



Analyzing the Effect of Drying Temperature and Storage Conditions on Properties of Amaranth Starch-based Biodegradable (Edible) Films

Narender Kumar Chandla^{1,2}, Gurjeet Kaur², Sukhcharn Singh¹, D.C. Saxena¹,
Sunil Kumar Khatkar², Nitin S. Wakchaure², Gajanann P. Deshmukh²

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ABSTRACT

Background: Edible films are emerging as a sustainable alternative to synthetic packaging, enhancing food preservation while minimizing environmental impact. Their functional properties depend on extrinsic factors like storage conditions, influencing quality and shelf life. Understanding drying kinetics is essential for optimizing production and ensuring consistency. Incorporating CMC with amaranth starch improves film transparency and structure, while optimizing drying parameters enhances quality. This study investigates the drying characteristics and storage behavior of CMC-reinforced amaranth starch films to support large-scale commercialization.

Methods: Biodegradable edible films were developed using *Amaranthus hypochondriacus* Durga and CMC, forming transparent gel solutions. Filmogenic solutions with amaranth starch, CMC, and glycerol were prepared, cast, and dried. Drying trials at 45°C, 55°C, and 65°C identified 55°C for 13.5 hours as optimal for achieving desirable film quality and functionality.

Result: Our investigation enabled us to analyze the functional properties of edible films under varying temperature and humidity conditions. The functional properties of the edible films were examined after 90 days of storage at 30°C and 50% RH and 45°C and 75% RH. The study of functional properties and SEM analysis can help to standardize the production process parameters for commercial manufacturing of biodegradable edible films in the future.

Key words: Amaranth, Drying, Edible films, Starch, Storage.

INTRODUCTION

Amaranth grain is a nutritious food source with unique starch properties that can be used in the development of biodegradable and edible films for packaging (Silva-Sanchez *et al.*, 2008; Hoang *et al.*, 2019).

Understanding the scientific properties of amaranth starch could transform the starch industry and make it a valuable ingredient in food and allied processing industries (Singh *et al.*, 2014). Researchers recognize the potential of starches to develop biodegradable and edible films for packaging due to environmental concerns and food interaction with polymeric packaging. Drying techniques such as convective drying can be used to produce clear edible films with desired properties, reducing ecological problems caused by polymeric waste at 40-65°C (De Moraes *et al.*, 2015). The characteristics of the films are influenced by the raw material, formulation and drying process (Saini *et al.*, 2012). Films prepared by thermal heating and cold gelatinization from non-conventional starch sources possess good thermoplastic properties.

Edible films made from starch are popular due to their abundance, affordability and biodegradability (Al-Hassan and Norziah, 2012). However, they have poor mechanical properties and require additives and standardization of drying time and temperature to achieve sufficient strength and good quality films. Standardizing drying rates is a critical factor in scaling up starch-based film production, as it

¹Department of Food Engineering and Technology, Sant Longowal Institute of Engineering and Technology, (Deemed University), Longowal-148 106, Punjab, India.

²College of Dairy and Food Science Technology, Guru Angad Dev Veterinary and Animal Sciences University, Ludhiana-141 001, Punjab, India.

Corresponding Author: Narender Kumar Chandla, College of Dairy and Food Science Technology, Guru Angad Dev Veterinary and Animal Sciences University, Ludhiana-141 001, Punjab, India. Email: narenderchandla1@gmail.com

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affects the overall economics of film manufacturing. Along with coating applications on fresh produce. High temperatures can lead to brittle structures and caution must be taken in increasing drying rates (Nascimento *et al.*, 2012; Sukhija *et al.*, 2016; Sharma *et al.*, 2016; Jyothsna and Nair Reena, 2024). Additionally, extrinsic factors like storage conditions can influence the properties of edible films, which can affect the quality and shelf life of food products. By studying drying conditions and behavior, scientists and engineers can

scale up and accelerate the production of edible films. Adding CMC as a hydrocolloid with amaranth starch can result in clear films and standardizing drying time and temperature improves their quality.

