

Characterization of Edible Coating Formulated from Achi (Brachystegia Eurycoma) Hydrocolloid Incorporated with Ehuru (Monodora Myristica) Essential Oil

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Abstract

The development of biodegradable edible films has become a sustainable alternative to synthetic packaging in preserving food quality. This study investigated the physicochemical, mechanical, and structural properties of edible films formulated from Achi (Brachystegia eurycoma) hydrocolloid incorporated with Ehuru (Monodora myristica) essential oil. The films were prepared at different concentrations (1%, 3%, and 5%) using glycerol as a plasticizer. Characterization parameters included film thickness, moisture content, water solubility, swelling power, water vapor permeability (WVP), opacity, tensile strength, elongation at break, and structural analyses using thermogravimetric analysis (TGA), X-ray diffraction (XRD), scanning electron microscopy (SEM), and Fourier-transform infrared spectroscopy (FTIR). Results revealed that film thickness ranged from 0.059–0.119 mm, moisture content from 12.92–11.53%, and solubility from 31.06–18.86%, indicating improved compactness at higher concentrations. WVP reduced (0.26–0.05 g·mm/m²·kPa·h), demonstrating enhanced barrier properties. Tensile strength increased (9.52–11.50 MPa), and elongation at break ranged between 22.06–34.20%, showing good flexibility. FTIR spectra confirmed interactions between Achi polysaccharides and essential oil functional groups, while SEM revealed smooth, homogeneous surfaces at higher oil concentrations. These findings suggest that Achi hydrocolloid–Ehuru essential oil films possess excellent structural and functional integrity, supporting their potential as natural edible coatings for fresh produce preservation.

Keywords: Achi hydrocolloid, Ehuru essential oil, edible film, physicochemical properties, biopolymer, food preservation

Introduction

The use of edible coatings (ECs) has emerged as an effective and environmentally-friendly alternative to extend their shelf-life (Karaca *et al.*, 2014) and protect them from harmful environmental effects. Such films, applied as coatings, can create semipermeable barriers to gases and water vapour, reducing respiration and weight loss and maintaining the firmness of the fresh product while providing gloss to the coated products. In addition, coatings are able to act as carriers of a wide variety of functional ingredients, such as antimicrobials, antioxidants, anti-browning agents, nutrients or flavouring and colouring compounds (Fagundes *et al.*, 2015; Raybaudi-Massilia, 2016), enhancing food stability, quality and safety, thus promoting the coatings' functional performance beyond their barrier properties (Mariniello *et al.*, 2010).

An Edible Coating is a thin layer of edible material coated directly on a food surface, applied in liquid form (film-forming solution/dispersion) on the food, usually by immersing or spraying (Kang *et al.*, 2013). The film-forming solution or dispersion contains a polymeric material with

filmogenic capacity (Campos *et al.*, 2011). The efficiency and stability of ECs depend on their composition. Polysaccharides, including starch, cellulose, gums, alginates, chitosan and others, are naturally occurring polymers, widely used for this purpose (Hassan *et al.*, 2017), and are compatible with a broad range of functional compounds (Mehyar *et al.*, 2014) whose aim is to improve their properties. Hydrocolloid is a promising polysaccharide for food coating/packaging purposes, when taking into accounts its filmogenic capacity, ready availability and low cost. The development of edible and/or biodegradable films is an alternative for the total or partial substitution of synthetic polymers in the manufacture of packaging, a use which is coherent with concerns for environmental conservation and a healthier life style (Dick *et al.*, 2015).

The incorporation of glycerol, a common plasticizer, into the edible coating formulation is crucial for ensuring the flexibility and durability of the film. Plasticizers such as glycerol reduce the brittleness of the film by increasing its elasticity, which is essential for handling and storing cucumbers without damaging the coating (López *et al.*, 2019). Furthermore, glycerol improves the water vapor permeability of the film, balancing moisture retention with the need for controlled transpiration in fresh produce.

Achi seed (*Brachystegia eurycoma*) hydrocolloids, derived from the seeds of a leguminous plant native to West Africa, have shown great potential as a natural biopolymer for the production of edible coatings. Their gel-forming properties make them an attractive option for coating applications, where they create a semi-permeable barrier that controls respiration rates and reduces moisture loss in fruits and vegetables (Aremu *et al.*, 2015). Although Achi seeds are commonly used in traditional cooking, their application in food preservation, especially as an edible coating component, is still relatively novel and warrants further exploration. Similarly, Ehuru (*Monodora myristica*), known for its aromatic properties, yields essential oil rich in antimicrobial compounds such as eugenol, α -pinene, and linalool (Owolabi *et al.*, 2014). These natural compounds can be incorporated into edible coatings to enhance their preservation efficacy. Combining the film-forming capability of Achi hydrocolloid with the antimicrobial activity of Ehuru essential oil may provide a synergistic effect, improving the shelf life and safety of fresh produce like cucumber during storage at ambient temperature.

