FOOD REVIEWS INTERNATIONAL, 17(1), 1–44 (2001)

IMPLEMENTATION OF HAZARD ANALYSIS CRITICAL CONTROL POINT (HACCP) SYSTEM TO THE ALCOHOLIC BEVERAGES INDUSTRY

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ABSTRACT

Alcoholic beverages (fermented or not) have been consumed for more than three thousand years and, generally, they have been considered safe because of their alcohol content. However, in recent years adulteration (i.e., use of low-cost, inappropriate alcohol) has made rapid progress in this field. Food and drink control and safety can be assured within the frame of strict adherence to quality and safety systems (ISO 9000 series, HACCP and TQM). The flow diagrams for the production of several alcohol drinks were shown, and an extensive hazard analysis critical control point (HACCP) analysis was carried out in order to reveal the weaknesses of the production line and to suggest the critical limits in compliance with legislation and the corresponding preventive and corrective measures.

Key Words: HACCP; Alcoholic beverages; Hazard; CCP; Ouzo; Gin; Vodka; Brandy; Distilled spirits; Wine; Sake; Beer.

INTRODUCTION

It has taken almost 30 years (since 1971 when it was officially presented for the first time) for the concept of Hazard Analysis Critical Control Point (HACCP) to become universally accepted as one of the most rigorous preventive programs whose strict implementation can assure food safety (1,2). Although HACCP is a system

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aiming at zero defect products, it is well known that this is not feasible and the real target is the minimization of unacceptable unsafe products. When a company decides to adopt HACCP it should be able to set controls at each point of the production line at which safety problems (physical, chemical, and microbiological) are likely to occur (3).

Prior to initiating a HACCP system, a company must endeavor to put together a HACCP plan, most often described by the five following steps (1,3,4,5): a) identify HACCP resources and assemble the team, b) describe the food and its distribution method, c) state clearly intended use and consumers, and d) develop a process flow diagram and e) verify the validity of this diagram in practice (operation).

The regulatory requirements for Sanitation Standard Operating Procedures (SSOPs) in conjunction with Good Manufacturing Practices (GMPs) should also be considered as a prerequisite to HACCP. The following seven HACCP principles constituting the major steps to writing an HACCP (6,3,7):

1. Conduct a hazard analysis
2. Identify critical control points (CCPs) by applying the HACCP decision tree (8, Fig. 1)
3. Establish critical limits (CLs) for each CCP
4. Establish monitoring actions
5. Establish corrective actions
6. Establish record-keeping procedures
7. Establish verification procedures

Today, HACCP is continuously gaining importance and worldwide acceptability, being implemented by most countries all over the world. The implementation of HACCP in the EU in particular was introduced by the Council Directives 91/43/93 and 92/5/92. HACCP's implementation is considerably facilitated when other complementary quality assurance systems (ISO 9001/2) are already in place (9). The current tendency is integrating HACCP and ISO 9001 or ISO 9002 (10,11) within the frame of Total Quality Management.

Since the two most important stages for the drink industry are fermentation and bottling, where hazards are likely to occur, special care is required (trained personnel, sanitation, equipment maintenance, GMP).

This review article aims to present an overview of HACCP implementation to alcoholic beverages through the production and distribution chains and to pinpoint the current CCPs, CLs and preventive and corrective actions due to be undertaken in case any deviations are observed.

BEER

Introduction

Beer is an alcoholic beverage produced by the fermentation of wort obtained from barley malt flavored with hops. The alcoholic content of beer ranges from 4%