Therefore, objective of this study was to analyze the drying characteristics and storage behavior of CMC enforced amaranth starch based edible films. Developed edible films were evaluated for various functional properties at varied drying time and temperature conditions.

MATERIALS AND METHODS

Material

Amaranth grain namely *Amaranthus hypochondricus* Durga was procured from National Bureau of Plant Genetic Resources (NBPGR), regional station, Phagli, India. CMC and analytical reagent (AR) grade glycerol were purchased from Merck Specialities Pvt. Ltd., India.

Physicochemical analysis

For each parameter, samples were analyzed in triplicate. The amylose content was evaluated by the method given by Morrison and Laignelet (1983) using iodine/potassium iodide solution (I₂-KI) and then, recording absorbance at 635 nm. Swelling power was determined according to procedure used by Adebooye and Singh (2008) as modified by Unnikrishnan and Bhattacharya (1981). In brief, approximately 500 mg of the sample was cooked in water, centrifuged and the supernatant was collected for evaporation and drying to determine swelling power, with residue weighed for analysis. Particle size distribution (PSD) was measured using laser diffraction particle size analyzer Malvern Mastersizer 3000 (Malvern Instruments Ltd, Worcestershire, UK). Water binding capacity was determined using Medcalf and Gills (1965) method involving agitation of a 5 g starch suspension, centrifugation, draining and weighing of wet starch. The pasting properties of the starch powder were determined by Rapid Visco Analyzer (RVA 4500, PerkinElmer, USA). A starch slurry (3 g starch in 25 mL water) was analyzed using an RVA system, heating from 50 to 95°C and cooling back to 50°C within 12 minutes, with stirring at 160 rpm to estimate peak viscosity, trough, setback viscosity, final viscosity, pasting temperature and peak viscosity time. The Differential Scanning Colorimetry (DSC-821, Mettler Toledo, Switzerland) was used to measure thermal properties and peak temperature was evaluated using method by Sandhu and Singh (2007), where 3.5 mg starch (70% water suspension) was sealed in aluminum pans, heated from 20°C to 100°C at 10°C/min and calibrated with indium and gelatinization parameters were determined.

Drying method of edible films

Edible films were prepared using a modified method by Valderrama and Rojas (2014). Amaranth starch of 10 g per 100 ml of double distilled water and CMC (0.17% w/v) were dispersed in water and heated, then glycerol (2.5/ 100 g of amaranth starch) was added and the resulting solution

was poured into a tray and dried in an oven (Fig 1). The drying kinetics was studied at different temperatures (45, 55 and 65°C) by periodically weighing samples until the equilibrium moisture content was reached. The effects of process variables on drying kinetics were also investigated.

Storage conditions of edible films

In this study, an accelerated storage study was conducted at specific temperature and relative humidity conditions of 30°C and 50% RH and 45°C and 75% RH, respectively.