2 Material and Methods

Materials

Achi seeds and Ehuru seeds used for this research work were purchased from a local market (Eke Awka) in Awka, Anambra State. Glycerol (99.5%) and other chemicals used were of analytical grade and were obtained from the Head Bridge chemical market Onitsha, Anambra State. The equipment and other materials were obtained from Alpha Research Laboratory Awka.

Methods

Sample Preparation of Achi and Ehuru

One kilogram of dehulled Achi seeds were sorted and milled using CORONA attrition milling machine (Landers YCIA, S. A. Colombia) to generate Achi flour. The flour was sun dried and sieved using a 52 μ m mesh size (British Standard). The fine flour obtained was defatted using n-hexane and then air dried. The air-dried flour sample was dissolved in distilled warm water and hydrated continuously using a magnetic stirrer. It was kept for 6hrs, centrifuged at 2500rpm for 5mins. The dried sample was cooled in the desiccator and pulverized using a ceramic mortar and stored in air tight container (Nwakaudu *et al.*, 2017). Ehuru seed were sorted and milled using CORONA attrition milling machine (Landers YCIA, S. A. Colombia) to generate Ehuru

flour. The powdered sample was weighed using a digital weighing balance to determine the weight.

Extraction of Ehuru *Monodora myristica* oil

The *Monodora myristica* oil was extracted using soxhlet apparatus with N-hexane as the solvent (AOAC 2010). 300 ml of normal Hexane was poured into a round bottom flask and 100 g of ground sample were weighed and placed on a filter paper. The sample folded on the filter paper was then placed in the thimble and inserted into the centre of the extractor. The soxhlet heated at 60°C. This was allowed to continue for 60 minutes. It was then removed from the tube, dried in the oven, cooled in the dessicator and weighed again. The solution left in the round bottom flask was a combination of oil and solvent. The solvent was removed through distillation and oil recovered, weighed and recorded.

Characterization of Edible Films

Film thickness

The thickness of films was determined by using an electronic gauge (Multicheck FE, SODEXIM, France) with precision ranges between 0.1 and 1% as a function of thickness value (0–100 mm or 0–1000 mm). Ten replicates were made on each film-making (Ranganna, 2003)

Moisture Content

The AOAC (2010) method was used. 5g of the sample were weighed into clean, dried and pre weighed crucibles. The crucibles and their contents were dried in the moisture extraction oven at 105°C for 1 hour. The samples were then removed from the oven, cooled and reweighed. The samples were again put back into the oven and dried until a constant weight was obtained. This analysis was carried out in triplicate and the average value was recorded as moisture content.

$$\% \text{ Moisture content} = \frac{\text{Initial weight of sample} - \text{weight of oven dried sample}}{\text{Initial weight of sample}} \times 100$$

Solubility in Water

The water solubility of film samples was determined according to the method proposed by Nogueira *et al.*, 2015, with modifications. Film samples of 20 mm (in diameter) were cut, weighed, and dried in a hot air oven at 105 °C for 24 h. The dried samples were stored in a desiccator to stabilize for 15 min and weighed. The dehydrated samples were immersed individually into 50-mL beakers filled with distilled water and maintained under slow agitation (75 rpm) for 24 h at 25 ± 2 °C in an isothermal reciprocal water bath shaker (SB 302, Double Eagle Enterprise Ltd., Taiwan). Then, insoluble samples were removed and dried at 105 °C for 24 h to determine the final dry mass. Solubility in water were calculated according to equations below

$$\text{Water Solubility (\%)} = \frac{W_i - W_f}{W_i} \times 100$$

Where:

W_i' is the initial weight of dry film before immersion (g)

W_f' is the weight of final insoluble dry films (g).

Water Vapor Permeability (WVP)

The water vapor permeability (WVP) was determined according to the method proposed by Abdillahi and Charles (2013). Films of known thickness were tied with rubber bands over the mouth of glass cups (depth = 70 mm, diameter = 30 mm) containing 10 g of dry silica gels.

