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Original article

Characteristics of tiger nut milk: effects of milling

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Summary A standard method for the extraction of tiger nut milk has been introduced. It has been shown that, although milling duration improves the yield of tiger nut milk solids and its nutrient composition, there is a quantifiable loss of nutrient in the pressing residue during milk extraction. Milling duration improved the colloidal stability of the milk against creaming during 16 h of storage. A higher milling intensity resulted in the aggregation of biological polymers which resulted in colloidal destabilisation. Milling improved the lightness and stability and reduced browning rate of the tiger nut milk during storage. This report is important for the production of tiger nut milk of consistent and comparable characteristics. Milling has been introduced as a processing method for the qualitative and quantitative modulation of the properties of tiger nut milk. It is recommended to develop further strategies to improve the colloidal stability of tiger nut milk as a beverage.

Keywords Colloidal stability, colour, milk extraction, milling, nutrient transfer, tiger nut.

Introduction

Tiger nut (*Cyperus esculentus*) milk is the aqueous extract derived from the sweet nut-like tuber of the perennial grass-like tiger nut plant (Coşkuner *et al*., 2002). The nut is nutritious, and milk from the nuts has found wide application, especially in Spain, where it is used for the commercial preparation of *horchata de chufa* (Sánchez-Zapata *et al*., 2012). It has also been used as flavouring in ice cream and bakery products and for the preparation of *Kunnu*, a nonalcoholic beverage with low viscosity, a sweet-acidic taste and a milky appearance (Akoma *et al*., 2000; Coşkuner *et al*., 2002; Belewu, 2008; Sanful, 2009). Methods that have been reported for the extraction of tiger nuts after milling them into a smooth mush include the use of tissue or cotton bags or layers of cheese cloth, and the application of gravitational force or hand pressing; the use of centrifuges has also been proposed (Akoma *et al*., 2000; Belewu, 2007, 2008; Djomdi *et al*., 2007). Most of the processes mimic traditional tiger nut milk extraction methods, and they are useful for understanding the properties of milks obtained from tiger nuts. However, the methods are not standardised so that it is difficult to compare reports on composition and properties of tiger nut milk. For instance, the use of a centrifuge might influence total solids content of the tiger nut milk and facilitate creaming because of the centripetal force (Letki, 2000; Belewu, 2007). Different types of membrane filters and different magnitudes of applied pressure may also affect composition, yield and physical properties of the tiger nut milk (Akoma *et al*., 2000; Djomdi *et al*., 2007; Belewu, 2008).

In this study, the use of a pneumatic press is presented. This approach uses a defined pressure (6.55×10^5 Nm^{-2}) and a defined filtration pore size (4 μm) for milk extraction, and the extraction yield and the distribution of nutrients between the tiger nut residue and the milk upon pressing as influenced by the duration of milling are investigated. Ejoh *et al*. (2006) provided important information regarding the effect of milling on tiger nut milk performance. It was, however, not indicated how milling affected nutrient recovery and the physical characteristics of the tiger nut milk. The aim of this study is to investigate the effect of milling of tiger nuts on the properties of tiger nut milk with respect to nutrient transfer, and the colloidal and colour stability of the milk.

Materials and methods

Sample collection, preparation and reagents

Freshly harvested tiger nuts (brown variety) were directly obtained from farmers in the Central Region of Ghana (Twifo Praso). The nuts were rubbed together in a sack to remove sand and root hairs, washed and

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dried for 2 months at room temperature. Darkened, broken or deteriorated tiger nuts were withdrawn, and the bulk of the nuts was then packed and stored in a cool room (5–6 °C) prior to analysis. Reagents including anhydrous sodium hydroxide, sulphuric acid and petroleum ether were obtained from Carl Roth GmbH & Co KG (Karlsruhe, Germany). Hydrochloric and boric acid were supplied by VWR GmbH (Darmstadt, Germany). All reagents used were of analytical grade.

