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# Comparative Study on the Tensile Properties of Thermoplastic Corn Starch Films Reinforced with Cornhusk and Stalk Fillers

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# دراسة مقارنة على الخواص الشدّية لأغشية النشا الذرة الحرارية المدعمة بحشوات قشور وسيقان الذرة

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**Abstract:**
With rising concerns regarding non-biodegradable waste in today's environment, there has been growing interest in naturally available fiber-reinforced bio composites, especially those derived from agro-waste resources. In this regard, this study presents a comparative evaluation of the tensile properties of thermoplastic corn starch (TPS) films reinforced with two types of filler materials obtained from corn: Corn Husk (CHF) and Corn Stalk (CSF). Bio composite films were developed by solution-casting using fructose as a plasticizer, with 2% to 8% fiber content. Results indicate distinct reinforcing behaviors between these two types of filler materials. In contrast to composites reinforced with CHF, a higher tensile modulus of the films reinforced with CSF can be due to the rigid nature of the stalk fibers. At the same time, however, higher brittleness and lower elongation at break of the CSF-reinforced films suggest that despite rigidity, there is a reduction in flexibility. CHF-reinforced films tended to have better tensile strength and higher elongation at break values, which would demonstrate higher flexibility than CSF-reinforced films. Based on 8% CHF content, films achieved an optimal balance between tensile strength and flexibility. Thus, from an efficiency standpoint, it can be concluded that Corn Husk is more suitable than Corn Stalk for creating flexible and environmentally friendly food packaging materials.

**Keywords**: Tensile Properties, Bio composites, Thermoplastic Starch, Interfacial Adhesion, Corn Husk/Stalk, Fracture Behavior.

**الملخص**
مع تزايد المخاوف بشأن النفايات غير القابلة للتحلل في البيئة، كان هناك اهتمام متزايد بالمواد المركبة الحيوية المدعمة بالألياف المتاحة طبيعيًا، خاصة تلك المشتقة من موارد النفايات الزراعية. في هذا الصدد، تقدم هذه الدراسة تقييمًا مقارنًا للخصائص الشدلل أفلام النشا الحرارية البلاستيكية (TPS) المدعمة بنوعين من مواد الحشو المستخرجة من الذرة: ألياف قشرة الذرة (CHF) وألياف ساق الذرة (CSF). تم تطوير الأفلام المركبة الحيوية عن طريق الصب باستخدام الفركتوز

كمادة ملدنة، مع محتوى ألياف يتراوح بين 2% و8%. تشير النتائج إلى سلوكيات تعزيز متميزة بين هذين النوعين من مواد الحشو. على عكس المركبات المدعمة بـ CHF، وجد أن معامل الشد يكون أعلى في الأفلام المدعمة بـ CSF بسبب الطبيعة الصلبة لألياف الساق. في الوقت نفسه، ومع ذلك، تشير القيمة الأعلى للهشاشة والقيمة الأقل للاستطالة عند الكسر للأفلام المدعمة بـ CSF إلى أنه على الرغم من الصلابة، هناك انخفاض في المرونة. تميل الأفلام المدعمة بـ CHF إلى أن تكون لها قوة شد أعلى وقيم استطالة عند الكسر أعلى، مما يدل على مرونة أعلى من الأفلام المدعمة بـ CSF. بناءً على محتوى CHF بنسبة 8%، حققت الأفلام توازنًا مثاليًا بين قوة الشد والمرونة. وبالتالي، من وجهة نظر الكفاءة، يمكن استنتاج أن قشرة الذرة أكثر ملاءمة من ساق الذرة لإنشاء مواد تغليف الأغذية المرنة والصديقة للبيئة.

**الكلمات المفتاحية:** الخصائص الشد، المركبات الحيوية، النشا الحراري البلاستيكي، الالتصاق البيني، قشرة الذرة/ساق الذرة.

## Introduction

The eventual accumulation of non-biodegradable plastics in the environment has become a pressing problem that highlights the need to replace them with biologically derived and sustainable materials [1,2]. Among the materials that have gained prominence in the packaging of products using biological principles is starch [3]. While the most commonly known source of starch is the corn plant, commonly referred to as "Zea Mays," the starch films are mechanically weak and water-sensitive [4].

