Research Article

Study on Physical Properties of Potato Starch-based Edible Film Incorporated with Orange Essential Oil

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Abstract

The orange essential oil contains wealthy antioxidant properties which are utilized in expanding the edible packaging material's usefulness. The primary objective of this study is to develop and characterize a novel biofilm using potato starch for food packaging incorporated with orange essential oil (0.4-1.2% v/v) that was added to potato starch (8-10% w/v) at different concentrations to prepare the film. The films were examined for physical and chemical properties in order to optimise the film concentration. The studies show that the thickness of the EF (Edible film) is between 200-270μm and the Water dissolvability of the films differed from 32.14 to 47.50% and it was essentially impacted by the convergence of glycerol. An increase in the concentration of starch and glycerol had decreased the tensile strength (8.8 – 2.4Mpa) and increased the water vapor permeability.

Keywords: Edible film, Potato starch, Response surface methodology, Antioxidant

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Introduction

There has been budding interest in developing biopolymeric packaging comprising of biodegradable and renewable material [1]. Edible films and coatings are prepared using different edible and food-grade additives. These edible film biopolymers can be proteins, polysaccharides (starches and gums), lipids, or a combination of these [2]. Edible films and coatings upgrade the nature of food items, ensuring them from the physical and organic disintegration [3]. The use of edible films and coatings can promptly improve the actual strength of food items, lessen particle clusters, and improve visual and material highlights on item surfaces [4]. It can likewise shield food items from dampness relocation, microbial development on a superficial level, light-prompted synthetic changes, and nutrient oxidation [5]. There is a developing need to comprehend the interactions between the film structure, water atoms, and different constituents utilized for edible film making. Starch, as perhaps the most significant and abundant polysaccharides in nature, has been the subject of various researches. It is generally utilized in industry to give functional properties, for example, gelling, thickening, bonding, and adhesiveness [6]. Starch is a high molecular weight (Mw) polymer of a hydro-glucose unit connected by a-glycosidic bonds. Starch atoms contain linear amylose and branched amylopectin. Amylose has a lower Mw, amylopectin has a higher Mw of 50–500 million [7]. However, the semi crystalline (20% to 45%) nature of native starch results in some undesirable drawbacks, such as its hydrophilic character, poor solubility, poor mechanical properties, uncontrollable paste consistency, and low freeze-thaw stability during film formation [8]. To survive these defects, and to change the starch film attributes, different change procedures can be utilized: physical, chemical, enzymatic, hereditary, and expansion of added substances or a blend of treatments. These would improve starch properties by modifying starch molecular structure. The addition of plasticizers is important to improve film adaptability. Glycerol is quite possibly the most mainstream plasticizer utilized in film-production procedures, due to its solidness and similarity with hydrophilic bio-polymeric packaging chains [9]. Although numerous essential oils are classified Generally Recognized as Safe (GRAS), high convergences of essential oils are for the most part expected to accomplish the viable antimicrobial activity indirect food applications, and accordingly, these focuses may surpass organoleptically satisfactory levels [10]. Specifically, the addition of these oils permits enhancements in food safety and shelf life by decreasing the development of pathogenic microorganisms. Citrus essential oil is currently the most common value-added product extracted from the citrus juice processing waste. This essential oil is mostly found in the citrus peel and flavedo and can be obtained using different extraction techniques. The essential oil of orange peel has antimicrobial properties against bacteria and fungi in both vapor and liquid states. Antimicrobial films and coatings for food packaging applications have been created by incorporating this oil into different polymer matrices [11].

Materials and Methods
Materials

Potatoes (*Solanum tuberosum L*) were purchased from Kwality big bazaar (Bengaluru, India). Glycerol (purity 99.97%, thickness 1.26 g/cm^3) was procured from Merck life science private limited (Mumbai, Maharashtra), the orange essential oil was purchased from Natural aroma world (Bengaluru, India) and glycolic acid of analytical grade was procured from NICE chemicals private limited (Kochi, Kerala).