Milk extraction

Approximately 50 g of previously dried and stored (5–6 °C) tiger nuts (initial moisture content: 0.088 ± 0.0012 g g^{-1}) was washed with demineralised water. To soften the nuts for effective comminution and extraction of the milk, the nuts were hydrated by soaking in a glass beaker with 400 mL water that was placed in a water bath for 24 h at 40 °C (Djomdi *et al.*, 2007). The hydrated tiger nuts were again washed with aqua demineralisation. until clear wash water was obtained. The nuts were then poured into the vessel of a Kult pro mixer (WMF AG, Geislingen, Germany), 200 g distilled water was added, and the mixture was comminuted for 1, 2 or 3 min using the 'smoothie mode' option to obtain the tiger nut mush. Finer particles of tiger nut mush was prepared by additionally dispersing the sample comminuted for 3 min using a T20 ultra-turrax mixer (IKA GmbH & CO. KG, Staufen, Germany) at 13 000 rpm for 20 min. The milling intensity needed for the milk particles obtained using the ultra-turrax was beyond the capability of the Kult pro mixer. Using 100 g water, the tiger nut mush was quantitatively transferred into the hopper reservoir of a pneumatic press with its frit being layered with a Whatman (GE Healthcare Europe GmbH, Freiburg, Germany) 4-μm-pore-size filter membrane. The retentate and the permeate fraction of the mush that is the pressing residue (PR) and the tiger nut milk (TNM), respectively, were obtained by applying a pressure of 6.55×10^5 N m^{-2} until flow from the press ceased. To prevent microbial growth in milk during storage at 5 °C, 0.2 gL^{-1} sodium azide was added to the tiger nut milk. To determine the soluble and insoluble solids of the milk, 10.0 g extracted tiger nut milk was filtered under gravity using a precalibrated 4–7-μm filter paper. The residual mass on the filter after drying (103 °C, 5 h) was determined, and the fractions of insoluble and soluble solids in dry mass (DM) were calculated using milk solids content.

Nutrient analysis of tiger nuts

Prior to analysis, 50-g tiger nuts were soaked as previously described, mopped dry, and their mass, M_{STN} (g), was recorded. This pretreatment step was performed to consider any transfer of compounds from the tiger nuts to the soaking water in the soaking step. To prevent microbial activity on the presoaked tiger nuts during nutrient analysis, the moisture content of the presoaked tiger nuts was reduced by oven-drying (60 °C, 24 h); subsequently, the mass of soaked, dried tiger nuts M_{SDTN} (g) was recorded. The nuts were then dry-milled into a fine powder using an EG100 coffee grinder (Electrolux Hausgeräte GmbH, Nürnberg, Germany) for 2 min to pass a 20 mesh sieve. Portions of the ground samples were used for the nutrient analysis.

Moisture content, crude protein, crude fat and ash content of these milled nuts was determined according to the methods described by Matissek *et al.* (1992) and Nielsen (2003). Crude soluble and insoluble fibre was determined using an enzymatic method (Megazyme International, Wicklow, Ireland), with the following modification: a 12- to 14-μm precalibrated Whatman filter paper was used for the filtration of insoluble fibres and alcohol-precipitated soluble fibres instead of a celite membrane. The carbohydrate content was estimated as the difference in dry mass. Carbohydrate estimates were analytically confirmed using the sulfuric acid–UV method proposed by Albalasmeh *et al.* (2013).

Analysis of tiger nut products

The moisture content of the wet pressing residue W_{WPR} (g g^{-1}), the mass of the wet pressing residue and the mass of the extracted tiger nut milk M_{TNM} (g) was determined. The material recovery R (%) and the relative amount of wet and dry pressing residue (M_{WPR} (g 100 g^{-1}) and M_{DPR} (g 100 g^{-1} DM), respectively) were calculated using:

$$R = 100 \cdot \frac{M_{TNM} + M_{WPR}}{M_{STN} + 300\,\text{g}} \tag{1}$$

$$M_{WPR} = 100 \cdot \frac{M_{WPR}}{M_{STN} + 300\,\text{g}} \tag{2}$$

$$M_{DPR} = 100 \cdot \frac{M_{WPR} \cdot (1 - W_{WPR})}{M_{SDTN} \cdot (1 - W_{SDTN})} \tag{3}$$

where W_{SDTN} (g g^{-1}) refers to moisture content of the soaked and subsequently dried tiger nuts. The nutrient composition of the wet pressing residue was then analysed using the methods as previously described.

