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# Tiger nut (*Cyperus esculentus* L.) oil: A review of bioactive compounds, extraction technologies, potential hazards and applications

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ARTICLE INFO	A B S T R A C T
<i>Keywords:</i> Tiger nut oil Extraction technologies Flavor Potential hazard Applications	Tiger nut is a tuber of a plant native in the Mediterranean coastal countries, which is of great interest in food industry due to its richness in carbohydrates, lipids, starches, minerals, etc. Recent studies have focused on the analysis of the phytochemical composition of tiger nut, including six essential nutrients, polyphenols, and the extraction of proteins, starches, and phenolic compounds from the by-products of tiger nut milk 'horchata'. Few works were focused on the possibility of using tiger nut oil, a nutritious oil comparable to olive oil, as an edible oil. Therefore, this review discussed some extraction technologies of tiger nut oil, and their effects on the properties of oil, such as bioactive compounds, oxidative stability and potential hazards. The information on the emerging applications of tiger nut oil was summarized and an outlook on the utilization of tiger nut oil by-products were also reviewed.

## 1. Introduction

Tiger nut (*Cyperus esculentus* L.) is a tuber of the underutilized perennial sedge plant, which is considered to originate in northeast Africa, Eastern Mediterranean. In ancient Egypt, the tiger nut was already cultivated as food (Pascual et al., 2000; Bamishaiye & Bamishaiye, 2011). Its' tuber was found in Pharaoh's tombs for embalming bodies (Pascual et al., 2000; Bamishaiye & Bamishaiye, 2011). Tiger nuts, not really nuts, are just 1–2 cm long almond-like tubers that have a globular or egg-like shape, when they are wet. An irregular appearance was found after dried (Maksim et al., 2021). Tiger nuts also have other names, for instance earth-almond, chufa, edible galingale, yellow nutsedge, Zulu nuts and rush nuts (Pascual et al., 2000; De-Vries, 1991).

Tiger nut has a high nutritional value, which contains 6.08-9.70% protein, 4.53-17.50% fiber, 19.79-37.83% fat and 30.90-59.18% carbohydrate (Nwosu et al., 2022; Gadanya et al., 2021; Oladele & Aina, 2007; Nina, 2019). The contents of lipid, protein, and carbohydrate are higher than those typical tuber crops like potato, cassava and sweet potato (Sánchez-Zapata et al., 2012; Coşkuner et al., 2002). Tiger nut is also rich in minerals, such as, sodium, magnesium, manganese, iron, potassium and calcium, among which potassium is the highest, up to 4,478.76 mg kg<sup>-1</sup> (Oluwakemi et al., 2021). And the high calcium content present in tuber is also sufficient for the formation of bone and

teeth in babies (Oladele & Aina, 2007). Besides, tiger nut tuber also has some medical values thousands of years ago (Negbi, 1992). It is generally believed that the consumption of tiger nut has a positive influence on activating blood circulation and preventing heart disease and cancer (Chukwuma et al., 2010). The tuber also helps reduce the risk of hypercholesterolemia and diabetes, and is benefit for human's gastrointestinal health (Viuda-Martos et al., 2010). Some bioactive compounds, such as alkaloids, tannin, phytates and flavonoids, have also been found. Some positive effects have also been reported in animal experiments in recent years. Tiger nut extract has been shown to facilitate the biosynthesis of testosterone, and prevent DNA damage in hepatocytes and against memory loss (Udefa et al., 2020; Sobhy et al., 2015; Umukoro et al., 2020). There might be due to the antioxidant properties provided by quercetin, vitamin E and vitamin C in tiger nut extract, which improve the antioxidant status of the cellular environment and thus alleviate problems caused by oxidative stress.

Tiger nut is mostly used in food industry because of the high edible value. In Spain, it has been served as a milk-like beverage called 'Horchata'. For the past few years, the popularity of horchata has expanded to other countries, such as Britain, France, America and China (Sánchez-Zapata et al., 2012; Alberto et al., 1972). In China, tiger nut milk has been used to promote normal menstrual period and cure dental ulcers. Tiger nut has a unique flavor, and its flour can be added to baits

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to improve its' taste and moisture (Bamishaiye & Bamishaiye, 2011). It has also been successfully applied as a caffeine-free coffee alternative and flavoring agent in ice cream and biscuits (Cantalejo, 1997).

However, the main research for tiger nut was focused on its biochemical, mineral composition, and pharmacology properties in recent years, which were concentrated on the processing of tiger nut beverage, tiger nut milk and their by-products as food products. As a matter of fact, tiger nut is one kind of plant species known to date that can store oil at large amounts in its tuber tissues, up to 35% (Ji et al., 2020). Tiger nut oil has a distinctive flavor with a sweet, nutty aroma. The oil is golden in color due to the high contents of pigments and phenolic substances (Bamishaiye & Bamishaiye, 2011). The fatty acid composition of tiger nut oil is similar with olive oil, and they are both rich in monounsaturated fatty acids and few polyunsaturated fatty acids. These suggested that tiger nut oil can be used as an alternative for olive oil (Pascual et al., 2000; Linssen et al., 1988). Besides these, tiger nut oil also contains many bioactive compounds, such as, vitamin E, phytosterol, and polyphenol (Ezeh, Niranjan & Gordon, 2016). And the content of phenolic substances in the cold pressed tiger nut oil was 3.3 times than that of sunflower oil (Rehab & Anany, 2012). The high contents of these natural antioxidants and monounsaturated fatty acids resulted in a good oxidative stability of tiger nut oil, which make the oil use as a good frying oil. Some studies have also shown that its physicochemical properties make it possible as a lubricant (Pascual et al., 2000; Nwosu et al., 2022). Furthermore, it was also used in the production of cosmetic, flavor additive and perfumed soap, which was attributed to the presence of aroma components, such as vanillin in tiger nut oil (Rehab & Anany, 2012; Lasekan, 2013).

Despite of some advantages of tiger nut oil in industrial applications, it is necessary to consider the impact of processing techniques, such as drying, pretreatment, extraction, and refining on oil quality, just like other vegetable oils. In general, the physicochemical and functional properties, fatty acid composition, and trace nutrient contents of the oil were changed during different processing technologies. On the one hand, the optimization of the process can improve the recoveries of oil and bioactive substances. However, under the unsuitable or dramatic processing conditions, some potentially harmful substances were formed, for example, polycyclic aromatic hydrocarbons (PAHs), 3monochloropropane-1,2-diol esters (3-MCPDEs) and glycidyl esters (GEs), etc. Therefore, in this paper, the effect of the processing of tiger nut oil on the oil quality was systematically discussed. And some possible hazards that have not yet been studied but may be formed during processing were also discussed. Finally, we summarized the potential applications of tiger nut oil with the aim of increasing the world's interest in this novel and underutilized oilseed crop.

## 2. Drying of tiger nut

Oilseeds are always dried to prolong their shelf life and prevent microbial contamination before processing. Freshly harvested tiger nut tuber has a high moisture content in the range of 32-42% (Abano & Amoah, 2011; Ibeogu & Eze, 2022). As a result, tiger nut is susceptible to spoilage, which has a serious impact on its nutritional properties and the quality of the oil. In addition, the moisture content of the tuber also has a significant impact on their physical properties, such as particle density, porosity and coefficient of friction, which have a considerable impact on the selection of subsequent processing equipment (Abano & Amoah, 2011). Moreover, it also showed that the moisture content of tiger nut has an effect on the oil recovery rate (Adejumo & Salihu, 2018; Ezeh, Gordon & Niranjan, 2016). At present, the commonly used drying methods are the traditional sun drying, and modern solar drying, hot air drying, and vacuum drying. The effect of four drying methods on indicators related to the quality of the oil, such as acid value, peroxide value, oil extraction rate, and lipid concomitant nutrient contents were

studied by Zhang, Jia, Li, Liu, Wei & Zhu. (2022). They found that drying method had a significant effect on the color, phytosterol and pigment contents of tiger nut oil, and vacuum drying is a good method for tiger nut drying. Notably, they found that the oxidative stability of tiger nut oil obtained by microwave drying was significantly higher than other drying methods. These were attributed to the production of the Maillard reaction products, which have strong antioxidant properties.

## 3. Extraction technologies of tiger nut oil

Oil extraction is the key step in the processing of oilseeds. The oil extraction techniques are mainly divided into the conventional and emerging technologies. The conventional technologies include mechanical expression (ME) and solvent extraction (SE), and the new developing technologies are supercritical fluid extraction (SFE) and aqueous enzymatic extraction (AEE). However, there are some issues with these methods. Although the quality of oil extracted by ME method is good, the extraction rate is low. SE method has the highest oil yield. However, there are some safety and environmental issues, due to the use of organic solvents. SFE and AEE methods are green and safe. However, the high cost and low efficiency limited their applications.

Pretreatment of oilseeds before oil extraction can solve some issues. The pretreatment methods currently reported in tiger nut as well as other oilseeds are soaking, roasting, germination fermentation, microwave processing, ultrasonic processing, enzyme treatment and high pressure treatment. Some reports found that these pretreatments could increase the oil recovery rate, nutrient contents, oxidation stability and antioxidant properties of the oil, and even form some new functional chemicals (Kaseke et al., 2021; Miao et al., 2022). The current technologies used to extract tiger nut oil are summarized in Table 1.

