PHYSICAL AND CHEMICAL PROPERTIES OF FRUCTO-OLIGOSACCHARIDE ENRICHED WALNUT CREAMS

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Abstract

Consumption of dietary fiber is essential to maintain a healthy digestive system. Walnuts are popular both raw and processed form for their positive physiological effects. The aim of our research was to compare the physical and chemical properties of walnut creams made from high nutrient walnut pellets, a by-product of walnut oil production, enriched with four different prebiotic fibers. In the experiment, the moisture content, CIE Lab color components, oil spreading and particle size of the nut creams were determined. The rheological measurements were performed by amplitude sweep to determine the LVE range limit and flow point, and rotational measurements were performed to determine the dynamic viscosity at constant shear rate. In the chemical measurements, water activity and total polyphenol content were determined.

The results showed that the fiber-enriched walnut creams have low water activity and moisture content, making them long-lasting products. Among the samples tested, the apple fiber enriched nut cream had one of the smallest particle sizes and the highest average dynamic viscosity at constant shear rate. Its oil spreading was one of the lowest and it is considered as a stable nut cream. Its color was darker, browner and richer than the other samples. The apple fiber enriched walnut cream had a close similar high total polyphenol content as the inulin enriched walnut cream.

Keywords

walnut, pellet, fiber, rheology, particle size

Introduction

Walnuts have numerous health benefits. They are a rich source of protein, dietary fiber, and micronutrients, as well as unsaturated fatty acids. Walnuts contain flavonoids, phenolic acids, and polyphenols, whose antimutagenic, anti-inflammatory, and anti-heterogenic properties benefit health. Nuts prevent coronary heart disease by reducing low-density lipoprotein in the blood and help maintain brain health (Pan et al, 2019).

Walnuts are an important crop in the food industry. The edible part of the fruit can be eaten fresh or roasted, used on its own or in other products, or pressed into walnut oil. An increase in demand for walnut oil leads to an increase in walnut oil residue, called walnut pellet, a by-product of walnut oil production. This by-product has a high nutrient content and is often used as animal feed or compost, however, in recent years, the food industry has looked to incorporate it into other foods.

For the experiment, we created walnut creams using high-nutrient walnut pellets enriched with four types of dietary fiber to enhance their nutritional value. The fibers used were inulin, glucomannan, apple fiber, and psyllium. These fibers are all good sources to enrich different foods for product development and are beneficial for human health. Inulin is a prebiotic fiber that is beneficial for gastric health and is also used as a replacement for fat or sugar (Shoaib et al., 2016). Glucomannan plays a significant role in treating and preventing various diseases (Devaraj et al., 2019). Apple fiber is indigestible by the human body, so it feeds probiotics in the small and large intestines (Kowalczyk et al., 2021). Psyllium is a great natural source of dietary fiber widely used in the food industry because of its physicochemical and functional benefits (Zhang et al., 2025).

Dietary fibers behave differently and affect food products in various ways. It is also important to compare the physical and chemical properties of products during product development.

Shakerardekani et al. (2013a) investigated pistachio spreads and found that the amounts and types of ingredients influence the product's color parameters. They observed correlations between shear work, G' and G'' values, and consumer acceptance, including taste, color, overall texture, overall acceptability, and spreadability.

Several studies have reported on the rheological properties of nut creams and spreads, some of which involved viscoelastic testing. It was found that the acceptability and rheological behavior of nut spreads are affected by the ingredients; heat treatment methods; and particle size distribution (Shakerardekani et al., 2013b). The research of Rao (2014) shows that the flow stress of any food product during manufacturing affects its structure and rheological characteristics.

The aim of our study was to compare the physical and chemical properties of walnut creams made from high nutrient walnut pellets, a by-product of walnut oil production, enriched with four different prebiotic fibers.

Materials and methods

Materials

For the experiments, we used walnuts that were purchased from a farmer in May 2024. 2.5 kg of walnuts were pressed using a 1500 W Sunhoa automatic oil press (Shenzhen, China) to extract walnut oil, maintaining a temperature between 40 and 50°C. The remaining nut pellets were used to produce walnut cream. The different dietary fibers were purchased from organic shops.

