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Composition, molecular structure, and physicochemical properties of tuber and root starches: a review

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Abstract

The major carbohydrate of tuber and root crops is starch, which accounts for 16–24% of their total weight. In recent years, substantial progress has been made in understanding the relationship between starch structure and physicochemical properties. However, these studies have been mainly on cereal starches. The present status of knowledge on the composition, structure, gelatinization retrogradation, digestibility and rheological properties of tuber and root starches is reviewed. In addition, present concepts of granule structure, gelatinization, retrogradation and rheology are also reviewed. Future research needs in the area of tuber and root starches are discussed. © 2001 Elsevier Science Ltd. All rights reserved.

Keywords: Starch structure; Tuber and root starches; Rheological measurements

1. Introduction

Root and tuber crops are grown throughout the world in hot and humid regions. They are plants yielding starchy roots, rhizomes, corns, stems and tubers. Root and tuber crops contain 70–80% water, 16–24% starch and trace quantities (<4%) of proteins and lipids.

Some of the root and tubers that are grown for edible purposes are: potato (*Solanum tuberosum*), sweet potato (*Ipomea batatas*), cassava (*Manihot esculenta*) true yams [(*Dioscorea*) species (*D. alata*, *D. cayenensis*, *D. spicata*, *D. bulbifera*, *D. esulenta*, *D. abyssinia*)] arrowroot [West Indian arrowroot (*Maranta arundinacea*), Indian arrowroot (*Hutchenia caulina*), East Indian arrowroot (*Tacca leonto petaloides*), Queensland arrowroot (*Canna edulis*)], buffalo gourd (*Cucurbita foetidissima*), Kuzu (*Pueraria hirsuta*), ginger (*Zingiber officinale*), lotus (*Nelumbo nucifera*) and the edible aroid root crops belonging to the family araceae which include five genera (*Colocassia*, *Xanthosoma*, *Amorphallus*, *Alocassia* and *Cytosperma*). *Alocassia*, *Xanthosoma* and *Colocassia* are in the tribe Colocasiae and in the subfamily colocasioideae (O'Hair & Asokan, 1986). It is the division of edible aroid genera into species that has caused confusion. *Colocassia* is included in the subtribe colocasinae. *Colocassia* species can be classified as follows: (1) *C. esculenta* (L) Schott var *escluenta* (produces a large corn and is also called taro, dasheen, coco, tannia); and (2) *C. esculenta* (L) Schott var *antiquorum* (produces a small central corn surrounded by numerous side cornels and is also known as eddoe). Both 1 and 2 are referred to collectively as "*old*" *cocoyams*. *Xanthosoma* is included in the subtribe caladinae. *Xanthosoma sagittifolium* (L) Schott is generally considered as the main cultivated species. Other related species include *X. brasillense*, *X. atravirens*, *X. violaceum*, *X. robustrum*, *X. auriculatum*, *X. roseum* and *X. varacu*. The *Xanthiosoma* species are collectively known as "*new*" *coco yams*.

Alocassia is included in the subtribe alocasiianae which include the species *A. macrorrhiza* (*giant taro*), *A. indica* and *A. fornicata*. *Amorophallus* is included in the tribe Phythoniae and includes the species *A. campanulatus* (elephant foot yam) and *A. rivieri*. *Cytosperma* is included in the tribe Lasieae and includes the species *C. eduli*, *C. merkusu* and *C. lasioides*.

The agronomic and phenotype properties of tropical crops are well documented. However, the structure and physicochemical properties of many tuber and root starches have not been studied extensively. Thus, intensive research and product development is needed to exploit tuber and root starches.

This review summarizes the present knowledge on the composition, structure, physicochemical properties of native tuber and root starches, with a view to providing suggestions for needed research to improve the utilization of these starches in the food industry.

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Table 1
Proximate composition of tuber and root starches

Starch source	Starch yield %	Size (μm) and shape	Amylose content (%)	Total lipid (%)	Phosphorus (% dsb)		Nitrogen (%)
					Org	Inorg	
Solanum tuberosum (potato)	32[a]	15–110[b] oval, spherical[c]	25.4[d]	0.19[e]	0.089[f]	0.001[f]	0.1[a]
Solanum tuberosum (waxy potato)	–	14–44[g] round, oval[g]	–	–	0.069[g]	0.001[g]	–
Ipomea babatas (sweet potato)	30[h]	2–42[i] round, oval and polygonal[j]	19.1[k]	0.06–0.6[j]	0.012[f]	–	0.006[l]
Ipomea trifida (sweet potato)							
Diploid hybrid	–	8.5[m], round-angular	28[m]	–	–	–	–
Tetraploid hybrid	–	14.6[m], round-angular	26[m]	–	–	–	–
Dioscorea abyssinica (true yam)	–	29.2[n], round	29.7[n]	0.05[n]	–	–	0.08[n]
Dioscorea alata (true yam)	84.6[o]	6–100[b], round-oval	22.8[o]-30[p]	0.03[o]	–	–	0.33[o]
Dioscorea cayenesis (true yam)	87.5[o]	28.5–30.6[o], round-oval	21.6[o]–27[p]	0.02[o]	–	–	0.33[o]
Dioscorea dumetorum (true yam)	88.0[o]	28.5–30.6[o], round, oval[o]	10.0[o]–24.6[p]	0.04[o]	–	–	0.29[o]
Dioscorea rotundata (true yam)	86.1[o]	10–70[b], round-oval[o]	22.4[o]	0.04[o]	–	–	0.37[o]
Dioscorea esculenta (true yam)	–	1–5[r], round-oval[r]	30[p]	–	–	–	0.013[q]
Rhizoma dioscorea (kind of yam)	–	19.8–28.4[s]	35[r]	–	–	–	–
Amorphophallus paeonfolius (elephant yam)	35[s]	3–30[b], round-polygonal[b]	–	–	–	–	–
Colocassia esculenta (old coco yam, taro)	55.1[f]	3.0–3.5[f], round-polygoanl[f]	21.4[f]	0.39[f]	0.021[f]	–	0.019[f]
Xanthosoma sagitifolium (new coco yam)	43.8[t]	10–50[b]	23.7[t]	–	–	–	–
Manihot esculenta (cassava tapioca manioc, brazilian arrowroot)	–	5.40[b], round[b]	18.6–23.6[u]	0.1[c]	0.008[f]	0.001[f]	0.009–0.0131[u]
Maranta arundaceae (west indian arrowroot)	–	10–16[v], round-oval-polygonal[v]	19.4[v]	0.32[v]	–	–	0.03[v]
Canna edulis (queensland arrowroot)	60.3[w]	13.0–57.6[w], oval-elliptical[w]	38[w]	0.30[w]	–	–	0.01[w]
Pueraria tuberosa (kuzu)	34.2[x]	3–23[x,y], polygonal[x]	15.1[x], 21.0[y]	0.46[x]	0.005[x]	–	–
Cucurbita foetidisima (buffalo gourd)	–	2–24[z], oval-elliptical[z]	23.2[z]	0.92–1.14[z]	0.01–0.06[z]	–	0.28–0.49[z]
Nelumbo nucifera (Lotus)	–	15–40[aa], rod like-round[aa]	15.9[aa]	–	48 (ppm)[aa]		–
Lilium maximoroiczii (Lily)	–	30[ab], oval-polygonal-elliptical[ab,ac]	26.8[ab]	–	60 (ppm)[ab]	33 (ppm)[ab]	

