

Cellulose synthesis from germinated tiger nut residue and its application in the production of a functional cookie

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Revised: 26 December 2023 / Accepted: 11 March 2024
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Abstract In this study, cellulose was synthesized from the residue obtained after germinating tiger nuts for 0, 48, 72 or 96 h. The influence of the synthesized cellulose (0%, 2%, or 5%) on the quality of clove extract laden-cookies was evaluated. The optimum structure, morphology, and thermal properties of cellulose were obtained after germinating tiger nuts for 72 h. Adding cellulose to the dough stabilized the total phenolic, flavonoid, and protein contents and radical scavenging activity during the baking operation. The addition of

2% cellulose generally enhanced the hydration, pasting, and viscoelastic properties of the dough. However, 5% cellulose negatively affected the highlighted properties, culminating in poorer textural and sensory properties of the cookies produced therefrom. Germination could be effective in modifying the properties of cellulose from tiger nuts; thus, enhancing its application in the production of a functional cookie.

Supplementary Information The online version contains supplementary material available at <https://doi.org/10.1007/s13197-024-05972-8>.

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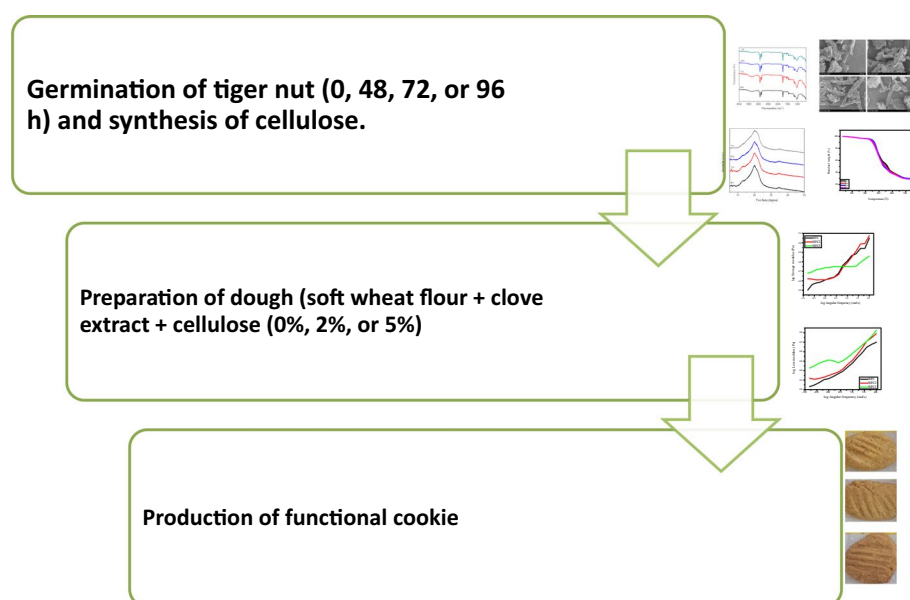
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Graphical abstract



Keywords Cellulose · Clove extract · *Cyperus esculentus* · Functional food · Germination

Abbreviations

XRD X-ray diffractometry
 TGA Thermogravimetric analysis
 WF Wheat flour
 CI Crystallinity index
 TPC Total phenolic content

Introduction

Tiger nut (*Cyperus esculentus*), a nut-like rhizome of the Cyperaceae family, is a popular raw material for making value-added products, such as imitation milk (*horchata de chufa*), oil, starch, and flour, in many parts of Europe and Africa (Ogundipe et al. 2021). During the processing of tiger nuts into these products, a high volume of solid residue, accounting for over 60% of the mass, is generated (Rosello-Soto et al. 2019). The residue is a rich source of α -cellulose and bio-active compounds (Rosello-Soto et al. 2019). Germination is an important technique employed to improve the nutritional and functional properties of tiger nut products ((Liu et al. 2022; Ogundipe et al. 2021). Previous studies (Babiker et al. 2021; Ogundipe et al. 2021)

revealed that the properties of the final products depend on germination conditions; however, little information is available on the impact of germination of tiger nut on the properties of cellulose synthesized from its residue.

Cellulose is an important ingredient in the food industry owing to its ease of biodegradability, non-toxicity, thermal stability, and excellent functional and dietary properties (Ren et al. 2020). Cellulose has attained the generally-regarded-as-safe status by the Food and Drug Administration and as a functional food ingredient by the European Union, permitting its use in the food industry (Khorairi et al. 2021). In the bakery industry, cellulose is mainly applied for improving dough rheology and a source of dietary fiber (Ren et al. 2020). Moreover, there is a continuous interest in exploiting cellulose for other novel applications in food processing.

The surge in consumers' desire for healthy diets has necessitated increased research to improve the bio-functionality of bakery products (Chinma et al. 2022). One such approach is by incorporating plant extracts, which are a rich source of phenolic compounds, with excellent antioxidant properties (Ranjbaryan et al. 2019). Accordingly, many studies have characterized the extract from clove (*Syzygium aromaticum*), a common spice belonging to the family Myrtaceae (Sharma et al. 2020), and applied it in food systems to enhance bio-functionality, storage

stability, and sensory properties (Harlina et al. 2018). However, the hydrophobic nature and poor thermal stability of plant extracts have limited their applications in food systems (Pilong et al. 2023). Therefore, the need to stabilize these materials in food systems is imperative, and using cellulose could be effective.