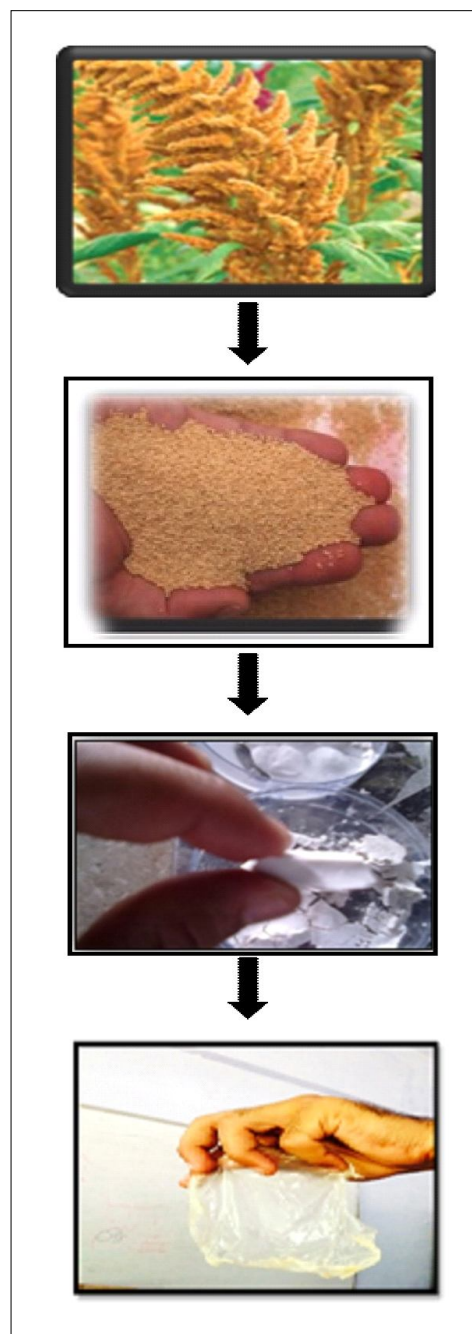


Fig 1: Stepwise process for developing amaranth starch (AHD)-based edible films.

The properties of the edible films were studied for three months under each set of conditions. The functional properties (thickness, tensile strength, solubility, water vapor permeation, transparency and surface morphology) of edible films were recorded every month. The thickness of the films was determined with a manual micrometer (Mitutoyo 2046F) with an accuracy of $\pm 1\mu\text{m}$. Tensile strength (TS) was determined using a texture analyzer (Model; TA.XT2i, SMS, Surrey, England) with a tensile grip attachment, where rectangular strips (5 mm $\times$ 50 mm) were tested at a 20 mm grip separation and a speed of 1 mm/s. Films solubility in water was determined according to the method proposed by Romero-Bastida *et al.*, (2005). Water vapor permeability of the films was determined at room temperature using a modified ASTM E96-00 procedure. Transparency was determined using a UV spectrophotometer (ID 5000 HACH, USA), measuring absorbance between 230-800 nm. Small film samples were placed in a micro plate without air gaps, with three replicates per film type and blank measurements taken for calibration. The transparency of the film was calculated by dividing A_{600} with x , where A_{600} represents absorption at 600 nm and ' x ' denotes film thickness. Surface morphological characteristics of film samples were analyzed by scanning electron microscope (SEM) (JEOL, JSM 6610-LV, Tokyo, Japan). Composite edible film samples were equilibrated for 24 hours in a desiccator, mounted on a grid with copper tubes and coated with gold for 1 minute using a 1 kV voltage. The samples were then observed under a scanning electron microscope (JEOL-JCM-6000, Japan) at magnifications ranging from 1000x to 7500x.

Statistical evaluation

All the analysis was determined in the triplicates and subjected to one- and two-way analysis of variance (ANOVA), followed by Duncan's by Mini Tab Statistica7. (State soft Inc., OK, USA). Graphs were plotted by using GraphPad Prism Software (Version 8, GraphPad, USA).

RESULTS AND DISCUSSION

Physico-chemical properties of amaranth starch

The amylose content of amaranth starch (AHD) was found to be 3.43%. This affects most of its physiochemical properties. AHD starch has a small average granule size of 2.852 μm , which affects its tendency to absorb water. The swelling power (SP) of AHD starch is 10.29%, which is related to its water binding capacity (WBC) of 199.47%.

The WBC of AHD starch is higher than that of other cereal starches such as wheat (0.44-0.76%) as reported by Li *et al.*, (2015) and Proso millet (138.43%) as reported by Singh and Adedeji (2017) resulting in better hydration rates within starch molecules and good quality gel formation (Kaur *et al.*, 2007). The peak temperature (TP) of AHD amaranth starch is 72.41°C, which is desirable for processing and cooking various food products. AHD amaranth starch has a peak viscosity (PV) of 1796 cp, which is of intermediate viscosity due to its high amylopectin content. The physico-chemical properties of AHD amaranth starch, such as high clarity and pasting characteristics, make it particularly suited for producing transparent edible films. These properties are advantageous for food packaging and preservation

Table 1: Effect of different drying temperatures on functional properties of freshly prepared AHD based edible film.