## 3.1. Solvent extraction method

Generally, achieving the highest oil extraction rate is the primary goal. As is known to all, the highest oil yield was obtained by SE method, which make it as a standard method for measurement of the oil content of oilseeds. The method has been widely used in industry (Aljuhaimi et al., 2018). For tiger nut oil, the highest oil yield of SE method reported in the literature was up to 41.2 g 100  $g^{-1}$  (Lasekan & Abdulkarim, 2012). The type of organic solvent also affected the oil yield. Yoon (2015) found that 21.92 g 100  $g^{-1}$  oil yield was obtained using the mixed solvents of chloroform and methanol (2:1, v/v), followed by 19.96 g 100 g<sup>-1</sup> for *n*-hexane and 18.64 g 100 g<sup>-1</sup> for diethyl ether. Other researchers found that the mixture of petroleum ether and acetone also worked well (Aljuhaimi et al., 2018). Controlling moisture was also an important key for SE method. Adejumo & Salihu (2018) found that the oil yield was decreased from 19.05 g 100 g<sup>-1</sup> to 12.14 g 100 g<sup>-1</sup> as the moisture content increased from 20% to 40%. The maximum oil yield of 25.89 g 100  $g^{-1}$  was obtained with 9.5% moisture content. Although the oil extraction rate of SE method was high, it was timeconsuming, generally requiring 3 to 6 h. Hu et al. (2018) solved this problem by combining microwave treatment and SE method to shorten the extraction time to 55 min, and the oil yield was still as high as 24.12  $g 100 g^{-1}$ .

## 3.2. Mechanical expression extraction method

ME is another conventional method of oil extraction. It is a green and safe method. It can be divided into cold pressing and hot pressing. Cold pressing is one kind of green oil extraction method in which the whole process is controlled in a mild state (generally not more than 60°C and without organic solvents or heat pretreatment of oilseeds). Although its oil yield is not as high as that of hot pressing, the high-quality oil with low acid value, good retention of nutrients and flavor is produced (Miao

Extraction technologies applied to tiger nut oil.

Origin of tiger nuts	Extraction methods	Process conditions	Key findings	Oil yield	References
Bauchi, Nigeria	SCDE	pressure (20–40 MPa), temperature (40-80°C) for (60–360 min)	SCDE was selective for the extraction of fatty acids.	63.8%	(Lasekan & Abdulkarim, 2012)
Fayoum, Egypt	SE	pretreatment: blanching (100°C water for 45 min), roasting (oven, $100 \pm 5$ °C for 30 min), soaking (25 ± 2°C water for 48 h)extraction: solid–liquid ( <i>n</i> -hexane) ratio1:3 (w/v)	The three pretreatments had little effect on oil quality.	_	(Adel et al., 2015)
Spain	ME	pretreatment: high pressure (50, 300, 500 and 700 MPa, 40°C for 15 min), enzymatic treatment (enzyme concentration of 0.5,1 and 1.5%, solid–liquid ratio 3:5 (w/v), pH = 8 and 40°C for 6 h) extraction: maximum pressure of 38 MPa, 30 min	High pressure pretreatment did not increase the oil yield, but enzymatic treatment did.	90%	(Ezeh, Gordon & Niranjan, 2016)
Gyeonggi-do, Korea	SE	diethyl ether, n-Hexan and Chloroform/methanol (2:1 v/v) for 16 h	The oil yield was affected by the slovent types, but the physicochemical property of the oil was not.	21.92 g/ 100 g	(Yoon, 2015)
Valencia, Spain	GAME	$\rm CO_2$ pressure (10, 20 and 30 MPa), $\rm CO_2$ flow rate 8.5 kg $\rm h^{-1}$ and $\rm H_2O$ pump pressure (10, 20 and 30 MPa) for 2 h	GAME had a higher extraction efficiency; GAME increased the polyphenol contents in the oil.	80%	(Koubaa et al., 2015)
	SCDE	$\rm CO_2$ pressure (10, 20 and 30 MPa), $\rm CO_2$ flow rate 8.5 kg $h^{-1}$ for 2 h			
Spain	EAP	enzyme pretreatment: enzyme concentration of 1%, solid–liquid ratio 1:1.7, 40°C for 6 h. pressing: maximum pressure of 38 MPa, 30 min	EAP increased the total phenolic content of the oil, especially <i>trans</i> -ferulic acid; EAP meals had the highest total DP3 and DP4 sugar contents of 82.5	89.3%	(Ezeh, Niranjan & Gordon, 2016)
	HPP-AEE	retreatment: mixture of water and 1,2-propanediol (70:30, v/v) as pressure transmitting fluid, 300 MPa and 25°C for 20 min. extraction: enzyme concentration of 1% (w/w), solid–liquid ratio 1:4, pH 8 for 4 h.	mg g <sup><math>-1</math></sup> .	-	
Niger, Nigeria	SE	sample moisture content (9.5, 20, 30 and 40%), solvent ( <i>n</i> -hexane), 30°C for 5–6 h	The moisture content of the sample significantly affect oil yield and some physicochemical properties of the oil.	25.89 g/ 100 g	(Adejumo & Salihu, 2018)
Hebei, China	MAE	microwave power (250–450 W), liquid–solid ratio (4–8 mL g $^{-1}$ ), temperature (65-85°C) for (40–60 min)	The oil obtained by MAE has higher nutrient contents, better physicochemical properties and oxidative stability.	80.27%	(Hu et al., 2020)
Mersin, Turkey	RASE	pretreatment: oven, $130^{\circ}$ C for 20 min. extraction: solvents (petroleum ether and <i>n</i> -hexane) for 5 h	Both solvent types and heat treatment affect the physicochemical properties of the oil; Heat treatment caused the loss of tocopherols.	-	(Aljuhaimi et al., 2018)
Neimenggu, China	MUAAEE	particle size (600–300 $\mu$ m), ultrasonic power (300–400 W), microwave power (300–400 W), radiation temperature (40–50 °C), radiation time (30–40 min), enzyme concentration (2–3%), enzymolysis temperature (30–40 °C), pH (3.5–4.5), liquid–solid ratio (10–12 mL g <sup>-1</sup> ) and enzymolysis time (180–240 min)	<ul> <li>(600–300 μm), ultrasonic power</li> <li>(b), microwave power (300–400 W),</li> <li>mperature (40–50 °C), radiation time</li> <li>h), enzyme concentration (2–3%),</li> <li>temperature (30–40 °C), pH (3.5–4.5),</li> <li>ratio (10–12 mL g<sup>-1</sup>) and enzymolysis</li> </ul>		(Hu et al., 2020)
Xin jiang, China	ME-SBE	Pressing temperature: ambient temperature SBE condition: 2400 mL of <i>n</i> -butane, 40°C for 50 min	The oil obtained by ME-SBE method has highest retention rate of total tocopherols and total	28.47 g/ 100 g	(Guo et al., 2021)
	ME-SCDE	Pressing temperature: ambient temperature SCDE condition: high purity CO <sub>2</sub> (99.99%), 40°C, 28 MPa for 90 min.	phenolic compound contents.	28.56 g/ 100 g	
Baoding, China	ME	Pressing times (1 and 3 times), pressure (40 and 60 MPa), for 60 min	The oil recovery increased with the increase of pressing pressure and number of presses; Cold pressing has less effect on the properties of the starch in the meals.	82.34%.	(Miao et al., 2022)
Hei Long jiang, China	MASBE	pretreatment: microwave oven, 560 W for (4–12 min) SBE condition: number of extraction cycles(1–5), liquid–solid ratio $(1-9 \text{ mL g}^{-1})$ , temperature (25-60°C) for (10–50 min)	Microwave treatment significantly increased the SBE efficiency.	96.9%	<b>(</b> Cai et al., 2022 <b>)</b>

SCDE: Supercritical carbon dioxide extraction; SE: solvent extraction; ME: mechanical expression; GAME: gas assisted mechanical expression; EAP: enzyme aided pressing; HPP-AEE: high pressure pretreatment aqueous enzymatic extraction; MAE: microwave-assisted extraction; RASE: roasting aided Soxhlet extraction; MUAAEE: microwave-ultrasonic assisted aqueous enzymatic extraction; ME-SBE: mechanical expression with subcritical *n*-butane extraction; ME-SCDE: mechanical expression with supercritical carbon dioxide extraction; MASBE: microwave assisted subcritical *n*-Butane extraction.

et al., 2022; Aydeniz et al., 2014). Furthermore, the oilcake, a byproduct of cold pressing, is rich in the nutrients, including high quality carbohydrates, fiber, protein, antioxidant ingredient and volatile constituents, which can be extracted separately as products (Miao et al., 2022; Karaman et al., 2015).

Currently, cold pressed tiger nut oil is commercially available as edible oil in China. However, the oil yield of cold pressing method is low. Miao et al. (2022) increased the oil recovery rate by increasing the pressing pressure and times of pressing. The oil recovery was increased by 4.96% and 8.38%, when the pressure was increased from 40 to 60

MPa and the times of pressing were increased from 1 to 3 times, respectively. In addition to the optimization of the pressing process, the mild pretreatment techniques can also be applied before cold pressing, such as high pressure and enzyme treatment (Patil & Singh, 2017). However, high-pressure pretreatment worked well in other oilseeds, but not in tiger nut (Ezeh, Gordon & Niranjan, 2016). This may be due to the presence of the dimeric structures of ferulic acid cross-linked arabinoxylans between cells and inside the cell wall, which resulted in the hard cell structure of tiger nut (Parker et al., 2000). Accordingly, the authors used enzymatic pretreatment to degrade cellular wall

components and cold pressing to increase the oil recovery to 90%. Moreover, this enzymatic pressing method did not have the emulsification issue in the process of oil extraction by AEE, which saves resources by eliminating the demulsification (Ezeh, Gordon & Niranjan, 2016).

