Yoghurt with candied chestnut: freeze drying, physical, and rheological behaviour

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Abstract As a novel product, yoghurt powder was produced by freeze drying and with added candied chestnut puree at ratios of 5, 10, and 20 % by weight. During the freeze drying process, mass loss, water activity, and the moisture content of the samples were determined and the colour (Hunter L, a, b) of the yoghurt powder products was measured. Results showed that increasing the percentage of candied chestnut puree resulted in an increase in water activity, moisture content, and colour change values of the end product. The drying behaviour, drying rate versus free moisture content, was also investigated. It was observed that yoghurt with or without added candied chestnut puree could be satisfactorily freeze-dried. Moreover, the performance of the dried product was observed in a ready-to-use, reconstituted form. For this purpose, the obtained powders were reconstituted to their original moisture contents. Shear stress and apparent viscosity against shear rate in a range of 1–1,000 (1/sec) was then measured by a Haake-Mars rotary viscometer. According to the results, the apparent viscosities of reconstituted products, as plain yoghurt and the one with an added 5 % chestnut puree were lower than that of fresh yoghurt. However, reconstituted yoghurts containing 10 % and 20 % chestnut puree had apparent viscosities higher than fresh yoghurt. Power Law explained well the rheological behaviour of reconstituted yoghurt samples for

the applied shear rate range. Based on rheological data and sensory analysis, it was concluded that the freeze dried yoghurt containing 10 % (w/w) candied chestnut puree was an acceptable novel product.

Keywords Freeze-drying behaviour · Yoghurt · Candied chestnut · Reconstitution · Rheology

Introduction

Yoghurt is a fermented dairy product obtained by lactic acid fermentation of milk by the action of yoghurt starter bacteria and is one of the most popular products throughout the world (Hayaloglu et al. 2007). The nutritional and therapeutic effects of yoghurt are well known and mainly attributed to fermentative changes in the milk and or the metabolic effects of the yoghurt micro flora (Venir et al. 2007). Yoghurt provides higher levels of protein, carbohydrate, calcium, and certain B vitamins than milk, since it is more concentrated. Better growth and increased nutrient utilization are associated with yoghurt consumption, due to increased protein digestibility. Milk fat also becomes easily digestible due to certain pre-digestion reactions during fermentation. Considering its therapeutic value, yoghurt is reported to have hypocholesterolaemic and antitumor effects (Kumar and Mishra 2004). Fresh yogurt is an example for naturally available probiotics; it contains live cultures of lactic acid-producing bacteria that can prevent traveler's diarrhea, antibiotic-induced diarrhea, rotavirus infection, and vaginal yeast infection (Rajasekaran and Kalaivani 2012). In addition, it may be possible to induce greater versatility in its sensorial properties by controlling texture and by the addition of ingredients such as fruit juices or fruit pieces, sweeteners, and flavours (Venir et al. 2007). Addition of such ingredients like pre-processed fruits or vegetables to dairy products allows production of novel foods.

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Sharma et al. (2011) developed a novel food product by combining freeze dried and conventionally dried broccoli sprouts powder with commercially available cheese powder. It was reported that freeze drying was more efficient in preserving the original nutritional and sensory characteristics of the dried broccoli sprouts. It was concluded that the blended cheese powder containing up to 20 % freeze dried broccoli powder was an acceptable product.

Chestnut (*Castanea sativa* Mill.) is a traditional produce imported from Europe to the Mediterranean countries (Chenlo et al. 2008). In Turkey, most chestnuts are used to produce candied chestnut for industrial purposes, and the remaining are consumed roasted or boiled. An average composition (wet basis) for chestnut is water 50.1 %, carbohydrates 41.2 % (starch 27.3 %), proteins 2.5 %, fats 1.9 %, and minerals 1.18 % (Chenlo et al. 2008). Because chestnuts have a high water content and high water activity they need to be preserved. Traditionally, freezing, cold storage, and drying are used to increase the shelf life of chestnuts.