Based on the composition of solids in the pressing residue, the transfer of total solids and of soluble and insoluble solids from wet-milled tiger nuts into tiger nut milk was calculated. The yield of tiger nut milk Y_{TNM} (g 100 g^{-1} DM) was calculated from the

difference in the relative amount of dry pressing residue M_{DPR} (eqn 3) up to 100% using:

$$Y_{TNM} = 100 - M_{DPR} \quad (4)$$

The nutrient content related to total solids of tiger nut milk NC_{TNM} (g 100 g^{-1} DM) was estimated for each analysed nutrient compound by applying

$$NC_{TNM} = 1 - \frac{NC_{WPR} \Big/ (1 - W_{WPR})}{NC_{SDTN} \Big/ (1 - W_{SDTN})} \cdot R \quad (5)$$

where NC_{SDTN} (g g^{-1}) refers to the nutrient content in soaked and subsequently dried tiger nuts, NC_{WPR} (g g^{-1}) refers to the nutrient concentration in the wet pressing residue and W_{WPR} (g g^{-1}) refers to the moisture content of the wet pressing residue. R is the material recovery from the press (eqn 1).

Particle size distribution

The starch granular composition, particle size distribution and droplet formation in the tiger nut milk were visualised using a light microscope (Carl Zeiss AG, Oberkochen, Germany) and measured by laser diffraction with a Helos/KR-H2487 (Sympatec GmbH, Clausthal–Zellerfeld, Germany). To 1 mL sample, 100 μL of Lugol solution (0.007% I_2, 0.014% KI) was added to stain the starch granules, and 15 μL of the stained samples were then observed in the microscope using 1000-fold magnification. For tiger nut milk droplet and particle size analysis, a relative refractive index of 1.101 was used based on the refractive index of tiger nut oil (1.464) and aqua demineralisation. (1.330) (Ekeanyanwu & Ononogbu, 2010). Tiger nut milk was diluted to obtain approximately 0.05% critical optimum particle concentration. The particle size of the milk was reported based on the volume-weighed mean diameter d_{43} using:

$$d_{43} = \frac{\sum n_i d_i^4}{\sum n_i d_i^3} \quad (6)$$

where n_i is the number of particles with a diameter d_i.

Colloidal stability

The effect of milling intensity on creaming as a measure of colloidal stability of the tiger nut milk was determined according to White *et al.* (2008). Briefly, 20.0 g tiger nut milk was transferred into glass tubes (1.5 cm internal diameter), plugged with a silicone stopper and stored in an environmental chamber at 50 °C for 44 h. Samples were observed every 2 h for emulsion breakdown by the formation of an upper oil layer. The creaming index CI (%) was determined using:

$$CI = 100 \cdot \frac{H_0}{H_e} \quad (7)$$

where H_0 is the height of the oil layer and H_e is the total height of the emulsion in the tube (White *et al.*, 2008).

Colour measurement

The colour stability of tiger nut milk was analysed over a period of 27 days using a LUCI100 CIE Lab colour space colorimeter (Hach Lange GmbH, Düsseldorf, Germany) working with D65 xenon light illumination and a 10° standard observer angle. The instrument was calibrated against black and white surfaces (standard LZM128). Pasteurised cow milk obtained from the supermarket was used for comparison. Mean values for lightness L^*, red–green intensity a^* and yellow–blue intensity b^* were calculated from the colour primaries. The Chroma $C^* = [(a^{*2}) + (b^{*2})]^{1/2}$ as an indicator for colour saturation and the hue angle $h_{ab} = \arctan(b^* \; a^{*-1})$ were also determined (Rohm & Jaros, 1996).

Statistical analysis

All the milk extraction experiments and analytical procedures were conducted in triplicate, and arithmetic means were used for analysis. One-way analysis of variance (ANOVA) combined with Tukey HSD and Games–Howell *post hoc* analysis was used to compare the mean values. All significance levels refer to $P < 0.05$. SPSS software package version 16.0 was used for performing the analyses (SPSS Inc, Chicago, IL, USA).

Results and discussion

Yield in tiger nut extraction

On average, the soaking of tiger nuts resulted in a mass increase from 50.21 ± 0.08 g to M_{STN} = 75.66 ± 1.16 g, and the mass of presoaked, dried tiger nuts M_{SDTN} was 49.6 ± 0.2 g. The corresponding mass of the wet pressing residue and that of the tiger nut milk M_{TNM} (g) from each extraction sample was 43.09 ± 1.21 g and 326.8 ± 0.7 g, respectively. The average material recovery in the extraction process was R = 97.86–98.97%. Milling intensity had no influence on the material recovery ($P > 0.05$) which implies that the extraction process was reproducible. It was observed that the wet mass of the pressing residue from the mush with 2 min of milling (Mush.2) and the mush with 3 min of milling plus ultra-turrax dispersion (Mush.3) was significantly lower than pressing residue mass after 1 min of milling (Mush.1; Table 1). Prolonged milling reduced the bulk of the