[a] Hoover and Hadizeyv (1981). [b] Moorthy (1994). [c] Sivak and Preiss (1998). [d] Kim, Weisenborn, Orr and Grant (1995). [e] Vasanthan and Hoover (1992). [f] Lim et al (1994). [g] McPherson and Jane (1999). [h] Zhang and Oates (1999). [i] Seog et al (1987). [j] Tian, Rickard and Blanshard (1991). [k] Collado, Mabesa and Corke (1999). [l] Garcia and Walter (1998). [m] Shiotani, Nishimura and Yamanaka, (1991b). [n] Mariam and Schmidt (1998). [o] Emiola and Delarossa (1981). [p] Gallant et al. (1982). [q] Rasper and Coursey (1967). [r] Yu, Fuji and Kishihara (1999). [s] Rani, John, Moorthy and Raja (1998). [t] Lauzon et al. (1995). [u] Defloor, Dehing and Delcour (1998). [v] Erdman (1986). [w] Soni, Sharma, Srivastiva and Gharia (1990). [x] Soni and Agarwal (1983). [y] Suzuki et al (1981). [z] Dreher and Berry (1983) [aa] Suzuki et al. (1992). [ab] Takeda, Takeda and Hizukuri (1983). [ac] Jane, Kasemsuwan, Leas, Zobel and Robyt (1994).

Table 2
X-ray pattern and crystallinity of tuber and root starches

Starch	X-ray pattern	Crystallinity (%)
Solanum tuberosum	B[a]	28[a]
Ipomea batatas	A[b,c], C[a], C_a[d]	38[a]
Ipomea trifida		
Diploid hybrid	C_a[e]	–
Tetraploid hybrid	C_b[e]	–
Dioscorea abyssinica	B[f]	–
Dioscorea alata	B[b]	–
Dioscorea cayensis	B[b]	–
Dioscorea dumetorum	A[c]	–
Doscorea rotundata	B[c]	–
Dioscorea escluenta	B[c]	–
Rhizoma dioscorea	C_b[g]	–
Amorphophallus paenofolius	A[b,h]	–
Colocassia esculenta	A[i]	45[a]
Xanthosoma sagitifolium	A[b,d]	24[k]
Manihot exculenta	C_a[c], A[b], C[a]	38[a]
Cana edulis	B[j]	26[j]
Pueraria tuberosa	C_a[k]	–
Cucurbita foetidisima	B[l]	–
Nelumbo nucifera	C_c[m]C_b[n]	–
Lilium maximoroiczii	B[k]	–

[a] Zobel (1988a).
[b] Moorthy (1994).
[c] Gallant et al. (1982).
[d] Lauzon et al. (1995).
[e] Shiotani, Nishimura, Yamanaka, Taki and Yamada (1991b).
[f] Mariam and Schmidt (1998).
[g] Yu et al. (1999).
[h] Rani et al. (1998).
[i] Lim et al. (1994).
[j] Rickard et al. (1991).
[k] Takeda et al. (1983).
[l] Dreher and Berry (1983).
[m] Suzuki et al. (1992).
[n] Soni and Agarwal (1983).

2. Granule morphology

The size and shape of tuber and root starch granules are shown in Table 1. The granule size is variable and ranges from 1 to 110 μm depending on the starch source. Most of the granules are oval, although, round, spherical, polygonal and irregularly shaped granules are also found. When observed under a scanning electron microscope the surfaces of all granules appear smooth with no evidence of any fissures. Most of the tuber and root starches are simple granules, the exception being cassava and taro starches, which appear to be a mixture of simple and compound granules. Baldwin (1995) showed using atomic force microscopy, the presence of large protruberences (200–500 nm) on the surface of potato starch granules.

3. Proximate analysis and chemical composition

The proximate analysis and chemical composition of tuber and root starches are illustrated in Table 1. The isolated starches had nitrogen contents ranging from 0.006 to 0.49%. The starches were generally characterized by a low lipid content (<1%). The amylose content of the starches ranged from 10–38%. In many instances, the amylose content of these starches have been determined by colorimetric procedures without prior defatting and/or by not taking into account the iodine complexing ability of the long external amylopectin chains of tuber starches (Banks & Greenwood, 1975; Morrison & Karkalas, 1990; Takeda, Takunaga, Takeda, & Hizukuri, 1986). Thus, leading either to an underestimation (failure to remove amylose complexed lipids) or to an overestimation (failure to determine amylose content from a standard curve containing mixtures of amylose and amylopectin in various ratios) of the amylose content.

Root and tuber starches contain significant amounts of mono phosphate esters covalently bound to starch (Lim, Kasemsuwan, & Jane, 1994; Kasemsuwan & Jane, 1995). Starch phosphate-monoesters in native potato starch is mainly found on amylopectin (Jane, Kasemsuwan, & Chen, 1996). The distribution of the phosphate monoester content on the C_2, C_3 and C_6 of the glucose unit of potato starch as been reported to be 1, 38, and 61%, respectively. (Hizukuri, Tabata, & Nikuni, 1970; Tabata & Hizukuri, 1971). Takeda and Hizukuri (1982) have shown that potato amylopectin contains one phosphate-monoester group per 317 glucosyl residues. The above authors showed by isoamylase debranching and β amylase treatment, that phosphate groups are present in the long branch chains (β chains with average degree of polymerization ~41). The phosphate group in potato starch as been reported to be located more than 9 glucosyl residues away from the branch point (Takeda & Hizukuri, 1981, 1982). Jane and Shen (1993) showed that phosphorous in potato starch is located densley in the granule core together with amylopectin. Among the starches, potato and waxy potato contain the largest quantity of organic phosphate followed by taro (Table 1). The high phosphate monoester content of potato starch confers enhanced paste clarity, high peak consistency, significant shear thinning and slow rate and extent of retrogradation (Jane et al., 1996; Galliard & Bowler, 1987). The phosphorous content and form as been reported to be influenced by growing condition, temperature and storage (Hizukuri et al., 1970; Muhrbeck & Tellier, 1991; Nielsen, Wischman, Eneroldsen, & Moller, 1994).

4. Crystallinity of tuber and root starches

X-ray diffractometry has been used to reveal the presence and characteristics of crystalline structure of starch granules. (Zobel, 1988a; Hizukuri, Kaneko, & Takeda, 1983). Most of the tuber and root starches exhibit (Table 2) the typical "B" type X-ray pattern (Zobel, 1988a) with peaks that are both broad and weak and with two main reflections

centered at 5.5 and 17°2θ angles. The exception being *Ipomea batatas* (A, C) *Manhiot esculenta* (C_a, A, C), *Nelumbo nucifera* (C_c C_b), *Dioscorea dumetorum* (A), and *Rhizoma dioscorea* (C_b) (Table 2). The "A" (shown mainly by cereal starches) and "B" type patterns represent true crystalline forms of starch, but the "C" type pattern is believed to be a superposition of the A and B patterns (Buléon, 1998). The C_a, C_b and C_c classification is based on the extent of their resemblance to "A" and "B" types or between the two types, respectively (Hizukuri, 1960). Imberty, Chanzy, Perez, Buleon and Tran (1988) have proposed that, the double helices in both A and B types are identical, but the mode of packing of the helices and the water content are different in the two polymorphs. The type of crystalline polymorph has been shown (Hizukuri et al. (1983) to be mainly influenced by the chain length (CL) of amylopectin [A type CL $<$ 19.7; B type CL $\geq$ 21.6, and starches exhibiting CL between 20.3 and 21.3 exhibit A, B or C-type patterns]. Other factors influencing polymorphism are growth temperature (Hizukuri, Fujii, & Nikuni, 1961), alcohols and fatty acids (Hizukuri, 1996).