Isolation of Starch

Potatoes were purchased from Kwality Big Bazaar (Bengaluru, India). In that one kg of potatoes were washed completely stripped and ground for the extraction of starch. Then distilled water was added to the ground potato and the extraction process was carried out using a centrifuge at a speed of 5000 rpm for 5 min. The centrifuged samples were separated utilizing Whatman no. 1 filter paper and the supernatant neglected to get wet starch. The wet starch was dried at room temperature for 5 h, the solid starch was squashed into a fine powder utilizing pestle and mortar and stored in a glass container for additional utilization.

Preparation of edible film containing orange essential oil

Potato starch (8% w/v) was suspended in 100 ml of distilled water and mixed for 5 minutes at room temperature (25-28^{0}C). The dispersion was warmed at 85^{0}C in a water bath, for 30 minutes to prompt starch gelatinization and cooled down to room temperature [12, 13]. Glycerol (3% v/v) was added into the above dispersion as a plasticizer. The blend was moved to a water bath and heated at 90◦C for 15 minutes. This combination was cooled down to room temperature (25-28^{0}C). Orange essential oil (0.4% v/v) was added at the required extent and mixed for 2 to 3 mins to get a uniform dispersion [14]. These film-forming solution (20±1.5 ml) was poured into the borosil petri plates (7 cm dia) and put in a hot air oven at 70^{0}C for 3-4.5 hours. After complete drying of these films, they were removed from the Petri plates utilizing scalpel and forceps. Prepared film samples were packed in HDPE polythene sheets and put away in a fridge at 4^{0}C utilizing a hermetically sealed container to forestall dampness ingestion. A Box-Behnken statistical tool (rotatable or almost rotatable three factorial plan) under response surface methodology was used to decide the impact of three independent factors, at three levels each, in film properties. The total design comprising of 13 formulation combination and is presented in the **Table 1**. Prepared edible film samples are presented in the **Figure 1**.

Table 1 Total design comprising 13 analyses

Sl. No.	Starch (W/V)	Glycerol (V/V)	Orange essential oil (V/V)	Coded value
1	8%	3%	0.4%	08X3Y0.4Z
2	8%	2%	0.8%	08X2Y0.8Z
3	8%	4%	0.8%	08X4Y0.8Z
4	8%	3%	1.2%	08X3Y1.2Z
5	9%	2%	0.4%	09X2Y0.4Z
6	9%	2%	1.2%	09X2Y1.2Z
7	*9%*	*3%*	0.8%	09X3Y0.8Z
8	9%	4%	0.4%	09X4Y0.4Z
9	9%	4%	1.2%	09X4Y1.2Z
10	10%	3%	0.4%	10X3Y0.4Z
11	10%	2%	0.8%	10X2Y0.8Z
12	10%	4%	0.8%	10X4Y0.8Z
13	10%	3%	1.2%	10X3Y1.2Z

Film Thickness

The thickness of each composite film was measured by a thickness gauge, and 10 points were uniformly taken from the centre of the film and the average value was taken as the thickness value t of the composite film.

Figure 1 Prepared edible films

Water vapor permeability (WVP)

Permeation cells which are loaded with a lot of anhydrous silica gel (RH = 10%) provided with an air gap of under 6 mm, were fixed by the readied film utilizing parafilm. At that point, the penetration cells were set into desiccators with a consistent RH (52%) and temperature (30°C). Starting weight and weight of the film for each 2 h for 24 h were estimated. The changes in their weight were recorded and plotted against time [15]. Water vapor permeability was determined by the equation.

$$WVT=\frac{G}{tA}=\frac{G}{t}/A \quad (1)$$

Where WVT is the water vapor transmission ($g/h{\cdot}m^2$), G is the weight acquire (g), t is the time (h) and A is the test territory (penetration cell mouth territory m^2)