The glass cups were weighed and placed inside the desiccator with 100 mL of distilled water and a humidity and temperature sensor. The air inside the desiccator was removed and placed inside a digital humidity controller (RH = 50%, temperature = 25 °C). The weight of glass cups was measured, and relative humidity and temperature was recorded at 24-h intervals for 7 days. Water vapor permeability values are expressed in g·mm/m²·kPa·h and were calculated using:

Water Vapour Permeability (WVP) Formula:
$$\frac{W \times L}{A \times t \times \Delta P}$$

Where:

WVP = Water Vapour Permeability (g·mm/m²·kPa·h)

W = Amount of water vapor gained by the cup (g)

L = Thickness of the film (mm)

A = Area of film exposed to vapor transmission (m²)

t = Time of exposure (h)

ΔP = Partial pressure difference of water vapor across the film (kPa)

Film Transparency

Film transparency will be determined in a UV-Vis spectrophotometer. The film samples will be fixed in the cuvette such that the light beam passed through the film. The transparency will be determined at 600 nm in triplicate (Han and Floros, 2017) and the values, expressed in percentage, calculated from the following Equation:

Transparency (%) = Abs Thickness (600 nm)

Mechanical Properties

Tensile strength at breaking (TS) and percentage of elongation (%E) were measured using a Universal Testing Instrument (Instron UTTI 1122, Instron Ltd.) with a 5 kN load cell. Samples were stored at 57% RH over the sodium bromide saturated solution for 10 days at 25 °C prior to measurement. A total of 20 samples for each type of films were stretched at a constant rate of 100 mm min⁻¹. The effective dimension of the film was 20 x 60 mm² before the stretching.

Thermogravimetric Analysis

Thermal analysis was carried out using Differential Scanning Calorimetry (DSC). A heating cycle with a step of 5 °C is performed and simultaneously the heat flow is obtained. The temperature range for the measurements was selected based on research of the literature, with each coating being subjected to a different range. More specifically for starch, a range of 0 to 250 °C was chosen; The samples were stored for 24 h at 25 °C and 0% RH in order to minimize moisture content. The glass transition temperature T_g is identified from the heat flow-temperature diagram. The enthalpy of crystallization is derived from the area under the crystallization peak in the thermogram, which is calculated using integration software provided by the DSC instrument. This peak corresponds to the heat released or absorbed during the crystallization process.

Crystallinity (X-ray Diffraction)

An X-ray diffractometer (XRD, D8-ADVANCE, Bruker, Karlsruhe, Germany) was used to analyze the crystal structure of film. The samples were tested in dry state, so the powder wafer method and Cu target radiation were adopted. Powder X-ray diffraction (XRD) experiments were performed between 2° and 80° (2θ) with a step size of 0.02° and a measuring time of 0.8 s per step.

Microstructure Properties (SEM)

Film microstructure was observed by environmental scanning electron microscopy (ESEM, Phillips XL 30 ESEM, Japan). A 5x10 mm² film was fixed on the support using double side adhesive tape, with an angle of 90 to the surface, which allowed observing the film cross-section. No particular film preparation was necessary. Films were observed at different magnifications from x100 up to x3000.

FTIR analysis

FT-IR spectra were recorded using the IRTracer-100 (Shimadzu, Japan) equipped with an attenuated total reflectance (ATR) accessory. The samples were placed on a measurement plate and measurements were taken at 40 scans and at a resolution of 2 cm⁻¹ with Happ-Genzel apodisation. After each measurement, the plate was carefully cleaned from any previous residues by wiping using acetone and hexane and dried with a soft tissue before filling in with the next sample. Proprietary Lab Solutions software (Shimadzu, Japan) was used for FTIR data collection and processing. All spectra were recorded from 4,000 to 400 cm⁻¹ and read as absorbance in triplicate before taking the averaged value.

Statistical analysis

All data obtained were subjected to the proper statistical analysis using the MSTAT statistical software and the treatments means were compared by using the LSD at 0.05 level of probability as described by Snedecor and Cochran (2013).

Result and Discussion

Physicochemical Properties of Achi-Ehuru Edible Film

Thickness

In this study, the increases in film thickness significantly corresponded with the increases in Achi flour concentration ($p < 0.05$). Thickness is an essential physical property of films and coatings because it affects color, mechanical characteristics and barrier properties towards the permeation of water vapor and other gases. Therefore, this parameter can influence the packaged food product's shelf life. Film thickness, can change due to the amount of solid content of the films after dehydration of the film. Increasing the concentration of Achi flour, as well as, the incorporation of Ehuru seed oil increased film thickness. Moreover, the film thickness increased significantly ($P < 0.05$) with increase in the amount of essential oil. This phenomenon can be attributed to the inclusion of Ehuru essential oil into the Achi film matrix which changed material density and the amount of the dry compounds since the same volume of film solution was poured at each Petri dish.

Similar results were reported for quince seed films incorporated with oregano essential oil (Jouki *et al.*, 2014), The thickness values obtained in this study (0.05–0.12 mm) fall within the acceptable range for edible coatings (0.04–0.15 mm), making them suitable for food application without affecting product appearance.