The objective of this current study was to evaluate the influence of germinating tiger nut (0–96 h) on the surface properties of cellulose and to elucidate the impact of different concentrations (0%, 2%, and 5%) of cellulose on the quality of a clove extract-enriched cookie.

Materials and methods

Materials

Yellow-variety of tiger nuts were obtained from the Teaching and Research Farm, Federal University Wukari, Wukari, Nigeria. The nuts, on a dry basis, contained 6.21 ± 0.47 g/100 g, 52.74 ± 3.22 g/100 g, 25.11 ± 1.11 g/100 g, and 14.03 ± 1.66 g/100 g moisture, α -cellulose, lignin, and hemicellulose, respectively. Soft wheat flour (WF) and dried clove buds were obtained from CJ Cheiljedang, Korea and TRS Co. Ltd., Watford, UK, respectively. Analytical-grade reagents were used for the experiments. Soft wheat flour (WF) and dried clove buds were obtained from CJ Cheiljedang, Korea and TRS Co. Ltd., Watford, UK, respectively.

Germination of tiger nut and synthesis of cellulose

Tiger nuts (500 g) were soaked in 2 L 0.1% ascorbic acid solution and incubated at 27 °C for 30 h, and sprouted in humidity chamber (TH3-ME-065, Jeio Tech., Daejeon, Korea) at 85% relative humidity and 32 °C for different germination times, 0, 48, 72, or 96 h. The germinated nuts were wet-milled and the resulting mass was sieved with a 0.074-mm American standard sieve to obtain the residue. The residue was oven-dried (40 °C, 48 h) and dry-milled.

Cellulose was extracted from the germinated tiger nut residue (Melikoglu et al. 2019). Briefly, 50 g of the residue was treated with hot distilled water (95 °C) for 40 min and filtered. The residue was treated twice with 400 mL of 1 mol/dm³ HCl (Junsei Chemicals, Tokyo, Japan) at 80 °C in a water bath (BF-50SIR, Biofree Ltd., Gyeonggi-do, Korea) for 20 min and filtered. This was followed by the treatment with 5% 300 mL of NaOH (Duksan, Gyeonggi-do, Korea) at 60 °C for 1½ h. The residue was treated twice with 1% NaOCl (Duksan, Korea) at 80 °C for 2 h. The resulting cellulose was washed copiously with distilled water until the pH of the filtrate was neutral. The cellulose was oven dried at 40 °C until the moisture content was constant.

Preparation of ethanolic extract powder from clove buds

Ethanolic extract powder was prepared from the clove buds (El-Maati et al. 2016). Dry clove buds were ground to a fine powder (average particle size 0.150 mm) using a mortar and pestle. The ground clove bud (100 g) was poured into a conical flask and 400 mL of 80% ethanol was added. The mixture was incubated at room temperature (25 °C) for 1 h and filtered using Whatman number 1 filter paper. The filtrate was frozen at −45 °C for 24 h and freeze-dried at −80 °C for 72 h. The resulting pellet was ground into powder and kept at −20 °C. The TPC, flavonoid, and 1, 1-diphenyl-2-picrylhydrazyl (DPPH) radical scavenging activity of the powder were 137.22 ± 2.64 mg/g, 14.68 ± 0.84 mg/g, and $89.72 \pm 2.13\%$, respectively.

Formulation of wheat flour-cellulose blends and production of a functional cookie

Wheat flour (99 g) and 1 g of clove extract powder were blended in a bag mixer (Interscience, Nom, France) for 4 min. Subsequently, the WF-clove extract powder blend was substituted with 0 g, 2 g, or 5 g cellulose powder, and the mixture was further mixed for 5 min. Cookies were produced from the flour blends based on the AACC (2000) method, number 10-50D. Forty-five grams of sugar (Bua PLC., Lagos, Nigeria) and 65 g margarine (Blue band, Lagos, Nigeria) were mixed in a mixer (XL KVL4100S, Kenwood, Sheffield, UK) at 500×g for 4 min. Other ingredients, 100 g flour blend, 65 g skimmed milk powder (Wamco Nigeria PLC, Lagos, Nigeria), 1 g table salt (Dangote Salt Ltd., Lagos, Nigeria), and 25 mL egg white, were added to the sugar-margarine mixture and further kneading was done until a non-sticky dough was obtained. The dough was spread on a wooden board and divided with a 60-mm circular cookie cutter (Hulisen, Guangdong, China). The dough was rested at ambient temperature (25 °C) for 20 min and refrigerated at 4 °C for 12 h and was baked at 160 °C for 15 min.

Characterization of cellulose

For structural evaluation, 0.1 g of the cellulose was palletized with KBr and analyzed using FTIR (Nicolet iS5, Thermo Scientific, USA). The analysis was conducted at a 0.2 cm/s scan speed, 4 cm^{−1} resolution, and 4000–550 cm^{−1} wavelength (Melikoglu et al. 2019). Diffraction spectra of cellulose samples were obtained with the aid of an XRD device (EMPYREAN, Malvern Panalytical, Malvern, UK) with Cu as the X-ray source. Diffraction intensities were obtained at $2\theta = 5^\circ$ – 50° at 50 mA and 40 kV (Melikoglu

et al. 2019). Crystallinity index (CI) was calculated using Eq. 1.

$$CI (\%) = \frac{\text{Area of crystalline region}}{\text{Area of crystalline} + \text{amorphous regions}} \times 100 \quad (1)$$

To evaluate their morphology, the cellulose samples were gold-plated, and images were captured with a scanning electron microscope (Hitachi SU8030, Hitachi, Japan) at 5.0 kV and $\times 500$ magnification (Johar et al. 2012).

