ORIGINAL RESEARCH PAPER

IMPACT OF DRYING METHOD ON BIOACTIVE COMPOUNDS, FUNCTIONAL AND THERMAL PROPERTIES OF DURUM WHEAT (TRITICUM DURUM) SPROUTS

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Abstract
Sprouting is recognized as a green engineering tool for improving cereal's nutritional properties. Thus, the incorporation of sprouts in food formulation would be recommended. However, sprouting also increases the seeds’ water content which shortens their shelf life. Thus, drying is recommended to decrease moisture content. This research aimed to investigate the impact of different drying methods (Oven drying, lyophilization and micro-wave vacuum drying (MVD)) on durum wheat sprout’s bioactive compounds, physicochemical, functional and thermal properties through differential scanning calorimetry (DSC). Durum wheat was sprouted for 48h, then water content, water activity (a_w), color, bulk density, Water Absorption Capacity (WAC), Oil Absorption Capacity (OAC), Swelling Power (SP), Least Gelation Concentration (LGC) and thermal properties through DSC were assessed for raw and dried sprouts. Sprouting and drying significantly affected the evolution of all properties. In all cases, water content and a_w decreased to an acceptable level. Oven and MVD dried samples were darker than freeze-dried ones. Compared to the raw samples, bulk density and SP decreased while OAC increased. WAC decreased after lyophilization (-14.3%) while it significantly increased (+90.5%) after MVD. DSC measurement showed two endotherms with peak temperatures between 39.7 and 46.6 °C for the first and 101.9 to 108.5 °C for the second. Regarding nutritional properties, lyophilization preserved them the best. However, MVD induced lower losses than oven drying. Understanding changes occurring after drying would help choose the suitable drying technology and better use dried sprouts after knowing their properties.

Keywords: bioactive compounds, drying, functional properties, sprouts

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Introduction

Cereals play a key role in the human diet. Moreover, they are found in a varied range of products like bread, pasta and cookies. Sprouting is a green engineering tool contributing to improving cereals and pulses’ nutritional properties (Donkor et al., 2012). Thus, sprouts could be suggested as a potential functional ingredient for developing food products with added nutritional value. Unfortunately, sprouting increases seeds water content which may reduce their shelf life and make their use difficult in many food products, especially cereal ones. One of the ways of extending the shelf life of food products is drying. In fact, drying is one of the oldest ways used for food preservation (Maskan, 2000) as it reduces water activity and consequently extends shelf-life (Zhang et al., 2006), while quality degradation could occur (Maskan, 2000). In this context, studies have compared several drying methods’ impact on food’s functional properties, as reviewed by Dehnad et al. (2016).

Convective drying is an ancient and common method used (Dorofejeva et al., 2011) due to its simplicity and low cost. However, it might cause quality alteration (Giri and Prasad, 2007). The lyophilisation (or freeze drying) process is known for preserving plant material composition (Georgé et al., 2011). Consequently, freeze-dried products have high nutritional and sensory properties (Wojdyło et al., 2016). However, the use of this method might be restricted due to its long duration and cost.

The use of microwave drying has also been suggested (Bondaruk et al., 2007) since the drying coefficient and dehydration rate are improved with this method. However, product properties such as color, nutrient content and texture may be affected (Therdthai and Zhou, 2009). Researchers investigated Microwave-Vacuum Drying (MVD) as a potential alternative to optimize microwave drying (Giri and Prasad, 2007). It combines the advantage of both technologies: drying time is shortened and the process occurs at low temperatures (Therdthai and Zhou, 2009) which may contribute to improving the quality of the dried product.

