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An Assessment of the Effects of Different Soaking Pretreatments and Microwave Drying Conditions on Selected Phytochemical Properties of Tiger-nut (*Cyperus esculentus*)

Anyene, C. C. C.[1*], Nwajinka, C. O.[2], Nsofor, P. S.[3], Okpala, C. D.[4] and Ogbuagu, N. J.[5]

[1] Department of Agricultural and Bioenvironmental Engineering, Federal Ploytechnic, Oko, Anmabra State
[2]Department of Agricultural and Bioresources Engineering, Nnamdi Azikiwe University, Awka, Anambra State
[3] Department of Agricultural and Bioenvironmental Engineering, Federal Ploytechnic, Oko, Anmabra State
[4]Department of Agricultural and Bioresources Engineering, Nnamdi Azikiwe University, Awka, Anambra State
[5]Department of Agricultural and Bioresources Engineering, Nnamdi Azikiwe University, Awka, Anambra State

[*]Corresponding Author's E-mail: edunkpaanyene@gmail.com

Abstract
The effects of soaking pretreatments and microwave drying conditions on the phytochemical properties of Tiger-nut were assessed. A two factor, four level factorial designs in a completely randomized design was adopted for the study and the Design Expert 13 software was used to establish the experimental matrix for the study. Tiger-nut samples were soaked for 24 hr, 48 hr and 72 hr at room temperature and dried at 100 W, 140 W, 200 W and 280 W microwave powers until constant weight was attained. The resulting samples were reduced and phytochemical contents analyses were carried out on them using standard laboratory protocols. From the results it was observed that the pretreatments and drying conditions caused reduction in moisture content of Tiger-nut to safe storage levels and variation in the drying characteristics (drying rates, drying time, and equilibrium moisture. The drying rates and drying time increased with increase in microwave power. The highest drying rates obtained at 100W microwave power were 0.161 gH_2O/min for the 24hr soaked sample, 0.232 gH_2O/min for the sample soaked for 48 hr and 0.158 gH_2O/min for the sample soaked for 72 hr. The peak drying rates observed at 140 W microwave power were 0.325 gH_2O/min for 24 hr soaking pretreatment, 0.365 gH_2O/min for 48 hr soaking pretreatment and 0.449 gH_2O/min for the 72 hr soaking pretreatment. The peak drying rates obtained at 200 W microwave power were 0.443 gH_2O/min for samples soaked for 24hr, 0.323 gH_2O/min for sample soaked for 48 hr, and 0.323 gH_2O/min for sample soaked for 72 hr. The peak drying rates gotten at 280 W microwave power were 1.018 gH_2O/min for sample soaked for 24hr, 0.938 gH_2O/min for sample soaked for 48 hr, and 0.938 gH_2O/min for sample soaked for 72 hr. The mean drying duration at which EMC was attained for 100 W, 140 W, 200 W and 280 W microwave power dried Tiger-nut was 580 min, 330 min, 310 min and 130 min respectively. The ANOVA of the phytochemical contents showed that at $P < 0.05$, soaking pretreatments and microwave drying conditions caused significant alterations on the phytochemical properties of the Tiger-nut samples. There was a decrease in the flavonoids, alkaloids, tannin, and oxalate contents and a slight increase in the cardiac glycoside and phenol contents of the pretreated and dried samples when compared with the result obtained for the untreated sample (control).

Keywords: Phytochemical, Tiger-nut, microwave power, soaking time, pre-treatment

1. Introduction

Tiger-nut (*Cyperus esculentus*) belongs to the kingdom *plantae*, phylum *tracheophyta*, class *liliopsida*, order *cyperales* and family of *cyperaceace*, and was originally discovered in tombs located in Egypt over 4000 years ago (Kizzie-Hayford *et al.*, 2021; Malashree *et al*., 2021). It is an edible perennial nut-like C4 plant of the tropical and Mediterranean zones of the world

(Bando *et al.*, 2020) that is commonly referred to as yellow nut sedge, earth almond, chufa and Zulu nuts (Omale *et al*., 2020). The plant has a fibrous (rhizomatous) root system which spread to produce secondary shoots. Edible 8 mm to 16mm oblong to spherical tubers are developed and attached to the spread rhizomatous roots (Figure 1). Irrespective of the variety, young Tiger-nut tubers have whitish coloration and transform to either black, brown, yellow, etc., following their variety trait (Henry *et al.*, 2021; Idoia-Codina *et al*., 2014; Malashree *et al*., 2021). Preferably, the yellow variety is highly sought over the black and brown varieties as a domestic and industrial resource due to its fresher pericarp, attractive color and bigger size attributes.

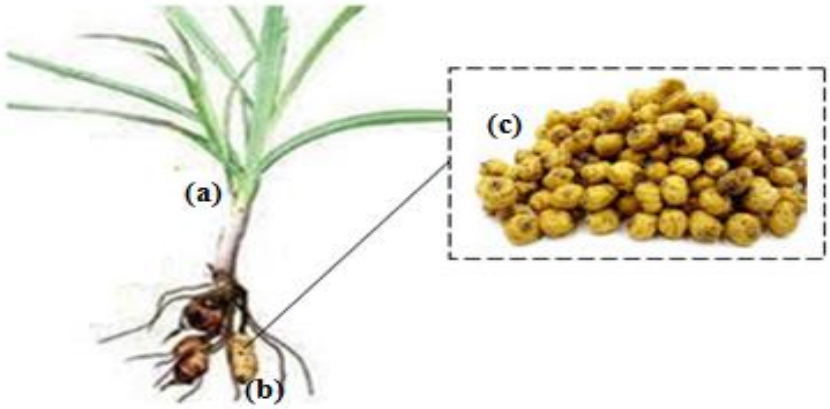

Figure 1: Tiger-nut plant: (a) shoot system (b) unharvested matured tubers (c) harvested and cleaned tubers. (Source: Bazine and Arslanoglu, 2020, *adapted*)

In the local markets, Tiger-nut is sold as either fresh tuber after soaking it for range of hr or drying the tubers (Fabunmi *et al*., 2016). Tiger-nut tubers are usually classified into small Tiger-nut of diameter 6 mm – 7 mm, standard Tiger-nut of diameter between 8 mm – 11 mm and large Tiger-nut of diameter 12 mm – 16 mm (Malashree *et al*., 2021). Due to the larger size feature of the yellow variety, it is highly sought for consumption as snack, indirect application as a raw material for the production of highly sweet and nutritional food products and drinks, as well as good animal feed resource (Asare *et al*., 2020; Djomdi *et al*., 2022; El-nagger, 2016; Martín-Esparza and González-Martínez, 2016; Onuoha *et al*., 2017; Yu *et al*., 2022).

According to Abdullahi *et al*. (2022), Tiger-nut plant resources present good health potentials due to the presence of these phytochemicals. Phytochemicals are bioactive compounds that are usually contained in plant resources and foster several health benefits. There are over 4000 of them that have been discovered (Velavan, 2015; Shakir *et al*., 2019, Awuchi *et al.*, 2020). Some of the active phytochemicals that are contained in Tiger-nut include tannins, alkaloids, saponins, flavonoids, phenols, phylates, oxylates, saponin, etc. (Ayo *et al*., 2016; Marchyshyn *et al*., 2021).