Moisture Content

The moisture content of the films decreased from 12.92% to 11.53%, showing a significant reduction ($p < 0.05$) as the concentration increased. This can be attributed to the stronger polymer network formed at higher hydrocolloid concentrations, which reduces the number of free hydrophilic sites available for moisture absorption. Comparable observations were reported by Campos *et al.* (2011) in composite films, and Akhtar *et al.* (2013) in starch–glycerol films, where moisture content decreased as polymer concentration increased. Lower

moisture content improves film stability, making it less susceptible to microbial attack and degradation during storage.

Water Solubility

Film solubility reduced significantly ($p < 0.05$) from 31.06% to 18.86%. The decrease in solubility indicates enhanced water resistance due to increased molecular interaction between the Achi hydrocolloid matrix and hydrophobic constituents of Ehuru oil.

A similar reduction in solubility was reported by Sapper and Chiralt (2018) for starch-based coatings containing essential oils and by Fabra *et al.* (2009) for polysaccharide–lipid films. This suggests that the inclusion of hydrophobic oil components contributes to forming a more compact and less permeable structure. Reduced solubility is desirable in edible coatings as it enhances durability and moisture barrier function.

Optical Properties (Opacity)

Opacity decreased slightly from 0.42 to 0.29 as polymer concentration increased, indicating more transparent films. Transparency is a desired characteristic for edible coatings as it ensures the product's visual appeal is maintained. Hypothetically, the lower the starch content or film thickness, the higher the amount of UV light transmitted. Comparable opacity values (0.25–0.40) were reported by Yin *et al.* (2019) in hydrocolloid-based films with essential oils. The slight reduction in opacity may be due to uniform dispersion of essential oil droplets within the polymer matrix, reducing light scattering (Atarés & Chiralt, 2016).

Water Vapor Permeability (WVP)

The WVP values decreased significantly ($p < 0.05$) from 0.26 to 0.05 g·mm/m²·kPa·h as concentration increased. The reduction in WVP suggests improved barrier performance against moisture transfer. The presence of hydrophobic essential oil components likely filled micropores in the hydrocolloid matrix, creating a tortuous path for water vapor diffusion.

This observation agrees with Perdonés *et al.* (2012), who found that chitosan–lemon essential oil films had reduced permeability compared to control films. Likewise, Bonilla and Sobral (2016) reported that polysaccharide–lipid blends showed decreased WVP due to hydrophobicity enhancement. The low WVP obtained here makes the film suitable for minimizing weight loss in fresh produce such as cucumber.

	Thickness (mm)	Moisture (%)	Water Solubility (%)	Opacity (A/mm)	WVP(g mm/m ² KPa) Day
A	0.059 ± 0.0016 ^c	12.92 ± 0.39 ^a	31.06 ± 0.24 ^a	0.42 ± 0.020 ^a	0.26 ± 0.004 ^a
B	0.092 ± 0.0014 ^b	12.18 ± 0.27 ^b	24.93 ± 0.14 ^b	0.40 ± 0.036 ^a	0.19 ± 0.002 ^b
C	0.119 ± 0.0050 ^a	11.53 ± 0.75 ^c	18.86 ± 0.22 ^c	0.29 ± 0.019 ^b	0.05 ± 0.001 ^c

Table 1: Physicochemical properties of Achi-Ehuru edible film

Results are means $\pm$ SD for three determinations

Figures in the same horizontal row that share the same superscript are not significantly different from control at $P > 0.05$

Key:

Sample A -1% Achi+1% Ehuru Oil

Sample B- 3%Achi+3% Ehuru Oil

Sample C-5% Achi+5% Ehuru Oil

Mechanical Properties

Tensile strength increased significantly from 9.52 MPa to 11.50 MPa ($p < 0.05$) as the concentration increased. This improvement can be linked to enhanced polymer chain interaction and uniform distribution of essential oil, which contributes to matrix reinforcement. Tensile strength in edible films is one of the important properties in packaging and coating of products, as it affects the flexibility of the film; high tensile strength would give lower flexibility and low tensile strength would give higher flexibility (Krishna *et al.*, 2012). Edible film with high tensile strength would be more suitable for products requiring stiffer packaging, while edible film with lower tensile strength would be more suitable for products requiring flexibility in its packaging (Katili *et al.*, 2013). The strength of the packaging should be specific and/or complement the properties of the food product.

The elongation at break ranged from 22.06% to 34.20%, increasing with polymer concentration. The higher elongation values indicate better flexibility and elasticity, likely due to effective plasticization by glycerol and molecular compatibility between the Achi and Ehuru components. The elongation values increase with increasing essential oil concentration due to the decreasing of the intermolecular bonds between the polymer chains and the formation of weaker hydrogen bonds which led to increasing elasticity (Sanyang *et al.*, 2015).

