# Morphology and selected properties of kenaf fiber/cornhusk reinforced corn starch hybrid biocomposites

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**Abstract:** The study examined the effect of the cornhusk fibers (CHF) content on the structure, physical, mechanical and water barrier properties of composites obtained on the basis of corn starch (CS) and kenaf fiber (KF), used in the amount of 6 wt%. Sorbitol in the amount of 30 wt% was used as a plasticizer. Hybrid biocomposite films were obtained using the solution casting technique. The addition of CHF reduced the water absorption and solubility of the film in water. Increasing the CHF content in the composite resulted in greater film thickness, weight and density. Thanks to good biocompatibility (FESEM) the composites showed higher tensile strength and Young's modulus. However, elongation at break was lower. FTIR results confirmed the intermolecular hydrogen bonding between the matrix and the fibers. The best properties were obtained at 6 wt% CHF content.

Keywords: corn starch, cornhusk fiber, kenaf fiber, hybrid composite, biodegradable films.

# Struktura i wybrane właściwości hybrydowych biokompozytów skrobi kukurydzianej wzmocnionej włóknem kenaf i włóknem z łusek kukurydzy

**Streszczenie**: W pracy zbadano wpływ zawartości włókien z łusek kukurydzy (CHF) na strukturę, właściwości fizyczne, mechaniczne i barierowe dla wody kompozytów otrzymanych na bazie skrobi kukurydzianej (CS) i włókna kenaf (KF), stosowanego w ilości 6% mas. Jako plastyfikator użyto sorbitol w ilości 30% mas. Folie biokompozytów hybrydowych otrzymywano techniką odlewania z roztworu. Dodatek CHF zmniejszał absorpcję wody i rozpuszczalność folii w wodzie. Zwiększenie zawartości CHF w kompozycie skutkowało większą grubością, gramaturą i gęstością folii. Dzięki dobrej biozgodności (FESEM) kompozyty wykazały większą wytrzymałość na rozciąganie oraz moduł Younga. Jednak wydłużenie przy zerwaniu było mniejsze. Wyniki FTIR potwierdziły obecność międzycząsteczkowych wiązań wodorowych między osnową polimerową i włóknami. Najlepsze właściwości uzyskano przy zawartości 6% mas. CHF.

**Słowa kluczowe**: skrobia kukurydziana, włókno z łuski kukurydzy, włókno kenaf, kompozyt hybrydowy, folie biodegradowalne.

In 2021, European production and consumption of plastic globally were 57.2 million tons [1, 2]. The world dumps 381 million tons of plastic trash annually, which is predicted to triple by 2034. The most frequently utilized plastic materials are polypropylene (PP), polyethylene (PE), polyurethane (PU), poly(vinyl chloride) (PVC), polystyrene (PS), poly(ethylene terephthalate) (PET), and phenolic resin, with PP and PE being broadly used polymers in everyday plastic goods especially disposable products like plastic packaging, sterile medical equipment, constructions, and recyclable plastic bottles [3–5].

Synthetic packaging materials pollution has contributed to the problem of environmental degradation, necessitating the development of eco-friendly packaging goods at a comparable cost in order to save our planet

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[6, 7]. As a result, natural biopolymers, such as poly(lactic acid) (PLA), poly(hydroxyalkanoate) (PHA), protein, and starch-based plastics, are being investigated as potential alternatives for non-biodegradable plastics [8–10].

Starch films have a long history of usage in both food and non-food applications. Due to their poor mechanical properties, specifically tensile strength, improving the characteristics of starch-based materials is a big challenge. To improve these mechanical properties, a variety of blending and compositing techniques have been devised, such as mixing with different polymers or reinforcing with natural fillers [11]. Several additive agents have been used to enhance the interaction between starch and other components [12]. Understanding the physicochemical properties of starch, its thermal properties, and the starch formulation development are some of the key elements in resolving these important issues [13].

