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*Research Article*

# Analysis of walnut pecan flour (*Carya illinoinensis* var. Shoshoni) for high-quality dried fruit leather production

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## Abstract

The objective of this study was the analysis of the physical, chemical, and functional properties of walnut flour pecan (WPF, *Carya illinoinensis var. Shoshoni*) extracted from oil. The study also involved producing high-quality dried fruit leather using apple juice and walnut flour pecan and evaluating its sensory and qualitative characteristics and their stability during storage. The approximate composition, color, water absorption, total polyphenols, and peroxide index of walnut flour pecan were determined. The WPF presented high polyphenols content (23.0±0.1 mg GAE/g), lipids (33.1%), and proteins (12.7%). The mixture of apple juice, WFP, and honey was dehydrated at 70°C for 4 hours, characterized, and subjected to storage. A sensory study was conducted to obtain information about the product's potential. The results showed that consumers rated the product as tempting, appetizing, and curious. Consumers characterized the taste of the dried fruit leather as sweet and slightly acidic, indicating a positive association with the product. During the 63 days of storage at 25°C and 75% relative humidity, the pH values ranged from 4.25±0.03 to 4.32±0.03, and the calculated peroxide index was not higher than 0.020 mequiv $O_2$/kg of dried fruit leather. A decrease in moisture content during storage, associated with a progressively increased fracture force, was observed. Total polyphenols content presented a significant loss but was preserved during storage. This product could be a healthy and innovative snack option and is also relevant to mention the added value of walnut flour pecan.

## 1. Introduction

Among the main trends, consumers look for foods with additional benefits. These changes in eating habits demand the challenge of designing attractive foods that incorporate different nutrients and generate healthier food choices. Fruit leathers restructured products that are widely consumed as tasty and healthy snacks in many parts of the world due to their natural ingredients and nutritional content. It is a convenient option to substitute fresh fruits and increase the intake of various nutritional elements such as dietary fiber, carbohydrates, minerals, vitamins, and antioxidants [1]. At present, several formulations for fruit leathers with different ingredients. In its preparation, purees, concentrates, or juices of apples, pineapple, pear, bananas, or plums have been used [2-6]; sweeteners such as sucrose, corn syrup, or honey and food additives such as citric pectin, glycerin, chemically modified starch, pectin, gelatin, alginate, gums, and cellulose derivatives have been incorporated to improve the spreading of the pulp and subsequent drying process [7-8]. The process of obtaining fruit leathers is carried out by eliminating the moisture content of the mixture, until reaching a moisture content of about 0.3 kg water/kg dry matter, which corresponds to a water activity of 0.70, sufficiently low in acid medium to inhibit mold growth at room temperature [1,9]. This is dried generally by hot air and being a dry product so that they do not usually require cold storage to avoid microbial growth.

Pecan nut cake is a by-product of low commercial value from the oil industry. Despite its high oil content, nutritional value, and pleasant sensory characteristics, the pecan nut cake is only used as animal feed [10]. Pecan nut [*Carya illinoinensis (Wangenh.*) *K. Koch*] cake is a source of carbohydrates, protein, dietary fiber, and minerals (Ca, Mg, K, Mn, Na, and Fe), it also contains polyunsaturated lipids (linoleic acid) and other bioactive compounds (phenols and tocopherol), and that content depends on the extraction method [11-12]. Some studies have reported the potential use of walnut cake in bakery products [13-14]. The production of press cake leads to walnut flour, with similar characteristics, being rich in polyunsaturated lipids, a source of protein, fiber, minerals, and other bioactive compounds [12]. Due to its qualities, it would be very beneficial in the food industry in a large number of foods such as, for example, fruit leathers.

Walnut flour has numerous qualities that make it a valuable ingredient in the food industry, particularly in products like fruit leathers. Its high nutritional value can enhance the nutritional content of these candies and provide important nutrients. The objective of this study was to analyze the physical, chemical and functional properties of walnut flour pecan extracted from oil. The study also involved producing high-quality fruit leather using apple juice and walnut pecan flour, and evaluating its sensory and qualitative characteristics, as well as its stability during storage.

