



MODELING OF DYNAMIC OSCILLATION AND CREEP-RECOVERY DATA OF GLUTEN-FREE BISCUIT DOUGH CONTAINING TIGER-NUT FLOUR

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ABSTRACT

This study investigates the impact of tiger nut flour (TNF) on the rheological properties of gluten-free biscuit dough, focusing on creep-recovery behavior. TNF is a rich source of dietary fiber and essential nutrients, making it a promising ingredient in gluten-free products. The dough samples, with varying TNF percentages (10%, 20%, 30%, 40%, and 50%), were analyzed for their thermal and rheological properties. Results show that as TNF content increases, the dough becomes more deformable, reflected in higher creep compliance and lower viscosity. However, the dough retained sufficient elastic recovery, making it suitable for applications requiring structural integrity and extensibility. The reduction in gelatinization enthalpy suggests that TNF enhances processing efficiency by lowering energy requirements for starch gelatinization. This study fills a gap in the literature on TNF's role in gluten-free dough systems, offering insights for future applications in gluten-free product development.

Keywords: Gluten-free biscuit, dynamic rheology, creep-recovery, thermal properties

YER BADEMİ UNU İÇEREN GLUTENSİZ BİSKÜVİ HAMURUNUN DİNAMİK SALINIM VE SÜNME-İYİLEŞME VERİLERİNİN MODELLENMESİ

ÖZ

Bu çalışma, yer bademi ununun (YBU) glutensiz bisküvi hamurunun reolojik özelliklerine, özellikle sünme-iyileşme davranışına olan etkisini araştırmaktadır. YBU, diyet lifi ve temel besinler açısından zengin olup, glutensiz ürünlerde umut verici bir bileşendir. Farklı YBU yüzdeleri (%10, %20, %30, %40 ve %50) içeren hamur örnekleri, termal ve reolojik özellikler açısından analiz edilmiştir. Sonuçlar, YBU içeriği arttıkça hamurun daha fazla deforme olduğunu, bunun daha yüksek sünme uyumu ve daha düşük viskozite ile yansıtıldığını göstermektedir. Ancak hamur yeterli elastik iyileşme kabiliyetini koruyarak hem yapısal bütünlük hem de uzayabilirlik gerektiren uygulamalar için uygun hale gelmiştir. Jelatinizasyon entalpisi azalması, YBU'nun nişasta jelatinizasyonu için enerji gereksinimlerini azaltarak işlem verimliliğini artırdığını göstermektedir. Bu çalışma, YBU'nun glutensiz hamur sistemlerindeki rolü üzerine literatürdeki bir boşluğu doldurarak, gelecekteki glutensiz ürün geliştirme uygulamaları için önemli bilgiler sunmaktadır.

Anahtar kelimeler: Glutensiz bisküvi, dinamik reoloji, sünme-iyileşme, termal özellikler

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INTRODUCTION

Tiger nut is a herbaceous, tuberous plant grown in Africa, Southern Europe, and America, though its consumption is not widespread (Adejuyitan et al., 2018; Bamishaiye and Bamishaiye, 2011). This tuber, known by its original names chufa, tiger nut, or earth almond, can be prepared and consumed raw, roasted, dried, baked, or as a vegan beverage (horchata de chufa) (Maduka and Ire, 2018). Tiger nut tubers are rich in energy (starch, fat, sugar, and protein), minerals (calcium, potassium, phosphorus, magnesium), and vitamins (C and E). Various studies have explored the use of tiger nut flour (TNF) in gluten-free bakery products, highlighting its nutritional benefits and its potential as a valuable ingredient in biscuits, bread, and snacks (Aguilar et al., 2015; Ahmed and Hussein, 2014; Gasparre et al., 2020; Ejiofor and Deedam, 2015; Çinar et al., 2023).

In recent years, gluten-free products have gained significant attention due to the growing number of people with celiac disease and gluten intolerance. However, many gluten-free products lack the fiber and nutritional value found in wheat-based products, which has led to efforts to improve these attributes. Ingredients like tiger nut flour, which are rich in dietary fiber, vitamins, and minerals, offer a promising solution to enhance the nutritional profile of gluten-free baked goods (Pellegrini and Agostoni, 2015; Theethira and Dennis, 2015; Rybicka, 2018; Niro et al., 2019). Tiger nut flour's high fiber content contributes to better digestive health and adds valuable nutrients, making it an attractive alternative for gluten-free formulations (Rybicka and Gliszczynska-Świgło, 2017).