Recently, increasing emphasis has been placed on the development of polymer biocomposites and the addition of filler reinforcement to starch-based polymers to produce biodegradable packaging [11, 14]. This natural filler reinforcing technique has been proven to be a good way to improve these characteristics while also adding new features to starch-based materials [15]. Due to their distinct environmental advantages, plant-based cellulosic fibers and polymers have recently played significant roles as raw materials in a variety of applications [7]. The significant relationship between cellulose fiber and starch matrix is important for improving mechanical effectiveness and water sensitivity [16, 17]. An effective method for improving the composite films functional qualities is using natural fibers as reinforcing agents while creating starch-based composites. As a result, researchers have carried out several studies to enhance the mechanical properties and water barrier of materials made of thermoplastic starch without sacrificing their biodegradability properties [18, 19].

Corn (or maize) is a useful crop both for humans and animals. Corn is the second most abundant crop produced worldwide annually [20]. In the United States it is estimated that 200 million tons of agricultural residues mainly consist of cornhusks (8%), cobs (15%), leaves (28%), and stalks (48%) that are left on the farms after harvest [21]. Different fibers to reinforce biopolymers, including waste from potato skins, cassava pomace [14], and sugar palms have been used [15, 22]. Starch reinforced with natural fibers showed good interface compatibility. This interaction creates a three-dimensional structure of hydrogen bonds between starch and fiber. Thus, the mechanical properties and water absorption of the biocomposites improve. It has been reported [23] that corn Zea mays fiber contains a significant amount of cellulose, thus serving as a source of cellulose fiber. Additionally, Zea mays tubers have a high starch content and a high green fiber content that can be obtained from bagasse fibers which enhance the water vapor barrier and the mechanical properties of the film.

The main objective of the work was to investigate the effect of the content of corn husk fibers (CHF) on the

structure, physical, mechanical and water barrier properties of hybrid composites based on corn starch. The natural fibers used in this study were not chemically or thermally processed, opening the door for the creation of more environmentally responsible and economical materials. This study is also designed to evaluate the utilization of agricultural leftovers, which are inexpensive and widely available, as a means of minimizing waste disposal and boosting economic growth through the wasteto-wealth transition.

# **EXPERIMENTAL PART**

# Materials

In Sungai Buloh, Selangor, Malaysia, a small factory called Thye Huat Chan Sdn. Bhd. provided the commercialized corn starch for fabrication. Evergreen Engineering & Resources Sdn Bhd, Semenyih, Selangor, Malaysia provided the sorbitol plasticizers. The starch was graded in the sieve machine Matest A060-01 to a size of 0.25 mm. Kenaf fiber is obtained from Lembaga Kenaf dan Tembakau Negara (LKTN), Kota Bharu, Kelantan, Malaysia. The corn husks from the market in Selangor, Malaysia were used. The layers covering the corn cob were cleaned with water, dehydrated, sieved and ground on a Pulverisette 19 machine to a size of 0.25 mm from which the corn husk fiber was extracted. Table 1 shows the chemical composition of both fiber used in this experiment which are kenaf and cornhusk fibers.

T a b l e 1. Chemical composition and physical properties of kenaf and corn husk fibers

Content	Kenaf	Cornhusk	
Moisture, %	8-12	7.81	
Lignin, %	2.8	4.03	
Density, g/cm <sup>3</sup>	1.4	1.49	
Crystallinity, %	23.8	30.1	
Cellulose, %	69.2	45.7	
Hemicellulose, %	27.2	35.8	
Ash, %	0.8	0.36	

#### Preparation of hybrid films

The preparation of corn starch-based hybrid films was made using the conventional solution casting procedures according to Sanyang et al [24]. Sorbitol plasticizer (3 g) was added to a beaker containing 180 ml of distilled water. For 20 min, the beaker was immersed in a hot water bath with steady stirring at a temperature of  $85 \pm 5^{\circ}$ C. Stirring the starch granules resulted in the production of a homogenous solution. Next, 10 g of pure CS was added to the solution and additional 20 minutes of the heating process was continued at the same temperature.