Based on the fiber-enriched walnut cream recipe defined in the preliminary experiments, walnut creams were formulated with the following composition per 100 g of product (*Table 1*):

Table 1 Recipe for different fiber-enriched nut creams

Ingredients	Inulin enriched walnut cream	Glucomannan enriched walnut cream	Apple fiber enriched walnut cream	Psyllium enriched walnut cream
Walnut pellet	36.7 g	36.7 g	36.7 g	36.7 g
Sunflower oil	25.3 g	25.3 g	25.3 g	25.3 g
Icing sugar	23.3 g	23.3 g	23.3 g	23.3 g
Lecithin	0.4 g	0.4 g	0.4 g	0.4 g
Inulin	14.3 g	-	-	-
Glucomannan	-	14.3 g	-	-
Apple fiber	-	-	14.3 g	-
Psyllium	-	-	-	14.3 g
TOTAL	100 g	100 g	100 g	100 g

The spreads were prepared using a SilverCrest SSMS 600 E6 INOX kitchen grinder (Lidl, Neckarsulm, Germany). The chopping was carried out in 400 g portions at speed 5 for 2x1 minutes.

For simplicity, the nut spreads are named after the different types of dietary fiber used in them.

Moisture content

The moisture content of the nut creams was determined using a Sartorius MA 100 rapid moisture analyser (Sartorius Lab Instruments GmbH & Co. KG, Göttingen, Germany), measured at 105°C to constant weight in 5 replicate measurements per sample.

Color

Color measurement was performed using a ColorLite sph850 spectrometer (ColorLite GmbH, Katlenburg-Lindau, Germany), where the CIELAB L*, a* and b* color coordinates were determined with three replicates of all samples at 24.5 ± 1.0°C.

Oil spreading

The oil content of the walnut creams was determined at 24.5 ± 1.0°C using White Ribbon S&S filter paper with a diameter of 150 mm (Macherey-Nagel, Düren, Germany), with three replicates for each sample. During the measurement, 2.0 ± 0.05 g of the sample was placed on the filter paper and left to rest for one hour, covered with a beaker to prevent the surface from drying out. After one hour, the walnut cream was gently removed, after which the weight of the oil-soaked filter paper was measured without residue. The oil loss results were expressed as a percentage.

Particle size

Particle size was determined optically using an XSP-181T-LED-PLAN biological microscope (Scopium, Hungary), microscope ToupView camera and software. To measure the samples, a small amount of paraffin oil was diluted to five times its volume and, after homogenization, a drop of the suspension was added to the microscope slide in three parallel measurements and the diluted samples were examined at 100x magnification. The microscope image was used to identify the FERET_MAX (maximum diameter over all measuring direction) and FERET_MIN (minimum diameter over all measuring direction) values of each sample tested by five parallel measurements.

Amplitude sweep

Measurements were performed with a modular compact rheometer MCR302 (Anton Paar, Graz, Austria) with 5-5 parallel measurements at 25 ± 0.2°C. A layer thickness of 1.1 mm was chosen for the rheological measurements based on the particle size of the walnut creams, as the maximum particle size should not exceed 10% of the layer thickness (Mezger, 2020).

The instrument's RheoCompass 1.33 software recorded the measurement data points. During the experiment, curves were recorded using a PP50 stainless steel probe with 50 mm diameter, at shear deformation values between 0.01-100%, with 25-25 data points at a rate of 5 s/data, at a constant angular rate of 10 rad/s. Each sample was measured with five replicates at 25 ± 0.2°C. Data point recording began when the 1.1 mm sample layer remained at the same temperature as the measurement temperature for 60 s. Due to the low water content and high fat content of the samples, it was feared that the probe would slip on the sample layer, so 320 waterproof sandpaper was glued to the face of the probe to match the surface of the probe.

By scanning the amplitude of the viscoelastic material during the measurement, we can determine the behaviour of the material and decide which measurement to continue with. The storage modulus G' gives information about the elastic property measured, while the loss modulus G" gives information about the viscous property. During the evaluation of the recorded curves, where the maximum value of G' drops to 97%, the value of the linear viscoelastic shear limit deformation (γLVE) and the value of the shear stress at the yield point of the samples (τf) were determined, which is given by the intersection of the G' and G" curves, where G' = G". Beyond this point, the viscous properties clearly dominate.

