Food Packaging and Shelf Life

A Practical Guide

Edited by

Gordon L. Robertson

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11.1 COFFEE QUALITY ATTRIBUTES

The word "coffee" is a general term that comprises roasted coffee (including decaffeinated coffee) and derived beverages, as well as a wide variety of convenience and semimanufactured products such as instant coffee and coffee concentrates. Thus, this term is synonymous with coffee products and encompasses many technological processes responsible for the great compositional complexity of the derived products.

Figure 11.1 shows the key technological steps in obtaining the most important coffee products. Conversion of green coffee beans into a beverage involves three main operations: roasting, grinding, and extraction. Roasting is the key technological operation to transform a green bean, characterized by poor or even unpleasant sensory properties, to the dark one with typical flavor and aroma. Conceptually, roasting is the unit operation in which coffee beans are put in contact with hot surfaces or gases to raise their temperature to 220°C. Exhaustive descriptions of the roasting operation have been given by Clarke (1987a) and Eggers et al. (2001). The complex chemical and physical changes that occur during roasting convert green beans into a very unstable and reactive system. The main reactions occurring in coffee beans during roasting are water removal and carbohydrate

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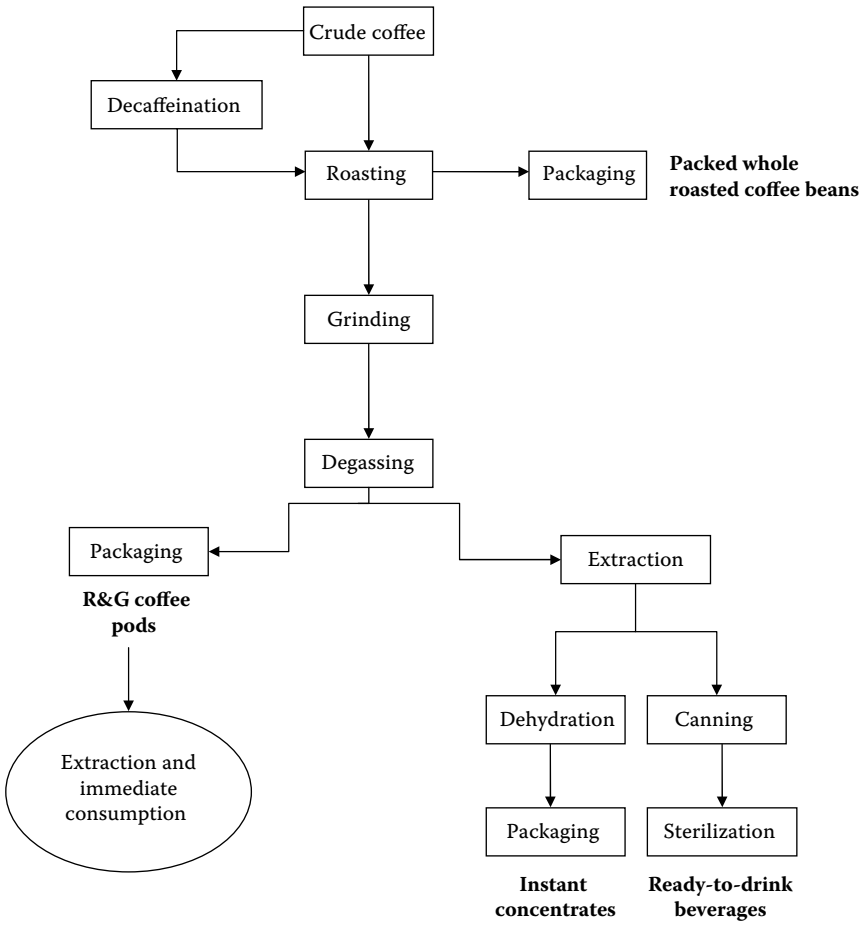

FIGURE 11.1 Key technological steps to obtain the most important coffee products present in the market.

fragmentation and polymerization, via nonenzymic browning (NEB) reactions, and pyrolysis. The chemical composition of the beans is drastically modified, with release of large amounts of CO_2 and the formation of hundreds of substances associated with coffee aroma and taste. There are a large number of volatiles and nonvolatiles such as melanoidins and their precursors among the reaction products. A list of volatile compounds deemed to be of particular importance for coffee flavor is provided by Czerny et al. (1999) and Clarke (2001). Melanoidins are responsible for bean color changes from green-yellow to brown-black. Detailed insight into the roasting chemistry is given by Trugo (1985), Macrae (1985), Clifford (1985), Da Porto et al. (1991), and Homma (2001).

The formation of volatiles and CO_2 during roasting causes the expansion of the beans due to internal buildup of gases, which, along with the high temperatures, allows internal pores and pockets to be formed. However, as the beans become very brittle, they progressively lose their ability to entrap and retain volatiles, which are partially released during storage. For this reason, a degassing step is carried out on ground coffee before packaging in order to avoid the swelling of the packages during storage.

Grinding is the operation that converts whole roasted beans into smaller fragments in order to increase the specific extraction surface area and thus facilitate the transfer of soluble and emulsifiable substances from the coffee matrix to water (Petracco, 2005a, 2005b). Additional coffee-related operations include caffeine removal, which is generally carried out on green coffee beans prior to

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roasting in order to minimize flavor and aroma losses. Decaffeination procedures include (a) decaffeination with organic solvents such as methylene chloride or ethyl acetate, (b) water decaffeination based on the use of green coffee extract with equilibrium quantities of noncaffeine solubles, and (c) supercritical carbon dioxide extraction (Heilmann, 2001).