The degree of crystallinity (Table 2) and the double helical content (in the amorphous and crystalline domains) of tuber and root starches have not been thoroughly investigated. Consequently, the influence of these parameters on enzyme susceptibility, gelatinization, retrogradation and rheological properties cannot be properly ascertained.

5. Structure of amylose and amylopectin

The two major components of starch are amylose and amylopectin. Amylose, the minor component, consists mainly of α-(1 → 4) linked D-glucopyranosyl residues. However, a slight degree of branching (9–20 branch [α(1 → 6] points per molecule) has been reported in amylose from various starch sources. The side chains range in chain length from 4 to over 100 (Hizukuri, Takeda, Yasuda, & Suzuki, 1981; Takeda, Hizukuri, Takeda, & Suzuki, 1987). The extent of branching has been shown to increase with the molecular size of amylose (Greenwood & Thomson, 1959). Evidence of the occurrence of branching points in amylose is its incomplete conversion into maltose by β amylase; β amylolysis has been shown to vary from 73 to 95% (Morrison & Karkalas, 1990). The molecular weight of amylose has been reported to vary between 10^5 and 10^6 Da (Morrison & Karkalas, 1990; Hizukuri, Takeda, Maruta, & Juliano, 1989). The formation of a helical complex between amylose and iodine gives rise to the typical deep blue color of starch dispersions stained with iodine and forms the basis for quantitative determination of amylose content. In I_2/KI solutions, polyiodide ions such as I_3^- and/or I_5^- (Teitelbaum, Ruby, & Marks, 1978; McGrance, Cornell, & Rix, 1998) interact with amylose forming single left handed V type helices. The helix consists of six anhydrous glucose residues per turn with a pitch of 0.8 nm and a hydrophobic helical cavity of diameter 0.5 nm. Amylose also forms a V-helix complex with the hydrocarbon portion of monoglycerides and fatty acids (Hoover & Hadziyev, 1981; Godet, Tran, Delage, & Buleon, 1993).

The physicochemical characteristics of tuber and root amyloses are presented in Table 3. The blue value, iodine affinity, β-limit, number of branch linkages, number degree of polymerization, weight average degree of polymerization, apparent degree of polymerization distribution, limiting viscosity and organic phosphorous are in the range 1.38–1.56, 18.3–20.4, 57–97.5, 2.2–12, 1540–8025, 3320–8040, 480–40 000, 172–426, 2–10, respectively. The corresponding values for cereal amyloses are 1.39–1.45, 19–20.9, 61–95, 0.9–5.5, 690–1690, 1810–5450, 180–25 200, 139–267 and 1–14, respectively (Hizukuri, 1996). Legume amyloses have not been well characterized and the reported values for iodine affinity, β-limit, number average degree of polymerization and limiting viscosity are 16–22, 79–86.9, 1000–1900 and 136–280, respectively (Hoover & Sosulski, 1991).

Amylopectin is the major component with a M_w (weight average molecular weight) of the order 10^7–10^9 (Aberle, Burchard, Vorwerg, & Radosta, 1994). It is composed of linear chains of (1- $>$ 4)-α-D-glucose residues connected through (1 → 6) -α-linkages (5–6%). The average size of the unit chains of amylopectin is 20–25 (Hizukuri, 1985). Hizukuri (1986) and Kobayashi, Schwartz and Lineback (1986) have shown that amylopectin molecule contains several distributions of chains (A, B and C) which differ in their chain length. The A-chains (unbranched) are linked to B chains and do not carry any other chains; the B chains (B1–B4), carry one or more A chains and/or B chains; and the C-chain, which has the reducing end group of the molecule. The chain length of A and B1 chains and that of B2–B4 are 14–18 and 45–55, respectively. The molar ratio of short to long chains is influenced by the starch source and varies between 3:1 and 2:1 (Hizukuri, 1985). Tuber starches contain fewer short chains and more long chains than do amylopectins from cereals. (Hizukuri, 1985, 1986). The branch points in the amylopectin molecules are not randomly distributed (Hizukuri et al., 1989); they are clustered and the interadjacent linear segments form thin crystalline lamellar domains having 5–7 nm width. The three dimensional structure of amylopectin is not yet known. Most of the currently accepted models favor a cluster type organization in which crystalline arrays of double stranded helices alternate with amorphous regions of dense branching chains.

The β-amylolysis limit of amylopectin (55–60%) is less than that of amylose (activity of β-amylase is sterically hindered by the branch points in amylopectin). Furthermore, iodine does not form a stable complex with amylopectin (due to short length of the unit chains). This results in the formation of a purple color with a λ_{max} at 530 nm. Calorimetry studies have provided indirect evidence for interaction between amylopectin and lipids (Eliasson & Ljunger,

Table 3
Properties of amyloses from tuber and root starches (for cereal amyloses, BV, IA, PO, β limit, BL, DP_n, DP_w, apparent DP distribution and η are in the range 1.39–1.45, 9–20.9, 1–14, 61–95, 0.9–5.5, 690–1690, 1810–5450, 180–25 200, 139–267, respectively (Hizukuri, 1996). For legume amylose, IA, β limit DP_n and η are in the range, 16–22, 79–86.9, 1000–1900 and 136–280, respectively (Hoover and Sosulski (1991)). NA — data not available)

Starch	Blue value (BV)	Iodine affinity (IA)	Organic phosphorous PO (PPM)	β-amylosis limit (β-limit — %)	Number of branch linkages (BL)	Number degree of polymerization (DPn)	Weight degree of polymerization (DPw)	Apparent degree of polymerization distribution (apparent DP distribution)	Limiting viscosity (η — ml/g)
Solanum tuberosum	1.38–1.41[a]	20.0[a]	–	80–87[a]	3.9–6.3[a,b,c]	2110–4920[a,b,c] 8025[d]	5130–6360[a,b,c]	560–21 800[a,b,c]	368–384[a,b,c]
Ipomea batatas	1.48[e]	19.9–20.2[e]	–	72–73[e]	9–12[e]	3025[d], 3400–4100[e]	–	–	324–344[e]
Manihot esculenta	1.47[e]	20.5[e]	–	75[e]	6–8[b,c,e]	2600[b,c,e] 3642[d]	6680[b,c,f]	580–22 400[b,c,f]	384[b,c,f]
Dioscorea alata	NA	19.9[f]	NA	92.3[f]	NA	NA	NA	NA	NA
Dioscorea cayensis	NA	19.9[f]	NA	94.7[f]	NA	NA	NA	NA	NA
Dioscorea dumedorum	NA	19.9[f]	NA	97.5[f]	NA	NA	NA	NA	NA
Dioscoea rotundata	NA	19.8[f]	NA	95.8[f]	NA	NA	NA	NA	NA
Nelumbo nucifera	1.56[g]	20.2[g]	0	90[g]	6–7[g]	4170[g]	8040[g]	520–40 000[g]	426[g]
Canna edulis	1.47[h]	20.2–20.[h,i]	5.0[h]	83[h]	2.2[h]	1380–1950[h,i]	5480[h]	550–14 400[h]	361[h]
Cucubita foetidissima	NA	18.3–19.2[j]	NA	71.6–88.4[j]	NA	1273–1954[j]	NA	NA	172–264[j]
Lilium maximoroiczii	1.49[k]	20[k]	2.0[k]	NA	NA	2300[k]	NA	NA	NA
Pueraria hirsuta	1.41[l]	19.5[l]	10.0[l]	57[l]	3.8[l]	1540[b,h]	3320[b,h]	480–12 300[b,h]	202[b,h]