Unlike cold pressing, hot pressing generally requires a thermal preprocessing prior to pressing (Dun et al., 2019). It is considered to have higher recoveries of oil and bioactive substances than cold pressing, because heat treatment can change the cellular structure of the oilseeds and facilitate their releases (Suri et al., 2021). The thermal preprocessing techniques mainly include conventional roasting, steaming and the emerging microwave and infrared treatment. Compared to traditional thermal treatment, the microwave roasting is time-saving and energy efficient, because it can heat the material from inside and outside (Lukešová et al., 2009). However, thermal treatments should also be controlled, because excessive heating might result in the superheating and the carbonization of plant materials, and a poor oil extraction yield (Liu et al., 2018). Zhang et al. (2023) found that the tiger nut oil yield of pressing after heat treatment at 180 °C for 20 min was lower than that of the cold pressing. The authors suggested that this might be due to the changes of starch structure caused by the dextrinization and Maillard reactions at high temperatures, which made the starch bind more tightly to the oil and restrict the free migration of the oil. Therefore, the heat treatment process should be optimized. However, as far as we know, there are no literature on tiger nut. Tiger nuts are rich in carbohydrates, which can be used as the precursor substances for caramelization and Maillard reaction to enhance the flavor and the antioxidant capacity of the oil (Liu et al., 2011).

## 3.3. Aqueous enzymatic extraction method

AEE method is a safe and environmental-friendly emerging oil extraction technology. The cell wall and internal structure of the plant material were destroyed through enzymatic degradation, and the cell inclusions such as proteins, oil and small molecule active compounds were released (Liu et al., 2020; Prommaban et al., 2021). Because of the special mechanism and benign operating conditions, high quality protein, cellulose and polysaccharide could be obtained simultaneously after the oil extraction. In addition, the phospholipids were also separated from the oil with the aid of water, which could save some oil refining steps (Yusoff et al., 2016).

The screen of enzymes is crucial, which should be conformed to the characteristics of the oilseeds. Alkaline protease, widely used in some oilseeds, was also applied to the extraction of tiger nut oil. However, the oil yield was only 14.6 g  $100 \text{ g}^{-1}$  (Zhang et al., 2023). The reason for the low oil yield may be due to the low protein content of tiger nut, and the single protease enzyme can't completely destroy the cell structure, thus limiting the release of oil. The combination of the  $\alpha$ -amylase, alcalase and celluclast was used to break the tiger nut cell wall, and 61.3% oil recovery was achieved (Ezeh, Niranjan & Gordon, 2016). However, this oil yield is still unsatisfactory. This may be due to the fact that the mixed enzymes themselves are proteins, and alcalase as an alkaline protease can inactivate other enzymes, which resulted in a low oil extraction rate. The optimization of key control points and the combination of multiple technologies could solve the low oil yield and time-consuming issues. Hu et al. (2020) used microwave-ultrasonic to assisted AEE of tiger nut oil. The oil recovery of 85.23% was achieved with only 30 min.

Besides, there are some other issues of AEE method, particularly emulsification of the system that severely limited the oil yield. Some researchers believed that emulsion removal was facilitated if the upstream process was optimized to produce a less stable emulsion. Furthermore, some specialized de-emulsifying methods, such as saltassisted microwave, freeze and thaw treatment, solvent and surfactant-assisted, were also studied. Another issue is the high cost of the enzyme production. For this issue, enzyme recycling is a development direction. It has been found that the enzyme could be recycled for three times and the oil recovery rate was still higher than 80% in sacha inchi seed oil extraction (Nguyen et al., 2020). In this regard, the immobilization of enzymes can improve the stability of enzymes and facilitate their recycling. However, no available information was reported in tiger nut oil extraction, which should be given more attention in future.

### 3.4. Supercritical and subcritical fluid extraction methods

Another green, non-toxic, non-flammable and safe method is SFE technology, as an alternative to SE and ME methods. The most widespread SFE is supercritical carbon dioxide extraction (SCDE) method with CO<sub>2</sub> as a solvent. CO<sub>2</sub> is an excellent solvent for the extracting lipidsoluble ingredients, which has high oil recovery (Ramazanov & Shakhbanov, 2019). The potential of using SCDE to extract tiger nut oil was evaluated by Lasekan & Abdulkarim (2012). The oil recovery of 63.79% was achieved after 210 min of extraction at 60°C and 30.25 MPa. Koubaa et al. (2015) found that the combination of the ME and SCDE could avoid the long-time consuming issue. The results showed that this gas-assisted mechanical expression (GAME) could achieve 80% oil recovery within only 120 min. However, the use of CO<sub>2</sub> as a critical fluid also has some disadvantages. One is that the partial dissolution of CO<sub>2</sub> might cause high free fatty acid value and also affected the oil yield. Second is that supercritical fluid was selective for fatty acid extraction, and high pressure was also required for a good dissolution of triacylglycerol in the critical CO<sub>2</sub> (Guo et al., 2021; Trentini et al., 2017).

Subcritical *n*-butane extraction (SBE) method is free of these concerns. The SBE method requires less critical pressure and temperature, and the solvent *n*-butane has the properties of high density, high diffusivity and better solubility of triacylglycerol compared to  $CO_2$  (Sun et al., 2018). It has been reported that oil yield could reach 28.47 g 100 g<sup>-1</sup> using SBE, and this extraction process could be finished in 50 min (Guo et al., 2021). Microwave pretreatment SBE was used by Cai et al. (2022) to improve the oil yield and extraction efficiency, and 96.93% oil recovery was achieved in 32 min. In addition, no *n*-butane solvent residue was found in the tiger nut oil, which indicated that microwave pretreatment SBE is a safe and green method.

## 4. Fatty acid composition and triacylglycerol profile of tiger nut oil

The fatty acid composition of tiger nut oil is influenced by many factors. The species and geographical origin of tiger nut were shown to have a significant influence on the fatty acid profile of tiger nut oil (Nina et al., 2021). Besides, climate, edaphic condition, harvest season and maturity of crop also changed the fatty acid constituent of the oil (Yu et al., 2022). Table 2 shows the fatty acid profile of tiger nut oil obtained using different extraction processes. The main fatty acid of tiger nut oil was monounsaturated fatty acid oleic acid, with a content of 64.4-75.60%. Other fatty acids were saturated fatty acids palmitic acid (11.65–16.60%) and stearic acid (2.0–6.6%), and polyunsaturated fatty acid linoleic acid (8.28-13.40%). These showed that tiger nut oil is similar to that of olive oil. The low content of polyunsaturated fatty acids made it less susceptible to oxidation and has a good storage stability. The predominant triacylglycerols of tiger nut oil are triolein (OOO) and palmitodiolein (POO) with the contents of > 60% (Table S1). The content of other triacylglycerols is strongly influenced by the origin of tiger nuts (Linssen et al., 1988; Dong et al., 2023; Hu et al., 2020). The sn-2 position fatty acids of triacylglycerols have a strong relationship with human digestion and absorption. It is worth noting that the fatty acids in the sn-2 position of triacylglycerols of tiger nut oil are mainly oleic acid and linoleic acid, with the contents of 75.1-77.5% and 18.5-23.1%, respectively (Linssen et al., 1988; Yeboah et al., 2012; Kim et al., 2007). The high contents of unsaturated fatty acids in the sn-2 position of tiger nut oil made it as a healthy and nutritious edible oil (Sánchez-Zapata et al., 2012; Yeboah et al., 2012).

Fatty acid composition (%) of tiger nut oil by different extraction methods.