## 2. Materials and methods

*2.1 Materials*

Walnut flour pecan (*Carya illinoinensis var. Shoshoni*) was obtained from local producers in the Entre Ríos Province, Argentina (Noyer®). It was packaged and stored at 4 °C until use. A gel (a product based on gum gellan low acyl) (Biotec ®, Argentina). The apple juice (45º Brix, Baggio®, Argentina) and honey of flowers (La Entrerriana®, Argentina) were purchased from a local supermarket. All reagents used were of analytical grade from Sigma-Aldrich (St. Louis, MO, USA).

*2.2 Walnut walnut flour pecan: physicochemical and functional characterization*

*Proximate composition:* performed according to AOAC [15] considered the following methodologies: moisture content (925.09), lipids (920.85), ash (923.03), crude protein (920.87) and crude fiber (991.43), carbohydrates were determined by difference.

*Color measurement:* was determined with a portable photocolorimeter (HUNTER LAB, MiniScan EZ, USA) after calibration with black and white reference mosaics. Color functions were calculated for illuminant D65 at 2° from the standard observer, and L*, a*, and b* (CIELab) parameters were obtained. The sample was placed in a black box during the measurement to avoid light entering the optical beam.

*Water absorption capacity (WAC, %):* was determined using the method proposed by Chandra et al., [16]. One gram of sample was mixed with 10 mL of distilled water and allowed to stand at room temperature (30±2 °C) for 30 min, then centrifuged at 3000 rpm for 30 min. WAC was expressed as per cent water bound per gram flour.

*Determination of total polyphenols content (TPC):* was determined using the Folin-Ciocalteu reagent as described by Maciel et al., [11] and Sarkis et al. [17]. The results were expressed as mg gallic acid equivalent (GAE) per g sample when compared to a calibration curve (0–0.20 mg GAE/mL, $R^2 = 0.99$).

*Peroxide value:* was determined by the official method Cd 8-53 of the AOCS [18].

*2.3 Apple leathers with walnut flour pecan development*

In a previous investigation, the formulation was optimized using a Box-Behnken experimental design. The designed formulations varied in contents of walnut pecan flour, gel, and apple juice. The formulations were completed with 30.3% of honey (ingredients in Section 2.1). In the study, several product properties were evaluated, including moisture content, puncture resistance, color, and peroxide value. It resulted in a formulation that contained 5.0 % (p/p) of walnut pecan flour, 0.7 % (p/p) of gel, and 64% (p/p) of apple juice. In addition to validating these levels, a qualitative sensory and acceptability analysis was carried out, thus ensuring that the fruit leather responds to consumers' expectations.

*2.3.1 Preparation*

First, the gel and walnut flour pecan were mixed in the specified proportions. The honey was then heated to 105°C for 1 minute to increase the viscosity and immediately cooled to 50°C. The apple juice and the flour-gel mixture were added when they reached temperature and mixed until homogenized (10 minutes). It was then heated to 80°C for 1 minute to activate the gelling power of the gel used, which is based gellan gum low-acyl and requires this temperature to activate. After this step, the mixture was distributed evenly (15 g) into glass Petri dishes (60 x 15 mm) and dried using hot air at 70±2°C to achieve a final moisture content of 0.28 g of water per gram of dry solid (4 hours). The fruit leather samples were stored in a dry place at 25±2°C and protected from sunlight for further analysis.

The time heated honey was less than 2 minutes to avoid increasing the ratio of hydroxymethylfurfural (HMF) present.

*2.3.2 Sensory study*

*Qualitative sensory study*: Participants were recruited through an online survey distributed via social networks and email (n=220). The survey presented a description of the product together with an illustrative photograph (Fig. 1) and they were instructed to express in one word the first impression or sensation generated by the product.

**Figure 1** Apple leathers with walnut flour pecan.

All the words provided by the participants were taken into consideration. The frequency for each word was determined, and a word cloud was created with the Word Art web-based free program. Finally, the opinion of the consumer about this product was investigated, providing response options from positive to negative, using an eight-point scale ("I dislike it very much" to "I like it a lot").