Water and acid solubility

The solubility of edible film in water was determined by the modified technique proposed by Gontard [16]. A two-centimetre diameter sample was cut from the edible film and the initial weight of the sample was weighed after drying the sample for 4 hours in an air circulation oven at 105 ◦C. At that point, samples were soaked in 100 mL of distilled water mixed gently and kept undisturbed for 24 h. The swollen samples were then removed and dried at 105 ◦C for 4 hours and the final dry weight was noted down. The solubility of the films was determined by utilizing the equation.

$$\text{Solubility} = \frac{W1-W2)}{W1} \times 100$$

W1- Initial dried load of the film, W2- Final dried weight of the film.

Acid solubility of the sample was determined after the same procedure depicted above yet the film was submerged in a measuring glass containing hydrochloric acid (1 M) for 24 h, and then dried and weighed to determine the final dry weight [17].

FRAP (Ferric Reducing Antioxidant Power) Radical Scavenging Assay

A FRAP was carried out according to previous research with slight modification [18].

One gm of film sample was immersed in 20 mL methanol (80%) to get the extract. Consequently, 0.2 mL of film extract was added to the 1.8 ml FRAP reagent. It was then mixed thoroughly and was incubated in a dark room at normal room temperature for 30 min. The absorbance was measured at 593 nm and PC- Based Double Beam spectrophotometer 2201 was used for absorbance measurement is shown in the **Figure 2**. The antioxidant capacity (*mg AEAC/100g)* was measured using the following equation:

Antioxidant capacity=(b)lightness X Total volume X 100/Assay volume X Weight of sample(g) X 1000

Figure 2 PC- Based Double Beam Spectrophotometer- 2201

Tensile strength (TS)

The rigidity of the film was determined by a Universal testing machine (Stable Micro Systems-Texture analyzer instrument) and as per the procedure recommended [19]. The machine utilized for the estimation was kept up at a constant rate of transverse of a grip head and is shown in the **Figure 3**. Edible film samples were cut as strips of appropriate size (15 mm width x 50 mm length) and one end of the strip was firmly grasped in the upper clasp and the opposite end in the lower cinch after changing the arrangement as straight as possible in the machine. The machine was turned on at a pre-adjusting speed of 500 (mm/min). The load range utilized was such that the maximum load of the test pieces fell between 15 – 85 percent of the full-scale reading. The result of every individual strip was recorded and the tensile strength was calculated and expressed as MPa from the original area of the cross-section.

Figure 3 Stable Micro Systems-Texture analyser

Statistical Analysis

The box-Behnken technique under response surface methodology was utilized to determine the interaction of independent variables on dependent parameters. Design Expert Version 6.0.8.1 Programming was utilized for analysing the data related to edible film, for statistical significance. All preparations were carried out in 13 variations.

Results and Discussion

Thickness

The impact of the addition of glycerol, sucrose, and orange essential oil on the thickness of the edible film is presented in **Figure 4**. It was seen that film thickness increased significantly (P<0.05) with the addition of starch and glycerol concentration. This might be because of the increase in the overall mass of the material, which thus might have increased the thickness of the film. The incorporation of orange essential oil diminished the film thickness, yet the impact was most certainly not huge (P<0.05). The incorporation of OEO increased the thickness of the films and Luis also reported an increase in film thickness with the addition of Liquorice essential oil (*Glycyrrhiza glabra L.*) and ascribed this behaviour to the entrapment of essential oil micro droplets into the polymeric matrix, subsequently increasing the compactness of the starch matrix structure [20].