Brewing, or, more correctly, solid-liquid extraction, is the operation in which roasted and ground (R&G) coffee is mixed with hot water to give a beverage that may be consumed as such or may constitute a semimanufactured product. Coffee brews prepared at the domestic and catering level are generally consumed immediately after preparation. The extraction methods adopted can vary greatly from country to country, strongly affecting the sensory properties of the cup of coffee (Petracco, 2001, 2005a, 2005b; Pictet, 1987).

Although, in term of volumes sold, roasted whole and ground coffee are the main coffee products present in the market, instant coffee and, more recently, coffee concentrates and ready-to-drink coffee brews represent an interesting and dynamic area. In the past 15 years, the production and sale of instant coffee has increased markedly in most countries, with a noticeable increase in the number of different brands. The success of these products is largely because they come in an attractive and convenient physical form and have a very long shelf life. At present, instant coffee and instant coffee-based products include spray-dried or freeze-dried powder or granules. Instant coffee technology includes extraction and further dehydration carried out by freeze drying or thermal concentration (i.e., spray drying). A comprehensive description of the process and equipment involved in instant coffee manufacture is given by Clarke (2001).

Coffee concentrate technology includes solid-liquid extraction and subsequent partial water removal through thermal or cryo-concentration. Partial dehydration results in concentrates with a solids content of up to 22% and a water activity (a_w) of up to 0.84 (Manzocco and Nicoli, 2007). These products are used as ingredients for the food industry or as semimanufactured products for vending machines and in catering. More recently, ready-to-drink coffee beverages have become very popular, especially in Asian countries, where the tradition of consuming freshly prepared coffee brews is not widespread. They are generally pasteurized and chilled or sterilized and stored at ambient temperature. They include the so-called coffee drinks obtained by using 5 g or more of R&G coffee per cup/can and the soft coffee drinks based on coffee drinks with added sugar, dairy products, emulsifiers, and other additional ingredients (Petracco, 2001).

11.2 DETERIORATIVE REACTIONS AND INDICES OF FAILURE

As previously mentioned, coffee is a term that encompasses a wide number of products that undergo different deteriorative reactions, have different sensitivities to environmental variables, and thus have different shelf lives. For this reason, stability problems relevant to roasted, instant, and liquid coffees will be discussed separately. However, a common element in all these coffee products is their compositional complexity. At the moment, the structures of the majority of the compounds present in roasted coffee, most of which end up suspended or solubilized in the coffee beverage, are unknown. This is the reason for the still inadequate scientific knowledge on the causes and variables affecting coffee product quality loss during storage. In general terms, it is well known that coffee products lose their peculiar sensory properties during storage, but in most cases, very little is known about the chemical and physical events underlying these undesirable changes.

11.2.1 Roasted Coffee

Although generally recognized as a shelf-stable product, roasted coffee undergoes important chemical and physical changes that greatly affect quality and acceptability of the coffee brew. From a sensory point of view, coffee staleness is defined as "a sweet but unpleasant flavor and aroma of roasted coffee which reflects the oxidation of many of the pleasant volatiles and the loss of others."

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(Buffo et al., 2004). The most important physical and chemical events involved in roasted coffee staling during storage are as follows:

- Volatile release
- Carbon dioxide release
- Surface oil migration
- Oxidation reactions

Volatiles and CO_2 are easily released into the vapor phase due to diffusion mechanisms enhanced by the fact that the pressure inside the bean pores is greater than atmospheric. The evolution of CO_2 is of particular importance because, although it can be considered an index of the aroma richness of roasted coffee, if it is released after coffee packaging it produces overpressure inside the package, with possible bursting and loss of package integrity. The presence of a pressure gradient between bean pores and outside is also responsible for the migration of lipids, together with their lipophilic solutes, onto the bean surface. As a consequence of the increased exposed surface area, these compounds become more prone to oxidation. Volatile and CO_2 release and oxidation reactions are considered the main causes of coffee staling.

The sensitivity of coffee to oxidation reactions originates from the roasting stage. The majority of compounds formed via NEB during roasting are strong antioxidants able to react both as chain breakers and as oxygen scavengers (Nicoli et al., 1997). It is interesting to note that just after roasting, coffee has a remarkably negative redox potential value, indicating its strong reducing properties (Nicoli et al., 2004). For this reason oxygen availability causes the oxidation of a wide number of coffee compounds. The development of the typical stale flavor of aged coffee is the result of two different reactions: the release of volatiles in the vapor phase and the development of off-flavor as the result of oxidation of some of them (Nicoli and Savonitto, 2005). Steinhardt and Holscher (1991) found that the aroma freshness of whole roasted coffee was mainly determined by certain low-boiling-point components, namely low molecular weight sulfur compounds, Strecker aldehydes, and α-dicarbonyls. Aroma staling is caused by the loss of most of them. The heavier compounds, such as furfuryl mercaptan, remain in the coffee, causing the "aging" note. As coffee is a dry product with a_w values ranging from 0.1 to 0.4, an additional cause of coffee staling is the sorption of moisture. As the moisture content and hence a_w increases, the roasted coffee bean, which is initially brittle and easy to grind, becomes very elastic due to a decrease in the glass transition temperature T_g (Figure 11.2) (Anese et al., 2005; Pittia, 2007). These changes also have a great impact on volatile retention, as shown in Figure 11.3.