*Evaluation of the level of taste of ingredients with a consumer:* considering that the different ingredients in foods influence the acceptability of the product, we proceeded to evaluate the perception of flavor and texture among potential consumers. According to Huang and Hsieh [19], sweetness and acidity were positively correlated with the overall liking of leather fruit, while consumers perceived the hardness attribute negatively. The leathers were prepared the day before the test and packaged in an aluminum container. They were subjected to sensory evaluation by an untrained panel of 74 panelists between 18 and 62 years of age and members of the university staff. Sensory attributes evaluated were flavor (acid, sweetness, nut, honey) and texture (hardness: the force required to bite completely into a sample placed between molars and stickiness: the amount of product adhering to the teeth after chewing the product). Before carrying out the sensory evaluation, the panelists were instructed in the terminology of each attribute. A 9 cm linear scale was used with defined scores: little, medium, and much. Evaluators were asked to make a vertical mark on the point representing their score.

*2.3.3 Fruit leather characterization*

*Moisture content:* was determined by the method N° 925.09 [15].

*pH*: the samples were crushed and immersed in distilled water (10%, w/v) for 24 h at 25°C, then filtered through filter paper. Aliquots were used to measure pH with a laboratory pH meter (MicropH 2001, Crison, Barcelona, Spain).

*Color measurements:* It was determined with a portable photocolorimeter (HUNTER LAB, MiniScan EZ, USA) after calibration with black and white reference mosaics. Color functions were calculated for illuminant D65 at 2° from the standard observer, and parameters L*, a*, b* (CIELab) were recorded and the browning index (BI) was calculated according to Quintero Ruiz et al., [5]. Reference values used were $X_n$= 80.03, $Y_n$= 84.83 and $Z_n$= 88.77, respectively.

*Texture property:* The analysis was performed with an Instron 3342 texture analyzer (Norwood, USA). The leathers were cut into 40×30 mm rectangles and placed on a plate with a perforation in the center. A cylindrical probe with a diameter of 3 mm was used, the measurement speed was 0.5 mm/s and the force load was 500 N. Force-displacement curves were plotted and recorded the point of maximum force at fracture ($F_{max}$), which refers to the force required to pierce the fruit leather.

Determination of total polyphenols content (TPC) and Peroxide value: were determined as described in point 2.2

*2.3.4 Storage studies*

The samples were kept at 25°C and 75% relative humidity in a controlled temperature and humidity oven for 63 days, using an aluminum wrap to provide adequate protection from light. The samples were withdrawn for analysis every seven days to evaluate the changes in the characteristics of maximum force at fracture ($F_{max}$), pH, moisture content, and BI, obtaining a total of 10 points to be analyzed. In the case of the peroxide index and total phenols, the analyses were proposed every ten days, resulting in seven analysis points.

## 3. Results and discussion

*3.3. Walnut flour pecan physicochemical and functional characterization*

The proximal composition of WFP on a dry basis is shown in Table 1.

**Table 1.** Physicochemical and functional characterization of walnut flour pecan and fruit leather

| Parameters | Walnut flour pecan | Fruit leathers |
|---|---|---|
| Lipids (%) | 33.1 | 0.21 |
| Proteins (%) | 12.7 | 0.64 |
| Crude fiber (%) | 8.18 | |
| Moisture (%) | 6.92 | 21.6 |
| Ashes (%) | 4.10 | 0.45 |
| Carbohydrates (%) | 35.0 | 77.1 |
| WAC (%) | 210±12.0 | |
| Total phenol contents (mg GAE/g sample) | 23.0±0.1 | 0.058±0.001 |
| Peroxide value (meq$O_2$/kg sample) | 1.10±0.20 | 0.015±0.001 |
| pH | | 4.32±0.03 |
| L* | 40.7±0.05 | 16.4±0.47 |
| a* | 7.93±0.01 | 5.33±0.41 |
| b* | 19.8±0.02 | 13.0±0.61 |