Extraction methods	Pretreatment methods	Palmitic acid C16:0	Palmitoleic acid C16:1	Stearic acid C 18:0	Oleic acid C18:1	Linoleic acid C18:2	Linolenic acid C18:3	Arachidic acid C20:0	Significance of difference	References
SE	soaking	15.14 $\pm$	$0.30\pm0.25$	5.31 $\pm$	68.97	$\textbf{8.37} \pm$	$0.20\pm0.33$	ND	NSD	(Adel et al.,
		0.33		0.22	$\pm 0.02$	0.18				2015)
	blanching	15.21 $\pm$	$0.33\pm0.34$	5.16 $\pm$	69.09	8.42 $\pm$	$0.19\pm0.28$	ND		
		0.38		0.20	$\pm 0.12$	0.12				
	roasting	15.05 $\pm$	$0.28\pm0.18$	5.20 $\pm$	69.33	8.28 $\pm$	$0.20\pm0.30$	ND		
	-	0.12		0.26	$\pm 0.06$	0.04				
ME	enzymolysis	14.5 $\pm$	$0.3\pm0.00$	$6.6 \pm$	66.0 $\pm$	11.0 $\pm$	$0.1\pm0.01$	$0.6\pm0.01$	NSD	(Ezeh, Niranjar
		0.08		0.08	0.04	0.07				& Gordon,
AEE	raw	$13.9 \pm$	$0.0\pm0.00$	6.4 ±	66.1 $\pm$	11.6 $\pm$	$0.1\pm0.00$	$0.7\pm0.01$		2016)
		0.06		0.04	0.07	0.05				
	high pressure	13.7 $\pm$	$0.3\pm0.00$	$6.2 \pm$	66.0 $\pm$	12.0 $\pm$	$0.1\pm0.00$	$0.7\pm0.01$		
	0 1	0.00		0.03	0.41	0.05				
ME-SBE	raw	12.5 $\pm$	ND	4.90 $\pm$	73.81	$8.89 \pm$	$0.34\pm0.00$	ND	NSD	(Guo et al.,
		0.02		0.01	$\pm 0.05$	0.04				2021)
ME-SCDE	raw	$12.09~\pm$	ND	4.86 $\pm$	73.91	8.84 $\pm$	$0.30\pm0.00$	ND		
		0.00		0.00	$\pm 0.00$	0.00				
MUAAEE	raw	11.86 $\pm$	$0.25\pm0.00$	$2.37~\pm$	74.73	9.46 $\pm$	$0.20\pm0.00$	$0.42\pm0.00$	NSD	(Hu et al.,
		0.23		0.07	$\pm 0.46$	0.18				2020)
SE	raw	$11.65 \pm$	$0.26\pm0.00$	$2.43 \pm$	74.52	$9.63 \pm$	$0.21\pm0.00$	$\textbf{0.45} \pm \textbf{0.00}$		
		0.28		0.05	$\pm 0.51$	0.12				
MAE	raw	12.08 $\pm$	$0.22\pm0.00$	$2.24 \pm$	75.60	8.85 $\pm$	$0.19\pm0.00$	$0.41\pm0.00$	NSD	(Hu et al.,
		0.16		0.44	$\pm 0.58$	0.13				2018)
SE	raw	$12.07~\pm$	$0.28\pm0.00$	$2.14 \pm$	74.75	9.47 ±	$0.22\pm0.00$	$\textbf{0.40} \pm \textbf{0.00}$		
		0.23		0.05	$\pm 0.65$	0.18				
SCDE	raw	$15.60 \pm$	$0.46\pm0.0$	ND	69.69	12.81 $\pm$	$0.26\pm0.0$	ND	SD	(Lasekan &
		3.5			$\pm$ 8.1	2.1				Abdulkarim,
SE	raw	16.60 $\pm$	$0.57\pm0.0$	ND	66.30	13.40 $\pm$	$0.19\pm0.0$	ND		2012)
		2.4			$\pm$ 7.3	3.2				
SE	raw	12.26 $\pm$	ND	3.41 $\pm$	72.67	10.24 $\pm$	$0.11\pm0.00$	$0.45\pm0.01$	SD	(Özcan et al.,
		0.06		0.08	$\pm 0.06$	0.03				2021)
	sprouting	$11.83 \pm$	ND	$3.65 \pm$	72.47	10.49 $\pm$	$0.15\pm0.00$	$0.51\pm0.01$		-
		0.07		0.01	$\pm 0.06$	0.03				
SE	raw	$15.67 \pm$	$0.35\pm0.07$	5.38 ±	67.47	$11.03 \pm$	$0.42\pm0.03$	$0.58\pm0.09$	SD	(Aljuhaimi
		0.73		0.23	$\pm 1.27$	0.54				et al., 2018)
	roasting	$17.86 \pm$	$0.51\pm0.03$	5.93 $\pm$	68.16	$11.56 \pm$	$0.49\pm0.07$	$0.67\pm0.05$		
		1.17		0.28	$\pm 1.19$	0.47				

NSD: no significant difference; SD: significant difference; ND: not detected; SCDE: supercritical carbon dioxide extraction; SE: Soxhlet extraction; ME: mechanical expression; AEE: aqueous enzymatic extraction; MAE: microwave-assisted extraction; MUAAEE: microwave-ultrasonic assisted aqueous enzymatic extraction; ME-SBE: mechanical expression with subcritical *n*-butane extraction; ME-SCDE: mechanical expression with supercritical carbon dioxide extraction.

## 4.1. Effect of extraction methods on fatty acid composition of oil

## process (Özcan et al., 2021).

5. Bioactive compounds in tiger nut oil

Table 2 shows that most processes applied to the tiger nuts did not significantly affect the fatty acid profile of the tiger nut oil. These indicated that the oil extraction process was not selective for fatty acids, which was beneficial from a nutritional perspective. However, it is still controversial whether the fatty acid composition of tiger nut oil is changed by processing. The fatty acid profile of tiger nut oil extracted by the SCDE method was compared with that of SE method by Lasekan & Abdulkarim (2012). They found that there were some significant differences between the oils extracted by the two methods. Several main fatty acid contents were increased with increasing temperature at 40 MPa. The authors noted that this was due to the high selectivity of the supercritical carbon dioxide fluid for these fatty acids. In the application of the SE method, it had been found that the type of solvent also had an influence on the fatty acid profile (Aljuhaimi et al., 2018; Yoon, 2015). Some intense processing techniques, such as roasting, also affected the fatty acid composition of tiger nut oil (Aljuhaimi et al., 2018; Adel et al., 2015). The excessive microwave treatment could result in a decrease in linoleic acid content and the increase in oleic, palmitic and stearic acid contents for rapeseed oil. These were due to the thermal degradation of polyunsaturated fatty acids (Rekas et al., 2017). But this phenomenon was not significant in tiger nut oil (Adel et al., 2015). In addition, the sprouting pretreatment also significantly altered the fatty acid composition of tiger nut oil compared to raw tiger nuts, which was due to the changes in the basic composition of the seeds during the germination

The quality of the oil is relative to its own physicochemical property and the contents of mineral, vitamins, and small fat-soluble bioactive substances. The main bioactive substances currently reported in tiger nut oil were summarized (Fig. 1).

Tocopherols are a group of phenolic compounds naturally present in plant oils, and have been reported to have antioxidant, antibacterial, and anti-tumor effects (Zhang, Zhu, Chen, Su, Chen & Cao, 2022). Tiger nut oil was rich in tocopherols, with a total tocopherol content of 142–348.9 mg kg<sup>-1</sup>. The predominant tocopherol in tiger nut oil was  $\alpha$ -tocopherol (110.3–329.76 mg kg<sup>-1</sup>), which was similar to olive oil  $(45.4-414.5 \text{ mg kg}^{-1})$ , but they were lower than sunflower oil (708–780) mg kg<sup>-1</sup>) and rapeseed oil (91–973 mg kg<sup>-1</sup>). The content of  $\beta$ -tocopherol in tiger nut oil was 27.8–74.3 mg kg<sup>-1</sup>, which was significantly higher than that of other vegetable oils. The contents of  $\delta$ -tocopherol and  $\gamma$ -tocopherol in tiger nut oil were both very low (Ezeh, Niranjan & Gordon, 2016; Fine et al., 2016; Guo, Wan, Huang & Wei, 2021; Hu et al., 2018; Qian et al., 2022; Zhang et al., 2023; Zhang, Jia, Li, Liu, Wei & Zhu, 2022). However, Aljuhaimi et al. (2018) found that one tiger nut oil with relative high levels of  $\gamma$ -tocopherol and  $\delta$ -tocopherol was found in Turkey, ranging from 342.7 to 364.4 mg kg  $^{-1}$ and 507.7–549.1 mg kg $^{-1}$ , respectively. The possible reasons for these results may be due to the varieties of tiger nut, growth environment and

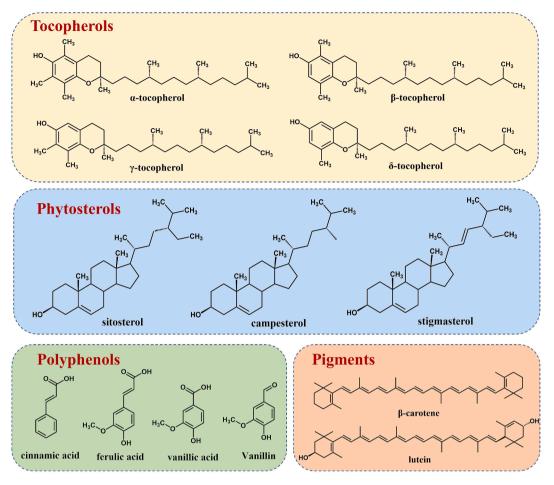


Fig. 1. Bioactive substances in tiger nut oil.

extraction methods. Besides, four kinds of tocotrienols were also found in tiger nut oil at low concentrations (Zhang, Jia, Li, Liu, Wei & Zhu, 2022). Tiger nut oil is rich in polyphenols, and 78 of which have been identified, including catechol, vanillin, and ferulic acid (Ezeh, Niranjan & Gordon, 2016; Koubaa et al., 2015). The total phenolic content of tiger nut oil was 16.5 mg GAE/100 g (mg of gallic acid equivalents per 100 g of oil), which was significantly higher than that of sunflower oil (5 mg GAE/100 g) (Rehab & Anany, 2012). Phenolics are the main chain breaking antioxidants in free radical chain reactions or in the conversion of lipid radicals to more stable molecules, which boosted antioxidant capacity of plant oil (Fruhwirth et al., 2003). Tiger nut oil also contains amounts of phytosterols. Phytosterols have a wide range of pharmacological properties, such as anti-inflammatory and anti-cancer effects (Nattagh-Eshtivani et al., 2021). The total phytosterol content of tiger nut oil was 171.42–685.68 mg 100  $g^{-1}$ . It was higher than some vegetable oils (olive oil 107.5–427.6 mg 100 g<sup>-1</sup>, peanut oil 190.4–281.6 mg 100 g<sup>-1</sup>, sunflower oil 221.2–414.6 mg 100 g<sup>-1</sup>, and soybean oil 150.0–432.8 mg 100 g<sup>-1</sup>). The main phytosterol of tiger nut oil was  $\beta\text{-sitosterol}$  with a content of 112.43–518.26 mg 100 g  $^{-1}\text{,}$  which was higher than that of the oils mentioned above. The others were stigmasterol and campesterol, with the contents of 33.22-70.42 mg 100 g<sup>-</sup> and 23.70–63.53 mg 100  $g^{-1}$ , respectively (Fine et al., 2016; Guo, Wan, Huang & Wei, 2021; Hu et al., 2018; Idrissi et al., 2022; Qian et al., 2022; Zhang, Jia, Li, Liu, Wei & Zhu, 2022). The phytosterol content of tiger nut oil was significantly varied with different origins. It has been reported that high growing temperature could lead to high phytosterol content (Ezeh et al., 2014). In addition, 2.78% squalene has also been found in the unsaponifiable matter of tiger nut oil, which was higher than olive oil (2.40%) and soybean oil (1.41%) (El-Naggar, 2016).