One of the problems encountered in the handling of Tiger-nut is their high perish-ability that usually results in high postharvest losses. To improve its shelf-life, storability and diversified utilization, process operations such as pretreatments, heat application and resource conversion are carried out on products after harvest. The common pretreatments that are relative to Tiger-nut handling and processing are soaking, steeping, blanching, fermentation (Muniandi and Hidetoshi, 2018), malting, (Deepika, 2017), heating/roasting (Ogunsina, 2017), etc.

Soaking is an essential unit operation in food processing that is usually carried out on biomaterials at different temperatures and time. It has been reported to improve the sensory characteristics of food materials and reduces the soluble bioactive compounds which could pose significant health hazards to the consumer (Adebayo, 2014; Howard *et al*., 2018; Ravoninjatovo *et al*., 2021). The different drying conditions that this biomaterial is usually put through in order to improve its shelf-life include *microwave drying*, open sun drying, solar drying, convective oven drying, freeze drying and osmotic dehydration. Subjecting Tiger-nut to microwave drying at different power levels promotes flavour development, improves the nutritional contents and causes the removal of such anti-nutrients that are heat labile in nature. Pretreatments and drying conditions have been reported to pose adverse effects on the nutritional, mineralogical, phytochemical and sensory properties of biomaterials (Abubakar *et al.*, 2018; Emurigho *et al.*, 2020; Omale *et al.*, 2020; Umaru *et al.*, 2018; Usman *et al.*, 2019).

Sound knowledge of the effects of pretreatments and drying conditions is very essential for effective preservation, storage and processing of biomaterials. In order to effectively carry out these operations without any impairment on agricultural and food materials, keen understanding of the interactions of the process variables and the biomaterial properties is paramount. Hence, there is a need to embark on such research that examines the effects of pretreatments and heat applications on the nutritional, mineral and phytochemical characteristics of Tiger-nut. Despite the fact that studies have been devoted to the behavior of the Tiger-nut during soaking with respect to water absorption kinetics and the characteristics of unstable bioactive components when roasted or dried in a convective oven, there is dearth of information on the effects of soaking and microwave drying on the essential phytochemical compounds of Tiger-nut.

This paper therefore assesses the effects of soaking pretreatments and microwave drying on selected phytochemical properties of Tiger-nut. The choice of the phytochemicals considered for the study was made in relation to the fact that such bioactive compounds have the deterrence potentials against diseases and chronic health conditions, and their essence in the nutrient quality prospects of Tiger-nut resources in both local and industrial applications. The study also helps to establish the best pretreatment(s) and microwave drying conditions that will still maintain the phytochemical integrity of the product while sustaining improved shelf life.

2.0 Material and methods

2.1 Materials

The biological material (Tiger-nut) that was used for the study was sourced, identified and prepared prior to the pretreatment and drying operations.

2.1.1 Materials sourcing and botanical identification

Fresh Tiger-nut tubers were purchased from the Eke local market at Ekwulobia in Aguata Local Government of Anambra State. The bought tubers were introduced into polyethylene bags and transported to the crop processing laboratory where the study was carried out. Randomly selected stock of Tiger-nut tubers were sent to a botanical laboratory for identification. This was necessary to ascertain that the sourced material was actually the intended biomaterial to be researched upon.

2.1.2 Material preparation

The Tiger-nut stock was sorted to remove stones, pebbles, dirt materials, rotten stems and broken tubers and cleaned in water to remove adhering dirt. The samples were selected considering the visual inspection of uniform colour, size ($7.12 \pm 0{:}77$ mm geometric mean diameter/thickness), and approximately spherical in geometry for the drying operations.

2.2 Methods

The methods of the study featured the use of equipment and reagents, phytochemical screening, experimental design and execution, phytochemical content analyses, and data analyses and inference.

2.2.1 Equipment and reagents for experiment

The equipment that were used for the analysis include spectrophotometer (Jasco ultra violet spectrophotometer model V-630), electronics weigh balance (Schimadzu AUN 220D. UniBloc), analytical flour mill, power variable laboratory microwave, temperature variable convective oven, sieve apparatus, filter flask, gas cover, stainless utensils, etc.. The major reagents that were used for the laboratory experiments included hydrochloric acid, sulfuric acid, 0.3% ammonium thiocyanate solution, sodium chloride, ethanol, copper sulphate (hydrate), methyl red, boric acid, diethyl ether, hexane, sodium carbonate, $1.5NH_2SO_4$, $0.1NKMnO_4$, Anhydrous sodium sulphate, etc.

2.2.2 Phytochemicals screening

20 g of the reduced Tiger-nut flour samples were introduced into 100 ml of ethanol in a continuous extraction instrument. The recovered extracts were further filtered using chess cloth and the filtrate was utilized for the qualitative screening to ascertain the phytochemical that are present in Tiger-nut. The methods described by Bando *et al.* (2020) were adopted in the individual test for the fundamental food products phytochemicals.

2.2.3 Preliminary drying operation

100 g of fresh Tiger-nut was introduced into the oven set at 70 ^{o}C and run till the material gained equilibrium moisture content (EMC). This was carried out in order to establish the drying time interval that was feasible for the intended experimental temperature ranges.

2.2.4 Experimental design

A two factor, four level factorial designs in a completely randomized design was adopted for the study. The Design Expert 13 software was used to establish the experimental matrix for the determination of the effect of soaking time and microwave power on the phytochemical properties of Tiger-nut samples. Microwave power and soaking time were chosen as the two design factors. The two factors were rooted on the impact of different soaking time and the imposing microwave power effect on Tiger-nut, while the adopted levels were based on pre-experimental trials.

2.2.5 Sample Soaking and Drying Treatments

Three different portions of the cleaned products were respectively soaked for 24 hr, 48 hr and 72 hour in water at room temperature. The soaking time were adopted with the consideration of having enough time for water molecules to permeate the hard pericarp of the Tiger-nut tubers into their interior. Each treated Tiger-nut was further dried in microwave oven at 100 W, 140 W, 200 W and 280 W and the material weight variations were measured at the drying time intervals of 15 min. After respectively soaking and drying to constant weight, samples were randomly drawn from the pretreated and dried stock, ground in an analytical flour mill, and sieved with a 160 μm mesh sieve to get fine powder. The obtained Tiger-nut flour was stored in properly labeled plastic sterile until they were required for phytochemical properties analyses.

2.3 Determinations of phytochemical composition

The presence of tannin, alkaloid, saponin, flavonoid and phenol were determined by respective Price and Butler method, Folin-ciocalteau method, The Aluminum (III) Chloride ($AlCl_3$) method, and gravimetric method. The results of the phytochemical analyses were expressed in milligram per gram (mg/g).