The rheological and thermal properties of dough are crucial for determining the final product quality of bakery goods such as bread, cakes, and biscuits. Understanding the dynamic rheology and the thermal behavior of gluten-free doughs is essential for optimizing texture, structure, and mouthfeel (Horstmann et al., 2017; Mariotti et al., 2009; Megušar et al., 2022). These properties, particularly storage and loss moduli, provide insight into the dough's elasticity and viscosity, which are critical for achieving desirable textural

properties in the final baked product (Larrosa et al., 2013; Ren et al., 2020).

This study focuses on the dynamic rheological modeling of gluten-free biscuit doughs containing tiger nut flour, specifically through creep recovery measurements. Currently, there are limited studies on the rheological behavior of TNF in gluten-free dough systems, particularly with respect to creep-recovery behavior. This research aims to fill a critical gap in the literature by providing insights into how TNF impacts the design and quality characteristics of gluten-free baked products (Aguilar et al., 2015; Ahmed and Hussein, 2014; Gasparre et al., 2020; Ejiofor and Deedam, 2015; Çinar et al., 2023).

MATERIALS AND METHODS

Materials

The rice flour used in the formulation of biscuit samples was obtained from Selva Gıda San. A.Ş. (Konya, Turkey). Tiger nut tubers (*Cyperus esculentus*, cv. Balyumru and Sarışeker) were kindly supplied by Eastern Mediterranean Agricultural Research Institute (Adana, Turkey) and they were ground into flour using a laboratory mill (Foss Tecator, 1095 Knifetec, Hoganas, Sweden). The flour sample was sieved using 0.5 mm sieve. Table salt, ammonium bicarbonate, sodium bicarbonate, powdered sugar (Dr. Oetker, İzmir, Turkey), skimmed milk powder (Pınar A.Ş., İzmir, Turkey), and vegetable margarine (Unipro, İstanbul, Turkey) were purchased from local markets. High-fructose corn syrup (HFCS) was supplied by Cargill (Bursa, Turkey), and xanthan gum by Sigma Aldrich (Steinheim, Germany).

Preparation of biscuit dough

The biscuit dough samples were prepared following the AACC 10-54.01 method (AACC, 1990), with some modifications (Table 1). The biscuit formulation was based on 40 g of flour, and to investigate the effect of tiger nut flour, mixtures of rice flour and tiger nut flour in varying proportions (10, 20, 30, 40, and 50%) were used. Additionally, a control dough containing 100% rice flour was prepared (Table 1).

Table 1. Biscuit dough formulation

Ingredients	Amount
Rice flour (g)	40.0
Shortening (g)	8.0
Powdered sugar (g)	16.8
Corn syrup (g)	0.6
Skim milk powder (g)	0.4
Water (ml)	8.8
NH ₄ HCO ₃ (g)	0.2
NaHCO ₃ (g)	0.4
NaCl (g)	0.4
Xanthan gum (g)	0.5

In dough preparation, powdered sugar, sodium bicarbonate, salt, skimmed milk powder, and vegetable margarine were mixed using a household mixer (Kitchen Aid, 5K45SS, Elk Grove Village, USA) at 135 rpm for 3 minutes, with 1-minute intervals until a creamy texture was obtained. Subsequently, a pre-prepared mixture of ammonium bicarbonate, water, and HFCS was added to the dough and mixed for 1 minute, with 14-second intervals, at 135 rpm. In the final step, the pre-prepared flour mixtures and xanthan gum were added and mixed at 95 rpm for 30 seconds, with 10-second intervals, until a homogeneous dough structure was achieved.

Differential scanning calorimeter (DSC)

A differential scanning calorimeter (DSC Q20, TA Instruments, USA) was used to analyze the thermal properties of tiger nut flour, rice flour, and biscuit dough samples. First, the dough samples were frozen at -80°C and then freeze-dried in a lyophilizator (Christ2B, Osterode am Harz, Germany). 4 mg flour or dried biscuit sample was placed in hermetically sealed aluminum pans, to which three times the amount of pure water was added. During the measurement of starch gelatinization enthalpy values, the temperature was increased from 25°C to 130°C at a rate of 10°C/min. The onset (T_o), peak (T_p), and conclusion (T_c) temperatures were obtained from the resulting thermogram. The results were reported as the average of two replicates.