## 5.1. Effect of processing on bioactive compounds of oil

The content and structure of bioactive compounds were susceptible to the oil processing. The content of oil concomitants can be lost or increased, and new compounds can be generated by chemical reactions during processing. The nutrient contents of tiger nut oil obtained by different extraction technologies are summarized in Table 3.

According to Table 3, traditional oil extraction methods, ME and SE were used as control methods. Researchers tend to improve the retention of nutrients in the oil by the combination of novel techniques. The SCDE assisted ME method was used by Koubaa et al. (2015) to increase the total phenolic content (TPC) of oil by 90.2% compared to the ME method. This was due to the synergistic effect of the combination of SCDE and ME, which increased the cell disruption and the contact of supercritical fluid with phenolics. Microwave and ultrasound-assisted oil extraction have also been used widely in recent years. They disrupt the adhesion of oil and other contents such as proteins and starch by high-frequency vibrations, resulting in the release of some nutrients. They have been showed good performance in the extraction of tiger nut oil (Hu et al., 2018; Hu et al., 2020).

The application of pretreatment can also improve the recovery of nutrients. The total polyphenol content in the high-pressure pretreated oil was increased by 69.7%, and the  $\alpha$ -tocopherol content was also increased by 54.4% and to 170.3  $\mu$ g g<sup>-1</sup> (Ezeh, Niranjan & Gordon, 2016). In addition, enzyme pretreatment can release some phenolics by enzymatic digestion of specific cell structures. For tiger nut, Viscozyme L was found that it could effectively disrupt the cross-linked structure of ferulic acid and arabinoxylans, leading to a significant increase of ferulic acid content in the oil (from 1  $\mu$ g g<sup>-1</sup> to 6.2  $\mu$ g g<sup>-1</sup>) (Ezeh, Niranjan &

Bioactive compound contents of tiger nut oil obtained by different extraction technologies.

Bioactive	Extraction technologies											
compounds	ME (CM)	GAME	ME (CM)	EAP	AEE (CM)	HPP-AEE	SE (CM)	MASE	SE (CM)	MUAAEE	SBE (CM)	ME-SBE
$\alpha$ -Tocopherol (µg $g^{-1}$ )			145.7	159.5 (†) 9.5%	110.3	170.3 (†) 54.4%	253.49	315.27 (†) 24.4%	246.17	329.76 (†) 34.0%	216.5	224.0 (†) 3.5%
$\beta$ -Tocopherol (µg g <sup>-1</sup> )			29.7	27.8 (↓) 6.4%	31.7	34.7 (†) 9.5%						
$\gamma$ -Tocopherol (µg $g^{-1}$ )											82.4	82.9 (†) 0.6%
TPC ( $\mu g \text{ GAE } g^{-1}$	52.9	100.6 (†)	17.9	13.2 (↓)	14.5	24.6 (†)	58.37	92.53 (†)	57.84	83.57 (†)		
oil)		90.2%		26.3%		69.7%		58.5%		44.5%		
TPC (mg SAE 100 g <sup>-1</sup> oil)											27.91	28.79 (†) 3.15%
$\beta$ -carotene (mg kg <sup>-1</sup> )							17.15	25.43 (†) 48.3%	20.26	28.85 (†) 42.4%		
$\beta$ -sitosterol (mg 100 g <sup>-1</sup> )							432.19	518.26 (†) 19.9%	445.69	502.75 (†) 12.8%	217.70	214.17 (↓) 1.6%
Stigmasterol (mg 100 g <sup>-1</sup> )							55.86	70.42 (†) 26.1%	51.51	65.83 (†) 27.8%		
Campesterol (mg $100 \text{ g}^{-1}$ )							23.70	25.78 (†) 8.8%	23.78	30.45 (†) 28.0%	58.48	55.36 (↓) 5.3%
Brassicasterol (mg											63.28	63.98 (†)
$100 \text{ g}^{-1}$ )												1.1%
References	(Koubaa	et al., 2015)	(Ezeh, N	liranjan & Gor	don, 2016)		(Hu et al.	., 2018)	(Hu et al	., 2020)	(Guo et a	l., 2021)

CM: controlled method; ME: mechanical expression; GAME: gas assisted mechanical expression; EAP: enzyme aided pressed; AEE: aqueous enzymatic extraction; HPP-AEE: high pressure pretreatment aqueous enzymatic extraction; SE: Soxhlet extraction; MASE: Microwave-assisted extraction; MUAAEE: microwave-ultrasonic assisted aqueous enzymatic extraction; SBE: subcritical *n*-butane extraction; ME-SBE: mechanical expression with subcritical *n*-butane extraction; TPC: total phenoic content; (†): increase; (↓): decrease.

Gordon, 2016). Besides, moderate heat treatment is also believed to increase the bioactive substance content of the oil. Oven heat treatment in the range of 250–290 °C also greatly increased the total phenolic content of tiger nut from 40.73 to 300.44 mg GAE 100 g<sup>-1</sup> (mg of gallic acid equivalents per one hundred gram of extract) (Djikeng et al., 2022). Therefore, it should be expected that the concentration of phenolic compounds in the oil can be increased correspondingly after roasting process. To the best of our knowledge, there is still a blank in the study of tiger nut oil.

In conclusion, the published studies on the extraction of tiger nut oil were mainly focused on the oil extraction rate. Study on the influences of extraction methods on the nutrient contents of tiger nut oil was not available. The combination of new technologies that can solve the problems of extraction efficiency and nutrient retention, should be studied in the future.

## 6. Stability of tiger nut oil

The stability of the oil is related to the shelf life and the applicability of the oil. The stability of an oil is generally evaluated by two methods, including antioxidant activity and oxidation stability assays. Antioxidant activity is an important index to analyze the content of the antioxidant components in the oil, including natural and newly generated antioxidants (Zhang et al., 2021). The main methods used to evaluate the *in vitro* antioxidant activity of the oil were DPPH, ABTS, FRAP and ORAC assays. Among them, the ORAC assay is based on hydrogen atom transfer, and FRAP is based on single electron transfer. ABTS and DPPH are the mixed assays involving hydrogen atom transfer and single electron transfer (Prior et al., 2005). Therefore, various antioxidant tests are often used together to explore the action mechanism of antioxidants.

## 6.1. Antioxidant activity

Although the fatty acid composition of an oil has an important effect on the antioxidant activity of the oil, the content of minor bioactive compounds in the oil also showed significant effect on the antioxidant activity of the oil. For the tiger nut oil extracted with methanol, the DPPH and FRAP radical scavenging activity were significantly correlated with the total phenolic content (r = 0.846, 0.930, p < 0.001) and total tocopherol content (r = 0.736, p < 0.05; 0.864, p < 0.001), which indicated that phenolic compounds were the main bioactive compounds that contributed to the antioxidant of tiger nut oil (Guo et al., 2021). Therefore, the oil extraction processes that enhance the recovery of phenolic compounds could theoretically improve the antioxidant properties of the oil. The oil with the highest antioxidant activity was obtained by the combination of SBE and ME methods, with 51.34 µmol TE 100 g<sup>-1</sup> (µmol of Trolox equivalents per one hundred gram of oil) of DPPH radical scavenging activity and 111.19 µmol TE 100 g<sup>-1</sup> of FRAP radical scavenging activity (Guo et al., 2021). Other bioactive substances in the oil, such as flavonoids, phytosterols chlorophyll and carotenoids, also have a positive impact on the antioxidant activity (Munteanu & Apetrei, 2021). Therefore, these bioactive substances should be retained as much as possible.

The antioxidants that contribute to the antioxidant capacity are not only endogenous antioxidants but also the new generated antioxidants during processing. Maillard reaction products (MRPs) could be generated by using thermal pretreatment, such as Melanoidins, which is considered to have a certain free radical scavenging capacity (Wu et al., 2018). Therefore, although the heat treatment resulted in the losses of tocopherols and polyphenols by thermal cleavage, the antioxidant capacity increased due to the new generation of MRPs. In addition, heat treatment did not always decrease the tocopherol contents. MRPs were considered to be more active and are oxidized prior to tocopherols, which could provide the protection against tocopherols and resulted in an increasing recovery of tocopherols in the oil during extraction processing (Vaidya & Eun, 2013). Therefore, the roasting process should be optimized in order to achieve a balance between endogenous and generated antioxidants, which could form a maximum antioxidant activity. Ndiaye et al. (2022) found that the highest antioxidant activity of tiger nut flour was achieved after roasted at 147°C for 38 min, and the contents of MRPs and phenolics were both at high levels, which could provide a reference for the processing of tiger nut oil.

## 6.2. Oxidative stability

Oxidative stability reflects the overall quality of the oil, which can be

used to estimate the shelf life of the oil product. The oxidative stability of the oil is related to the fatty acid composition and the content of antioxidants. The induction period (IP) is generally used to evaluate the oxidative stability of the oil. For tiger nut oil, the IP had a strong correlation with the total content of phenolic compounds.

