RESEARCH ARTICLE

Response Surface Methodology Study of the Synergistic Effects of Soaking Pretreatment and Microwave Power on the Proximate Composition of Tiger nut (*Cyperus esculentus* L.)

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Abstract

Despite its nutritional potential, Tiger nut remains under-utilized. This study examined the interactive effects of soaking time (24–72 h) and microwave power (100–280 W) on its proximate composition using response surface methodology. Commercial tubers (14.33% moisture) were rehydrated to 38.6–46.8% moisture via soaking and then microwave-dried at 2.45 GHz. Proximate composition was determined by AOAC methods. Ash, fat and carbohydrate followed linear models ($p < 0.0001$); fiber followed a two-factor interaction model ($p < 0.0001$); protein showed quadratic behavior ($p = 0.0006$). Multi-response optimization identified 48 h soaking and 190 W power as optimal (desirability=0.87). Five validation runs confirmed model predictions (no significant differences, $p > 0.05$; relative errors 0.56–2.62%), yielding: 4.18% ash, 5.62% fiber, 11.53% fat, 14.69% protein, 61.07% carbohydrate, and 2.91% moisture. Mechanistically, protein degradation exhibited non-thermal microwave effects (quadratic model), while fat followed thermal oxidation kinetics (linear model, $r = -0.87$). Dielectric loss factor (ε'') rose from 1.0–1.5 to 7–10 as moisture increased from 14.33% to 46.8%, and penetration depth (≈15–20 mm) exceeded tuber diameter (7.12 mm), confirming volumetric heating. These findings demonstrate synergistic interactions between soaking and microwave drying, providing a validated framework for industrial Tiger nut processing balancing nutrient retention and shelf stability.

Keywords: tiger nut, microwave drying, soaking pre-treatment, proximate composition.

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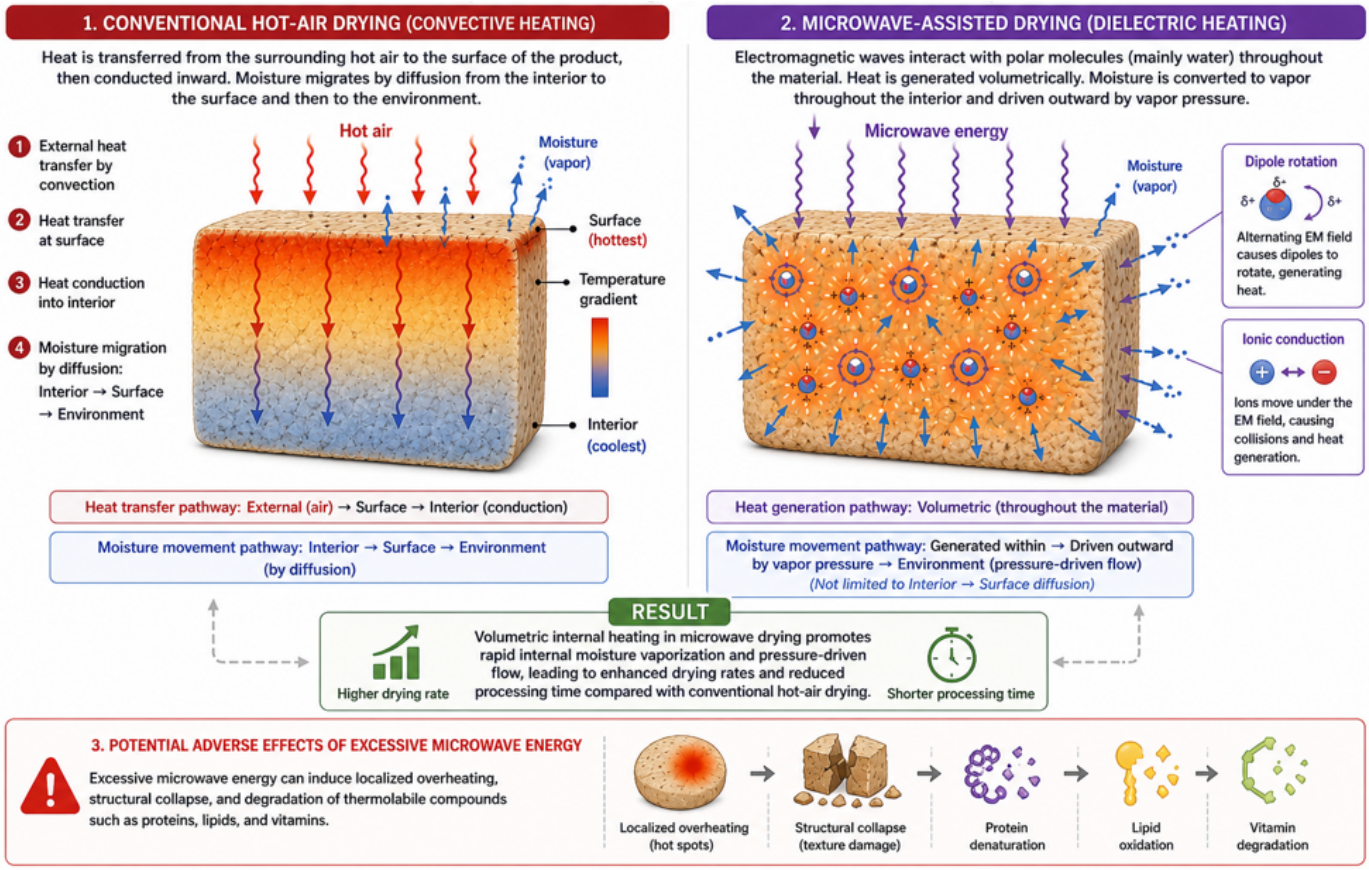

Figure 1. Schematic comparison of heat and mass transfer in conventional hot-air and microwave drying, highlighting moisture removal mechanisms and potential damage from excessive microwave energy.

1 Introduction

Tiger nut (*Cyperus esculentus L.*) is an underutilized perennial sedge widely cultivated across tropical and subtropical regions, valued for its edible tubers and adaptability to marginal environments. Although its use dates back to ancient civilizations [1, 2], its modern industrial application remains limited despite increasing global production and interest. The Valencia region of Spain is the leading producer of Tiger nut, with an annual output of about 5.3 million kilograms. Other nations where production is significant include Nigeria, Ghana, Niger, Burkina Faso, China, India, Chile, the United States, and Brazil [3].

Nutritionally, Tiger nut tubers are substantially rich in carbohydrates (especially starch and soluble sugars), monounsaturated fatty acids (comprising lipid), a reasonable amount of protein, and a significant content of dietary fiber [4]. The tubers also contain micronutrients (calcium, magnesium, potassium, and iron) and bioactive phytochemicals (flavonoids, alkaloids, tannins, phenolic compounds, etc.). The robust biochemical composition of Tiger nut makes it a unique food material with desirable antioxidant capacity and such physiological benefits as anti-inflammatory, hypocholesterolemic, anticancerous, and glycemic regulatory effects [5–8]. Conversely, the biochemical composition of Tiger nut is liable to both primary and secondary postharvest and processing conditions.

The crop has gained attention due to its resilience and potential role in food security, particularly in developing regions. In recent years, research has focused on improving its processing and utilization, especially through techniques that enhance its nutritional quality and shelf stability [9, 10]. An important factor often overlooked in Tiger nut processing studies is the significant moisture loss that occurs during postharvest handling. Freshly harvested Tiger nut tubers typically contain >40% moisture on a wet basis, yet in typical Nigerian supply chains, farmers and traders partially dry the tubers to 12–18% moisture content before marketing. This practice, rooted in indigenous knowledge, serves three critical purposes: (1) it substantially reduces postharvest losses by inhibiting microbial growth; (2) it decreases transportation weight and associated costs; and (3) it

allows for extended storage without refrigeration.

Drying, being one of the food processing unit operations, entails the simultaneous transfer of heat and mass that causes the transfer of moisture from food materials' interior structural matrices to the surface, and subsequent evaporation of the ejected moisture into the surrounding environment [11, 12]. The rate at which drying occurs is governed by capillary flow, internal diffusion mechanisms, and external convective mass transfer [13–16]. Pretreatment processes, particularly soaking, introduce another layer of complexity to the system. Soaking facilitates hydration of the cellular matrix, resulting in swelling, increased porosity, and modification of cell wall permeability [17, 18]. From a mass transfer perspective, soaking establishes a concentration gradient between the soaking medium and the intracellular components, leading to leaching of soluble solids such as sugars, minerals, and nitrogenous compounds [19]. Simultaneously, it promotes enzymatic activities that may alter the biochemical composition of the tubers [17, 20].

In conventional hot-air drying, heat is typically projected from a surrounding medium to the surface of the product, followed by absorption/conduction into the interior to create the temperature gradients that induce drying. Several studies hold that conventional hot-air drying is characterized by prolonged drying time and possible degradation of heat-sensitive nutrients [21, 22]. In contrast, microwave drying operates based on dielectric heating, where electromagnetic waves interact with polar molecules (primarily water) within the material. This interaction induces dipole rotation and ionic conduction, generating volumetric heat internally [23–25]. As a result, moisture evaporation occurs from within the material matrix, leading to enhanced drying rates and reduced processing time (see Figure 1). However, excessive microwave energy can induce localized overheating, structural collapse, and degradation of thermolabile compounds such as proteins, lipids, and vitamins [26, 27].