The lipid content of the sample was lower than reported by some authors [10, 17], but higher than that found in defatted pecan (*Carya illinoensis*) cakes by Maciel et al., [11]. In terms of color attributes, as shown in Table 1 the sample presented an L*-value, lower than the ones previously informed by Maciel et al., [11] for nut cake; this suggests a color brown dark that is likely to affect color when added to a product. The a*-value and b*-value; were higher than the ones obtained in cake nut. The WAC value for walnut flour pecan shown in Table X is higher than the records for wheat flour (140±12.3%) and wheat and rice flour blends [16] and for what was reported for derived flour *Tetracarpidium conophorum* (African walnut) defatted (108±40%) and non-fatted (103±3.4%) [20]. The protein content, as well as its degree of interaction with water and conformational characteristics [21], may be determining factors in its high water uptake. In addition, WAC plays an essential role in improving the texture, stability, and flavor of food products [11]. The total phenol content of the WFP was similar to those obtained by the authors Maciel et al., [11] (14.8±0.38 mg GAE/g) and higher than those obtained by the authors Sarkis et al. [16] (6.8±0.3 mg GAE/g). The presence of phenolic compounds would be related to the brown skin that covers the kernel, which is present in the press cake [12]. Walnut flour pecan presented a peroxide value was lower than that found by Salvador et al., [10] and by dos Santos, et al., [22] for pecan nut cake (Table 1).

*3.4. Evaluation of the consumers' expectations, characterization, and storage of the fruit leathers.*

The development of new products implies a sensory study, which allows linking the product formulation, storage conditions, and processing parameters with consumer responses and, therefore, obtains information about the potential of a product [23]. Since adding new ingredients to apple skin can affect color or flavor [6, 24], resulting in a less attractive product to the consumer, it is relevant to obtain this information.

*3.4.1. Qualitative sensory study*

The qualitative sensory study showed results on consumer opinion regarding the idea of apple leather with walnut flour designed. The analysis of the question: Would you buy this product? indicated that the surveyed 58.3% would buy the product (n = 127), 33.5% answered maybe, and only 8.3% responded that they would not buy the product. These results show interest in the product. Word association analysis showed that the categories mentioned by a higher percentage of the respondents were "tempting", "appetizer", and "curiosity" (Fig. 2). These indicate a positive idea associated with the product.


**Figure 2.** Word-cloud presenting the word of the first impression or sensation generated by the product on the consumer (n=220)

The evaluation of consumers' opinions about the leather is represented in Fig. 3. From 219 responses, 27.9% (n=61) of consumers responded that "I neither like nor dislike it", 25.1% (n=55) answered "I like it quite a lot", 20.1% (n=44) said "I like it a lot", and 13.7% (n=30) indicated, "I dislike it slightly".

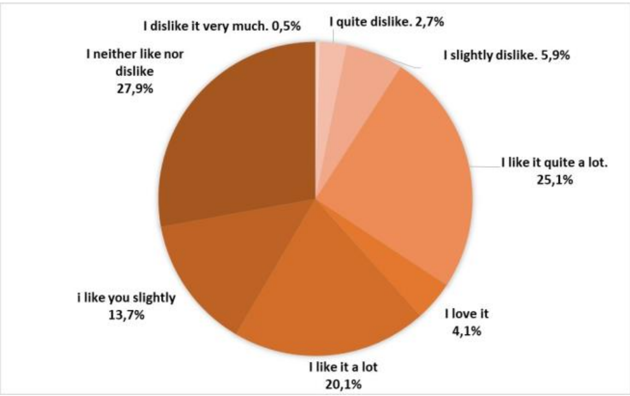


**Figure 3.** The evaluation of consumers' opinions about the leather

*3.4.2.Evaluation of the level of taste of ingredients with a consumer panel.*

The aim of the study was to determine how different ingredients affected the flavor and texture of the leather. Specifically, the study sought to determine whether consumers could perceive flavors like nuts and honey, and to define the level of acidity contributed by apple juice. The results showed that consumers perceived the leather as sweet, possibly due to the higher perception of honey flavor compared to walnut flavor, which was barely detectable (Fig. 4). The high content of apple juice in the leather may have masked the flavors of the other ingredients (walnut and honey). Fig. 4 shows the results of the texture properties of leather which was found to be desirable by consumers, as it required little force to cut and did not stick to teeth after chewing [3]. Overall, the study provides valuable insights into the sensory properties of fruit leather.