The thermal stability of the cellulose samples between a temperature range of 10–600 °C was evaluated using thermogravimetric equipment (Q500, TA Instruments, New Castle, DE 19720, USA) (Sanchez-Gutierrez et al. 2020).

Characterization of flour blends and cookies

Determination of total phenolic content, flavonoids, and antioxidant activity of flour blends and cookies

The TPC, flavonoids, and antioxidant activity, based on the DPPH radical scavenging assay, of flour blends and cookies were determined according to the procedure described by Babiker et al. (2021). For the analyses, sample extracts were prepared using 80% methanol. For the determination of TPC, 1 mL of sample extract was mixed with 1 mL of Folin-Ciocalteu reagent (Sigma-Aldrich, St. Louis, MO, USA), and 10 mL of 7.5% sodium carbonate (Junsei Chemicals, Japan) in a 25-mL test tube. The mixture was vortexed for 5 min and made to 25 mL with distilled water. Absorbance was read at 750 nm and TPC was estimated from a gallic acid standard curve, and data were expressed as mg gallic acid equivalent (GAE)/g. Flavonoid was determined based on an aluminum chloride colorimetric assay using quercetin (Sigma-Aldrich, USA) as the standard. Data were expressed as mg quercetin equivalent (QE)/g. For the determination of antioxidant activity, 1 mL sample extract and 10 mL DPPH solution (Sigma-Aldrich, USA) were mixed and kept in a dark cupboard at 25 °C for 30 min, and the absorbance reading was taken at 517 nm.

Determination of viscoelasticity of the dough

The viscoelasticity of the dough prepared from the flour blends was evaluated with the aid of a parallel-plate viscometer (AR2000, TA Instruments, DE, USA), with a geometry of 1-mm gap size and 20-mm diameter (Millar et al. 2019). To determine the linear viscoelastic range of the dough, an oscillation amplitude test was performed at different strain values (0.1–100.0%) and constant angular frequency (ω , 6.28 rad/s) and temperature (25 °C). After sample loading, a 5-min resting period was allowed, and sample dehydration was prevented by adding silicon oil at the edges of the geometry. At a 0.05%

constant strain, the frequency sweep test was conducted based on the previously established linear viscoelastic range. The data obtained, storage (G') and loss (G'') moduli, were fitted into viscoelasticity models (Eqs. 2–4).

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

$$G'' = K''(\omega)^{n''} \quad (3)$$

$$G^* = A_F(\omega)^{1/z} \quad (4)$$

where K' and G'' are viscoelasticity constants, n' and n'' are frequency indices. G^* , A_F , and z are complex modulus, gel strength, and interaction constant.

Determination of proximate and dietary fiber composition of cookies

The proximate composition of cookies was determined based on the AOAC (2005) methods. The parameters, moisture, protein, ash, and fat were determined using method numbers 930.15, 978.04, 930.05, and 930.09, respectively. Carbohydrate was obtained by difference and total dietary fiber was determined using the AOAC (2005) method, number 962.09.

Determination of physical properties of cookies

Dough yield (%) was obtained as the ratio of the weight of the cookie to that of the dough, and the spread ratio was estimated by dividing the diameter of the cookie by its thickness (Chinma et al. 2022). The textural profile of the cookies was analyzed based on the method described by Tarancon et al. (2015), with the aid of a universal testing machine (Z1010, Zwick-Roell, Ulm, Germany). The experiment was conducted at a pre-loading speed of 30 mm/min under a 10-g load using dual compression with a 5-s waiting time between cycles. The color attributes of cookies were evaluated using a color meter (CR-series, Konica-Minolta, Tokyo, Japan) according to the manufacturer's instructions.

Evaluation of sensory attributes of cookies

A 9-point hedonic scale was used for the assessment of sensory parameters of cookies, taste, texture, color, aroma, and overall acceptability, based on a protocol reported by Chinma et al. (2022). Fifty panelists, who were familiar with cookies, were selected among the undergraduate and postgraduate students of the Faculty of Agriculture and Life Sciences, Federal University Wukari, Wukari, Nigeria. The selected panelists gave their consent to participate in the

experiment. The samples were served to the panelists in separate booths in a sensory evaluation laboratory, illuminated with fluorescent light. Potable water was provided to the panelists to rinse their mouth before evaluating another sample. The Ethics Committee of the Federal University Wukari, Wukari, Nigeria approved the study.

Statistical analysis

All experiments and analyses were conducted three times. The first phase of the study (cellulose synthesis) has one independent variable, germination time (0 h, 48 h, 72 h, and 96 h). The second phase (formulation of blends for cookie production) also has one variable, the inclusion level of cellulose (0%, 2%, and 5%). The data generated were subjected to one-way analysis of variance and Duncan multiple range test using the statistical package for the social scientists (version 23.0, IBM Statistics, Armonk, NY, USA).

