# Characterization of the Physical, Mechanical, and Morphological Properties of Films Generated from Cassava Pomace

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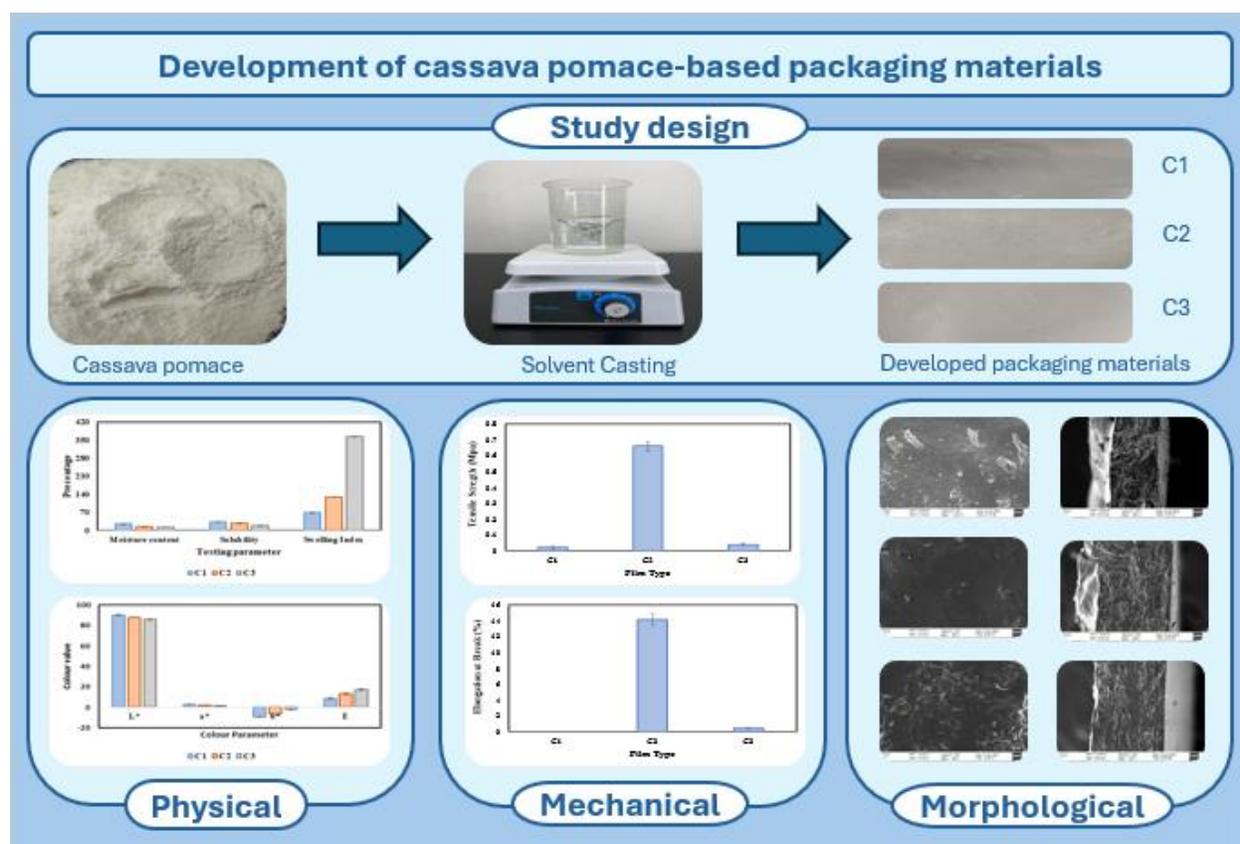
Abstract

The pollution of the environment caused by conventional packing materials such as plastic has driven the need for biodegradable alternatives. Although starch is an important component in the development of such materials, starch is not suited for use as a biodegradable packaging material due to global hunger challenges. The current study successfully utilized the major industrial waste of the cassava starch processing industry to develop biodegradable films. Three

