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Preparation and characterization of edible film from Barnyard millet starch

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Abstract

Due to the cynical environmental impacts of synthetic plastics, there is an essential need for the development of edible films for industrial and many food applications. Researchers are creating various edible films based on starch for various food product applications. The present research work investigates the starch from barnyard millet and developed the edible film for packaging applications. These were developed by using barnyard millet starch, pectin, and glycerol with various amounts. The effect of the amount of additive on edible film thickness, moisture, solubility, swelling power, color, and transparency was investigated. Among all four samples B₄ having the highest thickness which provides a thickness of 1.5mm, tensile strength 1.15MPa. B₁ having the lowest solubility, swelling power, and transparency values. This research work contributes to the development of the barnyard millet starch edible film properties as an alternative food packaging by the addition of a different percentage of pectin. From this research the results show the application. This result shows the suitability of Barnyard millet starch for packaging applications.

Keywords: Barnyard millet starch, solubility, swelling power, tensile strength, transparency

1. Introduction

Edible films are made from Starch extracted from renewable biomass (Azahari *et al.*, 2011) [3]. The application and development of edible film in food applications are helpful to minimize plastic usage, as well as environmental pollution. The various properties of edible films provide a positive impact in society, and create awareness of edible film packaging and also fascinate the researchers and plastic packaging industries for the development of edible films (Siakeng *et al.*, 2019) [24]. Due to various properties of the film mainly the biodegradability nature of the film helps to encourage the researchers to develop the edible film for packaging purposes (Muscat *et al.*, 2012) [19]. In industries and household appliances plastics play a vital role. Increased use of polymers has contributed to significant ecological problems in recent years due to total non-biodegradability, particularly packaging materials and off-set plastic bags and cups. During the manufacturing of these polymers, the emission of carbon and many other dangerous gases cause environmental concerns. (Jain *et al.*, 2020) [14]. Commonly, polythene plastics such as low-density polyethylene and high-density polyethylene are typically used for the manufacture of different polyethylene plastics, and the major problem of these plastics is its non-degradability and cause environmental pollution. It was predisposed to over 1000 million tons of plastic waste as undesirable material, and it may take more days to decay. Once plastic waste materials are in the ground or water, they mix with water and form toxic chemicals, which can also pollute the quality of drinking water (Emadian *et al.*, 2017) [9]. Efforts are also being made to examine the creation of edible films in order to minimize the use of synthetic plastics and facilitate the use of edible films.

Present days biodegradable and edible films are prepared from starch. The production of starch-based edible films is simple, and they are widely used for packaging applications (Islam *et al.*, 2015) [12] & (Gadhav *et al.*, 2018) [10]. The tensile properties and thickness of the starch are suitable for the production of packaging materials, and glycerol and pectin are added into the starch as a plasticizer. By fine-tuning the quantities of the additives, the necessary features were obtained. This polysaccharide is generated as an energy store by most green plants. This carbohydrate is also part of the human diet, and it is present in large amounts in primary foods, including rice, cassava, corn, wheat, potatoes, and millets.

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The most significant starch among them is cassava starch, which contains cassava starch. Nowadays the biodegradable millet film also emerging due to these also having similar properties to other starches. pure starch is white. The starch powder does not provide any taste and odor (Sanyang *et al.*, 2016) [21]. When starch is exposed to heat, starch becomes a thick paste and it increases the viscosity of starch. In packaging materials, high amylose starch is a smart reserve for use as an obstacle. Because of the low price, renewability, ample availability of resources, and respectable mechanical properties, it was used to produce edible films to partly or completely substitute the plastic polymers to overcome the environmental pollution from these plastic solid waste. When the amylose content increases the tensile properties of bioplastic or edible film were increased (Ceseracciu *et al.*, 2015) [7]. As the barnyard millet have a higher amount of amylose content, the present work investigated that the films produced from pure starch were brittle and difficult to handle (Ghanbarzadeh *et al.*, 2011). This problem was solved by adding the plasticizers with varying concentrations. (Muscat *et al.*, 2012) [19] In order to produce the films, they researched the effect of low and high amylose starches. The tensile strength increases as the amount of plasticizer increases. Higher tensile strength is a note in films with high amylose content too. In this analysis, bulberry powder was used (Luchese *et al.*, 2018) [18]; corn starch and glycerol and bioplastic films were produced using the casting process, and the results showed that the film is used for food packaging. In this research, they used a casting technique to create the potato starch film for us (Zakaria *et al.*, 2018) [30]. They studied the tensile strength and morphological properties of the film by varying the mixing temperature. Through various studies were carried out on the starch for the development of edible films for the packaging applications (Siracusa *et al.*, 2008) [25] & (Jabeen *et al.*, 2017) [13], The analysis of barnyard millet starch for packaging applications is not included in the literature. Hence, the barnyard millet starch with varying concentrations of pectin along with glycerol in the present job. The main objective of this study is to produce edible films from starch extracted from the millet of the barnyard. This would be very useful for the developing countries where environmental pollution occurs by plastic usage and it creates an impact on the economy. The edible film prepared from barnyard millet starch was to exhibit properties that are comparable to the existing commercial packaging materials. The edible films were also found to be soluble in water and easily degradable, hence these can be used in edible soups bags, edible straws, pouches, thereby making it environmentally- friendly. These types of edible film formulations can be effectively used in packaging applications, due to their advantageous characteristics.