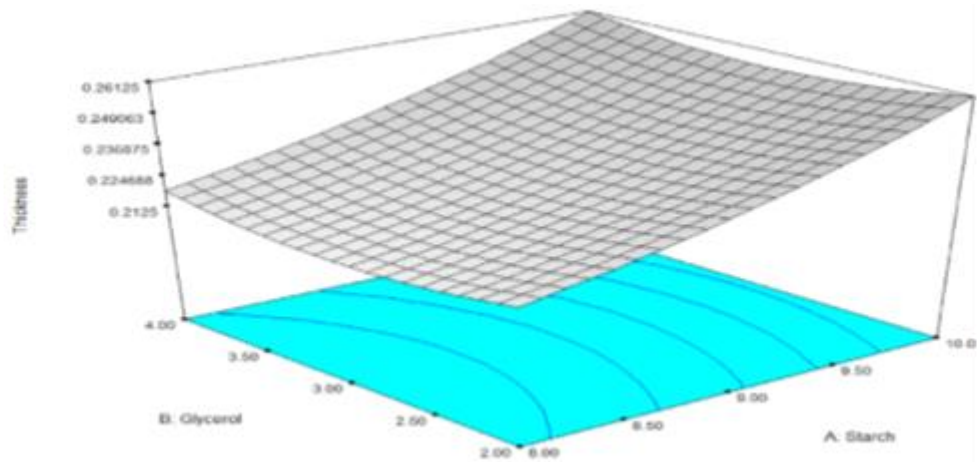

Figure 4 The thickness of the film at different concentrations of starch, glycerol, and orange essential oil.

Water vapor permeability

The impact of the addition of glycerol, sucrose, and orange essential oil on the water vapor permeability of the edible film is presented in **Figure 5**. The water vapor permeability (WVP) of films is a very important factor for estimating product shelf-life, as a result of water could transfer from the internal or external environment through the film, leading to the deterioration of product quality and shelf-life [21]. In this study, an increase in the concentration of starch and glycerol might have increased the affinity of the film towards the water, thereby permitting the moisture migration once the orange essential oil was incorporated into the film, it might have increased the heterogeneity of the film which in turn could have raised the WVP. The water permeability increased significantly with an increase in glycerol and starch concentration.

WVP is proportional to the concentration of plasticizer, this behaviour has already been reported in cassava starch film [22], cassia gum [23], and rice starch films [24], which might be because of increase in plasticizer concentration, that influence on three-dimensional molecular organization of the polymer. These organizations consequently change, the intermolecular attractive forces and increases the free volume of the system, consequently the network becomes less dense, allowing water permeation through the structure [25].

Water solubility (WS)

Water solubility is one of the important properties of biodegradable packing film using starch and glycerol. Packaging film requires water resistance for packaging material. The water solubility of the films at different concentrations of glycerol starch and OEO is presented in **Figure 6**. The WS values of potato starch films with different levels of glycerol ranged from 34.31 to 51.54%). The films with OEO, regardless of OEO concentration, showed higher solubility than the films without OEO. Overall, the solubility of films depends on the type and concentration of the compounds as well their hydrophilicity and hydrophobicity indices. The film solubility increased with an increase in starch concentration but the effect was not significant. It might be due to the lesser concentration of starch content. Glycerol had a significant (P<0.05) effect on increasing the film solubility [26]. Adding glycerol to starch films enhanced their water solubility in all concentrations, according to the findings by farhanky [27]. Generally, plasticizers increase the hydrophilic nature of the film which in turn might have increased the film solubility. An increase in glycerol concentration from 2-4% increases the water solubility.

