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Comparative Study of the Physiochemical Composition and Techno-Functional Properties of Two Extracted Acorn Starches

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Abstract

Due to the increase of search for new promising ingredients with interesting properties to develop new industrial food products, the valorization of undervalued resources became a challenge. Considering this, various species of genus *Quercus* acorns represent new resources of highly-valued food ingredients such as starch which encourage its extraction and valorization in food industries. In this regard, collected data from the literature provide an evidence review on the physiochemical and techno-functional properties of different acorn starches extracted from Tunisian species, especially; *Quercus ilex* L. and *Quercus suber* L. The reported data on X-ray diffraction analysis are, also, discussed. Data highlighted the possibility of using the extracted *Quercus* starches to develop new functional food products and improve technological properties and shelf life of products solicited by consumers.

Keywords: acorn starch, physiochemical composition, techno-functional properties, X-ray diffraction analysis

1. Introduction

Genus *Quercus* acorns belong to the family *Fagaceae*, which includes several species such as *Quercus robur*, *Quercus petraea*, *Quercus suber*, *Quercus ilex*, and *Quercus pubescens* [1]. These species produce a widely known fruit, named acorns, which are of vital importance for both humans and animals. The acorn fruits composition varies with species and origin. Acorn fruits are good source of carbohydrates (starch), proteins, fats, minerals, essential amino acids, vitamins (mostly A and C), unsaturated fatty acids (oleic acid) and sterols [2]. The nutritional composition of acorn fruit is comparable to many cereal grains. Moreover, acorns are a good source of active compounds, such as phenolic acids, and flavonoids, with an interesting

antioxidant activity [1]. Acorns also contain a high content of tannic acid, a mild toxin giving them a bitter taste that can be removed by many methods such as soaking in water, boiling, or roasting [3]. Traditionally, acorns were used in the human diet, generally as flour for bread production, or as a coffee substitute beverage (after a roasting process) [4]. Recently, acorn fruit flour was included in many other food preparations such as pasta, biscuits, hot beverages, cakes, and cookies [5]. Among all the nutrients present in acorn fruit, starch was the predominant component with content ranging from 31 to 55% [1]. Thus, further interest must be given to acorn starch extraction and valorization. Starch is a biodegradable carbohydrate polymer which has been widely studied due to its availability, price, and extensive industrial use. Recent research has shown that acorn starch can be used as an ingredient for food and nonfood applications [6, 7]. Acorn starch was used in many industrial applications like in the cases of paper, plastics, textile, pharmaceutical, and cosmetic industries [8]. Also, starch is a raw material representing the principal component of many food formulations being responsible for important functional and textural properties and nutritional characteristics of the many food products [9]. Owing to its interesting properties such as high resistance and paste consistency, acorn starch can be used as thickening and stabilizing agents in food formulations [2, 7]. The chemical composition and physicochemical features of starch are mainly characteristic of their biological origin. Starch from all plant sources has many similar properties but they do also differ in many aspects. Variation in structure, crystallinity, chemical composition and functional properties of starch granules are depending on their botanical origin and growth conditions. For the selection of the specific use of acorn starch, it is necessary to understand the physicochemical and functional properties of extracted starches from various acorn species. From all the above, collected data related to the chemical and technological properties of acorn starches extracted from *Q. ilex* L. and *Q. suber* L. species as well as characteristics of other starches obtained from various botanical sources such as corn, potato and cassava are discussed to highlight the importance to valorize acorn starches and their potential applications. Thus, it will be interesting to valorize acorn known as a Tunisian under valued resource by providing promising new ingredients to formulate new food products.

2. Starch extraction methods

Generally, fresh acorn fruits were manually collected from the North West of Tunisia. *Q. ilex* L. is abundant in Bizerte (north east of Tunisia) region, while, *Q. suber* L. is provided from the mountainous region of Ain Drahem from Jendouba in the north west of Tunisia. Due to their short shelf life, acorn fruits are, first, hand-peeled, dried in mild conditions and then milled into fine flour.