Fig. 1. Corn husk fiber extraction and film fabrication process

Following that, KF was added to the solution and the parameters were selected based on our previous work [25]. The CHF at various loading (2, 3, 6, and 8 wt%) based on dry starch was used as a hybridized agent. The heating process was kept for an additional 20 minutes before pouring the solution into a thermal casting dish. The casting dish was then weighted to 45 g to preserve film thickness consistency. The casting dish was then dehydrated in an air circulation oven for 15 hours at a temperature of 65°C. The obtained films were labeled according to their composition and concentrations of CHF as shown in Table 2.

# Methods

The thickness was measured to within 0.001 mm using a digital micrometer (Mitutoyo Co., Japan). The average of five measurements was taken as the result.

The density was determined using a densimeter (Mettler-Toledo (M) Sdn Bhd, Malaysia). To prevent water absorption by the hydrophilic samples, distilled water was replaced with xylene as the submersible solvent in this procedure. The density of the xylene should be lower than water. The density of the film was determined using its volume (V) and weight (m) according to Eq. (1).

$$\varrho = \frac{m}{V} \tag{1}$$

where: m = mass, V = volume.

Moisture content was determined using fiber samples. Samples were weighed before  $(W_i)$  and after  $(W_j)$  being heated at a temperature of 105°C for 24 hours. To calculate the moisture content, a digital scale and Eq. (2) were used. The average of three measurements was taken as the result.

Moisture content = 
$$\frac{W_i - W_f}{W_i} \cdot 100$$
 (2)

The method of Shojaee-Aliabadi *et al.* [26] was used. A strip of the material ( $30 \times 10 \text{ mm}$ ) was dehydrated for 24 hours at a temperature of  $105^{\circ}$ C before being immediately weighted ( $W_{i}$ ). Following that, the sample was placed in a lab beaker containing distilled water and stirred for 12 hours at room temperature. The insoluble part of the sample was dried at  $105^{\circ}$ C until it reached a constant weight ( $W_{o}$ ).

Before being placed in the drying oven for 24 hours at 105°C, three samples of 30 x 10 mm film were weighed ( $W_i$ ). The material was weighed again after drying ( $W_o$ ). Using Eq. (3), each sample's water solubility was determined.

T a ble 2. Composition of the films at different CHF loading

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Sample	Sorbitol %/dry starch	CS g/180 ml distilled water	KF %/dry starch	CHF %/dry starch
CS	30	10	0	0
CS-K	30	10	6	0
CS-K/CH2%	30	10	6	2
CS-K/CH4%	30	10	6	4
CS-K/CH6%	30	10	6	6
CS-K/CH8%	30	10	6	8



Fig. 2. Block diagram of film making by solution casting

Solubility(%) = 
$$\frac{W_i - W_o}{W_i} \cdot 100$$
 (3)

The ASTM D 570-98 standard was used to conduct the water absorption test. The initial mass ( $M_i$ ), of the film samples was calculated. The films were then dried at a temperature of 50°C for 24 hours and placed in a desiccator. Next, the film samples were weighed and soaked in distilled water at room temperature. Following the immersion, the soaked samples were removed from the distilled water and wiped dry with a dry and clean cloth before reweighing, ( $M_f$ ). Previous research [27, 28] revealed that WA could be assessed after 30 minutes and 3 hours of immersion time. Three test replicates were completed, and the mass changes between the initial and soaked films were calculated using Eq. (4):

Water absorption (%) = 
$$\frac{M_f - M_i}{W_i} \cdot 100$$
 (4)

An infrared spectrometer model was utilized to identify the presence of functional groups (Bruker vector twenty-two, Lancashire, UK). The FTIR spectrum of 4000 to 400 cm<sup>-1</sup> with a spectral resolution of 4 cm<sup>-1</sup> was presented. The samples were covered with a layer of potassium bromide, and the resulting mixture was pressed into thin, transparent sheets, which were subsequently analyzed.