Viscosity

Viscosity was measured using an Anton Paar MCR302 Modular Compact Rheometer, an oscillating rheometer with a combination of a PP50 measuring system and waterproof sandpaper. Each sample was measured with five replicates at 25 ± 0.2°C. A low maximum shear rate was chosen for the measurement because the samples were dispersions with inhomogeneous behaviour - due to the high concentration of solid particles in them, at higher shear rates the creams started to break up and part of the sample was washed out under the measuring head.

The measurements were therefore carried out in three stages, even after holding the temperature for 60 seconds. In the first stage, the samples were pre-sheared at a shear rate of 10 1/s for 60 s. In the second stage at constant shear rate of 10 1/s where the apparent viscosity curves were rded with 60 points at a rate of 1s/data. Finally, the flow curve was measured by reducing the shear rate from 10 1/s to 0.1 1/s in a linear scale, where 80 data points were recorded at a frequency of 0.125 s. The average dynamic viscosity and its percentage change at constant shear rate were determined.

Water activity

The water activity of the walnut creams was measured with a Novasina Ms1 (Novosina AG, Switzerland) water activity meter with 5-5 replicate measurements of each sample at 24.5 ± 1.0°C.

Total polyphenol content

The total polyphenol content of the walnut creams was determined spectrophotometrically at 760 nm according to the method of Singleton and Rossi (1965) using the Folin-Ciocalteu reagent.

Statistics

To evaluate the results, the Microsoft 365 Excel software was used to plot the bar graphs of the different measurements, the color chart, the particle size distribution curves and the rheological measurement curves, including the fitting of the rheological models using the Excel Solver extension. For amplitude scanning, the flow point values were determined using RheoCompass 1.33 software for the rheometer. Using the parameters extracted from the measurements and calculations, IBM SPSS 29.0 statistical software was used to search for significant differences between each nut combination using the ANOVA test followed by post-hoc testing (Tukey test or Games-Howell test).

Results and discussion

Moisture content

In all cases the moisture content of the walnut creams was between 3 and 3.5% and no significant differences were found between them. It was found that the different fibers used did not have a significant effect on the moisture content of the samples (*Figure* 1).

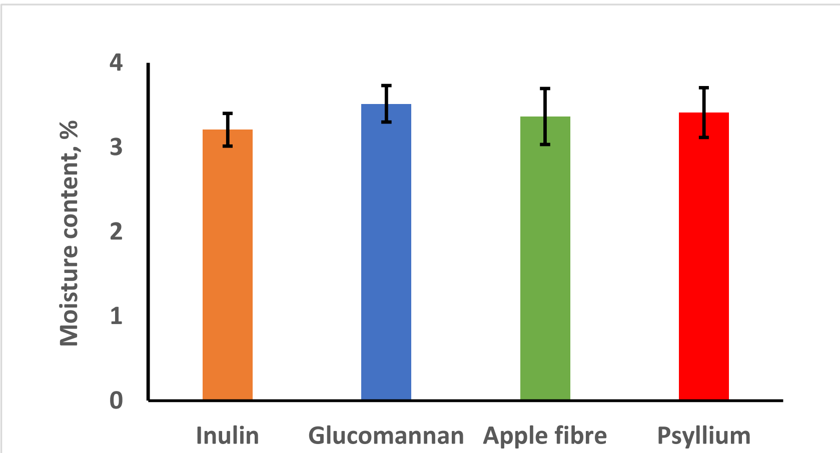


Figure 1 Average moisture content of the different walnut creams ($p < 0.05$)

Color

The CIE L*a*b* color parameter values of the different walnut creams were almost identical, except for the apple fiber-enriched cream, which had significantly lower lightness (L*) values and significantly higher red-green (a*) and blue-yellow (b*) color parameter values than the others. This was due to the darker color of the apple fiber compared to the other fibers used, which affected the color of the walnut creams (*Figure* 2).

