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Manuscrit of Food Processing Technologies-Practical works
Intended for L3 Agri-food and Quality Control students

Food Processing Technologies (Practical works)

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Preamble

Food processing technologies (TIAA) is taught in two sequential modules in the third year of the bachelor's program in food science and quality control. The first module (TIAA1), offered in semester five, introduces core concepts and unit operations for the processing and preservation of foods of animal origin (dairy, meat, fish, and eggs) and of cereal grains and edible fats and oils. Laboratory classes and industry site visits are integrated to consolidate theoretical learning.

The second module (TIAA2), offered in semester six, builds on TIAA1. It covers fundamental and applied aspects of the processing and preservation of fruits and vegetables; sugars and sugar-based products; and beverages and fruit juices. The judicious and regulated use of food additives is included among the learning outcomes. Additional site visits reinforce links between theory and industrial practice.

Target audience

This laboratory handout is intended for third-year students specializing in Agri-food and Quality Control students.

Learning objectives

The food processing technologies sequence prepares students to apply scientific principles to industrial food operations while ensuring product quality and safety. On completion, students should be able to: understand and select appropriate unit operations for specific product and process goals; implement analytical methods for quality control; interpret key biochemical and physicochemical reactions relevant to processing and storage; apply hygiene and food safety principles consistent with hazard analysis and critical control points (HACCP) and relevant standards; consider environmental impacts and resource efficiency in process design; and identify, assess, and control process and product hazards.

Prerequisites

Recommended prior knowledge includes chemistry, biochemistry, microbiology, physics, thermodynamics, and energy balances.

Laboratory component

This handout details seven laboratory exercises that address major topics in the agri-food sector, with emphasis on cereal processing, fruit juices, and canned foods:

1. Gluten extraction
2. Falling number (Hagberg–Perten) test
3. Chopin alveograph analysis
4. Determination of moisture content in flour
5. Determination of ash content in flour
6. Determination of reducing sugars in foods
7. Evaluation of the stability of canned foods

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

General Introduction

Food processing technologies encompass the scientific principles and unit operations used to convert perishable agricultural commodities into safe, stable, and nutritious foods, while maintaining economic efficiency and regulatory compliance. As a field, they integrate food chemistry, microbiology, engineering, and quality management to control transformation, preservation, and packaging so that products meet explicit safety and quality specifications throughout the value chain (Fellows, 2017).

Archaeological and biomolecular evidence indicates that foundational preservation practices—drying, smoking, and fermentation—emerged with early sedentary societies in the Neolithic. Mixed fermented beverages have been documented at Jiahu (Henan, China) as early as ca. 7000 BCE, illustrating the long-standing role of biotransformations in extending shelf life and diversifying diets. Similar techniques, together with drying and smoking of meats and fish, preceded modern thermal and packaging innovations by millennia. These records anchor the historical continuity between preindustrial practices and contemporary, process-controlled fermentation and dehydration technologies (McGovern et al., 2004; Penn Museum, 2004; FAO overviews; archaeological syntheses).

The industrialization of food processing accelerated from the late 18th to early 20th centuries with step-change innovations that created today's factory-based "agrifood" sector. Thermal processing in hermetically sealed containers—pioneered by Nicolas Appert and later adapted to metal cans—provided the first robust pathway to commercial sterility and global distribution. Parallel advances included the extraction and refining of beet sugar following Marggraf's 1747 discovery and Achard's early-19th-century factory-scale production, as well as the mechanization of chocolate manufacture with hydraulic pressing and conching. These developments exemplify how scientific discovery, engineering design, and organizational change transformed artisanal production into modern industrial manufacture (Appert; Marggraf and Achard; van Houten press; Lindt conche).

In contemporary economies, the agrifood industry functions as a system linking upstream input suppliers and primary producers to midstream processors and downstream distributors and

retailers. Framed as an agrifood value chain, it comprises coordinated activities—production, aggregation, processing, distribution, consumption, and disposal—governed by quality and safety standards and informed by risk-based regulation. Because food demand is universal, the sector is a major driver of employment, trade, and rural development; upgrading processing capacity and logistics is central to food security and loss reduction agendas (FAO value- chain guidance; UNIDO methodological briefs; KPMG sector mapping).

Within this system, Food Processing Technologies (FPT) aim to design, validate, and operate transformations that preserve nutritional value and sensory quality while ensuring microbiological safety and regulatory compliance. Core tasks include characterization of raw materials and intermediate streams; selection and modeling of unit operations; specification of process controls and critical limits; verification through analytical, microbiological, and statistical methods; and continuous improvement under recognized quality management frameworks. These functions align technological choices with public health goals and market requirements across diverse product categories and processing scales (Fellows, 2017; FAO, 2023–2024).

1 Gluten Extraction

1.1 Introduction

Glūten is Latin for “glue.” In cereal science, gluten denotes the cohesive, viscoelastic protein complex formed when wheat flour is hydrated and mixed. This complex underpins dough elasticity, gas retention, and bread volume in baking (Shewry, 2024). Wheat is the most widely consumed gluten source globally; *Triticum aestivum* (common or “soft” wheat) predominates in breads and pastries, while *Triticum durum* (durum wheat) is milled to semolina for pasta and couscous. In North Africa, including Algeria, wheat-based foods such as bread, couscous, and pasta are dietary staples, which elevates the importance of robust quality control for consumer protection and process standardization.

1.2 Historical background

Beccari first isolated and described “gluten” in 1745 during studies of wheat dough (Shewry, 2024). The clinical entity now known as celiac disease dates to Aretaeus of Cappadocia, who in the 1st–2nd century CE described patients with chronic malabsorption he called *koiliakos*. In 1887–1888, Samuel Gee provided the first modern clinical account and emphasized diet as central to management. During and after World War II, Willem Karel Dicke demonstrated that wheat ingestion precipitated relapse and that a gluten-free diet induced remission, establishing the causal role of cereal proteins (Gee; Dicke; historical syntheses).

1.3 Definition

Gluten is the cohesive, viscoelastic protein network that remains after the starch fraction of wheat dough is washed out with water. This network—formed during mixing and kneading—confers extensibility and elasticity to dough, enabling gas retention during yeast fermentation and baking. In wheat, gluten comprises two major protein classes: gliadins and glutenins. Gliadins are monomeric prolamins that primarily confer viscosity and extensibility, whereas glutenins are polymeric proteins responsible for dough elasticity and strength. In clinical contexts, the term *gluten* is often extended to prolamin fractions from related cereals—hordeins

in barley, secalins in rye, and avenins in oats—because these proteins can elicit gluten-related disorders in susceptible individuals.

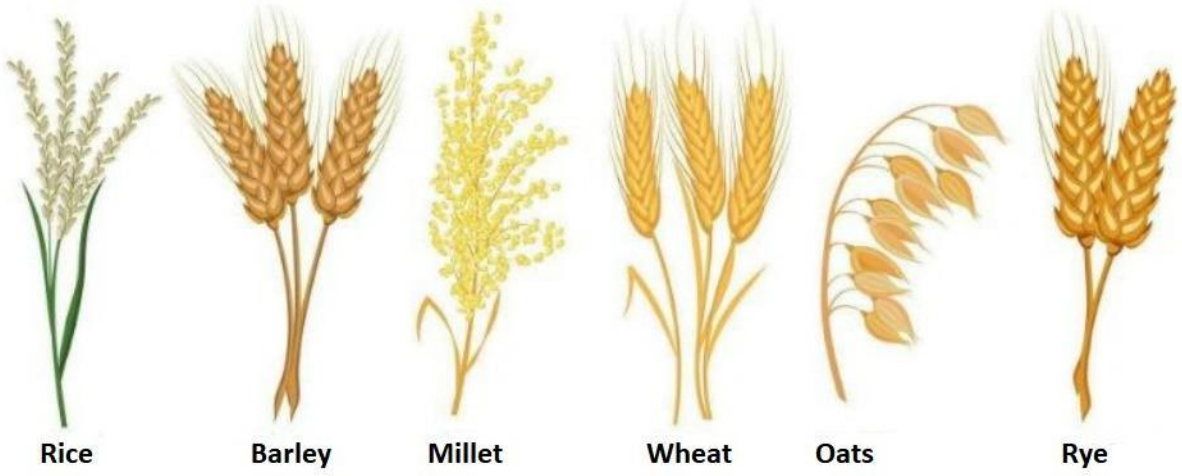


Figure 1. Representative cereal grains relevant to gluten and gluten-related disorders

1.4 Sources of Gluten

1.4.1 Dietary sources

Gluten is naturally present in several cereals and in foods made from them. Table 1 lists common dietary sources.

Table 1. Common dietary sources of gluten

Category	Examples
Wheat and derivatives	Wheat flour, whole wheat, wheat starch, wheat bran, wheat germ, spelt, semolina, bulgur, breadcrumbs, modified wheat starch, pasta, breads, pastries
Barley and derivatives	Barley flour, barley flakes, malt, malt extract, malt syrup, malt vinegar, beer
Rye and derivatives	Rye flour, rye bread, mixed wheat–rye breads

Oats and derivatives	Oat flour, oat bran, rolled oats (unless certified purity-tested gluten-free)
Processed foods commonly containing these cereals	Couscous, croutons

Note. The regulatory threshold for a "gluten-free" claim is commonly ≤ 20 mg/kg (20 ppm) gluten in many jurisdictions; verify local regulations when interpreting labels.

1.4.2 Hidden or less obvious sources

Because of its functional properties—binding, thickening, film-forming, stabilizing, and texturizing—gluten or gluten-containing derivatives may appear as ingredients or processing aids in diverse products (Table 2). Awareness of these "hidden" sources is essential for gluten-free diets, especially in medically indicated cases such as celiac disease or non-celiac gluten sensitivity.

Table 2. Examples of hidden or less obvious sources of gluten

Product category	Examples and notes
Processed foods	Soups and soup bases; bouillon; sauces and gravies; marinades; seasonings and spice mixes; confectionery and some chocolates; processed meats and deli products
Ingredients and additives	Malt and malted flavorings; wheat-based thickeners; hydrolyzed vegetable or plant proteins when the botanical source is unspecified; modified starches when the botanical source is unspecified
Beverages	Beer and some flavored malt beverages; certain flavored coffees and teas with cereal-based flavorings
Non-food exposures	Selected medicines (excipients), mouthwashes and toothpastes, and some cosmetics such as lipsticks or balms when wheat-derived excipients are used
Fermentation by-products	Brewer's yeast products that may contain barley residues

Notes. (i) Labeling rules determine whether the cereal source must be declared; check local law. (ii) "Wheat-free" is not equivalent to "gluten-free" when barley or rye derivatives are present.

1.4.3 Vital Wheat Gluten

Vital wheat gluten is a dried, concentrated gluten protein obtained from wheat flour by dough washing, separation of the insoluble protein fraction, and low-temperature drying to preserve functional properties. Upon rehydration, vital gluten reconstitutes a viscoelastic network that strengthens dough. Industrial production typically involves hydration of wheat flour, wet separation of gluten from starch, mechanical dewatering, and drying below temperatures that would denature the proteins and impair functionality.

Vital wheat gluten is widely used in baking and cereal-based products to increase dough strength, water absorption, and gas retention. It is particularly useful in:

- **Enriched or high-fat doughs** where added lipids weaken gluten development.
- **Cereal-blend breads** or formulations with non-gluten flours where network reinforcement is needed.
- **Frozen dough systems**, in which ice crystal formation and freeze–thaw cycles weaken gluten structure; added vital gluten helps maintain volume and crumb quality.

Table 3. Indicative protein composition of wheat gluten fractions

Protein group	Typical proportion in conventional gluten	Typical proportion in vital wheat gluten
Gliadins (prolamins)	~40–45%	~45–50%
Glutenins (polymeric)	~40–45%	~50–65%

Note. Ranges vary by wheat cultivar, milling and washing conditions, and drying method.

1.5 **Wheat Grain**

Wheat refers to several *Triticum* species in the *Poaceae* family cultivated primarily for flour production. The dominant species is common wheat (*Triticum aestivum* L.), used for breads and a wide range of baked products; within this species, hard classes are typically milled for bread flour, whereas soft classes are suited to cakes and biscuits because of lower protein and weaker gluten networks. Durum wheat (*T. turgidum* subsp. *durum*) is milled into semolina for pasta due to its vitreous endosperm, strong gluten, and high yellow pigment. Wheat also contributes to alcoholic beverages and distilled spirits, although barley is more common in brewing. These use patterns reflect functional differences in kernel composition, gluten-forming protein quality, and milling behavior (Shewry, 2009; Kent & Evers, 2016).

The wheat kernel comprises three morpho-anatomical fractions that differ in mass and composition (Figure 2). The endosperm constitutes about 82–85% of kernel weight and is rich in starch with storage proteins that become gluten-forming upon hydration and mixing. The bran accounts for roughly 13–15% and encompasses the pericarp, seed coat (testa), hyaline layer, and the aleurone; this fraction concentrates dietary fiber, phenolic compounds, minerals, and B vitamins. The germ **(**embryo**)** represents about 2–3% and is nutrient dense, providing lipids, vitamin E, and micronutrients (Shewry & Halford, 2002; Belitz et al., 2009). In milling, removal of bran and germ improves flour stability but reduces fiber, vitamin, and mineral content.

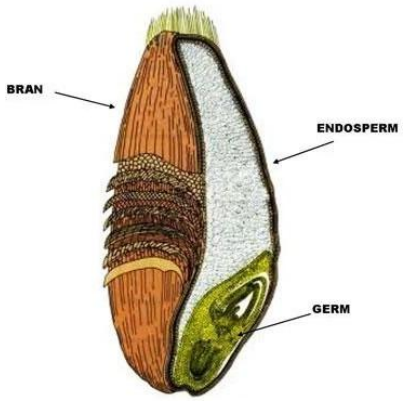


Figure 2. Morphological components of the wheat kernel

Whole wheat is an important source of macronutrients and micronutrients (Figure 3). Starch is the predominant carbohydrate and the principal energy source; native sugars are present in smaller amounts. Protein typically ranges from about 10% to 15% of kernel mass in common wheats and consists chiefly of gluten-forming fractions—gliadins and glutenins—that determine dough rheology and gas retention during breadmaking. Total lipid content of whole grain is modest (≈2–3%) and is concentrated in the germ, with triacylglycerols and polar lipids that contribute to flavor and oxidative stability considerations. Dietary fiber is concentrated in the bran, where arabinoxylans, β-glucans, cellulose, and lignin support gastrointestinal function and satiety. Wheat also supplies B-group vitamins (thiamin, niacin, pyridoxine, folate), vitamin E from the germ, and minerals including magnesium, phosphorus, iron, zinc, and manganese. The energy value of whole wheat grain is typically around 330–360 kcal per 100 g, depending on cultivar and moisture content (Belitz et al., 2009; USDA, 2024; Shewry, 2009).

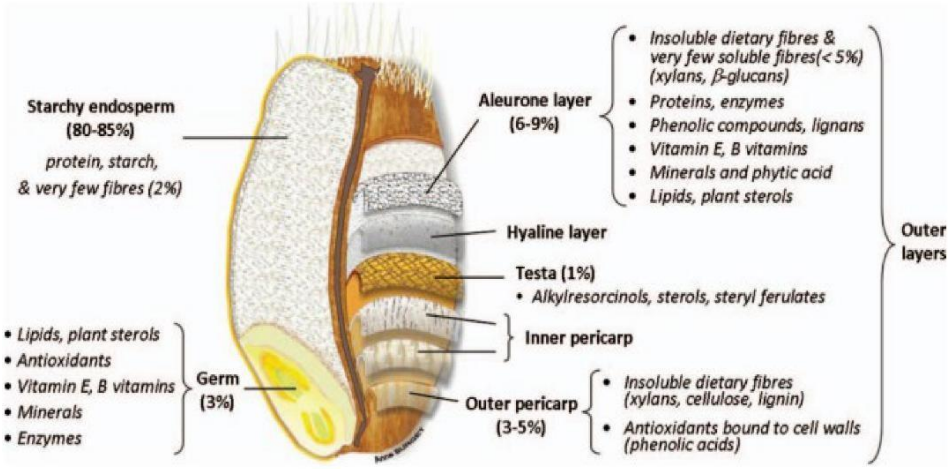


Figure 3. Typical proximate and micronutrient profile of whole wheat grain

1.6 **Gluten-Related Disorders**

Scope**.** *Gluten-related disorders* comprise immune-mediated conditions triggered by ingestion or exposure to gluten-containing cereals, principally wheat, barley, and rye. The major entities are celiac disease, wheat allergy, and non-celiac gluten (wheat) sensitivity. Extraintestinal manifestations such as dermatitis herpetiformis and gluten ataxia form part of this spectrum (ACG, 2013; AGA, 2021; EAACI, 2021).

1.6.1 Celiac disease

Celiac disease is a chronic, immune-mediated enteropathy precipitated by dietary gluten in genetically susceptible individuals who carry HLA-DQ2 and/or HLA-DQ8 haplotypes. It is pathophysiologically distinct from IgE-mediated food allergy. Gluten ingestion induces autoimmune responses involving tissue transglutaminase (TG2), leading to small-intestinal mucosal inflammation, crypt hyperplasia, and villous atrophy, with consequent malabsorption (ACG, 2013; Lebwohl et al., 2018).

Clinical presentation is heterogeneous. Gastrointestinal features include abdominal pain, diarrhea, bloating, and constipation; many patients present with extraintestinal or subtle signs such as iron-deficiency anemia, fatigue, headache, cognitive complaints, infertility, osteoporosis, or neuropathy, and some are asymptomatic ("silent" celiac disease). In children, growth failure and delayed puberty can occur. Long-term untreated disease increases risks of micronutrient deficiencies, low bone mineral density, adverse pregnancy outcomes, and, rarely, enteropathy-associated T-cell lymphoma (ACG, 2013; Ludvigsson et al., 2014).

Diagnosis relies on serology—total IgA with IgA anti-tissue-transglutaminase (tTG-IgA) and, if needed, IgG-based tests (deamidated gliadin peptides) in IgA deficiency—followed by duodenal biopsy showing characteristic histopathology while the patient consumes gluten. HLA typing has high negative predictive value when serology and histology are discordant. In select pediatric cases, *no-biopsy* pathways are accepted when tTG-IgA is ≥10× the upper limit and endomysial antibodies are positive on a second sample, per ESPGHAN criteria (ESPGHAN, 2020). Treatment is a lifelong strict gluten-free diet, with dietetic counseling, monitoring of clinical response and serology, and correction of deficiencies; vaccination considerations apply in hyposplenism (ACG, 2013; AGA, 2021).

1.6.2 Wheat allergy

Wheat allergy is an IgE-mediated hypersensitivity to wheat proteins. Reactions typically occur within minutes to hours after exposure and can involve the skin, respiratory, gastrointestinal, or cardiovascular systems, including anaphylaxis. Relevant allergens include gliadins and glutenins

as well as albumins and globulins. Clinical phenotypes include classic food-induced IgE allergy, wheat-dependent exercise-induced anaphylaxis (often linked to ω-5 gliadin), baker's asthma and rhinitis from inhalational exposure, and contact urticaria. Diagnosis uses history with skin-prick testing, serum specific IgE, component-resolved diagnostics where available, and oral food challenge when indicated. Management centers on wheat avoidance and emergency preparedness with epinephrine for systemic reactions (EAACI, 2021; Sicherer & Sampson, 2018).

1.6.3 Non-celiac gluten (wheat) sensitivity

Non-celiac gluten sensitivity (NCGS; also termed non-celiac wheat sensitivity) describes symptom onset after gluten-containing foods in individuals without celiac disease or wheat IgE allergy. Symptoms span gastrointestinal complaints (pain, bloating, altered bowel habits) and extraintestinal features (fatigue, headache, "brain fog"). Pathophysiology remains unsettled; proposed triggers include gluten, amylase–trypsin inhibitors, and FODMAPs within wheat. Diagnosis is clinical and by exclusion, ideally demonstrating symptom improvement on a gluten-free diet and relapse with blinded gluten challenge, though such challenges are rarely performed in routine practice (Biesiekierski, 2014; Catassi et al., 2015; AGA, 2021). Management is individualized dietary modification, often starting with supervised gluten elimination and consideration of low-FODMAP strategies to avoid unnecessary restriction.

1.6.4 Extraintestinal immune manifestations

Dermatitis herpetiformis is the cutaneous manifestation of celiac disease, characterized by pruritic vesicles and granular IgA deposition in dermal papillae with anti-transglutaminase-3 autoimmunity. Skin lesions improve on a gluten-free diet; dapsone provides rapid symptom control during diet transition (Salmi et al., 2019).

Gluten ataxia is an immune-mediated cerebellar ataxia associated with anti-gliadin and anti-transglutaminase-6 antibodies, with or without enteropathy; early diagnosis and a strict gluten-free diet may stabilize or improve neurological outcomes (Hadjivassiliou et al., 2014).

1.7 Gluten extraction

Gluten extraction isolates the wheat storage proteins—gliadins and glutenins—from starch and water-soluble fractions. In the standard wet process, flour is hydrated to form a cohesive dough, then washed with water under controlled shear. Washing removes dispersed starch granules and solubles through sieving or centrifugation, leaving a viscoelastic residue identified as wet gluten. Dewatering and low-temperature drying of this residue yield vital wheat gluten for functional or analytical use.