A Shimadzu LabX XRD 6000 was used for the XRD analysis (Shimadzu Corporation, Kyoto, Japan). The system measurements were performed with the X'Pert HighScore Plus software package, which is an integrated software platform with measurement and analysis capabilities. The experiment was carried out using a scattering angle speed of 1° ( $\theta$ ) min<sup>-1</sup> in the range of angular values from 5° to 40° (2 $\theta$ ). The tube voltage and current were set at 40 kV and 35 mA, respectively according to Ibrahim *et al.* [18]. Eq. (5) was used to calculate the crystallinity index (%) of the samples:

$$X_{c}(\%) = \frac{I_{c} - I_{o}}{I_{c}} \cdot 100$$
(5)

The ASTM D882-2002 [29] standard was used to test the tensile properties of hybrid samples using a 5kN Instron 3365 tensile machine (Instron, Norwood, USA). The test was done at room temperature. The tensile machine clamps were fastened to a film strip ( $70 \times 10$  mm) that was stretched at a crosshead speed of 2 mm/min, with

Composite	Thickness, mm	Weight, mg	Density, g/cm <sup>3</sup>	Moisture, %	Water solubility, %
CS/K	$0.17 \pm 0.02$	$0.08 \pm 0.02$	$1.45 \pm 0.04$	5.99 ± 2	33.67
CS-K/CH2%	$0.18 \pm 0.02$	$0.08 \pm 0.01$	$1.46 \pm 0.05$	$9.36 \pm 1$	38.55
CS-K/CH4%	$0.19 \pm 0.02$	$0.08 \pm 0.01$	$1.45 \pm 0.05$	$7.00 \pm 1$	37.60
CS-K/CH6%	$0.20 \pm 0.02$	$0.09 \pm 0.01$	$1.42 \pm 0.04$	$6.80 \pm 1$	35.24
CS-K/CH8%	$0.20 \pm 0.02$	$0.09 \pm 0.01$	$1.41 \pm 0.03$	$5.98 \pm 1$	31.73

T a bl e 3. Physical properties of hybrid composites\*)

\*) values with different letters in the same column are significantly different (p < 0.05).

an effective grip distance of 30 mm. The tensile machine was connected to the computer program Bluehill 3, which calculated the average values from ten duplicates of each specimen to determine the outcomes of tensile strength, tensile modulus, and breakpoint elongation.

The morphological behavior of the film was studied using a field emission electron microscope (FEI NOVA NanoSEM 230, Oregon, United States). The entire specimens were coated with gold using an argon plasma metalized model to prevent inadvertent charging (sputtercoater K575X, Edwards Limited, Crawley, UK) [30]. The final results of the FESEM experiment were obtained at an acceleration voltage of 3kV. The scans were conducted with an accelerating voltage of 3kV to observe the micro and nanostructure surfaces of the corn husk fiber, kenaf fiber and corn starch longitudinal cross sections.

The analysis of variance (ANOVA) in SPSS software was used to analyze the experimental results. The Duncan test was used to compare the average values at a significance level of 0.05 with ( $p \le 0.05$ ). The mean average and standard deviation (SD) for each sample were reported.

# **RESULTS AND DISCUSSION**

#### Physical properties of hybrid films

Table 3 shows the thickness, weight and density of CS based hybrid films. The addition of CHF significantly increased the thickness but decreased the density of hybrid composite films. During the casting process, the dry matter content per unit area of the film-forming solutions was carefully monitored and different thickness were obtained for control CS layers and hybrid biocomposites. In comparison to the other CHF fiber loading of 2 and 4%, the composite film samples CHF6% and CHF8% had a maximum thickness of 0.2 mm. Consequently, the hybrid samples of CHF8% had the lowest density. The results were related to the intermolecular interaction of both fibers with the polymer matrix. In comparison to biocomposite film, the bigger CHF concentration will result in rigidity of the structure and lower density. In this way, a thicker and coarser film is formed [31].

Fiber addition had a similar effect on the thicknesses and densities of biocomposite films, according to Edhirej *et al.* [14] who studies cassava bagasse reinforced thermoplastic cassava starch, sugar palm starch biocomposite films [32], seaweed/sugar palm fiber reinforced thermoplastic sugar palm starch/agar hybrid composites [33], thermoplastic films based on *Dioscorea hispida* starch [34] and arrowroot fiber reinforced arrowroot starch biopolymer composites [35]. Nonetheless, the lower densities of composite samples made them desirable materials, particularly for applications that demand compactness and ease of use.