The combination of candied chestnut and yoghurt, both of which are of traditional foods of Turkey, was investigated to produce a novel food product. Since the shelf life of plain yoghurt or with the candied chestnut puree addition is short, yoghurt can be dried by freeze, spray, and microwave drying methods to improve the shelf life by lowering its water content. The primary objective of yoghurt drying is to preserve the product in a shelf stable powder form of high quality without the need for refrigeration (Koc et al. 2010; 2012). Freeze drying, among all drying methods, allows food products to be dried at low temperatures leading to high quality products (Sharma et al. 1992; Sharma and Arora 1995; Kumar and Mishra 2004). Dried yoghurt can reduce transportation and storage costs because no refrigeration is required. However, balanced and substantial retention of the original properties is desirable for better consumer appeal.

The freeze drying process allows nutritional and sensorial qualities to be retained, together with extreme reduction in weight, high solubility, long shelf life at moderate temperature, and the possibility to perform rehydration at any desired level (Venir et al. 2007). Freeze drying is a process of high operational cost and up to now used only on an industrial scale for the drying of high value foods and food ingredients (Ratti 2001). For a comparison between the freeze drying cost and other methods of food preservation such as freezing or refrigeration, the energy used by the home storage freezer should also be taken into account (Flink 1977; Ratti 2001). With the increasing concern about food quality, this process could be considered as a valuable alternative to preserve foods or to produce a lyophilized bacterial culture (Dhewa et al. 2011). Freeze drying of yoghurt has been described by many researchers as having the best retained nutritional and sensorial qualities in comparison to those obtained using other conventional drying methods (Kumar and Mishra 2004).

The textural characteristics of dairy products are important consumer acceptance (Kumar et al. 2011). The consistency of yoghurt was studied by Sanz et al. (2008). In their study, yoghurts were enriched with fibre obtained from the non-edible part of asparagus shoots to assess how the method of extraction (water or ethanol) and then drying (oven or lyophilisation) affect their performance. It was found that fibre incorporation increased the consistency of the yoghurts, especially if the fibres had been extracted in ethanol and lyophilized.

In their study, Rascón-Díaz et al. (2012) showed that the yield stress displayed a significant irreversible decrease after spray drying of yoghurt containing hydrocolloids. It was concluded that spray drying disrupted the hydrocolloid polymeric network, thus causing drastic changes in the microstructure of the gel matrix despite the affinity of caseins for some hydrocolloids. Viscosity curves of this sample indicated that it decreased with increasing shear rate, because shear causes the disentanglement of yoghurt aggregates.

The objective of this study was to produce a novel product by combining candied chestnut with yoghurt, to determine the applicability of the freeze drying process to this product and to evaluate the drying behaviour and physical quality of the powders. Rheological behaviour of reconstituted powders was also investigated to evaluate the performance of the powder in a ready to use, reconstituted form.

Materials and methods

Yoghurt samples and preparation of samples containing candied chestnut

The white plain skim yoghurt, containing 14 % (± 0.1) dry matter, was purchased from a local producer (Pinar Dairy Products Inc., Izmir, Turkey). The candied chestnuts, containing 23.4 % (± 0.4) total sugar, were also purchased from a local supermarket (Kardelen Inc., Bursa, Turkey) and then ground to obtain a puree. The samples were then prepared by mixing the yoghurt with the candied chestnut puree at levels of 5, 10, and 20 % by weight. The initial dry matter content of the plain yoghurt and yoghurt samples containing candied chestnut 5, 10, and 20 % was found to be 14.0 (± 0.0), 15.4 (± 0.2), 19.2 (± 0.2), and 24.0 (± 0.1)%, respectively, by the standard gravimetric method (AOAC 1990).

Freeze drying

Samples of a 5 mm thickness were frozen in petri dishes in an air blast freezer (Frigoscandia, Helsingborg, Sweden) and freeze dried at the minimal vacuum chamber pressure reached by the freeze-drier apparatus (Armfield Model-FT33 Vacuum Freeze Drier, England) at a condenser temperature of -50°C . The

freeze-drying experiment was replicated twice for each sample. During the freeze drying process, samples were taken at pre-determined time intervals and a physical analysis on these samples was applied using two parallel measurements, the details are given in Section “Physical analysis; reconstitution of powder and sensory analysis”.

Physical analysis; reconstitution of powder and sensory analysis

Mass loss (%) during drying was calculated by measuring the weight loss of yoghurt after drying for a certain period of time. The water activity was measured by using a water activity measurement device (Testo AG 400, Germany) with ± 0.001 sensitivity and the moisture content (w/w) was determined by the standard gravimetric method (AOAC 1990). These analyses were performed as 2 parallel measurements, and the drying experiment was replicated two times. The drying rate (kg/h/m^2) was calculated as the amount of average moisture lost (kg moisture) per unit of drying time (h) per unit of drying surface area (m^2) of the yoghurt in the petri dishes and was presented as a function of average free moisture content (kg water/kg dry matter).