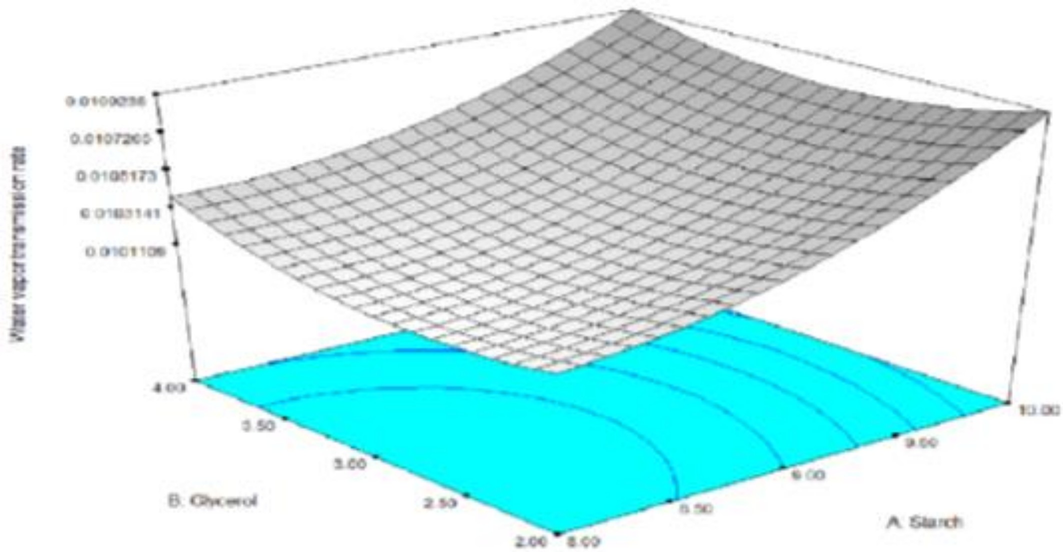

Figure 5 Water vapor transmission rate of the film at different concentrations of starch, glycerol, and orange essential oil

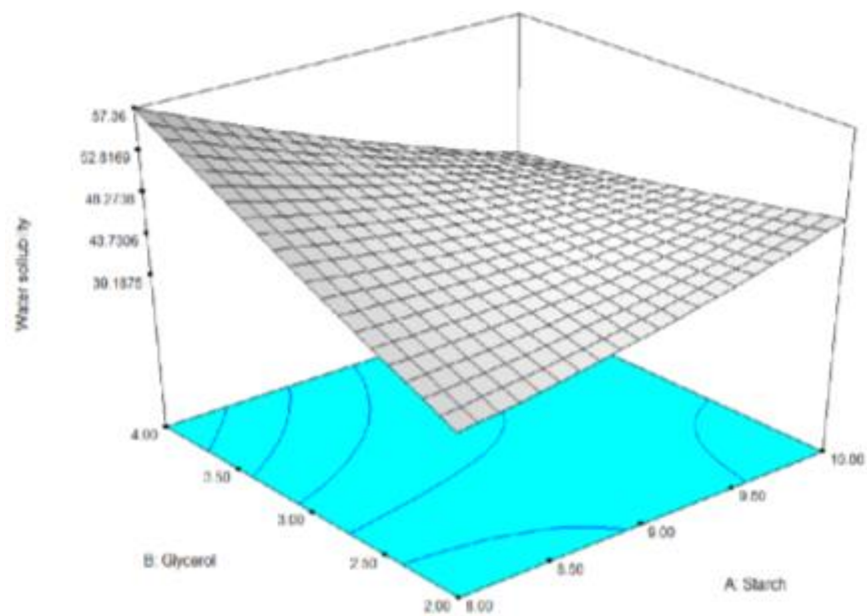

Figure 6 Water solubility of the films with different concentrations at glycerol, starch, and orange essential oil

Total Antioxidant

The impact of the addition of glycerol, sucrose, and orange essential oil on the total antioxidant of the edible film is presented in **Figure 7**. Antioxidant activity of packaging films turned into studies through FRAP (Ferric Reducing Antioxidant Power) radical scavenging activity into potato starch edible films: 80% methanol simulant. The film confirmed the highest % inhibition in the 80% methanol. This result may be defined by the lack of affinity and solubility of edible films in water. The film additionally shows the highest percentage of inhibition in the 80% methanol. Lopez-de-Dicastillo even analysing the active antioxidant packaging films, additionally observed that orange essential oil became notably soluble in methanol and had been released to an excellent quantity in 80% methanol [28]. However, with the increase in the concentration of antioxidants (orange essential oil), % inhibition will increase on these solvents [29]. Films having higher antioxidant activity when the orange essential oil is incorporated. This is probably viable due to the fact that the film incorporates greater phenolic contents which can be accountable for the better FRAP radical scavenging activity [30].