# Moisture content

Despite its hydrophilic nature, the moisture content is an essential aspect to consider when selecting natural fibers as reinforced materials to create new composites. When it comes to mechanical performance, the creation of porosity and water holding capacity, a low moisture content is necessary. High water content may have a detrimental impact on the dimensional stability of the composite material [36].

However, Table 3 demonstrates that the hybrid composites' moisture content was slightly reduced because of the CHF integration. This phenomenon was explained by Jawaid *et al.* [36]; Ilyas *et al.* [37]; Ibrahim *et al.* [18]; Ramesh *et al.* 2020 [38] through the function of cellulose in the hybrid composite. This means that the inclusion of another fiber enhances the cellulose content of the composite, lowering the moisture content rate. A hydroxyl group that is present in the structure of cellulose, makes it insoluble in water [39].

#### Water absorption study

Composites made of natural fibers are less resistant to moisture because hydroxyl and polar groups are moisture-absorbers [18, 25, 35, 40, 41]. Therefore, the influence of the water absorption rate on the CS-K/CH hybrid composites properties was investigated. According to Jawaid et al. [36], the tensile strength, porosity formation, dimensional stability, and swelling index of the final product are all significantly influenced by the physical characteristics of hybrid biocomposites, particularly the water absorption character. Bodur et al. [41] list three strategies for controlling water diffusion in polymer composites. The first two are the diffusion of water into the tiny spaces between neighboring polymer chains, capillary action into the spaces at the fiber-matrix interface, and transport through tiny fractures in the matrix. Figure 4 (c, d) displays the results of the water absorption test. The



# Fig. 3. Thickness (a) and density (b) of the films

240

220

200

180

160

140

120 100

80

60

40

20 0

Waer absorption, %

a b

CS/K

a a,b



ā

a b

b





CS-K/CH2% CS-K/CH4% CS-K/CH6% CS-K/CH8%

30 min

140 min

a a



45 40 35 30 25 20 15 10 5 0 CS/K CS-K/CH4% CS-K/CH6%  initial assessment of water absorption was made after 30 and 140 minutes of immersion when the samples reached the maximum saturation level. For the first 30 minutes, you could see a stunning result in hybrid films due to their hydrophilic nature, this is one of the disadvantages of bio-based materials. After 50 minutes, CS-K/CH8% absorbed about 215% amount of water, while CS-K/CH2% had total absorption of 231%.

After one hundred minutes of immersion, the variance in water absorption rate became more stable, with the hybridized composites showing the best resistance to water uptake. The fibers role in the creation of strong interfacial bonding in the structure, which prevents water from penetrating through the matrix, may have contributed to the decrease in absorption rate in the presence of CHF [28]. Additionally, the presence of lignin as a key ingredient in the creation of fiber, together with wax and fatty compounds, improves composites' resistance to water [42]. In addition, Ibrahim *et al.* [18] showed that gradual loading of SPF significantly reduced the rate at which hybrid composites based on cornstarch absorbed water.

# **X-Ray Diffraction**

X-ray diffraction analysis is commonly used to determine the percentage of crystallinity and the structure of materials like those used in this study. The XRD curves of CS-K/CH at different CHF loading, are shown in Figure 5. All samples displayed patterns of behavior that were comparable to those in the CS/K film from the prior investigation [25], the only difference was that after the addition of fiber loading, the main peaks' intensity improved. Sharp 20 peaks diffracted at angles of 16.70°, 16.82°, and 1.86° were established by the gelatinization and retrogradation of starch molecules in the crystalline structure of the CS-film without lignocellulosic fiber. These results were in line with the typical A-type pattern of the naturally derived starches, as shown in [43–45]. As it was expected, the addition of fibers raised the main peaks' intensity, which improved the hybrid composites' relative crystallinity. This finding demonstrated that the basic amorphous structure of starch and fiber was mostly preserved in the CS-K/CH sample [46–49]. However, a slight difference in the diffraction patterns still can be observed. The crystallinity ranged from 10° to 20° reflectance in the CS films, which is similar to the B-type diffraction pattern [50, 51]. Zhong et al. [52] also found a B-type crystal double helix with  $2\theta$  peaks at  $17^{\circ}$ .