The colour values of fresh, powdered, and reconstituted yoghurt samples containing 5, 10, and 20 % by weight candied chestnuts were measured with a colorimeter (Hunter Lab, Colour Flex, USA) (Hunter 1975) and expressed as Hunter parameters L , a , and b , as an average of 4 parallel measurements with standard deviations.

The reconstitution of yoghurt powder was performed by mixing the powder with water manually, with a spoon, until the initial water content was reached, and was always done at the same speed and in the same direction in order to achieve reproducible results.

Sensory analysis of stirred fresh yoghurt and reconstituted samples was conducted as per the methods of Sakin et al. (2008) and Koc et al. (2010), in two parallels. The overall acceptability of the product was carried out by adopting the classic standard scoring test (Altug and Elmaci 2005) by a panel of 8 semi-trained people, in the age range of 24–45 years, who were from the academic staff and post graduate students of the Food Engineering Department. The overall sensory acceptability of the reconstituted powder was evaluated taking into account the parameters such as consistency in the mouth, consistency on the spoon, appearance, and taste and flavour, each on a 5-point scale (Altug and Elmaci 2005) as details were given by Sakin et al. (2008) and Koc et al. (2010).

The results were analysed for mean values and standard deviations. Also, “Univariate Analysis of Variance and the Duncan post hoc test” by SPSS (version 15.0) were applied to analyse and evaluate statistically (5 % levels) the effect of the candied chestnut puree ratio on the colour values and at

the same time, the chestnut puree ratio and drying time on the mass loss, water activity, and moisture content of fresh, powdered, and the reconstituted form of yoghurt samples.

Rheological behaviour

In order to observe the rheological behaviour, fresh and reconstituted yoghurt samples were subjected to the measurement of apparent viscosity (Pa.s) and shear stress (Pa) against shear rate in an increasing range of 1–1,000 (1/s) using a Haake-Mars rotary viscometer, with a peltier temperature controller at 20 °C, as two parallels. Prior to measurement, the fresh sample was subjected to mixing in the same way, as with the reconstituted samples, in order to give the same thermo-mechanical history and to satisfy comparability. Shear rate-shear stress relation for the increasing shear rate range of 1–1,000 was expressed by power law (Eq. 1), for all yoghurt samples, fresh and reconstituted, as plain and candied chestnut added;

$$\text{Shear stress (Pa)} = K \times \text{shear rate}^n \quad (1)$$

where, K is the consistency index (Pa.s^n) and n is flow behaviour index; the regression analysis was performed by using MS Office Excell, 2007. The samples of 1.5 ml were tested each time and a cone and plate probe at a 60 mm radius and 1 ° inclination were used. One-way Analysis of Variance of the means was applied to the results for different yoghurt samples to determine whether the means of n and K are different significantly (5 % levels).

Results and discussion

Freeze drying was applied until there was no significant change in weight, which was approximately 8 h. The mass loss during freeze drying was 87.05 (± 0.10), 83.80 (± 0.10), 80.39 (± 0.25), and 74.79 (± 0.91) % on an average for plain yoghurt and samples containing candied chestnut at 5, 10, and 20 %, respectively (Fig. 1a). As the candied chestnut ratio increased from 0 (plain) to 20 %, the final mass loss percentages were observed to reduce significantly ($p < 0.05$).

Although, the moisture content of the yoghurt samples decreased with the addition of candied chestnut, the drying time of candied chestnut added yoghurt samples was observed to be longer compared to plain yoghurt ($p < 0.05$) (Fig. 1b). There exist several studies related with freeze drying of yoghurt; however, because of the differences in experimental conditions the comparability of the results with ours is low. Freeze drying of yoghurt at -23 °C and 400 mmHg for 18 h with a final moisture content of 2–3 % (Kumar and Mishra 2004) and at -34 °C and 20 Pa (Venir et al. 2007) to obtain an anhydrous product are some examples.