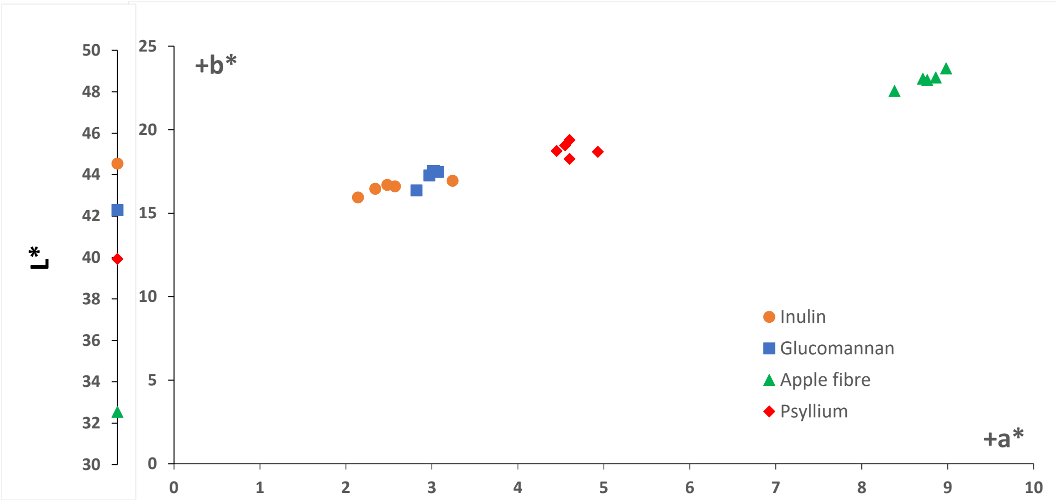


Figure 2 CIE L*a*b* results of the different fiber-enriched walnut creams ($p < 0.05$)

Oil spreading

It was found that the walnut cream enriched with inulin had the highest oil spreading and the lowest oil retention, with an oil loss of 11.33%. By contrast, the psyllium-enriched walnut cream lost only 6.40% of its oil. The apple fiber sample showed a similar value, while the walnut cream with glucomannan lost 9.21% of its weight. Using different fibers significantly influenced the oil retention capacity of the walnut creams.

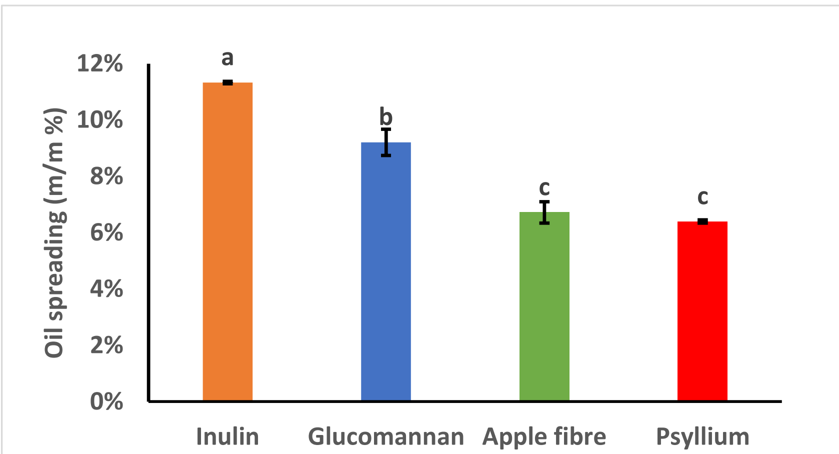


Figure 3 Oil spreading of the different fiber-enriched walnut creams ($p < 0.05$)

Particle size

Based on the results, it can be stated that the glucomannan-enriched nut cream contained the largest number of particles and the largest difference between the FERET_MIN and FERET_MAX values, which indicates inhomogeneity within the sample (*Figure 4*).