packaging materials were developed using the casting procedure, which involved combining various quantities of cassava pomace (CP) and plasticizer combinations. The developed films were analyzed to evaluate their characteristics, such as color, thickness, density, moisture content, solubility, swelling index, mechanical properties, microscopic, and FT-IR characteristics. In contrast, concerning multiple aspects, each of the films demonstrated unique characteristics. The film with the lowest CP (C1) appeared to be thinner and lighter in color; however, it tended to contain a greater amount of moisture. The C1 film exhibited an adhesive property that was well-suited for use as cling film. The intermediate CP film (C2) stands out because of its excellent mechanical characteristics, including high tensile strength and elongation at break. These attributes make it particularly well-suited for packaging applications, such as the production of biodegradable bags. Conversely, the swelling index and thickness of the highest CP film (C3) outperform both other films, suggesting that it may have the capacity to absorb higher moisture content. The scanning electron microscopic images revealed a uniform surface for all three samples. However, the cross-sectional images of C3 indicated internal cracks that were consistent with the lowest mechanical characteristics and flexibility. Therefore, the C3 film is more suitable for packaging items like plates. These films can serve as a viable, environmentally friendly, and biodegradable alternative to conventional packaging materials.

1.0 Introduction

Packaging plays a critical role in the food industry since it serves as a protective barrier while providing important information regarding ingredients, nutritional content, and traceability (Marsh & Bugusu, 2007). Plastic packaging contributes significantly to environmental pollution, second only to climate change. However, they are frequently used due to their minimal manufacturing cost, ease of handling, and stability. Food-grade plastic is used for food safety, shelf-life extension, and transportation protection. Most of the plastic pollution comes from food and beverage packaging, primarily from Asia. Significant quantities of single-use plastic packaging are responsible for the annual release of millions of tons of plastic waste into the environment after evading collection systems. Only 2% of plastic packaging trash is recycled, with some recycled into secondary plastic and the rest dumped or burned. This problem is more noticeable in developing countries, where recycling infrastructure is inadequate. The 'sachet economy' complicates problems further, as sachets are a prevalent form of product distribution in developing nations. These small, flexible, and soft plastic products are marketed to those with low incomes, making them difficult to recycle. Over 115 countries have either enacted or are in the process of implementing legislation to ban the usage of plastic bags that are only used once and other similar items (Phelan et al., 2022).

Biodegradability is a waste management strategy that employs microorganisms to eliminate products from the environment in a timely, safe, and effective manner. Biodegradable plastics are a subset of a larger class of materials known as bioplastics. The biodegradability of materials is dependent on the environmental conditions, chemical composition, final product structure, and raw materials. There are two types of biodegradable polymers. The first category consists of biodegradable plastics derived from petroleum resources that have the potential to biodegrade, and the second category comprises biobased plastics produced from biomass

feedstock material. There are several subclasses of bio-based plastics, including those composed of starch or cellulose or protein, polylactic acid (PLA), and polyhydroxyalkanoates (PHA). Subcategories of biodegradable plastics derived from fossil fuels include polybutylene succinate, polybutylene adipate terephthalate, polycaprolactone, and polyvinyl alcohol (Havstad, 2020). The growing interest in biodegradable polymers is attributed to its positive environmental impact and potential use across several industries. Biodegradable plastics are commonly used in various applications such as recyclable garbage bags, biodegradable compost film, catering goods, film wrapping for perishable food products, stiff packaging, and special biodegradable plastics used in medical technologies. These materials have various advantages, including less waste disposal, improved composting processes, and cost savings. Starch has acquired popularity among biodegradable plastic categories because of its abundant availability, affordable price, and ability to completely biodegrade without leaving behind any toxic residuals (Rujnić-Sokele & Pilipović, 2017).

Cassava pomace, a byproduct of the industry that processes cassava starch, is made up of trace amounts of protein and fat, fibers, moisture, pectin, and starch (Agustina et al., 2019; Bussolo de Souza et al., 2018). Cassava pomace may have a substantial quantity of residual starch as a result of insufficient processing conditions. Several factors impact the dietary content and visual attributes of cassava pomace, including harvest period, plant variety, extraction technique, and industrial apparatus (Rojan, 2009). Each metric ton of processed root yields around 900 kg of pomace, which has a moisture content of 85%. In the year of 2015, Brazil's cassava industry yielded 750 thousand tons of cassava starch and obtained 696 tons of cassava pomace as a major waste (Garcia et al., 2019). Despite its value, cassava pomace is often thrown away without proper treatment, contaminating air and water (Wicaksono et al., 2013). This versatile substance can be utilized to produce a wide range of valuable products, including biodegradable films, nanoparticles like nanofibers, organic acids such as ethanol, biofuel, enzymes, biologically active primary and secondary metabolites, and other high-value items (Pandey et al., 2000). The presence of residual starch and natural fibers in cassava pomace creates optimal circumstances for the production of thermoplastic starch, resulting in it being appropriate for the manufacturing of food packaging materials (De Morais Teixeira et al., 2005). Researchers have investigated the utilization of cassava pomace throughout packaging materials using several methods, such as reinforcing into starch films, recovering fibers from cassava pomace and incorporating them into packaging, developing active packaging films, and blending with polymers, as documented in the literature (Akmeemana et al., 2024). However, no literature exists on the direct use of cassava pomace in developing packaging materials using casting techniques. This study investigates the utilization of cassava pomace, a waste product of the cassava starch manufacturing industry, as a material for biodegradable films. This study examines the effects of different combinations of plasticizers and concentrations of cassava pomace on the mechanical and physical properties of film materials generated from cassava pomace, including thickness, density, Fourier transform infrared (FT-IR) bands, color, moisture content, solubility, and scanning microscopic view.