Fig. 1 **a** Mass loss (%), **b** Moisture content (weight/weight), **c** water activity (a_w) of plain and candied chestnut added yoghurt samples during freeze drying ($n=4$)

Water activity is an important consideration for safety of food materials. At the drying times between 4 and 7 h, the water activities of the candied chestnut added yoghurt samples was observed to be higher ($p<0.05$) which was proportional with the amount of addition, than the plain yoghurt (Fig. 1c). However, at the end of 8 h of freeze drying, the samples were no different, in terms of water activity values statistically ($p>0.05$), and all of them were within the acceptable limits (<0.2) as expected for powdered milk products.

The drying curve (Fig. 2) as drying rate (kg moisture/ $m^2 \cdot h$) versus average free moisture content (kg water/kg dry matter) was obtained by the use of average moisture content data. The drying rate for all samples corresponded to the falling rate period throughout drying except the plain yoghurt at initial drying times. Plain yoghurt, which had a higher initial moisture content, showed an increase in drying rate at the beginning when the surface pores were just opened, later it followed a falling rate period. Addition of candied chestnut at ratios of both 10 and 20 % carried the drying rate beyond the line of

Fig. 2 Drying rate versus average free moisture content for plain and candied chestnut added yoghurt samples ($n=4$)

plain yoghurt, at the initial stages of drying. The sample containing chestnut puree at 20 % showed the highest drying rate profile despite its highest initial dry matter content. Moisture removal behaviours of these samples were mostly affected by the chestnut puree addition and its cellulosic structure, drifting apart from the yoghurt protein network at the initial stages of drying. After this stage, at nearly 1 (kg water/kg dry matter) and lower free moisture contents of material, the plain yoghurt lost moisture faster than the chestnut puree added samples.

According to the results of colour analysis, it was observed that both the process of drying and the amount of addition of the candied chestnut had an effect on the colour values (Hunter L, a and b) ($p<0.05$). Considering Hunter L values, which is an indication of lightness of the sample (Fig. 3a); L values decreased with the addition of candied chestnut, for all fresh, dried powder, and reconstituted samples ($p<0.05$).

Depending on the colour theory, +a is a representation of redness and -a is a representation of greenness. According to the results (Fig. 3b), the addition of candied chestnut changed the colour scale to redness as with plain yoghurt, drying also increased the “a” value; however, reconstitution of the dried powder decreased to a value slightly more than the original value ($p<0.05$). Although for the candied chestnut containing samples, drying decreased “a” values, this effect was reversed for the reconstituted samples ($p<0.05$).

When the results for “b” values (yellowness) were considered for all samples, dried powders had “b” values less than the original sample and reconstitution increased “b” values of powders to values slightly less than the original ($p<0.05$) (Fig. 3c).

The sensory panel evaluated the reconstituted samples, each having the same dry matter content of 25 % (w/w), this is a dry matter content level established by the panel to have an acceptable reconstituted product. The highest appearance and flavour scores were for the 10 % candied chestnut added yoghurt sample. For consistency in the mouth and on the spoon, other than plain reconstituted yoghurt, which had the

Fig. 3 Average Hunter **a** L, **b** a, **c** b values and standard deviations of plain and candied chestnut added yoghurt samples during freeze drying ($n=4$)

lowest scores (1.9, 1.7, respectively), all reconstituted yoghurt samples received scores higher than 3.5, whereas, the 10 % chestnut added sample scored 4.1 (± 0.8) and 4.2 (± 0.7) respectively, out of 5 total points. According to the panel's average scores, the product with 10 % candied chestnut was most acceptable.

Apparent viscosity values (Pa.s) of yoghurt samples as a function of shear rate (1/s) were given in Fig. 4 for the increasing shear rate range of 1–1,000 1/s. Reconstituted samples of plain and 5 % candied chestnut containing yoghurt had apparent viscosity values lower than that of fresh yoghurt. On the other hand, it was observed that the addition of candied chestnut to yoghurt at 10 and 20 % by weight resulted in an increase in apparent viscosity values of reconstituted powders.

Fig. 4 Apparent viscosity of yoghurt samples (Pa.s) as a function of shear rate (1/s) ($n=2$)

The shear rate-shear stress relationship was explained well by the power law for reconstituted yoghurt samples, for the applied shear rate range of 1–1,000 (1/s). However, for the reconstituted yoghurt sample containing 20 % candied chestnut, due to the existence of tiny chestnut particles which were not easily dissolved, there existed a deviation from the power law at the shear rates lower than 250 (1/s). The mean and standard deviations for the constants n and K for Eq. 1 are given in Table 1.