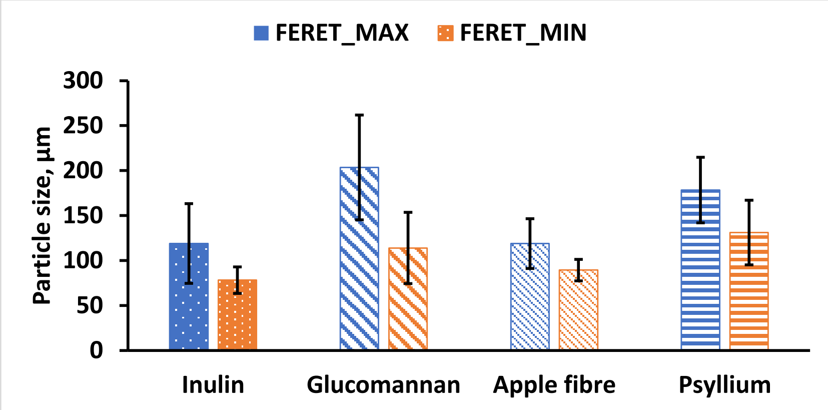


Figure 4 The particle size of different fiber-enriched walnut creams based on FERET_MIN and FERET_MAX values

Amplitude sweep

The *Figure 5* shows the variation of G' and G" of different dietary fiber enriched walnut creams with varying shear deformation. In all cases, G' was greater than G" at the initial value, so that all samples initially behaved as viscoelastic solids and then as liquids after the flow point. Significant variation was observed between the yield strength values of the samples. The results showed that the psyllium sample was the least fluid walnut cream, while the inulin sample was the most fluid.

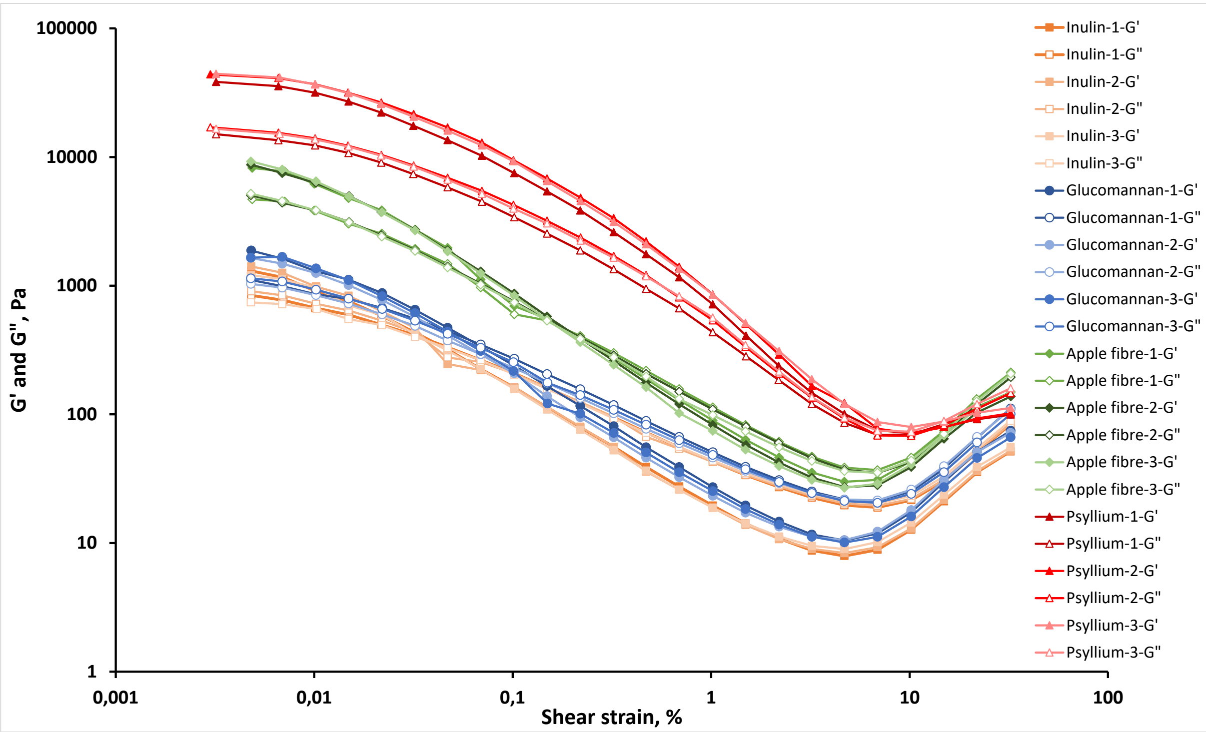


Figure 5 Storage modulus G' (Pa) and loss modulus G" (Pa) of different dietary fiber enriched walnut spreads in the function of the shear strain (%) ($p < 0.05$)

The average shear stress values of the walnut creams with standard deviation is shown in *Figure 6*. Significant variation was observed in the average shear stress values of the samples. The results showed that the psyllium-enriched walnut cream had the highest average shear stress value and the inulin-enriched cream had the lowest.