Dynamic oscillatory measurement and modelling

A parallel plate rheometer (AR 2000ex, Sussex, UK) was used for rheological measurements. Approximately 2 g of biscuit dough sample was placed between plates with a 3 mm gap and a 20 mm diameter. The excess dough that overflowed from the plates was scraped off with a spatula, and the sample was allowed to rest under a protective cover for 10 minutes. All measurements were conducted at 25°C. Before dynamic oscillatory measurements, the linear viscoelastic region was determined for the control biscuit dough and the dough samples containing various proportions of tiger nut flour by measuring at a frequency of 1 Hz and a strain range of 0.1% to 100%. Based on these data, a strain value of 0.5% was found to be within the linear region for each dough formulation, and the experiments were conducted at this constant value.

Dynamic oscillatory measurements were carried out in the frequency range of 10 Hz to 0.1 Hz at a constant strain of 0.5%. As a result, storage modulus (G') and loss modulus (G'') values were obtained. The experiments were performed in duplicate, and the results were averaged. The frequency dependence (ω) of these values was modeled using the power law model. The power law models for G' and G'' are presented in the following equations.

$$G' = K' \cdot \omega^{n'} \quad (1)$$

$$G'' = K'' \cdot \omega^{n''} \quad (2)$$

The K' and K'' values in Equations 1 and 2 represent the storage and loss modulus values at a frequency of 1 Hz, respectively. The n' and n'' values indicate the indices of the power law models and correspond to the slope values of the graphs plotted on a log-log scale (Taracon et al., 2015).

Creep and Recovery measurements and modelling

A parallel plate rheometer, which was used in oscillatory measurements, was employed for creep and recovery measurements. Approximately 2 g of biscuit dough sample was placed between plates with a 3 mm gap and a 20

mm diameter. The excess dough that overflowed between the plates was scraped off, and the sample was allowed to rest under a protective cover for 10 minutes. All measurements were conducted at 25°C. In preliminary trials, the measurement parameters were determined as a total measurement time of 1200 seconds (600 s for creep and 600 s for recovery) and a stress application of 30 Pa, which indicated steady viscous flow behavior in the biscuit dough. The measurements were performed in duplicate, and the results were reported as creep compliance (Pa⁻¹) corresponding to unit stress. Deformation is represented by the following equation (Equation 3).

$$J(t) = \frac{\gamma(t)}{\sigma_0} \quad (3)$$

The Burger model was used to model the creep portion of the data obtained from creep and recovery measurements (Equation 4).

$$J(t) = J_0 + J_1 \cdot (1 - \exp(-t/\lambda)) + t / \eta_0 \quad (4)$$

In this equation, J_0 represents instantaneous deformation, J_1 represents retarded deformation, t is time, λ is the mean retardation time, and η_0 represents Newtonian viscosity. The Levenberg-Marquardt algorithm, a mathematical model (KaleidaGraph 4.0, Synergy Software), was used for solving this non-linear equation (Laguna et al., 2013). In this iterative and non-linear method, an initial value was provided for each parameter. As

the iterations continued, the model progressively improved until a predetermined magnitude order was achieved.

Statistical analysis

A one-way ANOVA was performed using the MINITAB statistical analysis program (Minitab 18, State College, Pennsylvania, USA) to determine whether the data obtained from the analyses differed significantly from each other ($P < 0.05$). Tukey's multiple comparison test was also applied when significant differences were found.

RESULTS AND DISCUSSION

Thermal properties of flour and biscuit dough samples

In the differential scanning calorimetry (DSC) analyses, the onset temperature (T_o), peak temperature (T_p), and conclusion temperature (T_c) of starch gelatinization, expressed in degrees Celsius (°C), and the starch gelatinization enthalpy (ΔH) (J/g sample) based on dry sample weight were recorded. The T_o and T_c values indicate the temperatures at which gelatinization starts and finishes, respectively, while the T_p represents the temperature at which the starch gelatinization transformation occurs. The enthalpy value (ΔH) indicates the energy required for the completion of gelatinization. The results of the analyses performed on TNF and RF are presented in Figure 1.

