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Changes in the functional constituents and phytic acid contents of firiks produced from wheats at different maturation stages

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ABSTRACT

Three different wheat cultivars (Bezostaya, Eser and Cesit-1252) at four maturity stages were processed into firik which is a whole wheat-based ethnic food by using traditional cooking method. Some nutritional and anti-nutritional (moisture, ash, protein, fructans, dietary fibers, phenolic contents and antioxidant activity) properties of firiks were investigated. A significant increase was observed for the hectoliter and 1000 kernel weights whereas a decrease for ash and protein contents by increased maturation level. It was found that immature wheats especially at early stages of kernel development are rich sources of functional nutrients such as total dietary fiber (17.3–20.4%), fructans (4.1–7.2%), total phenolics (4602.5–4838.3 mgGAE/kg) and antioxidants (729.2–782.8 μmolTE/100 g) besides having lower phytic acid contents (498.6–604.9 mg/100 g).

1. Introduction

Firik (also known as frekeh) is one of the oldest and traditional whole wheat-based foods with a unique smoked flavor. The annual production amount is reported as about 200–300 thousand tons in the Middle East. The popularity of this immature wheat product is increasing gradually throughout the world as well as already being consumed in several countries of the Middle East and also North Africa (Al-Mahasneh & Rababah, 2007). Firik is mainly used as a substitute for rice and bulgur in pilav as well as the consumption of firik is quite similar with bulgur. Firik-pilav is a traditional and popular meal in some regions of Anatolia, it is consumed especially with meat, tomatoes by frying in fat with different spices. The product is generally home-made for domestic consumption or commercially produced by small-scale manufacturers (Maskan, 2001; Özboy, Özkaya, Özkaya, & Köksel, 2001; Özkaya, Özkaya, Eren, Ünsal, & Köksel, 1999).

Selection of the right time for harvesting, wheat cultivar and production method are crucial points for the nutritional quality and flavor characteristics of firik. The best harvesting time for firik production is generally accepted as interval from late-milk-ripe stage to mid-dough-ripe stage especially because of the better taste of those due to the higher content of free simple sugars when compared with the ones processed at the full ripe stage. From the point of wheat cultivar, Durum wheats (*Triticum durum*) have been primarily preferred more when compared with common hard wheats (*T. aestivum*) in firik production since the best firik is made from the hardest kernels (Özkaya et al., 1999; Özkaya, Özkaya, & Köksel, 1998).

Physicochemical characteristics of firiks produced from immature wheats can also considerably vary depending on the cultivar and the maturation period beside the nutritional composition. The previous studies indicated that test weight, kernel weight and kernel size increased while protein, ash, thiamin and riboflavin contents decreased with maturation. Furthermore, the phytic acid (PA) content increased and the total phosphorus content decreased during maturation (Abernethy, Paulsen, & Ellis, 1973; Preston, Kilborn, Morgan, & Babb, 1991; Skarsaune, Youngs, & Gilles, 1970; Özkaya et al., 1999). PA ($C_6H_{18}O_{24}P_6$, inositol hexaphosphates-IP_6) is the primary storage form of phosphorus in most legumes and cereal grains (Ma, Zuo, Sun, Wang, & Guo, 2013; Reddy & Sathe, 2001; Turksoy, Özkaya, & Akbas, 2010). PA has been regarded as an anti-nutrient since it forms stable complexes with dietary mineral cations (Ca^{+2}, Zn^{+2}, and Fe^{+2}), and reduces their bioavailability. Thus, diets with high phytate content may lead to deficiencies in micronutrients such as iron and zinc (Ma et al., 2013; Turksoy et al., 2010; Özboy et al., 2001). In contrast with this negative feature of PA, it may play important roles in human health as an antioxidant and anticancer agent with its relatively high binding affinity

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of iron (Ma et al., 2013).

In addition to the positive health effects of whole grains, immature wheat products like firik provide even further health benefits with its excellent health-related properties, such as high contents of dietary fibers, fructo-oligosaccharides (FOS) and fructose rich polymers and phytochemicals like phenolics and antioxidants. These compounds exhibit a wide range of differing biologically protective effects against obesity, type-2 diabetes, cardiovascular complications, gastrointestinal disorders and certain types of cancer. Many of these positive health effects can be attributed to the non-fructan compounds. Cereal products prepared from immature wheat grains such as firik can be classified as a functional food with their unique health related properties (Arrizon, Morel, Gschaedler, & Monsan, 2010; D'Egidio, Cecchini, Cervigni, Donini, & Pignatelli, 1998; Kendall, Esfahani, & Jenkins, 2010; Maskan, 2001; Roberfroid, 1999).

The maximum accumulation of these functional compounds occurs after the anthesis and lasts until the milk-ripe stage. During this period, the photosynthesis products that exceed the growth requirements are stored in the stems and then used in the following phase for grain maturation. Thus, it can be noted that the harvest of winter cereals at the milk-ripe stage provides many health benefits, so increasing the fructan level of wheat grain could be a suitable way to increase fructan intake and thereby improve iron and calcium availability for a large number of people (D'Egidio, Cecchini, Cervigni, Donini, & Pignatelli, 1998; Huynh et al., 2008).