Results and discussion

Yield, structure, and crystallinity of cellulose

The yield of cellulose increased by 23.34% and 40.97% following the germination of tiger nuts for 48 h and 72 h, respectively (Fig. S1), likely to the increased activities of enzymes, such as amylase, xylanase, and proteases, during germination that reduced the concentration of non-cellulosic compounds in the tiger nut residue (Liu et al. 2022), corroborating a report that germination of tiger nut for 72 h increased the crude fiber content of tiger nut flour (Chinma et al. 2009). There was a reduction in the yield of cellulose by 7.34% at the latter phase of germination (96 h), probably because of the production of glucanases and glucosidases that partially degraded cellulose into sugars (Khorairi et al. 2021).

The FTIR spectra (Fig. 1a) show typical cellulose bands at 3350 cm^{-1} , 2920 cm^{-1} , 1760 cm^{-1} , and 1010 cm^{-1} , indicating OH-stretching of intermolecular hydrogen bonding, C–H stretching, C=O stretching, and C–O–H stretching, respectively (Melikoglu et al. 2019). There was no visible variation in the absorption peaks of the samples, however, the intensities at 3350 cm^{-1} and 1010 cm^{-1} were slightly increased during 72-h germination and reduced after 96-h germination, likely because of changes in the organization and orientation of fibrils in the cellulose molecules (Johar et al. 2012), indicating that short-term germination (72 h) improved intermolecular hydrogen bonding that would enhance stability and compatibility with other macromolecules (Adedeji et al. 2022).

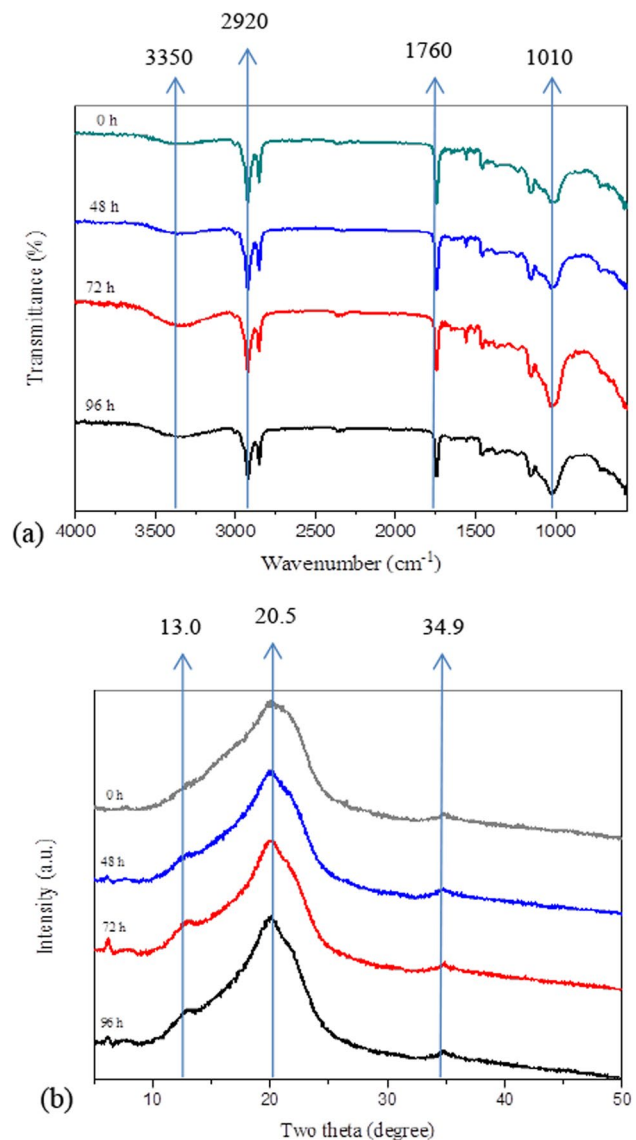
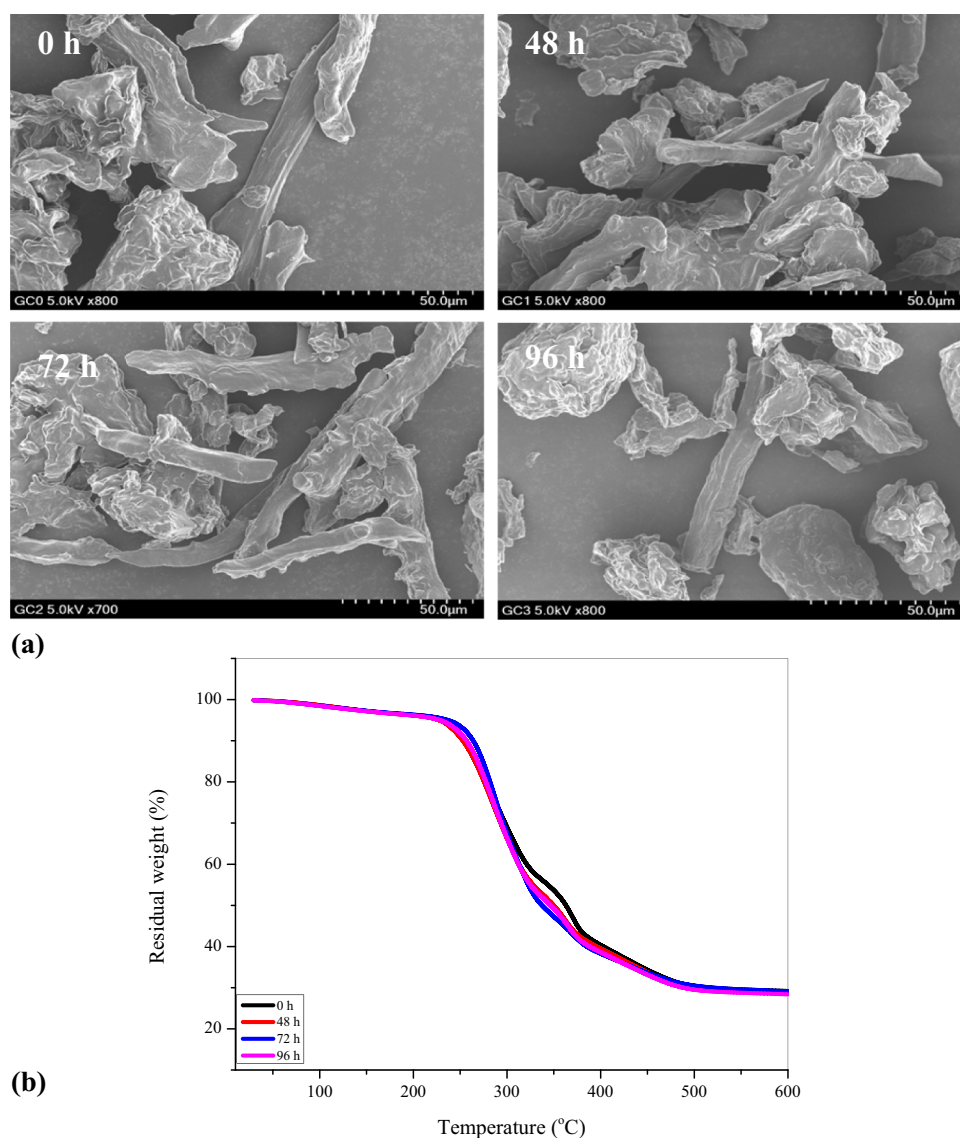