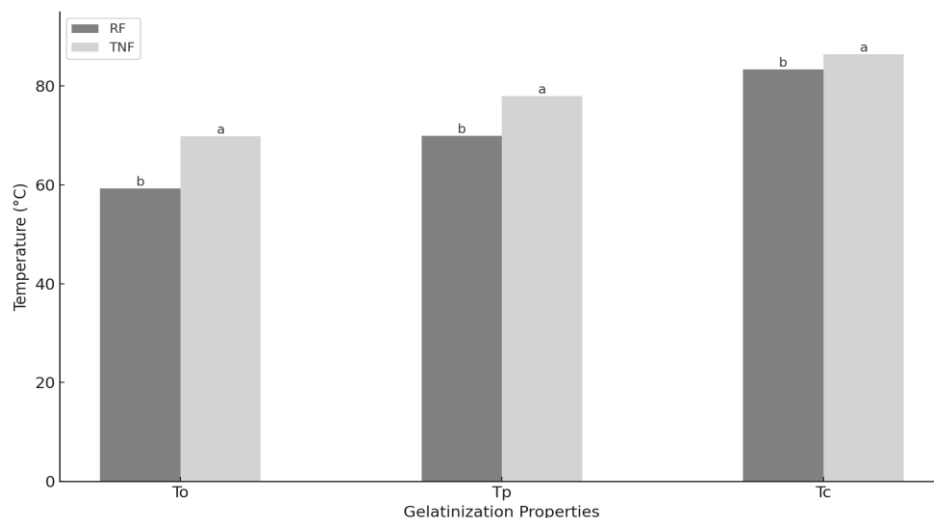


Figure 1. Onset (T_o), peak (T_p), and conclusion (T_c) temperatures of TNF and RF samples

For TNF, the T_o , T_p , and T_c temperatures were higher than those of rice flour, while the enthalpy required for gelatinization completion was significantly lower compared to rice flour ($P < 0.05$). Specifically, the gelatinization enthalpy for TNF was 2.989 J/g, whereas for RF it was 7.986 J/g (Figure 1). In the study conducted by Demirkesen et al. on TNF and RF-containing bread dough samples, the T_o , T_p , and T_c values were found to be higher for TNF, while the enthalpy value was lower (Demirkesen et al., 2013). The reason for this result is that the starch content in TNF is lower than in rice flour, which allows the complete gelatinization of all the starch in the structure to be achieved with less energy. In

the previous study by Çınar et al., the starch contents of TNF and RF were reported as 24.89% and 81.99%, respectively (Çınar et al., 2023).

The gelatinization values of gluten-free biscuit doughs containing different proportions of TNF are presented in Table 2. It was observed that there was no significant difference in the onset (T_o), peak (T_p), and conclusion (T_c) temperatures with increasing amounts of TNF in the dough formulations. However, the starch gelatinization enthalpy (J/g sample) showed a significant difference between the control group (0% TNF) and the formulations containing 30%, 40%, and 50% TNF ($P < 0.05$).

Table 2. Gelatinization values of biscuit dough samples containing different proportions of tiger nut flour

% TNF	T_o (°C)	T_p (°C)	T_c (°C)	Enthalpy (J/g _{dry solid})
0	68.49 ± 0.41 ^{a*}	74.90 ± 0.45 ^a	84.55 ± 1.39 ^a	4.95 ± 0.00 ^a
10	68.14 ± 0.04 ^a	74.46 ± 0.27 ^a	86.50 ± 1.17 ^a	4.52 ± 0.42 ^{ab}
20	67.87 ± 0.60 ^a	74.74 ± 0.35 ^a	87.21 ± 1.19 ^a	4.31 ± 0.33 ^{ab}
30	69.10 ± 0.20 ^a	75.03 ± 0.26 ^a	87.49 ± 0.79 ^a	3.74 ± 0.15 ^{bc}
40	69.26 ± 0.47 ^a	75.70 ± 0.20 ^a	88.56 ± 1.62 ^a	3.11 ± 0.18 ^{cd}
50	69.38 ± 0.88 ^a	75.37 ± 0.63 ^a	87.72 ± 2.90 ^a	2.41 ± 0.21 ^d

*The results are presented as mean values and standard deviation from two replicates. Values in each column having different letters indicate statistically significant differences ($P < 0.05$).