Although many of these changes are considered unavoidable, the rate at which they occur mostly depends on the exposed surface area of the coffee (i.e., whether whole or ground) and on environmental variables such as oxygen partial pressure and relative humidity. The greater the surface area exposed, the higher is the degradation rate. This is particularly evident for ground coffee contained in paper pods. Obviously, the influence of these variables is strongly dependent on the packaging solutions adopted. An additional environmental variable that can strongly affect coffee staling is temperature. The temperature dependence of the most important degradation reactions that take place in roasted coffee during storage can be well described by the Arrhenius equation (Manzocco and Nicoli, 2007).

Typical indices of failure (IoFs) for roasted coffee can be the headspace volatiles or CO_2 concentration (Anese et al., 2006; Nicoli and Savonitto, 2005). Among the volatiles, some specific indicators of coffee aroma freshness have been selected: (a) the M/B aroma index, the ratio between methylfuran and 2-butanone (Amstalden, 2001); (b) the flavor quality index, based on five key odorants (hexanal, vinylpyrazine, pyrrol, furfurylmethylketone, and pyridine), which shows an inverse linear relationship with the M/B index and has been used to follow coffee staling in industrial settings; and (c) the MM aroma index, the ratio of methanol to 2-methylfuran (Amstalden, 2001; Buffo et al., 2004; Holsher and Steinhartd, 1992). Steinhardt and Holscher (1991) suggested that the loss of coffee aroma freshness is due to the loss of certain aroma volatiles (mainly methyl mercaptan).

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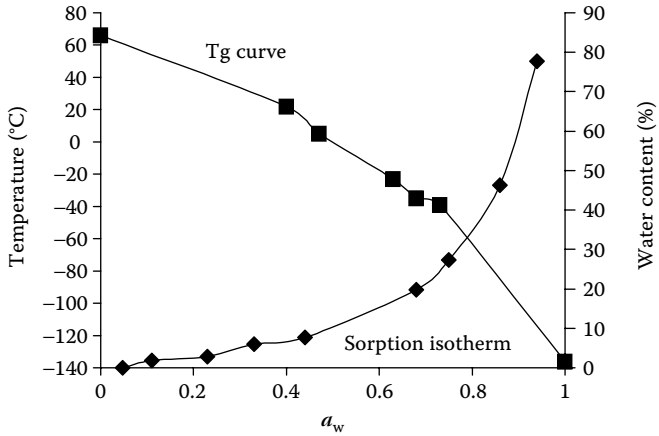

FIGURE 11.2 Modified state diagram of light-roasted coffee. (From Anese A., Manzocco L., Maltini E. 2005. Effect of coffee physical structure on volatile release. *European Food Research and Technology* 221: 434–438, with permission.)

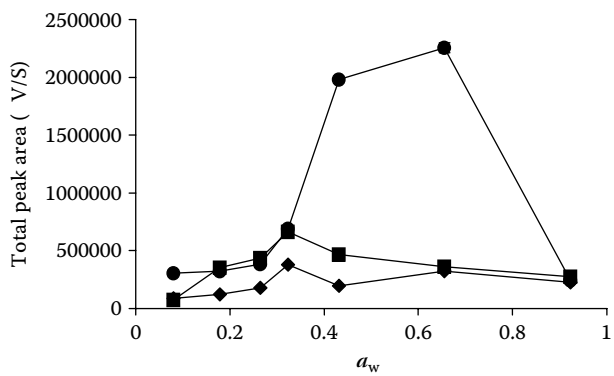

FIGURE 11.3 Total peak area of headspace volatile compounds of light- (◆), medium- (■), and dark roasted (●) soluble coffees as a function of a_w. (From Anese A., Manzocco L., Maltini E. 2005. Effect of coffee physical structure on volatile release. *European Food Research and Technology* 221: 434–438, with permission.)

which can be used as an indicator of freshness. Peroxide and acidity indexes have also been used to follow R&G coffee stability (Chafer et al., 1998). Additional IoFs can be represented by the redox potential value and sensory indicators (Cardelli and Labuza, 2001; Manzocco and Lagazio 2009; Nicoli et al., 2004). No literature data are available on chemical, physical, and sensory IoF values corresponding to acceptability limits for shelf life assessment. Consumer acceptability can also be used as an IoF, and survival analysis can be applied to obtain directly an estimate of the shelf life of the product (Anese et al., 2006).

11.2.2 Instant Coffee

Instant coffee has very long shelf stability if proper packaging solutions are adopted. It is probably for this reason that there are a limited number of studies on the stability and shelf life assessment of instant coffee. In contrast to what happens to R&G coffee, instant coffee does not have the problem of a steady loss of CO_2 during storage. However, volatile release may be responsible for a

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rapid quality loss of the product, as most of these volatiles are susceptible to oxidation reactions. The extent of oxidation can be enhanced when coffee oils are spread over the granule surface to improve product aroma. Being hygroscopic, instant coffee is also susceptible to the action of ambient moisture, which affects volatile liquid vapor partition. An increase in moisture content to 7–8% is responsible for "caking," where the powder or granules becomes a pasty or solid mass (Clarke, 1987b). Therefore, O_2 and moisture are the environmental variables that must be carefully controlled in selecting suitable packaging. In addition, low storage temperatures should be adopted in order to slow down volatile release rates and keep the product below the glass transition temperature.

