal properties. Glass transition temperatures of corn starch hybrid films were significantly lowered when cornhusk compositions were increased from 0.2% wt to 0.8% wt. Importantly, the current work has demonstrated that hybrid films made of corn starch can be a suitable biodegradable material for substitute synthetic plastic.

1. Introduction

The widespread usage of face masks and protective personal equipment (PPE) kits during the COVID-19 pandemic has resulted in an increase in plastic waste pollution. Theses waste are often thrown abandonly into streets, rivers, seas and all over the places of the environment. Plastic waste contamination currently has a harmful impact on the environment due to their long process in

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decomposition. The strong efforts to control the spread of COVID-19 result in a major slowdown in economic activity, which has a negative impact on the environment by lowering greenhouse gas (GHG) emissions, notably lower atmospheric CO_2 levels [1].

Plastics are quickly becoming the primary material accountable for land and marine pollution. Plastics are also used in the production of numerous products today due to its low cost, light weight, and excellent durability [2–5]. Despite the increased need for plastics, issue due to inappropriate disposal of plastic waste arises [6,7]. The widespread usage of plastic has bring the world to a significant environmental problem [8–11]. It is practically hard to keep track of the growing number of plastic wastes. For example, due to inefficient waste management, the maritime system gets an estimated 4.8–12.7 $\times 10^6$ tonnes of plastic waste per year [6,12,13].

Kenaf and corn fibres have long been utilised as reinforcing fibres with numerous applications [14–19]. Table 1 shows the chemical composition and physical properties of kenaf and corn husk fiber. Kenaf fibre has been explored to replace the use of wood in the pulp and paper sectors in order to promote to forest preservation. Furthermore, in the composites market, kenaf fibre has been utilised as reinforcement with a variety of matrices for a wide range of applications. For example, nonwoven kenaf mats have a long history of use in fiberboards, automotive parts, and textiles. As an alternative to wood, Hidayat et al. [20] created a polylactic acid/kenaf composite with characteristics similar to particle board. For high-performance biodegradable polymer composites, kenaf fibre can be a good reinforcement candidate [21]. In recent technological advances, it has been widely used in building platforms, automotives parts, safety equipments, bridges and low-cost houses [22]. Samaei et al. [23] examined how alkaline treatment affected the kenaf natural fibres' acoustical, morphological, tensile, and thermal characteristics. Their results are consistent with the hypothesis that kenaf fibres' tensile, acoustical, thermal, and morphological properties are improved by the alkaline treatment. These fibres can be used in the building and automotive industries to reduce energy consumption by reducing the rate of heat transfer. Ibrahim et al. [24] reported on a study that looked at the possibility of employing multiscale corn husk fibre as a reinforcing filler in biocomposites made of cornstarch. The results showed that adding cornhusk fibre generally improved the performance of the composite films. The density had significantly decreased, the moisture level had increased, and the soil burial assessment had revealed less biodegradation resistance. The enhanced tensile strength and young modulus, thermal stability as well as the crystallinity index were all a result of the morphological structure and outstanding compatibility between the reinforcement and matrix [25–27].

Hybrid composites are novel engineering materials, that incorporate two or more constituents (phases) that are mechanically and physically distinct [28,29]. Hybrid composites created by combining two or more reinforcing fibres into a single matrix [30]. There aren't many hybrid composites that are reinforced exclusively with natural fibres, but they could be useful in a variety of applications [31]. Chandramohan et al. [32] has researched the potential of hybrid roselle/sisal fibre reinforced polyester composites in automotive applications such rear-view mirrors and signal covers for car compartments. Chandramohan et al. [33] revealed the results of another investigation comparing roselle/sisal, sisal/banana and roselle/banana, which then concluded that the roselle/sisal combination makes one of the best materials to use for the fabrication of bone implants. Yusoff et al. [34] created three different kinds of hybrid green composites using a polylactic acid (PLA) polymer matrix composites made of kenaf-coir and PLA, bamboo-coir and PLA, and kenaf-bamboo coir and PLA. In terms of tensile and elastic modulus, the combination of kenaf-bamboo coir demonstrated the best hybridization result. Its performance was on par with that of composites made of glass and plant fibres.