Functional properties play a crucial role in food conception. These properties are affected by several factors such as storage conditions, ingredients composition (proteins, carbohydrate, lipid amounts) (Dehnad et al., 2016) and processing like drying. In this context, kilning or drying of germinated seeds is an essential step in the sprouting process in case of their further use instead of their consumption raw. It allows the product stability, handling and milling. Meanwhile, some biochemical changes may occur according to the drying parameters (Woffenden et al., 2002). The work of Shingare and Thorat (2014) focused on the effect of fluidized-bed drying of sprouted wheat with different operating conditions on color, physical properties, proximate composition and effective diffusivity. No literature was found on the comparison of the effect of different the drying methods on the properties of sprouted wheat seeds. Therefore, this research investigated the impact of drying method on the sprout's nutritional, functional and thermal properties.
Materials and methods

Materials

A Tunisian cultivar of durum wheat (*Triticum durum*) “Karim” provided by the National Institute of Cereal Crops (INGC) (harvested in June 2015) (Bou Salem, Tunisia) was used in this study.

Sprouting

Seeds were sprouted as described by Jribi *et al.* (2019). Seeds were disinfected with a hypochlorite sodium solution of 1% (V/V), soaked in distilled water for 40 min and finally, water was removed and seeds were spread on plates with “Blotting paper”. Samples were watered after 24 hours with distilled water. Sprouting was conducted at a temperature of 22 ± 1 °C for 48 hours.

Drying methods of sprouted seeds

Sprouted seeds were dried using three different methods: lyophilization, Microwave vacuum drying (MVD) and oven drying at 50°C.

Sprouted seeds were frozen at -80 °C then lyophilization was carried out in a freeze dryer (Christ Alpha 1-4 LCS, Germany) for 18 h (-55°C condenser temperature, 2.2 kPa chamber pressure).

Microwave vacuum drying (MVD) was performed with a custom-designed MVD dryer. The apparatus contains a cylindrical stainless steel vacuum chamber with a conical dome for better vapor removal. The samples were located in a rotary polytetrafluoroethylene (PTFE) tray. Microwaves were generated by two 850 W rated output magnetrons, operating at 2450 MHz. The vacuum was kept constant at 1 kPa by a rotary vane vacuum pump, connected to a shell and tube heat exchanger for vapor condensation. The cooling water for the heat exchanger a compressor was provided and was kept circulated by a pump (Ferenczi *et al.*, 2017). Intermittent drying was applied: samples were dried with 60 s microwave pulses followed by a 60 s break.

For convective hot air drying, a laboratory-scale hot-air dryer (L-MIM 320, Hungary) was used: Temperature was set at 50 °C and airflow was 0.9 m.s⁻¹.

For oven drying and MVD experiments were stopped when samples’ moisture content ranged between 10 and 15% (wet basis). All drying experiments were performed in triplicate. After drying, all samples were milled (Reisch Grindomix GM 200, Germany). Milled raw seed and dried sprouted wheat powders were hermetically packed and stored at 4 °C until analysis. Physico-chemical, functional and thermal properties, as well as bioactive compounds and antioxidant activity were investigated on both raw and sprouted wheat seeds.

Physico-chemical properties

The moisture content of all samples was analyzed using the AOAC oven method (2000). Water activity was measured by Novasina LabMaster aw (Switzerland) device at 25 °C. Color parameters were measured with a Konica Minolta Croma Meter CR 400 (Japan). The CIE LAB color space was used to express color
parameters, where L* stands for lightness (0 for black, 100 for white) –a* is green, +a* is red, –b* is blue and +b* is yellow tint.

**Functional properties**

Bulk density was assessed as described by Singh *et al.* (2017). Water Absorption Capacity (WAC) and Oil Absorption Capacity (OAC) were determined according to the procedure described by Kaushal *et al.* (2012). Swelling Power (SP) and Least Gelation Concentration (LGC) were evaluated as suggested by Singh *et al.* (2017).