To overcome these drawbacks, plasticizers like fructose are blended to create thermoplastic starch (TPS) [5]. However, TPS still has limitations in providing enough strength to cater to packaging demands [6]. There might be some scope to improve TPS by mixing natural fibers because their strength can be better than inorganic materials with similar densities [7].

From the viewpoint of the agricultural sector, the following is a description of the corn industry as a material generator: The corn industry produces post-harvest waste, specifically corn husks and corn stalks. These wastes are usually burned or disposed of, which results in pollution and contributes to the production of greenhouse gases [8]. The process of using the corn industry's byproducts as a filler material in starch composites reinforces the concept of a circular economy [8,9].

Although there has been considerable work reported on natural fibers such as jute, kenaf, and sisal, relatively less work has been done on utilizing different residues of corn in thermoplastic corn starch film matrices. Corn husk fibers can be expected to be cellulose-based soft fibers, while corn stalk fibers can be labeled as lignocellulosic rigid fibers [1 , 10]. Keeping these factors in mind, it is proposed in this study to develop and compare the tensile strengths of TPS films incorporating corn husk fiber (CHF) and corn stalk fibers (CSF). It is basically an effort to explore structural differences in properties between these two materials for optimal reinforcing agent requirements for sustainable food packaging materials.

## Experimental

2.1 Materials

corn starch (CS) was extracted from the grains of fresh corn ear through the wet milling process. Cornhusk was obtained from the leaves that surrounded the corn ear. While corn stalk was derived from stems of the corn plant. Both corn husk and stalk were ground and sieved to be used in powder form. Fructose supplied by evergreen Sdn. Bhd., Malaysia, and was employed as plasticizers.

### 2.2 Films preparation

The solution casting method was employed to prepare starch films by utilizing a film-forming solution that includes 5 g of corn starch/100 ml distilled water. In this case, fructose used as plasticizers at concentrations of 0.25 g/g dry starch. The corn husk and corn stalk were used as filler, with a particle size of less than 300 µm at different loading of 2, 4, 6, and 8 % of dry starch. The films contain 2, 4, 6, and 8 % of husk were coded as H2, H4, H6, and H8, respectively. The films with 2, 4, 6, and 8 % of stalk were termed as S2, S4, S6, and S8, respectively. The mixture was heated to 80±5 ºC in a thermal bath and kept at this temperature for 20 min under constant stirring. Air bubbles that were formed during heating were removed by placing the film-forming solution into a desiccator under vacuum until there was no bubble and poured homogenously onto circle Petri plates with 140 mm diameter. The plates with the film-forming solution were then dried in an oven with air circulation at 50 ºC. The dry films were taken off from the plates and kept at ambient circumstances in plastic bags for almost a weak before characterization.

**Film Characterization**

**3.1 Scanning electron microscopy (SEM)**

An electronic microscope device type (Hitachi S-3400N, Japan) was employed to scan the surface morphology of the films. Each sample was surrounded by a thin golden layer (0.01-0.1 μm) and mounted on a bronze griddle before applying (20 kV) acceleration voltage. The scanning was conducted under a high vacuum atmosphere included liquid nitrogen to freeze the films. The test resulted in a high-resolution image at different magnification factors.

**3.2 Tensile properties**

The mechanical behavior of the specimens was evaluated using a universal tensile machine (5KN INSTRON). The test was performed according to D882 (ASTM, 2002) standard at ambient conditions. A film strip (70mm×10mm) was fixed between the tensile machine clamps and pulled with 2 mm/min crosshead speed, and the effective grip separation was set to be 30 mm. The results of tensile strength, elastic modulus as well as the elongation at breakpoint were calculated from the average measurements of five replicates for each specimen.