The extraction technologies of acorn starch consist of dry and wet methods. The use of dry methods is shown unsuccessful for the elimination of protein, fat, and tannins from acorn flour, which need to use some other absorbents.

The acorn flour is used for starch extraction using different methods as alkaline washing, hot-water soaking, ultrasonic-assisted ethanol soaking and ultrasonic-assisted hot-water soaking [10]. The three later methods lead to starch granules with similar internal structure. However, starch granules isolated using hot-water methods are complete and glossy with a few numbers of pits. It's important to know that the ultrasonic technology became the most effective in food applications compared with conventional technologies.

3. Starch extraction yield

Numerous studies have already been conducted on the starch yield of acorn species originating from countries all over the world. It is stated in previous studies that starch is the main component of acorns and usually constitute more than 50% of the kernel [11]. The yield of starch extracted from acorn species cultivated in Tunisia and other countries of the world as reported in different studies is presented in **Table 1**. The starch yield varied from 17.3 to 89.83% in acorn species. The starch content in *Q. ilex* L. and *Q. suber* L. were reported to vary from 48.93 to 89.83% [14] and 86.9 to 88.5% [12], respectively. The values indicated that acorn fruits are promising crops as an alternative source of starch. In general, the authors concluded that starch content in *Q. suber*. L is higher than that in *Q. ilex* L. reporting a starch yield of about 34.5% [7]. As it can be also seen from **Table 1** that *Quercus palustris Muenchh* [15] contain the lowest amount of starch (17.3%) as compared to other acorn species. Correia et al. [13] and Masmoudi et al. [17] reported values of 88.5% and 63% of starch content in the Portuguese and Tunisian *Q. suber* L. fruits.

Irinislimane and Belhaneche Ben semr [18] and Correia et al. [12] isolated starch from *Quercus Suber* L. acorns and observed a starch yield accounting 21% and 31.4%, respectively. The starch content in potatoes, tubers and roots are reported to vary from 75 to 80% and 75.4 to 77.4% [5], respectively.

This variability in the starch yield was due to the difference in plant species, cultivation climate, ripening stage, harvesting time of fruits, and extraction method used [7].

The obvious retained conclusion is that the high content of starch makes the *Quercus* species particularly *Q. suber* L. ideal for starch extraction and valorization in many food and nonfood industries applications. Besides, the content of starch in acorn flour gave it good functional characteristics related to starch such as viscosity, swelling, and gelling [17]. In fact, it is suggested that acorn starch might be used as thickening and stabilizing agent, owing to its high paste consistency [2]. Since this polysaccharide is present as resistant starch in a high percentage, it can be very useful as a prebiotic growth promoter, constituting a good alternative to other current prebiotic agents such as fructo-oligosaccharides, inulin, isomalto-oligosaccharides, polydextrose, and lactulose [19].

Several studies have examined the effect of different methods using both physical and chemical methods on acorn starch yield. Differences in starch content are

Species	Starch yield (%)	Extraction methods	References
<i>Quercus suber</i>	86.9	Enzymatic treatment	[12]
	88.5	Alkaline method	[12]
	49	Alkaline method	[13]
	45–57	Alkaline method	[12]
<i>Quercus ilex</i>	34.5	Alkaline method	[7]
	48.93–89.83	Alkaline method	[14]
<i>Quercus palustris Muenchh</i>	17.3	Water method	[15]
<i>Quercus leucotrichophora</i>	54.7	Alkaline method	[16]
<i>Quercus rotundifolia</i>	48	Alkaline method	[13]

Table 1.
Extraction methods and starch yield of various acorn species.

observed using alcohols-based extraction, alkaline-based extraction, acetone-based extraction, hot-water soaking, ultrasonic-assisted ethanol soaking and ultrasonic-assisted hot-water soaking [10].