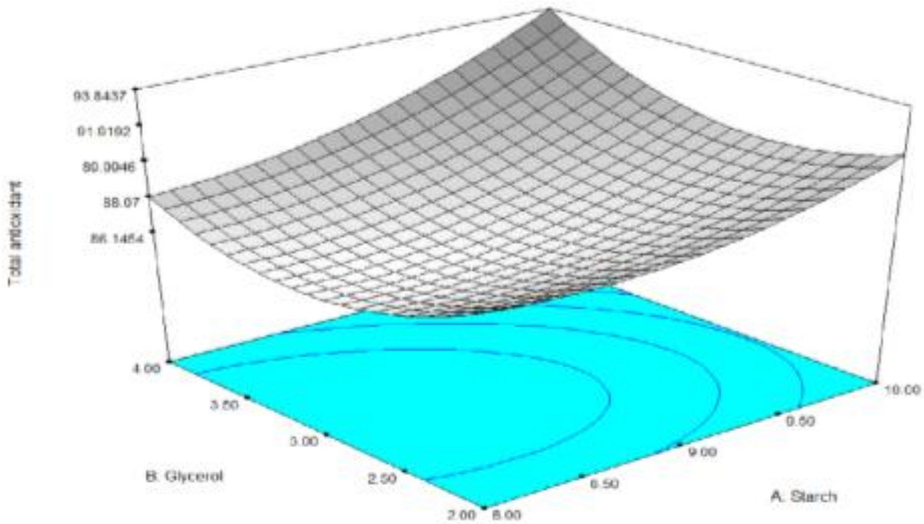

Figure 7 Total Antioxidant of the films with different concentrations at glycerol, starch, and orange essential oil

Tensile strength

The impact of the addition of glycerol, sucrose, and orange essential oil on the tensile strength of the edible film is presented in **Figure 8**. A texture profile analyzer was used to prepare the plasticized films and measure their tensile strength. Tensile Intensity can be calculated exactly with this analyzer in conjunction with ASTM D882 [31]. The effect of plasticizers weakens the intermolecular forces between adjacent macromolecule chains, raising the free volume and causing a reduction in mechanical resistance [32]. As a result of the decline in intermolecular interactions, raising the plasticizer concentration lowers the tensile strength TS [33]. Increased gelatin and starch concentrations resulted in increased elongation at break and tensile strength of the bioplastics. A study on biodegradable blends of poly(caprolactone) with native and modified corn starches found an increase in tensile strength with increasing starch concentration [34]. As a result, the films prepared with less glycerol had higher tensile strength than those prepared with more glycerol [35]. The highest TS of the film might be due to the hydrogen bond formation between glycerol and starch molecules which strengthened the film network.