pressing residue, possibly because of a more effective reduction of the size of tiger nuts, and an enhanced solids transfer across the filtration membrane during pressing. Milling also affected the moisture content of the pressing residue significantly. Specifically, the pressing residue from Mush.2 (PR.2) had higher moisture content than the pressing residues from Mush.1 (PR.1) and Mush.3 (PR.3) (Table 1). This may partly be ascribed to the contribution that comes from the soluble fibre content of the pressing residue. It was observed that PR.2 had higher soluble fibre content (Table 2), and soluble fibres are known to have a high water retention capacity (Elleuch *et al.*, 2011; Chen *et al.*, 2014). It is suggested that a specific milling intensity may be necessary to regulate soluble fibre content in high fibre-containing food materials such as tiger nuts.

The increase in milling intensity progressively increased the yield of milk solids from 35.1% to 41.2% ($P < 0.05$; Table 1). Djomdi *et al.*, (2007) reported a maximum yield of 30%. The type of the extraction method, the extent of size reduction, the pore size of the filter membrane, the magnitude of the pressure and the variety of tiger nuts used might account for the disparity in yield of milk (Ejoh *et al.*, 2006). Even though the yield of tiger nut milk increased, the fraction of soluble milk solids significantly decreased from 89.3% to 74.5% with the increase in milling intensity. A similar observation by Ejoh *et al.* (2006) was attributed to an increase in the fibre and lipid content of the milk. In this work, an increase in the proportion of insoluble starch granules was observed using light microscopy (data not shown). It is therefore proposed that insoluble starch granules might have also accounted for the decrease in percentage soluble solids in the milk. Compared to Ejoh *et al.* (2006), a higher ratio of soluble to insoluble solids was observed in this experiment. The occurrence of excessive unadsorbed granules of polysaccharides in colloids is known to promote flocculation through a depletion mechanism, resulting in creaming instabilities (Jenkins & Snowden, 1996; Chanamai & McClements, 2001). Therefore, although a high yield of milk solids from tiger nuts is desirable, the occurrence of a high fraction of insoluble polysaccharides may have implications on the stability of the milk.

Nutrient retention in the tiger nut pressing residue

The moisture content of presoaked, dried tiger nuts was 0.0856 ± 0.0002 g g^{-1}. Dry matter-related gross composition of the presoaked tiger nuts (Table 2) was within the range that has been reported in literature (Coşkuner *et al.*, 2002; Ejoh *et al.*, 2006; Adejuyitan *et al.*, 2009; Oladele *et al.*, 2009).

The analysis of the nutrient composition of the pressing residues is important to evaluate the efficiency of the extraction procedure and to determine the fraction of the respective compound that remains in the pressing residue after milk extraction. Table 2 shows that the fraction of fat and salt in the residue was comparable to what has been reported for the residue from a local horchata process (Sánchez-Zapata *et al.*, 2009). The transfer of individual nutrients from the wet-milled tiger nut to milk or pressing residue may be affected by the intensity of tiger nut milling, the duration of maceration and the number of washing steps that are applied in the extraction (Ejoh *et al.*, 2006). As concerns effects of milling intensity, PR.3 showed a significantly lower protein content than PR.1 and PR.2. This might be due to the solubilisation of tiger nut proteins, depicting that tiger nuts might have a high fraction of water-soluble proteins. Fat and ash content in the pressing residues was also reduced significantly when milling time was increased. Milling was accompanied by partial warming of the mush, which might have enhanced lipid hydration and the transfer to the aqueous fraction (Cevc & Marsh, 1985; Nielsen, 2003). The soluble and insoluble fibre content of the pressing residue also varied; PR.2 and PR.3 showed the highest fractions for soluble fibre and insoluble fibre, respectively. A milling duration of 2 min seemed most effective for extracting carbohydrates, whilst a more intense milling was effective for

Table 1 Influence of wet-milling intensity on mass transfer from tiger nuts mush to the pressing residue and the milk