**Bioactive compounds and antioxidant activity**

Total phenol content was determined using the Folin-Ciocalteu method (Aprodu and Banu, 2012) wherein gallic acid was used as standard. Total carotenoid pigments were determined according to the procedure described previously (Pasqualone *et al.*, 2017). DPPH (1,1-diphenyl-2-picrylhydrazyl) radical scavenging activity (DPPH RSA) was measured using the method proposed by Aprodu and Banu (2012) with slight modification during the extraction procedure for antioxidant activity measurement. In this study, the extraction was done with 80 % (v/v) aqueous methanol solution, for 2 h at 37 °C. Samples were afterward centrifuged at 3508 x g for 30 min. The supernatant was used for determining the antioxidant capacity. Antioxidant activity was calculated according to equation 1.

\[
\% \text{ DPPH RSA} = \left( 1 - \frac{A_{\text{sample}} (t=30)}{A_{\text{control}} (t=0)} \right) \times 100
\]

**Differential scanning calorimetry (DSC) measurements**

Thermal properties were determined using a DSC 131 (SETARAM, France). Flour samples (from raw and dried sprouted seeds) (20 mg) were weighed into aluminum pans. Samples were hermetically sealed and allowed to stand for 2h at room temperature before testing. An empty pan was used as a reference. DSC analyses were carried through 3 cycles of heating-cooling (Ciesla and Eliasson, 2007): Samples were kept at 30 °C for 5 min then heated from 30 to 110 °C at 5 °C·min\(^{-1}\) (heating cycle 1) (Martínez *et al.*, 2014). Samples were then cooled from 105°C to 30 °C at 20 °C·min\(^{-1}\). Two successive heating/cooling runs were conducted in similar conditions. The Universal Analysis2000 software was used to analyze the main endotherm of the DSC traces for onset temperature (T\(_0\)), peak temperature (T\(p\)) and enthalpy change (ΔH). As no significant differences were observed between the second and the third heating cycle, only the first and second heating cycle results will be presented.

**Statistical analysis**

Statistical analysis was carried out using the Minitab software (Minitab 17, USA). All experiments were carried out in triplicate and the average values were reported together with standard deviations. Analysis of variance (ANOVA) was performed using the Fisher test. Significance was defined at p < 0.05.
Results and discussion

**Physico-chemical properties**

Compared to raw seeds, sprouted ones have higher water content (56.74 %) as well as water activity (0.989) (Table 1). This significant increase ($p<0.05$) reaching 5 folds for moisture content, may reduce the shelf life of sprouted seeds as the conditions are optimal for microbial growth. For dried samples, the averages of moisture content were significantly ($p<0.05$) different according to the technology used. The lowest averages were obtained with lyophilization (6.89 %). The nature of transfers could explain the differences occurring during drying, mainly, heat and mass transfer.

**Table 1.** Effect of drying method on physical properties of sprouted whole wheat flour (n=3).

<table>
<thead>
<tr>
<th>Treatment</th>
<th>Water content (%)</th>
<th>Water activity ($a_w$)</th>
<th>Lightness index ($L^*$)</th>
<th>Redness index ($a^*$)</th>
<th>Yellow index ($b^*$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Raw</td>
<td>11.34±0.11 b</td>
<td>0.558±0.00 b</td>
<td>76.09±0.04 b</td>
<td>1.84±0.04 b</td>
<td>19.23±0.08 a</td>
</tr>
<tr>
<td>Sprouts</td>
<td>56.74±0.14 a</td>
<td>0.989±0.00 a</td>
<td>--</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>Dried sprouts</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Convective oven drying</td>
<td>11.17±0.05 bc</td>
<td>0.524±0.00 bc</td>
<td>76.13±0.05 b</td>
<td>1.42±0.01 c</td>
<td>18.12±0.04 b</td>
</tr>
<tr>
<td>Lyophilization</td>
<td>6.89±0.15 d</td>
<td>0.322±0.00 d</td>
<td>80.70±0.04 a</td>
<td>0.69±0.04 d</td>
<td>16.13±0.04 a</td>
</tr>
<tr>
<td>Microwaves vacuum drying</td>
<td>10.97±0.10 c</td>
<td>0.490±0.00 c</td>
<td>73.03±0.14 c</td>
<td>2.32±0.04 c</td>
<td>19.22±0.18 a</td>
</tr>
</tbody>
</table>

For each column, values represent the mean and standard deviation of three replicate samples. In a column, means with a common letter are not significantly different by Fisher’s test at $\alpha=5\%$

Water activity ($a_w$) level decreased significantly after drying following the same trend as moisture content. The highest averages were observed with convective drying (0.524) while the lowest corresponded to freeze-drying (0.322). In all cases, final levels are enough to stop bacterial growth.