Temperatures (°C)	Thickness (mm)	Tensile strength (MPa)	Solubility (%)	Transparency (%)	WVP (g/ms Pa) $\times 10^{-1}$
45	0.27 $\pm$ 0.04 ^c	2.70 $\pm$ 0.12 ^d	38.23 $\pm$ 0.20 ^a	98.68 $\pm$ 0.01 ^a	0.60 $\pm$ 0.10 ^a
55	0.26 $\pm$ 0.02 ^b	2.65 $\pm$ 0.03 ^a	37.50 $\pm$ 0.08 ^b	98.25 $\pm$ 0.40 ^a	0.55 $\pm$ 0.03 ^b
65	0.24 $\pm$ 0.03 ^a	2.56 $\pm$ 0.08 ^b	33.96 $\pm$ 0.03 ^c	98.12 $\pm$ 0.05 ^{ba}	0.54 $\pm$ 0.05 ^c

n = 3, Mean $\pm$ standard deviation values in the same column differ significantly.

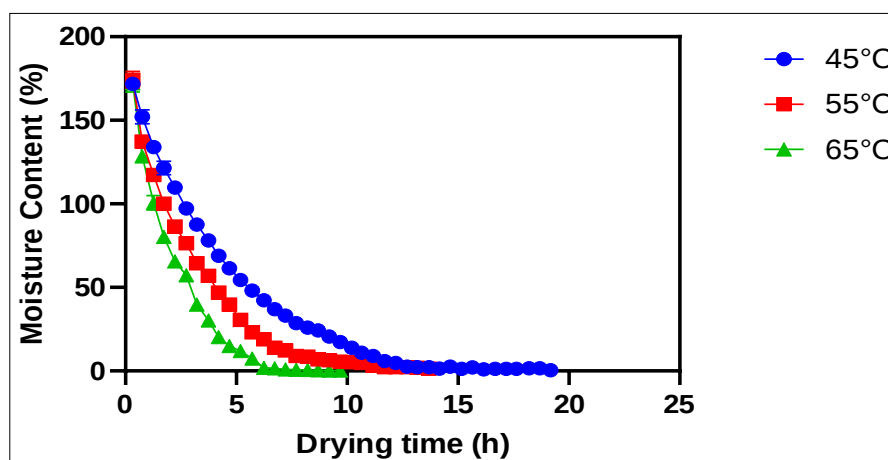


Fig 2: Drying curves of amaranth starch (AHD) based edible films at different temperatures.

applications. Notably, AHD amaranth starch requires a lower temperature for gel formation compared to conventional starches, enhancing its appeal as a suitable base ingredient for edible films formation (Singh *et al.*, 2014). In developing these films, glycerol was employed as a plasticizer to improve flexibility and carboxymethyl cellulose (CMC) was used as a cellulosic gum to enhance structural integrity. This combination resulted in edible films with superior quality, meeting the requirements for practical use in the food industry.

Drying kinetics

The effect of temperature variables on the drying characteristics of edible films were studied and different properties were evaluated at different drying conditions (Table 1). It was found that the total drying time decreased significantly with an increase in the temperature of hot air. As the temperature increased from 45 to 65°C, the drying rate also increased, as shown in Fig 2 and 3. Drying occurred in the falling rate period and faster drying was achieved at higher temperatures up to 65°C, with reduced time. The Arrhenius equation (eqn. 1) was further applied to analyze the dependence of drying rate on temperature.

$$\ln k = \ln A - \frac{E_a}{RT} \quad (1)$$

Where,

K = Rate constant.

A = Constant.

E_a = Activation energy.

R = Gas constant.

T = Temperature in Kelvin.