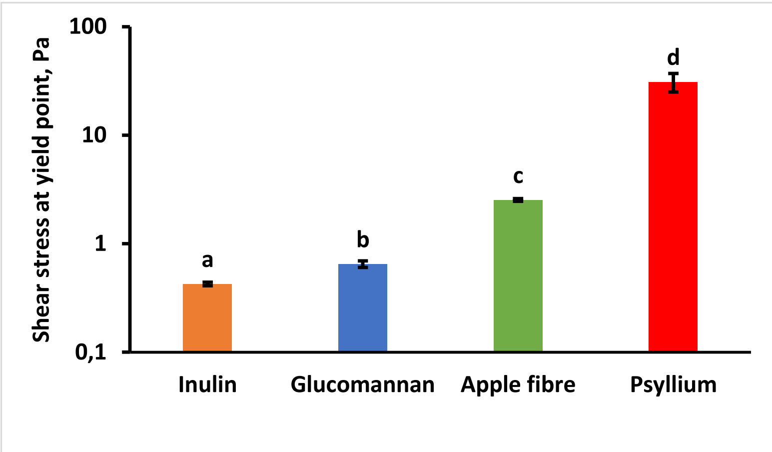


Figure 6 Average shear stress values of different fiber-enriched walnut creams with standard deviation ($p < 0.05$)

Viscosity

The correlation between the apparent viscosity and the shear rate of the walnut creams in the decreasing phase is shown in *Figure 7*. For all four samples it is observed that the apparent viscosity takes on a value of around 20 Pas at a shear rate of 10/s. The sample with the lowest initial viscosity is inulin enriched walnut cream, followed by glucomannan, psyllium and finally the apple fiber enriched walnut cream.

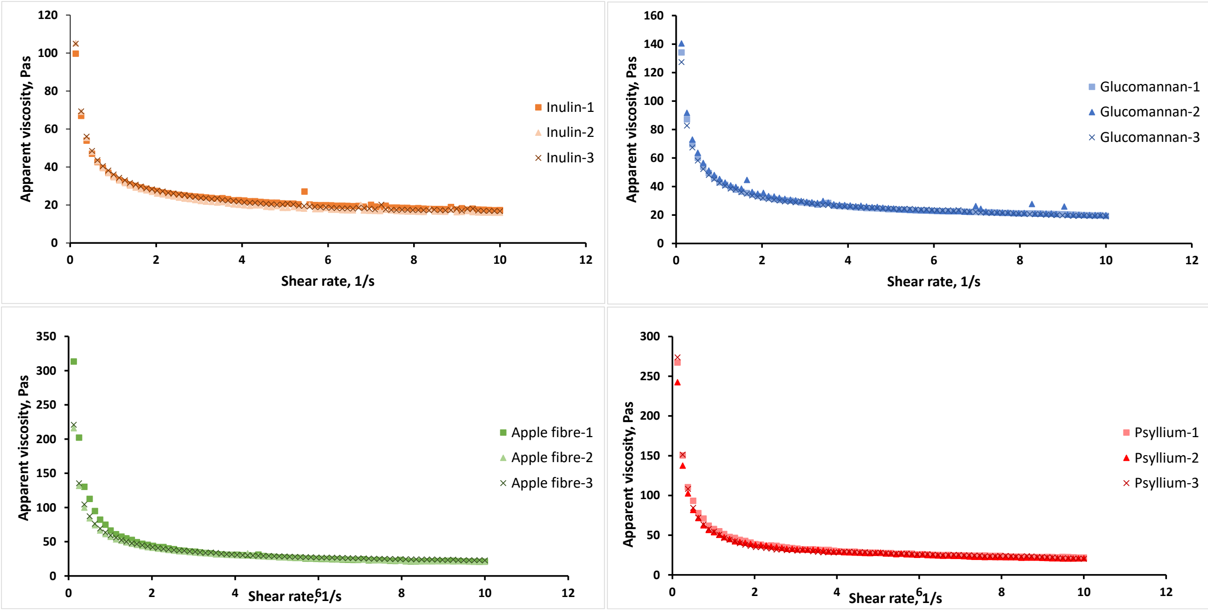


Figure 7 The apparent viscosity of different fiber-enriched walnut creams at increasing and decreasing shear rate