The drying method significantly affected the color development of the dried product (Table 1). MVD contributed to samples darkening mainly through a decrease in lightness ($L^*$) and an increase in redness ($a^*$). Contrarily, freeze-drying led to an increase in lightness ($L^*$) and a decrease in redness ($a^*$) and yellow index ($b^*$). However, in oven drying, there was only a decrease in redness and yellow index. These findings are similar to the study of Agrahar-Murugkar and Jha (2010) where microwave drying induced the browning of sprouted soybean. However, in the same study oven drying at 60°C after steaming for 10 min increased redness and yellow index. These differences might be related to the difference between wheat and soybean and also experimental settings (steaming step and higher temperature).

**Functional properties**

Bulk density, as an indicator of flour particles’ heaviness (Singh et al., 2017), is an essential factor in deciding whether the flour is more suitable for use in food
preparations (flours with high bulk density) or complementary products (low Bulk density) (Kaushal et al., 2012). As seen in Table 2, the dried samples’ bulk density ranged between 0.65 and 0.72 g/ml. This parameter decreased significantly (p<0.05) if compared to a raw seed. Results of Singh et al. (2017) dealing with flours obtained from germinated sorghum seeds showed that this bioprocess decreased bulk density. In this study, it could be suggested that the significant decrease seen was not only due to germination but also due to the drying method. According to these results, whole mill flour from sprouted wheat could be used for food preparations and complementary products (Kaushal et al., 2012). Compared to raw whole mill flour, the drying method was a determinant in Water Absorbance Capacity (WAC) evolution (Table 2). MVD increased this property; lyophilisation decreased it while no significant changes were seen after oven drying. According to Dehnad et al. (2016), oven drying at low temperatures (40-50°C) increases water retention capacity. These results are not necessarily contradictory, in this study, drying is combined with the effect of the germination process. The functional properties of dried products depend on the drying parameters used (mainly temperature and time). Moreover, these properties also depend on germination and conditions used (temperature, duration, and soaking time), which negatively affect WAC (Singh et al., 2017; Singh et al., 2001). Probably, for this reason, WAC does not show differences between raw seeds and oven-dried samples. However, the effect of germination on WAC was more seen in freeze-dried samples (Table 2). Lyophilization is a dehydration process that preserves product properties and quality. Thus, the effect of the germination process (decrease of WAC) will be highlighted more.

**Table 2.** Functional properties of raw (unspotted) and dried sprouted wheat flour (n=3).

<table>
<thead>
<tr>
<th>Treatment</th>
<th>Bulk density (g.ml⁻¹)</th>
<th>Water Absorption Capacity (WAC) (g.g⁻¹ dm)</th>
<th>Oil Absorption Capacity (OAC) (g.g⁻¹ dm)</th>
<th>Swelling Power (SP) (g.g⁻¹ dm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Raw</td>
<td>0.75±0.01ᵃ</td>
<td>1.05±0.01ᵇ</td>
<td>1.15±0.02ᶜ</td>
<td>6.67±0.11ᵃ</td>
</tr>
<tr>
<td>Dried sprouts</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Oven drying</td>
<td>0.72±0.00ᵇ</td>
<td>1.04±0.01ᵇ</td>
<td>1.18±0.01ᶜ</td>
<td>4.44±0.08ᵇ</td>
</tr>
<tr>
<td>Lyophilization</td>
<td>0.71±0.00ᶜ</td>
<td>0.9±0.01ᶜ</td>
<td>1.37±0.01ᵇ</td>
<td>4.15±0.03ᵈ</td>
</tr>
<tr>
<td>Microwaves vacuum drying</td>
<td>0.65±0.00ᵈ</td>
<td>2.00±0.05ᵃ</td>
<td>1.60±0.02ᵃ</td>
<td>5.62±0.08ᵇ</td>
</tr>
</tbody>
</table>

For each column, values represent the mean and standard deviation of three replicate samples. In a column, means with a common letter are not significantly different by Fisher’s test at α=5%. dm: dry matter basis.