Typical IoFs are similar to those previously mentioned for roasted coffee, such as volatile profile or selected volatiles as indicators of the oxidative status of the product, and moisture content. Recently, the Weibull hazard analysis has been applied to sensory acceptability data at different storage times to evaluate the shelf life of spray-dried instant coffee (Ocampo and Giraldo, 2006).

11.2.3 Coffee Concentrates and Drinks

These products, generally pasteurized or sterilized, and thus stable with respect to microbial spoilage, show a very low chemical stability characterized by a change in the flavor profile and an increase in perceived sourness. These changes are accompanied by a pH decrease, corresponding to an increase in titratable acidity. Quality depletion of liquid coffee as well as of coffee concentrates starts immediately after brewing and proceeds at a significant rate, even at subzero temperatures. At present, very little is known about the mechanisms underlying liquid coffee instability. It has been suggested that the decrease in pH could be the consequence of complex reactions, probably related to NEB pathways involving carbohydrates and amino acids. Additional mechanisms involving lactone hydrolysis could also contribute to pH decrease (Balzer, 2001). Similar reactions have been reported for coffee brew aroma staling, which was mainly attributed to interactions between odor-active thiols and melanoidins (Hofmann and Schieberle, 2002; Mueller and Hofmann, 2007). Typical IoFs for this class of product are headspace volatile profile; pH, and, hence H_3O^+ concentration; changes in antioxidant properties; and sensory attributes (Anese and Nicoli, 2003; Dalla Rosa et al., 1990; Nicoli et al., 1989; Perez-Martinez et al., 2008). Consumer acceptability can also be used as an IoF, and survival analysis can be applied to obtain an assessment of the shelf life of the product (Manzocco and Lagazio, 2009).

11.3 HOW PACKAGING IMPACTS INDICES OF FAILURE

11.3.1 Roasted Coffee

The oldest types of packaging used to store and sell roasted whole beans were simple jute or cardboard bags. At that time they perfectly fitted storage and selling, as the requested shelf life of roasted coffee was very short. However, the development of large-scale manufacturers and the increased complexity of the distribution chain demanded a longer shelf life for roasted coffee. Consequently, major attention had to be paid to coffee stability. Therefore, the choice of the packaging materials as well as of the packaging procedures became crucial for delivering the required shelf life. As previously mentioned, the shelf life of roasted coffee is the result of the interaction between the coffee matrix and the packaging, which influences the environmental conditions inside the package. The latter are affected by the permeability of the package to moisture, gases (mainly O_2 and CO_2), and coffee volatiles. In addition, the material should be greaseproof due to the presence of oil on the surface of the coffee beans. Furthermore, the headspace volume and the resistance to increases in internal pressure of the packaging can play a critical role.

Table 11.1 shows the most commonly used materials for the packaging of roasted whole and ground coffee. Tinplate or aluminum cans were the first commercial packages used by the coffee industry (Clarke, 1987b). The use of cans has considerable advantages, such as providing a total barrier to water, gases, and volatile compounds. On the other hand, their management is expensive,

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TABLE 11.1
Materials Commonly Used for Packaging of Roasted Coffee

Material	Advantages	Disadvantages
Tinplate	Total barrier Resistant to pressure Recyclable Sturdy Satisfactory ecobalance[a]	Expensive Its rigidity prevents an optimal use of space
Glass	Total barrier Resistant to pressure Recyclable Good ecobalance[a]	Fragile Expensive Heavy Its rigidity prevents an optimal use of space Difficult to sort from other wastes
Aluminum	Total barrier Resistant to pressure Recyclable	Expensive Its rigidity prevents an optimal use of space Difficult to sort from other wastes Poor ecobalance[a]
Combined flexible multiply polymer aluminum	Inexpensive Simple manufacturing technology Large flexibility and optimal use of space Satisfactory ecobalance[a]	Nontight barrier Resistant only to negative pressure Poor strength
Combined multiply aluminum cardboard	Inexpensive Total barrier Satisfactory ecobalance[a]	Nontight barrier Resistant only to negative pressure Poor strength Complex manufacturing technology Difficult to sort from other wastes
Flexible combined multiply polymer	Inexpensive Simple manufacturing technology Satisfactory CO_2 permeability No need to sort from other wastes	

Source: From Nicoli M.C., Savonitto O. 2005. Physical and chemical changes of roasted coffee during storage. In: *Espresso Coffee: the Science of Quality*. Viani R., Illy A. (Eds), 2nd edn. San Diego, California: Elsevier Academic Press, pp. 230–245, with permission.

[a] Ecobalance is defined as the total pollution resulting from energy consumption and that resulting from the product during manufacture, use, and disposal.

because their rigidity and generally cylindrical shape do not allow optimal use of space in the warehouse and during distribution. Similar considerations apply to glass containers, which have the additional disadvantage of fragility. Today, the most commonly used materials are the inexpensive and easy-to-manage flexible polymer–aluminum multiply laminates, which permit both hard and soft packs. These materials ensure an efficient barrier due to the presence of a central layer of aluminum foil. The other layers are a waterproof film on the inner side and a rigid film that gives mechanical strength on the outer side. A typical construction is poly(ethylene terephthalate) (PET)/alufoil/low density polyethylene (LDPE). Other materials used with comparable performance are metalized PET laminated to LDPE or four-ply structures based on biaxially oriented polypropylene (BOPP) or biaxially oriented nylon (BON) in addition to OPET, alufoil, and LDPE (Clarke, 1987b). These packages can be fitted with a one-way valve that opens at a preset pressure to release gases.