2.0 Material and Methods
2.1 Material
The major raw material Cassava (MU 51 variety) was purchased from the Piliyandala area. Glycerol and sorbitol of analytical grade and distilled water were used.

2.2 Production of cassava pomace

The procedure reported by (Gunathilake & Somendrika, 2024) was modified to create cassava pomace. The cassava roots (MU 51 variety) were cleaned to get rid of sand and dirt particles. The peeled cassava tubers were then pulverized with a grater, and the resulting pulp was blended for 2 min in a laboratory blender with a 1:4 water ratio. The blend was afterwards strained with a cotton cloth. The leftover filtrate on the muslin cloth was dehydrated at 60 °C to achieve a moisture content of 10 to 12% (w/w). The pomace was then mechanically pulverized and sieved.

2.3 Film preparation

The casting technique was used to develop three packaging materials using sorbitol and glycerol as plasticizers, and cassava pomace in proportions of 3% (w/v), 5% (w/v), and 7% (w/v) for C1, C2, and C3 films, respectively. The mixture was mechanically stirred through a hotplate stirrer (Lab Companion, Daejeon, Republic of Korea), at 700 rpm for 10 min at ambient temperature, then heated to 86 ± 2 °C and subjected to a constant temperature of that value for a duration of 20 min, while being stirred continuously at a speed of 700 rpm. The mixture was vacuum degassed then cast on nonstick pans and dehydrated at 50 °C in a dehydrator.

2.4 Film Color

Color measurements were conducted using an LC100 colorimeter manufactured by Lovibond, Tintometer Ltd, UK. The color properties examined for white paper (the standard) were as follows: (L = 94.30; a = 4.2; b = -17.9). The L* value represented the range of brightness from black to white, whereas the a* and b* values showed the range of green to red and blue to yellow accordingly. The color difference (E) was calculated using equation (1) utilizing white paper as standard:

$$\Delta E = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (1)$$

The values ΔL*, Δa*, and Δb* represent the differences between the standard and sample values of L*, a*, and b*, respectively (Carvalho et al., 2018).

2.5 Film thickness and density

The measurement of thickness was obtained using a digital micrometer (CH.MM.25D, China, resolution of 0.001mm) The specimen weight and volume were used to calculate the film density (g cm^{-3}) (Travalini et al., 2019a).

2.6 Mechanical properties of the film

The percentage of elongation at break, tensile strength, and Young's modules were determined using a dual-column tabletop universal testing system (Jinan Marxtest Technology Co. LTD, Model ETM-5) as explained in (de Carvalho et al., 2019) with minor modifications in the firing load. From each film, three constant strips (70 mm * 25 mm) were trimmed and mounted between tensile clamps. (The initial grip distance is 43 mm, the crosshead speed is 2 mm/s, and the firing load is 100 N) The dual-column tabletop universal testing machine was applied to break down films to capture the original properties of the film's cross-sectional surface.