Maslinda et al. [35] investigated the kenaf/jute and kenaf/hemp reinforced epoxy composites, susceptibility to moisture absorption. According to their findings, hybrid composites outperformed single system composites in both dry and wet situations in terms of tensile and flexural qualities. Jawaid et al. [36] discussed the hybridization of non-woven jute fibres and empty fruit bunches (EFB) from oil palm trees. After being hybridised with jute, EFB's thermal characteristics were enhanced. Additionally, the dynamic mechanical characteristics of the hybrid composites were improved. Similar result from Ref. [37] that studies on seaweed/sugar palm fibre reinforced thermoplastic sugar palm Starch/Agar hybrid composites. The results revealed that seaweed and sugar palm fibre were compatible and increasing intermolecular hydrogen bonding in the composites. Mechanical properties were also improved, and the impact resistance was reduced with the addition of SPF. Another study by Bachtiar et al. [38] in determining the tensile properties of hybrid sugar palm/kenaf fibre reinforced polypropylene composites. The researchers came to the conclusion that the hybrid composites with more kenaf fibres exhibit higher tensile strengths than the composites with more sugar palm fibres.

In this study, a novel hybrid biocomposite comprising cornhusk and kenaf fibers to reinforced cornstarch matrix were conducted. The main objective of the current contribution is to investigate the effect of multi-scales of corn husk fiber contents on the thermal and biodegradable properties of the cornstarch hybrid composite. The hybrid biocomposites was fabricated using traditional solution casting method with cornhusk loading from 0.2 % wt to 0.8 % wt.

Table 1
Chemical composition and physical properties of kenaf and corn husk fiber.

No	Content	Kenaf	Cornhusk
1	Moisture (% wt)	8.00–12.00	7.81
2	Lignin (% wt)	2.80	4.03
3	Density (g/cm^3)	1.40	1.49
4	Crystallinity (% wt)	23.80	30.10
5	Cellulose (% wt)	69.20	45.70
6	Hemicellulose (% wt)	27.20	35.80
7	Ash (% wt)	0.80	0.36

2. Materials and methods

2.1. Materials

Thye Huat Chan Sdn. Bhd. in Sungai Buloh, Selangor, Malaysia, is where the commercial Star Brand cornstarch (CS) was acquired. Cornhusk fibre (CHF) was bought at a night market in Serdang, Selangor, while native kenaf fibre (KF) was collected from a nearby kenaf farm in Lembaga Kenaf dan Tembakau Negara, Kota Bharu, Kelantan. Both materials were cleaned, dehydrated, ground using Pulverisette 19 and graded in a sieving machine. The starch and fiber were graded in the sieve machine Matest A060-01 to a size of 0.25 mm.

2.2. Preparation of hybrid films

The preparation of corn starch-based hybrid films was performed using the conventional solution casting procedures according to Hazrol et al. [39]. Sorbitol plasticizer (3g), 10g of pure CS and kenaf fiber 6 wt% based of dry starch. As a hybridising agent, the CHF was utilised with different loadings of (2 wt% - 8 wt%) dry starch. The heating process was kept at 85±∘C for an additional 20 min before casting on a thermal casting dish. The casting dish was then weighted at 45g to preserve film thickness consistency. The casting dish was then dehydrated in an air circulation oven for 15 h at 65 °C.

2.3. Soil burial test

The biodegradation experiments were carried out using ASTM G21-70 on all samples following the method described by these researchers [24,40,41]. Experiment was conducted and arranged as in Fig. 1. Before being buried in the soil, the samples were wrapped in an iron mesh to prevent degradation while allowing moisture and microbes access. All samples underwent drying for a straight 24 h at 105 °C and the weights were measured and recorded to determine the initial weight, (M_i). Then the sample was buried 5 cm deep in damp soil. The soil nutrient content contains 45–55% moisture, pH of 5.5–6.5, 30–40% of carbon, 1–1.5% of nitrogen, 1500–2000 ppm of phosphate, 1500–2000 ppm of potassium, 2000–3000 ppm of magnesium and 2000–7000 ppm of calcium. The samples were frequently moistened using distilled water to maintain the humidity of the soil. The sample evaluation was carried out in triplicate by taking soil samples at same times and gently rubbing them with a clean dry cloth. After that, the samples were dehydrated for 6 h at 105 °C and reweighed (W_f). All samples were soil-buried for prespecified durations of 1–14 days. Every day, the obtained samples were subjected to degradation analysis was determined using Eq. (1):

$$Weight\ loss\ (\%) = \frac{(W_i - W_f)}{W_i} \times 100 \quad (1)$$

2.4. TGA

Thermo-gravimetric analyzer Mettler Toledo AG, Analytical (Schwerzenbach, Switzerland) was used to investigate the specimens' thermal behaviour. This test was conducted in a dynamic nitrogen atmosphere. Temperatures ranging from 25 °C to 500 °C at constant heating rate (10 °C/min-1) were used throughout the tests. Each specimen, comprised of 5–15 mg composite, was placed in a sample pan and cooked. The weight loss % vs temperature was included in the created TGA curve.