2.3.1 Determination of condensed tannin content

The condensed tannin content of the various Tiger-nut samples was determined according to the method described by Umaru *et al.* (2018). Prepared I ml extract of each Tiger-nut sample was diluted to 10ml with distilled water, and mixed with constituted 0.5ml of 0.1 M FeCl3 in 0.1 NHCl and 0.5 ml of 0.008 $MK_3Fe(CN)_6$. The mixture was left for 1 min for the colour to develop, and the absorbance was then read at 250 nm. The tannin content was extrapolated from the standard curve that initially prepared using tannic acid at concentrations of 0.0, 0.01, 0.04, 0.08, 0.15, 0.20, 0.50 and 1.0 mg/mL) and expressed as the equivalent tannic acid (TAE/100 g).

2.3.2 Determination of total flavonoid

The Aluminum (III) Chloride ($AlCl_3$) method described by Shraim *et al.* (2021) was used to determine the total flavonoid contents in the Tiger-nut sample. Aliquot of 1.5 ml of each freshly prepared Tiger-nut sample was added to equal volumes of a solution of 2% $AlCl_3.6H_20$ (2g in 100ml methanol). The mixture was vigorously shaken and the absorbance was read at 367nm after 10min incubation period. 0.0, 0.02, 0.05, 0.10, 0.50, 1.50 and 2.0 mg/mL concentrations of garlic acid were used to prepare the flavonoid standard curve for. Flavonoid content was then extrapolated from the standard curve using the absorbance values and expressed as garlic acid equivalents (GAE/100 g).

2.3.3 Determination of alkaloid content

The gravimetric method was adopted in this study for determination of the alkaloid content (Nimenibo-Uadia *et al*., 2017; Dey *et al*., 2020). 5.0 g of ground Tiger-nut sample was dispersed with 50 ml of distilled water, 95% methanol, acetone/hexane, n-hexane/methanol/acetone and acetone/water/acetic acid solvents in 250 ml volumetric flasks. This was shaken vigorously and allowed to rest for 4 hr before being filtered through Whatman No. 5 filter paper. The filtrate was evaporated to one quarter of the original volume and concentrated ammonium hydroxide (NH_4OH) was added in drops to each of the alkaloid until the precipitate persisted. The mixture was again filtered through weighed filter paper, and the alkaloids residues washed with 1% ammonium hydroxide solution. The filter papers and contents (alkaloids) are oven-dried at 60 ^{O}C for 30 min. and reweighed to determine alkaloids contents using the expression:

$$\text{Percentage (\%) alkaloid} = \frac{W_2 - W_1}{W} \times \frac{100}{1} \quad (1)$$

Where, W = Weight of sample, g
W_1 = Weight of filter paper, g
W_2 = Weight of filter paper and precipitated alkaloid, g

2.3.4 Determination of total phenol

The determination of the total phenol content was carried out using Folin-ciocalteau method which allows for the estimation of all anthocyanins, flavonoids, and non-flavonoid phenolic compounds with tannins and phenols inclusive (Razola-Diaz *et al*., 2022). The total phenol content of the various Tiger-nut samples was determined by mixing 0.5ml aliquot of freshly prepared sample extract with equal volume of water, 0.5 ml Folin-Ciocalteu's reagent, and 2.5 ml of saturated solution of sodium carbonate (Na_2C0_3). The absorbance was determined after 30 min at 647 nm (Yamin *et al*., 2021). 0.0, 3.0, 6.0, 12.0, 18.0, 24.0 and 30.0 µg/mL concentrations of garlic acid were used to prepare the standard curve for total phenol. Total phenol content was then extrapolated from the standard curve using the absorbance values and expressed as garlic acid equivalents (GAE/100 g).

2.3.5 Oxalate content determination

The method described by Iwuozor (2019) was adopted for the determination of oxalate content in Tiger-nut samples. 2 g of the sample was weighed into a 250 ml volumetric flask and suspended in 190 ml of distilled water. 10 ml of 6M HCl was added and the suspension digested at 100 ºC for 1 hr. It was cooled, and then made up to 250 ml mark before filtration. Duplicate portion of 125 ml of the filtrate were measured into beakers and 4 drops of methyl red indicator added. This was followed by the addition of NH_4OH solution (drop-wise) until the test solution changed from salmon pink to a faint yellow colour (pH 4-4.5). Each portion of the duplicate sample was heated to 90 ºC, cooled and filtered to remove precipitate containing ferrous ion. The filtrate was again heated to 90 ºC and 10ml of 5% CaCl solution added while being stirred constantly. After heating it was cooled and left overnight at 25 ºC, the solution was then centrifuged at 2500 revolution per minute for 5 min. The supernatant was decanted and the precipitate completely dissolved in 10 ml of 20% H_2SO4 solution. It was made up to 300 ml. Aliquots of 125 ml of the filtrate was heated until near boiling and then titrated against 0.05 M standard $KMNO_4$ solution to a faint pink colour which persists for 30 s.

$$\text{Oxalate, (mg/100)} = \frac{T \times (Vme)(Df)}{(ME) \times Mf} \quad (2)$$

Where, T = titre value
Vme = volume-mass equivalent (i.e 1 ml of 0.05 M $KMN0_4$ solution is equivalent to 0.00225 g anhydrous oxalic acid).
D.f = Dilution factor (2.4)

ME = Molar equivalent of $KMNO_4$
Mf = Mass of sample used.

2.3.6 Cardiac glycoside content determination

The method that was described by Bando *et al.* (2019) was utilized in the determination of the cardiac glycoside content of the Tiger-nut samples. 5 g of the sample was weighed into beaker and 100 ml of distilled water were added. The sample was soaked for 3 hr and filtered to collect the filtrate. To 2 ml extract of the sample was added 1 ml of 2% solution of 3, 5-DNS (Dinitro salicylic acid) in methanol and 1 ml of 5% aqueous NaOH. It was boiled for 2 min (until brick-red precipitate was observed and the boiled sample was filtered. The weight of the filter paper was weighed before filtration. The filter paper with the absorbed residue was dried in an oven at 105 ^{o}C till dryness and weight of the filter paper with residue was noted.

$$\% \text{ cardiac glycoside} = \frac{\text{Weight of filter paper with residue} - \text{Weight of filter paper}}{\text{Weight of sample analyze}} \times \frac{100}{1} \quad (3)$$

2.4 Result Analysis

All data generated from the experiments were reported as Mean ± SD. One-way ANOVA was used to assess the significance of the levels of the factors on the Tiger-nut samples and the values with $p < 0.05$ were considered significant.

3.0 Results and Discussions

3.1 Results of Tiger-nut Samples Botanical Identification

The Tiger-nut tuber was of the mini yellow specie, sativus Boeckeler variety, JYD-35 ascension, morphiotype 2 category with a mean size of 7.12mm ($\emptyset < 10$mm) and an approximated round shape due to the relative mean sphericity, aspect ratio and flakiness ratio (Table 1).