Wet-milled tiger nuts*	M_{WPR} (g 100 g^{-1})†	W_{WPR} (g g^{-1})	Y_{TNM} (g 100 g^{-1} DM)	Milk solids (g 100 g^{-1} DM)	
				Soluble	Insoluble
Mush.1	11.83 ± 0.03^{a}	0.445 ± 0.002^{a}	35.06 ± 0.14^{a}	89.30 ± 0.20^{a}	10.70 ± 0.20^{a}
Mush.2	11.39 ± 0.01^{b}	0.456 ± 0.004^{b}	37.87 ± 0.17^{b}	86.13 ± 0.74^{a}	13.87 ± 0.74^{a}
Mush.3	11.16 ± 0.09^{b}	0.438 ± 0.003^{a}	41.23 ± 0.43^{c}	74.46 ± 1.49^{b}	25.54 ± 1.49^{b}

M_{WPR}, Mass of wet pressing residue; W_{WPR}, Moisture content of wet pressing residue; Y_{TNM}, Yield of tiger nut milk.

*Presoaked tiger nut wet-milled for 1 min (Mush.1), for 2 min (Mush.2) or for 3 min plus ultra-turrax dispersion (Mush.3).

†Values in the same column with different superscripts differ significantly ($P < 0.05$). Arithmetic means ± standard deviations are based on triplicate experiments.

Table 2 Influence of wet-milling intensity on nutrient retention (g 100 g^{-1} DM) in the pressing residue during extraction of tiger nut milk

Nutrient compound	Pretreated tiger nuts*	Tiger nut pressing residue (PR)†,‡		
		PR.1	PR.2	PR.3
Protein	4.95 ± 0.01	1.72 ± 0.46[a]	1.78 ± 0.16[a]	1.61 ± 0.09[b]
Fat	21.60 ± 0.12	10.32 ± 0.11[a]	8.78 ± 0.16[b]	8.15 ± 0.11[c]
Ash	1.75 ± 0.01	0.74 ± 0.01[a]	0.74 ± 0.01[a]	0.69 ± 0.01[b]
Insoluble fibre	18.37 ± 0.20	28.58 ± 0.23[a]	30.96 ± 0.48[b]	31.03 ± 0.11[b]
Soluble fibre	2.56 ± 0.15	0.85 ± 0.03[a]	1.48 ± 0.15[b]	0.56 ± 0.03[c]
Carbohydrate	55.08 ± 0.13	57.79 ± 0.25[a]	56.26 ± 0.28[b]	57.96 ± 0.05[a]

*Presoaked, oven-dried tiger nuts.
†Tiger nut pressing residue after 1 min milling (PR.1), after 2 min milling (PR.2) or after 3 min milling plus ultra-turrax dispersion (PR.3).
‡Values in the same row with different superscripts differ significantly ($P < 0.05$). Arithmetic means ± standard deviations are based on triplicate experiments.

extracting proteins, ash and fat but not necessarily fibre.

Nutrient distribution during tiger nut milk extraction

Tiger nuts have been reported as being rich in energy and minerals (Sánchez-Zapata *et al*., 2012); however, little is known about the efficiency of nutrient transfer during extraction of tiger nut milk. Table 3 shows the effect of wet-milling on the transfer of nutrient compounds into tiger nut milk; the difference to 100% is because material recovery R (eqn 1) was considered in the calculations. It was observed that the distribution of the nutrient between tiger nut milk and pressing residue depended on the intensity of milling, but also on compound type. For instance, the fractional distribution of protein, fat, salts and soluble fibre between the pressing residue and the milk was approximately 1:4, whilst the distribution of insoluble fibre was 6:1 and that of carbohydrates was 1:1. The composition of the milk was as follows: moisture, 93.65–94.58 g 100 g^{-1}; protein, 0.47–0.54 g 100 g^{-1}; fat, 1.88–2.27 g 100 g^{-1}; ash, 0.16–0.18 g 100 g^{-1}; insoluble fibre, 0.32–0.36 g 100 g^{-1}; soluble fibre, 0.21–0.29 g 100 g^{-1}; and carbohydrates, 2.31–2.74 g 100 g^{-1}. The carbohydrate composition was comparable to the results of Udeozor (2012). Apart from the total soluble solids, content of tiger nut milk which differed from that of horchata, the protein, fat and ash content was consistent with the report of Cortés *et al*. (2004). The results, however, imply that, during extraction of tiger nut milk, a greater fraction of carbohydrates and insoluble fibre is retained in the pressing residue, whereas a greater proportion of protein, fat, ash and soluble fibre are transferred into the tiger nut milk. Although a maximisation of the transfer of nutrients such as proteins, minerals and essential fatty acids into tiger nut milk during extraction is desirable, it is also worthwhile to consider the influence the milling process shows on the colloidal characteristics of the tiger nut extract.