The synergistic effect of soaking a product and subsequently subjecting it to microwave drying requires serious attention as it could advance beyond mere additive to interactive phenomenon. Soaking operations tend to alter the microstructural matrix and moisture distribution within the tuber [28]. This is likely to impact the rate of microwave energy absorption and heat generation patterns during the drying process. The increase in moisture content associated with soaking operations has been reported to improve dielectric properties [29, 30]. While such could improve microwave energy coupling, there are also chances that uneven heating and potential energy degradation may occur if the process is not properly controlled.

The comprehension of the synergistic effect of soaking pre-treatments and microwave drying requires a logical and assessable approach. In recent food processing undertakings, Response Surface Methodology (RSM) has emerged as a very reliable scientific and statistical tool for modelling and optimizing composite processes that often comprise dual or multiple variables and their corresponding interactions. In principle, RSM integrates experimental design, regression modelling, and optimization methodologies in order to create relationships between independent and response variables. The assessment tool also helps researchers to identify optimal processing conditions with a reduced number of experimental runs [31, 32].

Despite the growing body of research on Tiger nut processing, studies focusing on the combined influence of soaking time and microwave power on proximate composition remain scarce. Most existing works have examined these variables independently, thereby overlooking potential synergistic or antagonistic interactions. Given that proximate composition is a critical indicator of nutritional quality, there is a need to establish optimized processing conditions that preserve desirable attributes while minimizing losses. In food processing applications, RSM has been widely applied to optimize drying conditions, extraction processes, and formulation variables. The technique typically employs second-order polynomial models to describe system behavior, capturing both linear and interaction effects of process variables. For drying processes, RSM facilitates the evaluation of how factors such as temperature and microwave power influence responses like moisture content, nutrient retention, and energy efficiency.

This study, therefore, aims to investigate the combined effects of soaking time and microwave power on the proximate composition of Tiger nut using Response Surface Methodology. By integrating experimental analysis with statistical modelling, the study seeks to develop predictive models and identify optimal processing conditions that enhance product quality. The findings are expected to contribute to improved

processing strategies, promote value addition, and support the wider utilization of Tiger nut in food systems.

2 Materials and Methods

2.1 Raw material sourcing and preparation

Two categories of Tiger nut samples were obtained for this study. The first category, commercial samples, served as the primary experimental material. Fresh Tiger nut tubers of the mini yellow species (*Cyperus esculentus var. sativus Boeckeler*, JYD-35 accession) were purchased from a daily market in Ekwulobia town, Aguata Local Government Area, Anambra State, Nigeria. These tubers represent the typical raw material available to local food processors. Upon arrival at the laboratory, the samples were manually sorted to remove defective, damaged, or immature tubers. Only tubers that displayed uniformity in size, color, and structural appearance were selected, a precaution taken to drastically minimize experimental variability. Preliminary botanical characterization showed that the sampled Tiger nut tubers had a mean geometric diameter of 7.12 $\pm$ 0.77 mm and an approximately spherical geometry, with mean sphericity of 0.964, mean aspect ratio of 0.850, and mean flakiness ratio of 1.03.

The second category consisted of freshly harvested samples, which were used as reference material to establish the full postharvest moisture loss trajectory and to understand processing-induced changes relative to the true fresh state. These tubers were obtained directly from farms within the same local government area within 24 hours of harvest, then transported to the laboratory in insulated containers and analyzed within 48 hours.

The initial moisture content of the commercial tubers was determined to be 14.33% (wet basis) using the oven-drying method at 105°C until constant weight was achieved, while the freshly harvested tubers had an initial moisture content of 44.20% (wet basis). This difference of nearly 30 percentage points represents moisture loss during postharvest handling, including open sun drying, which is standard practice in the local supply chain to prevent rapid spoilage during transportation and storage. The commercial samples at 14.33% moisture served as the baseline control for all processing experiments because they reflect the actual raw material received by food processors, ensuring industrial relevance since processors rarely receive truly fresh tubers. The freshly harvested samples at 44.2% moisture were used only for comparative characterization to document the full moisture loss trajectory.

The postharvest handling protocol for commercial Tiger nut tubers in the study region follows established local practices. Freshly harvested tubers undergo open sun drying on elevated mats for a period of 5 to 10 days, depending on prevailing weather conditions. To promote uniform moisture removal, the tubers are turned manually two to three times each day. Overnight, the drying mats are covered with polyethylene sheeting to minimize moisture reabsorption from dew. Once sufficiently dried, the tubers are kept in breathable jute sacks under ambient storage conditions (temperature range 25–32°C, relative humidity 60–80%) for durations of 1 to 6 months prior to retail distribution. No chemical preservatives or anti-browning agents are employed during any stage of postharvest handling. This documentation provides a clear reference for the postharvest history of commercial Tiger nut tubers used in this study, thereby enhancing the reproducibility of the baseline compositional data reported.

2.2 Single-factor pre-experiments for design space determination

Prior to the main factorial experiment, single-factor pre-experiments were conducted to establish the approximate operating ranges for soaking time and microwave power. For soaking time, Tiger nut tubers (commercial baseline, 14.33% moisture) were submerged in distilled water at room temperature (27 $\pm$ 1.4°C) with a water-to-sample ratio of 4:1 (v/w) for durations of 0, 12, 24, 36, 48, 60, 72, 84, and 96 hours (single determination per time point). Moisture content was measured after each interval. Results showed rapid water absorption during the first 24 hours (reaching 38.6% moisture), followed by gradual uptake to 44.5% at 48 hours and 46.8% at 72 hours, with minimal additional increase beyond 72 hours (47.1% at 96 hours). Based on these results, soaking times of 24, 48, and 72 hours were selected for the main experiment to represent under-rehydration, full rehydration (matching fresh tuber moisture of 44.2%), and extended rehydration, respectively.

For microwave power, 100 g samples of soaked tubers (48 h soaking) were dried in the microwave oven at power settings of 50, 100, 150, 200, 250, 300, and 350 W (single determination per power level) until equilibrium moisture content. Power levels below 100

W resulted in excessively long drying times, while levels above 280 W caused visible scorching and case hardening. Therefore, microwave power levels of 100, 140, 200, and 280 W were selected for the main experiment to span the range from gentle drying to the upper limit before product damage.

2.3 Soaking pre-treatment

An important consideration in this study is that soaking serves as a rehydration step for the partially dried commercial tubers. Water absorption kinetics were monitored during soaking. After 24 hours of soaking, the moisture content of commercial tubers increased from 14.33% to 38.6 $\pm$ 1.2%; after 48 hours, to 44.5 $\pm$ 0.9% (effectively restoring the moisture content to that of freshly harvested tubers); and after 72 hours, to 46.8 $\pm$ 0.7%. This demonstrates that 48 hours of soaking is sufficient to fully rehydrate the commercially dried tubers to a moisture state approximating fresh material. Extended soaking beyond 48 hours results in marginal additional water uptake but may increase leaching losses.

For each of the soaking durations (24, 48, and 72 hours), cleaned Tiger nut tubers were submerged in distilled water at room temperature (27 $\pm$ 1.4°C) with a water-to-sample ratio of 4:1 (v/w). For samples designated for soaking beyond 24 h, the water was changed after 24 hours to minimize the chances of microbial proliferation. After soaking, absorbent paper was used to blot off surface moisture to ensure uniform pre-treatment conditions before the drying experiments.

2.4 Microwave drying process

For each experimental run, 100 g of the soaked Tiger nut samples (for each respective duration) were uniformly spread on the microwave turntable glass before initiating the designated microwave power treatment. A domestic microwave oven (CMV 20L-E model, Samsung Electronics, operating at 2.45 GHz frequency, with maximum output 800 W) was used for all drying experiments. The drying operation was continued until equilibrium moisture content was attained (defined as less than 0.1% mass change over three consecutive 30-minute measurements). It is acknowledged that this criterion may not represent true thermodynamic equilibrium, as residual bound water may remain within the tuber matrix. No independent verification (e.g., Karl Fischer titration) was performed to confirm that true equilibrium was achieved. Weight measurements were taken intermittently at 30-minute intervals using an electronic balance (Shimadzu AUN 220D; 0.0001 g sensitivity).

2.4.1 *Theoretical basis for microwave heating*

There are fundamental frameworks for the drying of Tiger nut. In microwave drying, Risman [33] reported that the power absorbed per unit volume (P) is given by:

$$P = 2\pi f \varepsilon_0 \varepsilon'' E^2 \tag{1}$$

where f is frequency of microwave radiation (2.45×10^9 Hz), ε_0 is permittivity of free space (8.854×10^{-12} F/m), ε'' is dielectric loss factor (material-specific, moisture- and temperature-dependent), E is electric field strength.

Several studies have reported values for similar tuber and oilseed materials [23, 34], and the dielectric loss factor (ε'') for food materials like Tiger nut is estimated as a function of moisture content [35]. At commercial baseline (14.33% moisture), ε'' ranges from 1.0–1.5, indicating low energy absorption with surface heating dominance. After 24 hours soaking (38.6% moisture), ε'' increases to 5–7, representing moderate energy absorption with transition to volumetric heating. After 48 hours soaking (44.5% moisture), ε'' reaches 7–9, confirming high energy absorption and efficient volumetric heating. After 72 hours soaking (46.8% moisture), ε'' reaches 8–10, indicating very high energy absorption with risk of localized overheating at higher power levels [36].