In general, the oils with high polyunsaturated fatty acid content are prone to become rancid. For example, the oxidation rate of linoleic acid was about ten times than that of oleic acid (Freire et al., 2012). Gao et al. (2021) found that there was a significant negative effect of linoleic acid in the walnut oil system on IP. Tiger nut oil has a high oleic acid content of 64.4-75.60% and low linoleic acid content of 8.28-13.40%, and it is also rich in tocopherols and polyphenols, which give it a good oxidative stability. For tiger nut oil, the IP had a strong correlation with the total content of phenolic compounds. Guo et al. (2021) found that there was a significant positive correlation between the IP and the total phenolic and tocopherol contents (r = 0.972, 0.824, p < 0.001). Miao et al. (2022) also found that the IP of tiger nut oil obtained by three times pressing (repeat pressing three times) was increased by 25.15% than that of one time pressing. The improvement in oxidative stability was due to the fact that more active compounds were squeezed out by increasing the times of pressing. Therefore, some oils with the low content of endogenous antioxidants were added with an appropriate content of exogenous antioxidants in order to improve their oxidative stability. However, tiger nut oil has good oxidation stability, and its IP was significantly higher than that of other vegetable oil, such as olive oil and rapeseed oil, which suggested that tiger nut oil did not require the addition of external antioxidants (Zhang, Jia, Li, Liu, Wei & Zhu, 2022). Besides, thermogravimetric analysis (TGA) can estimate the oxidation behavior of the oil at high temperature, and whether it could be used as frying oil or base oil for industrial lubricants. To our knowledge, there are no available information on this aspect of tiger nut oil, and the excellent oxidative stability of tiger nut oil might be given more attention in future.

## 6.3. Chemical indicators related to oil stability

In addition to the IP that directly reflects the oxidative stability of the oil, some other indicators, such as, acid value (AV), peroxide value (POV), *p*-anisidine value (*p*-AV) and total oxidation value (TOTOX), also reflect the quality and stability of oil. The AV of oil reflects the content of free fatty acids in the oil, which indicates the rancidity degree of the oil. The POV of oil reflects the content of peroxide and the primary oxidation product in the oil. The *p*-AV of oil reflects the content of aldehydes and the secondary oxidation product in the oil. In general, the primary oxidation products of oil can be easily decomposed to form secondary products. As time goes on, the POV of oil decreases and the *p*-AV of oil increases. Therefore, in order to accurately evaluate the degree of oil oxidation, TOTOX was introduced, a compound index calculated as 2POV + p-AV (Zhang et al., 2021).

The AV and POV of tiger nut oil were significantly affected by the moisture content of tiger nuts (Adejumo & Salihu, 2018). Thermal treatment was often used in the pretreatment stage to adjust the moisture content of the oilseeds, and thermal treatment also reduced the activity of the lipolytic enzymes, which resulted in the low AV and POV of the oil (Veldsink et al., 1999). However, unsuitable heat treatment could accelerate the oil oxidation and hydroperoxide formation, which resulted in a significant increase of POV. Similar phenomenon was also found in tiger nut oil (Aljuhaimi et al., 2018; Djikeng et al., 2022). For different extraction methods, mild operating conditions and short extraction times can reduce the exposure of the oil to oxygen and light. The AEE method has mild operating conditions. The AV and POV of the extracted tiger nut oil obtained using AEE method were both very low, and the lowest POV was only 0.13 meq  $O_2\ kg^{-1}$  (Ezeh, Niranjan & Gordon, 2016). The oil with a low AV can save the step of deacidification in the refining process. Tiger nut oil with an extremely low POV of 0.02–0.03 meq  $O_2\ensuremath{\,\text{kg}^{-1}}$  was obtained by SBE method. However, when SCDE method was used, the AV of the oil was increased to 2.86 mg KOH

 $g^{-1}$ , which is caused by the partial dissolution of CO<sub>2</sub> consuming more alkaline reagents, resulted in a high AV (Guo et al., 2021). In conclusion, novel oil extraction techniques such as AEE and SBE methods can yield oils with better oxidative stability.

## 7. Volatile compounds of tiger nut oil

Flavor plays an important role in the sensory of oil. It is an important factor in increasing consumer satisfaction. The key volatile compounds of tiger nut oil and the flavor are summarized in Table 4. According to Table 4, the flavor of raw tiger nut oil was mainly contributed by 14 aldehydes and 5 alcohols. Some critical compounds such as vanillin,  $\alpha$ -Pinene and Cedrol together contributed to the overall vanilla and sweet aroma of tiger nut oil. Mild extraction techniques have less effect on the flavor of the oil. Hu et al. (2020) compared the flavor of oil obtained by microwave-ultrasonic assisted AEE method and SE method, and found that, the flavor of oils extracted by the two methods were very similar. This was because the low temperature conditions hardly changed the chemical structure of the volatile substances. Besides, these volatile compounds are difficult to be detected by human senses due to their low quantity or high threshold.

Heat treatment of oilseeds or tubers is a widely used method to enhance the flavor of the oil. Because of the presence of carbohydrate, amino acid, and oil, during the heat treatment process, a series of complex chemical reactions happened, such as, lipid oxidation, caramelization, Maillard reaction, and Strecker degradation (Yin, Shi, Li, Ma, Wang & Wang, 2022). The intermediates of these reactions also react with each other. For example, lipid oxidation products have shown a role in the degradation of phenylalanine (Strecker degradation) (Hidalgo et al., 2005). These reactions worked together and resulted in a variety of flavor components in oil. According to Table 4, the key volatile compound species of roasted tiger nut oil were significantly higher than the oil of unroasted tiger nut. Oxidation of the oil at high temperatures can produce more small molecule aldehydes. Besides, when the roasting temperature was above 150°C, the contents of heterocyclic compounds were increased, which were contributed to the roasted nutty flavor to the oil (Gracka et al., 2016). Six pyrazine compounds (heterocyclic compound) have been detected in roasted tiger nut oil, which directly contributed to the overall caramel-like, roasty and nutty flavors of the oil. However, roasting process should be controlled. For example, if roasted temperature was higher than 220 °C, the structures of sugar and amino acid were destroyed, and the taste became burnt and bitter, which made the overall sensory evaluation very poor. However, the composition and flavor of the oils are different, and the roasting conditions should be optimized according to the specific species. Guan et al. (2022) found that, the flavor of the defatted tiger nut powder was significantly changed when it was baked at 150 °C for 8 min. And the content of aromatic flavor component was significantly decreased when the baking time was longer than 20 min. It should be noted that tiger nut is not suitable for intense roasting, cause burnt taste can be produced quickly at high temperature due to its high starch content.

However, there are a few studies on the flavor of tiger nut oil. Researches on the flavor of roasted tiger nut oil were not systematic and comprehensive. The origin and species have a significant influence on the basic composition of tiger nut, which consequently affects its oil flavor. In addition, there are many extraction methods for flavor substances, including headspace extraction, simultaneous distillation extraction and solvent-assisted flavor evaporation. The composition of flavor substances extracted by different extraction methods also differs greatly. In conclusion, the unique flavor of tiger nut oil makes it have extensive application prospect, which should be focused on in the future.

## 8. Formation of potential hazards in oil processing

During the processing of oil, some potential toxic hazards were formed due to the processing especially in the acute high-temperature

Compounds	Chemical structures	Odour descriptions	Oil samples	References
Aldehydes Vanillin	HO	Chocolate, vanilla	RO, RoO, SCDEO, AEEO	(Lasekan, 2013; Xin et al., 2022; Cantalejo, 1997)
5-Ethylfurfural		Caramel, spicy	RoO	(Lasekan, 2013)
Phenyl acetaldehyde		Honey-like	RoO	(Lasekan, 2013; Chen et al. 2023)
Octanal		Fatty, citrus-like	RO, RoO,	(Lasekan, 2013; Liu et al.,
oara-Hydroxybenzaldehyde		Bitter almond	RoO	2022; Cantalejo, 1997) (Lasekan, 2013)
Z)-14-Methyl-8-hexadecenal		Sweet	RoO	(Lasekan, 2013)
rans,trans-2,4-Decadien-1-al		Fatty, oxidized	RO, RoO	(Liu et al., 2022; Chen et al 2023)
0-Undecenal		Citrus-like, fatty	RO, RoO	(Liu et al., 2022)
-Nonanal Iexanal		Fatty, almond, flowery Green	RO, RoO, CPO, SCDEO, AEEO RO, RoO	(Liu et al., 2022; Chen et a 2023; Xin et al., 2022) (Liu et al., 2022; Chen et a
-Heptylacrolein		Fatty, mushroom	RO, RoO	2023) (Liu et al., 2022)
eptaldehyde		Fruity, nutty	RO, RoO	(Liu et al., 2022; Chen et a
-Undecenal		Citrus-like, fatty	RO, RoO	2023) (Liu et al., 2022; Chen et a
Oodecyl aldehyde	₀	Lily-like, fatty,	CPO, SCDEO,	2023) (Xin et al., 2022)
lcohols ,5-Dimethylcyclohexanol	$\bigwedge$	Citrus-like Musky	AEEO RoO	(Lasekan, 2013)
l-Octen-3-ol	ОН	Mold, earthy	RO, RoO	(Liu et al., 2022; Chen et a 2023)
Cedrol	HOUSE	Woody	CPO, SCDEO, AEEO	(Xin et al., 2022)
	H			
-Nonanol	но	Fatty, rose-like	RoO	(Liu et al., 2022)
-Octanol	0.H	Fatty, citrus	RoO	(Liu et al., 2022)
sters thyl hexadecanoate	Ŷ	Soapy, fatty	RoO	(Lasekan, 2013)
-Propyl 9,12-octadecadienoate		Fatty	RoO	(Lasekan, 2013)
		)		
	$\sim$			

(continued on next page)