2.7 Moisture content, solubility, and swelling index

Using a modified oven drying procedure (Senarathna et al., 2022), moisture content analysis was performed on the films. Each film sample was weighed after cutting into 2 x 2 cm sections (W0). The samples were subsequently reweighed (W1) after being subjected to drying at

a temperature of 105 °C until it reached a stable weight, it was then cooled to the surrounding temperature in a desiccator. To calculate the moisture content (percent), the subsequent equation was used:

$$Moisture\ content = \frac{(W1-W0)}{W0} \times 100 \quad (2)$$

The water solubility of these films was determined using a modified approach described by Senarathna et al., in 2022. The films were measured in weight after being divided into 2 by 2 cm sections and subjected to an 8 h drying period at a temperature of 105 °C (W1). After that, the samples were subjected to continuous agitation within 50 ml of distilled water at ambient temperature for a duration of 6 hours. When the leftover films were dried to a uniform weight at 105 °C, their weights were measured and recorded as W3. The following formula was used to calculate the films' water solubility.

$$Solubility = \frac{W1-W3}{W1} \times 100 \quad (3)$$

The swelling index of films has been evaluated using the procedure outlined previously (Senarathna et al., 2022). The films were divided into fragments measuring 2 x 2 cm, their weight was recorded as WO, and thereafter immersed in 50 ml of distilled water for a duration of 24 hours. Afterwards, the films were re-weighed (W2) once the excess water on the surface was removed by gently wiping the films with tissue paper. The swelling index was calculated using the given formula.

$$Swelling\ Index = \frac{W2-W0}{W0} \times 100 \quad (4)$$

2.8 Scanning electron microscope
A scanning electron microscope (Carlezeiss-evo18) was used to examine the films' interface and cross-sectional morphology. A gold and platinum mixture was used to metalize the samples that were mounted on carbon conductive tape. Images were taken with a 10 kV acceleration voltage, under high vacuum mode.

2.9 FT-IR
The ATR (attenuated total reflectance) mode of the Thermo Scientific Nicolet S10 FT-IR spectrometer was used to perform FT-IR analysis for the film samples with wavelengths from 600 to 4000 cm^{-1}.

3.0 Result and Discussion
The color of the product's packaging can influence the product's visual perception, making it a significant factor in the consumer's acceptance of the product. The color attributes of the films were discovered by measuring the L* (lightness), a* (green-red), and b* (blue-yellow) values (Senarathna et al., 2022). These measurements are presented in Figure 1. The C1 film exhibited the maximum lightness value, whilst the C3 film demonstrated the lowest lightness value. A substantial difference existed among the films. When the cassava pomace concentration of the films increased, there was a significant decrease in lightness. A trend consistent with the brightness value was also shown by the a* and b* values. Versino et al., in 2015 developed a biodegradable film using cassava root. The researchers also observed that when cassava pomace was incorporated

into the cassava starch matrix, the L* value ranged from 90.68 ± 0.59 to 88.83 ± 0.81 (Versino et al., 2015). Furthermore, the total color difference values ranged from 11.12 ± 0.97 to 16.48 ± 2.80, which aligns with our findings from the study.
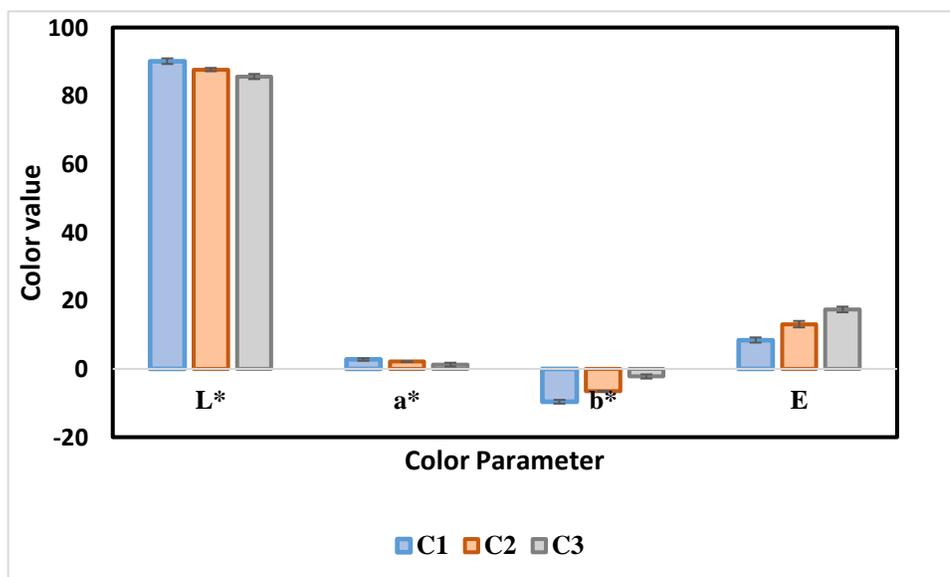
Figure 1: Color values of the C1, C2 and C3 films.