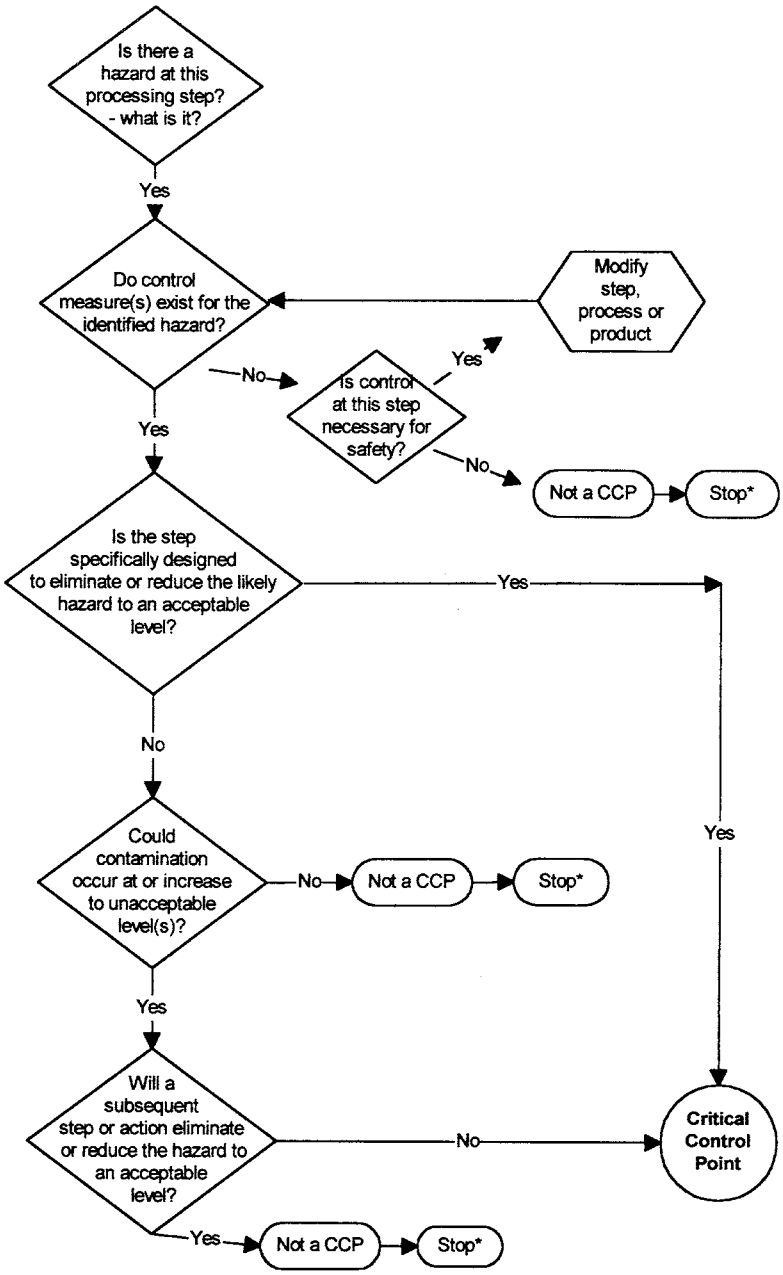

Figure 1. HACCP decision tree (102).

for ordinary beer up to 15%. Beer's first production in Mesopotamia by the Sumerians in the 5th millennium B.C. classifies it among the most ancient of alcoholic beverages. Towards the middle of the 3rd millennium B.C. there is documentary evidence of beer drinking by the Egyptians, who probably introduced beer technology

in Europe. Beer drinking in northern Europe dates back to early antiquity contrary to the Mediterranean countries, in which wine was the commonest drink. A critical point in its history was the works of Louis Pasteur, which greatly contributed to the understanding of beer production (12).

Beer Main Production Stages

The main stages for beer production are shown schematically in Figure 2, together with their critical control point (CCP) numbers.

Incoming Raw Materials (CCP1)

The principal raw materials used to brew beer, are water, malted barley, hops and yeast. Barley is required to be of sufficiently good malting quality in order to germinate and to produce a satisfactory product yield. Other factors such as dormancy and losses during malting have also to be considered (13). The malting or subsequent brewing characteristics are subtly affected by the weather conditions prevailing over the growing period. Some information regarding the quality of a batch of barley can be obtained by visual inspection, but usually it is complemented by analyses including moisture content, total nitrogen, 1000-grain weight and the portion of nongerminating grain. The National Institute of Agricultural Botany (UK) provides descriptions of the European malting varieties. Residues of certain pesticides used on malting barley survive through to the final malt and wort and can affect the process and quality of the end product (CCP). Fungicides and herbicides influencing enzyme synthesis during malting process can accumulate in the yeast, thereby affecting the next fermentation (14). The critical limits of these substances are prescribed by Codex Alimentarius and are presented in Table 1. Presence of heavy metals above the specifications of Directive 80/776/EC and mycotoxin production more than 0.04 mg/L, mainly from *Fusarium* species, such as aflatoxins, ochratoxine A, zearoleon, deoxyniralenol, constitutes a high risk for human health (CCP) (15). Temperature and relative humidity are two interacting parameters that define the germination of spores of different microorganisms (16). Visual inspection and biological plate methods detect the fungal contamination; for mycotoxin analysis employment of HPLC or ELISA is required (17).

The quality of the water used is a major factor affecting the beer quality (CCP). The development of strict water control standards was introduced by most breweries in which water is filtered through activated carbon as well as ion exchange resins to remove impurities (pesticides, herbicides and industrial wastes). Two ions of particular importance in water are calcium and carbonate/bicarbonate, which control the pH during brewing. Calcium also protects α-amylase from heat destruction, thereby permitting liquefaction of starch during mashing (18).