Following the application of Maxwell's equations to study how electromagnetic plane waves propagate through unbounded homogeneous media [37], the penetration depth (D_p), defined as the distance microwave energy can travel into a material, is given by:

$$D_p = \frac{\lambda_0}{2\pi\sqrt{2\varepsilon'}} \times \frac{1}{\sqrt{1 + \left(\frac{\varepsilon''}{\varepsilon'}\right)^2 - 1}} \tag{2}$$

where λ_0 is 122 mm (at 2.45 GHz), ε' is the dielectric constant.

For moist Tiger nut at 44.5% moisture ($\varepsilon'' \approx 8$, $\varepsilon' \approx 12$), the estimated penetration depth is approximately 15–20 mm. Since the geometric mean diameter of Tiger nut tubers is 7.12 mm (radius $\approx$ 3.6 mm), the penetration depth exceeds the tuber radius by a factor of 4-5, confirming that microwave energy penetrates completely through individual tubers at moderate moisture levels, enabling true volumetric heating [38].

2.5 Experimental design

A two-factor factorial completely randomized design (CRD) was adopted for this study. The experimental factors were soaking time (three levels: 24, 48, and 72 hours at room temperature of 27 ± 1.4 °C) and microwave power (four levels: 100, 140, 200, and 280 W), resulting in twelve different treatment combinations. Each treatment combination was carried out in triplicate, producing 36 experimental units. To reduce systematic bias, the order of all experimental runs was randomized using Design-Expert software (version 13, Stat-Ease Inc., Minneapolis, MN, USA). The resulting randomization sequence, along with the corresponding treatment codes (each indicating both soaking duration and microwave power), is shown in Table 1.

Table 1. Randomized Run Order for the 3 (Soaking Time) × 4 (Microwave Power) Factorial Experiment.

Run order	Soaking time (h)	Microwave power (W)	Treatment code
1	48	100	t48_P100
2	24	200	t24_P200
3	72	140	t72_P140
4	24	280	t24_P280
5	72	100	t72_P100
6	48	140	t48_P140
7	24	100	t24_P100
8	72	280	t72_P280
9	48	200	t48_P200
10	72	200	t72_P200
11	24	140	t24_P140
12	48	280	t48_P280

Each treatment code combines soaking time (t followed by hours) and microwave power (P followed by watts). The table displays one representative run order per treatment; all three replicates were randomized together in the full sequence.

While standard Response Surface Methodology designs (e.g., Central Composite or Box-Behnken) typically employ three or more factors, the present study restricted the investigation to two factors to manage experimental scope while still allowing second-order polynomial modelling of the selected responses.

2.6 Response surface methodology modelling

A second-order polynomial model based on Response Surface Methodology was employed to analyze the experimental data. The independent variables were soaking time (X_1: 24, 48, 72 h) and microwave power (X_2: 100, 140, 200, 280 W). The general second-order polynomial model used is:

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_{11} X_1^2 + \beta_{22} X_2^2 + \beta_{12} X_1 X_2 + \varepsilon \quad (3)$$

where Y is response variable (proximate composition parameter), β_0 is intercept, β_1, β_2 are linear coefficients, β_{11}, β_{22} are quadratic coefficients, β_{12} is interaction coefficient, ε is experimental error.

This model enables evaluation of both individual and combined effects of the process variables. All independent variables were coded to the range -1 to $+1$ for regression analysis to allow direct comparison of coefficient magnitudes. The applied coding formula is expressed as:

$$X_1(\text{coded}) = \frac{(\text{soaking time} - 48)}{24} \quad (4)$$

$$X_2(\text{coded}) = \frac{(\text{microwave power} - 190)}{90} \quad (5)$$

It is acknowledged that the selected microwave power levels (100, 140, 200, 280 W) and soaking times (24, 48, 72 h) are discrete and unevenly spaced. The coding formulae above assume continuous variables for regression purposes, which is a limitation of the present factorial design. Future studies employing a Central Composite Design with equally spaced levels would provide more robust second-order modelling. Nevertheless, the present approach is sufficient for identifying main effects and interaction trends within the tested ranges.

2.7 Proximate composition analysis

The nutritional quality of Tiger nut was evaluated using proximate composition analysis. All determinations were carried out in triplicate (n = 3) for each experimental unit (i.e., three analytical replicates per treatment replicate, totaling 108 analytical measurements) to ensure precision and statistical consistency, in accordance with the standardized methods of the Association of Official Analytical Chemists [39]. For each experimental replicate, a representative subsample was obtained, and all parameters were analyzed to maintain accuracy and reproducibility.

Moisture content was determined by oven-drying the samples at 105 °C until constant weight was achieved, following AOAC [39] Method 934.01. Ash content

was assessed by incinerating the samples in a muffle furnace at 550 °C for 5–6 hours, in line with AOAC [39] Method 942.05. Crude fat was extracted using a Soxhlet extraction system with anhydrous diethyl ether as the solvent, applying a continuous extraction procedure as specified in AOAC [39] Method 920.39. Crude protein (expressed as nitrogen × 6.25) was determined using the micro-Kjeldahl method, which involved digestion with concentrated sulphuric acid, followed by distillation and titration, in accordance with AOAC [39] Method 978.04. Crude fiber was analyzed through sequential digestion with 1.25% sulphuric acid and 1.25% sodium hydroxide solution, as described in AOAC [39] Method 978.10. Total carbohydrate content was subsequently estimated by difference using Equation 6:

$$\%\text{Carbohydrate} = 100 - (\%\text{Protein} + \%\text{Moisture} + \%\text{Ash} + \%\text{Fat} + \%\text{Fiber}) \quad (6)$$

2.8 Statistical analysis and model development

The experimental data derived from the factorial combination of soaking time and microwave power were analyzed using Response Surface Methodology with the second-order polynomial model expressed in Equation 2. All analyses were performed on the full dataset of 36 observations (12 treatments × 3 replicates) using Design-Expert software (version 13). The suitability of the developed models was appraised using ANOVA, coefficient of determination (R^2), adjusted R^2, predicted R^2, and lack-of-fit tests. Residual diagnostics (normality of residuals using Shapiro-Wilk test, homogeneity of variance using Levene's test) were performed to validate ANOVA assumptions.

Multi-response optimization was performed using the desirability function approach with desirability weights (protein: 5, fat: 4, fiber: 3, ash: 2, carbohydrate: 2) justified by industrial relevance and nutritional importance. The optimization constraints were: soaking time (24–72 h, target 48 h), microwave power (100–280 W, target 190 W), ash (maximize), fiber (maximize), fat (maximize), protein (maximize), carbohydrate (maximize), moisture (constraint 2–4%, experimentally determined).

2.9 Experimental validation of predicted optimum

Based on the desirability function analysis, the optimal conditions were predicted as 48 hours soaking and 190 W microwave power. It is noteworthy that 190 W was not among the original experimental power levels; it was interpolated between 140 W and 200 W. To validate this prediction and justify the interpolation, five independent validation runs (n = 5) were conducted at 48 h soaking and 190 W microwave power, following the identical procedures described in Sections 2.3 and 2.4. The proximate composition of the validation samples was analyzed in triplicate each. The experimental validation values were compared with the predicted values using two-tailed t-tests ($\alpha = 0.05$) to assess significant differences. The relative prediction error (RPE) was calculated as:

$$RPE(\%) = \frac{(\text{Predicted value} - \text{Experimental value})}{\text{Predicted value}} \times 100 \quad (7)$$

Model validity was considered acceptable if $RPE < 10\%$ for all responses and t-tests showed no significant differences ($p > 0.05$). Additionally, validation results at 190 W were compared with experimental data at adjacent power levels (140 W and 200 W, all at 48 h soaking) using one-way ANOVA with Tukey's HSD post-hoc test ($\alpha = 0.05$) to confirm statistical distinction.

2.10 Determination of activation energy for moisture diffusion

To understand the energy barrier for moisture removal during microwave drying, the activation energy (E_a) was derived from the temperature dependence of effective moisture diffusivity. For each soaking duration (24, 48, and 72 hours), the drying trials at four microwave power settings (100, 140, 200, and 280 W) provided the necessary data. During the constant-rate drying phase, the temperature of the tuber bed was recorded using a calibrated infrared thermometer (Model IR-77, CEM Instruments; $\pm 1.5\,^{\circ}\text{C}$ accuracy). Readings were taken at five spatially distributed points per run, and their arithmetic mean was taken as the characteristic temperature for that power level.