A recent innovation in the R&G coffee sector is the coffee pod, which contains the optimal quantity of coffee for a single-cup preparation. The Easy Serving Espresso pod, or ESE pod, is an example. It looks like a small disk and contains 7 g of coffee prepacked in its own paper filter. Coffee

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pods are generally individually sealed in a high-barrier metalized secondary package flushed with an inert gas (typically N_2) prior to sealing. More recently, coffee capsules have been introduced into the market. Designed for specific espresso machines, capsules have different shapes and may contain different amounts of coffee, ranging from 5.0 to 7.5 g. The capsules are generally made of aluminum coated on the inside with a protective film. Before sealing they are saturated with N_2 to improve shelf life.

11.3.2 Instant Coffee

Instant coffee has been commercialized for many years, and the prevalent package is glass jars or tinplate cans of various shapes. Moisture-proofness is ensured by a paper/plastic laminate or a metal foil sealed to the rim of the container, over which a tight-fitting metal lid is placed in the case of tinplate containers and a plastic or metal screw-cap in the case of jars (Clarke, 1987b). Today flexible materials such as LDPE, BOPP/BOPP, metalized PET/LDPE, and PET/alufoil/LDPE are also employed to package single doses (2 g) (Alves and Bordin, 1998; Robertson, 2006).

11.3.3 Coffee Concentrates and Drinks

Ready-to-drink coffee beverages are available in several packaging formats. Pop-top (easy-open) aluminum cans are a widely popular option, especially in Japan and other Asian countries. Other coffee drinks are available in plastic cups with different shapes. Some of these can be instantaneously heated or cooled due to the presence of an inside cavity containing endothermic or exothermic salts and water separated by a thin layer. By breaking this layer and shaking the container, the salts and water mix, releasing or adsorbing heat. Some manufactures also offer liquid coffee in plastic bottles to those consumers who need to purchase relatively large volumes. Plastic/alufoil/paperboard laminated cartons are also available. Coffee is also available as a liquid concentrate in plastic squeezable bottles or bag-in-box containers for industrial use.

11.4 SHELF LIFE OF COFFEE IN DIFFERENT PACKAGING MATERIALS

11.4.1 Roasted Coffee

11.4.1.1 Effect of Packaging Techniques on Whole and Ground Roasted Coffee Shelf Life

In order to obtain the required shelf life of roasted coffee, the choice of packaging technique is as crucial as the choice of packaging materials. Table 11.2 shows the packaging procedures together with the relevant materials that can be employed for the packaging of both roasted whole and ground coffee. The latter can be packed in multidose containers or in single doses (coffee pods). The table also reports some shelf life data. Unfortunately, very few data on coffee shelf life are available in the literature. In addition, most of the published papers do not specify whether the reported data refer to whole or ground coffee or give clear indications on the methodologies used for shelf life assessment. For this reason the data reported in Table 11.2 only provide an approximate indication of shelf life.

Packaging in air is the easier and older technique. It simply consists of filling and hermetically sealing the package. In this way, coffee is protected against humidity and flavor loss, but the presence of O_2 inside the package greatly shortens the shelf life by inducing the development of oxidative reactions. This technique can be applied to degassed coffee to prevent the swelling and, in extreme cases, the possible bursting of the package. The shelf life of the product is very low, typically around 1 month. Another possibility is fitting a one-way safety valve on the packaging. The valve opens at a preset pressure to release the CO_2 without letting air in. As CO_2 is heavier than air, it tends to stratify at the bottom of the package headspace. This implies that during [illegible] expelled through the valve, followed by O_2 and CO_2. The flushing effect of CO_2 contributes to the

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TABLE 11.2
Main Packaging Techniques and Relevant Materials Applied for the Packaging of Roasted Coffee and Reported Shelf Lives

Technique			Materials	Requirements	Shelf Life (Months)
Multidose packaging	Air packaging		Metal cans Flexible laminated materials	Degassing or one-way safety valve	1–3*
	Modified atmosphere packaging	Under vacuum	Metal cans Flexible laminated materials	Degassing	4–6*
		Inert gas	Metal cans Flexible laminated materials	Degassing or one-way safety valve	6–8*
		Active packaging	Metal cans Flexible laminated materials	Degassing or one-way safety valve	n.d.
		Pressurization	Metal cans	One-way safety valve	>18*
Single-dose packaging	Modified atmosphere packaging	Inert gas	Paper pods + flexible laminated materials Plastic capsules	Degassing	>18

Source: * Shelf-life data from Nicoli M.C., Savonitto O. 2005. Physical and chemical changes of roasted coffee during storage. In: *Espresso Coffee: the Science of Quality*. Viani R., Illy A. (Eds), 2nd edn. San Diego, California: Elsevier Academic Press, pp. 230–245, with permission.
n.d. = not determined

control of the internal atmosphere composition, reducing the level of O_2 to 10–12%, thus increasing the shelf life of the product for up to 3 months (Illy and Viani, 1995).

It should be noted that flavors also escape through the valve, leading to beverages with poor sensory quality (Nicoli and Savonitto, 2005). Such problems may be potentially overcome by packing the coffee under modified atmosphere conditions. The conditions may be based on the modification of both the overall pressure and the gas composition inside the container. The simplest way of modifying the atmosphere is based on vacuum application. In such a case, after filling, a high vacuum is created so that the level of O_2 is reduced to 4–6% (Nicoli and Savonitto, 2005). The shelf life of roasted coffee packed under vacuum is prolonged by up to 4–6 months. The vacuum packaging procedure can be applied to both cans and flexible materials. In the latter case, after the vacuum is applied, the package collapses onto the coffee to form the very popular "hard bricks," in which the brick shape is created by the intimate contact between coffee and packaging. To prevent pressure increase inside the container during storage due to volatile release, roasted coffee must be degassed before packaging. It should be noted that the vacuum level that can be reached in rigid containers is lower than that achieved in flexible bags (Robertson, 2006).