Sample	Tensile Strength (MPa)	Elongation at Break (%)
A	9.52 ± 1.10^c	22.06 ± 4.34^c
B	10.66 ± 1.00^b	32.65 ± 1.15^b
C	11.50 ± 0.44^a	34.20 ± 3.97^a

Table 2 Mechanical properties

Thermogravimetric Analysis

The thermogravimetric curves of the three edible films are showed in fig 4.3. The curves exhibited decreasing weight patterns and maximum decomposition temperatures of the edible film. Thermal properties indicate the heating and cooling transitions of packaging materials, particularly during freezing and pasteurization. According to Suriyatem *et al.*, [2023]. The first two stages were related to the vaporization of water molecules (35 to 150 °C). The final stage (279–315 °C) corresponded to the degradation of carbonaceous residues formed during the second stage, which occurred with the complete oxidation of these materials. The disintegration trend was similar in all the samples, although a significant shift toward lower temperatures was observed in the sample C; hence, recording lower weight loss. It started to disintegrate between

313 and 315 °C, while the Sample A film disintegrated at 279 °C. At about 550 °C, the final weight, which corresponded with the release process of the minerals and char residue, presented the stability of the films.

Achi Ehuru film samples displayed lower weight losses (~35.6%, 33.7%, and 36.4%, respectively) indicating their higher thermal stabilities. Similar findings were observed when cassava starch concentrations were increased in composite Cassava/rice-starch-based films. Nevertheless, Qin *et al.*, [2015] claim that for films to be recognized as suitable for food packaging material, they must exhibit stability when environmental temperatures are lower than 100 °C; hence, all edible films were thermally stable and could potentially be used as packaging material for perishable and/or cooled foods during storage and transportation.