Regarding the increase of WAC seen on MVD dried samples, this evolution might be related to the microwave impact on decreasing particle size and protein alteration, as demonstrated (Walde et al., 2002). Water holding capacity increases as particle size decreases (Protonotariou et al., 2016). The results of Berton et al. (2002) showed a significant negative correlation between particle size (d90) and water hydration capacity. It could be suggested that the decrease in particle size
increases specific surface area per unit weight. Consequently, there is a more available surface to interact with (Protonotariou et al., 2016).

As shown in Table 2, raw seeds flour and oven-dried ones showed the lowest averages of Oil Absorption Capacity (OAC). When lyophilization and MVD methods were used, a significant increase was observed on OAC. A previous study dealing with sprouted cereals showed that sprouting increases OAC (Singh et al., 2017; Elkhalifa and Bernhardt, 2010). This increase could be explained by protein degradation taking place during sprouting under proteolytic enzyme activity (Alvarez-Jubete et al., 2009). Moreover, the observed increase in OAC could also be associated with the role of gluten proteins. Previous results showed that freeze and micro-wave drying induced morphological and structural modifications in wheat proteins, particularly gluten (Gianfrani et al., 2017; Liao et al., 2013). However, temperature less than 60-75°C does not affect these proteins (Which is the case for oven drying in this study) (Singh and MacRitchie, 2004). This agrees with the results of this study, as the highest OAC was recorded after freeze drying and microwave vacuum drying. The increase in OAC would be an advantage in food formulation as flavor retention and palatability might be improved (Hussain and Uddin, 2012).

Recorded averages of Swelling Power (SP) (Table 2) ranged between 3.95 and 5.91 g/g dm. Raw seeds flour had the highest values, while a significant decrease was observed with sprouted, dried samples. Considering dried specimens, oven drying decreased the most the SP contrarily to micro-wave vacuum drying (Table 2). The significant decrease in SP is probably due to the starch degradation under amylasic enzyme activity (Jribi et al., 2020) and drying time. Oven drying is a slow process compared to MVD; free water availability at the first stage of drying contributes to more degradation of starch molecules under amylasic enzymes. Singh and Kayastha (2014) reported that purified α-amylase from germinated wheat has an optimum temperature of 68°C. In this study, the temperature setting for oven drying was 50°C, the closest one to the optimum. The decrease of SP after sprouting has been previously reported (Singh et al., 2017). In addition, Chinma et al. (2015) reported that the size of particles, genetic background, and types of processing methods or unit operations might influence the swelling flour capacity.

The Least Gelation Concentration (LGC) corresponds to the lowest concentration at which gel remained in the inverted tubes. It reflects the minimum amount required of starch or blends of starch to form a gel: an increase in gelation concentration means an increase in needed amounts of starch to form a gel (Eke-Ejiofor, 2015). It is also an indicator of proteins' ability to provide a structure able to hold water and thus create a gel (Appiah et al., 2011). LGC could be considered as a reflection of denaturized sample molecules (Appiah et al., 2011). Dried samples showed different results according to the drying method used (Table 3). Lyophilization and oven drying led to an increase in LGC (from 8% for raw seeds to 12% and 15%, respectively), while no difference between raw seeds and MVD sprouted seeds flour was observed. Gelation properties are related to flour composition, mainly protein and starch. Competition for water between starch
gelatinization and protein gelation is the main phenomenon influencing flour’s gelation properties (Kaushal et al., 2012). An increase in LGC is followed by decreased swelling power (Kaushal et al., 2012). As the swelling power reflects the starch granules’ ability to hold water, an increase in this indicator means that a low starch concentration will be needed to form a gel. The same trend was observed in this study. The germination process induces an increase in gelation properties as some macro-molecules like starch and proteins are degraded under enzymatic action (Singh et al., 2017). This effect was observed in freeze and oven-dried samples at different extents. This fact could probably be related to starch and proteins’ evolution during the drying process. However, the MVD samples showed the same LGC as in the raw seeds. Considering swelling power (SP) (Table 2), it is seen that the lowest decrease in this parameter is for MVD. It might be proposed that this method has a lower impact on starch compared to other methods used. Meanwhile, microwave affects gluten protein structure (Liao et al., 2013). Thus, it may suggest that a combined role between proteins and starch erased the effect of sprouting on LGC.