The capacity of packaging to resist outside forces is highly dependent on the mechanical characteristics of the materials used to construct it (Travalini et al., 2019). The mechanical properties results concerning tensile strength, elongation at break, and Young's module were presented in Table 1. When considering the mechanical properties C2 film obtained a considerably higher mechanical property when considering the other two films. There are two possible factors that could be accountable for the increase in tensile strength. They are the reinforcing agent's high compatibility with the polymeric starch matrix and the inherent characteristics of the starch amylopectin chains. On the outermost layers of the cellulose fibers in cassava bagasse, starch amylopectin chains can appear, thereby increasing the film's resistance (Carvalho et al., 2018). The least tensile strength was reported for the C3 film. This could be due to a higher concentration of larger particles at higher proportions (7% (w/v)) of cassava pomace, resulting in a less homogeneous material prone to mechanical defects (Edhirej et al., 2017; Versino et al., 2015). Furthermore, fibers had more crystallinity than starch, resulting in decreased extendibility also could cause a reduction in tensile strength (Prachayawarakorn et al., 2013). The same finding was observed by others (Edhirej et al., 2017; Ferreira et al., 2019; Versino et al., 2015) as the concentration of the substance increased. Similarly in the present study, structural defects are shown in the scanning microscopic images of the C3 film. The decrease in the mechanical characteristics of the C3 film may be attributed to these structural flaws. Elongation at break is a measure of the ability of a film to stretch before it reaches the point of breaking (Sanyang et al., 2015). This measure was used to evaluate the films' flexibility and stretchability, which was important for storing, transporting, and handling (Sanyang et al., 2015) The highest elongation at break was reported in the C2 film. The film's stiffness can be quantified by a property known as Young's modulus, with a higher value indicating more rigidity.

Maintaining a consistent thickness during the casting process is crucial since any deviation can affect the mechanical and barrier characteristics of the film (Perazzo et al., 2014). In the current study, an equal quantity of film-forming solution was added to nonstick pans according to the film variety. Thus, an increase in thickness is observed by the increase in the concentration of the substance. A previous study (Edhirej et al., 2017) intended to evaluate the characteristics of films composed of thermoplastic cassava starch, with various percentages of cassava pomace as a reinforcement agent. The study discovered that the films had a thickness ranging from 0.30 ± 0.09 to 0.49 ± 0.07 when the cassava pomace was incorporated. The current investigation revealed a similar range of results. The C1 film obtained a slightly lower value of 0.27 ± 0.03, whereas the C3 film obtained a slightly higher value of 0.54 ± 0.05 compared to the previous study. The variation may arise from the varying quantities of cassava pomace (3% and 7%) utilized in the film development process, in contrast to the aforementioned study which used a consistent 5 g of cassava starch with various percentages of cassava pomace. The increase in material concentration led to a higher quantity of fiber particles being submerged in the polymer matrix, resulting in an increase in the film's thickness (Edhirej et al., 2017).

Table 1: Mechanical properties, Thickness, and density values of developed films

Film Type	Mechanical properties			Thickness (mm)	Density (g cm^{-3})
	Tensile Strength (Mpa)	Elongation at break (%)	Young's model (Mpa)		
C1	0.02 ± 0.01^b	0.00 ± 0.00^b	0.00 ± 0.00^b	0.27 ± 0.03^c	0.69 ± 0.06^b
C2	0.66 ± 0.29^a	14.13 ± 3.16^a	5.03 ± 1.45^a	0.36 ± 0.04^b	1.36 ± 0.03^a
C3	0.04 ± 0.01^b	0.41 ± 0.11^b	0.00 ± 0.00^b	0.54 ± 0.05^a	1.41 ± 0.11^a

Data represented as mean ± standard deviation. Different letters in the same column indicate a significant difference.

The density of biodegradable films varied between $0.6 9 \pm 0.06$ to 1.41 ± 0.11 g cm^{-3}. The lowest density was observed for the C1 film. Density is the quantitative measurement of mass divided by volume for a given material. When the quantity of cassava pomace was raised from 3% to 7%, there was an increase in the concentration of cassava pomace. The thickness also increased according to the material concentration, resulting in an increase in the volume of the substance. Nevertheless, the increase in the quantity of cassava pomace (from 3 g to 7 g) had a greater impact than the increase in thickness (from 0.27 ± 0.03 to 0.54 ± 0.05). The results showed that when the content of cassava pomace increased, there was a corresponding increase in the density values. Similar results were revealed in (Travalini et al., 2019) with cassava starch films reinforced with nanoparticles.