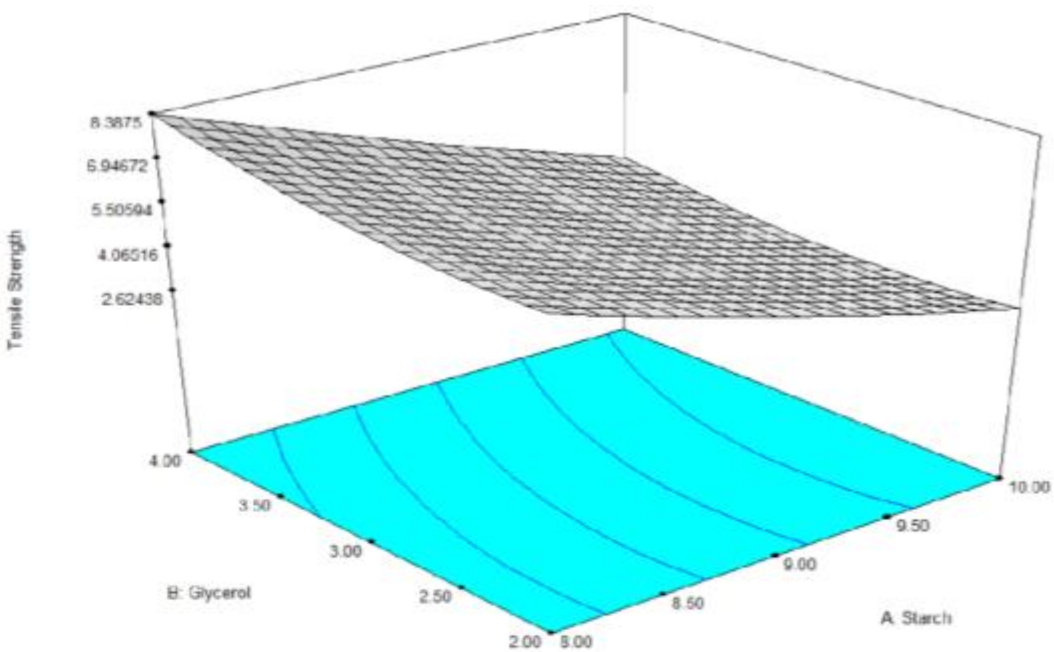

Figure 8 Tensile strength of the films with different concentrations at glycerol, starch, and orange essential oil

Optimization of edible coating solution formulation was conducted using the Box Behnken statistical tool. Optimum response in orange essential oil film formulation for each dependent did not fall in the same region. Hence the numerical optimization by the Box Behnken method was utilized to get the optimal solution. It gives the edible coating formulation with higher desirability consists of 9% starch, 3% glycerol, and 0.88% orange essential oil. The predicted dependent variables for optimized formulations consist of Thickness (millimeter) - 0.23mm, Tensile strength - 4.7 Mpa, Water solubility -45.03%, and lower permeability of 0.03 g/h·m^2

To verify the reliability of the response surface methodology experiment was conducted with optimized parameters such as 9% starch, 3% glycerol, and 0.88% orange essential oil. After three replications the mean value of tensile strength was found to be 4.56 Mpa. It confirmed the good correlation between the obtained tensile strength value and predicted tensile strength value.

Conclusion

Our results showed the potential of developing potato starch-based films reported with orange essential oil. Orange essential oil is rich in antioxidant properties and became utilized in increasing the edible films (EFs) functionality. Potato starch films have been made from potato starch formulated with glycerol as plasticizer through casting method, in the course of which the granular and crystalline shape of starch became normally obliterated through heat and shear stress under the help of glycerol, so flexible films have been formed. Such films have been smooth to deal with and that they have been not sticky. The addition of plasticizers became vital to increase film properties, especially the mechanical ones. In the existing study, the physical properties of the potato starch-based EFs prepared with different concentrations of glycerol, potato starch, and orange essential oil have been analysed. The water-resistance and mechanical properties of the edible films have been progressed with the addition of starch. This examination proved that the orange essential and glycerol performed a vital function in the physical and mechanical properties of potato starch films. Such active packaging with edible films may be used for numerous food products, particularly low to intermediate moisture foods, in which the active transport of the orange essential oil can help to enhance the quality. The edible coating formulation is optimized by analysing the physical properties of the film. Optimized edible film formulation contains 9% starch, 3% glycerol, and 0.88 % orange essential oil. Films with orange essentials can be successfully used for edible film/packaging of fruits because films have desirable physical properties for packaging and also increases the antioxidant value of the film. Which in turn amplifies the shelf life of fruits due to the antioxidant property of orange essential oil. Further studies can be carried out on finding out the antimicrobial properties of the film.

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flavor.

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