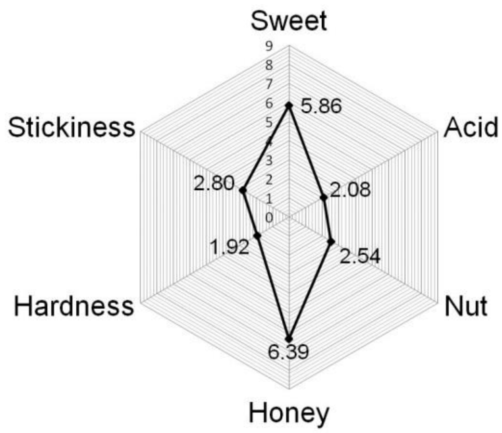


**Figure 4.** Scores of the attributes measured by consumers for taste and texture properties

*3.4.3. Fruit leather characterization*

The sample's moisture content was 21.6%, within the typical range for apple leather [5-6, 9]. The chemical composition of the fruit leather is presented in Table 1, highlighting that the contribution of lipids and proteins to the WPF are low. In contrast, carbohydrates represent a significant proportion of the total (77.1%), mainly due to their main components, apple juice and honey. The value pH (Table 1) of the fruit leather indicates that the leather was slightly acidic, which may confer a higher preservation quality [25]. In addition, the sample exhibits pH values similar to those of a wide range of fruit leather developed [5-6, 26]. Color parameters are influenced by the type of ingredient (fruit, additive, sweetener) in the formulation. The value parameters L*, a* and b* are presented in Table 1, where it is seen that the values in the fruit leather are lower than in the walnut flour pecan sample. It is evident that the L* value of the fruit leather is significantly lower compared to the values reported by the authors for apple leather [26-27]. This decrease in the L* value can be attributed to the incorporation of walnut pecan flour, as previously mentioned. Additionally, the value for parameter a* closely resembles the findings of Valenzuela and Aguilera [28], whereas the value for parameter b* is comparatively lower. From the force-displacement curves, the point of maximum fracture resistance for fruit leather was determined. The average value was the same as 0.752±0.048 N, which was lower than those found in the literature for these types of products [2, 28]. The peroxide value of leather apple with walnut flour pecan allows us to predict that the oil in the walnut flour would not undergo significant oxidation of the fatty acids present during drying (Table 1). The total polyphenols content (TPC) of the fruit leather, as shown in Table 1, indicates a noteworthy reduction in polyphenols compared to the values found in walnut pecan flour. This decline in polyphenols might be attributed to the drying temperature or oxidation reactions that potentially caused the degradation of these compounds. Interestingly, this effect differs from what was reported in the drying process of blueberry leather by Chen and Martynenko [28].

*3.4.4. Storage studies*

The pH values during storage did not change significantly, presenting a range between 4.25±0.03 to 4.32±0.03, as did the peroxide index calculated for the leathers, with values no higher than 0.020 mequiv $O_2$/kg leather. The puncture test to characterize the leather texture showed that the force at fracture increased progressively during storage time (Fig. 5a) and was not higher than reported for Valenzuela and Aguilera [27] in apple leather. The increase in fracture strength throughout storage is related to a decrease in the moisture content of the leather (Fig. 5b). According to Roudaut et al. [29], this effect could be interpreted by an anti-plasticizing mechanism, where the hydration facilitates molecular reordering, responsible for interactions between water and matrix macromolecules.

The color of the leather was probably due to the dark color of the pecan nut flour present in the formulation. The Browning index, calculated from the mean values of L*, a*, and b* (Fig. 6a), showed an increase in storage time. When comparing the data with those apple leathers elaborated by Torres et al., [5], it was found that the values obtained at the beginning of storage were similar, while at the end of storage (2 months), they were almost twice as higher than those reported. Therefore, the increase in BI could be due to browning caused by non-enzymatic reactions and oxidation of other molecules, including phenols present in walnut flour pecan and apple juice [30, 31], this would be related to the loss of total polyphenol content over storage. The loss of total content of polyphenols was significant after 10 days of storage,