In analytical applications, the method quantifies gluten content and assesses gluten quality. Reported metrics typically include wet gluten, dry gluten, and the gluten index, which together indicate flour suitability for bread-making. Higher gluten quantity and appropriate gluten quality correlate with dough elasticity, extensibility, and gas-retention capacity, thereby supporting leavening performance and loaf volume. For comprehensive flour evaluation, gluten measurements are interpreted alongside total protein content and dough rheology obtained by standard instruments (e.g., farinograph or extensograph) under controlled test conditions.

1.8 Key steps in extracting gluten from flour and semolina

1.8.1 Dough preparation

Prepare two test doughs separately from soft-wheat flour and durum semolina. Hydrate each test portion with approximately 48–52% water by weight to obtain a cohesive mass suitable for washing. Standardized mechanical methods wet 10.00 g flour with ~4.8 mL of 2% NaCl solution, which corresponds to ~48% hydration.

1.8.2 Hydration and kneading

Allow the dough to hydrate, then knead to promote formation of the gluten network from gliadin and glutenin fractions. Proper hydration and short kneading ensure a continuous viscoelastic phase prior to washing.

1.8.3 Gluten extraction (washing)

Wash the dough using 2% (w/v) NaCl solution, by manual or mechanical means (e.g., Glutomatic). Continue washing under controlled flow and shear until starch and solubles are removed and a cohesive wet-gluten mass remains. The 2% NaCl wash and cycle times are specified in AACC 38-12 and ISO 21415-2.

1.8.4 Drying for dry-gluten determination

For dry-gluten quantification, dry the wet gluten in a ventilated oven at 130 °C ± 2 °C for 6 h to constant mass, with intermediate incisions after 2 h, as specified in ISO 21415-3.

1.8.5 Expression of results

Report wet and dry gluten separately for flour and semolina. Use masses recorded during the procedure:

- **Wet gluten content**

$$\mathrm{TGH}\ (\%) = \frac{M_1}{m_0} \times 100$$

where m_0 is the mass of the test portion (g) and M_1 is the mass of wet gluten after washing (g). When required, adjust to a reference moisture basis using the AACC 38-12 conversion factor.

- **Dry gluten content**

$$\mathrm{TGS}\ (\%) = \frac{m_2}{m_0} \times 100$$

where m_2 is the mass of the dried gluten (g) obtained under ISO 21415-3 conditions.

Include the gluten index when relevant, as defined by the proportion of wet gluten retained on the sieve after standardized centrifugation.

1.8.6 Interpretation of results

Durum semolina typically exhibits higher protein and wet-gluten levels than common-wheat flours, supporting pasta firmness and couscous texture. Commercial semolina often targets ≥ 13% protein with strong gluten aggregation; common T55-type flours are usually lower to mid-range in protein and gluten strength. Interpret gluten quantity and quality together using gluten index or rheology.

Gluten levels vary with genotype, nitrogen fertilization, environment, and milling. French “T” numbers (T45, T55, T65, T110, T150) indicate ash content and refinement, not gluten content per se; wholemeal types (e.g., T110, T150) contain more bran, which mechanically disrupts gluten development despite sometimes higher total protein. Avoid inferring gluten percentage from T-number alone.

2 Hagberg Falling Number

2.1 Introduction

Bread is a staple food in most diets, including regions where wheat is not locally cultivated. Its persistent popularity reflects the sensory attributes of well-crafted bread. Consumers prioritize flavor, aroma, crumb and crust texture, and external appearance. These expectations have driven continuous innovation in formulas, processing conditions, and baking technologies. Bakeries therefore optimize ingredient functionality, dough handling, and thermal profiles to enhance sensory quality and ensure product consistency at scale.

2.2 Rheological Properties of Wheat Flour

Rheological properties describe the flow and deformation behavior of flour–water doughs under applied stress or strain. They are critical determinants of dough handling, gas retention during proofing, and the structure set during baking. Optimal rheology is product-specific: breadmaking requires doughs with sufficient gas-holding capacity and structural strength during final proof and the early stages of baking, whereas biscuit and cracker manufacture favors weaker gluten networks with high extensibility and low dough strength to limit spring and promote spread.

2.3 Principal Rheological Properties

2.3.1 Elasticity

Elasticity is the tendency of a deformed dough to recover its original shape upon removal of stress. In wheat doughs, elasticity arises primarily from the gluten network and supports gas retention during fermentation and oven spring.

2.3.2 Extensibility

Extensibility is the capacity of dough to stretch without rupture. Adequate extensibility facilitates moulding and expansion during proofing and baking; excessive extensibility with low strength can cause loss of shape, while insufficient extensibility can limit volume.

2.3.3 Dough strength (Tenacity)

Dough strength denotes resistance to deformation. Stronger doughs better withstand CO_2 pressure during proofing and maintain loaf geometry. In practice, strength must be balanced with extensibility to avoid tight crumbs or reduced volume.

2.3.4 Water absorption

Water absorption is the amount of water required to reach a target dough consistency. It is influenced by protein content and quality, damaged starch, and fiber. Accurate determination guides formula hydration and affects mixing energy, dough handling, and shelf-life.

2.3.5 Development time

Development time is the mixing duration needed to achieve optimal gluten network formation and dough functionality. Under-mixing yields weak structure; over-mixing causes gluten breakdown, stickiness, and reduced gas retention.

2.3.6 Stability

Stability is the time interval over which a dough maintains near-optimal rheological properties under mixing or fermentation conditions. Higher stability indicates better tolerance to process variability and delays.

2.3.7 Dough rheology in processing

Dough rheology integrates elastic, viscous, and viscoelastic responses across unit operations, from mixing and dividing to proofing and baking. Instrumental assessments (e.g., farinograph for absorption, development, and stability; alveograph for tenacity–extensibility balance; extensograph for resistance and extensibility; rheometers for fundamental moduli) support process control and product specification.

2.4 Factors Influencing Rheological Properties

Rheological behavior of wheat- flour doughs depends primarily on gluten quantity/quality and starch functionality, but multiple compositional and process variables interact.

- **Protein content and gluten quality.** Higher protein generally increases dough strength and gas-holding, provided glutenin polymer size and gliadin–glutenin balance support viscoelasticity. Weak or under-oxidized gluten reduces stability and loaf volume.
- **Damaged starch and native starch properties.** Milling-induced starch damage increases water absorption and amylase susceptibility, affecting dough stickiness and proof tolerance. Granule size distribution and amylose/amylopectin ratio influence pasting and set.
- **Enzymatic activity (α-amylase).** Excess activity lowers paste viscosity, weakens crumb, and increases stickiness; deficiency yields tight crumbs and low volume. The Hagberg Falling Number indexes this effect in slurry, informing expected dough behavior.
- **Particle size distribution.** Finer flours show higher surface area and faster hydration; very fine fractions can raise absorption and mixing energy yet risk over-development and stickiness. Coarser particles interrupt the gluten network and reduce gas retention.
- **Hydration level.** Water addition controls dough consistency, gluten development kinetics, and mixing energy. Over-hydration causes stickiness and shape loss; under-hydration yields crumbly dough and poor expansion.
- **Fiber and bran.** Insoluble fibers and bran disrupt the gluten matrix, dilute proteins, compete for water, and increase dough stiffness; soluble fibers (e.g., arabinoxylans, β-glucans) modulate viscosity and may improve water binding depending on molecular weight and extraction.
- **Lipids and surface-active compounds.** Endogenous lipids and added emulsifiers (e.g., DATEM, SSL) modify gas-cell stability and dough strength through gluten–starch–air interfacial effects.
- **Mineral content (ash) and ionic environment.** Salt strengthens gluten and improves gas retention by screening charges; reducing agents (e.g., L-cysteine) increase extensibility; oxidants (e.g., ascorbic acid) increase strength via disulfide formation.

- **Fermentation variables.** Yeast level, time, temperature, and pH alter dough rheology through CO_2 generation, proteolysis, and acidification, impacting extensibility and stability.
- **Thermal and mechanical history.** Mixing speed, specific mechanical energy, and dough temperature during make-up determine development time and tolerance.

2.5 Instruments for Measuring Rheological Properties

Empirical and fundamental tests quantify complementary aspects of dough behavior used in specification and process control.

1. **Farinograph.** Measures water absorption to a target consistency and reports development time, stability, and softening, indicating mixing tolerance and flour strength.
2. **Extensograph.** Quantifies resistance to extension and extensibility over resting times, reflecting the balance of tenacity and stretch relevant to moulding and proofing.
3. **Alveograph.** Provides P (tenacity), L (extensibility), and W (deformation energy). Useful for predicting bread volume and for differentiating bread, biscuit, and pasta flours.
4. **Viscometers and rheometers.** Fundamental small-amplitude oscillatory and steady-shear tests yield storage and loss moduli, yield stress, and flow curves. These characterize viscoelasticity beyond empirical devices and can assess temperature or enzymatic effects. (Mixograph or doughLAB may also be used where applicable.)

Note. The Hagberg Falling Number test evaluates α-amylase activity via slurry viscosity collapse; it complements, but does not replace, dough rheology tests.

2.6 Importance of Rheology in Manufacturing

Rheology links flour specification to process performance and product quality.

- **Flour selection.** Match P/L and W (alveograph), absorption and stability (farinograph), and extensograph profiles to the target product class.
- **Process optimization.** Set hydration, mixing energy and time, rest/proof schedules, and oxidant/reductant dosing to achieve the desired viscoelastic balance.

- **Quality control.** Monitor lot-to-lot variability, adjust formula or process in real time, and predict outcomes such as loaf volume, crumb structure, spread, or bite in bread, pasta, biscuits, and crackers.

2.7 Hagberg–Perten Falling Number

The Hagberg–Perten Falling Number (FN) quantifies α-amylase activity in cereals, principally wheat and rye, by measuring the viscosity loss of a heated flour–water slurry. Low FN indicates high α-amylase activity typically associated with pre-harvest sprouting or weathered grain; high FN indicates low α-amylase activity. For breadmaking, very low FN yields sticky crumb and poor loaf structure due to excessive starch hydrolysis, whereas excessively high FN can reflect insufficient enzyme activity and reduced fermentable sugar formation, limiting loaf volume.

2.7.1 Principle

A standardized mass of flour, corrected to 14% moisture, is dispersed in water in a viscometer tube and heated in a boiling water bath while being stirred with a plunger. Starch gelatinizes and increases viscosity, while endogenous α-amylase hydrolyzes starch and decreases viscosity. After a fixed mixing period, the plunger is released; the time for it to fall through the slurry depends on slurry viscosity and thus on net α-amylase activity. The FN is the total time in seconds (mixing period plus fall time) (ISO 3093; ICC 107/1; AACC 56-81).

2.7.2 Apparatus and method description

The apparatus comprises a thermostatic boiling water bath, a calibrated glass viscometer tube, a standardized stirrer/plunger assembly, and an automated or manual timing and lifting mechanism. Early methods were manual; current instruments automate lifting, mixing, release, and timing for improved repeatability and operator safety.

Figure 4. Schematic of FN apparatus

2.7.3 Procedure

1. **Sample preparation.** Mill wheat to test flour or use commercial flour. Adjust test portion to the mass equivalent of 7.0 g at 14% moisture (per method), weighed to ±0.05 g.
2. **Slurry formation.** Add distilled water to reach the prescribed volume (typically 25 mL). Insert the plunger and rapidly mix to disperse flour and initiate gelatinization uniformly.
3. **Heating and mixing.** Place the tube in a vigorously boiling water bath and mix for 60 s at the specified stroke rate. During this period, starch gelatinizes and α-amylase begins hydrolysis.
4. **Fall time measurement.** After 60 s, automatically lift and release the plunger. Record the time for the plunger to descend to the bottom mark. FN = 60 s (mixing) + fall time. Higher α-amylase activity lowers viscosity and shortens the fall time, reducing the FN.

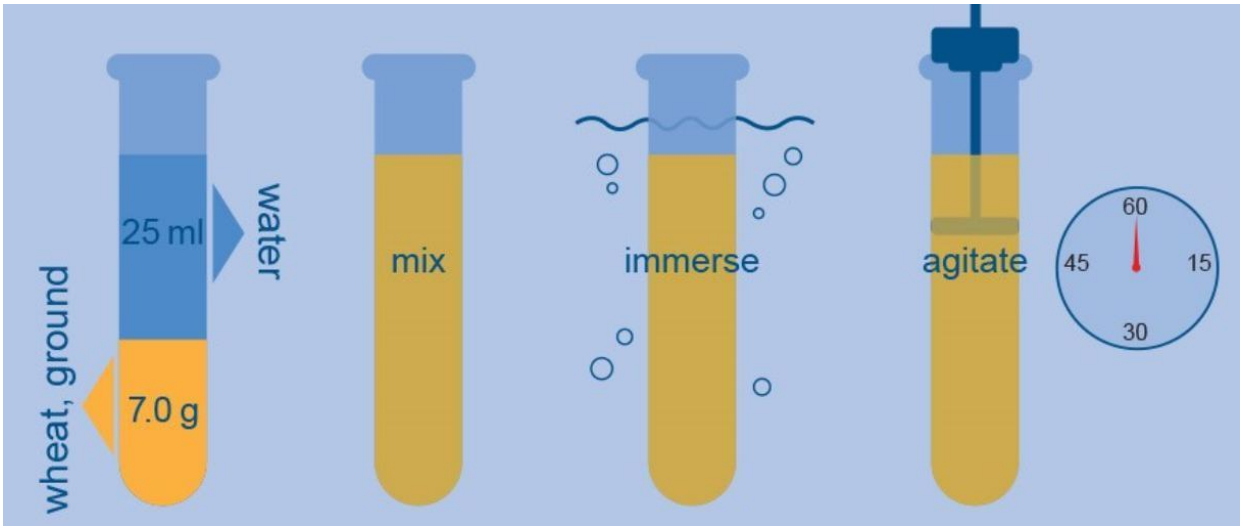


Figure 5. Steps for measuring the Hagberg Falling Number

2.8 **Interpretation**

FN values integrate both starch gelatinization and enzymatic thinning. Interpretation must consider cultivar, growing conditions, and target product.

- **Low FN (<≈170–200 s).** Strong α-amylase activity consistent with sprout damage; high risk of sticky crumb, coarse cell walls, and collapsed structure. Corrective options include blending with high-FN flour or using exogenous oxidants; gluten is not the primary target of α-amylase—the defect arises from starch hydrolysis.
- **Moderate FN (≈220–300 s).** Generally suitable for pan breads and rolls; often used as commercial acceptance ranges. Many markets use ≈220 s as a minimum specification for "sound" lots.
- **High FN (>≈350–400 s).** Very low α-amylase activity; potential for tight crumb and reduced volume unless diastatic malt or fungal α-amylase is added. High FN is not universally "better"; optimal depends on product and process.

Commercial note

FN is widely used for lot classification and pricing in grain trade. Thresholds such as 220 s for "sound" wheat are common, but specifications vary by region and end use. FN should be

interpreted alongside moisture, protein, ash, and dough rheology (farinograph, alveograph, extensograph) for robust quality control.

2.9 Role of α-Amylase in Flour

α-Amylase catalyzes endo-hydrolysis of α-1,4-glycosidic bonds in starch, producing dextrins, maltose, and glucose. In wheat systems, activity derives from endogenous grain enzymes and, where needed, added diastatic malt or microbial enzymes. Its technological value is indirect: by modulating starch gelatinization–viscosity and releasing fermentable sugars, α-amylase affects proofing kinetics, loaf volume, crumb structure, and staling behavior. Activity must remain within a narrow process window; excess causes loss of structure and sticky crumb, deficiency limits gas production and volume.

2.9.1 Functional contributions in breadmaking

1. **Generation of fermentable sugars.** Controlled hydrolysis increases maltose and glucose available to yeast, supporting CO_2 production during proofing and early baking. Sound wheats often require small additions of diastatic activity to reach target sugar levels; sprout-damaged wheats already exhibit high activity.
2. **Dough handling and viscoelastic balance.** In doughs, α-amylase does not act on gluten; effects on "extensibility" are secondary to changes in starch phase and water distribution. Moderate activity can reduce excessive dough tightness by lowering pasting viscosity of the starch fraction during mixing and proofing, improving moulding and gas-cell expansion. Overactivity thins the starch phase, destabilizes gas cells, and promotes spread and collapse.
3. **Crumb structure and volume.** Adequate activity promotes uniform cell walls and higher loaf volume by sustaining gas production into early oven spring. Excess activity weakens gelatinizing starch, producing gummy, under-set crumbs and wall rupture.
4. **Shelf-life and staling.** Short dextrins formed from limited α-amylolysis interfere with amylopectin retrogradation, modestly delaying crumb firming. This anti-staling benefit is formulation- and process-dependent. Heat-stable bacterial amylases risk post-bake crumb

gumming; fungal amylases are typically chosen for appropriate thermal inactivation profiles.

2.9.2 Practical control and limits

- **Optimal range.** For pan breads, flours with Falling Number ≈220–300 s generally indicate suitable endogenous activity. Lower FN implies excessive activity; higher FN suggests supplementation with malt or fungal α-amylase.
- **Dosing.** Typical fungal α-amylase additions are in the ppm range of flour weight; titrate against process conditions and product style.
- **Interactions.** Oxidants (e.g., ascorbic acid) adjust gluten strength independently of α-amylase. Proteolysis, not amylolysis, degrades gluten; distinguish faults accordingly.
- **Process sensitivity.** Dough temperature, pH, and fermentation time affect apparent enzyme action and should be held within validated ranges.

2.9.3 Clarifications of common misconceptions

- α-Amylase does not "degrade gluten." It acts on starch; gluten changes stem from mixing, oxidation–reduction chemistry, and proteases.
- Improved "extensibility" from α-amylase reflects starch-phase softening and gas-cell dynamics, not direct modification of the protein network.
- Shelf-life extension is due primarily to reduced starch retrogradation, not to "better heat dissipation" or moisture loss.

2.10 Germination of Wheat

Germination is the physiologic transition of a quiescent caryopsis into a seedling. It begins with water imbibition and requires suitable temperature, oxygen, and moisture. Signals from the embryo (notably gibberellins) induce the aleurone layer to synthesize and secrete hydrolytic enzymes into the starchy endosperm. These enzymes mobilize stored reserves—lipids, starch, and proteins—to fuel early growth. The same enzymatic activities, when present in harvested grain ("pre-harvest sprouting"), alter flour functionality and dough/bread quality.

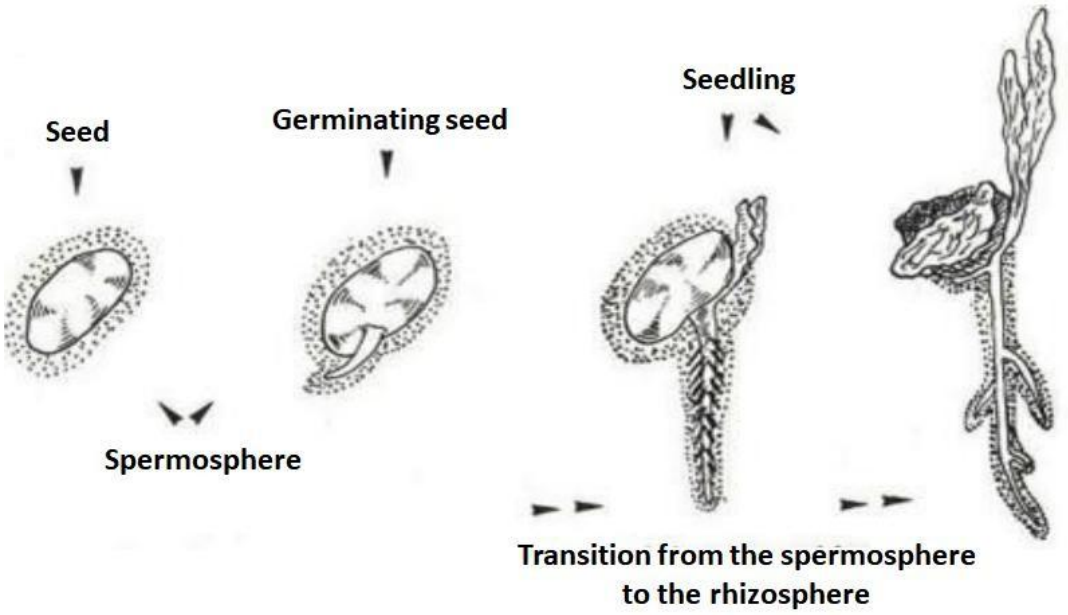


Figure 6. Stages of wheat germination

2.10.1 Lipases

Lipases hydrolyze triacylglycerols to free fatty acids and glycerol. Activity is concentrated in the embryo/scutellum, where lipid reserves are richest. Lipase action supplies energy substrates for β-oxidation in the germinating seedling. In wholemeal flour during storage, endogenous lipases and lipoxygenases may continue to act at low rates, promoting rancidity; they are not the sole active hydrolases under storage, but they contribute most to lipid hydrolysis.

Key points:

- Primary location: embryo and scutellum; induction during germination.
- Technological note: in flour, unchecked lipid hydrolysis/oxidation can impair flavor and dough properties.