Fig. 1. Soil burial testing.

2.5. Statistical analysis

The analysis of variance (ANOVA) on the acquired experimental results was performed using SPSS software. Duncan's test was performed to compare means at a significance level of 0.05 ($p \leq 0.05$).

3. Results and discussion

3.1. Degradation test

The research on biodegradation characteristics is essential for the use of biodegradable hybrid composite films in the environment. Soil burial tests were carried out in this study for a hybrid film containing 0.2 to 0.8 wt% cornhusk fiber. In a nutshell, biodegradation is the breakdown of chemicals by the action of fungi, bacteria, and microorganisms, as well as other biological processes. When these microbial species came into touch with the biodegradable polymer, the degradation of the polymer began [42]. These microbial organisms changed the polymer by an enzymatic or metabolic process that dissolved the polymers into smaller molecules with reduced average molecular weight. As a result, this facilitated material degradation in the surrounding environment [43].

Fig. 2 depicts the weight loss of the CS/K and CS-K/CHF hybrid composites as a function of biodegradation time following biodegradation analyses. At the end of 10 days, the weight of neat CS/K had lost 96.18%, whereas the CS-K/CHF 0.8 wt % hybrid composite had lost 83.82%. The average degradation rate were 9.62% per day and 8.38% per day, respectively for the CS/K and CS-K/CHF 0.8 wt %. It was observed that the weight loss CS/K biocomposites was higher compared to CS-K/CHF hybrid composite at any given time points. The control CS/K film disintegrated almost completely in 10 days, whereas hybrid composite films took 12 days to totally degrade. When compared to the hybrid composite films, the weight loss of the control matrix was greater. Water absorption of CS-K/CHF in hybrid composite films may be attributed to this condition.

The weight loss for the control film CS/K, was greater than that of the CS-K/CHF hybrid composite on day 1 and began to decrease consistently for the subsequent degrading tests. This could be attributed to the physical features of CS/K, as CS/K absorbed more water than CS-K/CHF films on the first day, leaving it more vulnerable to microbial attack [37,44]. These microorganisms attacked the CS/K when a moisture was present [45]. This can be explained by the water absorption characteristics of biocomposite films. For example, the water absorption for CS/K was around 84.00%, but the water absorption for the CS-K/CHF 0.8% hybrid composite film was 69.33%. The hydrophilic nature of kenaf and cornhusk may be responsible for the acceleration growth of microorganism [24,46]. Higher starch content samples would therefore possess greater biodegradability characteristics.

The concentration of CHF had an impact on the film's deterioration as well. Films with greater CHF concentrations deteriorated more slowly than those with lower CHF concentrations. This was most likely driven by the increased concentration of CHF, which was linked to a higher degree of crystallinity and made matrix degradation harder. The physicochemical properties of the substrate, such as polymerization of cellulose and the degree of crystallinity, which is a significant structural parameter for cellulose, have a significant impact on the ability of cellulolytic bacteria to destroy cellulose [47].

Higher crystallinity in CS-K/CHF than CS/K gave it greater resistance to microbial organism attacks than starch in comparison to control film CS/K. The weight loss between the CS/K and CS-K/CHF hybrid composites varied as a result of the microbial organism attacks that started with starch. In the case of CS-K/CHF hybrid composites, the accessibility of the microbial organism to the matrix was favored by the amorphous area [48].

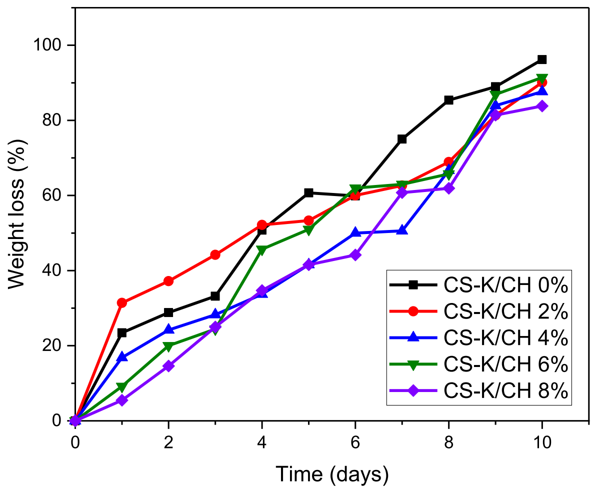

Fig. 2. Soil burial test of CS-K/CHF hybrid composite.