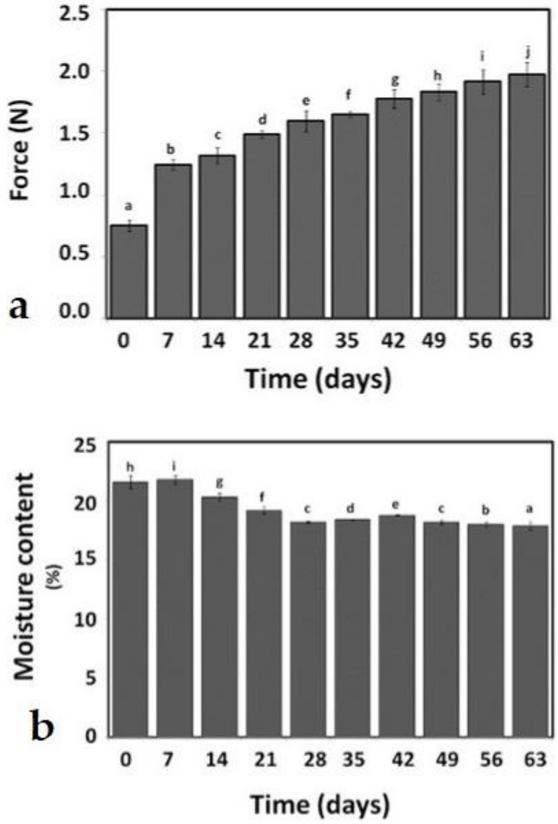


**Figure 5.** a) Fractured force maxima behavior of the fruit leather during storage. b). Content moisture of the fruit leather during storage. Different letters indicate significant differences $p \leq 0.05$

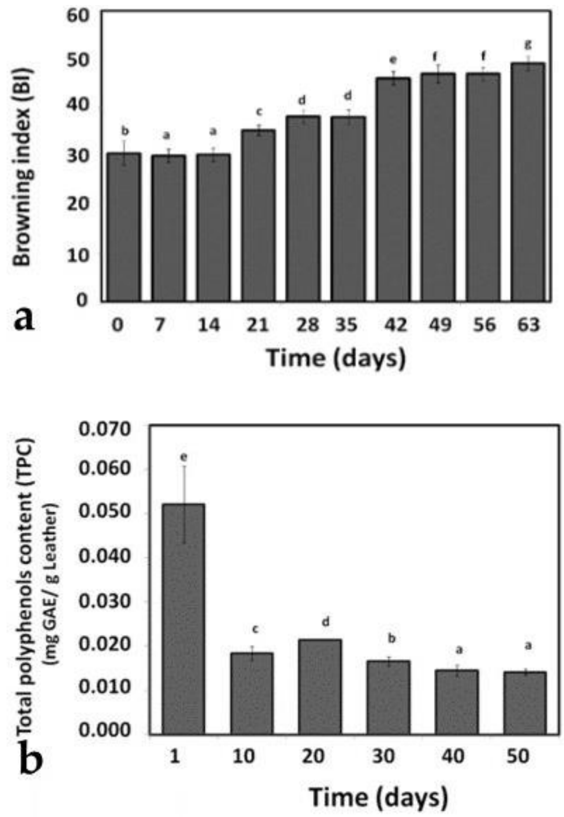


**Figure 6**. a) Color variation in leathers represented by the browning index (BI) as a function of time storage. b) Total polyphenol content of the fruit leather during storage. Different letters indicate significant differences $p \leq 0.05$

as shown in Fig. 6b. According to Torres et al. [6], the heat treatment produced during leather preparation would inactivate the polyphenol oxidase enzyme (PPO, EC 1.14.18.1) in this case, both the processing and drying temperatures of the leather were not sufficient to prevent polyphenol degradation during storage.

## 4. Conclusions

The qualitative and quantitative sensory study conducted with consumers showed that fruit leather made with walnut flour was well-received. There was a perception that the leather had a sweet taste without the walnut flavor. The lower hardness and stickiness of the leather were perceived as positives. The walnut flour contributes to the bioactive components of the leather, which shows that leather could become a functional food. During a two-month storage period, the pH and peroxide index remained unchanged. There were some changes in moisture content related to texture, but remained within the range for this type of product. The phenol content decreased over time. These findings suggest that fruit leathers with walnut flour could be a natural, innovative, and functional confectionery option for health-conscious consumers. The use of agro-industrial by-products, such as walnut flour, could add value to the walnut pecan oil and confectionery industries.

## Authors' contributions

Conceptualization, G.M. and C.N.; Methodology, G.M. and C.N.; Formal analyses, G.M. and C.N.; Investigation, G.M., C.N. and V. L.; Resources, G.M., C.N., and V. L.; Writing—original draft preparation, G.M., C.N., and V. L.; and Writing—review and editing, Supervision, G.M., C.N. and V. L.

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## Availability of data and materials

All data will be made available on request according to the journal policy

## Conflicts of interest

The authors declare no conflict of interest

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