Table 3 Effect of wet-milling intensity on the transfer of nutrient compounds into tiger nut milk NC_{TNM} (g 100 g^{-1} DM) during extraction of tiger nut milk. Values were calculated according to eqn 5

Nutrient compound	TNM.1*,†	TNM.2	TNM.3
Protein	80.34 ± 0.58[a]	80.48 ± 1.51[a]	81.71 ± 0.67[a]
Fat	73.49 ± 0.17[a]	77.89 ± 0.75[b]	79.14 ± 0.54[b]
Ash	76.42 ± 0.47[a]	77.32 ± 0.45[a]	78.18 ± 0.41[a]
Insoluble fibre	16.63 ± 1.03[a]	14.54 ± 2.61[a]	13.19 ± 1.83[a]
Soluble fibre	80.71 ± 1.72[a]	68.85 ± 4.41[b]	86.97 ± 0.11[c]
Carbohydrate	38.57 ± 0.36[a]	43.31 ± 0.63[b]	40.68 ± 0.51[a]

*Tiger nut milk after 1 min milling (TNM.1), after 2 min milling (TNM.2) or after 3 min milling plus ultra-turrax dispersion (TNM.3).
†Values in the same row with different superscripts differ significantly ($P < 0.05$). Arithmetic means ± standard deviations are based on triplicate experiments.

Milk characteristics

A visual observation using microscopy showed that the tiger nut milk generally had deposits of starch granules. The milk extracted after 3 min milling with additional ultra-turrax dispersion (TNM.3) showed starch granules with a less defined structure, indicating that they were partially hydrated (data not shown). These hydrated granules might have a higher solubility and may also increase the density of the tiger nut milk and so contribute to milk stability (Tadros, 2009). Starch granules have been reported to enhance emulsion stability depending on their size and concentration (Li *et al*., 2013). The granules of milk extracted from nuts that were milled for 1 min (TNM.1) or 2 min (TNM.2) were relatively intact and appeared in a lower concentration. Based on the concentration and

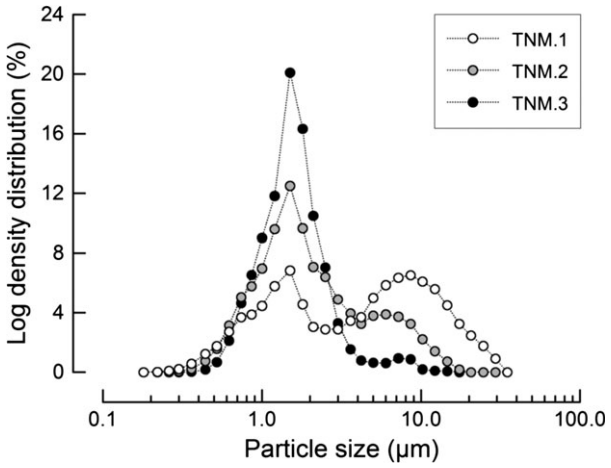

Figure 1 Effect of wet-milling intensity on particle size distribution of tiger nut milk. Milk was extracted from presoaked tiger nuts comminuted with cutting mill for 1 min (TNM.1), 2 min (TNM.2) or 3 min plus dispersion using an ultra-turrax (TNM.3).

the degree of hydration of the starch granules, TNM.3 may show a tendency towards a more stable colloidal suspension compared to TNM.1 and TNM.2. The differences in particle size of tiger nut milks were therefore ascertained using laser diffraction.

