Development of Teff-Starch based Edible Film: Mechanical and Optical Properties

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Abstract. Teff is a unique staple food in Ethiopia. Teff has not been investigated in detail with respect to biofilm base material. In this study, a biofilm was prepared using teff-starch. Further, water barrier, mechanical, and optical properties of the teff-starch based films were investigated. These properties were compared with the cassava-starch based biofilm that was developed with same combination of plasticizer, surfactant and agar. The result showed that teff-starch based edible film is good compactable for food packaging material. Also, the mechanical and optical properties compared with standard cassava-starch based biofilm showed teff-starch films are better appropriate as for a packaging material.

1. Introduction
Synthetic plastics are known to be good for different desirable properties such as mechanical and barrier properties. They can be produced in mass production at comparatively low cost [1]. Though having these advantages, most of the synthetic plastics are handled by incineration or landfill. Particularly, the synthetic plastic materials after use as food packaging may cause environmental pollution. The food packaging is mainly focusing to reduce food quality deterioration that occurs by biological and chemical parameters [2]. Oxidation of lipids from the food is one of the principal reasons deterioration of the foods. So, food industries use synthetic antioxidants for reducing lipids oxidation [3].

Due to consumer concerns with respect to safeness of synthetic antioxidants, food industries are focusing towards biofilms for food packaging materials since they are biodegradable and environmentally friendly. Biopolymers such as gum, wax, protein, and starch that are derived from raw food resources can be used as biofilm base materials [4]. Such environmentally-friendly materials are rapidly biodegraded when they are landfilling and harmless. Recently, starch-based biofilms are gaining huge interest among the researchers for developing bio-based food packaging materials due to its easy processing, and colorless and odorless nature [5]. Even though the biofilms obtained from starch have been widely investigated recently by several researchers, their mechanical and physical properties still need to be improved.

Teff is known to be a unique grain in Ethiopia. It is identified as a gluten-free grain. Even though several studies using teff starch have been carried out for substituting wheat in baking [6], starch from teff has not been subjected to investigate in detail as a biofilm base material. In specific, the blends concerning of teff-starch with major ingredients such as sorbitol as plasticizer, agar and
tween-80 as surfactant have not been studied yet. Teff has been characterized as 73% starch content that contains 18%–27% amylose. The starch content makes teff is suitable for preparation of biofilms [7]. In this study, biofilm was prepared using the starch that isolated from the teff with predetermined amounts of sorbitol as plasticizer, agar and tween-80 as surfactant. Further, the physical and barrier properties of the developed film were examined. The developed film was further compared with biofilm prepared with standard cassava starch that contain 82% by weight of amylopectin 18% by weight of amylose.

2. Materials and Methods

2.1. Materials
Teff powder was purchased from city market of Addis Ababa, Ethiopia. Food grade-Sorbitol, food grade-tween-80, agar, glass-ware and other required chemicals were procured from reputed chemicals at Addis Ababa.

2.2. Isolation of Teff-Starch
Starch from the teff was extracted using the procedure given by Bultosa et al. (2002) with some modifications. After proper purification, teff grains were grounded by grinder. Further, the powder was sieved with 600-mesh sieve. 100g of teff powder was taken 1000mL beaker and mixed with 500mL distilled deionized water. The blend was well-stirred at 30 °C for 1 hr. The resultant slurry was grounded well using wet milling. Further, it was filtered twice with a 300-mesh sieve. Using test-tube centrifuge, the filtrate was subjected to centrifuge at 900 × g, 30 °C, for 6 min. The gray layer was separated using a spatula after the supernatant was rejected. The resulted sludge was further well-dispersed using distilled water. These steps were repeated until the gray layer not to be visible. The resultant product was spread using deionized water and dried at 55 °C for 30 hr. The isolated starch was grounded by a mortar than it was filtered by passing through sieve (300-mesh). Then, it was stored at 4 °C and used for the preparation of biofilm.

2.3. Determination of chemical composition of isolated teff-starch
Chemical composition of isolated teff-starch such as moisture content (%), ash content (%), starch content (%), amylose (%), and amylopectin (%) were determined using AOAC methods (1995).

2.4. Standard cassava starch
Cassava starch that contains amylopectin-82% (by weight) and amylose-18% was obtained from Sigma-Aldrich as analytical grade.