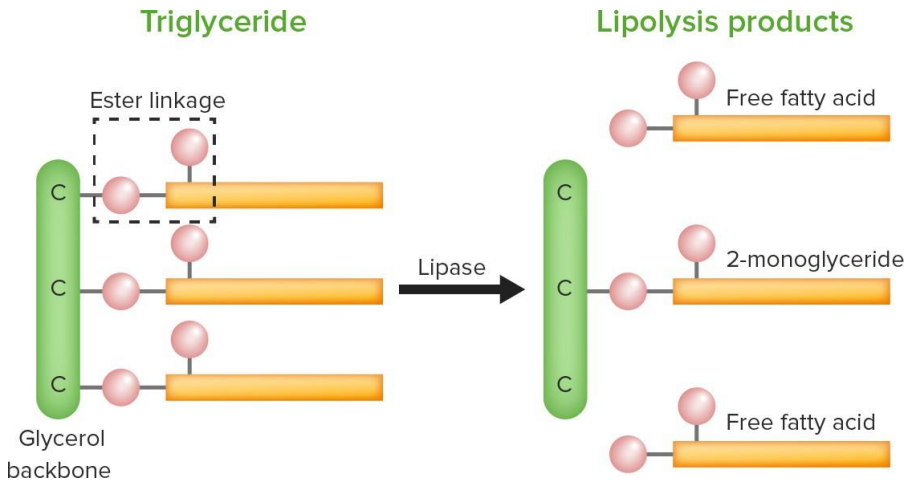


Figure 7. Schematic of lipase action on wheat lipids

2.10.2 Amylases and Proteases

Upon hormonal induction, the aleurone secretes amylases and proteases into the endosperm. Early fermentation in dough relies on a small native pool of fermentable sugars (~1–2% of flour); sustained CO_2 production requires enzymatic release of maltose/glucose from starch and peptides/amino acids from storage proteins.

- **Proteases/peptidases** hydrolyze gluten and other storage proteins into peptides and amino acids that support embryo growth and, in dough systems, modulate viscoelasticity. Excess proteolysis softens dough excessively.
- **Amylases** hydrolyze starch when gelatinized or partially swollen, reducing paste viscosity and providing fermentable sugars for yeast or lactic acid bacteria.

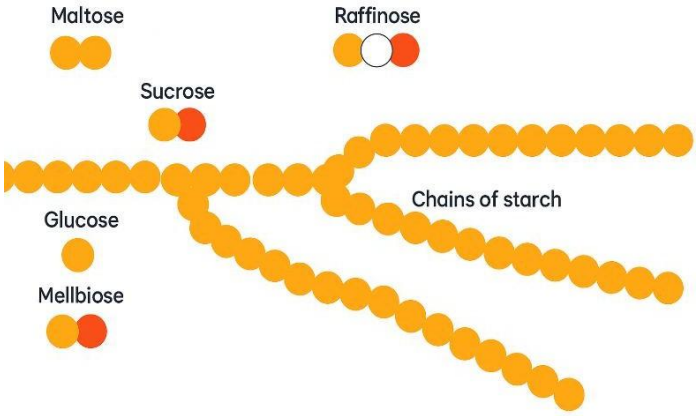


Figure 8. Simple versus composite carbohydrates in wheat grain

2.10.3 α- and β-Amylases

Wheat contains preformed β-amylase in the endosperm and synthesizes α-amylase de novo during germination.

- **α-Amylase (endo-amylase)** cleaves internal α-1,4 bonds in amylose and amylopectin, generating dextrins and reducing viscosity ("liquefaction").
- **β-Amylase (exo-amylase)** releases maltose units from the non-reducing ends of dextrins and native starch ("saccharification").
- **Complementarity.** α-amylase creates new chain ends; β-amylase converts those ends to maltose, increasing fermentable sugars.

Technological interpretation:

- Elevated α-amylase from sprouting lowers slurry viscosity, producing low Falling Numbers and risk of sticky crumb and collapse.
- β-amylase content changes little with sprouting; imbalance with high α-amylase yields excessive dextrinization without commensurate saccharification control, giving pasty crumbs.

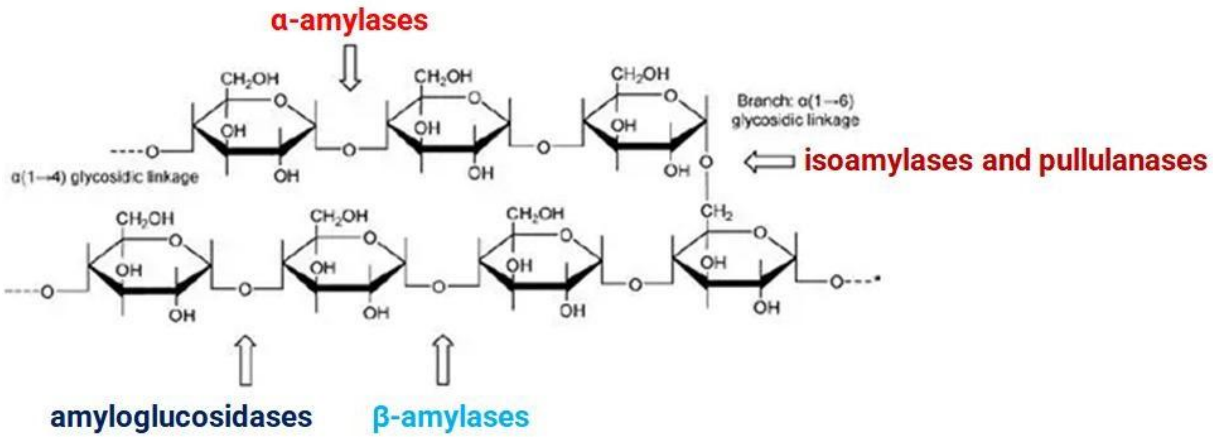


Figure 9. Partial structure of amylopectin with amylolytic enzymes action sites

2.10.4 Practical links to flour quality

- **Falling Number (ISO 3093/ICC 107/1/AACC 56-81).** Integrates the "liquefying" effect of α-amylase on a heated slurry. Low FN indicates high α-amylase and probable sprout damage; typical commercial thresholds near 220 s define sound lots, with product-specific targets.
- **Process control.** High-FN flours may need diastatic malt or fungal α-amylase; low-FN flours are blended upward. Diagnose softness due to proteases separately from amylase-driven viscosity loss.

3 Chopin Alveograph

3.1 Introduction

The technological value of wheat flour derives from a constellation of physicochemical attributes that determine its suitability for baked products such as bread, rusks, and biscuits. Among these attributes, the rheological behavior of dough formed by flour hydration and mixing is critical. The Chopin alveograph is a sector-standard instrument used to characterize dough plasticity and viscoelasticity. Its results support:

- Selection, evaluation, and commercial classification of wheat varieties and lots;
- Determination and adjustment of flour plastic properties, with or without additives, to meet downstream product specifications; and
- Rational blending of wheat or flour batches to reach target alveographic indices that obey mixture additivity, notably baking strength WWW, tenacity PPP, and extensibility LLL.

The alveograph is used both upstream in grain trading and cultivar development and downstream across baking and related processing industries.

3.2 I. Historical development

The alveograph design has evolved through successive innovations from the early twentieth century to the late twentieth century:

- **1907:** Jenö Hankóczy develops an early device for forming and assessing a single dough bubble.
- **1920:** Marcel Chopin introduces the “extensimeter” to evaluate breadmaking performance via rheological metrics (extensibility, resistance to extension, and elastic recovery). The instrument gains rapid adoption in milling and baking for flour and blend optimization and for testing new formulations.
- **1921:** The extensimeter is refined into the alveograph, enabling recording of a deformation curve as a dough sheet inflates under controlled air pressure.

- **1927:** An improved model with enhanced recording capability is commercialized.
- **Mid-1930s:** An integrated dough mixer is incorporated to standardize specimen preparation.
- **1982:** An air pump replaces the water column for bubble inflation, improving control and reproducibility.
- **1987:** A timer and temperature display are added to stabilize test conditions.
- **1995:** Bubble inflation is automated; the "NG" model is introduced alongside the Consistograph for complementary measurements.

Today, the Chopin alveograph remains widely used for wheat varietal selection and flour quality assessment.

3.3 **Definition and principle**

The Chopin alveograph (Figure 10) measures the deformation behavior of a thin, standardized dough sheet that is clamped and inflated by air until rupture. The instrument records the pressure–deformation curve, from which key indices are computed:

- **Tenacity P (mm H_2O):** maximum overpressure. It reflects dough resistance to deformation.
- **Extensibility L (mm):** total deformation at rupture. It reflects the dough's capacity to stretch without tearing.
- **Configuration ratio P/L (–):** balance between resistance and extensibility, indicative of dough "strength profile."
- **Baking strength (10^{-4} J):** area under the curve. It quantifies the overall energy required to inflate the bubble and is widely used as an integrative indicator of flour strength.

In viscoelastic terms, baking strength captures the combined contributions of elastic resistance and viscous flow during biaxial extension. Elasticity denotes the dough's ability to recover its shape after deformation, whereas extensibility denotes its capacity to undergo large strains prior to rupture. Together, these parameters inform millers and bakers about flour suitability for

specific products and process conditions, and they guide blending strategies to achieve targeted performance.

Figure 10. Chopin alveograph

3.4 Uses

The Chopin alveograph is routinely employed across the wheat–flour value chain by storage and trading operators, millers, and secondary processors.

- For storage and trading operators, the instrument serves to secure wheat and flour transactions against an internationally recognized analytical reference, to verify quality at intake, to sort and grade lots for subsequent use, and to screen for quality defects associated with insect-damaged kernels that can impair dough viscoelasticity.
- For millers, the alveograph supports prediction of flour quality from grist blends, calculation of blend ratios required to reach a target dough profile, precise adjustment of additives and processing aids to tailor flour to end uses, and continuous verification of in-process and outbound flour quality.
- For secondary processors (e.g., bakers, pasta and biscuit manufacturers), it is used to confirm the conformity of incoming flours to specifications, to assess new product

formulations, and to evaluate functional improvers by quantifying their effect and optimal dosage on dough behavior.

3.5 **Measurement Principle**

The alveograph quantifies the viscoelastic response of a standard dough sheet formed from flour and a saline solution under controlled conditions. A circular sheet of dough is clamped over an orifice and inflated at a constant air-flow rate. The instrument records the pressure required to inflate the dough "bubble" as a function of deformation until rupture. The resulting pressure–deformation curve captures two complementary properties: resistance to deformation (commonly termed tenacity, related to the curve's initial rise) and capacity for deformation before rupture (commonly termed extensibility, related to the descending portion of the curve). The total area under the curve represents the deformation energy **(**W**)**, a consolidated index of dough strength. In standard practice, a firm dough is prepared at 50% hydration to ensure reproducible measurements under defined temperature and mixing conditions.

3.6 **Operation and Curve Interpretation**

A test piece of firm dough is placed between two plates, each with a central aperture, and is sealed to prevent slippage. Air is then introduced at a constant rate through the orifice to inflate the dough sheet. During the initial phase, the dough shows little swelling and resists the applied pressure in a manner analogous to the early inflation of a balloon; on the alveogram this behavior is expressed by a steep pressure rise and reflects tenacity. As inflation proceeds, the dough bubble deforms, increases in volume, and the pressure rise slows; on the curve this is represented by the rounded apex followed by a gradual decline, reflecting extensibility as the dough accommodates further deformation. Rupture is indicated by a sharp pressure drop back to baseline. The full curve, often reported as P (maximum overpressure, an index of tenacity), L (the abscissa at rupture, an index of extensibility), P/L (the balance between resistance and stretch), and W (area under the curve, the energy required to inflate to rupture), provides a concise functional profile used to match flour to process requirements and product types.

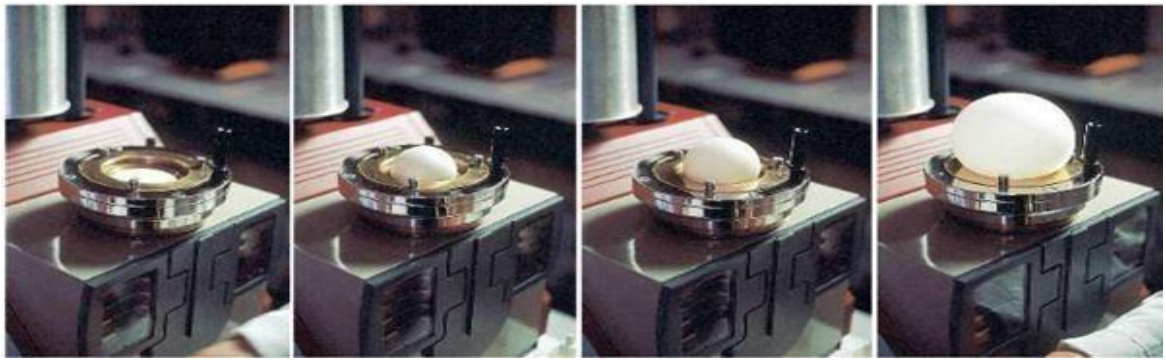


Figure 11. Stages of dough bubble development during alveograph measurement

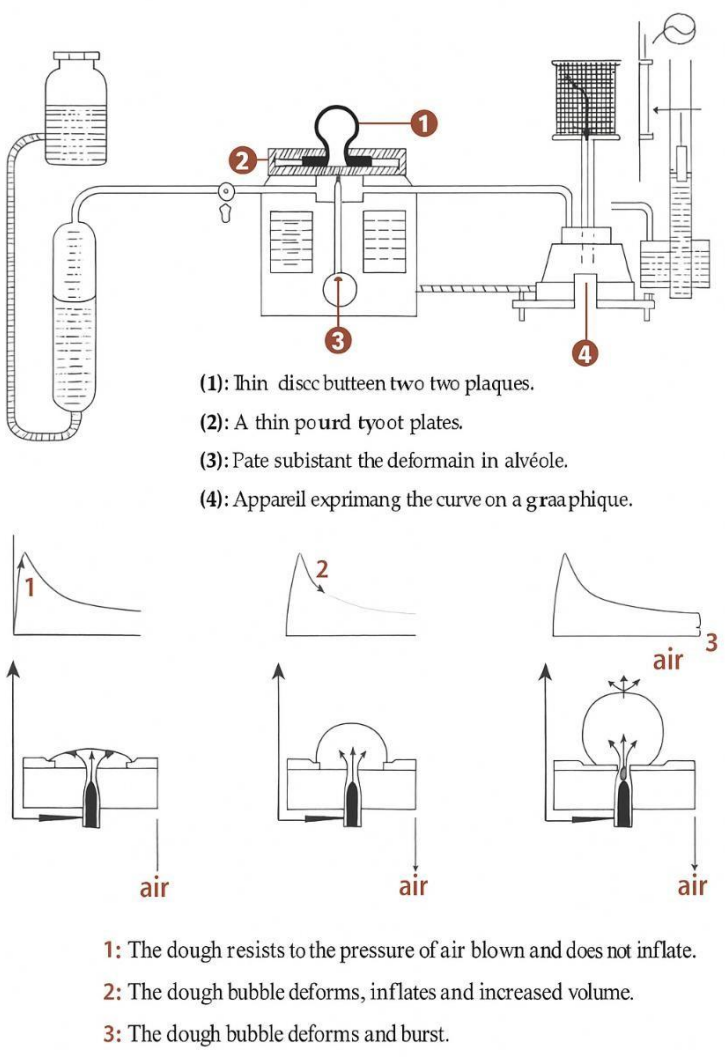


Figure 12. Representative Chopin alveogram

Results should be reported with test conditions (hydration, dough rest time, temperature, mixing protocol, and instrument model), and with the full set of alveograph indices (P, L, P/L, W). When specifications are contractual, refer to the applicable standard method and edition to ensure method equivalence. If figures are included, captions should follow the format above and units should be stated explicitly (e.g., P in mmH_2 O, W in 10^{-4} J).

3.7 Determination of Baking Strength with the Chopin Alveograph

The alveograph determines the baking strength of flour by inflating a standardized dough sheet with air at a constant flow until rupture, while continuously recording the pressure–deformation response (ISO, 2008). A cylindrical recorder generates an alveogram (Figure 13) whose size, shape, and rupture point reflect the dough's viscoelastic balance. From this curve, the instrument predicts the suitability of a wheat or flour for specific baked, pasta, or biscuit products by quantifying resistance to deformation and capacity to stretch before failure.

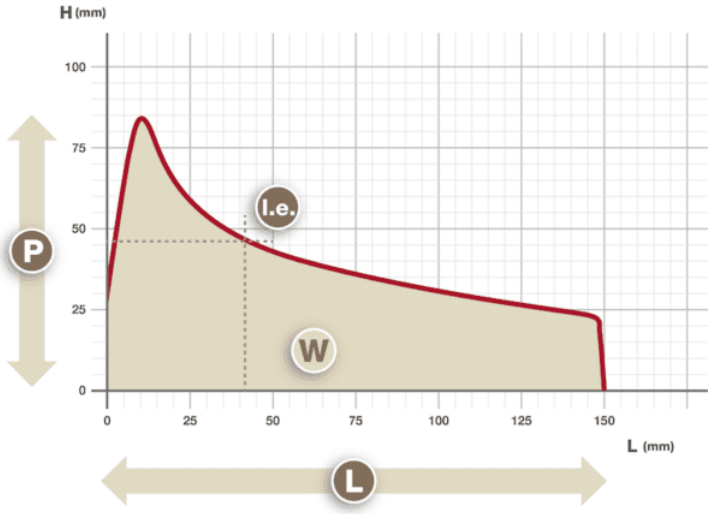


Figure 13. Standard alveographic curve

3.8 Principle of measurement

A firm dough is prepared from flour and saline solution under defined conditions. The dough is sheeted, clamped over an orifice, and inflated by an air stream at a fixed rate. Pressure increases as the dough resists deformation, then reaches a maximum as the bubble expands, and finally

drops to baseline at rupture. The pressure (mm H_2 O) is plotted against deformation; the area under the curve corresponds to the mechanical work required to inflate the bubble to failure, which is a consolidated index of dough strength (ISO, 2008).

3.9 **Alveographic indices and interpretation**

The alveogram is summarized by four primary indices and one ratio. Units must be reported.

1. **W (deformation energy, "baking strength")**: Area under the alveogram, expressed in 10^{-4} J per gram of dough. W quantifies overall dough strength. Higher W indicates greater capacity to withstand mechanical and gas-expansion stresses during processing and proofing. W correlates positively with flour protein quantity and quality.
2. **P (tenacity, maximum overpressure)**: Peak pressure on the alveogram, typically in mm H_2 O. P reflects dough resistance to deformation. Higher P denotes tighter, more elastic doughs that oppose stretching.
3. **L (extensibility)**: Abscissa at rupture, in mm. L represents the maximum deformation the dough sustains before failure. Larger L indicates greater stretchability.
4. **G (swelling index)**: A derived index of bubble expansion commonly calculated as $G = 2.2 \times \sqrt{L}$ (L in mm; G reported in arbitrary alveographic units). G summarizes volumetric swelling behavior of the dough sheet prior to rupture.
5. **P/L ratio (elastic–plastic balance)**: Captures the balance between resistance to deformation (P) and extensibility (L). Flours with identical W can behave differently in processing if their P/L differs; reporting W together with P/**L** is therefore essential for specification and end-use matching.

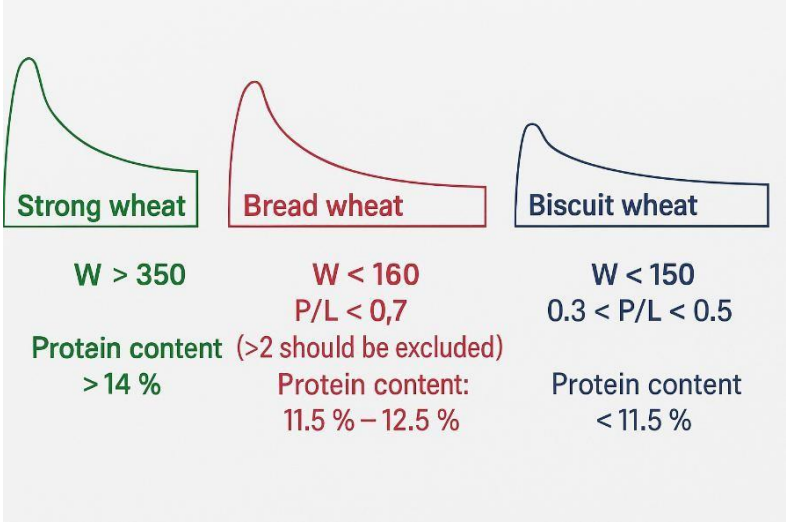


Figure 14. Schematic alveographic curves illustrating contrasting classes of wheat and flour behavior

3.10 **Typical specification ranges and indicative uses**

Report method conditions with results (hydration, rest time, temperature, instrument model, sheet thickness). The ranges below are indicative; industrial specifications vary by product style, process, and local standards.

Table 4. Potential uses of wheat according to their baking strength

Baking strength W (10^{-4} J)	**P/L ratio**	**Indicative end use**
< 150	0.3–0.5	Sweet and dry biscuits, cookies
160–250	0.5–0.7	Pan breads and flatbreads
250–300	0.5–0.9	Viennoiserie (croissant, brioche)
250–350	0.7–1.0	Sandwich and pan-baked breads

Notes.