**Results And Discussion**

**4.1 Scanning electron microscopy (SEM)**

The fracture surface images, as in Figure 1 show the difference among the prepared samples containing the various fiber loading. It can be observed that the film with husk presents coherent homogeneous surfaces and clearly coated by the starch matrix. The homogeneity of the matrix in the films is a good indicator of their structural integrity [11, 12]. For this reason, it was anticipated that the films containing husk would show better tensile properties. The films with stalk presented smooth surfaces and homogeneous texture with noticeable stalk needles at minimum loading. This probably due to the fiber agglutinations that are not desired, and they limit the stress transfer from the matrix to the fiber and hence decrease the mechanical strength of the composites [13]. However, when higher filler contents are used, the particle density increases, thus promoting the interaction between the filler particles, which could induce the formation of aggregates. The presence of these aggregates, together with the large size of fiber induces defects in the matrix, which affects the integrity of the film structure [14].

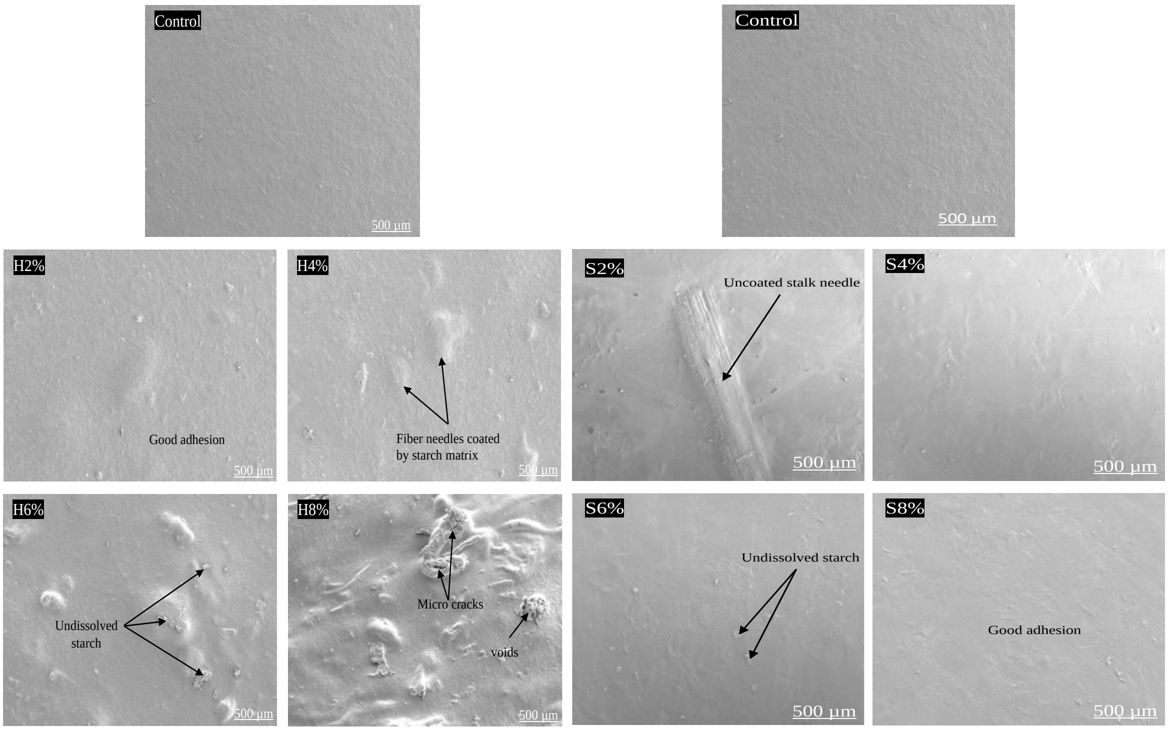



**Figure 1:** SEM images of cornstarch composite films with various loadings of husk and stalk.

**4.2 Tensile properties**

Adding the reinforcement agents into the starch materials is expected to improve the ultimate tensile strength. Figure 2 illustrates the tensile strength (a), tensile modulus (b), and elongation at break (c) properties for corn TPS films compared with corn husks and stalk bio composites at different levels of filler content.