The dynamic viscosity averaged over the constant speed section is plotted on a bar graph at *Figure 8*. Of the samples tested, the walnut cream enriched with apple fiber had the highest average dynamic viscosity, while the one with inulin had the lowest. We found that different dietary fibers significantly influenced the average dynamic viscosity of the walnut cream.

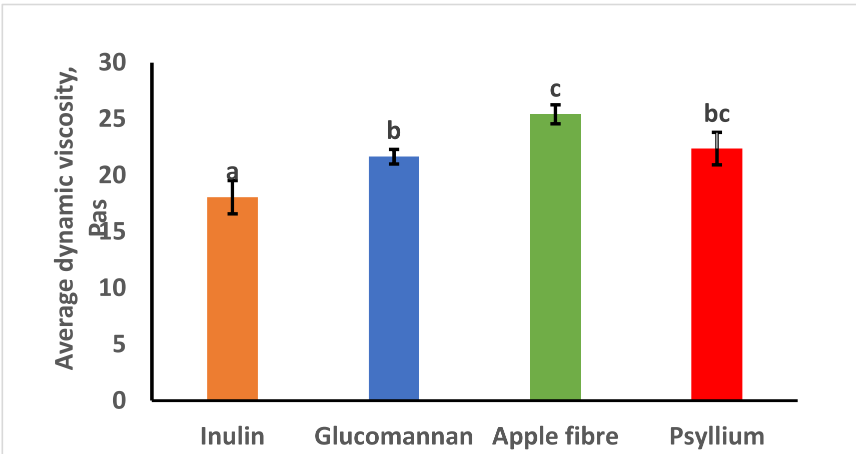


Figure 8 Average dynamic viscosity of different fiber-enriched walnut creams with standard deviation at constant maximum shear rate (10 1/s) ($p < 0.05$)

Water activity

Significant differences were found in the water activity of different dietary fiber-enriched nut creams (*Figure 9*). All walnut creams were found to have a water activity value below 0.6, classifying them as low-water-content foods. Water activity is also an indicator of shelf life, with lower values indicating a longer shelf life.

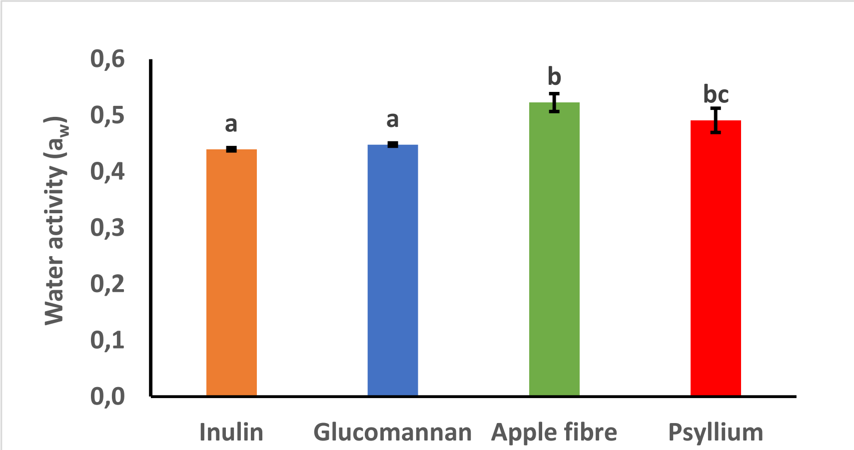
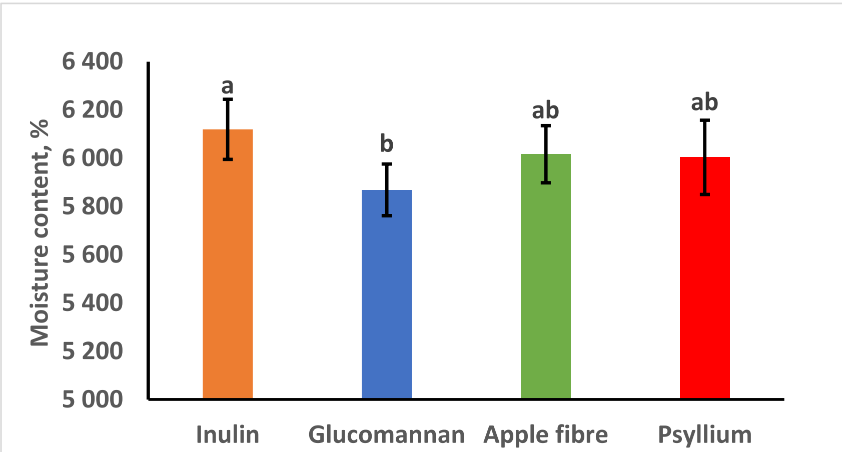