The moisture content of the films varies between $25.24 \pm 0.42\%$ to $12.34 \pm 0.44\%$ and the results are presented in Figure 2. The film exhibited a reduction in moisture content proportional to the quantity of the substance. The reason for this was an increase in solid content, causing the polymer network to be more dense. The C1 films, which had the lowest concentration of cassava pomace, demonstrated the highest moisture content. Conversely, the C3 films, which contained the highest concentration of cassava pomace, had the lowest moisture content. The study (Luchese et al., 2017) investigated the impact of varying moisture content on different ratios of cassava starch, including in the amounts of 20, 30, 40, 50, and 60 g kg^{-1}. The investigation documented moisture levels ranging from 12.2% to 33.0%. The cassava starch films containing the highest concentration exhibited the lowest moisture value, while those containing the lowest concentration demonstrated the highest value. The present investigation noticed a consistent pattern of outcomes within a comparable range.
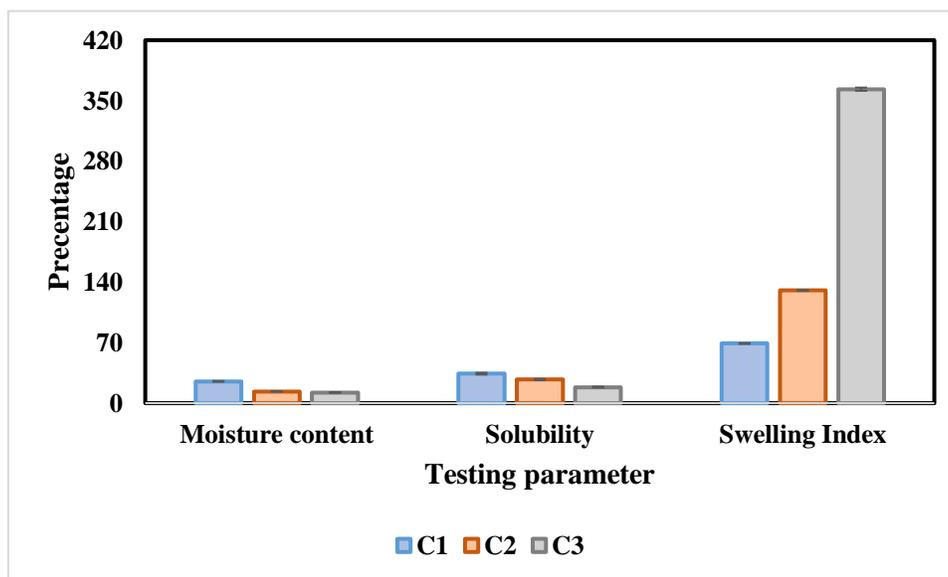

Figure 2: Moisture content, Solubility, and Swelling Index of developed films.

In the present study, water solubility ranged between $34.15 \pm 1.00\%$ and $18.589 \pm 0.49\%$. Water solubility declined as material concentration increased. The increase in the amount of cassava pomace material led to a subsequent increase in the presence of fibers in the films, as cassava pomace includes a greater concentration of fibers. It was assumed that the increase in film fibers formed a physical barrier that prevented the dissolution of biopolymers in water by interacting with the chains of the polymeric starch materials (Luchese et al., 2017). The study (Travalini et al., 2019) intended to evaluate the water solubility characteristics through the use of lignocellulose nanofibers derived from cassava bagasse. The study found a range of water solubility from 23.83 to 22.56 in the 4% cassava starch films. These films were reinforced with lignocellulose nanofibers derived from cassava bagasse, with percentages ranging from 0.65 to 1.3. The current investigation revealed elevated water solubility percentages for films based on 3% cassava pomace. The deviations observed may be attributed to factors such as the quantity and particle size of the cassava pomace incorporated, as well as the variations in the composition of the raw material. For instance, prior investigations combined cassava pomace and cassava starch when developing packaging material, whereas the current study exclusively utilized cassava

pomace. Nevertheless, both investigations reported a consistent decrease in water solubility as the concentration of cassava pomace increased.