2.5. Preparation of Biofilms
Solutions with starches (3g), plasticizer (0.8 ml), agar (0.8g) and surfactant (0.8 ml) were considered to prepare films by casting method. The clear-homogenous solution, used for film forming purpose, was first arranged by dissolving starch and agar in deionized water without plasticizer. Further, the surfactant and plasticizer were added to the solution. This solution was exposed to heat slowly up to 75 ± 5 °C with mild stirring (150 rpm). Further, the mixture was saved back for 35 min to develop a clear-homogenous film forming solution. This solution was subjected to degas using vacuum environment. The solution was carefully transferred into the Petri dishes placed with well-leveled surface and allowed to dry at 30 °C in a clean chamber with controlled temperature for 50 h. After the determined time, films were cautiously detached from the Petri dishes. The detached films were equilibrated at 30 °C for 80 h before they subject to analysis.
2.6. Characterization of the developed films

2.6.1. Mechanical Properties. The tensile properties such as, elongation (%), tensile strength (MPa), and Young’s modulus (MPa) were determined by a proper material testing equipment with appropriate load cell based on standard technique of ASTM, D 882-91(1996). Sample of film strips of appropriate size were cut before analysis. Film thickness of the prepared samples were determined by a digital micrometre.

2.6.2. Barrier Properties. The film thickness was determined by an appropriate digital micrometer. 30 trails for measurements of thickness were done at different locations randomly. The mean values were calculated and recorded. The examination for moisture content present in the film was carried out by gravimetric method. The important property, water vapor permeability for the prepared films was assessed based on the procedure given by Mali et al (2004) [8]. Thickness of the film was determined after tests, and Water vapor permeability was determined by the Eq. 1.

\[
\text{Water vapor permeability} = \frac{\text{Transmission rate of the water vapor}}{S(R_1 - R_2)D} (\text{g m}^{-1}\text{s}^{-1}\text{Pa}^{-1}) \quad \ldots \ldots (1)
\]

\(R_1\) refers to relative humidity of the desiccators, \(R_2\) refers saturation vapor pressure of water (Pa) at the room temperature, \(R_2\) refers the relative humidity of the permeation cell. \(D\) refers to the thickness of the film (m). Film solubility (SOL) was examined using the method marked by Romero-Bastida et al (2005). Using Eq. 2, the total soluble matter of the film sample was determined.

\[
\text{SOL} (%) = \left(\frac{M_0 - M_1}{M_1}\right) \times 100 \quad \ldots \ldots (2)
\]

Where, \(M_1\) and \(M_0\) refer to weight of the dry sample after and before the test, respectively. Another important property, swelling capacity of acquired films in water was determined based on the procedure explained by Hu et al (2009). The films with predetermined size were taken in the containers having 100mL (0.1 mol l\(^{-1}\)) NaOH and 100mL (0.1 mol l\(^{-1}\)) HCl solution, respectively. Then, the vessels were perfectly sealed and kept at 30 °C.

They were slightly shaken at regular time interval. The changes of sample appearance were noted to examine the stability of films at acid and alkali medium. Transparency of the film (TR) was examined using the procedure explained by Ozdemir & Floros (2008) [9, 10]. The rectangular predetermined-shapes of the films were positioned inside a spectrophotometer cell. The transparency of each film was determined at 560nm using Eq. 3.

\[
\text{TR} (%) = \left(\frac{I_1}{I_0}\right) \times 100 \quad \ldots \ldots (3)
\]

\(I_1\) refers to light intensity with the specimen in the beam and \(I_0\) refers to light intensity with no specimen in the beam. Sample of film strips of appropriate size were cut before testing and the thickness of the samples were determined by a digital micrometre [11].

3. Results and Discussions

3.1. Mechanical properties of developed films

Mechanical strength is needed to keep the structural integrity as well as barrier properties. The investigation on the mechanical properties having much importance because of their influence on performance and also the acceptance of the consumers. Edible films with acceptable mechanical
properties are great potential and ecological substitute for replacing synthetic packaging in various applications. This work focused starch-based biodegradable biofilms were developed and the mechanical properties, Young’s modulus, percentage elongation, tensile strength of the starch-based biofilms were studied.