According to Faruk *et al.* [53] and Hazrol *et al.* [54], the role of cellulose in enhancing crystallinity by building a crosslinked network and preventing composite disintegration by forming covalent bonds between fiber and matrix may help to explain the correlation between fiber loading and higher crystallinity. Table 4 shows the crystallinity values of corn-starch hybrid films as influenced by various fiber loadings. Natural fibers are orientated materials, hence an improvement in the crystallinity of starch-based composites is anticipated as the fiber content rises [5, 55].



Fig. 5. XRD curves of CS-K/CH hybrid composites with various CHF content

Sample	Crystallinity, %	
Control	18.0	
CS-K/CH2%	21.0	
CS-K/CH4%	28.4	
CS-K/CH6%	32.6	
CS-K/CH8%	39.4	

T a b l e 4. Crystallinity of CS-K/CH hybrid films

#### Fourier transform infrared spectroscopy (FTIR)

The functional groups in corn starch hybrid with various CHF loadings were examined using FTIR. Figure 6 shows the infrared spectra of plasticized CS-K composite and CS-K/CH hybrid composite films. The FTIR spectra properties of each hybrid film sample have the same absorbance peaks that are connected to fibers and polymer elements. Since the materials were from biological sources, their chemical composition comprises certain continuity, including cellulose, lignin, and hemicellulose in the case of the fibers, and amylose and amylopectin in the case of the starch. All films exhibit wide bands between 1700 to 2900 cm<sup>-1</sup>, which are due to the O-H hydroxyl groups in the starch and fiber structure vibrating in a stretched manner. Through the frequency shift of wide bands of the O-H group in CS, the H-bonds between CS, KF, and CHF were identified. This research finding was supported by Sanyang et al. [49]; Hazrol et al. [54]; Nordin et al. [56]; Galindez et al. [57]; Cerqueira et al. [58]; Jafarzadeh et al. [59]; Ili Balqis et al. [60].

The results from [56, 61, 62] were used to support the claim that the (C-H) methyl group was responsible for the peaks in the range of 2900 to 2950 cm<sup>-1</sup>. Meanwhile, the hydroxyl group of absorbed water within starch films was responsible for a strong absorption peak in the region of 1642–1645 cm<sup>-1</sup> [63]. This peak can also be attributed to the stretching vibrations of the starch water OH groups. Additionally, several functional groups, such as C-O(H) and C-O-C, contributed to the absorption bands at 1105 cm<sup>-1</sup> and 1150 cm<sup>-1</sup>, respectively. The vibrations of the polysaccharide compound of starch and fibers C-O stretching were linked to the band bigger than 993 cm<sup>-1</sup>[64, 65]. Ilyas *et al.* [10] noticed comparable peaks on the control and a plasticized film, in the ranges of 1020-995 cm<sup>-1</sup>, 1030–990 cm<sup>-1</sup>, 1082–1029 cm<sup>-1</sup>, 1150–1033 cm<sup>-1</sup>, and 1004 cm<sup>-1</sup> respectively. The interaction between the starch matrix molecules and the fibers was also confirmed by the band shifting upon fiber loading. For example, in Fig. 6, where the O-H stretching peaks at about 3200-3500 cm<sup>-1</sup> for CS film shifted to lower intensity bands after hybridization. Increased intermolecular hydrogen bonding in the hybrid structure is shown by the shifting of the band location, which results in stronger contact and bigger compatibility [66]. According to Ibrahim et al. [18] a similar interpretation was made,



Fig. 6. FTIR curves of CS-K/CH hybrid composites with multi-scale CHF

when corn starch-based composites were hybridized with sugar palm fiber.

### Mechanical properties

Figure 7 shows that the addition of CHF results in a noticeable increase in tensile strength (TS) and tensile modulus (E) and a markedly reduced elongation at break (EB). This suggests that hybridized composites have increased stiffness, as well as resistance to materials [67].