The shelf life of roasted coffee could be further improved by replacing the air inside the package with an inert gas such as N_2 or CO_2. Although the latter is not an "inert" gas, it is naturally present in the product. The procedure applied is based on the initial application of vacuum followed by its release with the desired gases. The pressure in the package is in equilibrium with the atmosphere at the moment of sealing. The final percentage of O_2 is expected to be 1–2% and the shelf life up to 6–8 months. Also in this case, coffee should be degassed before packaging to avoid an increase

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in pressure during storage. Similarly to air packaging, the prevention of increased internal pressure may be obviated by fitting a one-way valve on the package. Inert gas packaging can be performed employing both cans and laminated materials. The packs made with the laminated materials become the so-called soft or pillow packs.

In combination with vacuum and inert gas packaging, further atmosphere modification inside the container can be achieved using active packaging procedures. Although such systems are expected to extend coffee shelf life, to our knowledge no shelf life data are available in the literature. Active packaging is performed to achieve two different goals: (a) reducing the percentage of oxygen residue in the containers and (b) avoiding CO_2 emission from coffee. In the first case, oxygen scavenger systems are represented by sachets inserted into the package or bound by a label to the package wall. The most commonly applied oxygen scavengers in coffee packaging are based on iron powder oxidation (Vermeiren et al., 1999). This system could reduce the level of O_2 to a value lower than that reached by applying inert gas packaging. As an alternative tool to the one-way valve, the inclusion inside the package of sachets containing CO_2 scavengers has recently become quite widespread to avoid the problem of CO_2 release during storage (Vermeiren et al., 1999).

Pressurization is the modified atmosphere packaging that seems to most increase the shelf life of the roasted coffee (Nicoli and Savonitto, 2005). According to the producers, pressurization allows the shelf life of coffee to be extended for up to 18 months. However, no information is available on the criteria adopted to reach such an estimate. Pressurization consists of creating a pressure inside the package that is higher than atmospheric. Such high pressure can be established within the container by packing roasted coffee immediately after air cooling. In this case the pressure passively rises due to the large amount of gas released from coffee upon the degassing process, which, in this case, occurs inside the sealed container. The residual percentage of O_2 is less than 1% due to the relative increase in CO_2 concentration as well as the scavenging effect exerted by coffee melanoidins (Nicoli and Savonitto, 2005). The increase in the pressure inside the containers produces two further effects leading to the quality improvement of the final coffee beverage. As described by Clarke (1987b), the degassing rate decreases under pressurized conditions, reducing oil migration. The oil tends to remain inside the cell being protected against oxidative reaction. In addition, the pressure inside the package becomes higher than the partial pressure of most volatiles, so more of them are dissolved in the lipid phase or bound to melanoidins. Under such conditions the flavors are protected, even when the package is open, and pressurized blends retain their fragrance longer than those packaged using other techniques. However, in order to obtain these effects, the pressurized coffee should be temporarily stored before distribution for at least 15 days, which is the time generally required for the degassing process to reach equilibrium inside the container (Nicoli and Savonitto, 2005).

In contrast, pressure inside the package may also be conventionally increased by sealing the filled containers in the presence of gases at the desired overpressure. In order to withstand the internal pressure, the containers must be made of rigid materials (i.e., tinplate or aluminum), as pressure has been reported to rise to up to 2.2 atm (Illy and Viani, 1995). It should be noted that the packaging techniques reported in Table 11.2 can be used for packaging of both whole and ground roasted coffee. As previously mentioned, the higher exposed surface area of ground coffee compared to the whole bean is responsible for an increase in the release rate of CO_2 and volatiles. For this reason, at the moment of packing, ground coffee is expected to be not only poorer in volatiles, due to their lower concentration and oxidation level, but also more unstable because of the faster dynamics of volatile release. In this regard, Figure 11.4 compares the evolution of headspace volatiles of beans and ground coffee during storage. Thus, it can be inferred that, using the same packaging materials and techniques, ground coffee has a shorter shelf life than coffee beans.

Furthermore, as most CO_2 is released during grinding and degassing operations before packing, O_2 concentration inside the container becomes the most critical variable affecting the shelf life of R&G coffee. Thus, packing under modified atmosphere is required to obtain a longer shelf life. Similar considerations apply to coffee pods, whose large surface area makes ground coffee-

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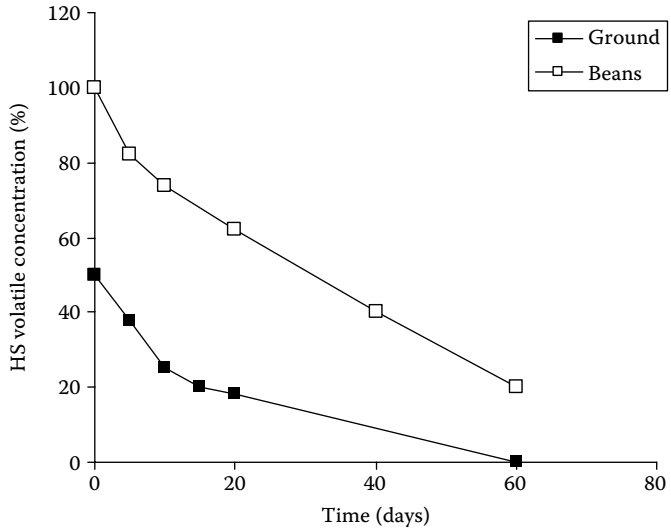