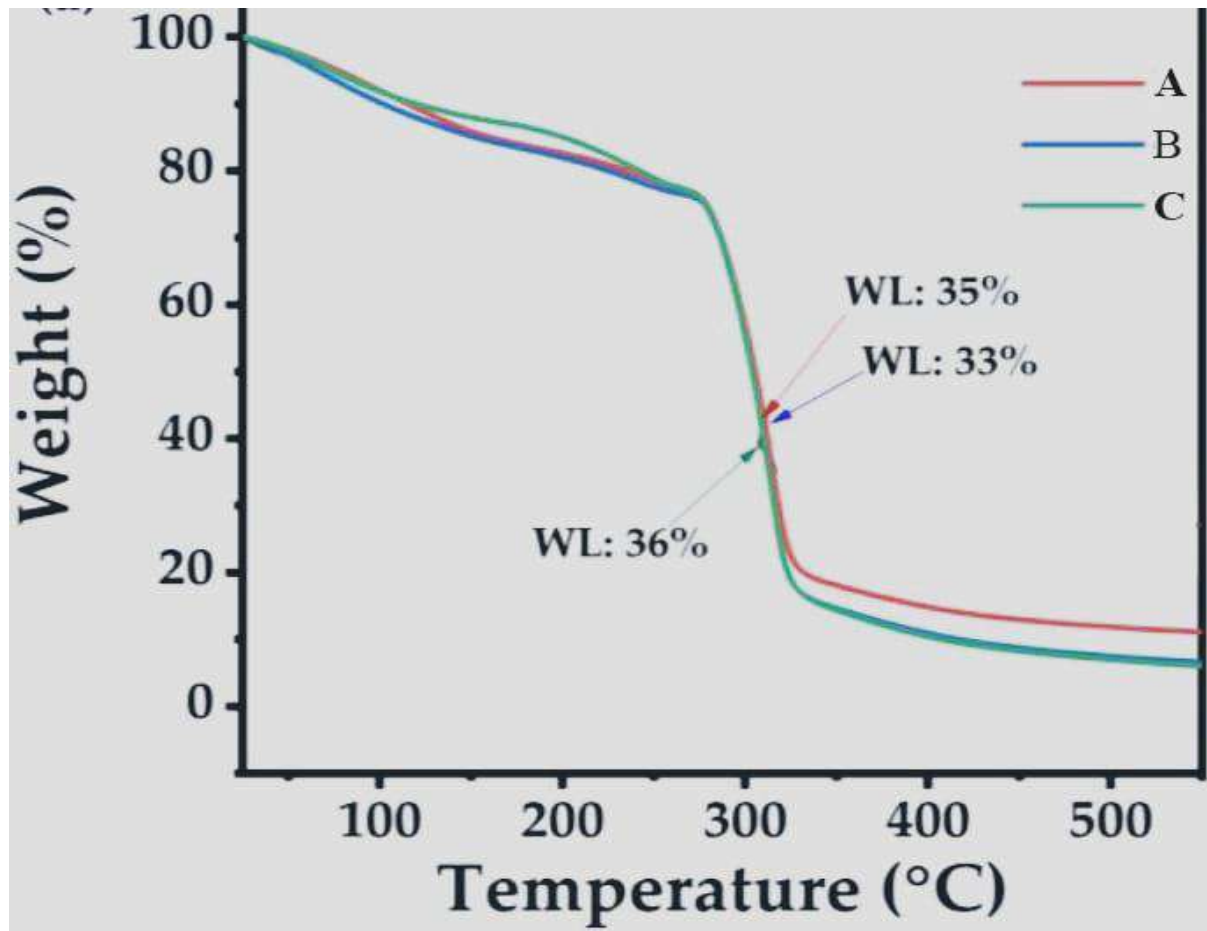


Fig 1. Thermogravimetric analysis (TGA)

Crystallinity (X-ray Diffraction)

The X-ray diffraction patterns of the three edible films produced are shown in fig 2. From the result, edible films displayed broad crystalline peaks at around $2\theta = 19.00^\circ$ (A), 19.13° (B), and 19.35° (C). The edible films exhibited similar diffractograms and depicted amorphous characteristics with small crystalline fractions 8.2% which were marginally different and interpreted as crystallinity being unaffected by ehuru oil. The amorphous character of the starch films was likely induced during the casting process (thermal treatment), where intermolecular hydrogen bonding between starch molecules was disrupted (thereby inhibiting starch retrogradation) by glycerol (plasticizer), which increased the chain mobility of the starch molecules (Hornung *et al.*, 2018). A previous study by Basiak *et al.*, (2017) similarly reported that low crystallinity exhibited smooth surface structure, which was confirmed by SEM

analysis. Moreover, films depicting amorphous patterns are characteristically flexible, soft, and workable; hence, all films were considered suitable for application in food packaging.

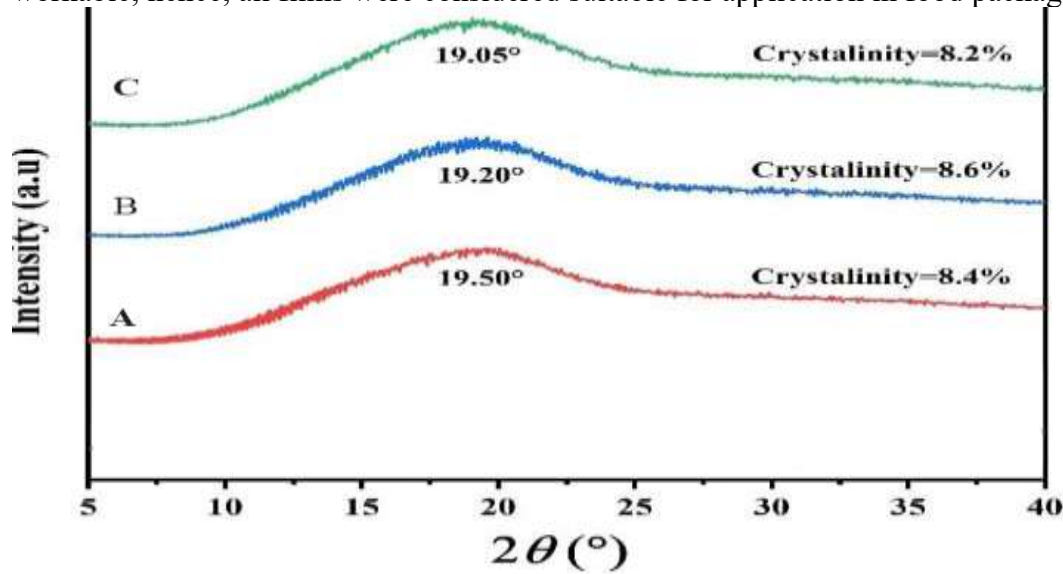
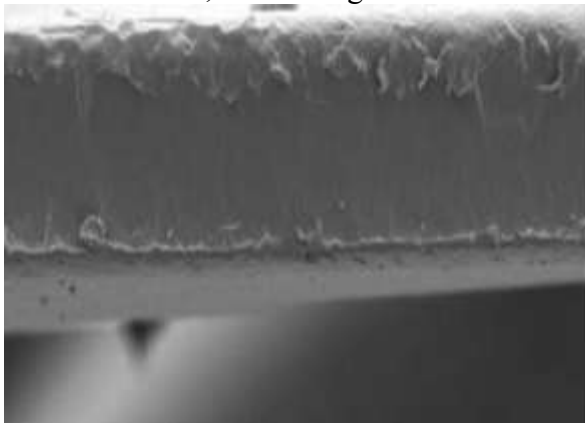