Table 1: Botanical identification result of Tiger-nut samples used for the study

Identification features	**Specifications**
Specie	Mini yellow
Variety	*Cyperus esculentus* var. sativus Boeckeler
Ascension	Cameroun (JYD-35)
Category	Morphotype 2 (yellow and small)
Size	7.12mm ($\emptyset < 10$mm)
Shape	Round (mean sphericity = 0.964; mean aspect ratio = 0.850; mean flakiness ratio = 1.03)

3.2 Drying Rate Curve at different microwave power

Figures 2, 3, 4 and 5 show the drying rate curves for differently soaked Tiger-nut tubers and dried at 100 W, 140 W, 200 W and 280 W conditions respectively.

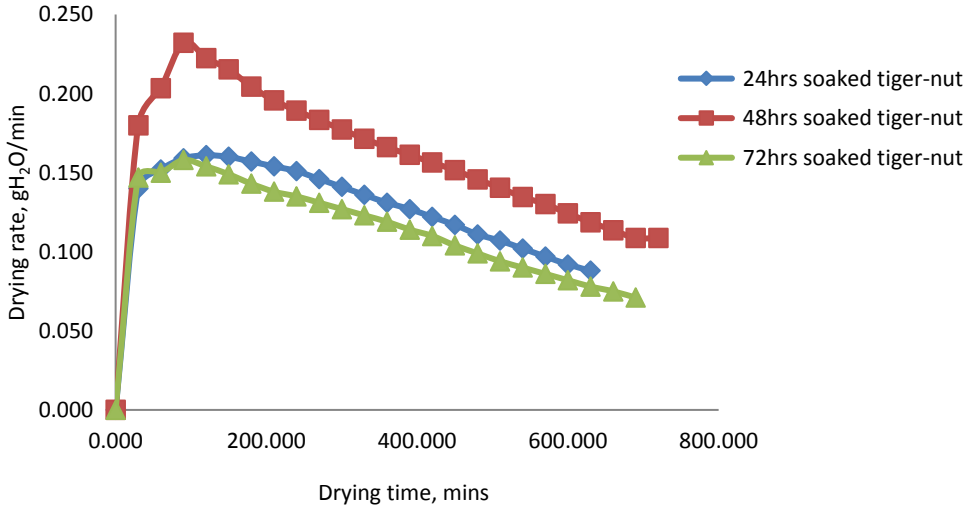

Figure 2: Drying rate curve for Tiger-nut sample dried in 100 W microwave power

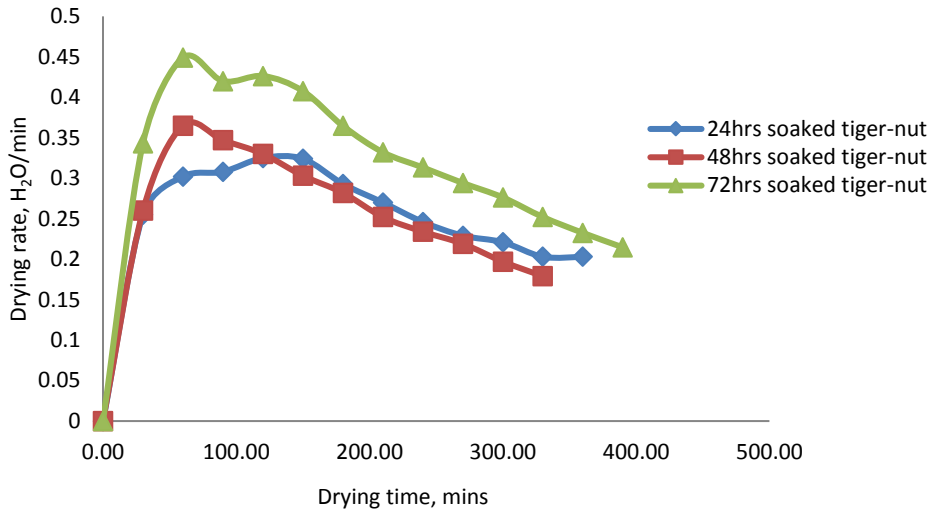

Figure 3: Drying rate curve for Tiger-nut sample dried in 140 W microwave power

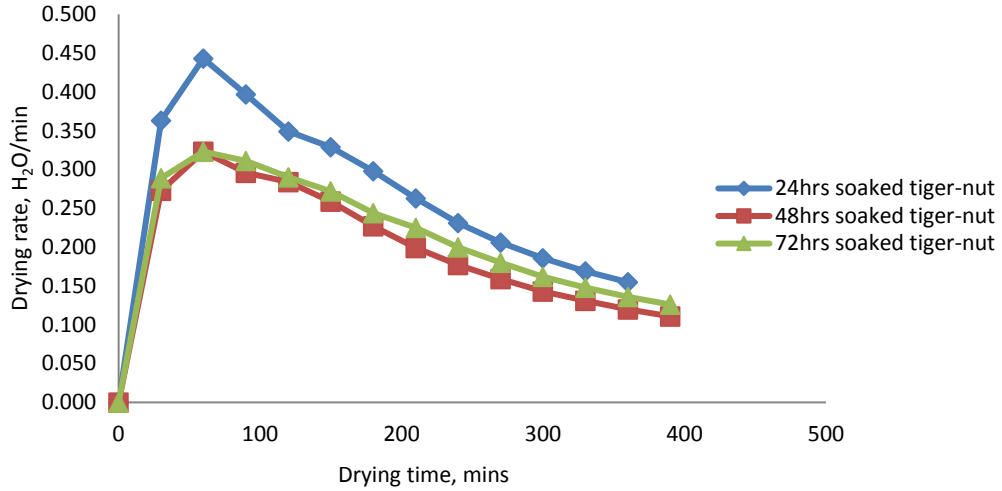

Figure 4: Drying rate curve for Tiger-nut sample dried in 200 W microwave power

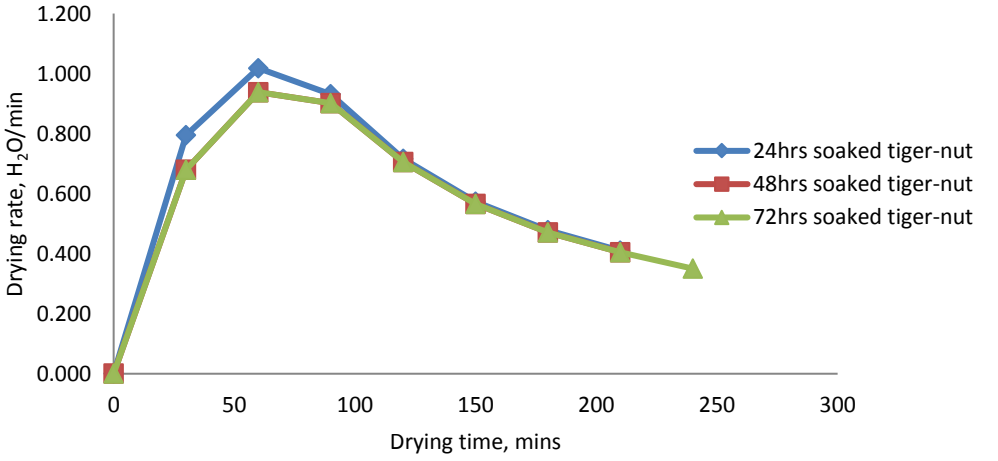

Figure 5: Drying rate curve for Tiger-nut sample dried in 280 W microwave power

3.3 Effects on Equilibrium Moisture Content (EMC) and drying time

Figures 6 and 7 represent the effects of microwave drying conditions on the equilibrium moisture contents (EMCs) and drying times for the differently soaked Tiger-nut samples.