Particle size distribution

The particle size distributions depicted in Fig. 1 imply that there is a progressively increasing effect caused by milling duration that was effective for producing finer particles in tiger nut milk. Milling duration caused a change in particle distribution from bimodal (TNM.1, TNM.2) to monomodal (TNM.3). Generally, the mean volumetric diameter decreased with milling intensity. Ninety per cent of the particles present in tiger nut milk (D_{90}) were smaller than 13.6 μm (TNM.1), 6.46 μm (TNM.2) and 2.45 μm (TNM.3). The corresponding D_{50} median values were 3.94 μm, 1.64 μm and 1.40 μm, respectively, and D_{10} was in the range of 0.72–0.79 μm. Therefore, TNM.3 had the most uniform size distribution of the tiger nut milks. For comparison,

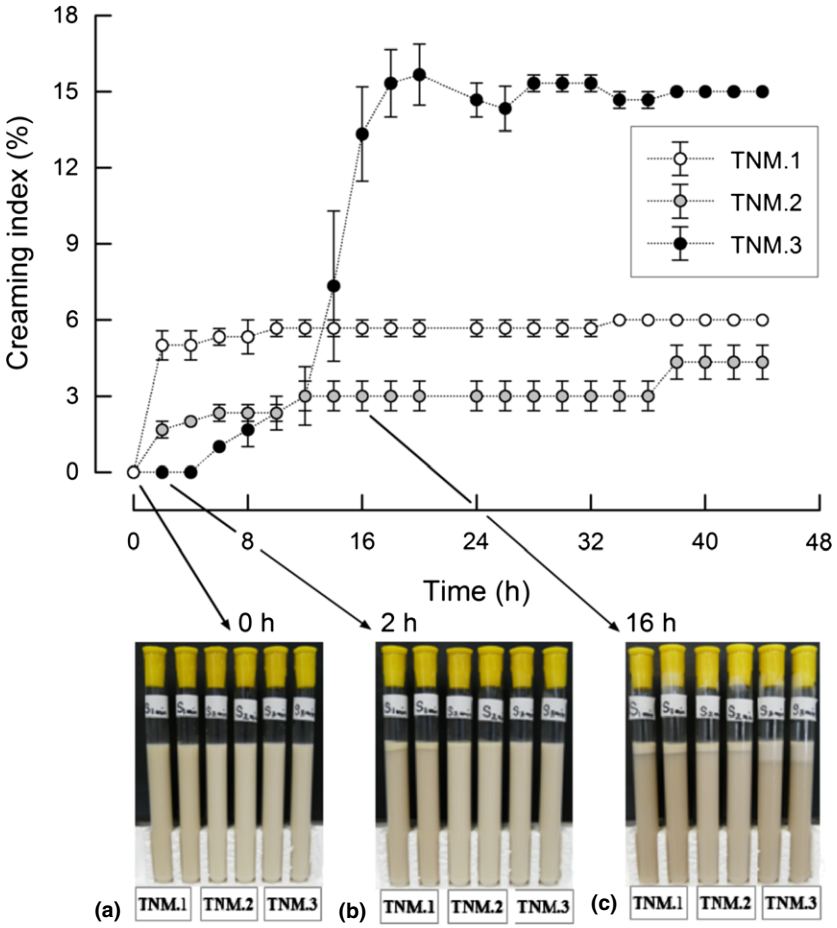

Figure 2 Effect of wet-milling intensity and storage period on the creaming rate of tiger nut milk. Milk was extracted from presoaked tiger nuts comminuted with cutting mill for 1 min (TNM.1), 2 min (TNM.2) or 3 min plus dispersion using an ultra-turrax (TNM.3) and stored at 50 °C. Photographs of milk (a, b, c) were taken at 0, 2 and 16 h respectively.

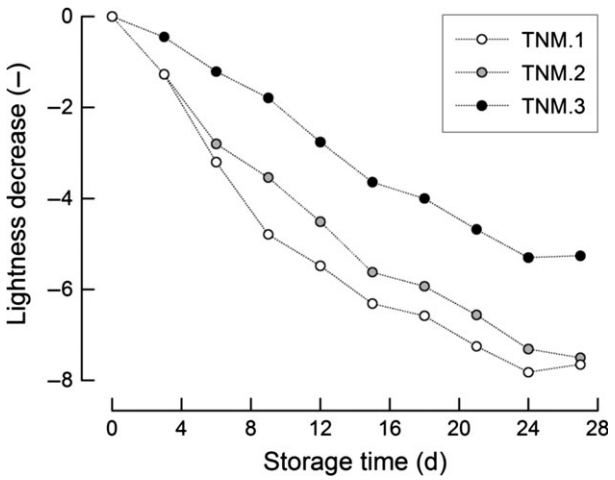

Figure 3 Effect of wet-milling intensity and storage period on lightness changes of tiger nut milk. Milk extracts from presoaked tiger nuts comminuted with cutting mill for 1 min (TNM.1), 2 min (TNM.2) or 3 min plus dispersion using an ultra-turrax (TNM.3). Storage conditions: 5 °C, 27 day.

commercial full fat cow milk had a D_{50} = 0.88 μm and a D_{90} = 1.46 μm. Consequently, the increase in milling duration increased the concentration of the milk constituents (see Table 3), improved the hydration, the solubility and the uniformity of the particles dispersed in the milk and reduced their average size. These properties are relevant for the stability of the tiger nut milk (Tadros, 2009).