2. Materials and Methods

Barnyard millet was procured from valwill sudesi farmers Producers Company, Tamil Nadu. The chemicals, solvents, and reagents were procured from Himedia, India. All the used additives and chemicals namely sodium hydroxide (NaOH), ethanol, pectin, glycerol, pectin, and glycerol were purchased from Suresh scientific co., Trichy.

2.1 Extraction of starch

Extraction of starch from barnyard millet by aqueous alkaline steeping method (Wang *et al.*, 2014) [18] & (Wani *et al.*, 2010) [29] with slight modification. One kilogram of the barnyard millet and rice was soaked deionized water (1:4) containing 4

g of alkali and kept at 4°C for 12 h. Wet milling by using a colloidal ball mill (Pilot smith India private limited) at a temperature of 28°C ±2°C with a speed of 3000 rpm for 10 minutes and followed by filtering the slurry through 60ms(mesh screen), and centrifuged at 5000 rpm for 10 Min. the precipitate was obtained after removing a yellow coating from the soil. The precipitate was infused and centrifuged with deionized water after removal. Again, the upper layer was removed by deionized water to remove alkali. Ethanol is added to the starch and keep in refrigerate condition subsequently washing with deionized water and centrifugation. Centrifugation is repeated up to clear white color appears. The extracted starch was dried in a tray drier at 60°C for 5 hours, then sieved through a 159-mesh screen and stored in an airtight container for further analysis.

2.2 Preparation of barnyard millet edible film

Using the casting process, the edible film was prepared. In different amounts, starch, pectin, and glycerol were added to 100 ml of distilled water. The mixture was stirred at a rate of 360 rpm for 15 min. then the mixture was heated on a hot plate at 70 °C, and manual stirring was done for 30 mins, continuously. It was then poured onto a glass tray and spread uniformly. Once set, the trays were kept in a tray dried at 30°C for 10 hours. After drying the edible film peeled out from the glass tray. Then, four samples were prepared for different compositions of pectin, as shown in table.3. The films were then analyzed (Aisyah *et al.*, 2018) [1].

Table 1: Composition of prepared edible films

Sample	Barnyard millet starch	Glycerol	Pectin	Water
B ₁	5%	1%	0.5%	100ml
B ₂	5g%	1%	1%	100ml
B ₃	5g%	1%	1.5%	100ml
B ₄	5g%	1%	2%	100ml

3. Characterization

3.1 Conditioning

All edible film samples were conditioned using a humidity chamber at RH 65 and 25°C. Films samples were transferred to zip lock covers after conditioning for further analysis. Samples were before subjecting them to permeability and mechanical tests according to a standard method, D618-61 (ASTM,1993). Films are used for testing moisture content, color, thickness, transparency, tensile strength (TS), and sensory properties (Sobral *et al.*, 2001) [26].