As the proportion of TNF in the dough increased, the enthalpy value per gram of dry sample decreased. This reduction in enthalpy is attributed to the decrease in starch content in the structure with increasing TNF proportion, which results in less energy required for the complete gelatinization of all the starch in the structure (Çınar et al., 2023). A similar finding was reported in a study by Laguna et al. (2011), where the performance of a resistant starch-rich ingredient in short-dough biscuits showed that increasing resistant starch content decreased gelatinization enthalpy in dough formulations.

Modelling of dynamic oscillatory measurements of biscuit dough

Rheology of dough is crucial for determining the final product quality, particularly in bakery products such as bread, cakes, and biscuits, making its characterization essential. In biscuit

doughs containing different proportions of TNF, changes in the rheological properties of the dough samples were observed based on varying TNF percentages. In our previous study (Çınar et al., 2023), graphical representations of the G' (storage modulus) and G'' (loss modulus) values obtained from dynamic oscillatory measurements were presented. According to these results, the G' values were higher than the G'' values, indicating that the dough samples exhibited elastic behavior. Similar elastic behavior has also been observed in the literature for dough samples of biscuits and cakes (Gao et al., 2017; Moiraghi et al., 2010; Zannini et al., 2012). On the other hand, besides obtaining the G' and G'' values, representing these values with different models is also important for evaluating the data.

When observing the rheological behavior of biscuit dough samples containing various

proportions of TNF, the Power law model parameters describing the dependence of storage modulus (G') and loss modulus (G'') on angular velocity provide critical insight. According to Table 3, the control sample exhibited the highest K' value, indicating the greatest capacity for elastic energy storage. As the TNF content increased, the K' value progressively decreased, with the 50% TNF dough sample showing the lowest K' value. Statistical analysis using Tukey's test indicated that the K' value for the 50% TNF sample was significantly lower than the control ($P < 0.05$), highlighting a significant reduction in the dough's ability to store energy as TNF content increases. This trend is consistent with findings by Xu et al.

(2017), where the incorporation of potato granules into wheat flour dough also led to reduced storage moduli, as the gluten network weakens with the inclusion of alternative ingredients such as dietary fiber. Tiger nut flour contains higher dietary fiber content than rice flour and it is widely known that incorporating fiber-rich ingredients into gluten-free dough network generally weakens the structure, which can result in a decrease in G' . Another study on gluten-free dough enriched with *Plantago* seeds also demonstrated that fiber-rich additives weaken the dough structure and reduce its resistance to deformation, further supporting the decrease in storage modulus (Pejcz and Buresova, 2022).

Table 3. Parameters of Power law functions describing dependence of storage (G') and loss moduli (G'') on angular velocity

Sample	$G' = K'w^{n'}$			$G'' = K''w^{n''}$		
	K' (Pa.s ^{n'})	n'	R^2	K'' (Pa.s ^{n''})	n''	R^2
control	123205.92 ^a	0.094 ^a	0.971	62054.00 ^a	0.214 ^a	0.979
10%	131425.65 ^a	0.013 ^b	0.829	63766.43 ^a	0.130 ^b	0.951
20%	119695.89 ^a	0.031 ^{ab}	0.937	60826.22 ^a	0.103 ^b	0.936
30%	111378.56 ^{ab}	0.013 ^b	0.958	51535.80 ^{ab}	0.144 ^{ab}	0.956
40%	100312.61 ^{ab}	0.064 ^{ab}	0.970	52164.19 ^{ab}	0.170 ^{ab}	0.971
50%	79395.74 ^b	0.065 ^{ab}	0.963	42150.51 ^b	0.152 ^{ab}	0.960

*The results are presented as mean values from three replicates. Values in each column having different letters indicate statistically significant differences ($P < 0.05$).