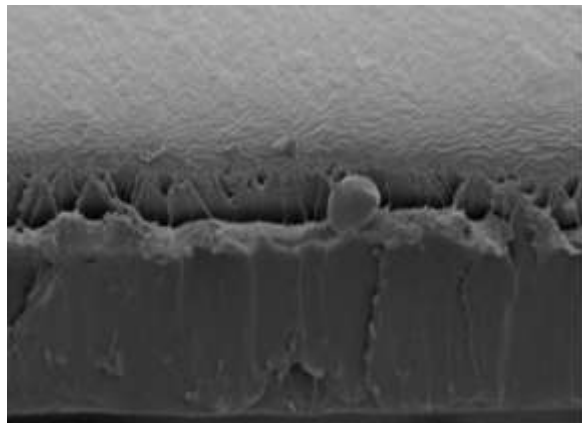
Fig 2: X-ray diffraction (XRD) of edible films

Microstructure Properties (SEM)

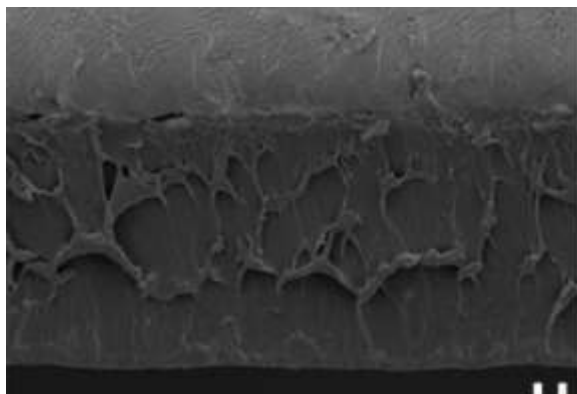
The surface morphology of all the edible films as shown in plate 1 showed the film had no cracks, pores, and bubbles, and displayed homogenous surfaces, thus indicating the effectiveness of the casting technique. Moreover, the sample A and B appeared smooth; however, sample C exhibited a wrinkled surface, which indicated the higher starch and oil concentration, which rendered the resin viscous and difficult to cast. This was influenced by the ingredients of the edible film being evenly mixed, both filler and plasticizer are added, judging from the highest mechanical test. Comparable morphologies were reported in starch–oregano oil films by Rhim and Wang (2013). The compact structure supports the low WVP values observed, confirming the micro structural basis for barrier effectiveness.



SAMPLE A



SAMPLE B

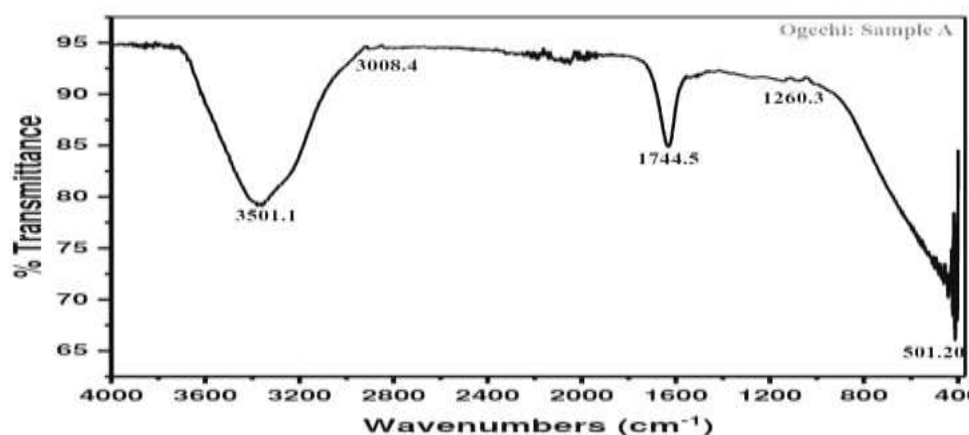


SAMPLE C

Plate 1: SEM images of edible film

FT-IR (Fourier Transform Infra Red)