Higher drying temperatures increase the collision rate and kinetic energy, reducing activation energy and validating the Arrhenius equation's applicability to correlate the rate constant (K) with temperature. For amaranth starch-based films, faster drying improves production efficiency, but non-

uniform thickness and surface heterogeneity cause evaporation rate variations, affecting film properties like strength, transparency and barriers. Optimizing drying conditions and addressing these inconsistencies can enhance film quality.

Functional properties of dried and stored films

Thickness

The thickness of edible films plays a crucial role in determining their mechanical properties and water vapor permeability. An increase in film formation temperature from 45°C to 65°C led to a reduction in thickness, attributed to the removal of free water (Table 1). Elevated temperatures enhance molecular collision rates, leading to non-uniform drying, which can result in variations in the functional properties of the films, although the final thickness remained uniform. These findings align with the results reported by Sharma *et al.* (2018) and Nascimento *et al.* (2012). Additionally, the thickness of edible films is influenced by temperature and relative humidity during storage. Films stored at elevated temperature and humidity conditions (45°C and 75% RH) exhibited increased thickness, whereas films stored at lower conditions (30°C and 50% RH) showed minimal changes. High humidity facilitates greater moisture uptake due to increased moisture availability and elevated temperatures accelerate this process.

Tensile strength (TS)

Tensile strength values of amaranth starch films ranged from 2.56 to 2.70 MPa, indicating good mechanical properties. This strength is a result of positive interactions between molecules during processing, such as those between amaranth starch, glycerol and cellulosic gum molecules, as well as the specific temperature and humidity conditions.

This study found that the characteristics of dried films are influenced by the rate of change in temperature,

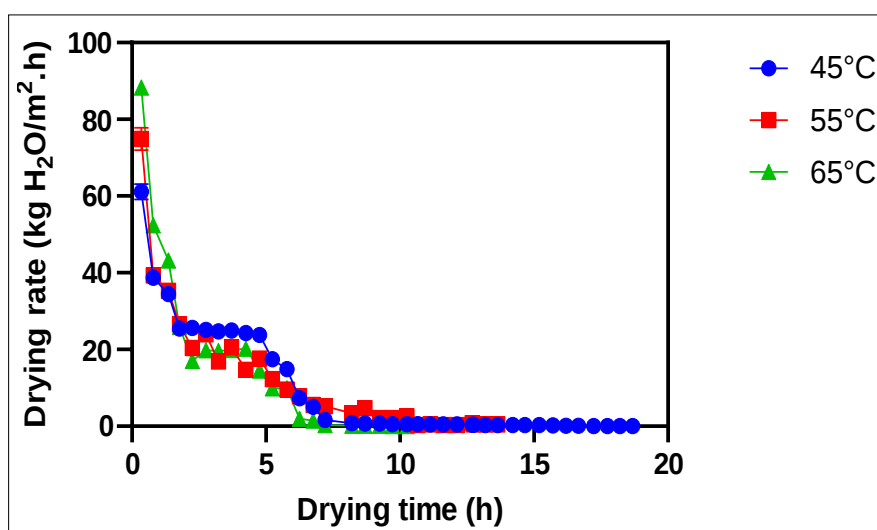


Fig 3: Drying rate versus drying time curves of amaranth starch (AHD) based edible films.

thickness and the molecules to molecule interactions during processing. Significant differences in tensile strength were observed and it was found that different combinations of temperature and relative humidity (RH) affected the strength of the films during storage, as presented in Table 2. The tensile strength of the edible films was observed to decrease over time. The results showed that storing the films under elevated temperature and humidity conditions resulted in better strength. Similar findings have been reported by Sukhija *et al.* (2016).