Fig. 1 Structural properties of cellulose synthesized from germinated tiger nut residue **a** FTIR, **b** XRD

The XRD spectra (Fig. 1b) show characteristic crystalline peaks of cellulose I of α -cellulose at $2\theta = 13.0^\circ$, 20.5° , and 34.9° (Melikoglu et al. 2019). Similar diffraction peaks were obtained for the samples at $2\theta = 20.5^\circ$ and 34.9° , however, there was an obvious increase in the diffraction peak $2\theta = 13.0^\circ$ as germination of tiger nuts progressed up to 72 h, likely because of increased alignment of cellulose fibrils in the tiger nut residue following the degradation of non-cellulosic components during germination (Liu et al. 2022). The crystallinity index of cellulose increased by 3.26–5.55% following the germination of tiger nuts for 48–72 h; beyond this, no significant change in crystallinity index was observed (Fig. S1). The increased crystallinity index during 72-h germination validates increased self-assembly of

Fig. 2 Morphology (a) and thermal properties (b) of cellulose synthesized from germinated tiger nut residue



cellulose fibrils owing to a reduction in non-cellulosic components of the residue that would otherwise have resulted in increased amorphization of ordered molecular structures (Liu et al. 2022).

Morphology and thermal properties of cellulose

The SEM images of cellulose samples are presented in Fig. 2a. The images depict the typical fibrillar structure of α -cellulose (Johar et al. 2012). Results show improved self-assembly of cellulose fibrils as germination of tiger nut progressed up to 72 h, thus, validating the FTIR and XRD data. At elevated germination time (96 h), disruption of the ordered fibrillar structure was observed, likely to the disruption of the ordered structure consequent to the increased activity of cellulose-degrading enzymes at the later stages

of germination that partially degraded cellulose (Khorairi et al. 2021).

The thermal properties, as depicted by the thermogravimetric curve, of cellulose samples are shown in Fig. 2b. Similar thermal characteristics were exhibited by the samples during the initial phase of the experiment (30–250 °C) where an average 7% weight loss mainly by the evaporation of bound and absorbed water (Sanchez-Gutierrez et al. 2020) was recorded, implying excellent thermal stability of cellulose synthesized from tiger nut residue and this agrees with the thermal property of cellulose from apple pomace (Melikoglu et al. 2019). During the first thermal degradation phase (250–400 °C), the cellulose produced from 72-h germinated tiger nut residue had the least weight loss, followed by the 48-h germinated sample, relative to the sample produced from the non-germinated tiger nut residue. A similar trend was observed during the second phase of degradation

Table 1 Total phenolic content, flavonoid, and antioxidant activity of flour and cookie samples

Parameter	Sample form	WFC	WFC2	WFC5
TPC (mg GAE/g)	Flour	0.65 ± 0.02 ^{b,A}	0.76 ± 0.03 ^{a,A}	0.73 ± 0.03 ^{a,A}
	Cookie	0.15 ± 0.01 ^{b,B}	0.44 ± 0.00 ^{a,B}	0.42 ± 0.02 ^{a,B}
	Reduction (%)	76.92	42.11	42.47
DPPH (%)	Flour	73.38 ± 0.86 ^{b,A}	77.74 ± 1.33 ^{a,A}	74.60 ± 2.12 ^{b,A}
	Cookie	29.46 ± 0.57 ^{b,B}	49.41 ± 0.54 ^{a,B}	46.56 ± 0.82 ^{a,B}
	Reduction (%)	59.85	36.44	37.59
Flavonoid (mg QE/g)	Flour	1.36 ± 0.04 ^{a,A}	1.39 ± 0.12 ^{a,A}	1.38 ± 0.08 ^{a,A}
	Cookie	0.07 ± 0.00 ^{b,B}	1.01 ± 0.04 ^{a,B}	0.98 ± 0.12 ^{a,B}
	Reduction (%)	94.85	27.34	28.78