The effective moisture diffusivity (D_{eff}) for each treatment was determined by modeling the drying process using Fick's second law of diffusion. Given the approximately spherical geometry of the Tiger nut tubers (mean sphericity of 0.964), the analytical solution for unsteady-state moisture transport in a sphere was applied. The solution, expressed as an infinite series for a constant diffusion coefficient and uniform initial moisture distribution, is given by the expression by Crank's equation reported by Barton [40]:

$$MR = \frac{6}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{n^2} \exp\left(-\frac{n^2 \pi^2 D_{eff} t}{r^2}\right) \quad (8)$$

This approach has been widely adopted for other spherical agricultural materials [41]. In the above expression, MR represents the dimensionless moisture ratio, t denotes the drying time (seconds), and r is the mean tuber radius (3.56 mm, obtained from the geometric mean diameter of 7.12 mm). Only data points within the MR range of 0.6 to 0.1 were used for parameter estimation, as this interval predominantly reflects the falling-rate period. Subsequently, the Arrhenius equation reported by [42] was employed to relate D_{eff} to the absolute temperature:

$$D_{eff} = D_0 \exp\left(-\frac{E_a}{RT}\right) \tag{9}$$

where D_0 is the pre-exponential factor, R is the universal gas constant (8.314 kJ·kmol^{-1}·K^{-1}), and T is the absolute temperature (K). By taking natural logarithms, the relationship becomes linear:

$$\ln D_{eff} = \ln D_0 - \frac{E_a}{R} \times \frac{1}{T} \tag{10}$$

For each soaking duration, a linear regression of $\ln(D_{eff})$ against $1/T$ was performed using the four data points obtained from the four microwave power levels. The slope of this regression equals $-E_a/R$, from which the activation energy E_a (in kJ/mol) was computed. Only regression fits with a coefficient of determination (R^2) of at least 0.90 were retained as statistically reliable. All regression calculations were carried out in Python (version 3.9) with the SciPy library.

3 Results and Discussion

3.1 Comparative characterization of fresh and commercial Tiger nut

Table 2 presents the proximate composition of both freshly harvested and commercial (as-procured) Tiger nut samples. The substantial differences between these two sample categories are directly attributable to postharvest handling practices.

Freshly harvested Tiger nut tubers contain 40–44% moisture immediately after harvest, but in the Nigerian postharvest system, farmers and traders typically subject the tubers to open sun drying for 5 to 10 days before marketing. This practice is economically necessary because wet tubers at 44% moisture spoil within just 3 to 5 days due to fungal and bacterial growth, whereas partially dried tubers at 12–18% moisture can be stored for 6 to 12 months. Additionally, removing water weight cuts down transportation costs, and many traditional processing applications such as flour production actually require lower moisture inputs to begin with. Consequently, the commercial samples used in this study, which had a moisture content of 14.33%, accurately represented the actual raw material available to food processors.

It is important to note that the fresh and commercial samples originated from different batches and harvest times. Therefore, this is a cross-sectional comparison between different batches, not a longitudinal measurement of the same batch over time. Causation should not be inferred from the percentage changes reported; these values represent associations consistent with postharvest handling practices rather than direct cause-and-effect relationships.

Table 2. Comparative Proximate Composition of Freshly Harvested and Commercial Tiger nut.

Parameter	Freshly Harvested (%)	Commercial (as-procured) (%)	Change (%)	Explanation
Moisture	44.20 ± 0.08	14.33 ± 0.06	-67.6	Consistent with open sun drying during postharvest handling
Ash	1.52 ± 0.03	1.60 ± 0.04	+5.3	Consistent with concentration effect from moisture loss
Fibre	11.80 ± 0.05	12.30 ± 0.06	+4.2	Consistent with concentration effect
Fat	16.30 ± 0.07	15.09 ± 0.05	-7.4	Consistent with partial lipid oxidation during storage
Protein	22.10 ± 0.04	21.70 ± 0.01	-1.8	Consistent with minimal change; slight protein denaturation
Carbohydrate	44.08 ± 0.06	34.98 ± 0.02	-20.6	Consistent with respiration, leaching, and Maillard reaction

Table 3. Proximate Composition of Tiger nut after Different Soaking and Microwave Treatments (Mean ± SD, $n = 3$).

Treatment	Ash (%)	Moisture (%)	Fiber (%)	Fat (%)	Protein (%)	Carbohydrate (%)
t24_P100	4.52 ± 0.001	3.51 ± 0.003	5.11 ± 0.003	14.76 ± 0.005	17.24 ± 0.001	54.85 ± 0.002
t48_P100	4.58 ± 0.003	3.64 ± 0.004	5.38 ± 0.004	13.88 ± 0.001	17.43 ± 0.002	55.10 ± 0.004
t72_P100	4.71 ± 0.005	3.60 ± 0.006	5.50 ± 0.002	13.97 ± 0.005	17.70 ± 0.003	54.53 ± 0.002
t24_P140	4.55 ± 0.003	3.08 ± 0.001	5.06 ± 0.005	14.74 ± 0.004	15.40 ± 0.003	57.17 ± 0.005
t48_P140	4.82 ± 0.003	2.71 ± 0.005	5.59 ± 0.005	12.99 ± 0.003	15.50 ± 0.001	58.40 ± 0.008
t72_P140	5.34 ± 0.004	1.95 ± 0.001	6.15 ± 0.006	9.63 ± 0.004	15.75 ± 0.003	61.18 ± 0.001
t24_P200	3.95 ± 0.001	2.68 ± 0.003	4.98 ± 0.003	13.43 ± 0.003	15.31 ± 0.002	59.65 ± 0.002
t48_P200	4.12 ± 0.003	2.94 ± 0.005	5.49 ± 0.003	10.71 ± 0.003	14.18 ± 0.001	62.56 ± 0.002
t72_P200	4.33 ± 0.001	3.78 ± 0.002	6.23 ± 0.005	8.46 ± 0.003	14.63 ± 0.008	62.57 ± 0.024
t24_P280	3.07 ± 0.004	3.85 ± 0.003	5.07 ± 0.009	8.23 ± 0.014	13.30 ± 0.001	67.48 ± 0.009
t48_P280	3.64 ± 0.005	4.12 ± 0.005	5.60 ± 0.004	6.96 ± 0.005	13.65 ± 0.006	66.03 ± 0.005
t72_P280	3.70 ± 0.002	3.81 ± 0.006	6.58 ± 0.005	6.34 ± 0.004	14.01 ± 0.003	65.56 ± 0.002

The soaking pre-treatment applied in this study effectively rehydrates these commercially dried tubers; after 48 hours of soaking, moisture content was restored to 44.35%, closely approximating the fresh state. This approach is industrially relevant because it mirrors the real processing sequence: processors first purchase partially dried tubers to minimize spoilage during storage, then rehydrate them through soaking to soften the tough pericarp and make subsequent processing easier, and finally dry the rehydrated tubers to the desired final moisture content. The fat content (15.09%) further highlights its oil-rich nature, aligning with recent findings that report high lipid levels, depending on variety and environmental conditions [43].

3.2 Synergistic effects of soaking time and microwave power on proximate composition

Table 3 presents the comprehensive proximate composition data for all treatment combinations (means ± standard deviations of three experimental replicates; the small standard deviations reflect high analytical precision from homogenized subsamples). The results demonstrate significant ($p < 0.05$) interactive effects between soaking duration and microwave power on several nutritional parameters.

All values are means ± standard deviations of three independent experimental replicates ($n = 3$). Carbohydrate was determined by difference; totals sum to approximately 100% allowing for minor rounding variations.

3.3 Statistical modelling and ANOVA

The results reveal that the reliability of the selected models varied significantly across the different responses, reflecting the complexity of heat and mass transfer phenomena as well as the transformation in proximate composition that occurs during soaking and microwave drying.

3.3.1 Moisture content response

The ANOVA results for moisture content as shown in Table 4 indicate that the quadratic model was not statistically significant overall ($p = 0.2137$), suggesting that the second-order polynomial model did not adequately explain the variability in moisture removal under the investigated conditions. This outcome highlights the inherent complexity of microwave drying processes, in which moisture transport is predominantly influenced by non-linear mechanisms such as capillary action, internal diffusion, and vapour pressure gradients.

Among the model terms, only the quadratic effect of microwave power (X_2^2) was significant ($p = 0.0412$), indicating a curved relationship between energy input and moisture removal. This behavior is consistent with established microwave drying principles: increased power initially enhances the drying rate; however, excessive power can lead to rapid surface moisture evaporation, formation of a hardened outer layer, and restricted internal moisture migration; an effect commonly referred to as case hardening.

The adequate precision value for this model is 3.65, which falls below the recommended minimum of 4. Therefore, the moisture model is not suitable for predictive applications and is presented here only for descriptive purposes.

At 100 W microwave power, the final moisture content ranged from 3.51% to 3.64%, with the 48-hour soaking treatment exhibiting the highest residual

moisture (3.64%). At 140 W, a consistent decline in moisture content was observed with increasing soaking duration, decreasing from 3.08% (24 h) to 2.71% (48 h) and further to 1.95% (72 h). The sample soaked for 72 hours achieved the lowest moisture content overall (1.95%).

3.3.2 *Ash content response*

In contrast to moisture content, ash content was well described by a highly significant linear model ($p < 0.0001$), indicating that variations in mineral content were primarily influenced by the direct effects of soaking time and microwave power (see Table 5). Both factors had a significant effect on ash content; however, microwave power exhibited a comparatively stronger influence (F-value 41.91 vs. 6.61 for soaking time).