Solubility

The study found that the solubility of the edible films at different drying temperatures ranged from 33.23 to 37.50%, as shown in Table 1. Increase in drying temperature led to decline in solubility. It may be attributed to decrease in interaction between the hydroxyl groups of AHD chains and other components, leading to a decrease in the availability of hydroxyl groups and thus reducing polysaccharide-water interactions. This in turn would result in a decrease in the films' solubility (Salarbashi *et al.*, 2013). The solubility values ranging from 33% to 38% are considered optimal for edible films prepared from starch and its derivatives. However, low solubility can result in a slower rate of degradation, while high solubility values can lead to the disposal of the material/packaging in a short time (Onyeaka *et al.*, 2022; Balaji *et al.*, 2022).

During the study, it was observed that as the thickness of the edible films reduced, the solubility of the films increased while water permeation also increased. Significant differences in solubility values were found when the films were exposed to different temperature and relative humidity (RH) conditions, as shown in Table 2. Edible films stored at high temperature (45°C) and high humidity (75%) exhibited a higher solubility compared to the films stored at lower temperature (30°C) and humidity (50%). This may be due to the availability of more moisture, more absorption in the edible films under high humidity and accelerated temperature conditions. As a result, the inter-molecular binding of the matrix developed from starch, plasticizer and CMC together becomes loosened.

Water vapor permeability

The water vapor permeability (WVP) values of the prepared amaranth starch-based edible films ranged from 2.03 to 2.94×10^{-1} g/ms Pa (Table 1). Moisture transfer significantly affects many functions of edible films and this attribute is controlled by the vapor permeation rate. A lower water vapor permeation property of a packaging material is of great importance as it offers numerous advantages to the packed food and also results in increased shelf life of the packaging material. The major application of the film is to act as a barrier against moisture, prevent deteriorative reactions and avoid shrinkage of unpackaged foods due to water loss (Sukhija *et al.*, 2016).

Edible films dried at 65°C exhibited minimum water vapor permeation and there were significant differences observed in water vapor permeation for different combinations of temperature and relative humidity. Films stored at high temperature and relative humidity (45°C and 75% respectively) showed higher water vapor permeation rates, which may be due to the plasticizing effect of moisture and migration through the capillary macro-porous structure of the films (Long *et al.*, 2023). WVP is influenced by both the relative humidity gradient on one side of the film and it increases with temperature as demonstrated by its dependence on temperature by Arrhenius equation at moderate temperatures (Chinma *et al.*, 2015). Moreover, WVP values linearly increased with an increase in the solubility of the films and water diffusion rate during storage, as shown in Table 2.

Light transmittance

The transparency of amaranth starch films is an important characteristic for customer appeal, especially in the packaging of fruits/vegetables. After drying, the films exhibited excellent transparency ranging from 98.12-98.68% (Table 1). During storage at accelerated conditions, transparency was not significantly affected by lower temperature and humidity conditions. However, high temperature and humidity conditions resulted in decreased transmittance due to greater water plasticization of the matrices that enhanced molecular mobility moisture migration and subsequent

Table 2: Impact of accelerated storage conditions on functional properties of dried AHD based optimized edible films.

Days	Storage condition (Temperature and RH)	Thickness (mm)	Tensile strength (MPa)	WVP(g/ms Pa) $\times 10^{-1}$	Solubility (%)	Transparency (%)
0	30°C and 50%	0.24±0.04 ^{cp}	2.61±0.12 ^{ap}	0.54±0.10 ^{dq}	36.23±0.20 ^d	98.68±0.01 ^a
	45°C and 75%	0.24±0.04 ^{cp}	2.52±0.10 ^{aq}	0.55±0.03 ^{dp}	35.23±0.20 ^d	98.68±0.01 ^a
30	30°C and 50%	0.25±0.02 ^{bq}	2.25±0.03 ^{bp}	0.56±0.05 ^{cq}	36.50±0.08 ^c	98.25±0.40 ^a
	45°C and 75%	0.28±0.02 ^{bp}	1.25±0.03 ^{bq}	0.60±0.01 ^{cpq}	41.50±0.08 ^c	97.25±0.40 ^b
60	30°C and 50%	0.26±0.03 ^{aq}	2.16±0.08 ^{cp}	0.62±0.02 ^{bp}	38.96±0.03 ^b	96.12±0.05 ^b
	45°C and 75%	0.29±0.03 ^{ap}	1.16±0.08 ^{cq}	0.63±0.03 ^{bq}	52.96±0.03 ^b	90.12±0.05 ^c
90	30°C and 50%	0.26±0.02 ^{abq}	2.04±0.05 ^{dp}	0.72±0.01 ^{ap}	39.48±0.03 ^a	95.24±0.01 ^c
	45°C and 75%	0.29±0.02 ^{abp}	0.54±0.05 ^{dq}	0.89±0.03 ^{aq}	61.48±0.03 ^a	85.24±0.01 ^d