Tensile Strength: As indicated by Figure 2(a), the tensile strength of TPS films increases with the inclusion of fibers. The corn husk fiber-reinforced films possessed a higher tensile strength compared to corn stalk fiber-reinforced films. The sample showing the largest tensile strength of around 12.84 MPa corresponds to H8, which is a remarkable improvement compared to the values from the plain film. This enhancement is a result of the compatibility and structural similarity of starch and cellulose since they are from the same biological origin [10]. This is evidenced by the scanning electron microscopy results shown, wherein the needle-like morphology of CHF encapsulated in the chitosan matrix with the absence of clusters shows evidence of compatibility and homogeneity of the CHF in the chitosan matrix.

Tensile Modulus (Stiffness): Analysis of the stiffness results provided in Figure 2(b) indicates a clear pattern for stalk reinforced composites. Corn stalk reinforced films have a higher tensile modulus compared to husks at the same weight fraction. This is clear in the fact that the S8 sample has a tensile modulus of around 880 MPa compared to the 640 MPa of the H8 sample.

This difference can be attributed to the inherent composition of corn stalks, having a considerable amount of lignin, thereby increasing the stiffness of the composite. Regarding stalk composites, SEM evidence of excellent adhesion between stalk fiber and starch matrix supports this high stiffness, as good interfacial interaction provides an excellent transfer of stress [15,16].

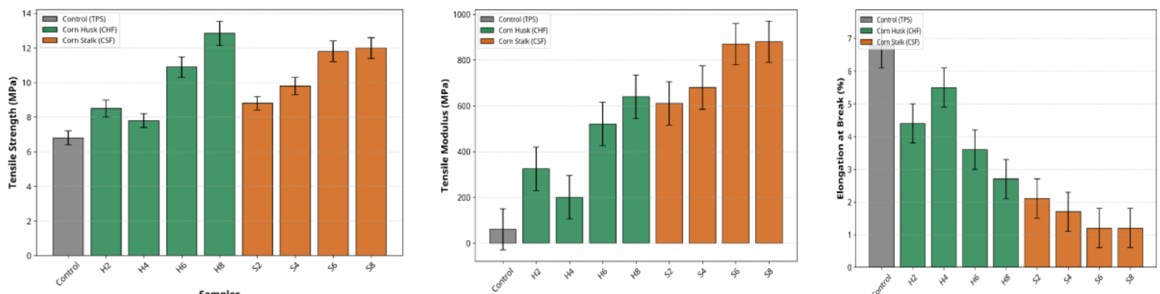
Elongation at Break: Elongation at break (Figure 2c) generally decreased with an increase in fiber loading in all the composites. The reduction in elongation was more in CSF films, which depicted higher stiffness. In contrast, the CHF films showed higher values of elongation, which emphasized that the husk fibers offered a more balanced effect of increased strength with less reduction in the values of film elongation. The observed decrease in film elongation with increased fiber loading is due to the fact that cellulose increases the intermolecular bonds of the starch matrix and thus, creates more hydrogen bonds between the fiber and starch molecules. Such reconstruction in the starch network induces rigidity and reduces the flexibility of films by hindering chain mobility [17].

## Conclusions

The effects of incorporating multiscale corn husk and corn stalk fibers as fillers to reinforce corn starch films' mechanical properties were assessed. Scanning electron microscopy results showed that both fillers were well dispersed within the polymer matrix. The husk fibers were more homogeneous with less agglomeration compared to the stalk fibers.

On comparison of the mechanical properties, the differences in the characteristics of the fillers were evident. The CSF fillers were more successful in enhancing the tensile modulus of the films, which could be attributed to the rigid nature of the lignocellulose filling material. The CHF fillers were more compatible with the starch matrix, thereby providing higher tensile strength and values of elongation at break.

Among these preparations, the composite film with 8% husk content (H8) had an optimal combination of strength (12.84 MPa) and flexibility. Noteworthy is that the particles of corn husk and corn stalk used in this study had not undergone any chemical processing. Thus, it can be observed that there is great potential for making this process more economical for production related to biodegradable packaging.




(a) (b) (c)

**Figure 2:** Tensile properties of cornstarch composite films with various loadings of husk and stalk.

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