- W thresholds overlap by product and process; artisan or long-fermentation breads may require higher L at moderate P (lower P/L), whereas laminated doughs often benefit from higher P at moderate L.
- For contractual acceptance, cite the exact method version and edition used for testing.

3.11 **Test Steps**

The alveographic test produces a standardized dough specimen that inflates into a bubble under controlled air pressure. This extension mimics dough deformation under carbon dioxide pressure during fermentation. The procedure comprises four stages (Figure 15).

Figure 15. Stages of the Chopin alveograph test

1. **Kneading a flour–saline mixture.** Weigh 250 g of flour. Condition ingredients and equipment at 25 °C. Start the mixer, then add saline water containing 2.5% NaCl (w/w in water) during the first 20 s. Adjust the added water for flour moisture to achieve the method's constant hydration. Continue mixing for up to 8 min to obtain a firm, homogeneous dough.
2. **Preparation of five calibrated test pieces.** Reverse the divider–former to eject the dough and discard the first piece. Laminate the remaining dough pieces with a lightly oiled roller using six back-and-forth passes to reach the specified sheet thickness. From each sheet, punch one circular test disc with the standard cutter.
3. **Resting the test pieces.** Transfer each disc to a resting plate and place it in the alveograph's isothermal chamber at 25 °C. At minute 28, center a disc on the

instrument's oiled test plate and flatten it with the designated pad to ensure uniform clamping.

4. **Automatic inflation to rupture.** Start the air generator to inflate the dough at a constant flow until rupture. The instrument records the pressure–deformation signal, from which P (tenacity), **L** (extensibility), P/L (balance), and W (deformation energy, baking strength) are computed.

Report all set points with results: flour mass, dough hydration, NaCl concentration, mixing time, rest time, test temperature, sheet thickness, instrument model, and method edition.

3.12 **Interpretation of Results**

The Chopin alveograph provides an indirect assessment of flour baking quality by quantifying dough viscoelastic behavior under biaxial extension. Unlike product-specific bake tests, alveograph indices predict processing performance from rheological principles and are calibrated against industrial observations and baking trials. As such, they serve as common specification metrics across the cereal value chain and help control both process consistency and product quality.

Reading the alveogram requires tracking the pressure that the dough withstands as it inflates. The initial steep rise reflects resistance to deformation (P, tenacity). The abscissa at rupture quantifies the maximum deformation before failure (L, extensibility). The curve area integrates the mechanical work to rupture (W, baking strength). The P/L ratio summarizes the balance between elasticity and stretch.

For typical breadmaking processes, P/L values near 0.5–0.8 indicate a balanced dough, assuming an appropriate W for the product style and processing conditions. Acceptable ranges remain application-dependent: laminated products often tolerate higher P/L, while long-fermentation or high-hydration breads may require lower P/L with higher L at moderate P. Figure 16 schematizes the relationship between tenacity and extensibility for contrasting dough behaviors.

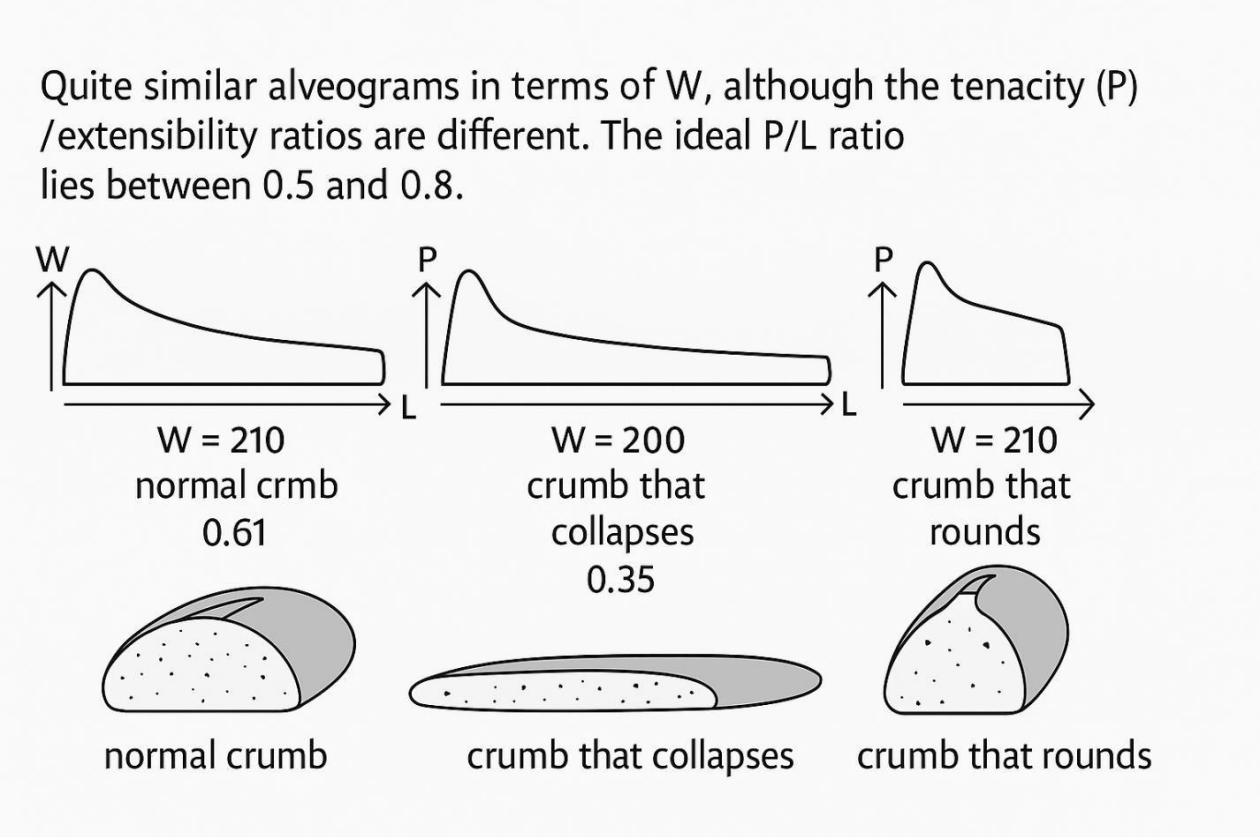


Figure 16. Schematic interpretation of tenacity (P) and extensibility (L) on the alveogram

4 Moisture Content of Flour

4.1 Introduction

Cereal products are staple foods in most countries and supply a substantial share of dietary protein and carbohydrates. Bakery products, particularly bread, occupy a predominant place in many diets, including in non-wheat-producing regions. Flour, the primary raw material for these products, is a powder obtained by milling cereal grains. Wheat flour consists mainly of starch and proteins. The water- and salt-soluble fractions are largely albumins and globulins, whereas gluten is formed in hydrated dough by the viscoelastic protein network of gliadins and glutenins.

Moisture content is a critical quality attribute that affects processability and preservation. Excessive moisture promotes caking and lump formation, supports microbial growth and enzymatic activity, and degrades baking performance. Conversely, overly low moisture yields flour that is dry, dusty, and less manageable during mixing and dough development. Moisture analysis is therefore integral to inspection, in-process control, storage management, and product development in the milling and baking industries. Finished products require moisture levels that optimize texture, sensory quality, and shelf life.

4.2 I. Definition of Water

Water (H_2O) is a transparent liquid ubiquitous on Earth. Its planetary cycling illustrates a biogeochemical cycle in which a chemical species moves through reservoirs via biological, geological, and meteorological processes. Solar energy drives evaporation from oceans, lakes, and soils; atmospheric transport and condensation form clouds; precipitation returns water to the surface; and runoff and infiltration close local hydrological loops (Figure 17). Although the global water cycle is often treated as closed with respect to total mass, small losses to the stratosphere and photodissociation occur.

The tendency of water to evaporate at a given temperature is described by its equilibrium vapor pressure, which rises steeply with temperature (Table V). At 100 °C, the saturation vapor pressure is approximately 101.33 kPa, equal to 1 atm. This temperature dependence underlies

practical phenomena important to food processing, including drying kinetics, water activity control, and storage stability.

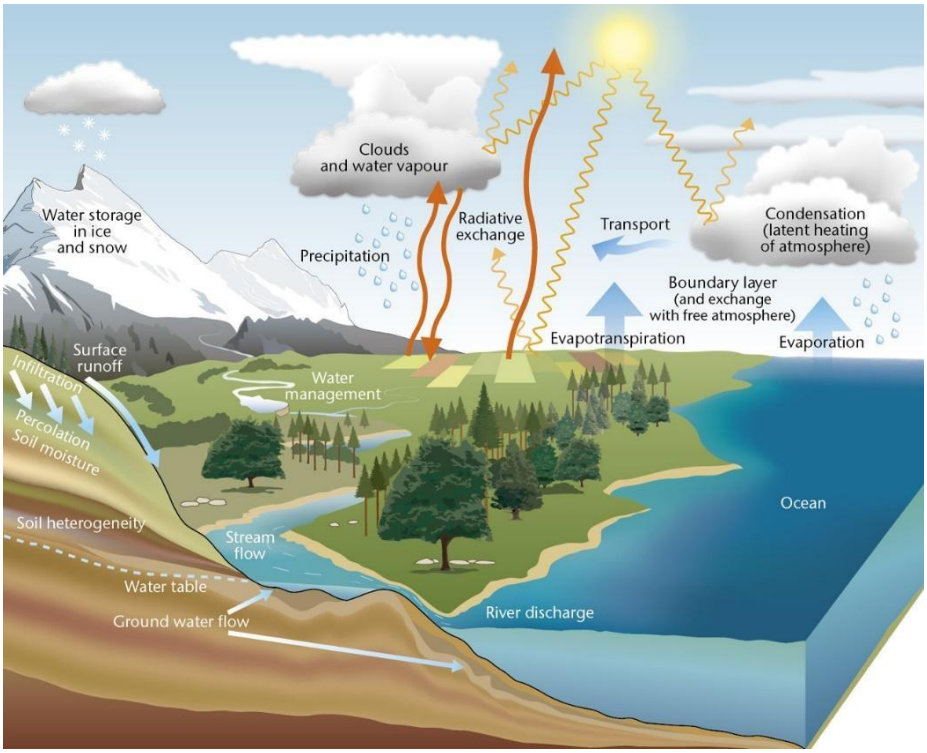


Figure 17. The water cycle in nature

Table 5. Saturation vapor pressure of water at selected temperatures

Temperature (°C)	**0**	**10**	**20**	**30**	**40**	**50**	**60**	**70**	**80**	**90**	**100**
Vapor pressure (kPa)	0.61	1.23	2.34	4.24	7.37	12.33	19.92	31.18	47.34	70.11	101.33

Water is the principal component of most living organisms. Biologically, it acts as a solvent and reaction medium, enables transport of nutrients and metabolites, and supports macromolecular structure and function. All known life depends on water to sustain biochemical reactions and reproduction.

4.3 Water in Food

Water in foods exhibits distinct physicochemical behaviors depending on how it associates with biopolymers, low–molecular weight solutes, and with other water molecules. These associations govern mobility, phase behavior, and availability for microbial growth and reactions.

4.3.1 Free (open) water

Free water is the mobile fraction located in pores and capillaries, present as solvent for dissolved solutes, or osmotically retained. It retains most properties of pure water, including the ability to crystallize on freezing and to participate in mass transfer and biochemical reactions. Its mobility underlies caking, stickiness, microbial proliferation, and rapid quality changes during storage.

4.3.2 Bound water

Bound water is closely associated with surface sites on proteins, polysaccharides, and other solids through hydrogen bonding, ionic interactions, and, to a lesser extent, hydrophobic effects. It forms the primary adsorbed monolayer, exhibits reduced mobility, and does not readily freeze at conventional food-freezing temperatures. Because interactions with the matrix exceed water–water cohesive forces, this fraction is effectively unavailable for most microbial and enzymatic processes.

4.4 Water Activity and Interactions Between Water and Other Constituents

4.4.1 Definition of water activity (a□)

Water activity quantifies the thermodynamic availability of water rather than its total content. It is defined as the ratio of the equilibrium vapor pressure of water above the food (p) to that above pure water (p^0) at the same temperature:

$$a_w = \frac{p}{p^0} = \frac{\mathrm{ERH}}{100}$$

where ERH is the equilibrium relative humidity (%) of the headspace at thermodynamic equilibrium with the product. By definition, $0 \leq a_w \leq 1$. Values near 1 indicate highly available water. Because both ppp and p^0 are temperature dependent, aw is temperature dependent. In foods, a_w is typically depressed below 1 by dissolved solutes (colligative effects) and by sorption to hydrophilic sites on the dry matrix.

Measurement principle: a sealed, temperature-controlled chamber allows the food to equilibrate with the headspace. Instruments sense ERH (e.g., chilled-mirror dewpoint or capacitive sensors), from which a_w is computed. Reporting should include temperature, instrument type, and equilibration protocol. Water activity is a critical quality and safety metric for moisture-sensitive products because microbial growth, enzymatic activity, nonenzymatic browning, lipid oxidation, and texture transitions each exhibit characteristic a_w dependencies.

4.4.2 Sorption isotherms (adsorption/desorption)

A moisture sorption isotherm relates water activity to equilibrium moisture content at a fixed temperature. It characterizes hygroscopic behavior and the strength and heterogeneity of water–solid interactions. Isotherms can be obtained by adsorption (rehydrating a previously dried sample) or desorption (drying a moist sample), typically revealing hysteresis between the two paths (Figure 18).

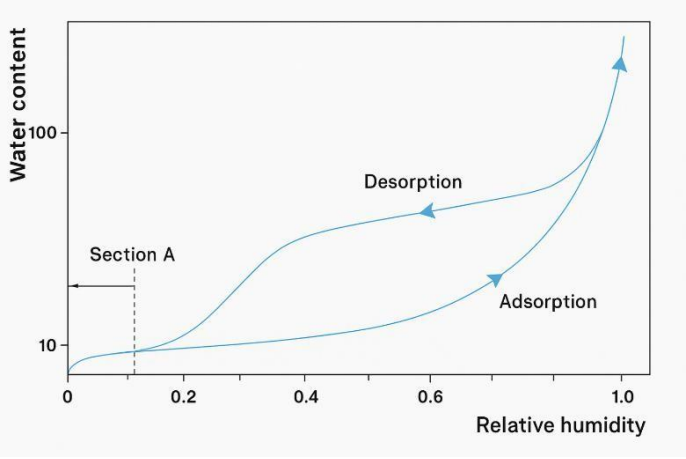


Figure 18. Representative moisture sorption isotherm

Foods most often display IUPAC Type II (sigmoidal) or Type III isotherms (Figure 19). For Type II isotherms, three functional regions are classically distinguished:

- **Region A (a□ < ~0.20).** Primary monolayer adsorption on polar sites. Water is strongly bound, unfreezable at typical temperatures, and largely unavailable for biochemical reactions. The moisture content at the inflection is the monolayer moisture content (M_0), a key parameter in the BET/GAB models.
- **Region B (~0.20 ≤ a□ ≤ ~0.70).** Multilayer adsorption with progressively weaker binding. Mobility and reactivity increase; many quality changes, including Maillard browning and certain enzymatic reactions, accelerate with rising a_w in this region.
- **Region C (a□ > ~0.70).** Capillary condensation in pores and interstices with minimal matrix–water interactions. Water behaves more like bulk liquid; microbial growth risk rises sharply, and caking/aggregation and texture softening become prominent.

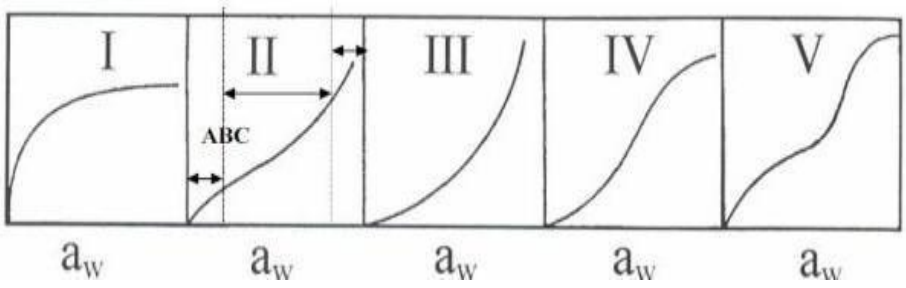


Figure 19. Moisture sorption isotherm types

4.5 Factors Influencing Water Activity

Water activity reflects the thermodynamic availability of water, not simply its total amount. Multiple variables act—often concurrently—to set a_w by altering vapor–liquid equilibrium, molecular binding, and phase state. Control of these variables determines product stability, safety, and sensory quality.

4.5.1 Temperature

At constant composition, a_w generally increases with temperature for aqueous solutions because the saturation vapor pressure of pure water (p^0) and the solution vapor pressure (p) change differently with temperature. Matrix transitions can create exceptions: near a glass transition or protein denaturation, altered binding and capillary condensation may shift a_w non-monotonically. Measurement therefore requires strict temperature control and reporting; standard practice is isothermal equilibration within ±0.2 °C.

4.5.2 Moisture content

Moisture content and a_w are related through the product's moisture sorption isotherm at a fixed temperature. The relationship is nonlinear and shows hysteresis between adsorption and desorption. Higher moisture tends to raise a_w, but the slope depends on matrix composition, porosity, and physical state. Consequently, moisture cannot substitute for a_w when predicting microbial growth or reaction rates.

4.5.3 Solute concentration

Dissolved solutes lower a_w by reducing the chemical potential of water (colligative effects). Nonelectrolytes (e.g., sucrose, glycerol) and electrolytes (e.g., NaCl) depress a_w with strength that scales with molality and, for salts, ion dissociation. Example: honey's high sugar content yields low a_w despite high total moisture compared with fresh fruit. The practical effect is slowed microbial growth and modified kinetics of browning and enzymatic reactions.

4.5.4 Physical structure and matrix state

Microstructure governs how water partitions among bound, multilayer, and capillary domains. Porous and fine-capillary matrices can promote capillary condensation at relatively low a_w, while compact or hydrophobic matrices limit sorption. The matrix state matters: glassy carbohydrates and proteins restrict water mobility and often keep a_w lower at a given moisture than rubbery counterparts. Hydrocolloids and proteins immobilize water via binding and viscosity increases;

note, however, that high–molecular weight binders mainly reduce mobility and do not substantially lower a_w unless they contribute low–molecular weight solutes.

4.5.5 Additional process and environmental factors

- **Hygroscopic equilibrium with ambient air.** A_w = ERH/100. Storage relative humidity drives moisture gain or loss until equilibrium, shifting a_w accordingly.
- **Drying.** Physical water removal lowers both moisture and a_w; the trajectory follows the desorption isotherm and depends on temperature and airflow.
- **Freezing.** Ice formation excludes solutes into the unfrozen phase, reducing the unfrozen water fraction and thereby lowering a_w of that phase, even though total water remains.
- **Formulation "hurdles."** Combining factors amplifies control of a_w and stability, e.g., salting plus drying of fish, or sugar addition plus low-temperature storage in confectionery.
- **Product type.** Each formulation exhibits a characteristic a_w range set by composition, structure, and processing history; specifications should be product- and process-specific.

4.6 Water Activity and Food Spoilage

Water activity is measured to assess product stability. Microbial growth, many chemical reactions, and most enzyme-catalyzed changes depend on water availability, not total water content. Each microorganism has a minimum a_w for growth; foods at or above that threshold permit proliferation, while foods below it are microbiologically stable for that organism.

No food is intrinsically immune to spoilage. Given suitable a_w, temperature, pH, and time, at least some bacteria, yeasts, or molds can grow. Organisms that tolerate low a_w are termed xerophiles (molds), osmophiles (sugar-tolerant yeasts), and halophiles (salt-tolerant microbes).

4.6.1 Typical microbial limits vs. water activity

Report temperature and pH with a_w because limits shift with conditions. The values below are widely used guides at ambient temperatures.

Table 6. Influence of water activity (aw) on microbial survival

Product examples	Typical a_w	Microbial implication
Dried powders (flour, starch, dried vegetables, whole egg powder)	0.20–0.50	No microbial growth; even xerophiles and osmophiles cannot grow below ~0.60
High-sugar products (honey, jams)	0.55–0.75	Bacteria inhibited; osmophilic yeasts and some xerophilic molds may grow
Intermediate-moisture foods (IMF; jerky, soft cookies)	0.70–0.85	Molds and some yeasts can grow; most pathogens inhibited below 0.90; *Staphylococcus aureus* growth minimum ≈0.86
High-moisture foods (fresh meat, milk, fresh doughs)	0.97–0.99	Bacteria, yeasts, and molds can grow rapidly

Minimum a_w for growth, approximate: most Gram-negative bacteria ≥0.95; many Gram-positive bacteria ≥0.90; *S. aureus* ≈0.86; yeasts ≈0.88, osmophilic yeasts ≈0.60; molds ≈0.80, xerophilic molds ≈0.65–0.70. Absolute lower bound for microbial growth in foods is near a_w≈0.60.