The films with higher amounts of cassava pomace exhibited a higher swelling index. The swelling behavior of a polymeric system may be affected by the structure and interaction capabilities of the polymeric structure. As the medium permeated the polymeric matrix, the solvent-free polymer initiated to expand (López-Córdoba et al., 2017).

The microstructure perspective of the surface and cross-section was assessed using scanning electron microscopy (SEM). The surfaces of the films are depicted in. Figure 4 (A-C). It is clear that no surface defects, such as cracks or bubbles, appeared. The surface films had a heterogeneous surface due to the fibers in the cassava pomace. Similarly, these fibers can be visually observed in the prepared films. Figure 4 (a-c) depicts a cross-section of the films.
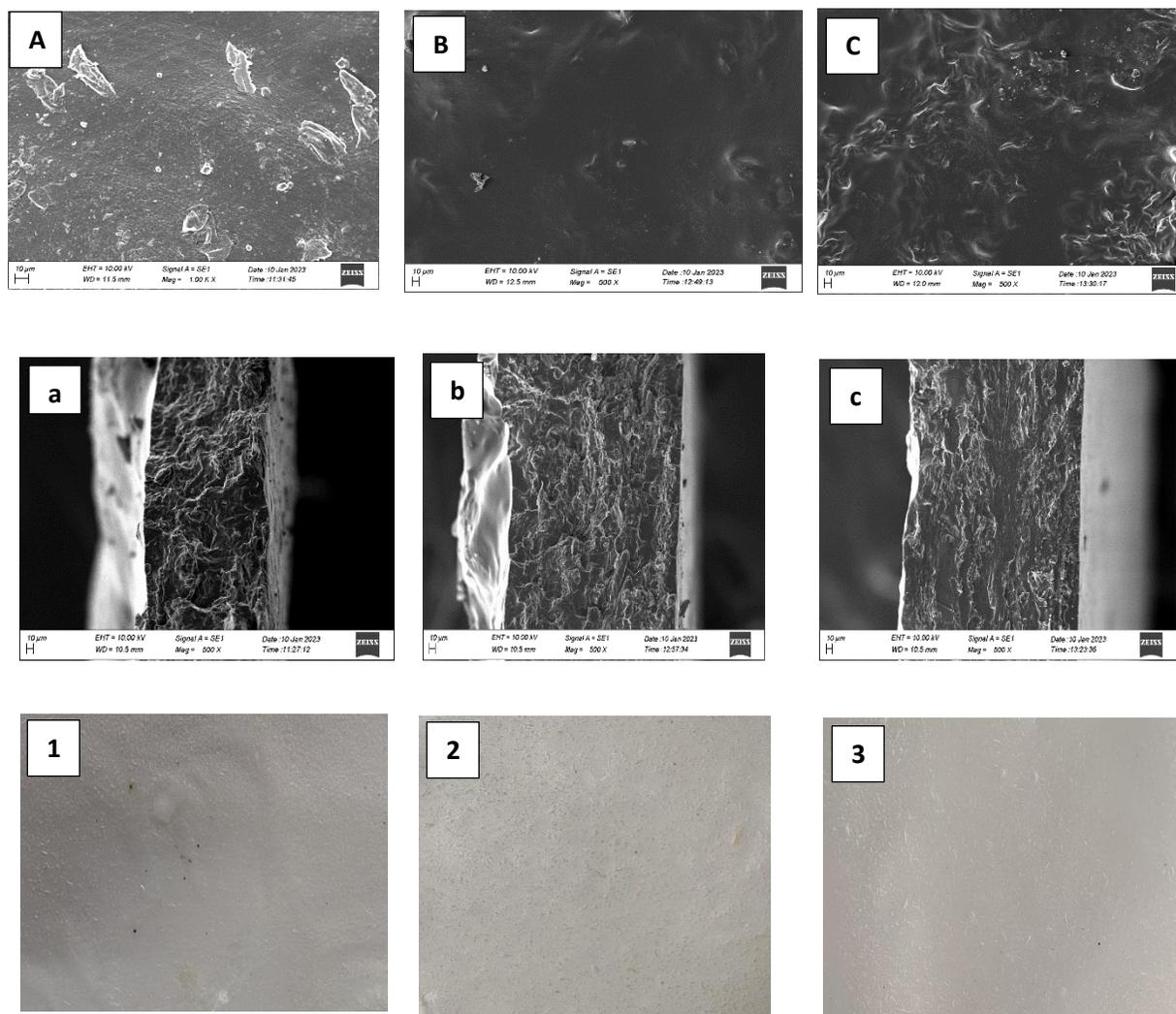
Figure 3: Scanning electron microscopic images of surface and cross-section and images of developed films. A, B, and C represent surface images, and a, b, and c represent cross-sectional images of C_1, C_2, and C_3 respectively. Visual images of C1, C2, and C3 films are demonstrated in 1,2,3 images respectively.