3.2 Moisture content

Moisture was estimated by (Lalnunthari *et al.*, 2019) [17] method. Two grams of barnyard millet sample was weighed in moisture dishes after noting down the empty weight of moisture dishes. The dishes were kept in a hot air oven at 130°C 3 hours. The dishes containing samples were cooled in a desiccator and the final weight was noted down. The above procedure followed for each sample such as barnyard millet edible film. The difference in initial and final weight was expressed as the percentage of moisture in the foodstuff as shown in the below equation.

$$\text{Moisture (\%)} = \frac{w_2 - w_3}{w_2 - w_1} \times 100$$

Where

W₃ is the weight of the dish with a dried sample.

W₂ is the weight of the dish with the initial sample.

W₁ is the weight of an empty dish

3.3 Colour

A Hunter Color lab (Model: D25 optical sensor; Hunter laboratory associates, Reston, VA) was used to test the color of the edible films and the findings were expressed in terms of $L^*a^*b^*$ values. (brightness 100), or lightness (0), a^* (0+ redness/-greenness), b^* (+ yellowness/-blueness) denotes the value L^* . For lightness (0), b^* (+ yellowness/-blueness), a^* (0+ redness/-greenness) (Sayanjali *et al.*, 2011) [22].

3.4 Thickness

The thickness was noted after conditioning the strip for 48 hours in a humidity chamber at 25°C and 65 RH. The three readings of strips were taken randomly using a precision digital caliper meter (INSIZE) and the average was noted as final thickness (Aisyah *et al.*, 2018) [11].

3.5 Tensile strength

Using the universal test machine, the calculation of tensile strength was carried out. The tensile strength values were determined by the division between the maximum force (F) and the surface area (A) of the edible film (Aisyah *et al.*, 2018) [11].

3.6 Film transparency

The transparency of the films was measured in accordance with (Hosseini *et al.*, 2013). The proportions of the film with the uniform dimensions preserved in the test cell for each film

$$\text{swelling power} = \frac{\text{weight of wet sediment}}{\text{weight of dried starch}} \quad \text{solubility index} = \frac{\text{weight of wet sediment}}{\text{weight of dried starch}} \times 100$$

3.8 Sensory analysis

Barnyard millet edible film was analyzed for its acceptability using 7 points hedonic rating scale by 20 semi-trained panelists. The developed barnyard millet edible film was judged by 20 semi-trained panelists to check the overall acceptability and also to find the suitable edible film for the edible packaging purpose (Kulawik *et al.*, 2019) [16].

3.9 Sealing properties of barnyard millet edible film

Bar sealing is often used for creating a seal from sealing machines; the strongest technique is band sealing. The important component in the heat-sealing process is the sealing pressure and sealing temperature. When making a decent seal, these two components are important. When the film sample is exposed to heat, the film will melt the sealing layer during the heat-sealing process to molten or partially molten impact on the film.

4. Results and Discussion

4.1 Moisture content for edible films

The Moisture content of various samples was calculated, the results are shown in the table. 2. B₁ having the least water absorption, however, B₂, B₃, and B₄ revealed higher moisture content. As the pectin concentration increases the moisture content of the film increases gradually. The hydrophilic compounds would increase the solubility of the film. However, the hydrophobic compounds would decrease it (Kavoosi *et al.*, 2013). The same trend was observed in the water solubility of barnyard millet starch. In this present study, the solubility of all starch films followed the same tendency as per the expectation, while the hydrophilicity of film increases with increasing pectin concentration. From this, relative to the B₄ sample, we can assume that the moisture content in sample B₄ has a higher moisture content. The B₄

and the empty cell used as reference in the UV spectrophotometer (Shimadzu, UV-1800). An average of three measurements for each film was taken at 600nm. The transparency was calculated by the following equation.

$$\text{Transparency} = \frac{-\log T}{x}$$

Where T = transmittance at 600 nm and x = thickness of the film (mm). This equation implies high transparency indicates less transparent and high opaque film.

3.7 swelling power and solubility index

Swelling power and solubility of Banayard millet edible were evaluated by the method of (Nwokocha *et al.*, 2009) & (Gautam *et al.*, 2016) [11] with slight modification, and results are expressed in g/g of dry starch. 0.1 g of the edible film was mixed with 10 ml of distilled water in a 50 ml centrifuge tube and heated in a water bath at a temperature ranging from 50, 60, 70, 80, and 90°C (Arowora *et al.*, 2013) for 30 min. After heating, the suspension was centrifuged at 1500rpm for 15 min. the supernatant was carefully removed and edible film part sediment was weighed. The supernatant was taken in a pre-weighed Petri plate and evaporated at 4 h at 120°C. The residue obtained after drying of supernatant determines the amount of film solubilized in water. The results are expressed by using the below equations.

sample has more solubility than the B₁ sample due to the hydrophilicity of pectin and a higher level of glycerol. We infer from this research that the moisture content in sample B₁ has the lowest value, which increases the shelf life of edible film in packaging materials.