4.6.2 Chemical, enzymatic, and microbial spoilage vs. a_w

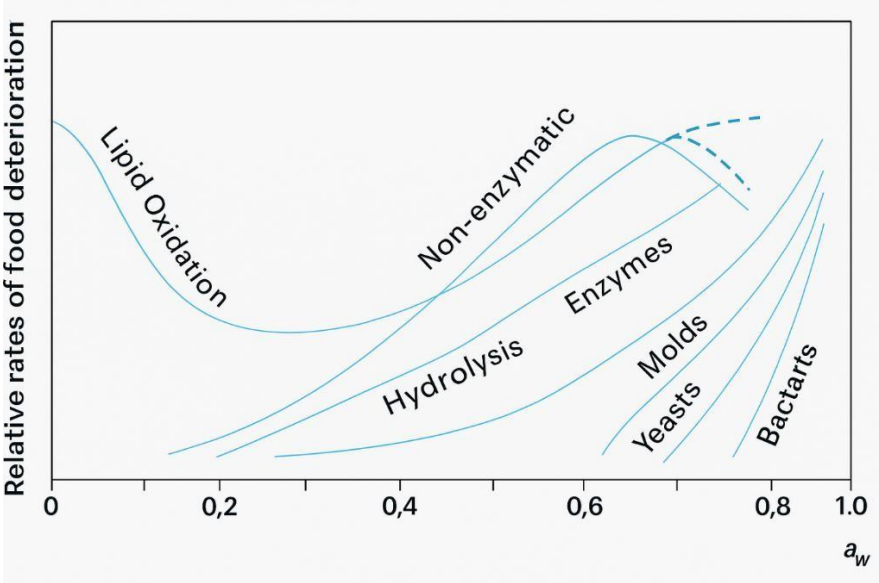


Figure 20. Principal spoilage risks as a function of water activity

1. **Lipid oxidation.** Rate is typically highest at intermediate–low a_w (≈0.2–0.4) because limited water at interfaces favors oxygen access and catalytic ion mobility; rates often decline as a_w rises further due to reduced oxygen solubility and increased radical quenching by water. Matrix composition, emulsification, metals, and antioxidants shift the peak.
2. **Non-enzymatic browning.** Maillard and related reactions show a bell-shaped dependence: slow at low a_w (<0.3–0.4) due to restricted molecular mobility, maximal around a_w≈0.5–0.7 where reactants are concentrated and mobile, then slower again at high a_w because of dilution and diffusion limits. Caramelization is primarily thermal and less governed by a_w than Maillard chemistry at comparable temperatures.
3. **Non-enzymatic hydrolysis.** Hydrolytic reactions (e.g., sucrose inversion, ester cleavage) accelerate with increasing a_w as water becomes more available as reactant; very low a_w suppresses these reactions.
4. **Enzymatic activity.** Requires sufficient unfrozen, available water for enzyme–substrate interaction. Activity generally increases with a_w up to an enzyme- and matrix-specific optimum; low a_w reduces catalysis and can inactivate some enzymes by promoting a glassy state.
5. **Microbial growth.** Probability and rate rise sharply above organism-specific minima. For safety, many shelf-stable foods are formulated at aw≤0.85 to inhibit pathogenic staphylococci and most pathogens; additional hurdles (pH, preservatives, heat treatment, packaging) are combined as needed.

4.6.3 Practical control strategies

- **Lower** a_w by drying, humectant addition (sugars, polyols, salts), or formulation changes that bind water.
- **Stabilize** a_w via moisture-barrier packaging and control of storage relative humidity (aw=ERH/100).
- **Combine hurdles**: e.g., salting and drying of fish; sugar plus low temperature in confectionery.

- **Specify and monitor** a_w at a defined temperature with calibrated instrumentation; record adsorption/desorption history due to hysteresis.

4.6.4 Typical Water Activity Limits for Microorganisms

Microbial growth depends on available water, not total moisture. Lowering a_w extends shelf life by inhibiting growth and, for some species, toxin production. Thresholds vary with temperature, pH, oxygen, preservatives, and substrate; report test conditions with a_w.

Minimum water activity for growth and typical toxin-production thresholds.

Table 7. Growth limits of microorganisms as a function of water activity

Group or species	Minimum a_w for growth	Typical a_w for toxin production	Notes
Most Gram-negative bacteria	≈ 0.95	—	Rapid growth near 0.98–0.99.
Most Gram-positive bacteria	≈ 0.90	—	Many are inhibited at ≤ 0.90.
Staphylococcus aureus	≈ 0.86	≥ 0.90–0.93 (enterotoxins)	Growth possible down to ~0.86; toxin formation needs higher a_w and favorable conditions.
Halophilic bacteria	≈ 0.75	—	Prefer high salt; non-halophiles inhibited.
Most yeasts	≈ 0.88	—	Growth slows markedly below 0.90.
Osmophilic yeasts	0.60–0.62	—	Tolerate high sugar (e.g., honey, jams).
Most filamentous molds	≈ 0.80	—	Many cease growth below ~0.80.
Xerophilic molds	0.65–0.70 (as low as ~0.61 reported)	—	Adapted to low a_w in dry foods.
Aspergillus flavus / *A. parasiticus*	≈ 0.80–0.82	≥ 0.85–0.87 (aflatoxins)	Temperature and substrate strongly affect thresholds.
Penicillium verrucosum	≈ 0.80–0.85	≥ 0.85–0.87	Important in cereals under cool

(formerly *P. cyclopium*)		(ochratoxin A)	storage.
Aspergillus ochraceus	≈ 0.77	≥ 0.85 (ochratoxin A)	Warm-climate commodities.
Penicillium expansum	≈ 0.82	≥ 0.95 (patulin)	Spoilage of apples and juices.

Notes. Values assume near-ambient temperature and permissive pH; minima shift with temperature, pH, oxygen, preservatives, and substrate.

Interpretation. Foods with $a_w \leq 0.85$ are generally hostile to pathogenic bacteria, including *S. aureus* toxin production, but yeasts and xerophilic molds may still grow above ~0.60–0.70. Use multiple hurdles (pH, preservatives, heat, packaging, low temperature) with a_w for robust stability.

4.7 Methods for Reducing Water Activity

Reducing water activity extends shelf life by limiting microbial growth and slowing moisture-dependent reactions. Interventions act by removing water or by lowering water's chemical potential. Use temperature control and packaging to maintain the achieved a_w

4.7.1 Direct methods (water removal)

- **Drying.** Evaporation removes unbound water and lowers a_w. Options include hot-air convective, vacuum, drum, spray, microwave-assisted, and infrared drying. Kinetics and final a_w depend on temperature, air velocity, humidity, particle size, and matrix state. Report end-point a_w, residual moisture, and drying profile.
- **Freeze-drying (lyophilization).** Product is frozen, then ice is removed by sublimation under vacuum. This yields very low a_w at modest temperatures with minimal thermal damage. Primary and secondary drying stages set the final a_w.
- **Freezing.** Ice formation removes liquid water from the unfrozen phase, which lowers the a_w of the unfrozen fraction. Microbial growth is effectively halted at frozen temperatures. Freezing is not a substitute for low a_w at ambient storage; on thawing, a_w returns to the prefreeze range.

- **Concentration and dewatering.** Reverse osmosis, ultrafiltration, or vacuum evaporation reduce water content before final drying, improving efficiency and enabling low a_w targets.
- **Osmotic dehydration.** Immersion in concentrated solute solutions (e.g., sucrose, NaCl) draws water out and partially replaces it with solutes, lowering a_w with mild heating.

4.7.2 Indirect methods (water potential reduction)

- **Humectants.** Low–molecular weight solutes depress a_w via colligative effects. Typical agents: sugars (sucrose, glucose, fructose), polyols (glycerol, sorbitol), salts (NaCl, KCl), and certain organic acids. Select by desired a_w, taste impact, and regulatory limits.
- **Formulation solids.** Increasing total soluble solids lowers a_w at a given moisture (e.g., jams, confectionery). In protein- or starch-rich systems, controlled gelatinization or denaturation can shift the sorption isotherm.
- **Hydrocolloids and proteins.** Gums and proteins immobilize water and slow migration. Note: high–molecular weight binders reduce mobility but have limited effect on a_w unless accompanied by added small solutes.
- **pH, preservatives, and hurdle design.** Lower a_w is combined with acidification, sorbates/benzoates, nitrites, mild heat, or modified-atmosphere packaging to raise safety margins. The same a_w can yield different stabilities depending on the other hurdles.

4.8 Determination of Moisture Content in Common Wheat Flour

Moisture content, expressed as mass percent, is determined by oven drying a test portion at 130–133 °C at atmospheric pressure after grinding if required (ISO 712). Perform two determinations on each laboratory sample and report the mean.

Principle. A known test portion is dried to constant mass under specified conditions. Loss in mass is attributed to moisture and other volatile matter defined by the method. Conditioning, drying temperature, drying time, and sample particle size follow the standard to ensure comparability.

Apparatus and conditions (summary). Air oven capable of 130–133 °C; weighing bottles with tight-fitting lids; desiccator; balance (0.001 g resolution); grinder for homogenization when necessary. Drying time follows the product category in ISO 712; for common wheat flour, the 130–133 °C reference schedule applies.

Calculation. Let

m_0 = mass of the test portion before drying (g)

m_1 = mass of the test portion after drying and cooling in a desiccator (g)

$$\text{Moisture (\% m/m)} = \frac{m_0 - m_1}{m_0} \times 100$$

Report the average of duplicate determinations and the absolute difference between duplicates; if the difference exceeds the repeatability limit in ISO 712, repeat the test. State the temperature, drying time, and whether results are expressed on a wet basis. Note that at 130–133 °C the method may include small amounts of other volatiles; this is inherent to the reference method and must be acknowledged when comparing with alternative methods.

4.9 **Standards**

Under CODEX STAN 152-1985, the maximum moisture content for soft (common) wheat flour is 15.5% m/m. Lower limits can be specified contractually for particular destinations to account for climate, transport duration, and storage period. Method references on certificates should cite the edition used (e.g., ISO 712) and the reporting basis to avoid ambiguity.

5 Ash Content of Wheat Flours

5.1 Introduction

Wheat flour, a staple in both savory and sweet applications, is produced by milling kernels of common (soft) wheat (*Triticum aestivum*). In several jurisdictions, flours are classified by "T-type," which denotes mineral residue (ash) per 100 g of dry matter measured after controlled incineration. Higher T-values indicate greater inclusion of outer kernel tissues and, therefore, higher ash content.

T110 is a semi-wholemeal flour in which some bran fractions have been removed. T150 is wholemeal (integral) flour that retains virtually all bran layers and the germ. In general, the higher the T-type, the higher the mineral content.

Whole-wheat flours contain markedly more minerals than refined white flours because the bran and germ are preserved. These fractions are nutrient-dense and are largely removed during refining, which lowers ash, fiber, vitamin, and lipid contents while increasing the proportion of starchy endosperm.

5.2 Wheat seed structure

The wheat grain is ovoid and exhibits a longitudinal ventral furrow that runs along its entire length (Figure 21). It is obtained after threshing, that is, once the chaff enveloping the grain has been removed.

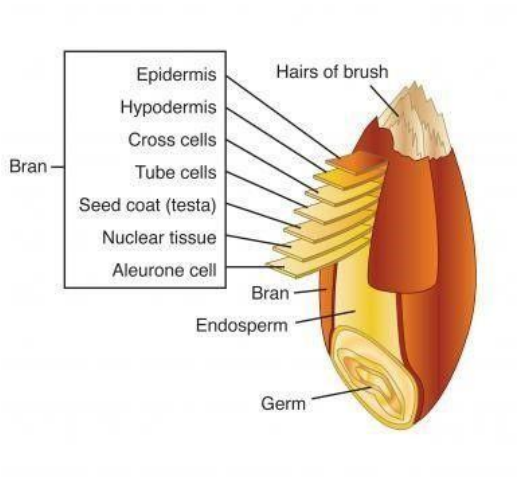


Figure 21. Structure of the wheat seed

At the dorsal base lies the germ, surmounted by a small "brush." Wheat grains typically measure 5–7 mm in length and 2.5–3.5 mm in thickness, with a single-kernel mass of approximately 20–50 mg. Grain color ranges from red to white, depending on genotype and growing conditions, including soil, agronomic practices, and climate. The anatomy of the wheat grain is summarized in Figure 22. The grain comprises three major components: the outer envelope (bran), the starchy endosperm, and the germ.

5.2.1 Envelope

The husk (bran) represents roughly 13–17% of grain mass in common bread wheat (the often-cited 20% is an overestimate for many modern cultivars). It consists of several layers observable from the outside inward:

- Pericarp: the outer envelope formed by multiple layers of thick-walled, lignified cells.
- Seminal integument (testa) and hyaline layer: the testa contains pigments responsible for red or white coloration; the hyaline layer appears translucent under the microscope.
- Aleurone (protein-rich base): composed of cells with thinner walls than the pericarp and less lignification; it is rich in proteins, minerals, enzymes, and B vitamins and is included with the bran in conventional milling.

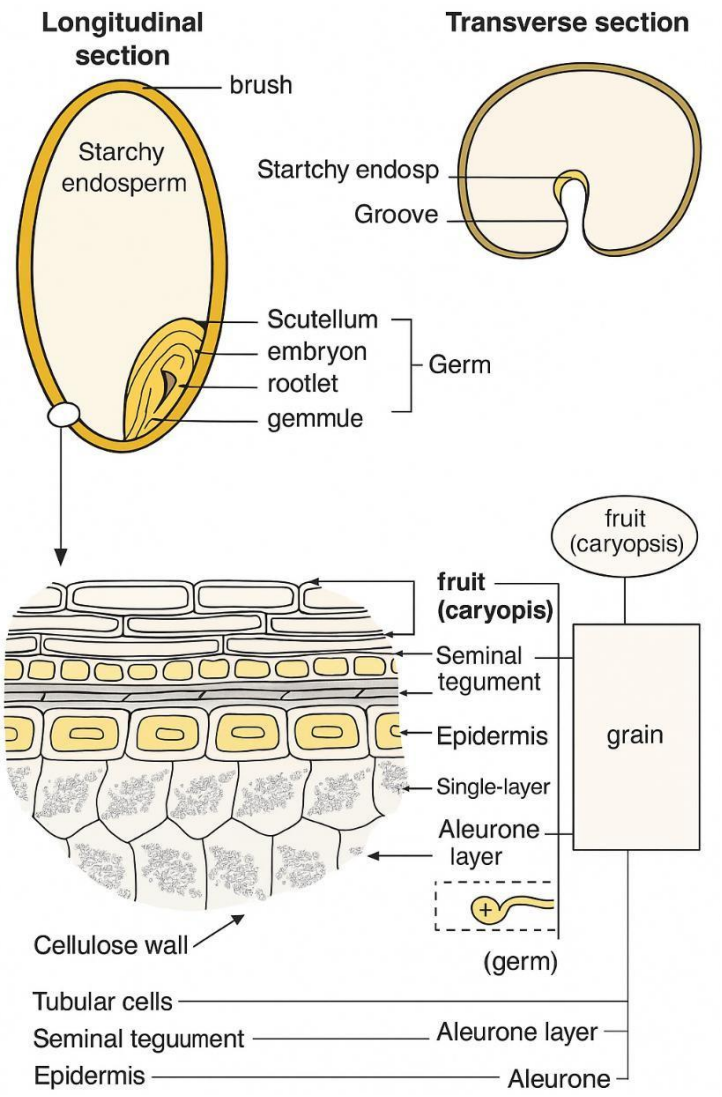


Figure 22. Anatomy of the soft wheat grain

5.2.2 Floury almond (endosperm)

Also called the endosperm, this fraction represents the major part of the grain, about 77–80% of its mass, and is bounded basally by the germ. It consists of parenchyma cells filled with starch granules embedded in a gluten-forming protein matrix (Figure 20). Upon hydration and mixing, gliadins and glutenins develop a viscoelastic network that yields an elastic dough. From the periphery toward the center of the endosperm, the number and distribution of starch granules increase and vary, influencing milling behavior and flour functionality.

5.2.3 Germ

The germ constitutes about 2–3% of the grain mass (Figure 20). It is the embryonic plant and is rich in lipids, sugars, and vitamins, notably vitamins B and E. Its inclusion increases ash and lipid contents but can reduce storage stability because of endogenous lipase and lipoxygenase activities.

5.3 Biochemical Composition of Wheat Grain

Wheat grain is a composite biological material comprising macronutrients and micronutrients distributed heterogeneously across the pericarp, aleurone layer, endosperm, and germ. For clarity, constituents are grouped into primary (major) and secondary (minor) components. Quantitative values vary with cultivar, environment, and moisture basis; typical ranges are provided from standard cereal chemistry sources.

5.3.1 Primary Constituents

5.3.1.1 Carbohydrates

Carbohydrates are the predominant constituents of wheat, consisting mainly of starch with smaller fractions of simple sugars and non-starch polysaccharides. Starch accounts for roughly 60–70% of kernel mass in common bread wheat (Triticum aestivum), while simple sugars such as glucose, fructose, and sucrose occur at low levels and support early germination and fermentation processes. Structural polysaccharides, including cellulose and arabinoxylans, are concentrated in the cell walls of the pericarp and aleurone; they are poorly digestible by monogastric species and contribute to dietary fiber in human nutrition.

5.3.1.2 Starch (reserve polysaccharide)

Starch is the principal energy reserve of the endosperm. It occurs as discrete granules of characteristic size populations and morphology embedded in a protein matrix. Native wheat starch typically comprises 20–30% amylose and 70–80% amylopectin. Its techno-functional attributes—gelatinization, pasting, retrogradation, and water binding—govern dough viscosity,

crumb structure, and product staling dynamics. In most hard wheats, total starch content ranges from approximately 60% to 72% of kernel mass.

5.3.1.3 Proteins

Wheat proteins occur as albumins, globulins, and, uniquely, the prolamin–glutelin system that forms gluten. Total crude protein in wheat grain typically averages 10–15%, compared with ~35–40% in soybean, ~10–12% in barley, and ~7–10% in maize. Amino-acid balance is limiting in lysine relative to animal requirements and FAO reference patterns, which has implications for complementary protein formulation in feeds and fortified foods.

5.3.1.4 Gluten (viscoelastic protein network)

Gluten is the hydrated, viscoelastic network obtained after washing wheat dough to remove soluble components and starch. On a dry basis, commercial vital gluten contains ~75–85% protein, with residual starch (~5–10%), lipids (~5–10%), reducing sugars (~1–2%), and mineral ash (~1%). The network arises from intermolecular bonding among gliadins and glutenins; gliadins impart viscosity and extensibility, whereas high- and low-molecular-weight glutenin subunits provide elasticity and strength. These properties underpin gas retention during proofing and define breadmaking quality.

5.3.1.5 Lipids

Wheat lipids are minor constituents at ~1.5–2.5% of kernel mass, concentrated in the germ and outer layers. Non-polar triacylglycerols predominate in the endosperm, whereas polar lipids (galactolipids and phospholipids) and free fatty acids are enriched in the germ and bran. Fatty-acid profiles are dominated by unsaturated species, chiefly linoleic (C18:2) and oleic (C18:1) acids, with lower proportions of palmitic (C16:0). These lipids influence starch–lipid complexing, dough rheology, crumb softness, and oxidative stability during storage.

5.3.2 Secondary Constituents

5.3.2.1 Pigments and Vitamins

Carotenoids (e.g., lutein) are the principal native pigments in wheat and are localized mainly in the endosperm and aleurone, imparting a creamy to yellow hue. Phenolic compounds in the bran contribute to color and antioxidant capacity. The germ and outer layers are also the major repositories of B-group vitamins (e.g., thiamin, riboflavin, niacin, folate) and vitamin E (tocopherols). Milling extraction rate therefore strongly affects micronutrient density of flours.

5.3.2.2 Enzymes

Cereal enzymes occur at low concentrations but have high technological relevance.

- **Amylases.** α-Amylase endo-hydrolyzes α-1,4 linkages to dextrins, while β-amylase exo-hydrolyzes non-reducing ends to maltose. Endogenous activities modulate fermentable sugar supply and crumb structure; excessive α-amylase (e.g., from preharvest sprouting) leads to sticky crumb and low loaf volume.
- **Proteases.** Endogenous proteases cleave storage proteins, affecting dough development, mixing tolerance, and extensibility. Increased proteolysis during germination softens doughs and can impair breadmaking quality.
- **Lipases and Lipoxygenase.** Lipases hydrolyze triacylglycerols to free fatty acids and glycerol, accelerating rancidity if bran or germ is present. Lipoxygenase catalyzes oxidation of polyunsaturated fatty acids and can bleach carotenoids, affecting flour and crumb color. Managing bran inclusion, moisture, and storage temperature limits off-flavor formation.

5.3.2.3 Water

Moisture content is a critical quality and safety parameter. For safe storage and to limit microbial growth and enzymatic spoilage, grain moisture is typically controlled to ≤13% at temperate conditions, with adjustments for temperature and storage duration. In processing, water acts as

solvent, plasticizer, and reactant, activating enzymes and enabling gluten development and starch gelatinization.

5.4 Classification of Common Wheat Flours

Ash content quantifies total mineral residue and serves as a proxy for degree of refinement. Higher ash indicates higher extraction rate and greater retention of bran- and germ-derived minerals and fiber. Lower ash indicates a whiter, more refined flour with reduced bran.