Fourier Transform Infrared Spectroscopy (FTIR), carried out on the edible film from Achi and Ehuru oil provided detailed molecular information about the composition of various materials. This analytical technique gave clear information regarding the functional groups characterizing each of the components used in the coatings. The FTIR analysis of the coatings is presented in fig 3. The results of the FT-IR analysis provide a spectrum with absorption in the area of 3361.17 cm^{-1} indicates the presence of a hydroxyl group (OH) or -NH group, also spectrum with an absorption of 3297.98 cm^{-1} indicating the presence of a hydroxyl group (OH) and in the area of wave number 2931.95 cm^{-1} derived from -glucose, Also a spectrum with absorption in the region of 3297.00 cm^{-1} indicating the presence of a hydroxyl group (OH) derived from glycerin and absorption in the region of wave number 2880.17 cm^{-1} indicating the presence of CH aliphatic, and 2939.52 cm^{-1} indicating the presence of an alkane group (CH) (Kumar *et al.*, 2020) . These characteristic peaks give information on the nature of compounds in the edible film owing to the presence of Achi flour and ehuru seed oil, and thus, they provide valuable information with respect to the secondary structure of the material used in the coating .These findings are similar to those reported by Bonilla and Sobral (2016) and Yin *et al.* (2019), confirming that essential oils interact with polymer matrices to enhance film compactness and structural integrity.



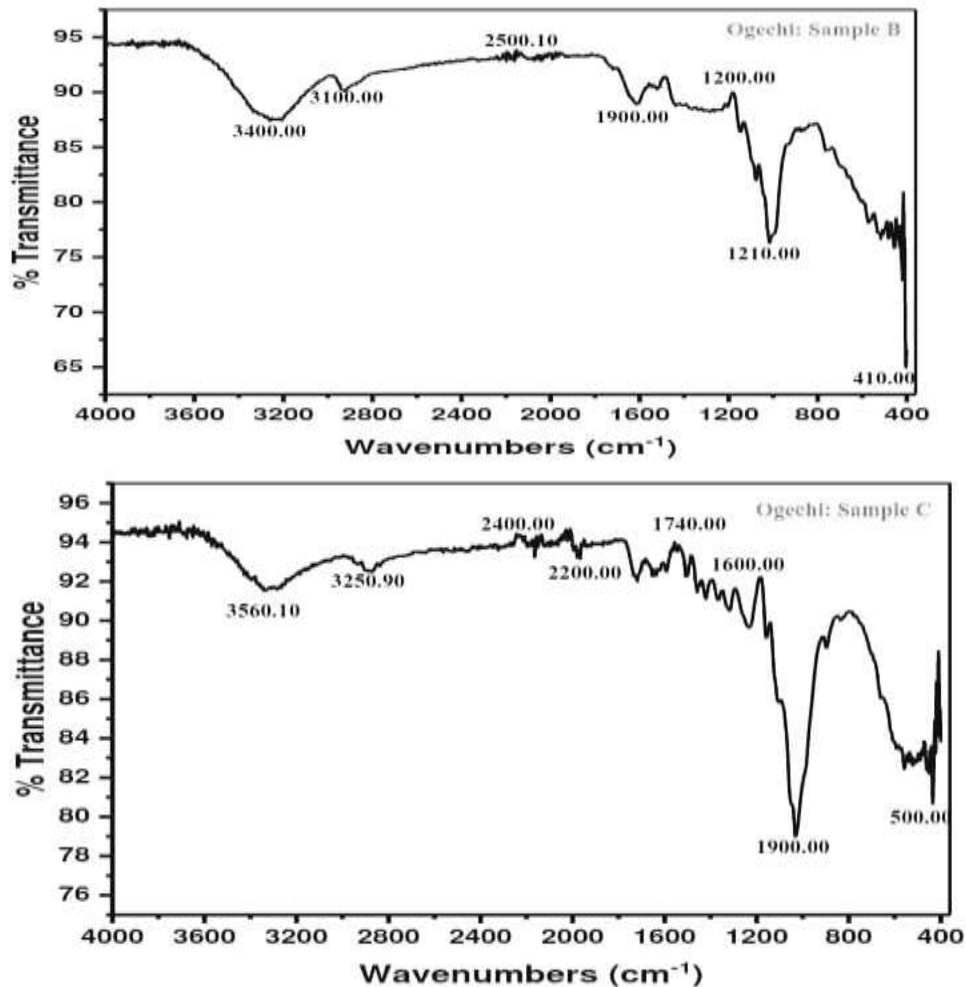


Fig 3. FTIR of Achi-Ehuru oil Edible Film

CONCLUSION

This study established that films produced from *Achi* hydrocolloid and *Ehuru* essential oil possess desirable physical, mechanical, and structural characteristics suitable for use as edible coatings in food preservation. Increasing polymer concentration improved the film's thickness, tensile strength, and water vapor resistance, while reducing solubility and swelling. The incorporation of *Ehuru* oil enhanced the film's compactness, thermal stability, and potential antimicrobial functionality. These findings highlight the potential of indigenous Nigerian materials in the development of eco-friendly, biodegradable packaging systems for the food industry.

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