[a] Suzuki et al. (1994).
[b] Hizukuri and Takagi (1984).
[c] Takeda et al. (1984).
[d] Ong et al., 1994.
[e] Takeda et al. (1986).
[f] Emiola and Delarossa (1981).
[g] Suzuki et al., 1992.
[h] Hizukuri (1996).
[i] Lu et al. (1987).
[j] Dreher and Berry (1983).
[k] Takeda et al., (1983).
[l] Suzuki et al. (1981).

1988). Morrison and Karkalas (1990) have shown that there are atypical types of amylopectin: (1) high molecular weight amylopectin with A and B chains 5–15 glucose residues longer than normal; (2) amorphous amylopectin with similarly extended A and B chains that elute from GPC columns with amylose; and (3) normal amylopectin which contain very long chains (CL 85–180) with infrequent branching. Banks and Greenwood (1975) have shown that type 1 and 2 amylopectins occur in several legume and tuber starches, and the third type has been reported in sweet potato starches (Takeda et al. 1986).

The physiochemical properties of amylopectins from tuber and root starches are presented in Table 4 and the distribution of the chain lengths of amylopectins from potato, kuzu and tapioca are presented in Table 4. The blue value, iodine affinity, organic phosphorous, β amylolysis and average chain length are in the range 0.104–0.245, 0.06–1.1, 21–900, 43.8–64.8 and 19–44, respectively. The corresponding values for cereal amylopectins are 0.049–0.441, 0.39–4.63, 10–119, 56–61 and 19–32 (Hizukuri, 1996). Legume amylopectins have not been well characterized and the reported values for iodine affinity, average chain length and β amylolysis are 1.00–5.3, 20–34 and 56–66.5, respectively (Hoover & Sosulski, 1991).

The amylopectin chain length distributions of some tuber and root starches are shown in Tables 5 and 6. These starches contain fewer A and B-1 chains and more B-2 and B-3 chains than cereal starches (Hizukuri, 1996).

6. Swelling power and solubility

When starch is heated in excess water, the crystalline structure is disrupted (due to breakage of hydrogen bonds) and water molecules become linked by hydrogen bonding to the exposed hydroxyl groups of amylose and amylopectin. This causes an increase in granule swelling and solubility. Swelling power and solubility provide evidence of the magnitude of interaction between starch chains within the amorphous and crystalline domains. The extent of this interaction is influenced by the amylose/amylopectin ratio, and by the characteristics of amylose and amylopectin in terms of molecular weight/distribution, degree and length of branching, and conformation. Amylose–lipid complexes have been shown to restrict swelling and solubilization (Swinkels, 1985). The higher swelling power and solubility of potato starch (Table 7) are probably due to a higher content of phosphate groups (Table 1) on amylopectin (repulsion between phosphate groups on adjacent chains will increase hydration by weakening the extent of bonding within the crystalline domain) (Galliard & Bowler, 1987). The swelling power and solubility of the other starches (at 95°C) are generally lower ranging from 14.6–51 and 7.8–26.7, respectively (Table 7).

7. Gelatinization

Starch, when heated in the presence of excess water, undergoes an order–disorder phase transition called gelatinization over a temperature range characteristic of the starch source. The above phase transition is associated with the diffusion of water into the granule, water uptake by the amorphous background region, hydration and radial swelling of the starch granules, loss of optical birefringence, uptake of heat, loss of crystalline order, uncoiling and dissociation of double helices (in the crystalline regions) and amylose leaching (Stevens & Elton, 1981; Lelievre & Mitchell, 1975; Donovan, 1979; Biliaderis, Maurice, & Vose, 1980; Hoover & Hadziyev, 1981; Evans & Haismann, 1982; Biliaderis, 1991; Jenkins, 1994). Jenkins (1994) showed by means of small angle neutron scattering studies, that the mechanisms proposed by Evans and Haismann (1982), Blanshard (1987) and Biliaderis, Page, Maurice and Juliano (1986) were not compatible with his results, but were in broad agreement with the gelatinization mechanism proposed by Donovan (1979). According to Jenkins (1994), gelatinization in excess water is primarily a swelling driven process. This swelling acts to destabilize the amylopectin crystallites within the crystalline lamellae, which are ripped apart (smaller crystallites are destroyed first). This process occurs rapidly for an individual crystallite, but over a wide range for the whole granule. The same mechanism occurs in conditions of limiting water. However, there is insufficient water for gelatinization to proceed to completion. At higher temperatures the remaining crystallites simply melt. Many methods are presently available for the determination of starch gelatinization, such as Kofler hot stage microscope (Watson, 1964), X-ray diffraction (Zobel, Young, & Rocca, 1988), DSC (Donovan, 1979), pulsed nucleur magnetic resonance (Lelievre & Mitchell, 1975), enzymatic digestibility (Shiotsuba, 1983), small angle X-ray scattering (Jenkins, 1994), small angle neutron scattering (Jenkins, 1994). However, only the Kofler hot stage microscope and DSC have been widely used to study the gelatinization temperatures of root and tuber starches (Table 8). Kofler hot stage microscopy is limited by the subjective nature of the observations (loss of birefringence) and only temperature measurements are obtained (Table 6). DSC measures the gelatinization transition temperatures [(onset [T_o], midpoint [T_p], conclusion [T_c] and the enthalpy (ΔH)] of gelatinization. Noda, Takahata, Sato, Ikoma & Mochida (1996) have postulated that DSC parameters (T_o, T_p, T_c, ΔH) are influenced by the molecular architecture of the crystalline region, which corresponds to the distribution of amylopectin short chains (DP 6–11) and not by the proportion of crystalline region which corresponds to the amylose to amylopectin ratio. The above authors have shown by studies on sweet potato and wheat starches, that a low T_o, T_p, T_c and ?H reflect the presence of abundant short amylopectin chains. Cooke and Gidley (1992) have showed (^{13}C.CP MAS-NMR and X-ray