3.2. Thermal properties

Fig. 3 (a, b) shows the TGA and DTG curves results of CS-K/CHF hybrid composites with multiple CHF loading. Fig. 3 also indicated four distinct stages of weight reduction, which illustrated by significant peaks on the DTG curve (Fig. 3b). The thermal characteristics of materials were determined using TGA and DTG curves as illustrated in Fig. 3a and b, respectively. This is to calculate the decomposition temperatures and the proportion of material remnants following the maximum degradation rate. Each event is connected to a distinct weight loss in the TGA graph and a conspicuous peak in the DTG curve. The water molecules being eliminated via evaporation and the dehydroxylation process. This initial weight loss happened at temperatures lower than 100 °C [49–51]. Table 2 depicts the effect of cornhusk fiber and concentrations on T_g of corn starch hybrid composites film. In accordance with the moisture content results in Table 2, the rate of weight loss during heating was greater for samples that contained more moisture.

The second weight loss occurred during the heating process, between 150 °C and 170 °C (as shown in Table 3), was caused by the thermal decomposition of sorbitol particles along with remaining water fragments. Most plasticizers start to evaporate at temperatures above 150 °C [52]. The third heat activity resulted in weight loss due to the hydrolysis of starch's amylopectin [53]. The forth weight loss occurred during the heating process, between 370 °C and 390 °C (as shown in Table 3), was caused by the thermal decomposition of natural fiber. The primary components of reinforcing fibers hemi-cellulose, cellulose, and lignin were decomposed at the fastest rate. Lomelí-Ramírez et al. [54] claims that, depending on the plant species and the percentages of fabric components, thermal decomposition begins with the decay of hemicellulose in the (200°C-260 °C) range, cellulose in the (240°C-350 °C) range, and lignin in the (280°C-500 °C) range for all lignocellulosic plant fibres. After lignin decomposes completely, inorganic materials like silica (silicon dioxide, SiO_2), which might be thought of as a char, are all that's left (mass residues).

In summary, the hybrid CHF-films demonstrates close onset degradation temperatures ranging from 298 °C to 300 °C which can be seen in Fig. 3a, as well in Fig. 3b. Table 3 also displayed the degradation temperature of CS-K/CHF hybrid composites. It was significantly lower than the 302 °C threshold for maximal deterioration of the CS/K composite film. In a similar manner, after completely decomposing the hybrid composite, there was a slightly increase in the mass of the residues and a significant rise in the weight loss. Evidence like this suggests that the thermal stability of CS-based hybrid composites was compromised by the addition of

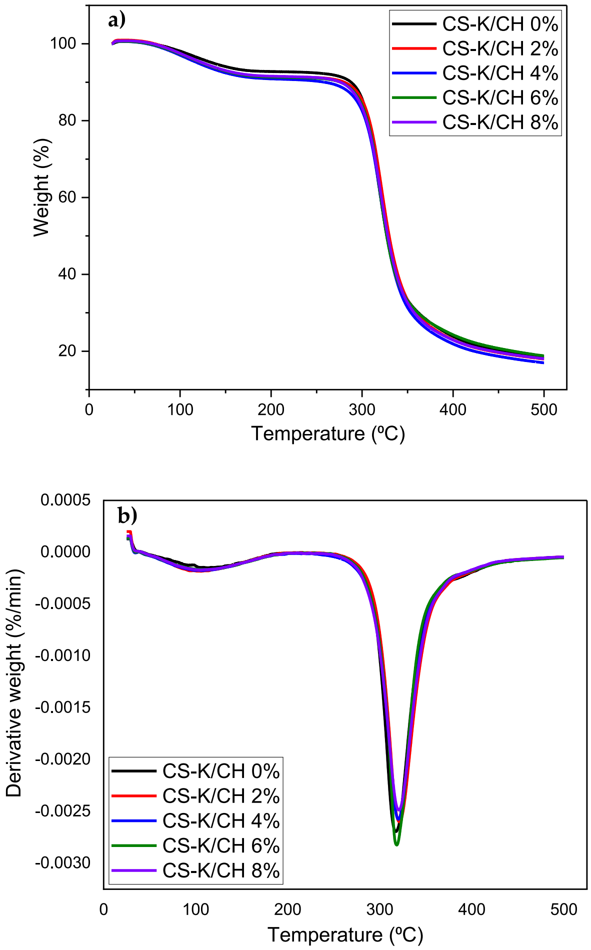

Fig. 3. Thermal analysis of CS-KF/CHF hybrid composite. (a) TGA and (b) DTG.