Values are means ± standard deviations of triplicate scores

WF: wheat flour; WFC: 100% WF; WFC2: 2% cellulose + 98% WF; WFC5: 5% cellulose + 95% WF

^{a–b}Means with different letters within the same row are significantly different at $p < 0.05$

^{A–B}Means with different letters within the same column are significantly different at $p < 0.05$

(400–600 °C). This implies reduced degradation of cellulose synthesized from 72-h germinated tiger nut residue, likely because of increased resistance of hydrogen bonding to decomposition following the improvement in the fibrillar structure of cellulose during germination, which would enhance the thermal stability of cellulose during elevated temperature processing such as baking.

Findings in this study show that the sample synthesized from the 72-h germinated tiger nut residue had the best structural, morphological, and thermal properties; consequently, it was selected for subsequent study.

Influence of cellulose inclusion on the total phenolic content and antioxidant property of the functional cookie

The TPC, DPPH radical scavenging activity, and flavonoid of the flour blends and the functional cookies are presented in Table 1. As expected, the TPC and DPPH of the flour blends were higher than those of the cookies, likely due to the decomposition of some phenolic components in the clove extract during the baking operation (Pilog et al. 2023); however, the addition of cellulose significantly reduced the impact of the high baking temperature. There was a 76.92%, 42.11%, and 42.47% reduction in the TPC of the samples produced using WF + clove extract (WFC), WF + clove extract + 2% cellulose (WFC2), and WF + clove extract + 5% cellulose (WFC5), respectively, indicating that cellulose addition preserved phenolics during baking. This is probably due to the high hydrogen bonding and fibrillar nature of cellulose that reduced de-conjugation of the aromatic particle ring of the phenoxyl components of the clove extract (Harlina et al. 2018), corroborating the findings of Ranjbaryan et al. (2019) that the addition of cellulose nanofiber preserved cinnamon essential oil in a sodium caseinate-based matrix. The DPPH result followed a similar

trend, due to the high TPC (El-Maati et al. 2016); suggesting that the thermo-protection of phenolic compounds by cellulose resulted in increased antioxidant activity of the cookies. Baking had the most effect on the flavonoid content of the WFC, as evident in 94.85% loss, meanwhile, the addition of cellulose resulted in 27.34–28.78% reduction, implying a 3.29–3.46-fold preservation of flavonoid, confirming the stabilizing effect of cellulose owing to its high concentration of hydrogen bonds (Melikoglu et al. 2019). The TPC, flavonoids, and DPPH of the samples were not significantly influenced by the concentration of cellulose, indicating that the antioxidant property of the functional cookie could be preserved using a low concentration of cellulose.

Influence of cellulose inclusion on the rheological properties of the dough

The dynamic viscoelasticity data (Fig. S2 and Table 2) show that the inclusion of cellulose significantly influenced the rheological properties of the dough, agreeing with previous rheological studies of WF and cellulose-rich matrices (Adedeji et al. 2022; Ren et al. 2020; Tarancon et al. 2015). At lower angular velocity (< 6.31 rad/s), the G' increased as the concentration of cellulose increased (Fig. S2a), likely to the intermolecular interaction between the constituent macromolecules that enhanced the mechanical properties of the dough under low oscillation (Tarancon et al. 2015); however, at higher angular velocity (> 6.31 rad/s), the G' of WFC5 reduced relative to WFC and WFC2, due to the exfoliation of cellulose from the WF matrix due to the disruption of the gluten-cellulose network at high oscillation (Tarancon et al. 2015), suggesting poor stability and elasticity of WFC5 during operations such as mixing and kneading (Millar et al. 2019). This corroborates a report that the addition of high-fiber *goji* berry interfered with the gluten network of WF dough (Bora et al. 2019). The G'' of the samples (Fig. S2b)

Table 2 Viscoelasticity parameters of the dough samples

Model	Parameter	WFC	WFC2	WFC5
$G' = K'(\omega)^{n'}$	K'	$28,339.16 \pm 10.13^c$	$30,853.33 \pm 17.92^b$	$45,572.02 \pm 39.63^a$
	n'	0.0295 ± 0.0010^b	0.0215 ± 0.0052^c	0.0752 ± 0.0054^a
	R^2 (%)	96.27	86.72	82.05
$G'' = K''(\omega)^{n''}$	K''	$14,388.72 \pm 16.57^c$	$15,549.75 \pm 3.53^b$	$23,458.77 \pm 6.74^a$
	n''	0.0794 ± 0.0007^a	0.0435 ± 0.00^b	0.0578 ± 0.0186^a
	R^2 (%)	96.98	91.59	87.32
$G^* = A_F(\omega)^{1/z}$	A_F (Pa.s)	$31,792.95 \pm 11.24^c$	$34,509.85 \pm 12.94^b$	$52,104.16 \pm 5.43^a$
	z	12.58 ± 0.42^b	12.97 ± 0.57^b	29.85 ± 0.21^a
	R^2 (%)	96.69	90.15	86.97

Values are means $\pm$ standard deviations of triplicate scores

WF: wheat flour; WFC: 100% WF; WFC2: 2% cellulose + 98% WF; WFC5: 5% cellulose + 95% WF

^{a-c}Means in rows with different superscripts are significantly different, $p < 0.05$

increased with increasing cellulose content, indicating increasing energy dissipation during deformation (Millar et al. 2019). Generally, a higher G' than G'' was obtained for all the samples throughout the experiment, indicating the solid-like property of the dough (Ren et al. 2020).