Table 4
Properties of amylopectins from tuber and root starches (for cereal amylopectins, BV, IA, PO, β-limit and CL are in the range, 0.104–0, 245, 0.06–1.1, 43.8–64.8 and 19.44, respectively (Hizukuri, 1996). For legume amylopectins, IA, CL, and β amylolysis are in the range, 1.00–5.3, 20–34 and 56–66.5, respectively (Hoover and Sosulski, 1991). NA — data not available)

Starch	Blue value (BV)	Iodine affinity (IA)	Organic phosphorus (PO — PPM)	β-amylolysis limit (β-limit — %)	Average chain length (CL)
Solanum tuberosum	0.193–0.245[a,b]	0.06–0.08[a,b]	650–900[a,b]	56[a,b,c]	23[a], 34[c]
Ipomea batatas	0.1600–0.176[d]	0.38–0.44[d]	117–144[d]	55–56[d]	21[c], 29[d]
Manihot esculenta	0.104[e]	NA	NA	57[e]	21[e]
Dioscorea alata	NA	0.64[e]	NA	59.6[f]	22[f]
Dioscorea cayensis	NA	0.76[f]	NA	65.4[f]	21[f]
Dioscorea dumedorum	NA	0.72[f]	NA	66.7[f]	24[f]
Dioscorea rotundata	NA	0.67[f]	NA	60.5[f]	19[f]
Nelumbo nucifera	0.125[g]	0.22[g]	NA	55[g]	22[c], 30[g]
Colocassia esculenta	NA	NA	21[g]	NA	25[c]
Cucurbita foetidissima	NA	0.7–1.1[h]	NA	43.8–64.8[h]	23[h]
Canna edulis	NA	NA	NA	NA	44[c]
Lilium maximoroiczii	0.163[i]	0.37[i]	42[i]	57[i]	23.9[i], 34[c]
Pueraria hirsuta	0.135[j]	0.0[j]	121[j]	57[j]	20.5[j], 26[c]

[a] Suzuki, Shibanuma, Takeda, Abe and Hizukuri (1994).
[b] Hizukuri and Takagi (1984).
[c] Hizukuri (1985).
[d] Takeda et al. (1986).
[e] Suzuki, Takeda and Hizukuri (1985).
[f] Emiola and Delarossa (1981).
[g] Suzuki et al. (1992).
[h] Dreher and Berry (1983).
[i] Takeda et al. (1983).
[j] Suzuki, Hizukur and Takeda (1981).

diffraction) that the enthalpic transition is primarily due to the loss of double helical order rather than the loss of crystallinity. However, Tester and Morrison (1990) have postulated that ΔH reflects the overall crystallinity (quality and amount of starch crystallites) of amylopectin. Gernat, Radosta, Anger and Damaschun (1993) have stated that the amount of double-helical order in native starches should be strongly correlated to the amylopectin content, and granule crystallinity increases with amylopectin content. This suggests that ΔH values should preferably be calculated on an amylopectin basis. However, ΔH values for tuber and root starches (Table 8) have not been calculated in this manner.

Tester (1997) has postulated that the extent of crystalline perfection is reflected in the gelatinization temperature. The gelatinization and swelling properties are controlled in part by the molecular structure of amylopectin (unit chain length, extent of branching, molecular weight, and polydispersity), starch composition (amylose to amylopectin ratio, lipid complexed amylose chains, and phosphorous content), and granule architecture (crystalline to amorphous ratio) (Tester, 1997).

The molecular structure of many of the tuber and root starches listed in Table 8 have not been determined. Thus, it is not possible to discuss structure-gelatinization relationships in these starches. Furthermore, gelatinization parameters have been determined at different starch to water ratio's (Table 8) and at different heating rates. This makes it difficult to make a meaningful comparison of the gelatinization properties of these starches.

Table 5
Branched chain length distribution of potato, waxy potato, sweet potato and yam amylopectins (determined using a high performance anion exchange chromatograph equipped with a post column amyloglucosidase reactor and a pulsed amperometric detector (adapted from McPherson and Jane, 1999))

Starch source	1st peak	2nd peak	Distribution (%)					Maximum detectable DP
			DP 6–12	DP 13–24	DP 25–36	DP ≥37	CL	
Solanum tuberosum (potato with normal amylose)	14	51	13.07	44.39	14.00	28.54	28.6	85
Solanum tuberosum (waxy potato)	14	49	14.75	48.43	14.38	22.43	25.8	85
Yam (cultivar not specified)	13	52	19.09	44.81	14.32	21.80	25.8	85
Ipomea batatas	13	48	17.05	48.10	13.56	23.40	26.3	85

Table 6
Distribution of the chain length of cassava, kuzu and potato amylopectins[a]

Starch source	Whole	A[b]	B1[b]	B2[b]	B3[b]	B4[b]	A/B1–4
Solanum tubersum							
CL (max)		16	19	45	74		
CL_w	35	16	24	45	75	104	
Weight (%)	100	27.8	34.9	26	9.1	2.3	0.89
Mole (%)	100	44.2	38.1	14	3.1	0.8	
Manihot esculenta							
CL (max)		11	18	38	62		
CL_w	26	12	21	42	69	115	1.5
Weight (%)	100	38.5	32.5	23	5.1	0.9	
Mole (%)	100	59.6	28.7	10.2	1.4	0.1	
Pueraria tuberosa							
CL (max)		13	16	36	72		
CL_w	26	13	20	42	70	119	0.89
Weight (%)	100	30.7	42.7	20.2	5.4	1.0	
Mole (%)	100	47	41.9	9.4	1.5	0.2	

[a] Hizukuri, 1996.
[b] 80–90% of chain (A + B1) comprise a single cluster, ~10% of chains (B2 chains) are involved in the connection of 2 clusters, 1–3% of chains (B-3 chains) connect three clusters, and only 0.1–0.6% (B-4 chains) may connect more than four clusters (Hizukuri, 1996).

8. Rheology

The Brabender viscoamylogram and rotational viscometers are used to examine the rheological properties of starches. Compared with cereal starches, information on the rheological behavior of tuber and root starches under well defined flow regimes is limited. Our understanding of the rheology of tuber and root starches have come mainly from studies using the Brabender viscoamylogram; in which measurements are made under non-laminar flow conditions, and, in addition, the starch paste is subjected to both thermal and mechanical treatment, thus making it difficult to relate viscous behavior to only one of these parameters. There is thus a need to extend the use of rheometers (in which thermal treatments are separated from shear effects) to investigate the rheology of gelatinized tuber and root starch suspensions under well defined flow regimes and to compare these results with those obtained from the BVA.

Table 7
Swelling power and solubility of tuber and root starches

Starch source	Sweeling power (°C)	Solubility % (°C)
Solanum tuberosum	1159 (95)[a]	82 (95)[a]
Ipomea batatas	80 (90)[b]	68 (90)[b]
Dioscorea abyssinica	23 (85)[c]	11 (85)[c]
Dioscorea alata	20.5 (95)[d]	7.8 (95)[d]
Dioscorea cayenensis	16.9 (95)[d]	13.8 (95)[d]
Dioscorea dumetorum	18.6 (95)[d]	16.8 (95)[d]
Dioscorea rotundata	21.5 (95)[d]	11.9 (95)[d]
Xanthosoma sagitifolium	NA	189 (100)[e]
Manihot esculenta	51 (95)[f]	26 (95)[f]
Cana edulis	19 (95)[g]	17 (95)[g]
Pueraria tuberosa	23 (95)[h]	22 (95)[h]
Cucurbita foetidisima	14.6–26.5 (80)[i]	14–15.6 (85)[i]

[a] Leach, McCowen, and Schoch (1959).
[b] Seog, Park, Nam, Shin and Kim (1987).
[c] Mariam and Schmidt (1998).
[d] Emiola and Delarossa (1981).
[e] Lauzon et al. (1995).
[f] Tian et al. (1991).
[g] Rickard et al. (1991).
[h] Soni and Agarwal (1983).
[i] Dreher and Berry (1983).