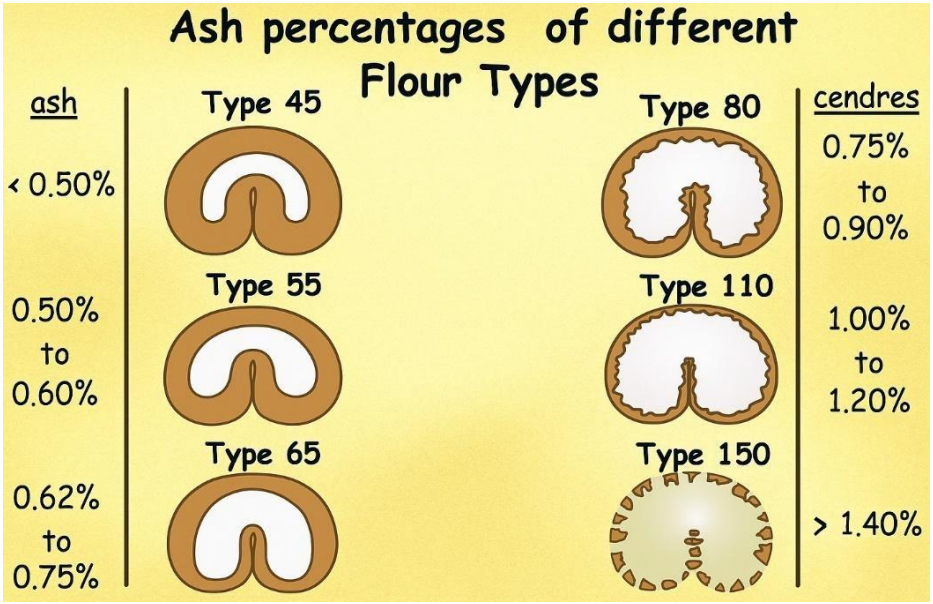


Figure 23. Ash content of selected wheat flour types (“T” classification)

The French “Type” designation (T) refers to ash mass per 100 g of flour. The numeral after “T” approximates the ash percentage multiplied by 100.

5.4.1 Type 45 (T45) — Very low ash

Maximum ash ≈ 0.50%. Ultra-white, very fine particle size. Typical use: fine pastries and cakes where low bran particles and low enzyme load favor light color and tender crumb.

5.4.2 Type 55 (T55) — Low ash

Maximum ash ≈ 0.65%. White flour with slightly higher mineral content than T45. Typical use: laminated viennoiserie, general pastry, and many “all-purpose” applications.

5.4.3 Type 65 (T65) — Moderately low ash

Ash ≈ 0.62–0.75%. Whiter bread flour with limited outer-layer material. Suited to crusty bread styles requiring stronger doughs and modest flavor from bran-associated compounds.

5.4.4 Type 80 (T80) — Semi-wholemeal (light)

Ash ≈ 0.75–0.90%. Less refined than T65. Balances nutritional density with sensory qualities; increased fiber and micronutrients with acceptable loaf volume and color.

5.4.5 Type 110 (T110) — Semi-wholemeal (dark)

Ash ≈ 1.00–1.20%. Enriched in bran and germ fractions. Higher fiber and micronutrient content (proteins, minerals, B-vitamins, tocopherols) relative to T65–T80, with darker crumb and more pronounced cereal flavor.

5.4.6 Type 150 (T150) — Wholemeal

Ash ≈ ≥ 1.50% per 100 g of flour. Wholemeal flour retaining all anatomical parts of the grain (bran, endosperm, germ). Highest fiber and mineral density, darker color, robust flavor. Preferred for rustic breads and nutrient-dense formulations.

5.5 Importance of Ash Content in Wheat Flour

Wheat grain is a caryopsis in which the pericarp (fruit coat) is fused to the seed coat, collectively forming the bran. The kernel comprises the starchy endosperm, a single-cell aleurone layer surrounding the endosperm, and the germ. Commercial flour is milled primarily from the starchy

endosperm; milling aims to maximize endosperm recovery while minimizing contamination by bran and germ, which concentrate minerals, enzymes, and phenolics.

Mineral matter is unevenly distributed across anatomical parts. The endosperm constitutes ~80–85% of grain mass but contains a minority of total minerals; the bran fractions (pericarp, testa, aleurone) and germ contain the majority. Ash content—the mass of inorganic residue after ashing at high temperature—therefore serves as a sensitive proxy for the extraction rate and the extent of bran and germ inclusion in flour (ISO/ICC methods typically use ~900 °C for cereals).

Ash content functions first as an indicator of refinement. Lower-ash flours have reduced bran carryover and appear whiter with fewer endogenous pigments and phenolics. Higher-ash flours contain more bran- and germ-derived material and thus higher levels of minerals and dietary fiber; they also carry more endogenous enzymes and phenolics that influence color and flavor.

Ash content also correlates with nutritional density. As ash rises, concentrations of minerals (e.g., Fe, Zn, Mg, P), B-group vitamins, tocopherols, and fiber generally increase because these nutrients are enriched in the aleurone, bran, and germ. Conversely, very low-ash flours have improved visual brightness and a finer mouthfeel but lower micronutrient and fiber levels on an equal-mass basis.

Technologically, ash impacts flour functionality. Higher ash is associated with darker flour color (lower L*), greater water absorption, stronger buffering capacity, and altered dough rheology due to bran particles and associated non-starch polysaccharides. These effects can reduce loaf volume and yield denser crumb, which is desirable in certain rustic and wholegrain products but detrimental for fine pastry. Lower-ash flours favor light-colored crumb, tender textures, and delicate layers in cakes and laminated products.

Culinary use follows from these relationships. Low-ash flours are selected for pastry and fine baked goods where color, tenderness, and extensibility are prioritized. Medium-ash bread flours balance machinability and volume with flavor. High-ash (semi-wholemeal to wholemeal) flours emphasize flavor, fiber, and micronutrients, producing darker, denser breads suited to wholegrain formulations.

5.6 Wholemeal Flour

Wholemeal flour is milled from the entire cereal kernel (e.g., wheat or rye) and retains all anatomical parts: bran (pericarp + seed coats, including the aleurone), endosperm, and germ. Relative to refined white flour, wholemeal flour delivers higher levels of dietary fiber, minerals (e.g., Fe, Zn, Mg, P), and B-group vitamins and tocopherols. Functional properties differ because bran particles, endogenous enzymes, and phenolics remain present.

Table 8. Typical composition (whole wheat flour, per 100 g)*

Nutrient	Typical value
Energy (kcal)	~330–350
Water (g)	~10–13
Protein (g)	~11–14
Carbohydrate (g)	~68–73
of which starch (g)	~55–62
Dietary fiber (g)	~9–13
Iron (mg)	~2–4
Magnesium (mg)	~80–150

*Values depend on cultivar, extraction rate, and moisture basis; use local analytical data for labeling and formulation.

5.6.1 Cardiometabolic relevance

Higher-extraction flours carry more fiber and phytochemicals. Viscous and fermentable fiber fractions increase fecal bile acid loss, modulate postprandial glycemia, and yield short-chain fatty acids that may influence lipid metabolism. In controlled diets and cohort studies, whole-

grain intake is associated with improved blood lipid profiles and lower risk of cardiovascular disease; flour ash and fiber rise together as extraction increases.

5.6.2 Iron: content and bioavailability

Wholemeal flour contains more total iron than refined flour, but most is non-heme and bound within bran matrices, reducing fractional absorption. Co-ingestion of vitamin C–rich ingredients enhances non-heme iron uptake. Heme iron from meat exhibits higher bioavailability; therefore, equal milligram amounts do not imply equal physiological iron gain. Use phytate-reduction strategies (preferments, sourdough acidification, enzymatic phytase) to improve bioaccessibility in wholegrain products.

5.6.3 Colorectal health

Higher dietary fiber from wholegrain flours increases stool bulk, dilutes luminal carcinogens, and produces butyrate via colonic fermentation. Observational evidence links wholegrain intake with lower colorectal cancer risk. Mechanisms are consistent with fiber physiology, but product-specific effects depend on particle size, fiber type, and processing.

5.6.4 B-vitamins and magnesium

The aleurone and germ supply thiamin, riboflavin, niacin, folate, and magnesium, which are depleted during refining. These micronutrients support energy metabolism, erythropoiesis, and neuromuscular function. Milling extraction rate and subsequent processing (e.g., heat, oxidation) determine retained levels.

5.7 Determination of Ash in Wheat Flour

Ash is the inorganic, non-combustible residue remaining after incineration of flour under controlled conditions. For wheat flours classified by “Type T,” ash is determined at high temperature, typically 900 ± 25 °C, and reported as g per 100 g flour (wet basis) and, when needed, on a dry-matter basis.

Figure 24. Steps for determining the ash content in soft wheat flour.

5.7.1 Principle

A known mass of flour is incinerated in a pre-weighed crucible in a muffle furnace until organic matter is destroyed. The crucible is cooled in a desiccator and re-weighed. Moisture content is determined separately to allow dry-basis expression.

5.7.2 Apparatus and reagents

Porcelain or silica crucibles; drying oven; muffle furnace capable of 900 ± 25 °C; desiccator with fresh desiccant; analytical balance (±0.1 mg); heat-resistant tongs.

5.7.3 Sample preparation

Mix the test flour thoroughly. If needed, pre-dry grossly moist samples gently (<60 °C) to prevent spattering; record any pre-drying loss separately (not typical for commercial flours).

5.7.4 Procedure

- **Prepare crucibles.** Ignite empty crucibles at 900 °C for 30 min; cool in a desiccator to room temperature; weigh (m_0).
- **Weigh sample.** Accurately add ~5.000 g flour to each crucible; record total (m_1 = m_0 + sample).

- **Char.** Place crucibles in a cool furnace. Open door slightly to allow smoke to escape. Raise temperature gradually to avoid flaming; once smoking ceases, close the door.
- **Ash.** Incinerate at 900 ± 25 °C for 90–120 min, or to constant mass (difference <0.5 mg upon re-ashing 15 min). Ash should be light gray to white.
- **Cool and weigh.** Transfer crucibles to a desiccator for ≥45 min; weigh (m_2 = m_0 + ash).
- **Moisture.** Determine flour moisture (H, %) on a separate test portion using an approved oven method (e.g., 130 ± 1 °C for 60 min or an equivalent standard).

5.7.5 Calculations

- **Ash mass (g):** $m_{ash}=m_2-m_0$
- **Sample mass (g):** $m_{sample}=m_1-m_0$
- **Ash, wet basis (% of flour as received):**

$$\mathrm{Ash}_{wb}\ (\%) = \frac{m_{\mathrm{ash}}}{m_{\mathrm{sample}}} \times 100$$

- **Ash, dry basis (% of dry matter):**

$$\mathrm{Ash}_{db}\ (\%) = \frac{m_{\mathrm{ash}}}{m_{\mathrm{sample}}\,(1-H/100)} \times 100$$

- **Reporting:** Refined flours to two decimals (e.g., 0.55%); wholemeal often to one or two decimals per method.

6 Determination of Reducing Sugars in Fruit Juices

6.1 Introduction

Carbohydrates are the most abundant class of biomolecules and perform structural, energetic, and signaling functions in living systems. As major constituents of foods, they occur as soluble mono- and oligosaccharides and as insoluble polysaccharides. Insoluble polymers such as cellulose and hemicelluloses form the plant cell wall. Glycosaminoglycans and glycoconjugates contribute to extracellular matrices, joint lubrication, and cell–cell recognition. Structure and conformation determine function, so precise nomenclature and classification are essential in food analysis.

The term carbohydrate includes reducing sugars and compounds that yield reducing sugars on hydrolysis. In plants, photosynthesis produces carbohydrates from carbon dioxide and water. Glucose is the most abundant monomer and serves as the precursor of the storage and structural polymers starch, glycogen, and cellulose.

A reducing sugar is a carbohydrate that can donate electrons from a free carbonyl in its open-chain form. This includes aldoses and ketoses that tautomerize under assay conditions. All monosaccharides are reducing. Among disaccharides, maltose, lactose, and cellobiose are reducing because their anomeric carbons are not both engaged in the glycosidic bond. Sucrose and raffinose are non-reducing because the glycosidic linkage involves the anomeric carbons and prevents ring opening until hydrolysis. The reducing activity of sugars under alkaline conditions is widely used for quantification in food analysis by reduction of Cu(II) to Cu(I), as in Fehling's solution, with the formation of cuprous oxide.

Dietary sources rich in reducing sugars include fruits, honey, certain vegetables, milk and dairy products that contain lactose, confectionery, and fruit juices.

6.2 Fruit Juices

Under the Codex standard (CODEX STAN 247-2005), fruit juice is the unfermented yet fermentable liquid obtained from the edible portion of sound, ripe fruit, whether fresh or preserved under hygienic conditions, and produced using operations consistent with Good Manufacturing Practices. Post-harvest surface treatments must comply with Codex provisions.

Juice may be expressed from fruit that contains seeds, stones, and peels, which are typically excluded. However, small amounts of these components that cannot be removed by Good Manufacturing Practices are acceptable. Processing should preserve the essential physical, chemical, sensory, and nutritional attributes of the source fruit. Juice may be cloudy or clarified. Volatile aroma fractions and flavor compounds recovered from the same fruit species may be restored by suitable physical means. Pulp and cells from the same fruit, obtained by appropriate physical processes, may be added.

A single-strength juice is derived from one fruit species. A blended juice is produced by mixing two or more juices or juices with fruit purées from different fruit species, provided all components comply with the same compositional and processing requirements.

6.3 Types of Fruit Juices

The fruit-juice category comprises three principal products defined by composition and process: fruit juice (direct expression), fruit juice from concentrate (reconstituted), and fruit nectar. All originate from fruit but differ in permitted ingredients, minimum fruit content, and processing steps (Codex Alimentarius Commission, 2005; European Parliament & Council, 2012).

6.3.1 Fruit juice (direct expression)

Fruit juice is obtained by mechanical processes such as pressing or extraction from the edible portions of sound, ripe fruit. No sugars, sweeteners, or additives that increase °Brix are permitted. Pulp and cells from the same fruit species may be restored. The product may be

cloudy or clarified, and aromas recovered from the same fruit may be added back by physical means. (Codex STAN 247-2005).

6.3.2 Fruit juice from concentrate (reconstituted)

Fruit juice from concentrate is produced by reconstituting concentrated fruit juice with potable water to the original soluble-solids content and organoleptic profile of single-strength juice. It remains a 100% fruit product with no added sugars or sweeteners. Volatile aromas captured during concentration may be restored, and pulp/cells from the same fruit species may be added. (Codex STAN 247-2005).

6.3.3 Fruit nectar

Fruit nectar is produced from fruit juice and/or fruit juice from concentrate and/or fruit purée, diluted with water. Sugars and/or permitted sweeteners may be added within regulatory limits to achieve palatability, particularly for pulpy or high-acidity fruits. Minimum fruit content is regulated by fruit type, typically 25–50% by volume. Examples include nectars from banana, apricot, peach, pear, and various acidic "red fruits." Orange or apple "nectars" exist as lower-cost products with reduced fruit content and added water and sweetening agents. (Codex STAN 247-2005).

6.3.4 Smoothies

"Smoothie" is a commercial rather than a universal regulatory term. Classification depends on composition and jurisdiction. Where a smoothie contains only fruit juice and/or fruit purée from the same fruit species, some authorities classify it within fruit-juice or nectar standards; the presence of added dairy, protein, or other non-fruit ingredients places it outside the fruit-juice standards. Always apply the local standard to determine the legal name. (Codex STAN 247-2005; European Parliament & Council, 2012).

6.4 **Labelling of Fruit Juices**

Labels must state the legal product name—"fruit juice," "fruit juice from concentrate," or "fruit nectar"—together with the name and address of the producer/packer**,** net quantity, date marking, and ingredient list in descending order by weight for blends. For nectars, the minimum fruit content must be declared. If sugars and/or sweeteners are added, the label must indicate this in accordance with applicable regulations. For fruit juice and fruit juice from concentrate, the addition of sugars is not permitted. (Codex STAN 247-2005; European Parliament & Council, 2012).

Table 9. Regulatory labelling and compositional allowances

	Fruit juice	**Fruit juice from concentrate**	**Fruit nectar**
Fruit content (v/v)	100%	100%	25–50% minimum*
Vitamins and minerals	Allowed	Allowed	Allowed
Pulp/cells (same fruit species)	Allowed	Allowed	Allowed
Lemon juice for acidification	Allowed	Allowed	Allowed
Added sugars	Not allowed	Not allowed	Allowed**
Preservatives and colourings	Not allowed	Not allowed	Not allowed***

* Exact minima depend on fruit type per standard.

** Within limits and with required declarations where permitted by local law.

*** Where preservation is needed, non-thermal processes or heat treatment are used; if additives are permitted locally, they must be declared and remain within standard limits.

Products that do not meet the compositional or process definitions cannot be labeled "fruit juice" or "fruit nectar." They must bear a generic name such as "fruit-flavoured beverage" or "fruit juice–based drink," supplemented by an accurate descriptor.

6.5 Juice Production Line

Industrial fruit-juice manufacture comprises raw-material preparation, juice extraction, clarification or stabilization, thermal processing, optional concentration, and packaging. Unit operations are selected to maximize yield and quality while ensuring microbiological safety and regulatory compliance. A generalized line includes washing and sorting, size reduction and maceration, pressing or other extraction, clarification or stabilization, pasteurization, optional concentration under vacuum with aroma recovery, cooling, and filling (Figure 25).

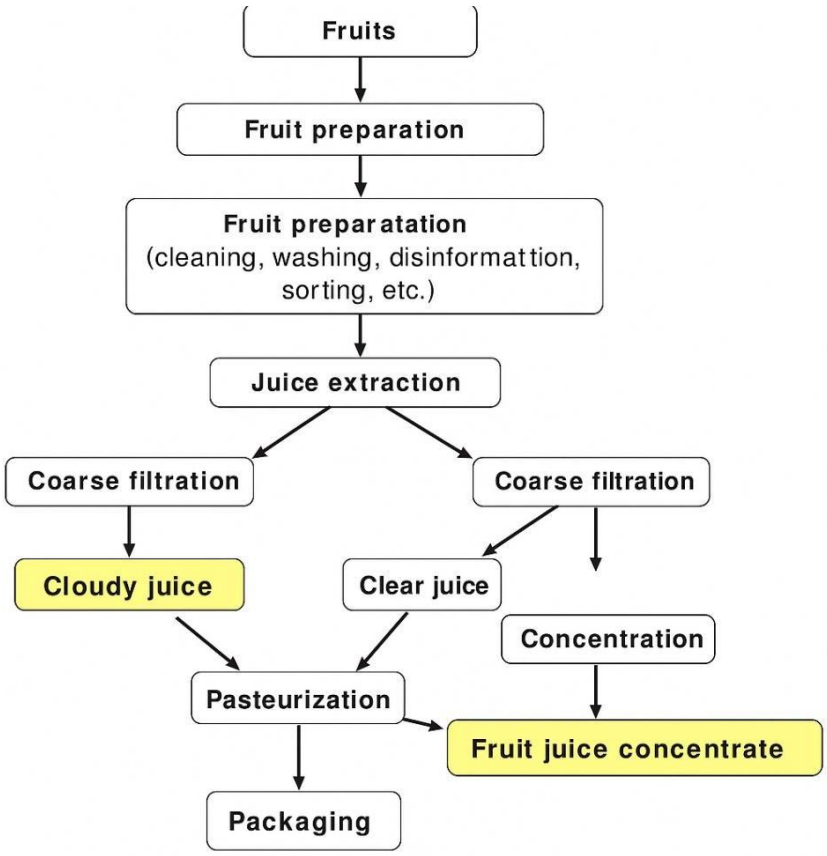


Figure 25. Schematic of a fruit-juice production line

6.5.1 Preparation of fruit

Preparation adapts fruit to processing while minimizing quality loss and contamination. The sequence varies by species and process design.

6.5.1.1 Sorting and grading

Fruit is sorted for soundness, maturity, and color, which strongly influence soluble-solids content, acidity, flavor, and yield. Defective fruit and extraneous matter are removed to protect product quality and equipment.

6.5.1.2 Washing

Washing removes soil, stones, leaves, insects, and residues of field sprays. Counter-current spray systems, flumes with agitation, and spray-plus-soak combinations are common. Potable water is mandatory; where legally permitted, sanitizers may be used at validated concentrations. Effective washing reduces initial microbial load and foreign material that would otherwise affect flavor and clarification.

6.5.2 Extraction

Extraction separates juice from comminuted fruit tissues.

6.5.2.1 Comminution (grinding/milling)

Fruits are cut or milled to form a mash that releases intracellular juice. Equipment selection depends on fruit morphology. For pome fruits and tomatoes, controlled comminution that preserves seed integrity is preferred to limit release of bitter or astringent compounds. Mash heating or enzymatic maceration may be applied prior to pressing to improve flow and yield.

6.5.2.2 Pressing and alternative extraction

Pressing is the predominant extraction method for many juices. After conditioning, mash is expressed using hydraulic, pneumatic, belt, or screw presses designed to achieve high yield while limiting oxygen uptake and turbidity. Good practice includes using permeable press cloths or screens that retain solids, applying uniform pressure on rigid surfaces, draining free-run juice before pressure increase, and periodically loosening or re-spreading the cake to sustain

permeability. Alternatives or complements include centrifugation for citrus or tropical purées, as well as diffusion for specific applications.