FIGURE 11.4 Headspace volatile concentration of beans and ground coffee as a function of storage time at 25°C. (From Nicoli M.C., Innocente N., Pittia P., Lerici C.R. 1993. *Staling of roasted coffee: volatile release and oxidation reactions during storage*. Proceedings of the 15th International Scientific Colloquium on Coffee, Montpellier, France: pp. 557–566, with permission.)

more prone to oxidation. Pods in modified atmospheres are generally attributed by their producers a shelf life higher than 18 months, not significantly different from that of R&G multidose coffee. However, such indications seem to contradict the shelf life data reported for bulk coffee packed under inert gas. It can be hypothesized that shelf life data reported for bulk coffee could be underestimated because they are based on actual product turnover and not on the assessment of IoFs.

11.4.1.2 Secondary Shelf Life of Coffee

During home or catering use, coffee is almost never consumed immediately after opening the package. When used by consumers, packages of roasted coffee are opened and closed frequently, so coffee degradation may suddenly speed up due to modification of conditions inside the package to those characteristic of an air package. In addition, the moisture content of coffee often increases due to equilibration with the relative humidity of the environment. As a consequence, degradation reactions during consumption proceed at much higher rates than those observed in the originally packed ground coffee, thus contributing to what is known as a secondary shelf life (Cappuccio et al., 2001). Secondary shelf life represents the length of time after opening of the package during which coffee maintains acceptable quality. The end of the secondary shelf life of R&G coffee at 30°C under different a_w conditions was determined by Anese et al. (2006) through consumer rejection methodology, choosing as the acceptability limit a 50% consumer rejection (Table 11.3).

The end of secondary shelf life was almost constant, at around 20 days, at a_w values lower than 0.36. At higher a_w values, the secondary shelf life greatly decreased, to about 13 days, corresponding to an a_w of 0.44. These results were in agreement with the high loss of volatile compounds of coffee equilibrated at the highest a_w. Independent of the hydration degree of the coffee, the end of shelf life was reached when the same total volatile pressure was measured in the coffee headspace. In particular, a loss of about 60% of the volatiles compared to those in the coffee at the time of opening was identified as a proper acceptability limit indicating the end of shelf life.

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TABLE 11.3
Secondary Shelf Life of Ground Roasted Coffee as a Function of a_w

a_w	Shelf Life (Days)
0.09	20±3
0.17	23±4
0.23	22±6
0.36	17±6
0.44	13±3

Source: From Anese M., Manzocco L., Nicoli M.C. 2006. Modeling the secondary shelf life of ground roasted coffee. *Journal of Agricultural and Food Chemistry* 54: 5571–5576, with permission.

11.4.2 Instant Coffee

The few shelf life data reported in the literature indicate that instant coffee maintains its original quality for years, provided that moisture content is less than 4–5% w/w to keep the product below its glass transition point, and provided oxygen present in the headspace is low enough to avoid flavor oxidation. The shelf life of unopened instant coffee varies from 18 to 36 months if stored in glass jars or tinplate cans. The Q_{10}'s for coffee staling and moisture absorption are quite low at 1.5–2.0 (Labuza, 1982).

Ocampo and Giraldo (2006) reported that instant coffee packed in glass was rejected by 50% of consumers after approximately 2 years of storage at 18°C. Flexible materials containing aluminum foil (i.e., PET/alufoil/LDPE) as an O_2 and moisture barrier may also be very effective, giving shelf lives of around one year. However, a much lower shelf life is obtained when instant coffee is stored in other plastic materials. Alves and Bordin (1998) investigated the shelf life of portion packs (25 and 50 g) of instant coffee at 30°C and 80% RH in three different plastic structures. The surface areas of the packs were 8 × 11 cm for 25 g and 10 × 16 cm for 50 g. The structure of the packages, their water vapor transmission rate (WVTR), and the shelf life of the coffee at 30°C and 80% RH are shown in Table 11.4. The end of shelf life was when the moisture content had reached 7.8%, at which time powder agglomeration occurred. Data on the shelf life of agglomerated and powdered instant coffee packed in PP and PET pots were presented in a later paper (Alves et al., 2000), with the shelf life in the former being up to 2.5 years, compared to 4 months in the latter.

11.4.3 Coffee Concentrates and Drinks

As previously reported, the extraordinary instability of coffee brews is due to the development of a number of different chemical reactions, leading to rapid off-flavor formation and a decrease in pH. The changes in pH of a dark roasted coffee brew are shown in Figure 11.5 as a function of storage time at different temperatures. The rate of pH decrease is strongly affected by temperature, and other environmental variables, potentially controllable by packaging, do not exert a critical role. Quality depletion is, in this case, strictly related to brew formulation. In other words, in the wide range of coffee derivatives, beverages are the ones whose shelf life is independent of the packaging.

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TABLE 11.4
Shelf Life of Instant Coffee in Plastic Packages

Package Structure	WVTR $g\ m^{-2}\ day^{-1}$	Shelf Life (Days)	
		25 g	50 g
LDPE 30 μm	6.1	15	16
BOPP/BOPPP 20 μm/40 μm	1.2	84	94
Met PET/ LDPE 12 μm/70 μm	0.9	108	112

Source: From Alves R.M.V., Bordin M.R. 1998. Shelf life prediction of instant coffee by mathematical method. *Ciencia e Tecnologia de Alimentos* 18: 19–24, with permission.