Tensile strength is defined as the ability of any material that withstands against tensile (pulling) force. Tensile strength can be determined in units of force per cross-sectional area. It was observed that the teff starch joined with agar molecule which induced the formation of biofilm with elevated tensile strength (4.8 MPa) [12]. The observed value is good for the development of dense structure due to establishment of inter-molecular H bonds between starch and agar. This molecular linkage directly improved the tensile strength of developed films. However, the film developed from starch derived from cassava-starch exhibited higher tensile strength (5.46 MPa) than the teff starch films. Elongation break point is widely used to explain the structural character of materials. The developed films were plasticized with sorbitol that exhibited lower value of elongation (33.12%). This value little high compared with cassava-starch biofilm (31.3 %) because of its interference with amyllose packing in starch chains and its further effect on chain attraction. Young's modulus explains material’s stiffness property, specifically for elastic materials. It can be defined as the ratio of the stress to strain on the linear part of the stress–strain curve.

The results from teff-starch biofilm characterization towards determination of Young's modulus showed that the teff-starch biofilm has reduced the Young's modulus (146 MPa). It showed the hydrophilic nature of the teff-starch. However, the cassava- starch biofilm had higher modulus value (158 MPa). Barrier and optical properties are important for the materials when they focused for permeable object transmits from one side to the other [13].

3.2. Optical and barrier properties of developed films

The knowledge on optical and barrier properties will be much helpful for packing as well as designing the structure of packing materials quite reasonably and efficiently. So, the optical and barrier properties such as thickness, water vapor permeability, moisture content, swelling capacity, anti-permeability against oil, solubility, stability in acid/alkali solution, and transparency were examined. The developed films were good transparent with no apparent bubbles, also they were easily separated from the Petri plates without apparent exudation of the surfactant on the film surface. They were easy to handle for both starch, surfactant, plasticizer, and agar utilized in this work.

Determination of moisture content provides the evidence with regards to the amount of water present in the films. The observed results provided that moisture content of developed films differed pointedly. Teff-starch based biofilm showed that the moisture content 18.24 %, but for cassava-starch films exhibited 14.35 %. Irrespective of the used plasticizer, the moisture content of the teff-starch based film was showed higher value than cassava-starch based film. Since the starch was more readily disintegrated by absorbing water and having poor barrier properties due to its low hygroscopic property. Water vapor permeability of the developed films is an important property. This property has great impact on utility of film in different applications. It was observed that the teff-starch in the film formulation showed $0.67 \times 10^{-10}$ g/msPa and cassava-starch showed $0.68 \times 10^{-10}$ g/msPa. These values were nearly same that could be the fact that, the hydrophilic nature of the starches favouring the adsorption of the water.

Water solubility and swelling capacity are important factors while choosing the films for specific uses. The properties of solubility and swelling capacity are much anticipated properties in various cases, in specific, the encapsulation. Solubility and swelling capacity of biofilms explain the pertinence of film to pack water-rich foods like unwrapped fruits. They are also much related to the biodegradability. So, the solubility and swelling capacity of films in water was studied [14]. It was observed that the predetermined film-formulation showed 31%-solubility and swelling capacity-82%. It was accredited to the occurrence of higher hydrophilic groups in the films that can attracted more water. Cassava-starch based films showed little lower value of solubility (29%) and swelling capacity (81%) than teff-starch films.
The edible starch-films prepared in this study for packing material to pack slight acid or alkali materials. Both materials could influence the stability of starch-based films. The determination of prepared starch-films in acid/alkali solution was examined. Starch-films were cut into 4 × 4 cm and submerged in 0.1 mol/L hydrochloric acid solution. The size of the films was increased little bit but such tendency did not go more beyond 3 days. After 35 days, the films still hold reliability. This kind of property showed that the starch films developed on both teff and cassava could withstand acidic condition. But, the teff-starch film exhibited a noticeable swelling at 1st day itself in 0.1 mol/L NaOH. The films start to break at end of 2nd day. It was fully collapsed in 4 days. The same kind of results were obtained for the cassava-starch biofilm. This was because of the OH− groups of starch molecules joined with NaOH, partially demolish the H-bonds and reduce the contact among the starch macromolecules that make the observed swelling and gelatinization property of starch. Transparency of films is much important while the biofilms are used for food packaging. The prepared films were observed to be transparent and homogenous. In addition, it was observed that, teff-starch based films showed higher transparency (up to 85%) than cassava-starch films (up to 81%). The higher transparency could be possibly related to its relatively higher amylopectin and lower amylose content.

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