The addition of candied chestnut puree at the 10 and 20 % levels resulted in a decrease of the flow behaviour index (n) values ($p < 0.05$), that is, they had values further removed from Newtonian behaviour ($n=1$), which indicated a greater resistance to the flow. On the contrary, reconstituted plain and 5 % candied chestnut puree added yoghurt samples had higher n values compared to fresh yoghurt ($p < 0.05$) indicating an apparently poor viscosity. K values of 10 and 20 % candied chestnut added yoghurt samples were higher ($p < 0.05$) than that of fresh yoghurt indicating an increased consistency at this level of addition.

A similar behaviour with our results was indicated by Sanz et al. (2008) for the fibre enriched yoghurts. They

Table 1 Determined n and K constants of Eq. 1 for yoghurt samples

	n	K (Pa.s ^{n})	R^2
Fresh	0.42 ± 0.01^a	2.20 ± 0.00^a	0.99
Plain	0.58 ± 0.00^b	0.42 ± 0.01^b	1.00
5 %	0.58 ± 0.00^b	0.47 ± 0.01^c	1.00
10 %	0.35 ± 0.00^c	4.47 ± 0.01^d	1.00
20 %	0.36 ± 0.00^c	3.74 ± 0.01^c	0.97

All data are reported as means $\pm$ standard deviations of two parallels ($n=2$). The different letters after the values in the same column indicate that one-way ANOVA of the means is different significantly ($p < 0.05$)

could adjust the shear stress–shear rate curves over the 0.1–100 s^{−1} shear rate range to the Herschel–Bulkley model and showed that the addition of fibre to yoghurt, with the exception of yoghurts enriched with fibre extracted in water and dried in the oven, brought about a lowering of the flow index. Also, they showed that the addition of all types of fibres significantly increased the value of the threshold force and of the consistency index.

Wen et al. (2012) obtained the flow behaviour for the set-yoghurt prepared from bovine milk, treated with horseradish peroxidase (HRP). The analysis results of flow behaviors of the yoghurt samples were fitted to the Ostwald de Waele model (Power law). They showed that HRP treatment of bovine milk enhanced consistency coefficient (K) significantly ($p < 0.05$), but had no influence on flow behaviour indices (n) of the yoghurt ($p > 0.05$).

Yoghurt is a complex material. It exhibits irreversible time-dependent shear behaviour, and shear rate thinning (van Marle et al. 1999 and Rascon-Diaz et al. 2012). Rascon-Diaz et al. (2012) suggested that, visual inspection of the viscosity curves of fresh yoghurt and reconstituted natural yoghurt (without added hydrocolloids) subjected to spray drying, indicates a decrease in viscosity in both yoghurt samples. This decrease in viscosity and shear stress peak in the flow curves of the reconstituted yoghurts suggest that the microstructure of the yoghurts may have been disrupted by the spray drying process. In addition, yoghurt was given as an example of thixotropic material by Magenis et al. (2006). In our measurements, fresh yoghurt showed time dependency and when the shear rate reduced from 1,000 to 1 (1/s), there also existed a thixotropy loop. However, since drying itself caused an irreversible loss of resistance in the yoghurt matrix, the reconstituted plain yoghurt samples showed a lower time dependency. Candied chestnut puree addition at a ratio of 10 % gave reconstituted yoghurt a plumpy structure and when reduced shear was applied, a higher time dependency than that of plain yoghurt was observed.

Conclusions

Results showed that increasing the percentage of candied chestnut puree resulted in an increase in water activity, moisture content, and colour change values of the end product. Yoghurt, with or without added candied chestnut puree, could be satisfactorily freeze-dried. The rheological measurements showed that although reconstituted powders lost consistency compared to fresh yoghurt, addition of candied chestnut at 10 % or more could increase apparent viscosity and improve the consistency. The power law well explained the rheological behaviour of reconstituted yoghurt samples for the applied shear rate range. The poor rheological behaviour, which is an important problem for reconstituted yoghurt powder, could be

improved with the addition of a chestnut puree at 10 % (w/w) which was also observed to be an acceptable product based on sensory analysis.

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