The molecular interactions that may occur between cassava pomace, water, and plasticizers (glycerol and sorbitol) throughout the film formation process can be examined using FT-IR (Zhai et al., 2020). The FT-IR spectrum of C2 film, which demonstrated greater mechanical properties, is depicted in Figure 5. A common wider peak was observed in all films at around 3300 cm^{-1}, indicating the stretching of O-H bonds. This stretching is caused by the film components, such as polysaccharides from starches, glycerol, sorbitol, and the remaining moisture content of the film (Costa et al., 2022; Navarro et al., 2016; Simona et al., 2021). The stretching of the C-H bond was responsible for the peak that appeared at 2925 cm^{-1} (de Carvalho et al., 2021). The peak at 1643 cm^{-1} was responsible for O-H blending, which is associated with starch-bound water molecules (Demash & Miyake, 2020; Prachayawarakorn et al., 2013). Peaks at 1350 cm^{-1} dominated the C-H vibration (Travalini et al., 2019b). Peaks at 1150 cm^{-1} and 1075 cm^{-1} are due to the stretching of C-O bonds within the C-O-H group (Edhirej et al., 2017). The peak around 990 cm^{-1} is due to the stretching of C-O in starch. The peaks between 990 and 1030 cm^{-1} represented the O-C stretch of the anhydroglucose ring (Edhirej et al., 2017). An observed frequency of around 920 cm^{-1} was attributed to the stretching of the C=O and C-O-C bonds in glucose within starch (Travalini et al., 2019).
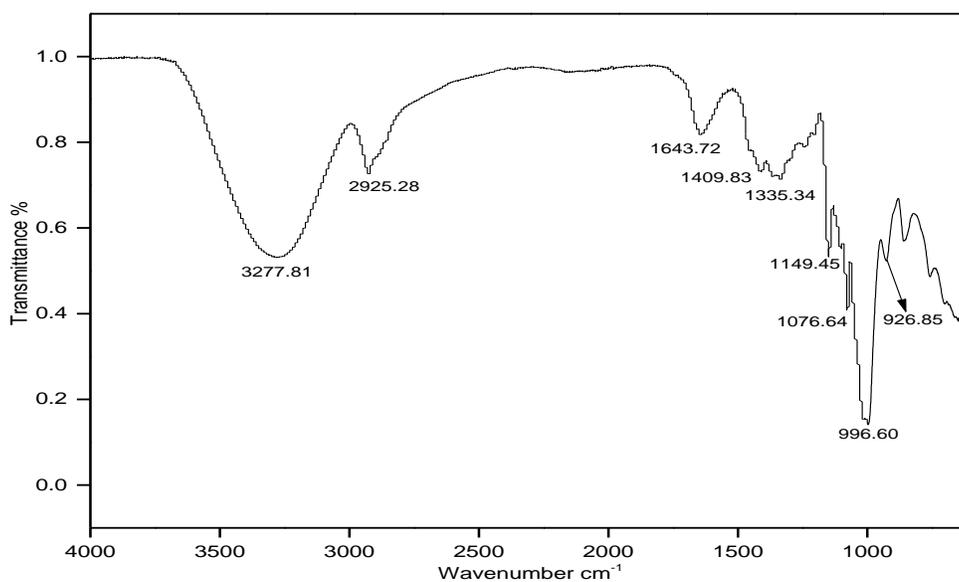
Figure 4: FT-IR spectrum obtained for C2 film

4.0 Conclusions

In the present study, cassava pomace-based films were successfully synthesized and characterized. In the morphological evaluations performed with an SEM, internal cracks were found in the C3 films. Similarly, internal cracks were found to have a negative impact on mechanical characteristics such as tensile strength. Film thickness, density, and swelling index increased as the amount of material increased, while solubility and moisture content reduced.

Acknowledgments

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