n = 3, Mean±standard deviation values in the same column as well as row (pq) (for individual properties), followed by different superscript, differ significantly (p<0.05) and RH; relative humidity.

swelling of the films during storage, as shown in Table 2. Similar results have been reported by Huntrakul *et al.* (2020) during storage of cassava starch based edible films.

Surface morphology (SEM)

Scanning Electron Microscopy (SEM) analysis of the optimized amaranth starch (AHD) edible films revealed a homogeneous and crack-free surface. The films exhibited a smooth and uniform starch matrix, with partially

gelatinized starch granules visible under higher magnifications (5000× and 7500 ×). These observations suggest strong interactions among the film components, including amaranth starch, the plasticizer and CMC, contributing to the film's integrity and structural properties. Intrinsic porosity in the film structure due to the formulation or processing conditions might be revealed at higher magnifications. The detailed microstructural characteristics are presented in Fig 4a.

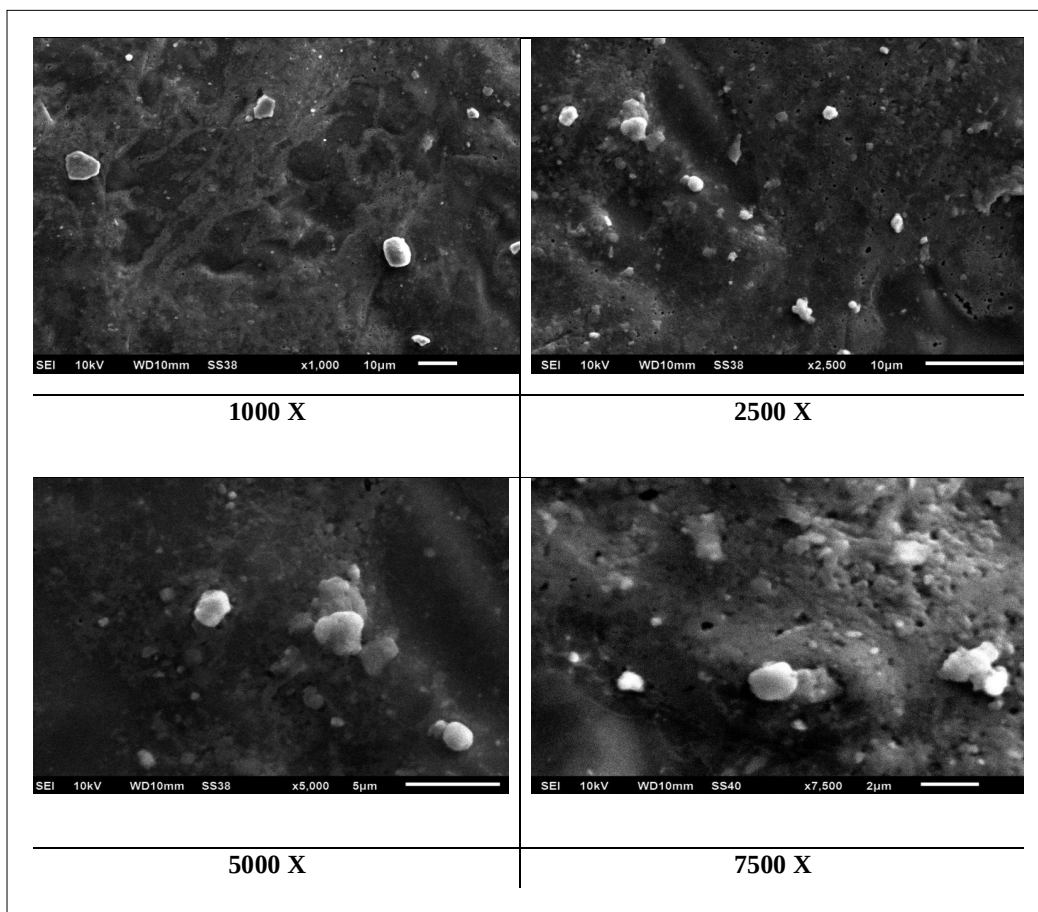


Fig 4a: Microstructures of dried edible film at different magnification.

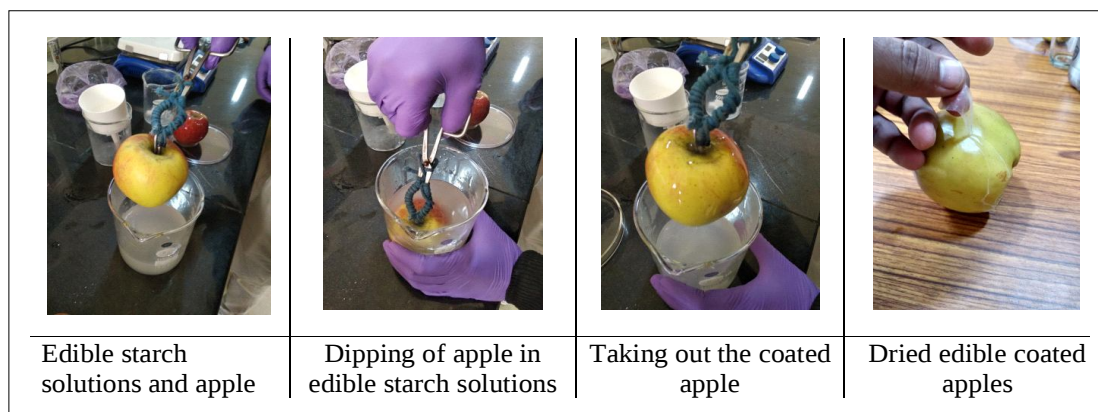


Fig 4b: Applications of edible filmogenic solutions to apple coating.

Applications

The developed edible films were evaluated for their effectiveness as a coating for apples. Edible coating solutions were prepared and applied by immersing apples in the optimized formulation (Fig 4b). After coating, the apples were dried at room temperature and the performance of the filmogenic solutions to form edible coating was assessed. The apples were successfully edible coated, demonstrating the feasibility of the edible solution in coating the apple by immersion method. Future research will focus on assessing the impact of the coating on apple quality during storage.

CONCLUSION

During the drying of amaranth starch based edible films, reduction in drying time as of increase in drying temperature resulted in improvement in the production process of films. Drying time and temperature to develop amaranth starch based edible films was standardized at 55°C and 13.5 h, respectively. At these conditions, the films exhibited fair and desirable properties, including adequate mechanical strength, controlled solubility, low water vapor permeability and favorable transparency. These attributes make them suitable as a biodegradable and sustainable alternative for primary packaging. Specifically, the films can be effectively used for premium fruits, such as apples, to maintain their quality throughout the supply chain. Further investigations into drying and storage conditions can enhance the films' performance and broaden their application in environmentally friendly packaging solutions, supporting the transition towards sustainable food packaging systems.

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Disclaimers

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information provided, but do not accept any liability for any direct or indirect losses resulting from the use of this content.

Informed consent

This study does not involve any animal procedures for experiments.

Conflict of interest

The authors declare that there are no conflicts of interest regarding the publication of this article. No funding or sponsorship influenced the design of the study, data collection, analysis, decision to publish, or preparation of the manuscript.

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