Table 3. Least gelation concentration (LGC) and its evolution after sprouting and according to drying method.

<table>
<thead>
<tr>
<th>Treatment</th>
<th>2%</th>
<th>4%</th>
<th>6%</th>
<th>8%</th>
<th>10%</th>
<th>12%</th>
<th>15%</th>
<th>20%</th>
<th>25%</th>
<th>30%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Raw</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
</tr>
<tr>
<td>Dried sprouts:</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Oven drying</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>±</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
</tr>
<tr>
<td>Lyophilization</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>±</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
</tr>
<tr>
<td>Microwave vacuum drying</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
</tr>
</tbody>
</table>

- : Not gelled; ±: Slightly gelled ; +: Gelled completely

Bioactive compounds and antioxidant activity

Final product quality is among the concerns during drying. As shown in Table 4, freeze-dried samples had the highest carotenoid content. This increase, compared to raw seeds, reflects the role of sprouting in enhancing carotenoid levels as reported previously (Plaza et al., 2003). The use of MVD induced losses leading carotenoids amounts to a similar level as raw seeds. However, oven drying drastically reduced its levels. Regarding total phenol content, freeze-dried samples had also the highest averages. However, MVD and oven drying affected these compounds as recorded levels are lower than raw seeds ones. The decrease is more significant with oven drying. The increase in total phenol content in freeze-dried samples could be attributed to the effect of sprouting as reported (Alvarez-Jubete et al., 2009). The difference seen between oven and MVD could be explained by the role of drying time on phenols and carotenoid degradation. Multari et al. (2018) investigated the effect of different drying temperatures (40, 50, 60, and 70°C) on the quinoa seed’s carotenoid and
phenol contents. The authors reported that the highest recovery of total phenolic compounds was at 70°C, while the highest recovery of cumulative carotenoids was 60°C.

**Table 4.** Bioactive compounds and antioxidant activity of raw and dried sprouted wheat (n=3).

<table>
<thead>
<tr>
<th>Treatment</th>
<th>Carotenoid (mg β carotene. Kg⁻¹ dm)</th>
<th>Total phenol content (mg GAE. g⁻¹ dm)</th>
<th>DPPH RSA (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Raw</td>
<td>15.31±0.28b</td>
<td>15.84±0.94b</td>
<td>18.64±0.34c</td>
</tr>
<tr>
<td>Oven drying</td>
<td>6.16±0.37c</td>
<td>6.54±0.03d</td>
<td>13.47±0.37d</td>
</tr>
<tr>
<td>Lyophilization</td>
<td>20.28±0.32a</td>
<td>35.14±0.16a</td>
<td>33.46±0.41a</td>
</tr>
<tr>
<td>Microwave vacuum drying</td>
<td>15.52±0.30b</td>
<td>11.62±0.03c</td>
<td>23.65±0.37b</td>
</tr>
</tbody>
</table>

For each column, values represent the mean and standard deviation of three replicate samples. In a column, means with a common letter are not significantly different by Fisher’s test at α=5%. dm: dry matter basis; GAE: Gallic Acid Equivalent; DPPH RSA (1,1-diphenyl-2-picrylhydrazyl) radical scavenging activity.