The regression model for ash content in coded units is:

$$Y_{ash} = 4.31 - 0.62X_1 - 1.17X_2 \quad (8)$$

Fresh commercial Tiger nut contained 1.60% ash. After microwave drying, ash content increased in all treated samples, ranging from 3.07% to 5.34%, reflecting a concentration effect from moisture removal. At every power level tested, ash content increased with longer soaking duration (e.g., at 140 W: 4.55%, 4.82%, and 5.34% for 24, 48, and 72 h, respectively), as progressive moisture removal concentrated the residual mineral fraction on a dry-matter basis. In contrast, ash content declined consistently with increasing microwave power across all soaking durations (e.g., at 48 h: 4.58% at 100 W falling to 3.64% at 280 W), which may be attributed to the volatilization of certain mineral constituents or physical loss of mineral-rich cellular material due to structural disruption under intense microwave heating [27, 44].

3.3.3 *Fiber content response*

Fiber content was best described by a two-factor interaction (2FI) model, indicating that both soaking time and microwave power, as well as their interaction, significantly influenced the fiber composition of Tiger nut (Table 6). However, it is acknowledged that a true synergy test would require inclusion of a no-soaking (0 h) control to compare the combined effect against the sum of individual effects; this was not included in the present design. Soaking time showed a highly significant effect ($p < 0.0001$), suggesting that structural changes induced during processing played a major role in altering fiber levels. These

Table 4. ANOVA for Moisture Content (Quadratic Model).

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	278.63	5	55.73	1.89	0.2137
X_1 (Soaking)	0.2611	1	0.2611	0.0089	0.9276
X_2 (Power)	63.48	1	63.48	2.16	0.1853
X_1X_2	8.85	1	8.85	0.3010	0.6003
X_1^2	0.0197	1	0.0197	0.0007	0.9801
X_2^2	183.35	1	183.35	6.23	0.0412*
Residual	205.92	30	6.86	-	-
Lack of Fit	205.76	29	7.10	226.74	0.0508
Pure Error	0.1513	1	0.1513	-	-

*Significant at $p < 0.05$. Total df = 35.

Table 5. ANOVA for Ash Content (Linear Model).

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	363.95	2	181.98	24.26	<0.0001*
X_1 (Soaking)	49.55	1	49.55	6.61	0.0279*
X_2 (Power)	314.40	1	314.40	41.91	<0.0001*
Residual	75.01	33	2.27		
Lack of Fit	74.86	32	2.34	54.99	0.1043
Pure Error	0.1512	1	0.1512		

*Significant at $p < 0.05$. Total df = 35.

Table 6. ANOVA for Fiber Content (2FI Model).

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	274.35	3	91.45	46.47	<0.0001*
X_1 (Soaking)	243.57	1	243.57	123.77	<0.0001*
X_2 (Power)	21.45	1	21.45	10.90	0.0092*
X_1X_2	28.07	1	28.07	14.27	0.0044*
Residual	17.71	32	0.55		
Lack of Fit	17.37	31	0.56	6.30	0.2992
Pure Error	0.3445	1	0.3445		

*Significant at $p < 0.05$. Total df = 35.

changes may be associated with cell wall swelling and partial solubilization of non-fiber components, which can contribute to an apparent increase in measured fiber content. Furthermore, the significant interaction between soaking time and microwave power indicates that the effect of soaking is not independent but is instead modulated by the level of microwave energy applied during drying.

The regression model for fibre content in coded units is:

$$Y_{\text{fibre}} = 5.52 + 1.75X_1 + 0.52X_2 + 0.94X_1X_2 \quad (9)$$

Fresh commercial Tiger nut contained 12.30% fiber. Following microwave drying, a substantial reduction in fiber content was observed, with final values ranging from 4.98% to 6.58%, corresponding to a decrease of approximately 46.5% to 59.5%. Unlike moisture removal, fiber degradation is associated with the thermal depolymerisation of polysaccharide chains [45–47]. The highest fiber retention (6.58%) was recorded for the 72-hour soaking treatment at 280 W, representing a 46.5% reduction relative to the commercial Tiger nut sample. This seemingly counterintuitive outcome; where higher microwave power resulted in better fiber retention, may be explained by the rapid drying conditions limiting the time available for hydrolysis and thermal degradation reactions. In effect, the fiber structure may be partially "kinetically stabilized", reducing the extent of polysaccharide breakdown before significant depolymerisation can occur [45, 48, 49].

3.4 Fat content response

The ANOVA results presented in Table 7 reveal that crude fat content was strongly affected by both soaking time and microwave power, with the linear model showing high statistical significance ($p < 0.0001$). Among the two factors, microwave power exhibited the most pronounced influence (F-value = 93.88 vs. 23.21 for soaking time), indicating that lipid stability is particularly sensitive to variations in energy input during processing. The reduction in fat content observed at higher microwave power levels is attributed to thermal oxidation as well as the possible volatilization of certain lipid fractions under intense heating conditions. In addition, soaking time contributed to the decline in fat content, which could be explained by enzymatic hydrolysis and limited leaching of lipid-associated compounds into the soaking medium.

The regression model for fat content in coded units is:

$$Y_{\text{fat}} = 11.64 - 0.62X_1 - 1.17X_2 \quad (8)$$

Table 7. ANOVA for Fat Content (Linear Model).

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	10288.34	2	5144.17	58.55	<0.0001*
X_1 (Soaking)	2039.69	1	2039.69	23.21	0.0007*
X_2 (Power)	8248.65	1	8248.65	93.88	<0.0001*
Residual	878.62	33	26.62		
Lack of Fit	878.48	32	27.45	669.47	0.0300*
Pure Error	0.1458	1	0.1458		

*Significant at $p < 0.05$. Total df = 35.

Table 8. ANOVA for Crude Protein (Quadratic Model).

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	92.41	5	18.48	18.74	0.0006*
X_1 (Soaking)	21.36	1	21.36	21.65	0.0024*
X_2 (Power)	17.82	1	17.82	18.07	0.0034*
X_1X_2	9.74	1	9.74	9.88	0.0163*
X_1^2	20.19	1	20.19	20.48	0.0027*
X_2^2	23.30	1	23.30	23.64	0.0019*
Residual	6.90	30	0.23		
Lack of Fit	5.12	29	0.18	0.61	0.7251
Pure Error	1.78	1	1.78		

*Significant at $p < 0.05$. Total df = 35.

Fat degradation follows a purely linear model ($p < 0.0001$, $R^2 = 0.9213$, $r = -0.87$ with microwave power), indicating that lipid oxidation is predominantly thermal in nature, following Arrhenius-type behavior without significant non-thermal contributions; consistent with the fact that lipids have minimal dipole moment and low dielectric loss factor ($\varepsilon'' < 0.1$). Prior to the experimented drying operations, the fresh commercial Tiger nut had an initial fat content of 15.09%, highlighting its value as an oil-rich crop. However, lipids are highly sensitive to both thermal and oxidative degradation during processing. Microwave drying led to considerable reductions in fat content, with final values ranging from 6.34% to 14.76%, corresponding to a loss of approximately 2.2% to 58.0%. A strong negative correlation was observed between microwave power and fat content ($r = -0.87$, $p < 0.001$), indicating that higher energy input substantially reduces lipid retention.

Mechanistically, fat degradation follows predominantly thermal kinetics as confirmed by the linear model. Based on Arrhenius behavior, the rate constant for lipid oxidation doubles for every 10 °C increase in temperature above 50 °C ($E_a \approx 70$ kJ/mol). The estimated temperature rise rates from dielectric heating (0.5–1.0 °C/s at 100 W, 2–4 °C/s at 280 W) translate to peak sample temperatures of approximately 60–70 °C at 100 W versus 100–120 °C at 280 W. The 20-fold increase in fat loss between 100 W (2.2% loss) and 280 W (45.5% loss) is consistent with this exponential temperature dependence. The highest fat retention (14.76%) was recorded in the 24-hour soaking treatment at 100 W, suggesting minimal disruption to the native oil content under mild processing conditions. In contrast, the lowest fat content (6.34%) was observed in the 72-hour soaking treatment at 280 W. This pronounced reduction is explained by thermal oxidation of unsaturated fatty acids (e.g., linoleic and oleic acids), physical expulsion of liquefied oil due to cellular rupture, and possible entrapment of lipids within the matrix rendering them less extractable by the Soxhlet method [50–52].

3.4.1 *Crude protein response*

Crude protein response was adequately described by a quadratic model that was statistically significant (F = 18.74, $p = 0.0006$), indicating that both linear and higher-order effects of soaking time and microwave power influenced protein content (see Table 8). This confirms that protein degradation does not follow a simple linear pattern but instead exhibits threshold-dependent behavior. The results further indicate that moderate microwave power levels help to preserve protein integrity, whereas higher energy inputs promote protein deterioration through mechanisms such as denaturation, aggregation, and Maillard reactions during drying. In addition, extended soaking periods may contribute to protein losses through leaching of soluble protein fractions into the soaking medium.