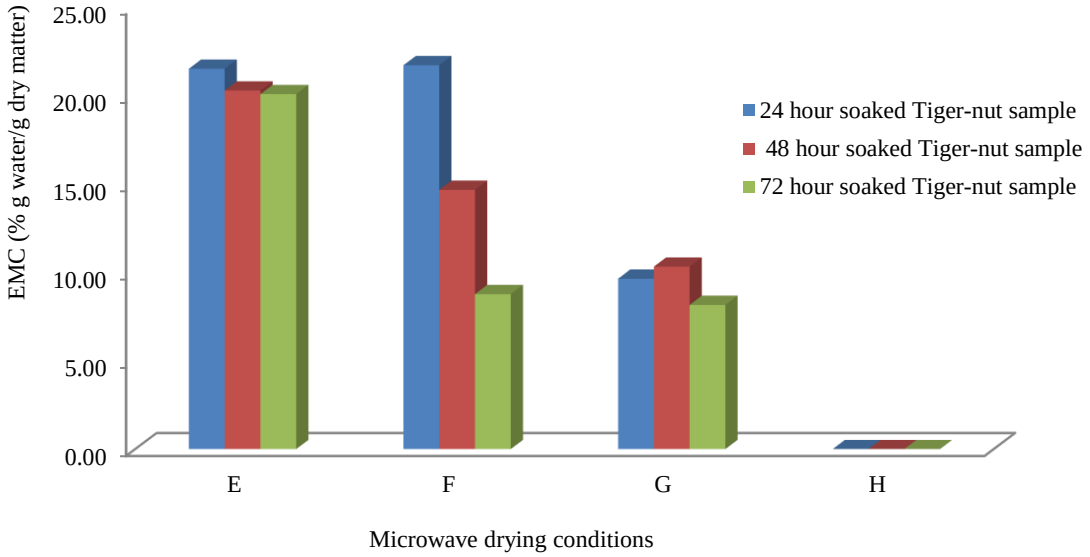

Figure 6: Effects of different treatments and microwave drying conditions on EMC
(E – samples dried at 100 W microwave power; F – samples dried at 140 W microwave power; G – samples dried at 200 W microwave power; H - samples dried at 280 W microwave power)

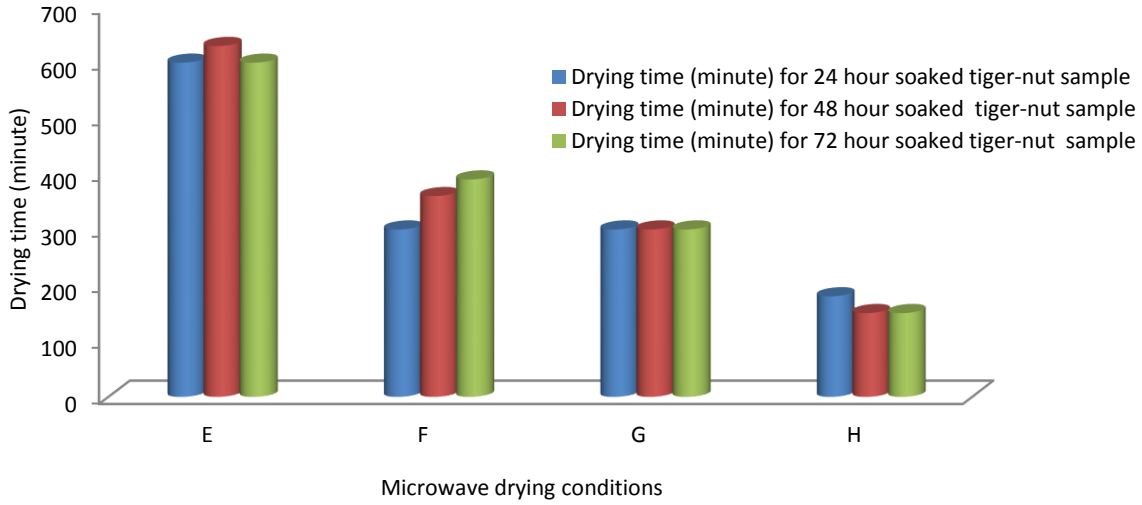

Figure 7: Effects of microwave drying conditions on drying time.
(E – samples dried at 100 W microwave power; F – samples dried at 140 W microwave power; G – samples dried at 200 W microwave power; H - samples dried at 280 W microwave power)

3.4 Effects of pre-treatments and drying conditions on phytochemical compositions

Table 2 showed the results of Analysis of variance (ANOVA) of effects microwave drying on the phytochemical properties of pretreated (soaked) Tiger-nut. Tables 3 outlined the computed mean and standard deviation of the effects of microwave power on the phytochemical compositions of pre-treated Tiger-nut sample.

Table 2: ANOVA of phytochemical properties of microwave dried pretreated Tiger-nut

Experimented samples	Source of Variation	SS	df	MS	F	P-value	F crit
E	Between Groups	26859.100	5	5371.830	13.012	1.900E-05	2.773
	Within Groups	7430.800	18	412.822			
	Total	34289.900	23				
F	Between Groups	29206.325	5	5841.265	15.964	4.549E-06	2.773
	Within Groups	6586.387	18	365.910			
	Total	35792.711	23				
G	Between Groups	28570.900	5	5714.170	15.182	6.500E-06	2.773
	Within Groups	6774.910	18	376.384			
	Total	35345.800	23				
H	Between Groups	26244.900	5	5248.980	11.775	3.700E-05	2.773
	Within Groups	8023.810	18	445.767			
	Total	34268.700	23				

Legend
E = samples dried at 100 W microwave power; F = samples dried at 140 W microwave power; G = samples dried at 200 W microwave power; H = samples dried at 280 W microwave power

Table 3: Mean phytochemical compositions of fresh and microwave dried Tiger-nut samples