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Compounds	Chemical structures	Odour descriptions	Oil samples	References
I-Dodecanolide		Sweet, flowery	CPO, SCDEO, AEEO	(Xin et al., 2022)
etones ,3-Dihydro-3,5-dihydroxy-6- methyl-4H-pyran-4-one	но но	Caramel-like	RoO	(Lasekan, 2013)
thanone, 1-(4-hydroxy-3-methoxy phenyl)		Faint vanilla	RoO	(Lasekan, 2013)
Iydrocarbons -Pinene	HO	Pine incense	CPO, SCDEO, AEEO	(Xin et al., 2022)
l-Limonene		Lemon-like	RO, RoO, CPO, SCDEO, AEEO	(Liu et al., 2022; Xin et al. 2022)
Heterocyclic compounds 5-Hydroxymethylfurfural	HO	Chamomile-flower	RoO	(Lasekan, 2013)
2-Pentylfuran		Caramel-like	RO, RoO	(Liu et al., 2022; Chen et a 2023)
3-Ethyl-2,5-diMethylpyrazine		Caramel-like	RoO	(Liu et al., 2022; Chen et a 2023)
2-Methylpyrazine		Roasty	RoO	(Chen et al., 2023; Cantale 1997)
2,5-Dimethyl pyrazine		Roasty, nutty	RoO	(Chen et al., 2023; Cantale 1997)
2,3-Dimethylpyrazine		Roasty	RoO	(Chen et al., 2023; Cantale

(continued on next page)

#### Table 4 (continued)

Compounds	Chemical structures	Odour descriptions	Oil samples	References
Trimethyl-pyrazine	N	Roasty, meat-like	RoO	(Chen et al., 2023; Cantalejo, 1997)
2-Ethyl-5-methylpyrazine	N	Roasty	RoO	(Chen et al., 2023; Cantalejo, 1997)

RO: raw tiger nut oil (Soxhlet); RoO: roasted oil; CPO: cold-pressed oil; SCDEO: supercritical carbon dioxide extraction oil; AEEO: aqueous enzymatic extraction oil.

treatment, such as, the thermal pretreatment of the oilseeds and the refining process. Besides, the introduction of some foreign impurities also resulted in some dangerous ingredients formation in the oil. These potential hazards have been listed as the carcinogens. Therefore, the monitor and control of these substances in oil processing are very important.

## 8.1. Polycyclic aromatic hydrocarbons

Polycyclic aromatic hydrocarbons (PAHs) are the organic compounds that contain more than one fused aromatic ring, which were mainly formed by the insufficient combustion of some organic substances, such as protein, carbohydrate and lipid (Ji et al., 2020). Currently, there are more than 10,000 types of PAHs found in the natural environment. Some PAHs have potential toxicity, such as, carcinogenic, mutagenic, skin irritation, and so on. Among which, benzo(a) pyrene (BaP) has been listed on the human carcinogenic group I by the International Agency for Research on Cancer (IARC) (Sun et al., 2021). Due to the lipophilic nature, PAHs can be easily migrated into the oil during the harvesting and processing of oilseeds. Therefore, the control of PAHs is particularly important, and some relevant regulations have been released. EU has stipulated that the total contents of four polycyclic aromatic hydrocarbons (PAH4) and BaP in vegetable oils should not exceed 10 µg kg<sup>-1</sup> and 2 µg kg<sup>-1</sup>, respectively (Sánchez-Arévalo et al., 2020).

The PAHs in the tiger nut oil are mainly from soil-atmosphere environment, harvest, package and transportation of oilseeds, as well as oil processing and storage conditions (Ji et al., 2020). The generation of PAHs during the processing of oils was mainly in the thermal pretreatment process. PAHs were also formed by the pyrolysis and cyclization of proteins, carbohydrates and oils under high temperature (Yin et al., 2022). Generally speaking, dramatic thermal treatment could result in the increase of PAHs content. Zhang et al. (2022) found that the BaP, PAH4 and the total contents of the fifteen polycyclic aromatic hydrocarbons (PAH15) in peanut oil were increased with the increasing of heating temperature and time, and reached the maximum at 190 °C for 30 min. Yin et al., 2022 found the similar results in sunflower oil. Although the issue of PAHs contamination was found in vegetable oils, some refining processes of oil could reduce the PAHs content. Ma et al. (2017) found that the content of PAH16 in the oil was decreased by 55.7%, 87.5% and 47.7% in the neutralization, bleaching and deodorization stages, respectively. The efficient removal of PAHs by decolorization had inspired researchers to use adsorbents for the removal of PAHs from oil. Currently, some adsorbent materials, such as cellulose aerogel, activated carbon, and modified activated carbon, have been found to be effective for the removal of PAHs from oils (Kim, Kim, & Shin, 2021; Zhang, Ji, & Sun, 2023). Tiger nut have been available for direct consumption as roasted snack for a long time. However, the PAHs contents in roasted tiger nut and its oil have not been determined, which

should be given more attention in future.

## 8.2. 3-Monochloropropane-1,2-diol esters and glycidyl esters

3-MCPDEs and GEs are considered to be harmful contaminants formed during oil processing. Of these, GEs were shown to be carcinogenic and pro-tumor growth in toxicological tests (Oey et al., 2019). When 3-MCPDEs were entered the intestine, they were hydrolyzed by enzymes to free 3-MCPD, which had been proved to be toxic (Bakhiya et al., 2011). Therefore, a limitation should be required and the European Food Safety Authority (EFSA) also recommended a Tolerable Daily Intake (TDI) of 2.0 µg kg<sup>-1</sup> bw/day for total 3-MCPD (including 3-MCPD and 3-MCPDEs) (Gao et al., 2020).

3-MCPDEs and GEs were generally formed during the refining of vegetable oils, and they could be interconverted under some conditions. In particular, the deodorization process was mainly responsible for their formations, which was due to the vigorous deodorization conditions. Under the high pressure and temperature, the precursor substance chloride ions attack the glycerides, replacing the hydroxyl group or fatty acyls on glycerol and other chemical reactions to form 3-MCPDEs. The epoxide intermediate of GEs were formed during these reactions (Yung et al., 2023). At present, the mitigation of 3-MCPDEs and GEs can be done by controlling the precursor substances (chloride ions, free fatty acid, diglycerides and monoglycerides) before the deodorization process. Reducing the use of chlorine-containing fertilizers and the amount of chlorine ions in the water from the oilseeds harvesting to the refining stages of oils can also reduce the production of 3-MCPDEs. In addition, it is reported that acid neutralization before deodorization could also reduce the formation of 3-MCPDEs (Yung et al., 2023). Besides, the mitigation of 3-MCPDEs and GEs could also be achieved by reducing or changing the conditions of the deodorization process. For example, Gao et al. (2022) found that the decreasing of the deodorization temperature and time could alleviate the formations of 3-MCPDEs and GEs. Currently, 3-MCPDEs have also been found in some refined edible vegetable oils, such as peanut oil, soybean oil, palm oil, and sunflower oil (Graziani et al., 2017). However, the refining of tiger nut oil is still in its infancy. Therefore, more attention should be paid in future.

## 9. Industrial application of tiger nut oil

Fig. 2 shows some industrial applications of tiger nut oil, some of which are already available in the market. The fatty acid profile of tiger nut oil is close to the recommended human fatty acid intake ratio, which makes it as a good edible oil. Tiger nut oil could also be used to produce soap. When the moisture content of tiger nuts was controlled in the range of 20 to 40 %, the saponification value of the obtained oil was higher than the recommended value of edible oil, and thus was suitable for soap production (Adejumo & Salihu, 2018). In addition, tiger nut oil has been used in high-end cosmetics and essential oils. Because of its

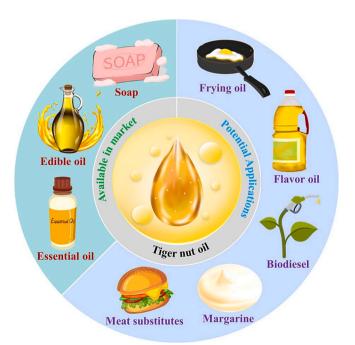
high contents of oleic acid and phenolic substances, it is easily absorbed by the skin and provides an antioxidant effect.

According to its composition and properties, tiger nut oil has the potential to be used as the frying oils, spray oils, and industrial lubricants. The frying properties of the mixed oil blended by tiger nut oil and sunflower seed oil was studied by Rehab & Anany (2012). They found that the mixed oil showed better frying properties when the ratio of tiger nut oil in the mixture increased, which showed that tiger nut oil could be used as a potential source of frying oil. In addition, tiger nut oil has a nutty flavor and can be developed into a strong-flavor oil if a thermal pretreatment was applied to enhance the flavor prior to oil extraction. Besides, there are also some other potential applications for tiger nut oil being studied (Fig. 2).

## 9.1. Biodiesel

In order to overcome the issue of the shortage of the petroleum oil, biodiesel has been produced from biomass resources through some technologies (Zhang et al., 1996; Tian et al., 2019). Biodiesel is a green and clean energy source, and the seeds, roots, stems and leaves of some crops can be used as the feedstock for biodiesel production (Barminas et al., 2001). Rapeseed oil and sunflower oil have also been used to produce high quality biodiesel, and corresponding production systems have been developed in Europe (Makarevičienė et al., 2013).