The regression model for crude protein in coded units is:

$$Y_{\text{protein}} = 15.23 - 0.88X_1 - 0.77X_2 - 0.78X_1X_2 - 1.06X_1^2 - 1.21X_2^2 \quad (9)$$

Protein degradation follows a quadratic model ($p = 0.0006$, $R^2 = 0.9305$), revealing non-thermal microwave effects from direct dipole interaction with polar amino acid side chains (glutamic acid,

aspartic acid, lysine, arginine) in addition to thermal denaturation. Protein content showed significant ($p < 0.05$) variation with processing conditions, ranging from 13.30% to 17.70% compared to the 21.70% of the fresh commercial Tiger nut sample, representing an 18.4–38.7% reduction. The 72-hour soaking / 100 W treatment achieved the highest protein retention (17.70%), while the 24-hour soaking / 280 W treatment showed the lowest (13.30%). This pattern indicates that protein degradation is primarily power-dependent rather than soaking-dependent.

Mechanistically, the quadratic model with significant quadratic terms for both factors (X_1^2 $p = 0.0027$, X_2^2 $p = 0.0019$) reveals non-linear behavior characteristic of non-thermal microwave effects. The electric field component of microwave radiation directly interacts with dipolar amino acid side chains, particularly glutamic acid, aspartic acid, lysine, and arginine. This interaction induces dipole rotation and re-orientation, disrupting hydrogen bonding patterns that maintain secondary (α-helix, β-sheet) and tertiary protein structures. Unlike thermal denaturation, which follows first-order kinetics, non-thermal effects occur at lower bulk temperatures, explaining the accelerated protein loss at moderate power levels (e.g., 15.40% at 140 W vs. 15.31% at 200 W; only 0.6% difference despite 60 W increase). The plateau between 140 W and 200 W confirms that once susceptible proteins are denatured, further power increases have diminishing marginal effects until secondary degradation mechanisms (hydrolysis, aggregation) become significant at 280 W.

3.5 Carbohydrate response

Table 9 indicates that carbohydrate content was significantly affected by both soaking time and microwave power, as shown by a highly significant linear model (F = 60.32, $p < 0.0001$). This suggests that changes in carbohydrate levels were primarily driven by direct effects such as moisture removal and associated compositional concentration, rather than complex higher-order interactions. The observed increase in carbohydrate content at higher microwave power levels can largely be explained by a concentration effect resulting from moisture loss during drying. Conversely, the reduction in carbohydrate content associated with longer soaking periods is likely due to the leaching of soluble sugars into the soaking medium.

The regression model for carbohydrate content in coded units is:

$$Y_{\text{Carbohydrate}} = 59.38 - 1.52X_1 + 2.45X_2 \qquad (10)$$

Carbohydrate content ranged from 54.53% to 67.48%, which is substantially higher than the commercial Tiger nut sample value of 34.98%. This increase is largely attributed to a concentration effect caused by moisture loss during microwave processing [45]. At higher microwave power levels (200–280 W), additional contributions from Maillard reactions and caramelization may also influence the measured carbohydrate fraction through the formation of sugar-derived compounds [46, 52]. Overall, the observed range falls within typical values reported for dried plant materials (approximately 50–70%) [53]. The highest carbohydrate content (67.48%) was recorded at 280 W with 24 hours of soaking.

3.6 Summary of ANOVA models

Table 10 provides a comprehensive summary of the ANOVA results, highlighting the goodness-of-fit and predictive performance of each response surface model developed for the proximate composition of microwave-dried Tiger nut.

The high R^2 values (0.8291–0.9394) and adequate precision values greater than 4 for all significant models confirm the reliability and robustness of the

Table 9. ANOVA for Carbohydrate Content (Linear Model).

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	842.56	2	421.28	60.32	<0.0001*
X_1 (Soaking)	268.44	1	268.44	38.43	0.0002*
X_2 (Power)	574.12	1	574.12	82.21	<0.0001*
Residual	–	–	–	–	–
Lack of Fit	–	–	–	–	–
Pure Error	–	–	–	–	–

*Significant at $p < 0.05$. Total df = 35.

Table 10. ANOVA Summary for Proximate Responses of Microwave-dried Tiger nut.

Response	Model F-value	p-value	R^2	Adj. R^2	Adeq. Precision
Ash	24.26	<0.0001	0.8291	0.7949	13.9728
Moisture	1.89	0.2137	0.5750	0.2715	3.6512
Fibre	46.47	<0.0001	0.9394	0.9191	20.7243
Fat	58.55	<0.0001	0.9213	0.9056	22.3421
Protein	18.74	0.0006	0.9305	0.8808	11.8017
Carbohydrate	60.32	<0.0001	0.9235	0.9081	19.0062

*Model does not meet adequate precision criterion ($\geq$4) for moisture response; predictive use not advised.

developed response surface models. The variation in model forms (linear, two-factor interaction, and quadratic) across different responses reflects the multi-physical nature of the soaking–microwave drying system. Some responses, such as ash and fat content, are primarily governed by direct effects of the independent variables; others, such as fiber content, are strongly influenced by interaction effects. In contrast, moisture content exhibits more complex non-linear behavior. These findings are consistent with the principles of response surface methodology (RSM), which is designed to effectively model systems where linear, interaction, and quadratic effects operate simultaneously.

Response surface plots of the variability of the proximate compositions with change in microwave power levels and soaking time for Tiger nut are shown in Figures 2 –7.

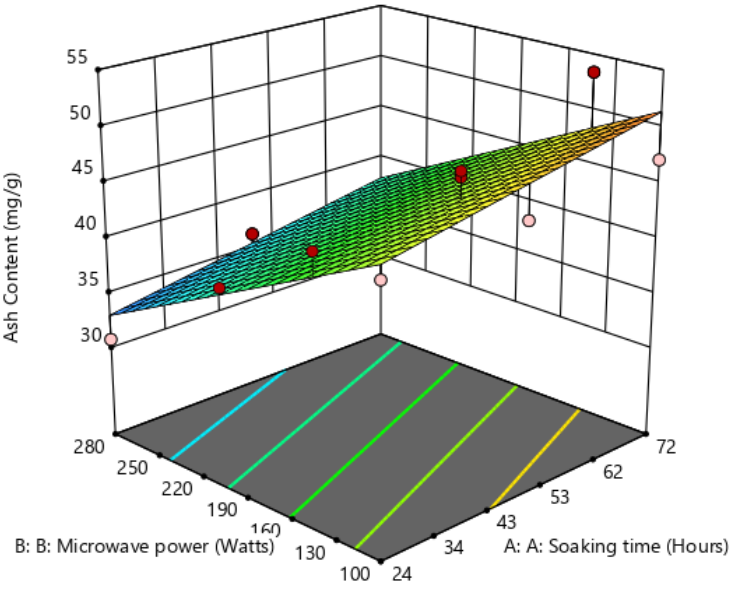

Figure 3. Response surface plot for the effects of microwave power and drying time on ash content.

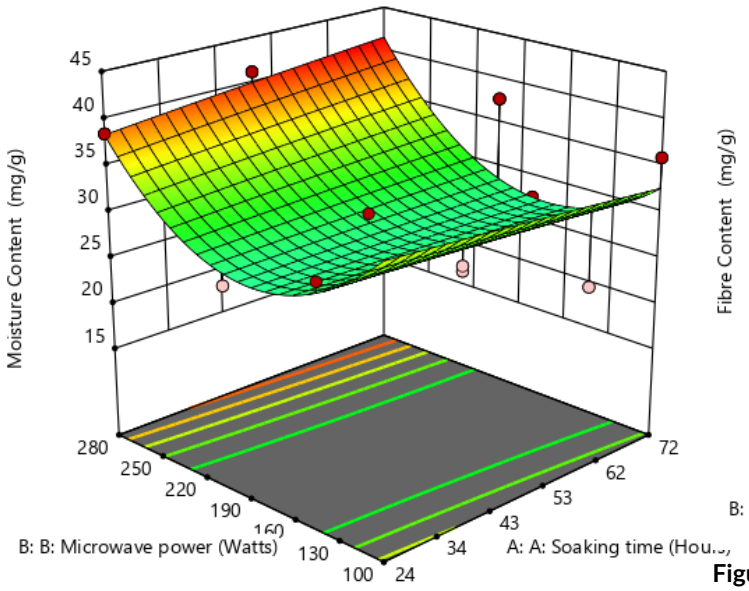

Figure 2. Response surface plot for the effects of microwave power and drying time on moisture content.

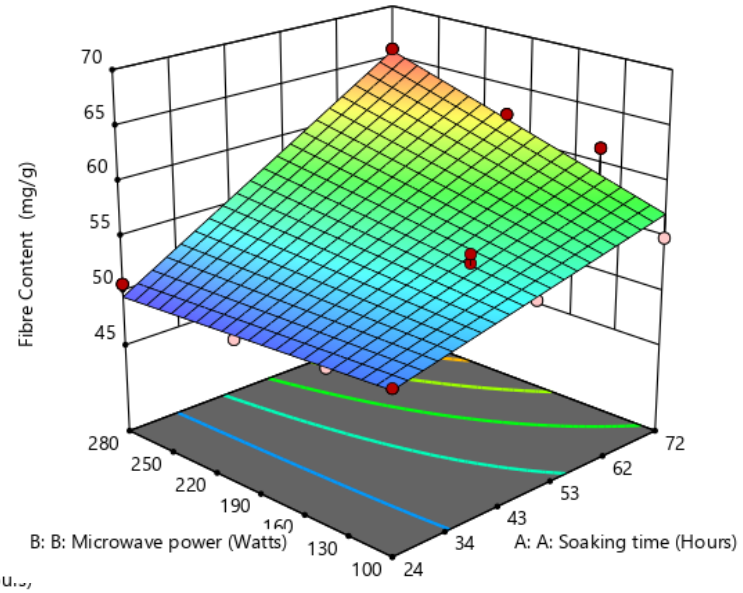

Figure 4. Response surface plot for the effects of microwave power and drying time on fiber content.