The mechanical properties of the hybrid composites did, however, significantly improve with the addition of CHF. For instance, the tensile strength and tensile modulus at CHF content of 6 wt% were 23.2 and 1486.1 MPa, respectively higher than that of CS/K composite, which were 17.7 and 1324.7 MPa. This enhancement could be attributed to cellulosic fibers' capability to control the mobility of starch molecules, promote interfacial bonding by creating a network that tightly ties the composites together and makes stress transmission easier, and dominate the mobility of starch molecules [68-71]. This is the relative crystallinity that Salaberria [72] described as a potential explanation for this improvement. Additionally, according to the researchers, an increase in crystallinity improves stiffness, which in turn improves system performance. In addition, the FTIR analysis revealed that the hydroxyl group and sorbitol formed a chemical link that improved the stress transmission from the matrix to the fiber and increased the fiber's tensile strength [73–75].

Despite this improvement, the tensile strength and modulus trended downward when CHF was added above 6 wt%. Too high fiber content led to the formation of agglomerates, which resulted in poor tensile properties [25, 76]. In contrast to elasticity modulus and tensile stress, fiber loading had a negative effect on the elongation of CS-K/CH hybrid composites. The composite's elongation dropped as the loading increased. This element controls how easily the material may be extended from its starting length to its breaking point [10, 77]. Film





elongation decreased as fiber loading increased because the presence of cellulose rebuilds the composite's structure by strengthening intermolecular bonds in the starch matrix. Through the elimination of chain mobility, this rebuilding of the starch network increases stiffness, hence reducing flexibility [78, 79].

#### Field emission scanning electron microscopy (FESEM)

Figure 8 displays the surface fracture FESEM images of the CS-K/CH hybrid composites, neat CS-plasticized film, and CS/K biocomposite. The CS-plasticized film had poor porosity, as well as compact, and comparatively smooth structure. Undissolved starch particles were also visible in the film. The addition of kenaf fiber to create a bio composite (CS/K) led to less uniform surfaces and a robust, uneven structure with a coarse texture. The addition of CHF to a hybrid composite, however, resulted in a more robust structure since the starch matrix completely encased the fiber particles. The absence of CHF clusters or aggregation within the CS matrix suggests that the interfacial interaction between the reinforcing fibers and polymer matrix was flawless. Strong interfacial interactions signal efficient stress transfer, which increases strength [80]. Due to the addition of CHF as a hybrid filler, the surfaces fracture of the CS-K/CH hybrid composite (2, 4 and 6%) were rougher than those of the CS-film. However, they seemed smoother than their CS-K/CH8% counterpart, indicating that the low concentration of CHF was more compatible with the CS-matrix than the high concentration. Due to the excessive usage of fiber, which negatively impacts structural integrity, the hybrid structure appeared to be less consistent in the case of the maximum CHF (CS-K/CH8%) addition, as demonstrated by the appearance of microcracks and CHF aggregation on surface fracture [54].

# CONCLUSIONS

New hybrid bio-composite films of corn starch (CS), kenaf (KF), and corn (CHF) fibers were obtained by solution casting and dehydration. Following the hybridization process, the performance of the composite film significantly improved according to testing and characterization data. The hybrid films' tensile strength and Young's modulus increased from about 18 to 23 MPa and from 1325 to 1486 MPa, respectively. The 6% (w/w dry starch) CHF shows the most prominent reinforcing loading among all. The density of CS-K/CH composites in water is slightly lower (2-2.7%) compared to CS-K composite. Following the fiber loading of CS-K/CH 6% and CS-K/CH 8%, which were 32.6% and 39.4%, respectively, the diffraction analysis showed a considerable improvement in the crystallinity degree. The addition of CHF improved the physical properties of the composites. Regarding the current initiative, which aims to use waste as a new source of production, it is feasible to see the significant capacity to repurpose raw materials into operations with fewer consequences for the environment, hence lowering the carbon footprint related to the use of raw materials. A potential application of the biocomposite is the packaging.

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50µm

a)

10 µm

b)



c0







Fig. 8. FESEM micrographs: a) CS, b) CS/K and CS/K/CHF composites: c) 2 wt% CHF, d) 4 wt% CHF, e) 6 wt% CHF, f) 8 wt% CHF

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