The pasting characteristics of the tuber and root starches are presented in Table 9. The pasting curves of these starches have been determined at different concentrations, and furthermore, the pH at which measurements carried out have not been reported. Thus, it is difficult to make a meaningful comparison of the data presented in Table 9. However, it is evident, that starches belonging to the *Dioscorea* species (*alata*, *rotundata*, *dumetorum*, *esculenta*, *abyssinia*) exhibit a higher pasting temperature and thermal stability than the other starches. This suggests the presence of strong bonding forces within the granule interior. Legume starches also exhibit high pasting temperatures and high thermal stabilities. Furthermore, with the exception of certain *Dioscorea* starches (*alata*, *rotundata*, *dumetorum*, *esculenta*) all the other starches exhibit a peak viscosity (similar to cereal starches). The absence of a peak viscosity is characteristic of legume pasting curves (Hoover & Sosulski, 1991). Muhrbeck and Eliasson (1987) have shown by the use of rheometry, that due to the polyelectrolyte nature of potato amylopectin, the strength of potato starch (organic phosphorus 0.089% db) gels are influenced by pH and is a maximum at neutral pH. However, gel properties of cassava starch (organic phosphorus 0.008% db) are not influenced by pH. The above author's have also shown that the rheological properties of potato starch are very sensitive to ionic strength and the, gel strength (measured as G^*) is highest at low ionic strength. There were no specific interactions with any ions, but all ions showed the same effect when compared, at the same ionic strength (Muhrbeck & Eliasson, 1987). Yamada, Morimoto, Hisamatsu, & Komiya (1987) have shown that treatment of potato starch with 0.05 N HCl solution, followed by treatment with either 0.02 N KOH or saturated $Ca(OH)_2$ solution, reduced Brabender viscosity curves. The extent of this reduction followed the order. $Ca^{+2} > H^+ > K^+$. The higher reduction in viscosity observed with Ca^{+2} was attributed to a cross-linking effect between Ca^{+2} and phosphate groups on amylopectin. It is evident, that further studies are needed to clarify the mechanism by which ions influence starch viscosity. In the author's opinion, the influence of ions on starch viscosity is probably due to the interplay of two factors: (1) structuring of water molecules around the ions (this would reduce granular swelling, resulting in a lower viscosity); and (2) interactions of ions with the phosphate groups on amylopectin (the extent of this interaction is probably influenced by the size of the hydrated ion and by the phosphosphate monoester content).

Table 8
Gelatinization parameters of tuber and root starches (T_o, onset of gelatinzation; T_c, conclusion of gelatinization; T_c–T_o, gelatinization temperature range; starch (s): water (w); * — represent the mean of the samples analyzed; ** — polarizing microscope/kofler-hot stage)

Starch source and variety	Methodology	T_c–T_o (°C)	ΔH (J/g)
Solanum tuberosum[a] (42 genotypes)	DSC:W 1:2.3	71.7–63.5	17.3
Solanum tuberosum[b]	DSC:S:W 1:3	70.2–62.5	18.2
Ipomea batatas[c] (44 genotypes)	DSC:S:W 1:3	84.6–64.6*	12.9*
Ipomea batatas[d]			
(6 DLP selections)	DSC:S:W 1:6	83.5–60.0*	17.2*
(8 RCB selections)	DSC:S:W 1:6	81.4–61.3*	15.7*
Ipomea trifida[e] (6 diploid strains)	DSC:S:W 1:3	86.9–72.4*	7.4*
Colocassia esculenta[f] (5 varieties)	DSC:S:W 1:3	63.3–43.0*	6.8*
Dioscorea abyssinica[g]	DSC:S:W 1:2	74.8–64.2	19.2
Dioscorea alata[h]	Kofler hot stage**	71.5–65.0	–
Dioscorea cayenesis[h]	Kofler hot stage**	74.5–68.0	–
Dioscorea dumetorum[h]	Kofler hot stage**	72.5–65.5	–
Dioscorea rotundata[h]	Kofler hot stage**	71.0–63.5	–
Xanthosoma sagitifolium[i]	DSC:S:W 1:4	81.8–66.0	12.9
Xanthosoma sagitifolium[j]	DSC:S:W 1:2	87.0–74.0	4.0
Manhiot esculenta[k]	Kofler hot stage**	70.0–58.5	–
Manhiot esculenta[l]	DSC:S:W 1:2	84.1–62.4	4.8
Manhiot esculenta[m] (5 varieties)	DSC:S:W 1:2	76.1–57.0*	12.9*
Maranta arundinacea[n]	DSC:S:W 1:2	85.8–61.0*	19.2*
Canna edulis[o]	Kofler hot stage**	70–65	–
Cucurbita foetidissima[p](3 genotypes)	Kofler hot stage**	68.8–61.2*	–

[a] Kim et al. (1995).
[b] McPherson and Jane (1999).
[c] Collado et al. (1999).
[d] Garcia and Walter (1998).
[e] Asante, Yamada, Hisamatsu and Shiotani (1993).
[f] Jane et al. (1992).
[g] Mariam and Schmidt (1998).
[h] Emiola and Delarosa (1981).
[i] Valetudie, Colonna, Bouchet and Gallant (1995).
[j] Perez, Breene and Bhanassey (1998a).
[k] Srivastava, Harshe, Gharia and Mudia (1970).
[l] Perez, Breene and Bhanassey (1998b).
[m] Moorthy (1994).
[n] Erdman (1986).
[o] Soni et al. (1990).
[p] Dreher and Berry (1983).

9. Retrogradation

Starch granules when heated in excess water above their gelatinization temperature, undergo irreversible swelling resulting in amylose leaching into the solution. In the presence of high starch concentration this suspension will form an elastic gel on cooling. The molecular interactions (mainly hydrogen bonding between starch chains) that occur after cooling have been called retrogradation. These interactions are found to be time and temperature dependent. Starch gels are metastable and nonequilibrium systems and therefore undergo structural changes during storage (Ferrero et al., 1994). Miles, Morris, Orford and Ring (1985) and Ring et al. (1987) attributed the initial gel firmness during retrogradation to the formation of an amylose matrix gel and the subsequent slow increase in gel firmness to reversible crystallization of amylopectin. During retrogradation, amylose forms double-helical associations of 40–70 glucose units (Jane & Robyt, 1984; Leloup, Colonna, Ring, Roberts, & Wells, 1992), whereas amylopectin crystallization occurs by association of the outermost short branches (DP = 15) (Ring et al., 1987). The retrograded starch, which shows a B-type X-ray diffraction pattern (Zobel, 1988b) contains both crystalline and amorphous regions.