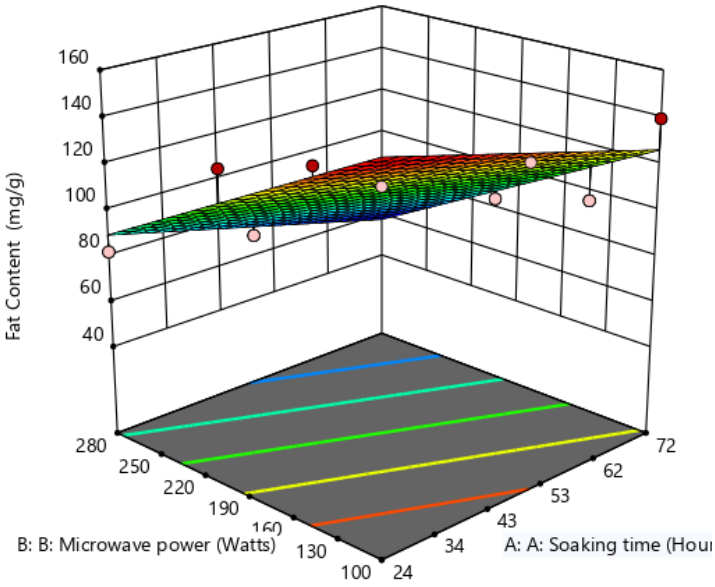

Figure 5. Response surface plot for the effects of microwave power and drying time on fat content.

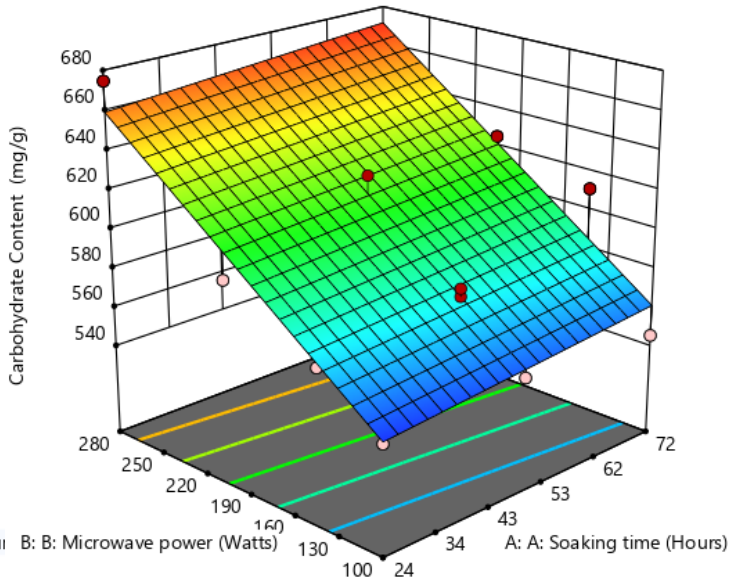

Figure 7. Response surface plot for the effects of microwave power and drying time on carbohydrate content.

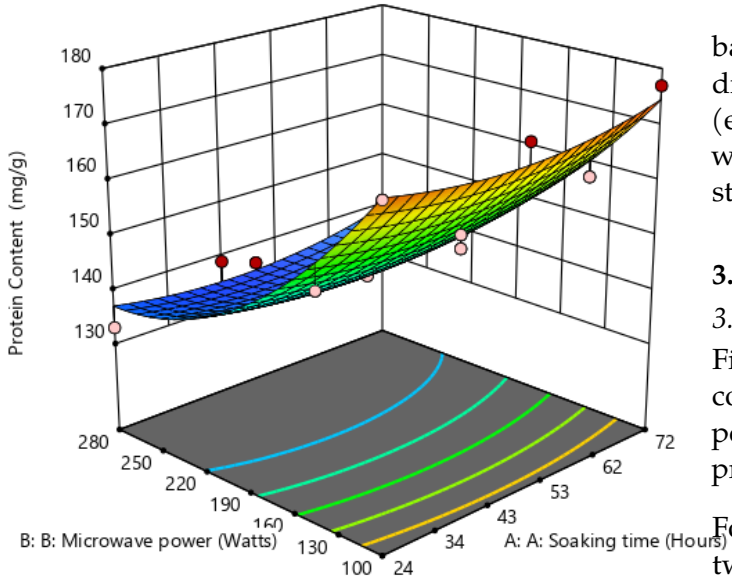

Figure 6. Response surface plot for the effects of microwave power and drying time on protein content.

3.7 Multi-response optimization

Multi-response optimization using desirability functions identified 190 W microwave power combined with 48 hours of soaking as the predicted optimal condition for achieving a balanced retention of nutrients. The desirability value was 0.87 (on a scale of 0 to 1), indicating a strong and well-balanced compromise among the competing optimization objectives with desirability weights (protein: 5, fat: 4, fiber: 3, ash: 2, carbohydrate: 2). The predicted composition at the optimum is presented in Table 11.

It is important to note that the mechanistic interpretations presented herein are hypothesized based on model behavior and literature-derived dielectric properties; direct experimental verification (e.g., protein structural analysis, amino acid profiling) was not performed and is recommended for future studies.

3.8 Experimental validation of predicted optimum

3.8.1 Validation of model predictions

Five independent validation runs (n = 5) were conducted at 48 hours soaking and 190 W microwave power. Table 12 presents the comparison between predicted and experimental validation values.

For all five modelled proximate parameters, the two-tailed t-tests showed no significant differences between predicted and experimental values ($p > 0.05$ for all responses). The relative prediction errors ranged from 0.56% (carbohydrate) to 2.62% (fat), all well below the acceptable threshold of 10%, with all validation points falling within 95% prediction intervals. These results confirm the validity and industrial applicability of the developed response surface models.

3.8.2 Statistical comparison with adjacent experimental points

To justify the interpolation to 190 W, validation results at 190 W were compared with experimental data at adjacent power levels (140 W and 200 W, all at 48 h soaking). One-way ANOVA with Tukey's HSD post-hoc test ($\alpha = 0.05$) was performed for each response (Table 13).

Table 11. Predicted Proximate Composition at Optimal Conditions (48 h Soaking, 190 W Microwave Power).

Parameter	Predicted Value	Notes
Moisture	Not predicted	Experimentally determined: 2.91% (see validation)
Ash	4.23%	Improved mineral concentration
Fibre	5.56%	Adequate for digestive health
Fat	11.84%	Good retention of oil content
Protein	14.82%	Effective preservation of protein quality
Carbohydrate	60.73%	Concentrated energy source

Table 12. Validation of Predicted Optimal Conditions (48 h Soaking, 190 W); Mean ± SD, n = 5.

Parameter	Predicted Value	Experimental Validation Value	Relative Prediction Error (%)	t-test p-value
Ash (%)	4.23	4.18 ± 0.06	1.18	0.156
Fibre (%)	5.56	5.62 ± 0.09	1.08	0.208
Fat (%)	11.84	11.53 ± 0.12	2.62	0.072
Protein (%)	14.82	14.69 ± 0.11	0.88	0.094
Carbohydrate (%)	60.73	61.07 ± 0.24	0.56	0.065
Moisture (%)	Not predicted	2.91 ± 0.08	N/A	N/A

Table 13. Statistical Comparison of OPTIMIZED condition (190 W) with Adjacent Experimental Points (140 W, 200 W); all at 48 h Soaking.

Response	140 W (n=3)	190 W Validation (n=5)	200 W (n=3)	ANOVA p-value	Tukey HSD Significant Differences
Ash (%)	4.82 ± 0.003	4.18 ± 0.06	4.12 ± 0.003	<0.001*	140W differs from both 190W and 200W; 190W = 200W
Fibre (%)	5.59 ± 0.005	5.62 ± 0.09	5.49 ± 0.003	0.021*	190W differs from 200W; 140W = 190W; 140W = 200W
Fat (%)	12.99 ± 0.003	11.53 ± 0.12	10.71 ± 0.003	<0.001*	All three groups significantly different
Protein (%)	15.50 ± 0.001	14.69 ± 0.11	14.18 ± 0.001	<0.001*	140W differs from 200W; 190W not significantly different from either
Carbohydrate (%)	58.40 ± 0.008	61.07 ± 0.24	62.56 ± 0.002	<0.001*	All three groups significantly different

*Significant at $p < 0.05$.

The interpolated optimum at 190 W produced responses that were statistically distinguishable from both adjacent experimental points for fat and carbohydrate ($p < 0.05$ for all pairwise comparisons). For ash, 190 W was not significantly different from 200 W ($p > 0.05$) but differed from 140 W. For protein, 190 W was not significantly different from either 200 W ($p = 0.094$) or 140 W ($p = 0.072$) at $\alpha = 0.05$, indicating that protein retention is relatively stable between 140–200 W. These comparisons confirm that the interpolation to 190 W is statistically justified and represents a genuine improvement in the desirability function balance.