Table 2: Moisture content of edible films

Sample	Initial weight W ₁ (In gram)	Final weight W ₂ (in gram)	Moisture content in percentage (%)
B ₁	10.2	10.3	10.2
B ₂	11.7	11.8	11.73
B ₃	12.3	12.2	12.26
B ₄	13.6	13.5	13.5

4.2 Colour profile analysis

The packaging films, which are used for wrapping food products have direct contact with the food product and significantly contribute to the appearance and consumer acceptance of the food products. The color value of the edible film is shown in table. 3.

The highest L value present in the B₁ compare to the B₂ and Highest redness is seen in the B₂ sample and the highest yellowness seen in the B₁ sample compare to the B₂, B₃, and B₄ samples. The lower lightness and redness value exhibited due to the transparent nature of the films. B₂ and B₄ samples having lower lightness values compare to the B₁ and B₃ samples. B₂ is light color material compare to other samples and it provides the transparent and translucent where the B₁ provides opaque.

The transparent features provide visibility to the packaged food. And it also attracts the customers due to its transparency it can easily notice the sample present inside the packet, by this we can easily identify the type of sample present in it

without tearing the packaging material (Sayanjali *et al.*, 2011) [22].

Table 3: Colour value of the edible film

Color measurements	L*	a*	b*
B1	53.66	0.02	6.09
B2	40.8	0.03	3.94
B3	50.91	0.16	0.47
B4	45.74	0.03	4.16

4.3 Thickness

The thickness of the edible film is measured by using a precision digital caliper meter (INSIZE) and the average is calculated and showed in the table. 4.

The results show the prepared edible films have a thickness range from 1.1 to 1.5mm. (Jouki *et al.*, 2013) [15] found that the thickness value of corn starch films was approximately 0.15mm. Other researchers by (Bakumov *et al.*, 2012) [4] studied the thickness of several starch films made up of potato, rice, wheat, and sorghum and found 53 to 63 microns, which much lower than the present study.

In the current work, the thickness is higher, which may be due to the presence of barnyard millet starch. And the increasing concentration of plasticizer leads to the higher edible film thickness due to plasticizers will inhabit the open pores present in the film network and interact with edible film to form a polymer, it results in increases the distance between polymers, hence enhancing the film thickness (Arham *et al.*, 2018) [2].

The thickness of the B4 is more than the remaining sample. The thickness of the film ranges between 1.1 mm to 1.5 mm. The difference in edible film thickness can be caused by several factors, such as the difference between the edible film being poured into the mould and the drying temperature. Edible film thickness can adjust by varying the amount of solution poured into the mould and the area of mould used. The more volume of edible film solution is poured, the thicker edible film will be obtained. It is because the total solid in the edible film solution gives more thickness (Bourtoom, 2008) [6].

Table 4: Thickness measurements of edible film

Sample	Thickness
B1	11. ±0.1mm
B2	1.2 ± 0.1mm
B3	1.4 ± 0.1mm
B4	1.5 ± 0.1mm

4.4 Transparency

Transparency films are important sensory indicators of edible films. The value of transparency Obtained by the method of (Shi *et al.*, 2013) [23] is inversely proportional to the degree of transparency of the edible film. The transparency value of edible film made from BMS (barnyard millet starch) plasticizers and additives can be seen in table.5.

The addition of plasticizer and pectin increases the value of transparency so that the degree of transparency edible film

decreases. B₁ Film with a low value of plasticizer and pectin produces clear transparent film, whereas the B₄ film with plasticizer and pectin to produce a film with opaque.

This may be because of high molecular weight and carboxyl groups present in pectin are relatively low. B₁ films having good sensory properties compared to B₄.