In terms of the loss modulus (G''), the control dough sample showed the highest K'' value, reflecting the greatest capacity for viscous energy dissipation. As the TNF content increased, the K'' values progressively decreased, indicating that the dough's ability to dissipate energy was reduced with higher TNF proportions. This trend is consistent with the findings of Moreira et al. (2013), who applied the Power Law model to gluten-free doughs made from chestnut and rice flour blends. Their study demonstrated that as the proportion of chestnut flour increased, G'' values decreased, showing the reduction in viscous properties seen in our TNF dough samples. Similarly, Tsatsaragkou et al. (2014) found that in gluten-free bread doughs containing carob flour, higher fiber content resulted in increased G''

values, highlighting the influence of fiber on the viscous behavior of doughs. However, in this study, the replacement of rice flour with TNF did not increase fiber content enough to strengthen the viscous properties, hence the lower G'' values with increasing TNF content. Furthermore, the study by Rezaei et al. (2017) on soy yogurt mixes reinforced that adding resistant starch and β -glucan could enhance G'' values, signifying a stronger viscous component in fiber-rich systems. In contrast, the results in this work suggest that TNF's lower starch and gluten content contributed to reduced viscous behavior, as indicated by the significant differences in K'' values compared to the control dough.

Creep and recovery measurements of biscuit dough

The creep-recovery behavior of biscuit doughs was analyzed to assess how increasing TNF content affects their viscoelastic properties. Figure 2 presents the shear compliance ($J(t)$) over time for various TNF-containing doughs, while Table 4 summarizes the parameters derived from the Burger model, including instantaneous compliance (J_0), retarded compliance (J_1), retardation time (λ_{ret}), and zero-shear viscosity (η_0) (Laguna et al., 2013).

From the creep curves in Figure 2, it is clearly seen that as the TNF content increases, the creep

compliance values also rise, indicating that doughs with higher TNF levels undergo greater deformation under applied stress. The control sample exhibited the lowest creep compliance, showing its resistance to deformation, whereas the 50% TNF dough showed the highest compliance. This suggests that increasing TNF content weakens the dough structure, making it more susceptible to flow under stress. These findings are consistent with those from Moreira et al. (2013), who observed similar trends in gluten-free doughs containing chestnut flour, where increasing fiber content led to greater deformation during creep.

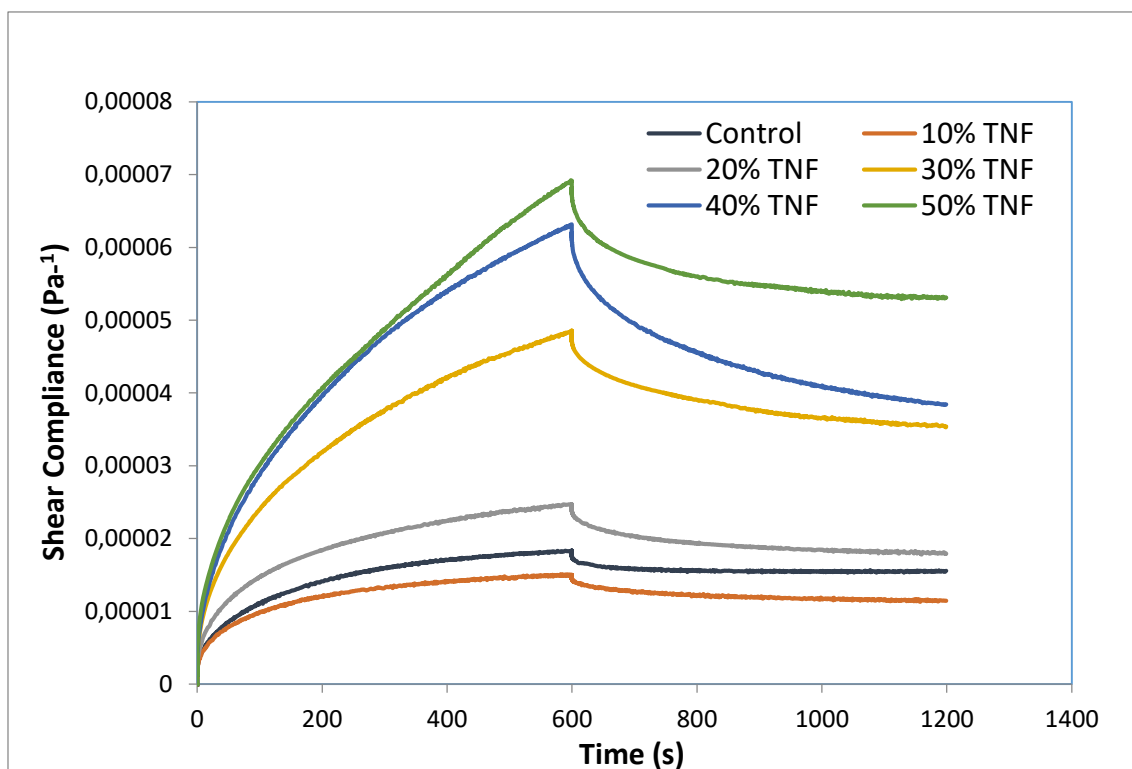


Figure 2. Shear compliance values (Pa^{-1}) of biscuit dough samples containing different proportions of tiger nut flour