DPPH Radical Scavenging Activity also increased after sprouting, as demonstrated by freeze-dried samples (Table 4), suggesting this bioprocess's role in improving antioxidant properties. Meanwhile, oven and MVD significantly affected DPPH RSA. In this study, DPPH RSA evolution is related to carotenoids’ evolution and total phenols ones (Pearson correlation coefficient 0.904 and 0.916, respectively).

**Thermal properties**

The effect of sprouting and drying methods on flour's thermal properties is shown in Table 5. In the tested temperature range (30-110°C), two events, two endotherms, were detected on the first and second scans. Previous studies dealing with starch and wheat flour at low water content also detected two endotherms (Liu et al., 2006; Wang et al., 2014). Noda et al. (2004) the first endotherm was attributed to starch gelatinization and the second to amylose-lipid complex dissociation. The gelatinization peak temperature (TP1) was significantly different among the tested samples. It ranged between 39.7-45.1°C for the first scan and between 46.6-41.8°C for the second one. Commonly, wheat starch gelatinization occurs at a range of 54-73°C (Liu et al., 2006). Measurement conditions and water content may affect endotherms (Liu et al., 2006). An increase in water ratio increases peak temperature (Wang et al., 2014), which was not the case in this study as no water was added.

In the second heating cycle, raw seeds flour and sprouted MVD dried seeds flours showed the highest averages for the first endotherm, followed by freeze-dried and oven-dried samples. These findings are consistent with our results on LGC analysis (Table 3), where the lowest concentrations were those of raw and MVD samples followed by freeze-dried and oven. An increase in LGC reflects that more starch is required to form a structured medium. Similar results obtained with raw and
sprouted MVD flours may reflect the microwave effect on starch molecules. Despite the effect of sprouting on starch degradation, the microwave may affect molecule structure. A previous study by Xie *et al.* (2013) showed that even a short time treatment with microwave modified potato starch crystalline structure might modify thermal properties. Similarly, Ndif *et al.* (1998) observed an increase in starch gelatinization degree after a short time of microwave treatment. The difference seen between freeze and oven-dried samples could be explained by drying history: sprouted seeds were frozen at -80 °C before lyophilization. Thus, enzymatic reactions were stopped. However, oven drying is a slow process (at least about 4 h to reach the required water content). Consequently, as long as there is available water, starch degradation could be continued during the first steps.