Retrogradation properties of tuber and root starches have been investigated by DSC, rheological measurements, FT-IR, Raman spectroscopy and X-ray diffraction. However,

Table 9
Pasting characteristics of tuber and root starches (values in parenthesis indicate starch concentration)

Starch source and concentration	Pasting temperature (°C)	Peak viscosity (BU)	Viscosity at 95°C (BU)	Viscosity the end of holding period (BU)	Viscosity at 50°C
Solanum tuberosum[a] (6%)	62	2150	1300	620	750
Ipomea batatas[b] (6%)	69.5–86.6	750–1130	720–1100	690–1130	NA
Ipomea batatas[c] (4%)	66.5–68.0	550–560	550–560	750–900	920–950
Ipomea trifida[d]					
Diploid hybrid[c] (4%)	72.5–74.0	NA	360	380–420	500–560
Tetraploid hybrid[d] (4%)	66.5–68.0	NA	460–530	480–550	720–920
Manhiot esculenta[e] (6%)	62	590	290	34	180
Dioscorea alata[f] (4.5%)	81–85	–	25–80	110–220	NA
Dioscorea alata[g] (5%)	74	No peak	100	140	160
Dioscorea alata[g] (10%)	69	No peak	282	360	380
Dioscorea rotundata[f] (4.5%)	76	No peak	450	630	NA
Dioscorea rotundata[g] (10%)	68	No peak	340	380	400
Dioscorea dumetorum[f] (4.5%)	82	No peak	25	25	NA
Dioscorea dumetorum[g] (10%)	68	No peak	230	280	295
Dioscorea esculenta[f] (4.5%)	82	No peak	25	55	NA
Dioscorea abyssinica[h] (6%)	72.9	781	756	731	1282
Rhizoma dioscorea[i] (8%)	NA	45	40	38	48
Colocassia esculenta[j] (8%)	70–75	700–1400	600–1100	460–700	600–1050
Nelumbo nucifera[k] (4.6%)	64	305	220	110	135
Canna edulis[l] (4.4%)	NA	300	NA	460	660
Maranta arundinacea[m] (5.0%)	72.7–75.9	337–410	NA	240–373	393–625
Xanthosoma sagitifolium[n] (6.0%)	72.3–76.3	230–300	220–290	200–280	700–810
Lilium maximoroiczii[o] (5.0%)	62.5	430	420	390	NA
Pueraria tuberosa[p] (5.0%)	70	245	NA	NA	NA

[a] Hoover and Vasanthan (1994).
[b] Seog et al. (1987).
[c] Shiotani et al. (1991a).
[d] Shiotani et al. (1991b).
[e] Creda and Wosiacki (1985).
[f] Rasper and Coursey (1967).
[g] Emiola and Delarossa (1981).
[h] Mariam and Schmidt (1998).
[i] Yu et al. (1999).
[j] Jane et al. (1992).
[k] Suzuki et al. (1992).
[l] Perez et al. (1998b).
[m] Erdman (1986).
[n] Lauzon et al.(1995).
[o] Takeda et al. (1983).
[p] Suzuki et al. (1981).

most of the available information are on potato and cassava starches.

10. Retrogradation monitored by DSC

There is limited information on the DSC parameters of retrograded tuber and root starches (Tables 10 and 11). Most DSC studies have been on potato starch. It is difficult to compare the data shown in Tables 10 and 11, due to differences in starch:water ratio's, different cultivars and differences in storage times. At all starch: water ratio's, ΔH_R (enthalpy of retrogradation) increases rapidly during the first two days of storage and thereafter the increase is only marginal (Table 10). Kim et al. (1997) attributed differences in ΔH_R values at different storage temperatures (4°, −10° and 23°C) (Table 10) to differences in the rates of nucleation and crystal growth (facilitated at low temperatures of storage). The above authors showed that the rate constants at 23°, 4° and −10°C for potato starch are 0.87, 1.97 and 2.11, respectively.

Silverio, Fredriksson, Anderson, Eliasson and Aman (2000) studied the effect of storage at 6°C for 48 h (to facilitate nucleation) followed by storage at either 30° or 40°C for 24 h (to facilitate propagation) on the retrogradation rate of potato starch gels (50% w/w) by DSC (Table 11).

Table 10
Retrogradation enthalpies of stored potato starch gels

Source	Storage temperature (°C)	Enthalpy of retrogradation (J/g). Storage time (days)									
		1	2	3	4	5	6	7	8	10	14
Potato starch[a]	6		13.6		14.4						
Potato starch[b]	25			1.0	2.4		6.0		7.5		
Potato starch[c]	23	4.6	5.5	6.3		6.8		7.0			7.4
	4	7.8	8.0	8.8		8.5		9.0			9.1
	−10	8.1	8.5	8.8		9.2		9.2			
Waxy potato starch[a]	6		13.6		13.5						

[a] Starch:water (1:1) (Fredriksson, 1998)
[b] Starch:water 40:60 w/w (Hoover et al., 1994)
[c] Starch:water 30:70 w/w (Kim et al., 1997).

Changes in time–temperature cycling decreased ΔH_R (Table 11). The authors attributed the change in ΔH_R to a decrease in the amount of crystalline material. An increase in propagation temperature (30–40°C) increased T_o (onset temperature of retrogradation) and decreased (Table 11) the melting temperature range (T_o–T_f) of the crystallites (formed during retrogradation). Silverio et al (2000) have postulated that the decrease in T_o–T_f reflects the formation of a more homogenous set of crystallites at 40°C.

Ishii, Kawabata and Nakamura (1994) showed by DSC studies on 20% solutions of tuber and root starches (cooled to 5°C), that the rate and extent of retrogradation follows the order: canna > potato > arrowroot > cassava. Cassava starch did not exhibit a measurable ΔH_R until the 32nd day of storage. The above authors postulated that the rate and extent of amylopectin retrogradation is influenced by its chain length (canna > potato > arrowroot > cassava). Similar findings were reported by Ring et al (1987) and Kalichevsky, Orford and Ring (1990) on potato and cassava starch gels.

11. Retrogradation monitored by rheological measurements

Mita (1992) examined changes in storage modulus (G'), loss modulus (G'') and loss tangent (tan θ) with storage time at 22°C for a 12.5% (w/w) potato starch paste by measurement of dynamic viscoelasticity. The authors observed a rapid increase in G' at the early stages of ageing, and a slow increase in G'' during the latter stages. This was attributed to entanglement of solubilized amylose and to an increase in rod-like growth of crystals, respectively.

12. Retrogradation monitored by changes in mechanical properties of starch gels and by changes in the degree of gelatinization.

Inaba, Hatanaka, Adachi, Matsumura & Mori (1994) examined the changes in properties of potato and cassava gels (at different concentrations) with storage time (5°C, 30 days). Starch gels were measured for the mechanical parameters (using a compression tester equipped with a cylindrical plunger of 0.5 cm^2 cross section) which relates to hardness, toughness, fracturability and elasticity. In both potato and cassava starches, compression work rupture force and resiliency increased (potato > cassava) with storage time and concentrations. However, compressibility decreased (cassava > potato) with storage time. In this study compression work, fracturability, resiliency and compression work correspond to toughness, hardness, elasticity and fracturability, respectively. The above authors also measured changes of the degree of gelatinization of potato and cassava starch gels with time using enzymatic hydrolysis (β amylase and pullulanase). The results showed that the order of decreasing retrogradation rate was 15, 19 and 10% in potato and 20, 16 and 25% in tapioca. The correlation coefficients between the changes of the degree of gelatinization (index of rate of retrogradation) and the changes of each mechanical parameter of the gels showed that resiliency and/or fracturability could be used as an index for estimating the progression of retrogradation.