Table 2
Effect of cornhusk fiber and concentrations on T_g of corn starch hybrid composites film.

Films	Moisture content (%)	T_i	T_f	T_g (∘C)	$t_{5\ days}$ (wt%)	$t_{10\ days}$ (wt%)
CS/K	5.99 ± 2	290.86	368.31	77.45	60.71	96.18
CS-K/CHF2%	9.36 ± 1	293.98	370.94	76.96	53.33	90.12
CS-K/CHF4%	7.00 ± 1	291.34	365.20	73.86	41.57	87.64
CS-K/CHF6%	6.80 ± 1	290.86	364.12	73.26	50.98	91.42
CS-K/CHF8%	5.98 ± 1	293.98	362.56	68.58	41.56	83.82

Table 3
Degradation temperature of CS-K/CHF hybrid composites.

Film sample	Onset degradation temperature (°C)				DTG peak temperature (°C)	Mass residue (%)	Weight loss (%)
	Phase 1 (Moisture)	Phase 2 (Plasticizer)	Phase 3 (Starch)	Phase 4 (Fibers)			
CS/K	80.57	153.92	287.51	386.74	317.83	18.47	81.94
CS-K/CHF2%	79.48	157.76	289.91	385.15	321.15	17.98	82.89
CS-K/CHF4%	64.51	159.55	290.86	372.50	318.33	16.98	83.43
CS-K/CHF6%	55.42	162.51	291.82	379.08	320.31	18.79	81.70
CS-K/CHF8%	55.66	166.74	292.94	376.33	320.83	18.07	82.50

CHF as a hybridising agent. Similar result from Izwan et al. [55] who studies on benzoylation treated sugar palm/kenaf fiber reinforced polypropylene hybrid composites. After incorporation with treated kenaf and sugar palm fiber, thermal properties of the hybrid composites were improved. Ghori et al. [56] who studied on date palm/kenaf fiber-reinforced epoxy hybrid composites also mentioned that addition of kenaf fiber has positively impacted on date palm fiber to improve the mechanical and thermal properties.

4. Conclusion

Innovative hybrid bio-composite films made by solution casting and dehydrating a mixture of maize starch (CS) and the fibrous remnants of kenaf fibre (KF) and cornhusk fibre (CHF). The experimental and characterization results demonstrated that the hybridization technique significantly enhanced the performance of the composite film. The thermal stability of the manufactured composites was slightly increased by the addition of CHF, de-spite the fact that the physical qualities were improved. The T_g value associated with the starch phase decreases as CHF concentration rises, this suggests that the presence of CHF increased the adaptability of the starch molecular chains. Although water has a strengthening impact on fibres, it was hypothesised that the moisture content of hybrid composite films increased the polymer's mobility between chains. The soil burial assessment revealed that fibre integration accelerated the biodegradation process because of the fiber's high hydrophilicity. It should also be highlighted that the molecules of kenaf and corn husk utilised as reinforcement in the current study have not undergone any chemical processing or modification, leading to the creation of ecologically friendly and economically advantageous materials. The hybrid composite film disintegrated completely in about 12 days as a result. In light of this, our study demonstrated the enormous potential of CS-K/CHF hybrid composite films for biodegradable packaging applications, practical applications and future developments.

Author contribution statement

M.D Hazrol: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

Mohd Sapuan Salit: Conceived and designed the experiments; Contributed reagents, materials, analysis tools or data.

R.A. Ilyas: Conceived and designed the experiments; Analyzed and interpreted the data; Wrote the paper.

E.S. Zainudin, M.Y.M. Zuhri, N.I Abdul Wahab: Conceived and designed the experiments.

Data availability statement

Data included in article/supplementary material/referenced in article.

Declaration of interest's statement

The authors declare the following personal relationships which may be considered as potential competing interests: R.A. Ilyas holds a position as one of the editorial board for Heliyon.

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