4. Physico-chemical composition

The physico-chemical composition of acorn starch extracted from *Quercus* species is affected by extraction and purification methods and the origin of raw materials [7]. The extracted acorn starch contained water and minor components such as lipids, proteins and ash (Table 2). The moisture content of acorn starch species varies from 7.22 to 15.91%. The moisture content of *Q. ilex* L. extracted starch from four different areas in Algeria varied from 2.2% to 15.9% [14]. The moisture content is very important parameters for the determination of the starch quality. A low moisture content of the acorn starch less than 20% is acceptable for commercial starch and a value less than 13% is recommended for safe storage [9]. Such values are close to those reported in cereal (10–12%) and some roots and tubers (14–18%) starches [20].

Several studies show low lipids, proteins, and ash contents in starches extracted from different acorn species.

Lipids have an essential role in the properties of starch, which is associated with the textural properties of various foods. The lipid content in all starches extracted from *Quercus* species is below 1%. Tunisian *Q. ilex* L. starch contains a high amount of lipid content (0.51%) than both potato and wheat starches [21].

The ash content of the extracted starch from *Q. ilex* grown in Tunisia (20.66%) is relatively high. The review of Taib and Bouyazza, [5], reported ash values ranged from 0.01 to 1.41% in different *Quercus* species. The low ash content illustrates the purity of starch after the extraction and isolation processes. The protein contents of starches obtained from different *Quercus* species ranged from 0.25 to 1.05%. These low values show a high extracted starch purity and quality [7]. The pH values in *Q. suber* was about 5.6 units, while it ranged between 4.73 and 6.43 units in *Q. ilex* starches. Such value is lower than that (6.22 units) reported for the potato starch [22]. High pH value could lead to undesirable protein modification as well as molecular

Components	<i>Quercus suber</i> . L		<i>Quercus ilex</i> . L	
	[7–12, 14]	[12]	[7]	[14]
Moisture (%)	12.95	7.22	10.17	2.2–15.9
Fat (%)	0.50–0.60	Nd	0.51	0.23–0.63
Protein (%)	0.25–0.3	Nd	0.92	0.91–1.05
Ash (%)	0.02–0.24	0.13	2.66	0.14–0.18
pH	5.60	Nd	5.3	4.73–6.43
L*	61.13	Nd	85.03	Nd
a*	0.84	Nd	0.52	Nd
b*	15.07	Nd	10.2	Nd

Nd: not determined.
L*, a* and b* are the color parameters.

Table 2.
Physico-chemical composition of acorn species starches.

cross linkage and rearrangements resulting in the formation of toxic compound [23]. A positive correlation was obtained between the pH value, the fat and the protein contents [24]. The variation observed in the chemical composition of starches is assigned to the extraction and purification methods, environmental conditions (climate and soil composition), growth stage of plant and genotype. During different seasons of the year (summer and autumn), protein and fat contents vary in four collected acorn species (*Q. suber*, *Q. ilex*, *Q. faginea* and *Q. pyrenaica*) [25]. From these findings, we conclude that the chemical content of starches is influenced by the botanical source and the extraction methods used.

Concerning the color parameters, the extracted starches from Tunisian *Quercus* species exhibited a slightly yellow-white color. Indeed, extracted *Q. ilex* L. starch showed a high lightness L^* value (85.03) [7] compared to the *Q. suber* L. (61.13). While, the obtained values of a^* (0.52) and b^* (10.2) were lower than those found in *Q. suber* L (0.84 and 15.07, respectively) [7]. These findings showed that acorn fruit is a good source of starch that can be used in food industry without the necessity of chemical or genetic modifications. This polysaccharide may be industrially applied as emulsifiers, stabilizers, and thickeners in food as well as prebiotic growth promoter [26].