Soaking time (hr)	Microwave power (W)	Responses					
		Flavonoids (mg/g)	Alkaloids (mg/g)	Tannin (mg/g)	Oxalate (mg/g)	Cardiac glycoside mg/g)	Phenol (mg/g)
Control	—	**100.037± 0.0024***	**110.34± 0.0105***	**6.400± 0.0027***	**0.00313± 0.0022***	**83.210± 0.0055***	**0.0055± 0.0012***
24	100	52.320± 0.0051*	22.110± 0.0314*	5.591± 0.0139*	0.00171± 0.0278*	89.760± 0.0036*	0.0082± 0.0064*
48	100	87.290± 0.0085*	26.760± 0.0485*	5.023± 0.0086*	0.00160± 0.0417*	49.750± 0.0042*	0.0197± 0.0177*
72	100	68.950± 0.0101*	34.880± 0.0223*	5.013± 0.0154*	0.00194± 0.0300*	69.170± 0.0015*	0.0090± 0.0185*
24	140	79.490± 0.0311*	21.780± 0.0552*	6.359± 0.0254*	0.00168± 0.0425*	85.740± 0.0103*	0.0109± 0.0115*
48	140	87.160± 0.0111*	27.670± 0.0337*	6.352± 0.0186*	0.00207± 0.0493*	49.160± 0.0098*	0.0184± 0.0118*
72	140	69.170± 0.0362*	32.080± 0.0413*	6.112± 0.0188*	0.00302± 0.0389*	74.940± 0.0111*	0.0163± 0.0208*
24	200	56.240± 0.0051*	21.860± 0.0327*	3.885± 0.0285*	0.00188± 0.0385*	84.980± 0.0087*	0.0107± 0.0169*
48	200	84.660± 0.0169*	27.850± 0.0381*	3.014± 0.0255*	0.00183± 0.0545*	62.160± 0.0100*	0.0109± 0.0228*
72	200	66.130± 0.0095*	30.140± 0.0254*	2.003± 0.0279*	0.00199± 0.0466*	80.420± 0.0120*	0.0102± 0.0196*
24	280	47.080± 0.0610*	23.670± 0.0562*	1.557± 0.0288*	0.00108± 0.0511*	81.400± 0.0176*	0.0109± 0.0058*
48	280	74.610± 0.0477*	22.890± 0.0497*	0.844± 0.0302*	0.00280± 0.0558*	54.730± 0.0204*	0.0197± 0.0276*
72	280	59.190± 0.0495*	21.760± 0.0490*	1.005± 0.0409*	0.00211± 0.0534*	88.430± 0.0108*	0.0091± 0.0213*

The asterisked (*) values show significance difference in their respective mean values

3.5 Discussion

Generally, the drying rate decreased with increase in drying time. The drying conditions followed two drying rate patterns – a relatively brief initial rise in drying rate and prolonged falling rate till equilibrium moisture is attained. This is in agreement with Alonge and Gilbert (2019) report in their study on the effect of drying methods on the quality of Tiger-nut. Figure 2 showed the drying rate of the soaked samples dried at 100 W microwave power. The curves showed that there was an increase in drying rate from 0 – 0.161 gH_2O/min between 0 – 120 min and a decrease from 0.161 – 0.088 gH_2O/min between 120 – 630 min for the sample soaked for 24 hr. For the 48 hr soaked sample, there was a rapid increase in drying rate from 0 – 0.215 gH_2O/min between 0 – 150 min and a decrease from 0.215 – 0.109 gH_2O/min between 150 – 690 min. For the sample soaked for 72 hr, there was an increase in drying rate from 0 – 0.158 gH_2O/min between 0 – 90 min and a decrease from 0.158 – 0.071 gH_2O/min between 90 – 690 min.

Figure 3 showed the drying rate of the soaked samples dried at 140 W microwave power. The curves showed respective increase in dry rates from 0 – 0.325 gH_2O/min, 0 – 0.365 gH_2O/min and 0-0.449 gH_2O/min between 0 – 120 min, 0 – 90 min and 0 – 60 min for Tiger-nut soaked for 24 hr, 48 hr and 72 hr. There were respective decreases in dry rates from 0.325 – 0.203 gH_2O/min, 0.365 – 0.179 gH_2O/min and 0.449 – 0.215 gH_2O/min between 120 – 330 min, 60-330 min and 60 – 390 min for Tiger-nut soaked for 24 hr, 48 hr and 72 hr.

Figure 4 showed the drying rate of the soaked samples dried at 200 W microwave power. The curves show that there was increase in drying rate from 0 – 0.443 gH_2O/min between 0 – 60 min and decreases from 0.443 – 0.155 gH_2O/min between 60 – 360 min for Tiger-nut soaked for 24hr. The drying rate increases from 0 – 0.323 gH_2O/min between 0 – 60 min and decreases from 0.323 – 0.103 gH_2O/min between 60 – 420 min for Tiger-nut soaked for 48 hr. For the sample soaked for 72 hr, the drying rate increases from 0 – 0.323 gH_2O/min between 0 – 60 min and decreases from 0.323 – 0.109 gH_2O/min between 60 – 450 min.

Figure 5 showed the drying rate of the soaked samples dried at 280 W microwave power. The curves indicated that between 0 – 60 min, the drying rate increases from 0 – 1.018 gH_2O/min and between 60 – 210 min, there was a decrease in drying rate from 1.018 – 0.410 gH_2O/min for Tiger-nut soaked for 24 hr. For Tiger-nut soaked for 48hr, the drying rate increases from 0 – 0.938 gH_2O/min between 0 – 60 min and decreases from 0.938 – 0.404 gH_2O/min between 60 – 210 min. For Tiger-nut soaked for 72 hr, the drying rate increases from 0 – 0.938 gH_2O/min between 0 – 60 min and decreases from 0.938 – 0.350 gH_2O/min between 60 – 240 min.

The mean drying time at which EMC was attained for 100 W, 140 W, 200 W and 280 W microwave power dried Tiger-nut was 580 min, 330 min, 310 min and 130 min respectively. This showed that the higher the microwave power the lower the mean drying time and vice versa. The Tiger-nut that was soaked for 72 hr before drying in microwave had the least EMC. The EMCs obtained from Tiger-nut dried at 200 W and 280 W presented unique trends. The higher EMCs recorded for samples dried at microwave power of 100 W and 140 W could be attributed to possible case hardening of the surface of the Tiger-nut samples. The 200 W microwave drying condition gave a more stable EMC for the samples soaked for 24 hr, 48 hr and 72 hr; while the highest microwave power (280 W) resulted in degradation of Tiger-nut tubers by causing burning of the products almost before the equilibrium moisture was attained.

At 0.05 probabilities, it was observed that soaking pretreatments and microwave drying conditions had significant effects on all the understudied phytochemical properties. The respective p-values of 1.900E-05, 4.549E-06, 6.500E-06 and 3.700E-05 obtained for soaked Tiger-nut dried at 100 W, 140 W, 200 W and 280 W which are less than the experimental $p<0.05$ level of confidence further showed that soaking and microwave drying significantly affected the biomaterial. The effects were greatest for the samples dried at 140 W and 200 W microwave powers. The average flavonoids, alkaloids, tannin, oxalate, cardiac glycoside and phenol contents obtained for the fresh Tiger-nut (control) samples were 100.037 ± 0.0024 mg/g, 110.340 ± 0.0105 mg/g, 6.400 ± 0.0027 mg/g, 0.00313 ± 0.0022 mg/g, 83.210 ± 0.0055 mg/g and 0.0055 ± 0.0012 mg/g respectively. After the experiments, there were relatively significant alterations on the phytochemical parameters. The microwave drying at different power resulted to decrease in flavonoids, alkaloids, tannin, oxalate, cardiac glycoside and phenol contents.