### Table 5. Thermal properties of raw and dried sprouted wheat (n=3).

<table>
<thead>
<tr>
<th>Heating cycle</th>
<th>Treatment</th>
<th>$T_{01}$ (°C)</th>
<th>$T_{p1}$ (°C)</th>
<th>$\Delta H_1$ (J/g)</th>
<th>$T_{02}$ (°C)</th>
<th>$T_{p2}$ (°C)</th>
<th>$\Delta H_2$ (J/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>1</strong></td>
<td>Raw</td>
<td>32.5&lt;sup&gt;a&lt;/sup&gt;</td>
<td>40.2&lt;sup&gt;a&lt;/sup&gt;</td>
<td>3.1&lt;sup&gt;a&lt;/sup&gt;</td>
<td>95.3&lt;sup&gt;c&lt;/sup&gt;</td>
<td>105.0&lt;sup&gt;b&lt;/sup&gt;</td>
<td>5.4&lt;sup&gt;bc&lt;/sup&gt;</td>
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<td></td>
<td>Dried sprouts:</td>
<td></td>
<td></td>
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<td></td>
<td></td>
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<tr>
<td></td>
<td>Oven drying</td>
<td>34.4&lt;sup&gt;cd&lt;/sup&gt;</td>
<td>41.5&lt;sup&gt;d&lt;/sup&gt;</td>
<td>3.2&lt;sup&gt;a&lt;/sup&gt;</td>
<td>95.7&lt;sup&gt;c&lt;/sup&gt;</td>
<td>105.1&lt;sup&gt;b&lt;/sup&gt;</td>
<td>4.6&lt;sup&gt;de&lt;/sup&gt;</td>
</tr>
<tr>
<td></td>
<td>Lyophilization</td>
<td>33.3&lt;sup&gt;de&lt;/sup&gt;</td>
<td>39.7&lt;sup&gt;e&lt;/sup&gt;</td>
<td>2.8&lt;sup&gt;b&lt;/sup&gt;</td>
<td>96.0&lt;sup&gt;c&lt;/sup&gt;</td>
<td>105.3&lt;sup&gt;b&lt;/sup&gt;</td>
<td>4.8&lt;sup&gt;cd&lt;/sup&gt;</td>
</tr>
<tr>
<td></td>
<td>Microwave vacuum drying</td>
<td>37.1&lt;sup&gt;b&lt;/sup&gt;</td>
<td>45.1&lt;sup&gt;b&lt;/sup&gt;</td>
<td>2.0&lt;sup&gt;b&lt;/sup&gt;</td>
<td>93.5&lt;sup&gt;d&lt;/sup&gt;</td>
<td>101.9&lt;sup&gt;e&lt;/sup&gt;</td>
<td>2.9&lt;sup&gt;f&lt;/sup&gt;</td>
</tr>
<tr>
<td><strong>2</strong></td>
<td>Raw</td>
<td>40.5&lt;sup&gt;a&lt;/sup&gt;</td>
<td>46.6&lt;sup&gt;a&lt;/sup&gt;</td>
<td>1.9&lt;sup&gt;b&lt;/sup&gt;</td>
<td>99.3&lt;sup&gt;a&lt;/sup&gt;</td>
<td>108.0&lt;sup&gt;a&lt;/sup&gt;</td>
<td>6.2&lt;sup&gt;ab&lt;/sup&gt;</td>
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<td>Dried sprouts:</td>
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<td>Oven drying</td>
<td>35.5&lt;sup&gt;c&lt;/sup&gt;</td>
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<td>1.9&lt;sup&gt;b&lt;/sup&gt;</td>
<td>98.1&lt;sup&gt;b&lt;/sup&gt;</td>
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<td>3.9&lt;sup&gt;e&lt;/sup&gt;</td>
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For each column, values represent the mean and standard deviation of three replicate samples. In a column, means with a common letter are not significantly different by Fisher’s test at α=5%. $T_{o1}$, gelatinization onset; $T_{p1}$, peak temperature; $\Delta H_1$, enthalpy.

Peaks of the second endotherm ranged between 101.9-105.3°C during the first heating cycle and 104.8-108.5°C during the second. The trend observed was the same: no significant difference between samples except in the MVD that showed the lowest values. Admitting that this peak corresponds to amylose-lipid complex dissociation, the results of this study seem in agreement with those of Noda *et al.* (2004). It is inferred that two days of germination did not affect the amylose-lipid complex. However drying method, mainly MVD, may affect the structure of this complex and thus peak temperature. A previous study dealing with gamma-irradiated wheat starch samples showed a difference in the peak temperature of the amylose-lipid complex transition between control and irradiated samples (Ciesla and Eliasson, 2003). During the first heating cycle, MVD induced a significant decrease in enthalpy if compared to raw seeds. However, no differences were seen among all samples during the second heat scan. The impact of MVD on enthalpy during the first heating cycle could be attributed to the structural changes (mainly...
on starch and protein) induced by this process. Since all the samples experienced the same heating-cooling steps, no differences were seen.

Conclusions
In this study, sprouted wheat seeds (Triticum durum) were dried according to three technologies: freeze drying, convective oven drying and Microwave vacuum drying (MVD). This study clearly showed that the evolution of bioactive compounds, physical, functional and thermal properties were strongly related to the germination process and the technology used. Lyophilization led to the highest nutritional quality. Interestingly, MVD induced fewer losses in bioactive compounds than oven drying (-23.5% for carotenoids, -67% for TPC, -29.32% for DPPH RSA). Considering drying time and energy consumption, to reach the desired moisture content (10-15%), 24 hours were required for lyophilization, 4 hours for oven drying, and only 25 minutes for MVD. Thus, MVD could be suggested as the most suitable technology to use for drying sprouts.

References