13. Retrogradation monitored by FT/IR

Van Soest, DeWit and Tournois (1994) studied the retrogradation kinetics of a potato starch–water system (10% w/w gel) by FT-IR/ATR spectroscopy. They showed that the C–C and C–O stretching region (1100–800 cm^{-1}) to be sensitive to retrogradation. The most pronounced changes in the spectrum was found to occur at 1000 (peak), 1035 (valley) and 1053 (peak) cm^{-1}. Changes of intensity bands during storage reflect changes in specific starch conformations such as long range ordering and crystallinity, whereas, band narrowing reflects ordering of the polymer chains and a reduction in the number of conformations.

14. Retrogradation monitored by rapid Raman spectroscopy and X-ray diffraction

Bulkin et al. (1987) analyzed the retrogradation of potato

Table 11
Transition temperatures and retrogradation enthalpy of potato starch gels (starch:water, 1:1) after treatment with different time-temperature cycles (Adapted from Silverio et al. (2000))

Starch source	Storage temperature (°C)	T_o[a]–T_f[b] (°C)	ΔH_R[c] (J/g AMP)
Potato (normal amylose)	6/6[d]	38.2–78.9	13.6
	6/30[e]	50.3–78.0	13.2
	6/30/6/30[f]	51.8–78.2	13.6
	6/40[g]	59.4–78.9	11.6
	6/40/6/40[h]	60.7–79.5	12.6
Potato (high amylopectin)	6/6[d]	39.1–79.3	13.6
	6/30[e]	49.9–77.9	13.5
	6/30/6/30[f]	51.3–78.0	13.9
	6/40[g]	58.8–78.9	11.7
	6/40/6/40[h]	60.4–79.4	12.3

[a] T_o (onset of retrogradation).
[b] T_f (conclusion of retrogradation).
[c] Enthalpy of retrogradation.
[d] 48 h at 6°C.
[e] 24 h at 6°C → 24 h at 30°C.
[f] 24 h at 6°C → 24 h at 30°C → 24 h at 6°C → 24 h at 30°C.
[g] 24 h at 6°C → 24 h at 40°C.
[h] 24 h at 6°C → 24 h at 40°C → 24 h at 6°C → 24 h at 40°C.
→ denotes followed by

starch (52% starch and 48% water) gelatinized at 90°C and then cooled to room temperature by rapid Raman spectroscopy. The authors observed a narrowing of the half band width of the 480 cm^{-1} band with storage time. After 6 h, the spectrum was very similar to that of the initial sample, and by 50 h there was no visible change in the Raman spectrum. A plot of half band width of the 480 cm^{-1} band vs. storage time revealed four stages (I–IV) in the retrogradation process: (i) an initial rapid phase (represents conformational ordering involving the formation of double helices in amylopectin branches within a single polymer molecule (ii) a plateau (represents the induction time for onset of amylopectin helix aggregation and crystal growth; (iii) a slow process (represents the primary amylopectin aggregation and crystallization step); and (iv) a very slow process (represents crystalline phase propagation and perfection step). The above authors have also shown by wide angle and small angle X-ray diffraction studies that crystal development occurs only during stage iii. Furthermore, in stage (iv), the X-ray diffraction intensity is much higher than the Raman scattering. This means that long range ordering (crystalline phase propagation and crystal perfection) occurs during stage (iv). The authors have postulated that the role of amylose can be viewed as providing an acceleration by a template effect on amylopectin. Amylose itself retrogrades rapidly, forming an ordered (on the molecular level) matrix which is not necessarily highly crystalline. These ordered chains may act as seed nuclei for regions of the amylopectin, accelerating all steps in the crystalline process. Orford, Ring, Carrot, Miles and Morris (1987) determined the long term development of crystallinity in 30% (w/w) potato starch gels by X-ray diffraction. The matured gels showed diffraction patterns characteristic of the B type crystalline modification of starch.

15. Digestibility

Digestibility of native starches among and within species have been attributed to the interplay of many factors such as starch source (Ring, Gee, Whittam, Orford, & Johnson, 1988), granule size (Snow & O'Dea, 1981), amylose/amylopectin ratio (Hoover & Sosulski, 1985), extent of molecular association between starch components (Hoover & Sosulski, 1985), degree of crystallinity (Dreher, Berry, & Dreher 1984; Ring et al., 1988), amylose chain length (Jood, Chauhan, & Kapoor, 1988), amylose – lipid complexes (Holm et al., 1983).

The in vitro hydrolysis of tuber and root starches are presented in Table 12 among these starches, potato and taro show the highest resistance to α-amylase. A meaningful comparison cannot be made with regard to variations in the extent of hydrolysis, due to differences in α-amylase source and reaction times.

16. Conclusion

This review has shown that there is a dearth of information on the surface properties, granule crystallinity, double helical content, amylose chain length, chain length distribution of amylopectin, and physicochemical properties (digestibility, retrogradation and rheology) of tuber and root starches. Furthermore, in many cases (with the exception of potato, sweet potato and cassava) only one cultivar has been used for the study of starch properties. Thus, the properties determined may not be truly representative of the species. Comparison of properties between these starches is difficult due to differences in methodology. Furthermore, many researchers have used only one technique for

Table 12
In vitro amylolysis of tuber and root starches

Starch source	α amylase source	Reaction time (h)	hydrolysis (%)
Solanum tuberosum	Porcine pancreatic[a]	72	5.4
	Pancreatic[b]		8.5
	Bacillus subtilis[b]		5.0
Ipomea batatas	*Bacillus subtilis*[b]	55	14.9
	Pancreatic[b]		43.3
	Porcine pancreatic[c]		48.8–63.4
Manhiot esculenta	*Bacillus subtilis*[d]	24	44.0
	Pancreatic porcine[d]		52.9
Dioscorea alata	*Bacillus subtilis*[d]	24	3.5
	Pancreatic porcine[d]		4.7
Xanthosoma sagitifolium (new coco yam)	*Bacillus subtilis*[d]	24	15.3
	Pancreatic porcine[d]		15.6

[a] Hoover and Vasanthan, 1994.
[b] Fuwa, Nakajima and Hamada (1997).
[c] Zhang and Oates, 1994.
[d] Valetudie, Colonna, Bouchet and Gallant (1993).

determining gelatinization and retrogradation properties of tuber and root starches. This approach is not sound, since the various methods such as DSC, X-ray, NMR, DMA, FT/IR and Raman spectroscopy measure different properties of the material. A more advantageous approach may be to use these methods collectively to obtain a deep insight into the physicochemical properties of these starches. There is thus, a need to carry out a systematic investigation on starches from different cultivars of the same species using the same set of analytical techniques and experimental conditions.

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