From the results it was obvious that the drying conditions resulted in drastic reduction in moisture content of the samples. This finding followed similar trends with the studies carried out by Omale *et al.* (2020) and Ibeogu and Eze (2022) who reported that drying process reduces the moisture content of the food to a safe storage limit. The phytochemical contents obtained in this study for dried Tiger-nut is higher than those reported by Alonge and Gilbert (2019). They agreed to the fact that microwave oven drying pose significant effect on moisture and maintained that it had the lowest moisture (0.05) when compared with sun drying and oven drying. The contents of dried Tiger-nut were significantly reduced among other drying methods when compared with the control. The results further showed that soaking in water under room temperature and microwave drying caused reductions in flavonoids, alkaloids, tannins and oxalate contents and increase in cardiac glycoside and phenol contents of Tiger-nut *w*hen compared with the fresh sample. This was also confirmed by Alonge and Gilbert (2019) who reported in their study that cyanide, oxalate, phylate and tannin contents were reduced due to different drying condition and more significant with the microwave drying.

4.0 Conclusion

Tiger-nut is rich in such phytochemicals as saponin, flavonoids, alkaloids, tannin, oxalate, phylate, Hemaglutinin, cardiac glycosides and phenol that are of great health and therapeutic importance. The effects of soaking at different duration of 24, 48

and 72 hr, and drying at 100 W, 140 W, 200 W and 280 W microwave powers on selected phytochemical contents of composition of Tiger-nut were assessed. For all the samples, the pretreatments and drying conditions caused the reduction in moisture content of Tiger-nut to a safe storage level, variation in the drying characteristics (drying rates, drying time, and equilibrium moisture) and relatively significant alterations on the tested phytochemical parameters. The drying rates and drying time increased with increase in microwave power.

The mean drying time at which constant weight and equilibrium moisture (EMC) were attained for 100 W, 140 W, 200 W and 280 W microwave power dried Tiger-nut samples was 580 min, 330 min, 310 min and 130 min respectively. At 0.05 probabilities, all the understudied phytochemical properties were significantly affected and the effects were more for the samples dried at 140 W and 200 W microwave powers. There was decrease in the flavonoids, alkaloids, tannin, and oxalate contents and slight increase in the cardiac glycoside and phenol contents of the pretreated and dried samples when compared with the constituents of the untreated fresh sample. The best microwave power for drying Tiger-nut sample to conserve the useful phytochemicals was observed to be 100 W. Moreover, soaking treatment and microwave drying improves Tiger-nut tubers to have unique fermented features and nutty taste. These features present the product to be suitable for the production of Tiger-nut food drink and infant food supplement.

5.0 Recommendation

With the information divulged in the study, it becomes pertinent to hint on the following recommendations.

i. Agricultural processing and food extension agencies should strategize on formidable awareness creation schemes on the essence of Tiger-nut as phytochemical fortified and dietry resource to alleviate its present status as "under-utilized food product".
ii. Prior to secondary value addition and diversified utilizations, Tiger-nut should be subjected to such treatments as soaking, and dried at 100 W microwave power to improve the sensory properties of the product.
iii. A follow up study should be serviced on the determination of the effects of soaking and microwave drying conditions on the vitamins and mineral contents of Tiger-nut to further add to the full scale quality analysis prospects of the biomaterial.

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References

Abdullahi, N., Badau, M.H., Umar, N.B., Yunusa, A.K., Rilwan, A, Jibril, H. & Iliyasu, R., 2022. Tiger-nut: A nutrient-rich underutilized crop with many potentials. *FUDMA Journal of Sciences (FJS)*, 6, 2, 10 –115.

Abubakar, H., Atiku, M., Alhassan, A. & Sa'id, A., 2018. Effects of processing treatments on the chemical composition of tiger nut (*Cyperus esculentus*) milk products. Department of Biochemistry, Faculty of Basic Medical Sciences, Bayero University Kano, Kano State, Nigeria. 12 pages. doi:10. 20944/preprints201802.0054.v1

Adebayo, S.F., 2014. Effect of soaking time on the proximate, mineral compositions and anti-nutritional factors of lima bean. *Food Science and Quality Management*, 27, 1–5. https://core.ac.uk/download/pdf/234683781. pdf

Alonge, A.F. & Gilbert, E., 2019. The effect of drying methods on the quality of Tiger-nut (*Cyperus esculentus* lativum). Proceedings of the 2019 International Joint Conference on JSAM and SASJ, and 13th CIGR VI Technical Symposium joining FWFNWG and FSWG Workshops. 11 pages.

Asare, P.A., Kpankpari, R., Adu, M.O., Afutu, E. & Adewumi, A.S., 2020. Phenotypic characterization of Tiger-nuts (*Cyperus esculentus* L.) from major growing areas in Ghana. *The Scientific World Journal (Hindawi)*, 1, 1 – 11.

Awuchi, C.G., Echeta, C.K., Anyanwu, C.O., Nwagboso, O. & Amagwula, I.K., 2020. Medicinal plant phytochemicals; the biochemistry and the uses if the pharmacologically active Alkoloids, Terpens, Polyphenols and Glycosides. Conference paper presented at the 44th NIFST Conference held at October, 2020. No. AB 013; 3 pages.

Ayo, J.A., Adedeji, O.E. & Ishaya, G., 2016. Phytochemical composition and functional properties of flour produced from two varieties of Tiger-nut (*Cyperus esculentus*). *FUW Trends in Science and Technology Journal*, 1(1): 261-266.

Bando, D.C., Tutuwa, J.A., Ogu, E.O., Nuhu, I. & Mbaragbog, S.J., 2020. Phytochemical screening, proximate nutritional analysis and anti-nutrient analysis of Tiger-nut (*Cyperus esculentus*). *International Journal of Advanced Scientific Research*, 5, 4, 53–60.

Bazine, T. & Arslanoglu, S.F., 2020. Tiger nut (*Cyperus esculentus*); Morphology, products, uses and health benefits. *Black Sea Journal of Agriculture*, 3, 4, 324–328.

Deepika, B., 2017. Malting: An indigenous technology used for improving the nutritional quality of grains – A review. *Asian J. Dairy & Food Re*., 36, 3, 179–183

Dey, P., Kundu, A., Kumar, A., Gupta, M., Lee, B.M., Bhakta, T., Dash, S. & Kim, H.S., 2020. Analysis of alkaloids (indole alkaloids, isoquinoline alkaloids, tropane alkaloids). In: Recent Advances in Natural Products Analysis, Silva A. S., Nabava, S. F., Saeedi, M & Nabavi, S. M.. *Elsevier*, 505–567.

Djomdi, I, Bakari, H., Gibert, O., Tran, T., Ejoh, R., Christophe, G.,Michaud, P. & Ndjouenkeu, R., 2022. The influence of soaking and sprouting on the physicochemical characteristics of Tiger-nut tubers (*Cyperus esculentus* L.). *Applied Chem*., 2, 48–58. https://doi.org/10.3390/appliedchem2020003

El-Naggar, E.A., 2016. Physicochemical characteristics of Tiger-nut tuber (*Cyperus esculentus* Lam) oil. Middle East J. Appl. Sci., 6, 4, 1003–1011.