6.5.3 Refining (finishing)

Refining—often termed finishing—separates seeds, peel fragments, and coarse pulp from the expressed juice. Centrifugal finishers or vibrating screens with defined aperture sizes are used. In some processes, mild heating lowers viscosity and facilitates separation; extensive polysaccharide hydrolysis is avoided when viscosity retention is desired for mouthfeel.

6.5.4 Juice processing: Clarification and stabilization

Clarification provides the degree of transparency required by product specifications. Enzymatic depectinization with pectinolytic preparations reduces viscosity and breaks colloidal stability, enabling subsequent solid–liquid separation by decantation, centrifugation, fining, or filtration. Amylolytic or proteolytic enzymes may be applied where starch or haze-forming proteins are present. Fining agents and filters (e.g., bentonite, PVPP, diatomaceous-earth filtration) are selected according to legal and label constraints. For juices marketed as "cloudy," stabilization focuses on controlling phase separation without full clarification.

6.5.5 Pasteurization

Pasteurization inactivates enzymes and destroys vegetative microorganisms to achieve the intended shelf life and safety targets. High-temperature short-time regimes are validated per product type, °Brix, pH, and packaging. Typical juice processes heat rapidly to a target in the 80–95 °C range for seconds to tens of seconds, followed by rapid cooling to minimize quality loss. Lower targets may suffice for high-acid juices when combined with hot-fill, whereas higher targets or longer holding may be required for more viscous products. Time–temperature combinations are established by hazard analysis and thermal validation rather than a single fixed recipe.

6.5.6 Concentration

Concentration removes water to reduce storage and transport costs. Falling-film or plate evaporators operating under vacuum limit thermal damage by enabling evaporation at reduced temperatures, often 40–65 °C. Aroma recovery systems capture stripped volatiles for later restoration during reconstitution. Target concentration is usually expressed as °Brix. Process residence times and temperatures are optimized to balance flavor retention, color stability, and energy efficiency.

6.5.7 Cooling and packaging

Post-process handling aligns with the preservation strategy.

- **Sterile (hot-fill) packaging.** Immediately after pasteurization, high-acid juices are filled at elevated temperature into containers that tolerate hot-fill. Containers are preheated where necessary. Closure occurs promptly, followed by inversion or equivalent treatment to sanitize the headspace and closure.
- **Aseptic packaging.** Juice undergoes validated thermal treatment and is cooled and filled under aseptic conditions into pre-sterilized containers. Product and container sterility are maintained without reliance on post-fill heat treatment.
- **Bulk storage under inert gas.** Pasteurized juices intended for later packaging can be held in aseptic or hygienic tanks under nitrogen or carbon dioxide to limit oxidation. If stored non-aseptically, repasteurization prior to final packaging is required; if stored aseptically, no repasteurization is performed, provided sterility is maintained and validated.

6.5.8 Heat and enzymatic treatment of the mash or juice

6.5.8.1 Heat treatment effects

Controlled heating during maceration softens tissues, promotes pectin dehydration and gelation, reduces elasticity of cell walls, and thereby enhances juice release. The specific time–

temperature profile is tailored to the fruit species, cultivar, maturity, and desired rheology and flavor outcomes.

6.5.8.2 Enzymatic maceration

Pectinolytic enzyme preparations increase yield, reduce viscosity, and improve pressability, particularly for pectin-rich fruits. Dosage, temperature, pH, and contact time are optimized for the enzyme specification and fruit matrix. Where clarity is a target, depectinization is carried through to completion; where a natural cloud is desired, partial treatment may be chosen to balance stability and mouthfeel.

6.6 Composition of Fruit Juices under Codex Standards

Fruit-juice composition is governed primarily by the *General Standard for Fruit Juices and Nectars* (CODEX STAN 247-2005). References to sugars use definitions from the *Standard for Sugars* (CODEX STAN 212-1999). The points below harmonize both. Use APA 7th style for citations.

6.6.1 Basic ingredients

For directly expressed fruit juice, the °Brix is that of the juice as obtained from the fruit. Soluble-solids content must not be altered, except by blending with juice from the same fruit species to manage natural variability. Restored aroma, pulp, and cells must originate from the same fruit species and be added by suitable physical means. (Codex STAN 247-2005).

For fruit juice from concentrate, reconstitution with potable water must restore the product to the composition and organoleptic profile of single-strength juice, meeting the Codex minimum species-specific Brix values. Calculation excludes solids from any optional ingredients. (Codex STAN 247-2005).

For reconstituted juices and nectars, the water used must be potable and at minimum conform to the latest WHO Guidelines for Drinking-water Quality. (Codex STAN 247-2005; WHO, latest ed.).

6.6.2 Other permitted ingredients and constraints

Sugars and sweeteners**.** Under current Codex and many national frameworks, added sugars and sweeteners are not permitted in fruit juice or fruit juice from concentrate. In fruit nectar, sugars and/or permitted sweeteners may be used within regulatory limits, and must be declared in the ingredient list. When sugars are used in foods covered by Codex sugar definitions, they follow CODEX STAN 212-1999 terminology (e.g., sucrose, glucose [dextrose], fructose) and specifications. (Codex STAN 247-2005; Codex STAN 212-1999).

Acidification. Where authorized by the importing country's legislation, lemon and/or lime juice may be used for acidification. Limits are expressed as anhydrous citric-acid equivalents and must comply with local law. Declaring such additions is mandatory where required. (Codex STAN 247-2005).

Botanical purity. Single-species juices must derive exclusively from the named fruit. Incorporation of other species' juices is not permitted unless expressly provided in the relevant standard and declared in the legal name. In practice, adding mandarin (Citrus *reticulata*) to orange juice is not allowed under Codex for a product labeled "orange juice" unless a specific allowance exists in the competent jurisdiction; otherwise the product must be labeled as a blend. *(Codex STAN 247-2005).*

Tomato juice seasonings. For tomato juice, salt, spices, and aromatic herbs (and their natural extracts) may be added, subject to national limits and labeling. (Codex STAN 247-2005).

Vitamins and minerals. Nutrient fortification for restoration or enrichment may be allowed where national legislation permits. Added nutrients must be approved, present at authorized levels, and declared. (Codex STAN 247-2005).

Simultaneous sugar and acid addition. In systems where sugar addition is permitted only for nectars, combining acidifying agents and sugars must follow national rules. For fruit juice and fruit juice from concentrate, this point is moot because sugars are not permitted. (Codex STAN 247-2005).

Ingredient naming and specifications. When ingredients such as sugars are allowed in a given product category, they must meet Codex identity and purity criteria (e.g., moisture content for crystalline sugars per CODEX STAN 212-1999) and be declared under ingredient-listing provisions. (Codex STAN 212-1999).

6.7 Determination of Reducing Sugars in Industrial Juices

6.7.1 Definition

Reducing sugars are carbohydrates that possess a free or potentially free carbonyl group capable of electron donation in alkaline media. In aqueous solution many aldoses and ketoses interconvert to an open-chain form with an accessible carbonyl at the anomeric carbon. Under alkaline, heated conditions these sugars reduce metal ions such as Cu(II) to Cu(I). This redox property underpins classical analytical tests, including Fehling's and Benedict's reagents, where cuprous oxide forms a brick-red precipitate. In fruit-juice analysis, results are expressed as glucose equivalents unless otherwise specified.

6.7.2 Quantification by the Fehling (Lane–Eynon) Method

6.7.2.1 Principle

A fixed aliquot of Fehling's solution—an alkaline cupric tartrate reagent—is titrated at boiling with a sugar solution until complete reduction of Cu(II) to Cu(I). The endpoint is detected by a color change of the supernatant or by an auxiliary indicator system. Prior calibration with a standard glucose solution establishes the factor that relates delivered volume to mass of glucose. The same thermal profile, timing, and reagent volumes are used for both standard and sample to ensure comparability.

6.7.2.2 Reagents and apparatus

Fehling's solution is prepared as separate solutions and mixed immediately before use:

- **Fehling A:** $CuSO_4 \cdot 5H_2O$ solution of defined molarity.
- **Fehling B:** Alkaline potassium sodium tartrate (Rochelle salt) in NaOH.

- **Standard glucose solution:** 5.00 g/L anhydrous D-glucose, freshly prepared.
- **Optional endpoint systems:**
    - Causse–Bonnans modification**:** add 5 mL of 5% K_4 [$Fe(CN)_6$] to form soluble cuprous ferrocyanide; the supernatant shifts from yellow to brown at the endpoint.
    - Alternative visual indicators (e.g., methylene blue) may be used where validated.
- **Boiling water bath or controlled hotplate**, **Erlenmeyer flasks (100 mL)**, **burette**, and **inert boiling aids** (pumice or anti-bumping granules). Use CO_2 -free distilled water for dilutions.

6.7.2.3 Calibration of Fehling's liquor

Pipette 10.0 mL Fehling A and 10.0 mL Fehling B into a 100 mL Erlenmeyer. Add two pumice grains. Bring to a gentle boil. From a burette deliver the 5.00 g/L glucose standard dropwise into the boiling reagent with continuous boiling. Allow transient cuprous oxide to settle between additions. The endpoint is reached at persistent decolorization of the supernatant; with the Causse–Bonnans modification, take the stable transition from yellow to brown as the endpoint. Record the delivered volume V_1 (mL). This defines the Fehling factor under the chosen conditions.

6.7.2.4 Preliminary test on sample

Prepare the test solution **S** from the juice. Filter or clarify as needed. If necessary, dilute so that its titration endpoint volume is close to V_1 to minimize differential errors from heating and timing.

6.7.2.5 Titration of sample

Repeat the calibration procedure exactly—same glassware, reagent volumes, boiling rate, and timing—using the sample solution **S**. Record the delivered volume V_2 (mL) at the same endpoint criterion used in calibration. Perform duplicate or triplicate titrations and use the mean value if results agree within predefined limits.

6.7.2.6 Calculation and expression of results

Let C = 5.00 g/L be the concentration of the glucose standard used in calibration. The concentration of reducing sugars in the analyzed solution, χ (g/L as glucose), is:

$$\chi = C \times \frac{V_1}{V_2}$$

If the original juice was diluted by a factor d to obtain S, express the concentration in the original juice as:

$$\rho\ (\text{g/L as glucose}) = \chi \times d$$

Report to an appropriate number of significant figures and state the basis (e.g., as-is juice, or on a °Brix- or moisture-corrected basis if required by specification).

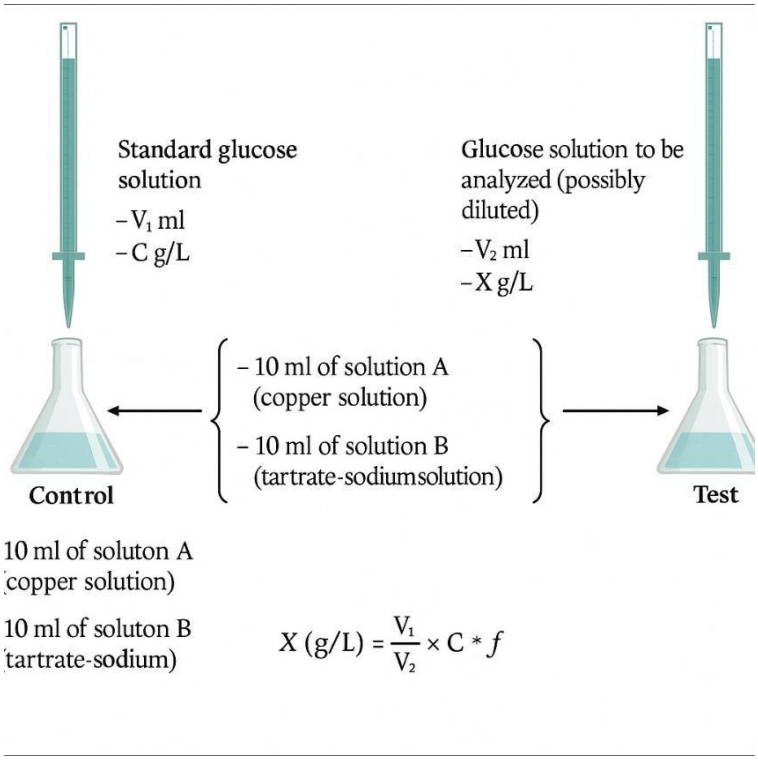


Figure 26. Determination of glucose and expression of the results

6.7.2.7 Method performance, interferences, and quality control

This titration is non-specific for glucose; it quantifies the total reducing sugars present (e.g., fructose, glucose, maltose, lactose if present) and reports them as glucose equivalents. Matrix components that chelate copper or strong oxidants/reductants can bias the endpoint. Maintain constant: reagent composition, mixing order, boiling intensity, flask geometry, and endpoint criterion. Control oxygen exposure and use boiling aids to avoid bumping. Validate recovery with a spiked juice and confirm day-to-day stability with an in-house control sample. Where precise sugar profiling is required, use enzymatic assays (e.g., glucose oxidase/peroxidase for D-glucose) or HPLC with RI/UV for individual sugars; the Fehling method then serves as a screening or legacy specification test.

7 Stability Tests of Canned Foods

7.1 Introduction

Human societies have always preserved food to bridge periods of scarcity. Early practices were empirical and climate-dependent: drying in arid regions, smoking in wooded areas, and natural freezing in cold climates. Salt curing, brining, and storage in fat, oil, honey, or concentrated sugar solutions limited microbial growth and moisture activity. Fermentation also emerged as a robust preservation strategy and likely played a formative role in the development of agriculture.

From the nineteenth century onward, industrial methods such as canning, pasteurization, refrigeration, freezing, and later ionizing radiation enabled large-scale, reliable preservation and long-distance distribution. These technologies reduced dependence on local seasonality while imposing new requirements for process validation and stability testing.

7.2 Food Storage

Food preservation comprises processes that maintain safety and retain sensory quality and nutritional value. Preservation acts by controlling or inactivating biotic factors—microorganisms, insects and other pests, and plant metabolic activity (e.g., sprouting)—and by mitigating abiotic stressors including light, oxygen, heat, moisture, irradiation, and ultraviolet exposure.

Effective preservation integrates:

- **Process control:** validated thermal or nonthermal treatments to achieve target lethality or stability.
- **Packaging:** barriers to oxygen, moisture, and light; mechanical integrity to prevent contamination and reentry of microorganisms.
- **Storage conditions:** specified temperature and humidity ranges, protection from light and mechanical stress, and inventory rotation.

For canned foods, the objective is commercial sterility: absence of viable microorganisms capable of growing under normal nonrefrigerated storage. Stability testing therefore evaluates process adequacy and post-process integrity under defined conditions to ensure shelf life and safety.

7.3 Principle of Food Preservation

Effective preservation controls the biochemical and microbiological activities that drive spoilage and loss of safety in meats, fish, fruits, vegetables, and dairy products. The targets are microbial growth, enzymatic autolysis, and oxidative rancidity.

Microorganisms require assimilable nutrients, available water (water activity, a_w), suitable temperature, and an appropriate redox environment with or without oxygen depending on species. Preservation acts by removing or restricting one or more of these necessities and by maintaining the modified state throughout storage. Core levers include thermal inactivation; reduction of a_w by drying or addition of humectants; acidification or fermentation to inhibit growth and enzymes; redox control via vacuum or modified atmospheres; refrigeration or freezing to slow metabolism; and approved antimicrobials. Packaging then sustains these hurdles by providing barriers to oxygen, moisture, and light and by preventing post-process contamination. Hygienic design, GMP, and validated sanitation keep background loads low so that the applied hurdles are effective and stable.

7.4 Appertization (Canning Sterilization)

Appertization is thermal sterilization of foods sealed in hermetic containers to achieve commercial sterility—the absence of viable microorganisms capable of growing under normal nonrefrigerated conditions. Products of animal or plant origin are filled into metal cans or other hermetic packages, sealed, and heated so that target microorganisms, including spores of *Clostridium botulinum* in low-acid foods, are inactivated to a validated probability level. Process specifications are expressed by time–temperature combinations and equivalent F-values referenced to an appropriate lethality model (e.g., F_0 at 121.1 °C, z-value defined for the organism of concern). Container integrity and rapid, controlled cooling prevent recontamination

and minimize quality loss. Continuous verification uses heat-penetration studies, biological indicators where appropriate, and closure integrity tests to confirm that the scheduled process consistently delivers the required lethality.

7.5 **Historical Overview**

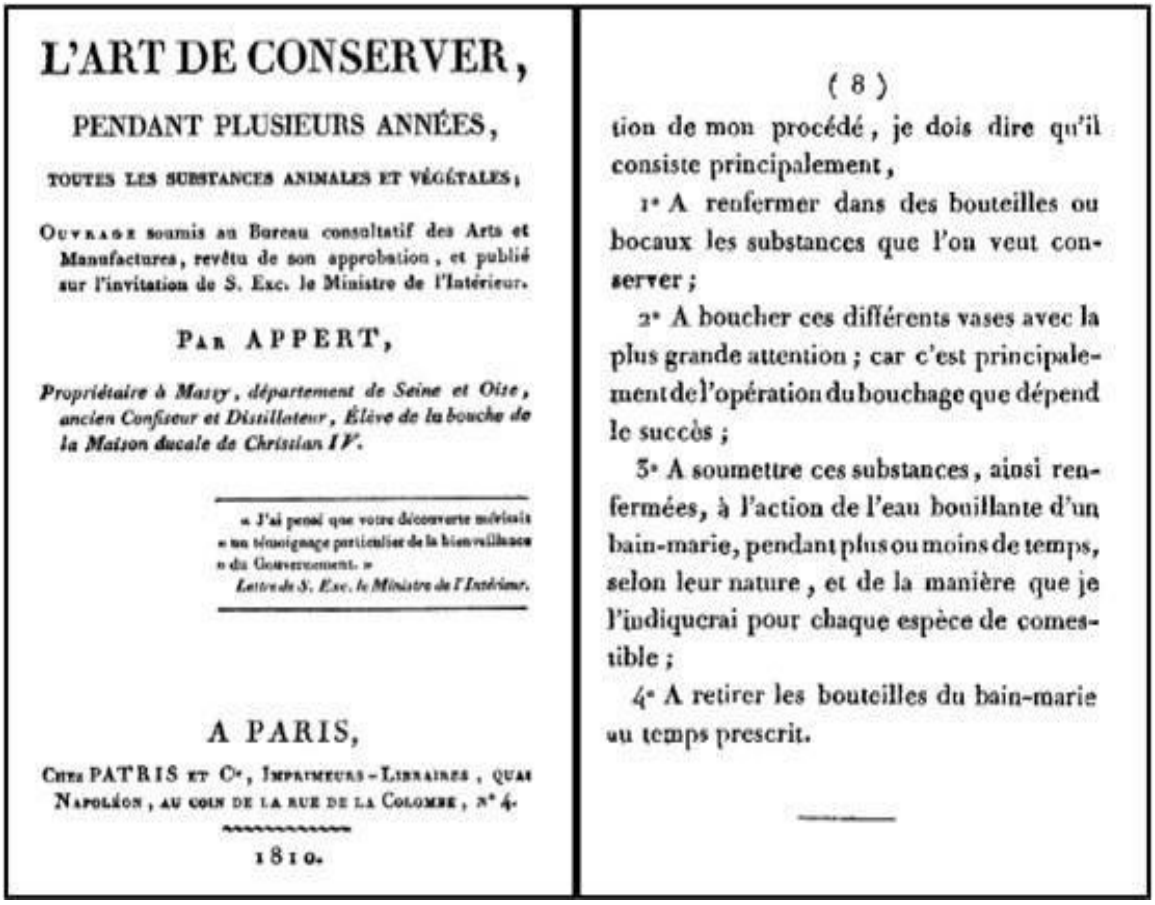
The canning process takes its name from Nicolas Appert (1750–1841), a French confectioner and caterer who, after extensive experimentation beginning in 1795, described a method for preserving foods by enclosing them in airtight containers and applying heat in boiling water. His 1810 treatise, *L'Art de conserver, pendant plusieurs années, toutes les substances animales et végétales*, is the first published monograph on heat preservation in sealed containers (Figure 27).

Later, Louis Pasteur (1822–1895) demonstrated that heating could inactivate microorganisms in beverages such as beer and wine, establishing the microbial basis for heat preservation. He developed what is now called pasteurization, involving brief heating at sub-boiling temperatures in the relative absence of air. In modern usage, pasteurization denotes processes—especially for acid foods (pH < 4.5)—that reduce viable microorganisms using temperatures below 100 °C, as distinct from full sterilization.

In the early twentieth century, thermal process calculation methods were formalized. W. D. Bigelow (1921) introduced a framework to integrate time–temperature data and express lethality via thermal death time and *z*-value concepts. C. Olin Ball (1923) proposed practical solutions for simultaneous heat-conduction modeling at the container's geometric center and lethality calculation, forming the basis of contemporary process scheduling and F-value determination.

Technological advances proceeded in parallel. Early nineteenth-century work explored elevated-temperature processing in sealed vessels, recognizing that temperatures above 100 °C require operation under pressure. Patents in France and elsewhere, including those associated with the Chevallier-Appert lineage, described pressure vessels (autoclaves) fitted with gauges that enabled controlled sterilization at temperatures exceeding the boiling point of water at atmospheric pressure. Subsequent innovations included improved container closures, agitation during heating to enhance heat transfer (applied notably in dairy sterilization in the late

nineteenth century), continuous pressure sterilizers with spiral conveyors introduced by the mid-twentieth century, bulk (continuous) sterilization followed by aseptic packaging, and rotary/agitating retorts (circa 1950) that reduced process times and quality losses.