Figure 9 Average water activity of the different fiber-enriched walnut creams with standard deviation ($p < 0.05$)

Total polyphenol content

We conducted total polyphenol content (TPC) studies to demonstrate the effect of different fiber-enriched nut creams on total polyphenol content. As shown in



Figure, we found that the total polyphenol content of the different prebiotic fiber-containing nut creams did not differ significantly. The highest total polyphenol content was found in the inulin enriched walnut cream, and the lowest in the glucomannan nut cream, among the samples tested.

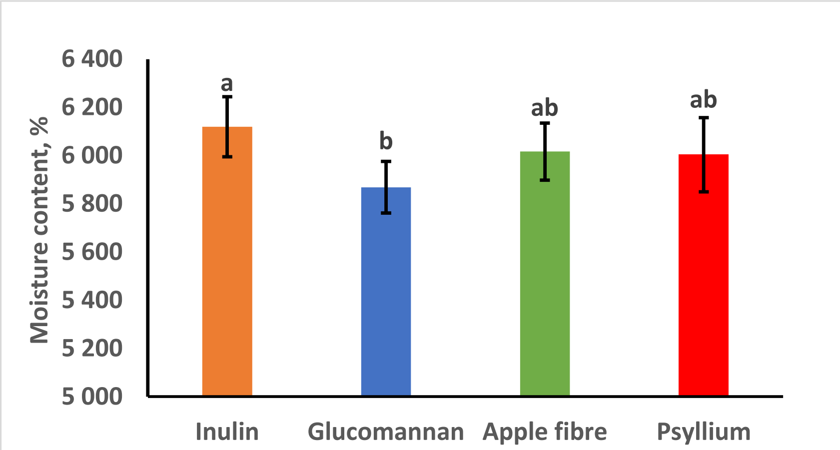


Figure ***10*** Average total polyphenol content of the different fiber-enriched walnut creams with standard deviation ($p < 0.05$)

Conclusion

Walnuts are high in nutrients and are used by the food industry in many forms. Our research aims to utilize the nut pellet, a by-product of walnut oil production, to produce walnut cream, which has high nutritional value and health benefits. We added four different dietary fibers to the walnut creams to further increase their nutritional content. We investigated the physical and chemical properties of the fiber-enriched creams, which are important for product development.

We found that all four walnut cream samples behaved as viscoelastic solids, and then liquids after the flow point. These products have a lower water activity and are therefore long-lasting. The results showed that the type of fiber significantly affects the oil retention, yield strength, and average dynamic viscosity of walnut cream. As Shakerardekani et al. (2013a) found, the type of ingredients influences the product's color parameters, this explains why the darker apple fiber caused a significant difference between the apple-fiber enriched walnut cream and the other samples. There were no significant differences in total polyphenol content, moisture content, or the FERET_MIN and FERET_MAX parameters among the particle sizes of the walnut creams.

The results showed that the apple fiber-enriched nut cream had one of the smallest particle sizes and the highest average dynamic viscosity at a constant shear rate. It was also considered a stable nut cream due to its low oil spreading.

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