Colloidal stability

The influence of milling and storage period on the colloidal stability of tiger nut milk is illustrated in Fig. 2. Milks underwent two stages of destabilisation: creaming of the milk (2–16 h) resulted in the formation of a lipid-rich top layer. The second destabilisation stage (starting at 16 h) was characterised by an extensive aggregation of milk solids, and the formation of a two-layer system: an oily biological polymer-rich upper layer and a biological polymer-depleted aqueous layer at the bottom. The increase in the duration of milling reduced the initial creaming rate of the tiger nut milk. TNM.2 and, especially, TNM.3 had a smaller average particle size (see Fig. 1) and a higher concentration of solutes (essentially proteins, fats and sugar polymers) (see Table 1, Table 3). In support of the hypothesis by Ye (2008), it is suggested that the size reduction coupled with homogenisation of the tiger nut milk may have enhanced the hydrophobic surface characteristics of the proteins in tiger nut milk through molecular unfolding. The proteins might have then adsorbed competitively or cooperatively with carbohydrates to form multiple layers on oil-in-water droplets reducing the interfacial tension (Dickinson, 2011). This may account for the higher stability in TNM.3 and TNM.2 as compared to TNM.1

In the second stage (≥16 h), TNM.3 formed aggregates at the lipid-rich upper layer consisting of proteins, lipids and carbohydrate. The biological polymers in the upper layer might have aggregated because of an increase in interactions of the partially unfolded protein (due to the energy intake during milling and dispersion), the lipid and carbohydrates. It is reported in literature that when interactions in the biological polymers are stronger than that in the biological polymer-aqueous layer in thermodynamically unstable colloids, the polymers aggregate, which result in phase separation (Doublier *et al.*, 2000; Dickinson, 2011; Cheetangdee & Fukada, 2014). Thus, the optimisation of milling may be expedient to obtain tiger nut milk which has a better resistance to aggregative phase separation.

Colour stability

The average lightness L^* of freshly prepared tiger nut milk was 66.2 ± 0.30 for TNM.1, 67.6 ± 0.29 for TNM.2 and 66.2 ± 0.21 for TNM.3. For comparison, commercial full fat cow milk had 76.9 ± 0.35. A higher range of lightness for horchata products has been reported in literature (Mosquera *et al.*, 1996). The initial colour intensity ranged between C^* = 6.0 (TNM.1) and 6.7 (TNM.3), indicating that milling intensity and particle size distribution contribute to colour. The initial h_{ab} ranged between 1.4° and 1.5°. During storage, we observed a significant decrease in L^*, whereas C^* and h_{ab} remained almost constant. The effect of milling intensity on the fate of tiger nut milk lightness is depicted in Fig. 3. L^* of cow milk during storage that was also evaluated for control purpose remained constant (76.7 ± 0.59 after 27 day).

The increase in total milk solids caused by higher milling intensity may account for a lower decrease of the lightness observed for TNM.3. However, beyond a specific milling intensity, nonenzymatic browning of the milk due to the formation of Maillard products might cause a reduction in milk lightness with a corresponding increase in colour intensity (Cheetangdee & Fukada, 2014). Conversely, protein–sugar conjugates have also been reported to enhance colloidal stability through enhanced interfacial droplet properties, whilst they function simultaneously as potent antioxidants, reducing the rate of further decrease in lightness at lower storage temperature (Cheetangdee & Fukada, 2014).

Heat treatment of tiger nut milk has been reported to retard the rate of change from the characteristic nutty, sweet and vanilla-like flavour of fresh horchata to an earthy and bitter taste accompanied by darkening of the milk during storage (Mosquera *et al.*, 1996). It is proposed that the optimisation of the milling

intensity of tiger nuts may have prospects for an increase in the lightness and for a reduction in the brown characteristics of tiger nut milk and an overall improvement in the shelf life of the milk.

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Conflict of interest

The authors declare that there is no conflict of interest.

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