L'ART DE CONSERVER,

PENDANT PLUSIEURS ANNÉES,

TOUTES LES SUBSTANCES ANIMALES ET VÉGÉTALES;

OUVRAGE soumis au Bureau consultatif des Arts et Manufactures, revêtu de son approbation, et publié sur l'invitation de S. Exc. le Ministre de l'Intérieur.

PAR APPERT,

Propriétaire à Massy, département de Seine et Oise, ancien Confiseur et Distillateur, Élève de la bouche de la Maison ducale de Christian IV.

« J'ai pensé que votre découverte méritait « un témoignage particulier de la bienveillance « du Gouvernement. »

Lettre de S. Exc. le Ministre de l'Intérieur.

A PARIS,

CHEZ PATRIS ET Cᵉ, IMPRIMEURS-LIBRAIRES, QUAI NAPOLÉON, AU COIN DE LA RUE DE LA COLOMBE, N° 4.

1810.

(8)

tion de mon procédé, je dois dire qu'il consiste principalement,

1° A renfermer dans des bouteilles ou bocaux les substances que l'on veut conserver;

2° A boucher ces différents vases avec la plus grande attention; car c'est principalement de l'opération du bouchage que dépend le succès;

3° A soumettre ces substances, ainsi renfermées, à l'action de l'eau bouillante d'un bain-marie, pendant plus ou moins de temps, selon leur nature, et de la manière que je l'indiquerai pour chaque espèce de comestible;

4° A retirer les bouteilles du bain-marie au temps prescrit.

Figure 27. Facsimiles of the first cover and page 8 of Nicolas Appert's 1810 book

Figure 28. Chevallier-Appert advertising campaign for canned goods

7.6 **Principle**

Sterilization aims to render a packaged food commercially sterile, i.e., free of viable microorganisms—including spores—capable of growing under normal nonrefrigerated storage. Two elements act together: a hermetic container that prevents ingress of liquids, gases, and microorganisms throughout processing and storage, and a lethality treatment—typically thermal—that inactivates target microbes and spoilage/quality- degrading enzymes. Containers must maintain seal integrity during retorting and cooling and provide an adequate barrier thereafter.

In canning, sterilization is achieved by heating sealed containers in pressurized retorts at temperatures commonly 110–130 °C; many legacy schedules for low-acid foods center near 115–121 °C, with the exact time–temperature combination determined by heat-penetration studies and lethality targets. The objective is to meet the scheduled F-value **(**e.g., F_0 at 121.1 °C for proteolytic *Clostridium botulinum*) while minimizing nutrient loss and sensory damage. Process design therefore balances microbial safety with retention of texture, color, flavor, and nutritional value.

7.7 Appertization Processes

Appertization comprises thermal sterilization of foods sealed in containers and the maintenance of sterility through container tightness. Because absolute sterility is unattainable in practice, processes are validated to achieve commercial sterility, expressed by a specified log-reduction of reference organisms under defined storage conditions. Low-acid foods (pH > 4.6) require spore-targeting sterilization; acid foods (pH ≤ 4.6) require milder treatments, sometimes hot-fill or pasteurization, because spores do not germinate and grow at typical storage pH.

Sterilization versus pasteurization. Pasteurization applies sub-boiling temperatures (generally < 100 °C) for times sufficient to reduce vegetative cells and enzymes but not bacterial spores; products then rely on accompanying hurdles such as acidity, refrigeration, or short shelf life. Sterilization applies higher temperatures under pressure to inactivate spores, enabling ambient storage.

Packaging sequence. For appertized products, hermetic containers—commonly lacquered tinplate cans or glass jars with validated closures—are filled and sealed before retorting to prevent post-process contamination. After the scheduled process, rapid controlled cooling prevents thermal shock, preserves quality, and avoids vacuum loss that could compromise the seal.

7.8 Foods Covered

Appertization is applied broadly to fruits, vegetables, meats, fish, and composite ready-to-eat meals, with product-specific preprocessing to control enzymatic activity, texture, and microbial load before filling and retorting. For vegetables, common canned items include peas, green beans, sweet corn, mushrooms, tomatoes, broccoli, endive, and celery. These may be packed as single-ingredient items or as mixed preparations such as jardinière packs and ready-to-serve salads, with prior steps tailored to firmness targets and blanching requirements. Fruit products encompass berries (raspberries, redcurrants, blackberries, strawberries) and pome or stone fruits (apples, pears, peaches, apricots), offered as whole or sliced fruit in syrup, water packs, purées, compotes, and jams; formulation controls pH, soluble solids, and heat exposure to preserve color

and flavor. Seafood items include tuna, salmon, sardine, mackerel, crab, shrimp, and scallop products, packed in water, brine, oil, or sauces; process schedules account for lipid content, piece size, and potential heat-stable pathogens. Canned soups, pâtés, and sauces require precise rheology and headspace management to ensure uniform heat penetration. Ready meals such as spaghetti with sauce, meatballs, chili, and ravioli combine multiple components of differing thermal properties; container geometry, agitation retorts where available, and validated lethality targets are used to achieve commercial sterility without excessive quality loss.

7.9 Appertization (Thermal Canning) Operations

Appertization is the preservation of foods by hermetically sealing them in containers and applying a validated thermal process to achieve commercial sterility. Figure 29 summarizes the sequence of unit operations.

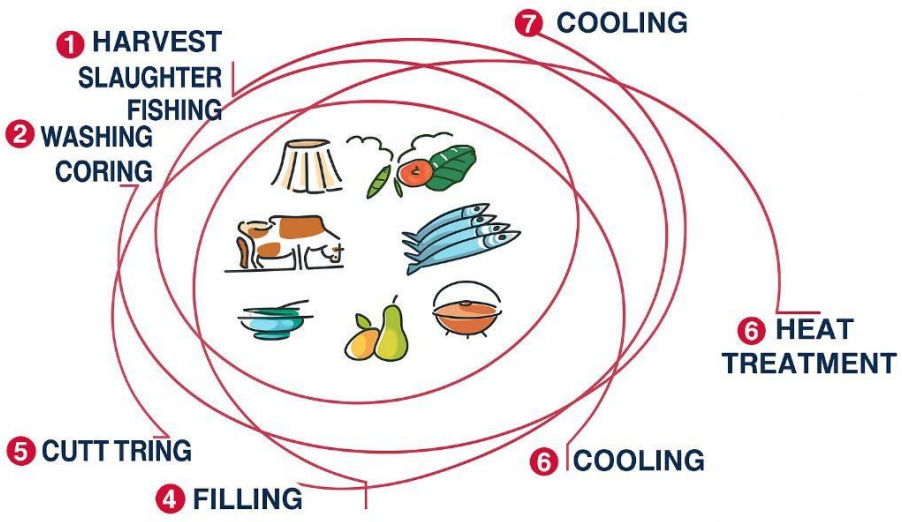


Figure 29. Sequence of appertization operations

7.9.1 Raw Material procurement: Harvesting, Slaughter, and Landing

Product freshness is critical to final quality and process efficiency. Raw materials should be canned as soon as possible after harvest or landing; for example, tender vegetables such as peas

are often processed within 2–3 hours of harvest when logistics permit. Time–temperature exposure before processing should be minimized through chilled storage or rapid transfer.

7.9.2 Preparation

Preparation is product-specific but typically includes cleaning, trimming, defect removal, and sizing.

- **Washing.** Performed by spray (sprinkling) or immersion to remove soil and debris.
- **Peeling.** Common for root and some fruit/vegetable tissues. Methods include mechanical abrasion, steam peeling (brief high-pressure steam followed by dewatering and washing), and chemical lye peeling followed by neutralization and thorough rinsing.
- **Grading/Calibration.** Size grading improves fill uniformity and heat transfer. Examples include belt graders, perforated drum graders, and inclined roller graders.
- **Fish preparation.** Heading, gutting, trimming, and portioning are usually performed manually or with automated lines depending on species and product form.
- **Meat preparation.** Trimming, deboning, defatting, and slicing to specification.

7.9.3 Blanching and pre-cooking

Blanching is a short heat treatment, typically by immersion in hot water or exposure to steam for ~1–2 minutes, followed by rapid cooling. Objectives:

1. Inactivate quality-degrading enzymes such as polyphenol oxidase and pectin methylesterase;
2. Soften tissues and reduce bulk;
3. Expel entrapped gases to limit oxidation and improve subsequent exhausting;
4. Reduce surface microbial load and, in some cases, reduce certain surface residues;
5. Adjust moisture content before filling.

Pre-cooking is applied to meats and some fish to standardize texture, reduce free water and fat, and improve pack integrity.

7.9.4 Filling (Container loading)

Filling accuracy must ensure that the declared drained weight and net content match label claims. Geometry and particle size distribution affect packing efficiency.

- **Manual filling** is reserved for delicate products (e.g., asparagus, celery hearts). To limit microbial growth and protect handlers, such operations are conducted either cold or under controlled hygienic conditions.
- **Automatic filling** commonly uses volumetric or gravimetric dosing. Volumetric systems assume uniform density; gravimetric systems are preferred when density varies.

7.9.5 Addition of covering liquid (Brine/Syrup/Sauce)

A hot covering liquid (brine, syrup, or sauce) is added to improve heat transfer during sterilization, to distribute salt/sugar/spices and permitted additives uniformly, to protect particulates against mechanical damage, and to control headspace. For buoyant products, filling may occur under vacuum or with deaeration to reduce floating and entrapped air.

7.9.6 Container closing: Double seaming and vacuum closures

Containers are closed promptly after hot filling and exhausting to minimize oxygen and contamination.

- **Metal cans.** Lids are attached by double seaming, forming an interlocked, hermetic seam between flange and end. Seam integrity is verified by teardown, seam height/overlap checks, and leak testing to ensure resistance to internal overpressure or vacuum.
- **Glass jars.** Closures commonly use vacuum “twist-off” caps with plastisol seals. Proper vacuum is verified to ensure seal integrity.

7.9.7 Thermal Processing (Retorting)

Thermal processing in batch or continuous retorts achieves commercial sterility by delivering a validated lethality (e.g., expressed as F0 for reference conditions). Schedules are product- and

container-specific and consider acidity, particle size, brine viscosity, and heating behavior. Processing also cooks the product to the desired texture and flavor. All processes must follow documented schedules and verification procedures.

7.9.8 Cooling

Cooling arrests the thermal process and prevents overcooking. It is performed rapidly, often within the retort via water spray or immersion with pressure control to avoid container distortion and to maintain vacuum.

7.9.9 Labeling and Storage

Labels identify product and provide required information. After labeling, cases are stored under clean, dry conditions. Handle to avoid seam or closure damage. Use first-in/first-out inventory rotation.

Table 10. Mandatory label elements for canned foods

Category	Required information
Product identity	Product name; standardized identity where applicable; intended use or instructions when needed
Contents	Full ingredient list in descending order by weight; quantitative ingredient declarations when required; net quantity; drained weight (when applicable)
Origin and traceability	Lot/batch code (coded or clear); country of origin or provenance where required or to avoid consumer confusion; establishment or health mark where required for animal products; name and address of the producer or packer
Date and storage	Date of manufacture if required; best-before or use-by date; storage instructions after opening, if applicable
Nutrition and claims	Nutrition Facts/Information Panel; permitted nutrition or health claims with conditions of use

Packaging and symbols	Barcode; recycling symbols where applicable; can-recycling logo where used

Note. Specific wording, placement, and format vary by jurisdiction. Align with the governing standard or regulation for the target market.

7.9.10 In-Process and final product control

Quality and safety controls are applied throughout:

- **Physical–chemical measures.** Net and drained weights, firmness/texture indices, pH, salinity or °Brix where relevant.
- **Sensory evaluation.** Appearance, color, aroma, taste, and texture against specifications.
- **Composition checks.** Fat content, brine/syrup strength, particulate ratio, and declared ingredient proportions.
- **Microbiological stability.** Incubation tests on control packs and routine verification of retort records. Corrective actions follow any deviation. Accelerated shelf-life studies are used when appropriate.

Table 11. Typical unit operations by commodity category prior to appertization

Operation	Vegetables	Fruits	Fish and seafood	Meats and meat products
Preparation	Washing; cleaning; peeling (as needed); trimming; size grading	Washing; labeling/grading; size grading; pitting or coring; peeling as required	Washing; heading; gutting; trimming; optional skinning; portioning; netting as relevant	Trimming; deboning; defatting; slicing; calibration to piece size
Pre-treatment	Blanching	Not usually required	Pre-cooking as specified	Pre-cooking as specified
Filling	Yes	Yes	Yes	Yes

Covering liquid	Brine or water	Light to heavy syrup or juice	Brine, oil, or sauce	Broth, gravy, gel, or brine
Closing	Double seam or vacuum closure	Double seam or vacuum closure	Double seam or vacuum closure	Double seam or vacuum closure
Thermal processing	Yes	Yes	Yes	Yes
Cooling	Yes	Yes	Yes	Yes

7.10 Metal cans

Modern food cans are fabricated from tinplate (low-carbon steel electroplated with tin) or tin-free steel **(**TFS; electrolytic chromium/chromium-oxide–coated steel), and from aluminum alloys for many small and easy-open formats. Multilayer polymer containers exist but are distinct from metal cans and require different process validation. Contemporary food cans employ an internal food-contact coating (organosol, epoxy, polyester, acrylic, or BPA-non-intent systems) that prevents metal–product interactions, corrosion, sulfur staining, and flavor changes. Coating selection is product-specific and must meet regulatory migration limits for the target market.

Material, geometry, and capacity affect thermal process design. Wall thickness, alloy, and bead pattern influence mechanical strength and heat transfer. Container shape and size determine the heating mode (conduction- vs. convection-dominant), come-up time, cold-spot location, and thus the delivered lethality (e.g., F0F_0F0 for low-acid foods). Small-volume aluminum cans heat and cool rapidly; large round or rectangular steel cans may require agitation or modified recipes to achieve uniform sterilization without quality loss. All commonly used metals are highly recyclable, and both steel and aluminum maintain material properties through repeated cycles, which reduces life-cycle environmental burden when recovery systems are in place.

7.11 Benefits of canned foods

Properly processed canned foods offer several validated advantages.

- Safety is achieved through hermetic sealing and a scheduled thermal process that attains commercial sterility for the stated shelf life.
- Nutritional value is largely retained, especially for macronutrients and most minerals; some heat-labile vitamins (e.g., vitamin C, folate) decrease, whereas others remain stable or become more bioavailable depending on the matrix and process.
- Sensory quality and availability benefit from year-round access to seasonal commodities with minimal post-process handling; product-specific recipes and covering liquids help preserve texture and flavor.
- Convenience follows from ambient storage, long shelf life, standardized drained weights, and compatibility with ready-to-use formats.
- Hygiene is supported by closed-system operations, reduced cross-contamination risk after sealing, and single-use food contact.
- Sustainability is enhanced where metal recovery is effective, since steel and aluminum cans are fully recyclable and often contain significant recycled content.

7.12 Stability control of canned foods

National surveillance data indicate that botulism is rare but severe. In France from 1991 to 2012 the incidence was about 0.43 cases per million inhabitants per year. Foodborne outbreaks have included canned products; for 2013–2016, 39 outbreaks of TIAC were recorded with roughly 70 cases. These figures underscore the need for validated thermal processes and systematic post-process stability controls.

7.12.1 Objective

The stability test assesses whether a shelf-stable, hermetically sealed product remains commercially stable at ambient temperature. The test detects post-process microbial growth by bacteria, yeasts, or moulds that would manifest as gas production, acidification or deacidification, turbidity, off-odours, or loss of vacuum.

Safety is addressed upstream and depends on product category and validated barriers:

- **Low-acid foods** (equilibrium pH > 4.6 at 25 °C) require a scheduled thermal process delivering adequate lethality against *Clostridium botulinum* spores (e.g., an F0 appropriate for container size and product heating characteristics).
- **Acid and acidified foods** (equilibrium pH ≤ 4.6) control vegetative pathogens and spores through pH, sometimes combined with water activity and preservatives; pasteurization is validated accordingly.
- **Pasteurized, chilled products** are outside the appertization scope and are controlled by validated time–temperature–pH–a_w parameters and continuous cold-chain management.

7.12.2 Procedure

Stability testing is performed in accordance with NF V08-408 using incubation (proofing) at 55 °C and 37 °C for 7 days. After incubation the following examinations are performed to confirm absence of microbial development and maintenance of container integrity:

- **Visual and olfactory examination.** Inspect for swelling, paneling, leakage, turbidity, color changes, or abnormal odours. Examine seams or closures for corrosion or defects.
- **Vacuum (depression) measurement.** Use a suitable vacuum gauge to verify that headspace vacuum remains within specification for the product and pack style. Loss of vacuum or overpressure suggests gas production.
- **pH measurement.** Measure with a calibrated pH meter. Compare to the product specification and investigate any abnormal drift from the target equilibrium pH.

Testing is carried out on three units of the same product and lot, drawn from the retort cold point load and/or from units with the lowest initial product temperature at the start of the heat treatment (often the first containers filled). Containers must be sound and undamaged before testing.

Acceptance criteria. No swelling or leakage; vacuum within specified limits; no abnormal odour or turbidity; pH within the validated tolerance for the formulation. Any deviation triggers confirmatory microbiological analysis and a process-deviation review, including verification of retort records, seam integrity, and raw-material and formulation checks.

7.13 Timing and frequency of stability testing

Stability testing begins once the formulation has cleared sensory acceptance in the development phase. Test units must be produced under the same conditions intended for routine manufacture—same equipment, fill volumes, thermal process, closures, and handling—to ensure representativeness.

In routine production, establish a documented testing frequency as part of self-monitoring. Use a risk-based schedule aligned with HACCP and prerequisite programs:

- Baseline periodicity by product family and container size (e.g., per lot, every *n* production runs, or at defined calendar intervals).
- Event-triggered testing after any relevant change: raw-material source, formulation, particle-size distribution, fill weight, covering liquid, container/closure system, retort schedule, loading pattern, or line speed.
- Verification testing following any process deviation or maintenance affecting heating, exhausting, double seaming, or closures.

Maintain records linking each stability test to batch identity, retort records, seam or closure checks, and calibration of pH meters and vacuum gauges. Nonconformities must trigger root-cause analysis and documented corrective actions before release.

7.14 Standards

In Algeria, microbiological criteria and stability requirements for canned foods are set by the Interministerial Order of 2 Muharram 1438 (4 October 2016) establishing microbiological criteria for foods, published in the Official Journal of the Algerian Republic, No. 39, 8 Shawwal 1438 (July 2017). The decree specifies:

1. **Article 9.** Canned foods, regardless of packaging type, must satisfy the stability tests prescribed by applicable regulations before release for consumption.

2. **Article 10.** Stability tests are not performed on canned products in metal, glass, plastic, metalloplastic, or composite (cardboard–metal–plastic) packaging that show major defects such as bulging, flocking, or leakage; such units are nonconforming.
3. **Article 11.** After the prescribed tests:
    - No apparent defects, especially bulging or leakage, may be present.
    - The difference in pH between incubated sample units and the control unit stored at ambient temperature over the selected periods must not exceed 0.5 pH units.
4. **Article 12.** Provisions contrary to this Order are repealed, including the Decree of 14 Safar 1415 (23 July 1994), as amended, on microbiological specifications for certain foods.

8 Chaoual 1438
2 juillet 2017 JOURNAL OFFICIEL DE LA REPUBLIQUE ALGERIENNE N° 39 21

7- Conserves et semi-conserves

Catégories des denrées alimentaires	Micro-organismes/ métabolites	Plan d'échantillonnage		Limites microbiologiques (ufc/g)	
		n	c	m	M
Semi-conserves pasteurisées d'origine animale (1)	Germes aérobies à 30 °C	5	1	10^4	10^5
	Coliformes totaux	5	0	Absence	
	Anaérobies sulfito-réducteurs	5	0	Absence	
	Staphylocoques à coagulase +	5	0	Absence	
	Salmonella	5	0	Absence dans 25 g	
Semi-conserves non pasteurisées d'origine animale (anchois au sel ou à l'huile…) (1)	Germes aérobies à 30 °C	5	1	10^5	10^6
	Coliformes totaux	5	0	Absence	
	Anaérobies sulfito-réducteurs (2)	5	0	Absence	
	Staphylocoques à coagulase +	5	0	Absence	
	Salmonella	5	0	Absence dans 25 g	
Semi-conserves d'origine végétale	Germes aérobies à 30 °C	5	2	10^4	10^5
	Escherichia coli	5	2	10^2	10^3
	Staphylocoques à coagulase +	5	2	10^2	10^3
	Salmonella	5	0	Absence dans 25 g	
Conserves	Epreuves de stabilité	Se reporter à la procédure prévue par la réglementation en vigueur			

(1) Revivification de la suspension mère pendant deux (2) heures à la température du laboratoire pour les semi-conserves pasteurisées et pendant 30 mn à 45 mn pour les semi-conserves non pasteurisées.

(2) Cas particulier des anchois au sel : Anaérobies sulfito-réducteurs : m = M = moins de 10 ufc/g.

Figure 30. Numerical values of the microbiological criteria for canned foods per the Interministerial Order of 2 Muharram 1438 (4 October 2016)

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