5. Swelling power, solubility and water absorption

When starch is heated in excessive amount of water, its crystalline structure is disrupted, and water molecules become linked by hydrogen bonding to the exposed hydroxyl groups of amylose and amylopectin [5]. These phenomena results in the swelling, solubility and increasing volume of starch granules. The swelling power, solubility, and water absorption values of extracted acorn starches from *Q. ilex* and *Q. suber* species are presented in **Table 3**. These parameters increased progressively with the increase of temperature from 60 to 90°C. The solubility of the extracted *Q. ilex* starch is higher than that of *Q. suber*, and ranged from 0.2 to 12.95% at 60°C and 4–64.22% at 90°C.

However, the swelling power and the water absorption values are lower in *Q. ilex* starch compared to the *Q. suber* starch. Diversity in swelling, solubility and water absorption of acorn starches has been observed. Boukhelkhal and MoulaiMostefa. [14] reported low solubility and swelling power of four species of acorn starches at temperature of 90°C ranging from 4 to 14% and 11 to 13 g/g, respectively. Values related to the swelling power are low compared to those found by Singh et al. [27] and Elmi Sharlina

Temperature (°C)	<i>Quercus ilex</i> . L starch [7–11, 14]				<i>Quercus suber</i> . L starch			
	60	70	80	90	60	70	80	90
Solubility (%)	0.2–12.95	1.8–16.35	2.4–29.28	4–64.22	3.3	4.4	7.5	20.2
Swelling power (g water/g starch)	3.9–8.95	8.4–10.53	10–13.3	11–20.76	6	12.5	13.03	21.51
Water absorption (g water/g starch)	3.68	5.7	9.3	10.1	4.5	8.5	11.6	15

Table 3.
Swelling power solubility and water absorption of acorn species starches.

et al. [22] on sweet potato starch (35 to 40 g/g) and chestnut starches (13.6–17.3 g/g), respectively. These low values are attributed to the amylose content of the acorn starch species [28], starch molecule’s ability to hold water, hydrogen bonding, and the degree of crystallinity [29]. According to Jiang et al. [30], the solubility values of starches extracted from five different *Dioscorea* L. species, which are *D. opposite Thunb*, *D. alata Linn*, *D.nipponica Makino*, *D. bulbifera Linn* and *D.septemloba Thunb*, varied from 11.14 to 30.04% at the temperature of 95°C. A comparative study showed that swelling power and solubility of acorn starch at 90°C were higher than those of black wheat, buckwheat, coix and naked oat starches and lower than those of corn, jiaoyu, kuzhu, and longya lily starches [31].

The solubility suggests that additional interactions may have occurred between amylose-amylose and amylopectin-amylopectin chains [32]. Concerning, the water absorption capacity of starch, it corresponds to the hydrogen bonding between water molecules and hydroxyl groups in the starch molecules and starch chains as well as diversification of the starch granule structures [33]. In general, the starch extraction methods have important effect on swelling power, water absorption and solubility parameters of starch. Zhang et al., [10] reported a relatively higher value of swelling power (24.99 g/100 g) and solubility (15.22%), at temperature of 90°C, in acorn starch extracted by an ultrasonic-assisted ethanol soaking method. Variation of these parameters in extracted starches is associated to various factors such as: amylose content, granule size, structure of starch granules, viscosity patterns, and presence of non-starch compounds (lipids, ash and proteins) [5].

6. Refrigeration and freezing stability

In order to evaluate the stability of starch during storage, it was necessary to verify the expulsion of water, expressed by syneresis, contained in gels as a consequence of the reorganization of starch molecules [34]. Collected syneresis values during refrigeration and freezing time are grouped in **Table 4**. Results showed that *Q. suber* starch lost less water than the *Q. ilex* starch under refrigeration (4°C) with the increase of the storage time. However, the latter presents higher syneresis values already from the first freeze time, showing low stability to freezing under the conditions used in the studies and a richness in amylose content [9]. It is known from the literature, that starches with high amylose content such as potato (20.1–31.0%), maize (22.4–32.5%), taro (28.7–29.9%), and cassava (18.6–23.6%) present high syneresis, due to the large amount of water expelled during the retrograding process [25]. It known that during freezing of the starch paste, the cohesive portion of the starch formed a layer and the rest separated into a water layer.