Results (Table 2) show an increasing K' and K'' with an increasing concentration of cellulose, implying an increase in viscosity and elasticity following electrostatic interaction between the macromolecules of the constituting polymers (Adediji et al. 2022). A higher K' than K'' was obtained for all the samples, confirming the solid-like nature of the dough samples (Ren et al. 2020). Despite its higher K' and K'' , WFC5 had a higher n' and n'' , compared to WFC2, indicating higher dependency of its G' and G'' on frequency that might negatively affect its stability during kneading and baking operations (Tarancon et al. 2015). WFC2 had lower n' and n'' than WFC, suggesting improved stability of the dough, due to the fibrillating effect of cellulose that enhanced intermolecular crosslinking through hydrogen bonding and hydrophobic-hydrophobic interaction (Ren et al. 2020). The weak gel model (Eq. 4) revealed an increasing A_F , a measure of dough stiffness, with an increasing cellulose concentration, validating an improvement in the solid-like nature of the dough. The z of WFC5 was different from WFC and WFC2, probably because of the higher interacting rheological units that led to high K' and K'' (Tarancon et al. 2015).

Influence of cellulose inclusion on the proximate composition of the cookie

The proximate compositions of the cookie samples varied according to the concentration of cellulose (Table 3). The moisture content of the cookie samples was generally low (1.52–1.7 g g/100 g), indicating good shelf stability of the samples (Cheng and Bhat 2016). The addition of 2% cellulose did not influence the moisture content of the cookie;

however, as the concentration increased to 5%, the moisture content increased by 10.69%, due to the electrostatic repulsion of the gluten-starch-cellulose matrix due to the exfoliation of the excess cellulose that reduced its water-binding capacity (Tarancon et al. 2015). The protein content of the cookie increased by 22.39% and 17.27% following the addition of 2% and 5% cellulose, respectively, due to the preservation of globular proteins and peptides, mainly from gluten, milk, and egg white, by cellulose; suggesting the encapsulating efficacy of cellulose on protein molecules (Ranjbarian et al. 2019). This implies that the nutritional quality of bakery products could be improved by exploiting the thermal property of cellulose. The reduction in protein content following 5%-cellulose addition indicates a probable expulsion of excess cellulose, compromising the hydrogen bonding and the electrostatic cohesion of the matrix (Tarancon et al. 2015). The ash content of the samples followed a similar trend as the protein content; however, fat content increased with increasing cellulose content implying the encapsulating effect of cellulose on fat globules that would improve textural and sensory qualities (Naseer et al. 2021). Carbohydrate was reduced by 13.96–15.72% following cellulose addition in the cookie matrix, due to the increase in protein, ash, and fat contents. The total dietary fiber increased by 38.10–67.77% as the concentration of cellulose in the cookie matrix increased, implying an improvement in the bio-functionality of the product since dietary fiber produces key health-promoting biomarkers following its degradation by gut's microorganisms (Chinma et al. 2022). Previous studies also reported an increase in the dietary fiber content of bakery products following the addition of cellulose-rich residues (Adegunwa et al. 2020; Bora et al. 2019; Naseer et al. 2021).

Table 3 Proximate composition, texture profile, and color attributes of the cookies

Parameter	WFC	WFC2	WFC5
Moisture content (g/100 g)	1.59 ± 0.01 ^b	1.52 ± 0.04 ^b	1.76 ± 0.07 ^a
Protein content (g/100 g)	4.69 ± 0.05 ^c	5.74 ± 0.10 ^a	5.50 ± 0.05 ^b
Fat content (g/100 g)	30.63 ± 0.52 ^c	35.77 ± 0.46 ^b	37.66 ± 0.18 ^a
Ash content (g/100 g)	3.13 ± 0.23 ^b	4.60 ± 0.13 ^a	4.38 ± 0.10 ^a
Carbohydrate (g/100 g)	59.61 ± 0.81 ^a	51.29 ± 0.32 ^b	50.24 ± 0.02 ^b
Total dietary fiber (g/100 g)	0.39 ± 0.01 ^c	0.63 ± 0.09 ^b	1.21 ± 0.27 ^a
Dough yield (%)	92.64 ± 0.47 ^a	91.04 ± 1.35 ^a	90.77 ± 0.30 ^a
Spread ratio	4.45 ± 0.11 ^b	5.90 ± 0.09 ^a	5.67 ± 0.01 ^b
Hardness (N)	73.80 ± 0.76 ^c	76.59 ± 0.45 ^b	82.46 ± 0.61 ^a
Springiness (mJ)	0.30 ± 0.01 ^a	0.32 ± 0.02 ^a	0.21 ± 0.01 ^b
Gumminess	9.99 ± 0.69 ^c	13.78 ± 1.28 ^b	18.35 ± 2.44 ^a
Chewiness	3.18 ± 0.36 ^c	4.89 ± 0.79 ^b	8.25 ± 1.13 ^a
Cohesiveness	0.24 ± 0.01 ^b	0.27 ± 0.15 ^b	0.14 ± 0.03 ^a
<i>L</i> *	51.98 ± 0.01 ^a	49.24 ± 0.02 ^b	44.89 ± 0.03 ^c
<i>a</i> *	−4.23 ± 0.01 ^a	−1.91 ± 0.02 ^b	−1.20 ± 0.08 ^c
<i>b</i> *	17.75 ± 0.68 ^b	18.53 ± 0.01 ^a	18.80 ± 0.07 ^a
Color intensity	37.52 ± 1.33 ^a	35.97 ± 0.02 ^b	31.28 ± 0.05 ^c