Table 5: Transparency of edible film

Sample	B1	B2	B3	B4
Transparency	2.34±0.05	2.48±0.05	2.51±0.05	2.74±0.05

4.5 Tensile strength

The value of tensile strength increased with the increase of pectin concentrations. The highest tensile strength was 1.16 Mpa, obtained by the addition of 2 g of pectin. Glycerol as a plasticizer, made the film more flexible as the intermolecular bonds between the polymer chains were reduced and mechanical properties were modified. In the present research, we can conclude that along with starch food additives also play major a role in enhancing the tensile strength of the edible film. In table 6. it is shown that the B₄ having good tensile strength and having more mechanical resistance compare to compare to B₁, by this research we can conclude that by increasing the tensile strength of edible film by increasing the amount of pectin concentration (Aisyah *et al.*, 2018) [1].

Table 6: Tensile properties of different samples

Sample	B1	B2	B3	B4
Tensile strength	0.65±0.05MPa	0.73±0.05MPa	1.04±0.05MPa	1.16±0.05MPa

4.6 Solubility index

The solubility of edible films in water constitutes a dry weight of edible films that has dissolved in water at a different temperature like 50°C, 60 °C, 70 °C, 80 °C and 90 °C for 10mins. The water solubility of all samples was observed in Fig 1. The Edible film with the lowest solubility indicates the best quality of the edible film. The edible with 0.5g concentration pectin having the lowest solubility compare to the 2 g of pectin concentration. The solubility of the edible film in water is influenced by the composition of the materials used. The solubility of the edible film in water is increasing with increasing the amount of pectin percentage. The plasticizer having a hydrophilic character due to this nature it will increase the solubility as we increasing the amount of plasticizer. (Bourtoom, 2008) [6]. In present research shows that the solubility was observed to be associated with an increase in the temperature and solubility will increase with increasing temperature. The highest solubility was observed at 90°C. The solubility mainly is related to the two-stage of relaxation of bonding forces within the starch granules at the second stage and also low amylose that influences higher starch granule disintegration. Since pearl millet, finger millet, and corn having low amylose content, these starch granules are easily disintegrated at a temperature below 65°C (Beverly *et al.*, 2008) [5].

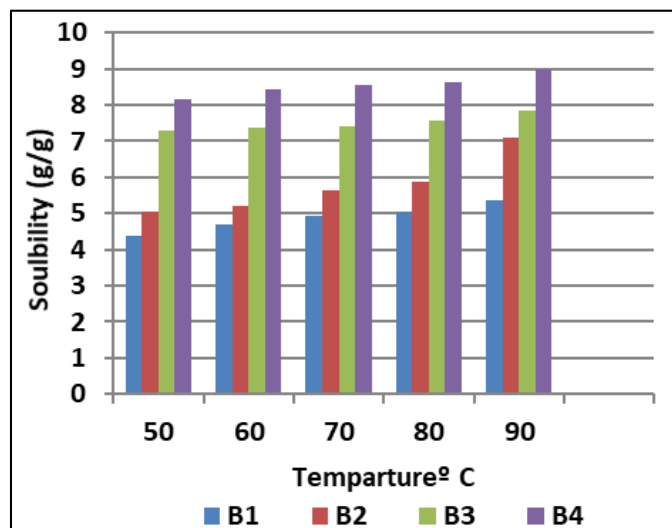


Fig 1: Effect of temperature on solubility (g/g) of barnyard millet edible film

4.7 Swelling power

The swelling power of edible films in water constitutes a dry weight of edible films that have dissolved in water at a different temperature like 50°C, 60 °C, 70 °C, 80 °C and 90 °C for 10mins.

The water solubility of all samples was observed in Fig 2. The Edible film with the lowest swelling indicates the best quality of the edible film. B₁ having the lowest solubility at 50 °C. The solubility of the edible film in water is influenced by the composition of the materials that are used. The increased concentration of plasticizer added will increase the solubility of the edible film in water (Bourtoom, 2008) [6].

It is because the addition of a higher amount of plasticizer which has a hydrophilic character will increase the swelling of the edible film in water. The highest swelling power was observed in B₄.