Emurigho, T.A., Kabuo, C.O.O. & Ifegbo, A.N., 2020. Determination of physical and engineering properties of Tiger-nut (Cyperus esculentus) relevant to its mechanization. *International Journal of Engineering Applied Sciences and Technology*, 5, 8, 82–90.

Fabunmi, T.O., Adigbo, S.O., Odedina, J.N. & Akinsanya, Y.I., 2016. Effects of tuber size, soaking hr and sprouting media on sprouting of tiger nut (Cyperus esculentus L. var. sativa) tubers. *Acta agriculturae Slovenica*, 107, 2, 345–354.

Henry, G.M., Elmore, M.T. & Gannon, T.W., 2021. *Cyperus esculentus* and *Cyperus rotundus*. 1st edition. In: Chauhan, B.S. (eds.), Biology and Management of Problematic Crop Weed Species. London: Academic Press, 151–172.

Howard, L.R., White, B.L., Uebersax, M.A., & Siddiq, M., 2018. Dry beans processing, quality evaluation and nutrition. In M. Siddiq & M. A. Uebersax (eds.), *Handbook of vegetables and vegetable processing* (2nd ed.,). John Wiley & Sons, 559–587. https://doi.org/10.1002/ 9781119098935.ch24

Ibeogu, I. H. and Eze, J. I. 2022. Effect of drying temperature on the proximate, vitamin and mineral compositions of Tiger-nut (Cyperus esculentus). *International Journal of Advances in Engineering and Management (IJAEM)*, 1(5): 1053-1059.

Idoia-Codina, T., Buenaventura, G. &Antonio, J., 2014. Characterization and comparison of Tiger-nuts (Cyperus esculentus L) from different geographical origin, physicochemical characteristics and protein fractionation industrial crops and products. In: D.T. Ray, M. J. Pascual-Villalobos, M.N. Belgacem, M. Berti, Loftsgard H. and E. Frollini (2015). *Industrial Crops and Products*. *Elservier*; 65, 406–414. Available online at http:www.Tiger-nut.com

Iwuozor, K.O., 2019. Qualitative and quantitative determination of anti-nutritional factors of five wine samples. *Advanced Journal of Chemistry – Section A*, 2, 2, 136 – 146.

Kizzie-Hayford, N., Ampofo-Asiama, J., Zahn, S., Jaros, D. & Rohm, H., 2021. Enriching Tiger-nut milk with sodium caseinate and xanthan gum improves the physical stability and consumer acceptability. *Journal of Food Technology Research*, 8, 2, 40–49.

Malashree, L., Prabha, R., Ramachandra, B. and Sushmitha, P., 2021. Tiger-nuts (*Cyperus esculentus*) – palieo but today's super food. *International Research Journal of Modernization in Engineering Technology and Science*, 3, 01, 1172–1178.

Marchyshyn, S., Budniak, L., Slobodianiuk, L., and Ivasiuk, I., 2021. Determination of carbohydrates and fructans content in *Cyperus esculentus* L. Pharmacia, 68, 1, 211–216. https://doi.org/10.3897/pharmacia.68.e54762

Martín-Esparza, E. & González-Martínez, C., 2016. Horchata de Chufa: a traditional Spanish beverage with exceptional organoleptic, nutritive, and functional attributes. In: Kristbergsson, K., Otles, S. (eds.), *Functional Properties of Traditional Foods. New York*, NY: Springer, 371–375.

Muniandi, J. & Hidetoshi, M. (2018). Transformation of traditional fermented foods of South India. *International Journal of Research in Agricultural Sciences*; 5(3):145–149.

Nimenibo-Uadia, R., Ugwu, I., Erameh, T. & Osunde, E., 2017. Estimation of tannins, alkaloids, saponins and proximate composition of Vernonia amygdalina (Del) root. *International Journal of Herbal Medicine*, 5, 3, 88–92.

Ogunsina, D. (2017). The Chemical analysis of food (Chap. 4). *Churchill living Edinburgh London*, 78–80.

Omale, P.A., Iyidiobu, B.N. & Ibu, E.J., 2020. Effect of drying temperature on the nutritional quality of Tiger-nut (Cyperus sculentus). *International Journal of Engineering Applied Sciences and Technology*; 4, 9, 399–403.

Onuoha, N.O., Ogbusua, N.O., Okorie, A.N., & Ejike, C.C., 2017. Tiger-nut (*Cyperus esculentus* L.) "milk" as a potent "nutri-drink" for the prevention of acetaminophen-induced hepatotoxicity in a murine model. *Journal of Intercultural Ethnopharmacology*, 6, 3, 290–295.

Ravoninjatovo, M., Ralison, C., Servent, A., Morel, G., Achir, N. Andriamazaoro, H. & Dornier, M., 2021. Effects of soaking and thermal treatment on nutritional quality of three varieties of common beans (*Phaseolus vulgaris* L.) from Madagascar. *Legume Science*, 4, e143. https://agritrop.cirad.fr/601715/ 7/601715.pdf

Razola-Díaz, M.C., Gómez-Caravaca, A.M., Guerra-Hernández, E.J., Garcia-Villanova, B. and Verardo, V., 2022. New advances in the phenolic composition of Tiger-nut (*Cyperus esculentus* L.) by-products. *Foods*, 11, 343. https://doi.org/10.3390/foods11030343

Shakir, U., Inam, U., Robeena, N., Mohammad, S., Mohsin, I. and Fozia, A., 2019. Phytochemicals screening and chromatographic separation of bioactive compound from the roots of Berberis lyceum. *Journal of Biotechnology & Bioinformatics Research*, 1, 1, 1–5.

Shraim, A.M., Ahmed, T.A., Rahman, M. & Hijji, Y.M. (2021). Determination of total flavonoid content by aluminum chloride assay: A critical evaluation. *LWT – Food Science and Technology*, 150, 111932.

Umaru, H.A., Umaru, I.J., Aminu, A. and Umaru, K.I., 2018. Influence of different processing methods on proximate and anti-nutritional value of Tiger-nuts (*Cyperus esculentus* L.). *GSC Biological and Pharmaceutical Sciences*, 3, 3, 29–34

Usman, D.D., Adanu, E.O., Jahun, B.G. and Ibrahim, K., 2019. Effect of moisture content variation on thermo-physical properties of brown variety Tiger-nut (*Cyperus esculentus*). *ARID Zone Journal of Engineering, Technology & Environment*, 15, 3. 714–724.

Velavan, S., 2015. Phytochemical techniques – A review. World Journal of Science and Research, 1, 2, 80–91.

Yamin, R., Mistriyani, S., Ihsan, S., Armadany, F. I., Sahumena, M.H. & Fatimah, W.O.N. 2021. Determination of total phenolic and flavonoid contents of jackfruit peel and in vitro antiradical test. *Food Research*, 5, 1, 84 – 90.

Yu, Y., Lu, X., Zhang, T., Zhao, C., Guan, S., Pu, Y. and Gao F., 2022. Tiger-nut (*Cyperus esculentus* L.): nutrition, processing, function and applications. *Foods*, 11, 4, 601. doi: 10.3390/foods11040601. PMID: 35206077; PMCID: PMC8871521