Tiger nut oil has been proposed as a fuel in twentieth century, because some physicochemical properties of tiger nut oil, such as viscosity, cloud point, specific gravity, and energy content, are similar to rapeseed, soybean, and sunflower oils. Short-term experiment showed that direct blend of tiger nut oil and diesel could be used in combustion engines, and the long-term effects of such blends on engines are similar to other blends of vegetable oils and diesel (Zhang et al., 1996). In addition, the ultimate analysis also showed that the tiger nut tuber has exceptionally low contents of N and S with just 0.9% and 0.1%, respectively, which produced less contamination after the combustion and also effectively avoided the catalyst deactivation caused by sulfur poisoning during biodiesel production (Tian et al., 2019). However, almost no finished oil was directly used for biodiesel in real-life applications. Finished oil should be transesterified with the low molecular alcohols to enhancing its properties, such as low viscosity and low



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toxicity. The transesterification reaction of the crude tiger nut oil was studied by Ofoefule et al. (2013). And the effect of mixing different ratios of transesterified oil with diesel on the physicochemical properties of blends was also evaluated. They found that the properties of the products with 10% or 20% contents of tiger nut oil were similar to the standard diesel. However, this two-step biodiesel production method including oil extraction and transesterification is time and organic solvent consuming. Cutting-edge research has focused on the direct catalytic reaction of biomass into biofuel. The effect of four factors (reaction temperature, time, catalyst type and the ratio of ethanol to feedstock) on Lewis acid-catalyzed the transesterification/esterification reactions of tiger nut in sub/supercritical ethanol was studied by Tian et al. (2019). They found that the highest crude biodiesel yield was obtained when the catalyst SnCl<sub>2</sub> was used. However, the carbon element in biomass (carbohydrates and proteins) is not fully utilized with this method. A thermochemical conversion method, hydrothermal liquefaction that could convert carbon in biomass into hydrocarbons, was used by Shi et al. (2023) to produce tiger nut biodiesel. The MgAl-layered double hydroxides was used to catalyzed the reaction, and the highest biodiesel vield of 38.7% was obtained.

## 9.2. Animal fat products replacement

With the changes of people's lifestyle and dietary habit, customers realized that meat products are no longer the best choice, because they contained more saturated fatty acids and cholesterol (Hygreeva et al., 2014). In this regard, scientists are trying to replace meat products entirely or partially with healthy plant-based ingredients (Carvalho et al., 2019). Tiger nut oil has more than 70% unsaturated fatty acids, which makes it a good alternative for animal meat products.

Currently, liquid oils are mainly converted into emulsified gels to replace sausage and hamburger meat. Recent researches have focused on the emulsion hydrogels due to their similar properties to animal meat, low price and easy to operate (Domínguez et al., 2021). Vargas-Ramella et al. (2020) and Barros et al. (2020) found that there was no significant texture variation when the animal fat was replaced by tiger nut oil emulsion. And the addition of tiger nut oil emulsion as a replacement for beef fat could significantly reduce the content of saturated fatty acids, which is beneficial for human. Besides, the oil content of this emulsified hydrogel was<40%, which greatly reduced the caloric value, and is suitable for weight loss group.

Margarine and shortening are widely used in bread spreads and bakery products. Since margarine is comprised of at least 80% fat, the selection of fat has a significant influence on the margarine quality (Dong et al., 2023). Vegetable oils should be modified by hydrogenation before used in the manufacture of margarine. However, partially hydrogenated oils (the main component of margarine) are facing safety issues, such as high contents of trans fatty acids and saturated fatty acids, that have negative effects on human health. Some new technologies should be developed to meet the requirement of margarine fat. In our previous report, we found that high quality margarine oil could be produced by the interesterification of tiger nut oil with palm stearin (Dong et al., 2023). When the oil system contained saturated fatty acids (14%-25%) and high levels of oleic acid (>60%), the obtained margarine was of superior quality. And the fatty acid contents of tiger nut oil are in this range, which make it as a good choice for plant-based oil for margarine manufacturing. In addition, the results of differential scanning calorimetry and solid fat content also indicated that the quality of interesterified oils were similar to that of the oils used in commercially available margarine. We also found that, under the optimal process, the modified oil was free of trans fatty acids, and the properties of oil met the requirements of oils for margarine (Dong et al., 2023).

## 10. By-products of tiger nut oil processing

Fig. 2. Industrial applications of tiger nut oil.

The meal or oil-cake produced during the extraction of vegetable oil

is also a non-negligible part of the oil processing. The effective utilization of this residue is in line with the concept of resource conservation and the requirement of oil factory to make full use of the oilseed in order to maximize profit. Generally speaking, the defatted meal produced in the oil exaction process contained some minerals, such as N, P, K, organic matter proteins and polysaccharides, which were often used as animal feeds (Sidohounde et al., 2014). The chemical composition and digestibility of tiger nut by-products obtained by different oil extraction methods (expeller-pressing, cold-pressing and solvent method) were compared. Results showed that the expeller-pressed tiger nut cake was the better feed for growing pigs compared with other two methods from the perspective of energy contents and amino acid digestibility (Wang et al., 2021).

## 10.1. Starch

However, it is a waste to use defatted tiger nut meals only as livestock feedstuff. Unlike other oilseeds that contain amount of protein, tiger nut tubers contained about 30–40% of starch that could be extracted as a main by-product for application. Some properties of tiger nut starch, such as Carr's index, density, and Hausner ratio, are similar to potato and maize starch. The internal starch granules of tiger nut starch are more tightly bound, which makes it have potential to be used as a binder for pharmaceuticals (Manek et al., 2012). Tiger nut starch can also be used to make steamed bread. Lv et al. (2022) found that the structure of the steamed bread crumbs was significantly improved when 50% wheat flour was replaced by tiger nut starch.

Recent studies have focused on the effects of different oil extraction processes on the quality of tiger nut starch. Liu et al. (2019) compared the effects of several different oil extraction techniques on the physicochemical and functional properties of the starches in tiger nut meals. They found that the pressure and temperature in the process were the key factors that had a significant impact on the starch properties, which confirmed that the hot pressing had the great impact on the physicochemical properties of starch. They also found that the solubility and swelling capacities of starch were both improved during this process. However, these resulted in the low freeze-thaw stabilities of starch, because high syneresis values were observed in the processed starch, compared to the untreated starch. In addition, all indexes indicated that the physicochemical properties and functional characteristics of the starch obtained by cold pressing were similar to those of the original starch. In this regard, the cold pressing process of tiger nut oil and the separation of starch from the meal were studied by Miao et al. (2022). They found that the structure of the starch was slightly affected by pressing process, and the paste and gel properties of the starch did not significantly change, which allowed it to have a wide application.

## 10.2. Protein

In addition to starch, the protein in tiger nut meal is also a good byproduct. Four kinds of proteins were found in tiger nuts, of which, the content of glutelin was the highest (>47.5%), followed by albumin of 31.8%. In addition, globulin (4.7%) and prolamin (3.8%) were also found (Kizzie-Hayford et al., 2015). Although the protein content of tiger nut is not high, it contains eight essential amino acids required for human body, which makes it have the commercial value (Wang et al. 2021).

Currently, tiger nut protein has not been given much attention. The methods that have been reported for tiger nut protein extraction are ammonium sulfate precipitation and alkali extraction methods (Kizzie-Hayford et al., 2015; Cui et al., 2021). Future research should focus on the effect of oil extraction process on tiger nut protein, and expand the application of tiger nut protein. Besides, there is a tendency for the utilization of by-product meal from oil extraction. By proper enzymatic treatment, the polypeptides can be broken into dipeptides or free amino acids, and some pleasant flavor substances can also be formed. The

enzymatic products can be used as a flavor additive directly added to oils and foods to enhance flavor. Similar studies have been done in peanut and soybean meals (Weng et al., 2021; Zhang, Song, Chang, Wang & Meng, 2022). However, the enzymatic reaction should be controlled, because excessive enzymatic digestion resulted in the undesirable flavors formation.

## 10.3. Polyphenols

Tiger nut is rich in antioxidant bioactive substances. The decomposition and release patterns of bioactive substances of four tiger nut products (flour, oil, milk with and without added sucrose) along gastrointestinal digestion were studied by Hernández-Olivas et al. (2022). They found that antioxidants such as phenolic compounds, vitamin E and carotenoids from tiger nut products, had good bioaccessibility, which suggested that tiger nut products can be used as functional foods to alleviate some diseases, such as, age-related macular degeneration. In addition, the antioxidants in tiger nut, such as polyphenols and carotenoids, can be extracted as the plant-derived antioxidants to extend the shelf life of foods. Current researches have focused on the extraction of phenolic compounds from "Horchata" (a famous beverage product of tiger nut) by-products. The extraction of phenolics from the "Horchata" by-products by ultrasound-assisted ethanol/water was optimized by Razola-Díaz et al. (2022) and eighteen phenolics and phenolic precursors were identified and quantified.

However, there are no studies on the extraction of phenolics from the by-products, oil cake or meal of tiger nut. Koubaa et al. (2015) found that the oil remained in the meals contained a large amount of polyphenol. As a matter of fact, only a small fraction of polar compounds is dissolved in the oil during the oil extraction process, and most polar phenolic compounds are hold in the meal. More attention should be given in future.

## 11. Conclusions and future prospects

Tiger nuts, an underutilized plant tuber, will play a great important role in the current food scarcity situation. However, the chemical composition and the nutrients of different species of tiger nuts varied greatly, and future researches should pay more attention to these differences. Some oil extraction techniques have been shown to be available for tiger nut oil extraction. However, some novel oil extraction techniques still face some challenges, which limit these methods to be used only in laboratory. Besides, the researches on the refining process of tiger nut oil and the utilization of the meal and oil cake after oil extraction are still scarcity. Meanwhile, the generation of potential hazards in the oil processing should be controlled. In addition, the unique flavor of tiger nuts makes it as a flavor oil, which should also be given more attention.

## CRediT authorship contribution statement

Yiming Zhang: Writing – original draft, Visualization. Shangde Sun: Writing – review & editing, Supervision, Funding acquisition.

## **Declaration of Competing Interest**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

## Data availability

Data will be made available on request.

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## Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.fochx.2023.100868.

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