Values are means ± standard deviations of triplicate scores

WFC: 100% WF; WFC2: 2% cellulose + 98% WF; WFC5: 5% cellulose + 95% WF

^{a–c}Means in rows with different superscripts are significantly different, $p < 0.05$

Influence of cellulose inclusion on the physical, textural, and color attributes of the cookie

The physical, textural, and color attributes of the cookies are presented in Table 3. The addition of cellulose did not affect the dough yield of the cookie, likely to the stability of the hydrogen bonding of the gluten-starch-cellulose network during baking, corroborating a report that the addition of tiger nut fiber did not significantly alter the dough yield of cookie (Verdu et al. 2017). Irrespective of concentration, cellulose addition increased the spread ratio of the cookie by 27.42–32.58%, likely to the increase in stiffness and interacting rheological units brought about by the electrostatic interaction between gluten, starch, and cellulose, agreeing with a report that the addition of carboxyl methyl cellulose increased the spread ratio of a WF-based cookie (Naseer et al. 2021).

Hardness, gumminess, and chewiness increased with an increasing concentration of cellulose in the cookie matrix (Table 3), likely to the intermolecular interaction between gluten and cellulose, similar to the report that the addition of fiber-rich mango peel powder enhanced the 3D network of gluten of a bakery product (Chen et al. 2019). Hardness is the force required for cookie disintegration and chewiness indicates the energy required for mastication (Amin et al. 2021). The findings obtained in this study suggest that the addition of cellulose might increase the resident time of the cookie in the mouth. Meanwhile, the addition of 2% cellulose did not influence the springiness and the cohesiveness

of the cookie, unlike the significant reduction recorded following 5% cellulose addition, likely to the reduction in the electrostatic and cohesive forces of the 3D gluten network due to the un-reacting excess cellulose (Millar et al. 2019), indicating a slower rate of recovery after compression (Adediji et al. 2022). This agrees with a reduction in bread's springiness with an increasing level of cellulose fiber (Fendri et al. 2016).

The measure of lightness and color intensity decreased while measures of redness (a^*) and yellowness (b^*) increased with increasing cellulose concentration in the cookie (Table 3), probably because of Maillard reaction among the constituting polymers as obtained in a similar report (Fendri et al. 2016). The reduction in a^* and b^* following cellulose addition in the cookie might enhance the golden color of the product, improving its acceptability.

Influence of cellulose inclusion on the sensory attributes of the cookie

The sensory attributes of the cookies (Fig. S3) in terms of taste, crispiness, color, aroma, and overall acceptability are presented in Table S1. The WFC2 compared significantly with WFC in overall acceptability. The panelists showed a higher taste and crispiness preference for WFC2 than WFC and WFC5, due to its better textural quality brought about by the stable 3D network formed by the hydrogen bonding between gluten and cellulose (Ren et al. 2020). In terms of color, the panelists showed varying preferences

for the samples in the order WFC > WFC2 > WFC5, probably because of the contrasting color of cellulose and WF in the cookie as the concentration of cellulose in the matrix increased, agreeing with the reduced color preference of bakery products following the addition of cellulose-rich fibers (Adegunwa et al. 2020; Adeloye et al. 2020). The panelists show a low preference for the WFC5 relative to the WFC and WFC2, likely to the impact of the unreactive exfoliated cellulose.

Conclusions

This study showed an improvement of the structure, crystallinity, morphology, and thermal properties of cellulose after germinating tiger nut. Optimum cellulose properties were achieved following the germination of tiger nut for 72 h. Also, incorporating cellulose into WF matrix improved the antioxidant and physicochemical properties of the resulting cookie. The inclusion of 2% cellulose was adequate to preserve the TPC and antioxidant activity, induced by the addition of clove extract powder, of the cookie during baking. Also, adding 2% cellulose stabilized the nutrients, mainly protein, of the cookie and enhanced the viscoelastic, textural, and sensory properties. Using 5% cellulose in the dough matrix negatively affected the highlighted parameters.

Author contributions OEA: Conceptualization, Investigation, Writing—original draft, formal analysis, resources, writing—review and editing; OAA: Investigation, formal analysis, project administration, supervision, writing—review and editing; OGA: investigation, writing—original draft, formal analysis, resources; HJK, NI, and JHM: Resources, investigation; JAA and CEC: Resources, project administration, writing—review and editing; YHJ: Funding acquisition, Project administration, supervision, writing—review and editing.

Funding This work was supported by the Korea Institute of Planning and Evaluation for Technology in Food, Agriculture, and Forestry (Grant No. 321028–5).

Data availability All data generated or analyzed during this study are included in this published article [and its supplementary information files].

Code availability Statistical package for the social scientists (version 23.0, IBM Statistics, Armonk, NY, USA).

Declarations

Conflict of interest The authors declare that they have no competing interests.

Ethical approval Not applicable.

Consent to participate Not applicable.

Consent for publication Not applicable.

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