In terms of elastic and viscous properties, Table 4 reveals a significant increase in instantaneous compliance (J_0) and retarded compliance (J_1) as the TNF content increases. For the control dough, J_0 was the lowest ($2.05 \times 10^6 \text{ Pa}^{-1}$), while the 50% TNF dough exhibited the highest value ($4.27 \times 10^6 \text{ Pa}^{-1}$), indicating that TNF-enriched

doughs are more deformable and less resistant to both instantaneous and delayed elastic deformation. The retardation time (λ_{ret}) decreased with higher TNF levels, implying a slower recovery from deformation, a trend also noted by Chompoorat et al. (2018) in gluten-based doughs. The zero-shear viscosity (η_0)

decreased significantly, from 10.41×10^7 Pa·s in the control dough to 1.42×10^7 Pa·s in the 50%

TNF dough, indicating that TNF-enriched doughs are less viscous and more prone to flow.

Table 4. Parameters of Burger model for biscuit dough samples

Sample	$J_0 \times 10^6$	$J_1 \times 10^5$	λ_{ret}	$\eta_0 \times 10^{-7}$
control	2.05	1.10	68.74	10.41 ^a
10%	1.86	0.90	54.28	13.79 ^a
20%	2.51	1.31	53.59	6.71 ^{ab}
30%	3.78	2.17	68.03	2.48 ^b
40%	4.03	2.74	78.97	1.83 ^b
50%	4.27	2.19	45.04	1.42 ^b

*The results are presented as mean values from three replicates. Values in each column having different letters indicate statistically significant differences ($P < 0.05$).

Statistical analysis confirmed significant differences between the control and TNF doughs in terms of J_0 and η_0 ($P < 0.05$), with the most pronounced differences observed between the control, 10%, and 50% TNF formulations. These results are supported by similar findings in studies by Rezaei et al. (2017) and Laguna et al. (2013), where increased fiber and starch contents in dough systems enhanced deformation. However, the doughs retain some elastic recovery capacity, making TNF-enriched dough formulations potentially beneficial for applications requiring extensible yet structurally stable doughs. Overall, the creep-recovery results suggest that increasing TNF content decreases dough resistance to deformation, as reflected by higher creep compliance and lower viscosity values. However, the doughs retain some elastic recovery capacity, making TNF-enriched dough formulations potentially beneficial for applications requiring extensible yet structurally stable doughs.

CONCLUSIONS

Tiger nut flour (TNF) has shown significant potential as a valuable alternative for gluten-free formulations, primarily due to its high nutritional value and positive influence on dough properties. As a rich source of dietary fiber, healthy fats, and essential nutrients, TNF offers a promising alternative to traditional flours, particularly for individuals with gluten sensitivities or those seeking healthier food options. In this study, the inclusion of TNF in biscuit doughs resulted in

doughs that were more deformable, as evidenced by higher creep compliance and lower viscosity values, yet the doughs retained sufficient elastic recovery, making them suitable for products requiring both extensibility and structural integrity. The reduction in starch gelatinization enthalpy with increasing TNF content suggests that TNF requires less energy for starch gelatinization, enhancing processing efficiency. Additionally, the rheological behavior observed in dynamic oscillatory and creep-recovery tests demonstrates TNF's ability to maintain dough stability, despite the weakening effect typically associated with fiber-rich ingredients in gluten-free doughs. These findings highlight TNF's capacity to improve dough handling and textural properties, contributing to higher-quality gluten-free baked goods. The potential applications of TNF in a wide range of gluten-free products are significant, offering manufacturers an opportunity to create healthier, fiber-rich, and nutritionally superior alternatives to conventional gluten-containing products. Future research could further optimize TNF use in various gluten-free formulations, examining its effects on sensory properties and shelf life, thereby solidifying its role as a key ingredient in gluten-free food innovation.

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DECLARATION OF COMPETING INTEREST

There is no conflict of interest.

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