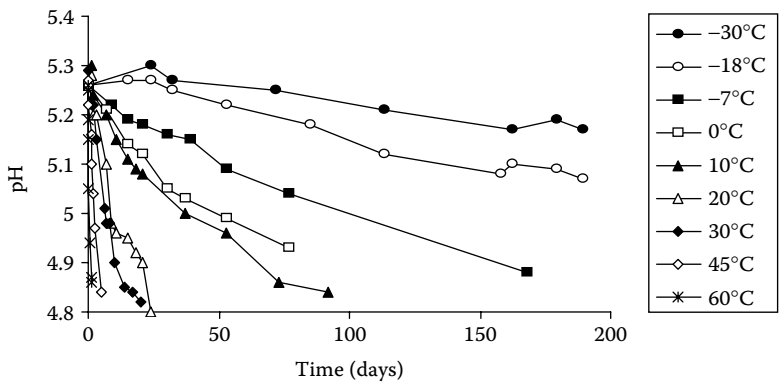

FIGURE 11.5 pH of a dark roasted coffee brew (1.8% w/w solid concentration) as a function of storage time at different temperatures. (From Manzocco L., Nicoli M.C. 2007. Modeling the effect of water activity and storage temperature on chemical stability of coffee brews. *Journal of Agricultural and Food Chemistry* 55: 6521–6526, with permission.)

Manzocco and Lagazio (2009) assessed the shelf life of a dark roasted coffee brew (initial pH 5.26) stored at room temperature by examining the probability of rejection expressed by consumers. Table 11.5 shows the shelf life of the coffee brew as a function of solids concentration and storage temperature. As consumer rejection strongly correlated with H_3O^+ concentration, and thus with pH, the relevant pH values at the end of shelf life were also calculated and are presented in Table 11.6. Such data may be exploited to predict, for instance, that when the coffee brew reaches a pH of 5.16, it is rejected by more that 25% of consumers, and this occurs after 4 ± 1 days of storage.

Coffee brews are also processed to obtain a number of coffee concentrates with different a_w values. Studies carried out on liquid coffee products with different solid content indicate that the rate of H_3O^+ formation was considerably lower for a_w values below 0.5 (0.94% w/w solid concentration). Beyond this critical boundary, the rate increased, reaching a maximum value of 0.8 a_w (78% w/w solids concentration), as shown in Figure 11.6 (Manzocco and Nicoli, 2007). If we consider an acceptability limit for the coffee concentrates as a consumer rejection higher than 25% (pH limit 5.16), the shelf life data can be calculated from the reaction rates reported in Figure 11.6 and are shown in Table 11.6. It can be seen that, at constant temperature, the shelf life of coffee concentrates decreases as solids content increases.

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TABLE 11.5
Shelf Life of Liquid Coffee with Increasing Solids Concentration Stored at Different Temperatures

Temperature (°C)	Shelf Life (Days)		
	Solids Concentration (% w/w)		
	1.8	15	78
0	17	14	12
10	14	9	8
20	4	3	1
30	3	2	1
45	1	1	<1
60	<1	<1	<1

TABLE 11.6
Shelf Life and pH Estimated for Increasing Percentages of Consumers Rejecting the Coffee Brew

Consumers Rejecting the Sample (%)	Shelf Life (Days) ± 95% Confidence Interval	pH ± 95% Confidence Interval
10	2 ± 1	5.22 ± 0.03
25	3 ± 1	5.19 ± 0.03
50	5 ± 1	5.14 ± 0.02
75	6 ± 1	5.12 ± 0.02
90	8 ± 2	5.08 ± 0.04

Source: From Manzocco L., Lagazio C. 2009. Coffee brew shelf life modelling by integration of acceptability and quality data. *Food Quality and Preference* 20: 24–29, with permission.

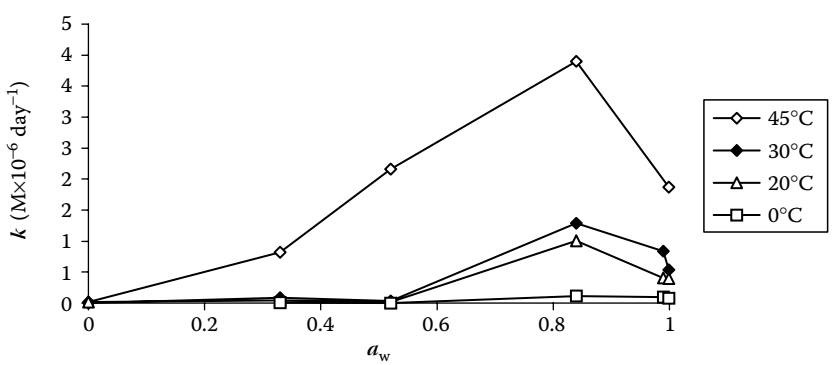

FIGURE 11.6 Apparent zero-order rate constants (k) of H_3O^+ formation as a function of a_w of coffee samples stored at different temperatures. (From Manzocco L., Nicoli M.C. 2007. Modeling the effect of water activity and storage temperature on chemical stability of coffee brews. *Journal of Agricultural and Food Chemistry* 55: 6521–6526, with permission.)

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