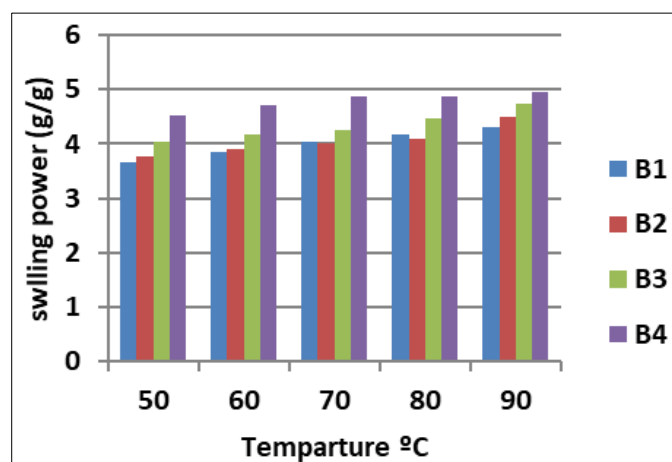


Fig 2: Effect of temperature on swelling power (g/g) of barnyard millet edible film

4.8 Sensory analysis

Barnyard millet edible film samples were analyzed by 20 semi-trained panelists as shown in figure.3 all the samples such as B₁, B₂, B₃, and B₄ were analyzed for their acceptability with a 7-point hedonic scale. The overall acceptability was high for B₁ followed by B₂, B₃, and B₄ with scores of 6.21, 6.1, 5.3, and 5.1 respectively (Cruz *et al.*, 2015) [8].

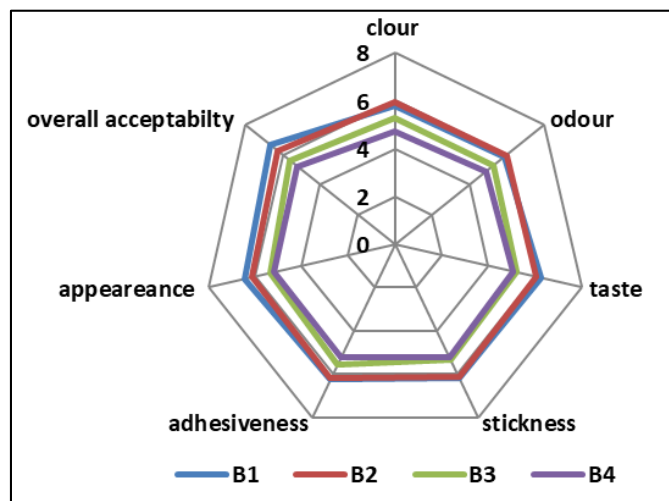


Fig 3: Sensory analysis of barnyard millet edible film

4.9 Sealing properties of barnyard millet edible film

It is observed that no single temperature is accepted for the heat-sealing process. This may show due to the reason that the plastic was usually sealed at moderate molten or melting conditions.

The acceptable range of temperature is different for different products. A particular range of temperature is set for different plastics as an acceptable sealing temperature, based on this temperature range, which good seal will be developed and prepared for the products within this temperature range. Dwelling time means the interval time that the coated film is brought into close contact with the heated film. The results indicated that the prepared edible film samples have good sealing capabilities. The main purpose of the sealing is to squeeze the two layers of film to get as great a molecular contact over as much of the sealing area as possible, by using these the bags/pouches were design. The heat-sealing of edible film samples were estimated through visual inspection. The sample was inspected manually. Since sealing properties are important for preparing edible film pouches, hence it is concluded that the edible films produced in this research we can be used to manufacture edible pouches or bags. B₁ has the best sealing properties compare to the B₄ sample. A sample edible film pouch is shown in fig.4



Fig 4: Edible film pouch

5. Conclusions

The results showed that the samples prepared from the barnyard millet starch have better properties compare to the existing biofilms and edible films. The varying composition of pectin increases the thickness and tensile strength of the edible film. B₁ having the best transparency values. The average moisture content is 11.92%. The maximum tensile strength of edible films is found to be 1.15 MPa. From the above results, it can be concluded that barnyard millet edible film can be used as packaging materials due to its solubility, tensile properties, and sensory properties. we can use as an alternative to LDPE and HDPE plastic bags, Due to the obtained properties of barnyard millet edible film, it would be interesting to prepare edible soup bags using these edible films with assumed lower cost. Development of the edible film with other materials, and with different proportions of plasticizers, would be an interesting scope for this research.

6. Acknowledgments

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7. Conflicts of interest

The authors declare that there is no conflict of interests.

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