It is certain that different wheat varieties and their various maturation stages would affect the chemical composition of the end products and also that of firik. Therefore, there is a large number of research reports in which the chemical composition and different kinetic parameters of different cereals at various conditions were investigated (Abernethy et al., 1973; Alfieri & Redaelli, 2015; Elgün, Ertugay, & Certel, 1990; Kean, Ejeta, Hamaker, & Ferruzi, 2007; Maskan, 2001; McCallum & Walker, 1990; Skarsaune et al., 1970; Xu et al., 2010). However, as far as we know, very little information is available about firik in the literature related to its physical, chemical and hydration properties under different processes (Al-Mahasneh & Rababah, 2007; Maskan, 2001; Özkaya et al., 1999). Therefore, the present study was undertaken to fill the gap in the literature about the relation between wheat maturation level and the nutritional benefits of end products. Thus, the main objective of the study is to determine the effect of different maturation stages on the composition and nutritional properties of firiks. The following properties of firik were investigated: physical parameters (test weight, 1000 kernel weight), basic descriptive chemical compounds (moisture, ash and protein contents), functional compounds (fructans, dietary fibers, antioxidants and phenolics) and also phytic acid.

2. Materials and methods

2.1. Materials

Three different wheat varieties cvs. C-1252, Bezostaya and Eser were used in this investigation, which were obtained from Research and Application Farm of Ankara University, Faculty of Agriculture, Ankara, Turkey. All wheat varieties used for firik production were harvested in 2015. Two of them are Hard Red Winter wheats and one of them is a durum wheat. The materials were sampled from the field at four different maturation stages, 10, 15, 20, 25 days after anthesis. The sample of each wheat cultivar harvested at full-ripe stage was used as respective control. All reagents and solvents used in this study were ordered from Sigma-Aldrich, USA.

2.2. Preparation of firik samples

The preparation method (Özkaya et al., 1999) for the Firik samples was given in Fig. 1.

For each cultivar these sample preparation methods were performed in duplicate and prepared samples were kept in sealed bags under cold temperatures until further analyses.

2.3. Extraction of free phenolic compounds

Free phenolic compounds of firik samples were extracted by mixing 0.5 g of sample with 5 mL of acetone: water mixture (1:1, v/v) for 1 h in stirring shaker at room temperature. After centrifugation at 2500*g* for 10 min, the supernatant was filtered through Whatman No. 42 filter paper and extraction was repeated two times. Supernatants were pooled and evaporated at 40 °C using a rotary evaporator (Buchi, Rotavapor R-210, Switzerland). The resulting solutions were kept at −20 °C under nitrogen gas in amber colored glass bottles (ISOLAB, Germany) until further analysis, after the extracts were dissolved in 2 mL DMSO (dimethyl sulfoxide solution) (Adom & Liu, 2002).

2.4. Extraction of bound phenolic compounds

The residues remained after extraction of free phenolics was digested with 2 N sodium hydroxide (1:40, w/v) at room temperature for 4 h by stirring. The pH of samples was adjusted to 2.0 with 6 M HCl solution. After neutralization, the mixture was extracted with 20 mL of hexane to remove lipids. After centrifugation at 2500*g* for 10 min, hexane was removed from the samples. This procedure was repeated one more time. The final solution was extracted five times with an appropriate amount of diethylether: ethylacetate mixture (1:1, v/v). After centrifugation at 2500*g* for 10 min the diethylether: ethylacetate fraction was pooled and evaporated at 40 °C using a rotary evaporator (Buchi, Rotavapor R-210, Switzerland). Samples were dissolved in 2 mL DMSO and were kept at −20 °C under nitrogen gas in amber colored glass bottles (ISOLAB, Germany) until further analysis (Adom & Liu, 2002).

2.5. Analytical methods

The test weight was determined by using on Ohaus test weight apparatus and reported on an 'as is' moisture basis. The 1000 kernel weight was determined by counting the number of seeds in 20 g of grain and reported on dry basis. Moisture and ash contents of the firik samples were determined using ICC Standard Methods 110/1 and 104/1, respectively (ICC., 2002). AACC Approved Method 46–10 was used for the determination of protein contents of the samples (AACC, 1999). Total phosphorus (P) content was estimated spectrophotometrically according to the method reported by Rickey and Evans (1955) after the samples were prepared by the wet ash method (Garcia, Blessin, & Inglett, 1972). Phytic acid content was determined according to the colorimetric procedure of Haug and Lantzsch (1983). Insoluble dietary fiber (IDF) and total dietary fiber (TDF) contents of firik samples were determined by using AACC Approved Method 32-07 (AACC, 1999). Soluble dietary fiber (SDF) was calculated as the difference between TDF and IDF. Fructan (fructo-oligosaccharide) contents of firik samples were determined using a Megazyme kit (K-FRUC), based on AOAC Method 999.03 (AOAC, 2012) and AACC Method 32.32 (AACC, 1999).

Phenolic content and antioxidant capacity of each sample were determined according to the methods described by Yu et al. (2002), after the extraction process mentioned by Adom and Liu (2002).

The phenolic contents of each extract were determined using Folin-Ciocalteau reagent. Briefly, 100 µL of extract was oxidized with Folin-Ciocalteau reagent, and the reaction was neutralized with sodium carbonate. The final volume was made up to 10 mL with pure water. After 2 h of incubation, the absorbance at 765 nm was measured and used to calculate the phenolic contents using gallic acid as standard. Results were expressed as milligram of gallic acid equivalent per kilogram of sample (Yu et al., 2002).

The antioxidant activities of samples were determined using 2,2-di-