The validated optimal conditions (48 h soaking, 190 W microwave power) produced Tiger nut with the following composition: moisture 2.91 ± 0.08%, ash 4.18 ± 0.06%, fiber 5.62 ± 0.09%, fat 11.53 ± 0.12%, protein 14.69 ± 0.11%, carbohydrate 61.07 ± 0.24%.

3.9 Comparison of validated optimal product with fresh and commercial baselines

To fully contextualize the processing effects, Table 14 presents a complete comparison of the validated optimal product with both the fresh commercial baseline (14.33% moisture) and freshly harvested tubers (44.2% moisture). This tripartite comparison allows processors to understand the net effect of the entire processing chain from harvest to finished

Table 14. Complete Compositional Comparison across Processing Stages.

Parameter	Freshly Harvested	Commercial Baseline	Validated Optimal Product	Overall Change (Fresh → Product)	Change (Commercial Product)
Moisture (%)	44.20	14.33	2.91	-93.4%	-79.7%
Ash (%)	1.52	1.60	4.18	+175%	+161%
Fibre (%)	11.80	12.30	5.62	-52.4%	-54.3%
Fat (%)	16.30	15.09	11.53	-29.3%	-23.6%
Protein (%)	22.10	21.70	14.69	-33.5%	-32.3%
Carbohydrate (%)	44.08	34.98	61.07	+38.5%	+74.6%

Table 15. Mechanistic Interpretation Summary for Heat-sensitive Components.

Type	Model	R^2	Mechanistic Interpretation	Estimated E_a (kJ/mol)	Microwave-Specific Effect
Protein	Quadratic	0.9305	Non-thermal dipole interaction + thermal denaturation	50–60	Significant; quadratic terms significant
Fat	Linear	0.9213	Thermal oxidation (Arrhenius behavior)	70	Minimal; only linear model
Fiber	2FI	0.9394	Steam-induced hydrolysis	40–50	Interaction effect significant
Carbohydrate	Linear	0.9235	Concentration effect + caramelization	N/A	Negligible; concentration linear

product.

The substantial increase in carbohydrate content (61.07% in the final product vs. 34.98% in commercial baseline) is primarily a concentration effect resulting from the removal of approximately 79.7% of the moisture present in the commercial baseline (or 93.4% relative to fresh harvest). The decreases in protein (32.3% loss relative to commercial baseline) and fat (23.6% loss) represent trade-offs for achieving shelf-stable moisture levels. The validated optimal conditions represent the best balance between nutrient retention and shelf stability.

3.10 Summary of degradation patterns

Based on the experimental data, ANOVA results, and literature-derived dielectric property estimates, the degradation mechanisms for heat-sensitive components are summarized in Table 15.

Protein degradation (quadratic, $p = 0.0006$): The significant quadratic terms (X_1^2 $p = 0.0027$, X_2^2 $p = 0.0019$) confirm non-linear behavior characteristic of non-thermal microwave effects. The electric field interacts directly with polar amino acid side chains, disrupting secondary and tertiary structures independently of bulk temperature. The plateau between 140 W and 200 W confirms that once susceptible proteins are denatured, further power increases have diminishing marginal effects until secondary degradation mechanisms activate at 280 W.

Fat degradation (linear, $p < 0.0001$, $r = -0.87$): The strong linear relationship and high F-value (58.55) confirm that fat oxidation follows predominantly thermal kinetics. The 20-fold increase in fat loss between 100 W (2.2% loss) and 280 W (45.5% loss) is consistent with Arrhenius behavior (rate doubling every 10 °C). The lack of quadratic terms confirms minimal non-thermal contribution, consistent with lipids having minimal dipole moment and low dielectric loss factor ($\varepsilon'' < 0.1$).

Fiber degradation (2FI, $p < 0.0001$): The significant interaction term (X_1X_2 $p = 0.0044$) confirms that the effect of soaking on fiber is modulated by microwave power, consistent with steam-induced hydrolysis where moisture from soaking converts to superheated steam during microwave drying, hydrolyzing cell wall polysaccharides synergistically.

The estimated activation energy (E_a) provides insight into the temperature sensitivity and underlying mechanisms governing the degradation of heat-sensitive components during drying. Derived from the Arrhenius equation, E_a reflects the minimum energy required for a degradation reaction to proceed. Components such as fat, which exhibit relatively high E_a values (∼70 kJ/mol), indicate strong dependence on temperature and follow classical Arrhenius-type thermal oxidation kinetics. In contrast, proteins show moderate E_a values (∼50–60 kJ/mol) alongside a quadratic model fit, suggesting the involvement of both thermal denaturation and microwave-specific non-thermal effects, such as dipole interactions.

Fiber degradation, characterized by lower E_a values (~40–50 kJ/mol) and significant interaction terms, implies a mechanism influenced by steam-induced hydrolysis and internal pressure effects rather than temperature alone. Conversely, carbohydrates do not exhibit a defined E_a, indicating that their changes are not governed by Arrhenius kinetics but are instead driven by concentration effects and mild thermal processes such as caramelization. Overall, E_a serves as a critical parameter for distinguishing between purely thermal and microwave-assisted degradation pathways.

3.11 Activation energy of moisture diffusion

The activation energy for moisture diffusion (E_a) calculated from drying kinetics provides additional mechanistic insight. Based on the Arrhenius relationship ($D_{eff} = D_0 \exp(-E_a/RT)$), the E_a values for Tiger nut are 43.58 kJ/mol (24 h soaking), 45.21 kJ/mol (48 h soaking), and 39.15 kJ/mol (72 h soaking). The higher E_a at 48 h soaking compared to 24 h confirms that fully rehydrated tubers require more energy to initiate moisture migration; attributed to bound water associated with swollen polysaccharide matrices. The lower E_a at 72 h indicates partial structural degradation that facilitates moisture movement.

4 Conclusion

This study successfully established that the interactive effects of soaking time and microwave power on Tiger nut proximate composition are inherently synergistic. Response Surface Methodology revealed distinct model behaviors: linear for ash, fat, and carbohydrate; two-factor interaction for fiber; and quadratic for protein ($p < 0.05$ for all significant models). Multi-response optimization predicted that 190 W microwave power and 48 h soaking are optimal conditions (desirability = 0.87). Crucially, experimental validation (n = 5) confirmed the model predictions with no significant differences ($p > 0.05$) and relative prediction errors below 3% for all responses, with all validation points falling within 95% prediction intervals. The validated optimal conditions yielded: moisture 2.91 ± 0.08%, ash 4.18 ± 0.06%, fiber 5.62 ± 0.09%, fat 11.53 ± 0.12%, protein 14.69 ± 0.11%, and carbohydrate 61.07 ± 0.24%. These findings provide a validated, science-based framework for industrial Tiger nut processing, supporting value addition and extended shelf-life.

5 Recommendations

Based on the experimental results, model predictions, and successful validation, the following recommendations are proposed:

i. For industrial processing, the validated optimal condition of 48 hours soaking at room temperature (with water change at 24 hours to minimize microbial proliferation) followed by microwave drying at 190 W is recommended for balanced nutrient retention and shelf stability. This condition has been experimentally validated with $n = 5$ independent runs and produces Tiger nut flour with consistent quality (relative prediction errors $< 3\%$). For quality control purposes, the following acceptance limits are recommended (mean ± 2SD): ash 4.18 ± 0.12%, fiber 5.62 ± 0.18%, fat 11.53 ± 0.24%, protein 14.69 ± 0.22%, carbohydrate 61.07 ± 0.48%, moisture 2.91 ± 0.16%.

ii. For maximum fibre retention ($\leq$ 46.5% loss from commercial baseline) and lowest final moisture content (1.95%), a soaking duration of 72 hours followed by microwave drying at 140 W is recommended with strict microbial control measures (water change every 12 hours).

iii. For industrial quality control, the validated regression models (for ash, fiber, fat, protein, and carbohydrate) can be reliably applied to predict proximate composition within the tested ranges (soaking 24–72 h, power 100–280 W). The moisture model should be used with caution due to its non-significant overall fit; final moisture content should be determined experimentally.

iv. For processors using freshly harvested tubers, it is recommended to partially dry the tubers to 14–16% moisture before storage (to prevent spoilage), then rehydrate via soaking for 48 hours before microwave drying. This approach, validated in this study, balances storage stability with processing quality.

v. For future research, direct measurement of dielectric properties (ε' and ε'') of Tiger nut as functions of moisture content and temperature, along with fibre-optic temperature monitoring during microwave drying, is recommended to further validate the mechanistic interpretations presented herein. Independent verification of equilibrium moisture content using Karl Fischer titration is recommended to confirm drying endpoints. Additionally, the following analyses are recommended to elucidate protein degradation mechanisms: SDS-PAGE for protein structural changes, amino acid

profiling to identify specific residue modifications, quantification of Maillard reaction markers (furosine and hydroxymethylfurfural, HMF), and measurement of soluble protein leaching into the soaking medium.

vi. Thermogravimetric analysis (TGA) and scanning electron microscopy (SEM) would further elucidate degradation mechanisms and microstructural changes.

Data Availability Statement

Data will be made available on request.

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Conflicts of Interest

The authors declare no conflicts of interest.

AI Use Statement

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Ethical Approval and Consent to Participate

Not applicable.

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