Time (h)	Syneresis to refrigeration 4°C (%)		Syneresis to freezing –20°C (%)	
	<i>Quercus ilex</i> .L starch [7]	<i>Quercus suber</i> .L starch	<i>Q. ilex</i> .L starch [7]	<i>Q. suber</i> .L starch
24	42.81	42	26	46.1
48	49.34	43.44	36.18	47.5
72	53.3	45.1	38.47	55.1
96	55.57	46	41.43	60.2

Table 4.
Refrigeration and freezing stability of acorn species starches.

7. X-ray diffraction analysis

Starch is a semi-crystalline material affected by amylose content and amylopectin chain length that consists of amorphous and crystalline regions. The amylose content directly affects the crystallinity degree of the starch, such that when there is a lack of amylose content, the crystallinity degree increases, whereas the longer chain amylopectin forms have a more stable crystalline structure [31].

Generally, starch is present in three different crystalline structures which are A-type, B-type, and C-type that depended essentially on the variety of starch source.

The difference between A- and B-types of starch granules is due to the arrangement of double helices. A-type starches form dense packing with four water molecules, whereas B-type starch is more open causing more water molecules (36 water molecules) to be located in the center of a hexagonal packing of helices. For this reason, it is indicated that the A-type is more stable and requires a higher temperature than B-type starch for gelatinization [31].

X-ray diffraction analysis was employed to observe the changes in the degree of crystallinity of starch as a result of gelatinization. **Figure 1** resume the X-ray diffraction patterns observed on acorn starches extracted from *Q. suber* and *Q. ilex*. The X-ray diffraction patterns provide a classification of the two acorn starches under an A-type crystalline structure, which characterized most cereal starches [35] showing two strong reflections at 15.2° and 22.7° . The X-ray diffraction patterns of both acorn starches showed four intense diffraction peaks at 15.2° , 17.2° , 19.52° , and 22.7° of 2θ . The strong reflections at 15.2° and 22.7° of 2θ were classified as the A-type crystalline structure, which characterized most cereal starches [35]. The observed peaks at approximately 17.2° and 19.52° of 2θ were characterized as the B-type pattern. However, the C-type crystalline structure consisted of A- and B-type crystallites. Thus, the X-ray diffraction pattern can contain various superpositions of the characteristic diffraction peaks depending on the ratio between the contents of these polymorphs [35]. In general, cereal starches have an A-type pattern, whereas tuber starches display the B-type pattern, and certain roots and legumes starches show a C-type pattern [36]. Such results were close to those found on starch from *Quercus glandulifera* Bl. [37] and *Dioscorea pyriformis* tubers [2]. Numerous studies have already

Figure 1.
X-ray diffraction pattern of different starches. A: *Quercus ilex*. L starch, B: *Quercus suber*. L starch.

been conducted on the X-ray diffraction of starches extracted from acorn species originating from countries all over the world. Deng et al. [26] reported that acorn starch from china was B-type. A-type polymorph was reported for acorn starch [38]. However, Zhang et al. [10] and Molavi et al. [39] noted also a C-type polymorph in acorn starches. The difference in the diffraction pattern of starch granules was mainly influenced by genotypic, agronomic, and growing conditions such as temperature [7]. According to Dereje, [40] the type of crystallinity of the extracted starch was influenced by growth temperature, alcohols, fatty acids, and the chain length of amylopectin.

8. Conclusion

Despite that acorns are underutilized fruits, they represent a good alternative source of starch. The acorn starch yield differs from one specie to another representing about 50%. It can be extracted using various methods. The acorn starch was characterized by a yellow color and good technological properties allowing its use during manufacturing of food products. Thus, acorn starch can represent an interesting functional ingredient capable to improve the properties of the final product.

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Conflict of interest

The authors declare no conflict of interest.

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