Hops not only provide bitter flavor to the beer but impart a hoppy character as well. These aroma components are derived from the essential oil. The brewing

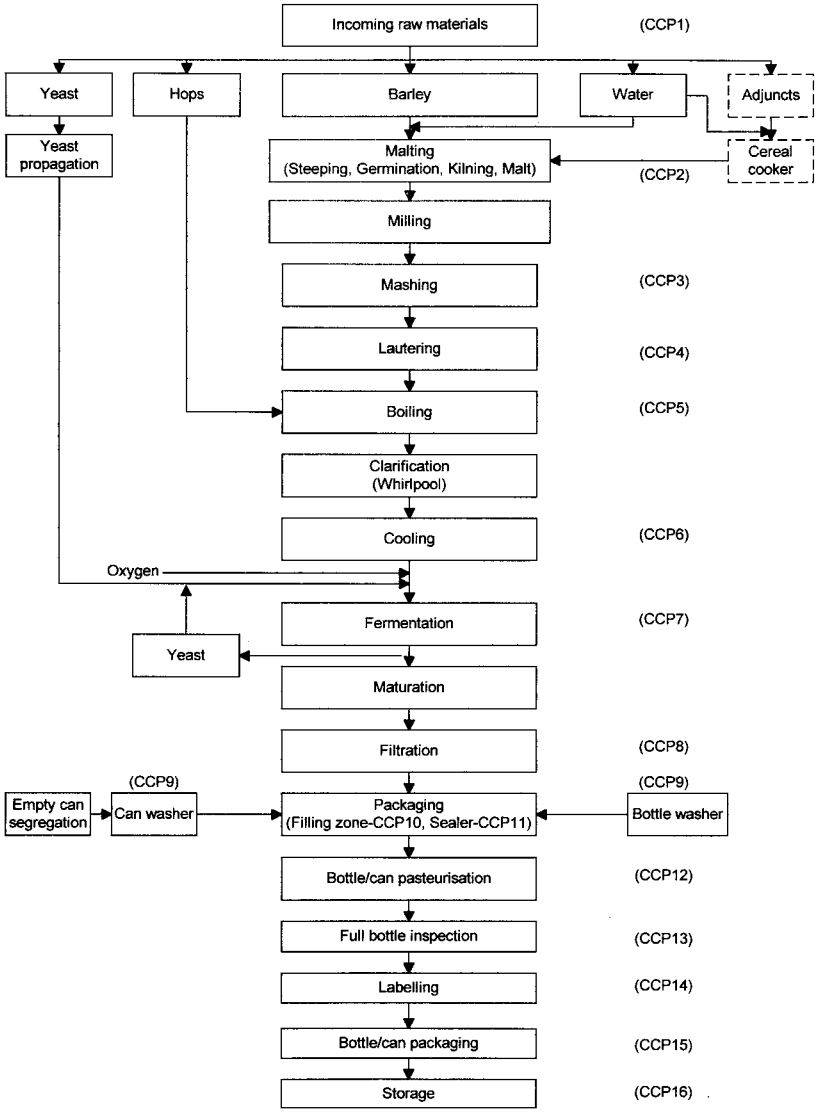

Figure 2. Process flow diagram of beer production (22,26).

value of hops depends on the resin fraction which amounts to 15%, and the essential oil, comprising ∼0.5%. Total resin is defined as the material soluble in both cold methanol and diethyl ether; "soft" resin is that proportion of the total which is soluble in hexane comprising mainly α and β-acids, while "hard" resin is insoluble in hexane. The α-acids that are the most significant bittering precursors can be distinguished from other soft resins from their ability to form a lead salt which is insoluble in methanol. The determination of moisture and seed content also provide useful conclusions about their quality (13). Adjuncts of carbohydrate origin other

Table 1. Summary of Hazards, CCPs, CLs, Monitoring, Corrective Actions, and Personnel Responsible for Beer Production

Process Step	Hazards (P, M, C)[a]	Control-Preventive Measures	CCP Parameter	Critical Limit	Monitoring Procedures	Corrective Actions	Responsible Personnel
Incoming raw materials (CCP1)	M	Control of fungi development, temperature and RH regulation during storage	Mycotoxin production	0.004 mg/L	Visual inspection of fungi development HPLC, ELISA, EPS analysis	Rejection of specific batch	Quality control manager
		Certified suppliers, schedule inspections	Presence of *Entero-bacteriaceae*	0	Microbiological analysis	Rejection of specific batch Change supplier	
		Stricktly following instructions	Contamination of microbial preparations	100% clean		Change preparation method	
	C	Efficient disease management system in use Certified suppliers	Pesticide residues in barley, hops, water	By pesticide as described by Codex	Specific chemical analyses	Rejection of specific batch	Quality control manager
		Proper water decontamination	Heavy metals presence	Within specifications prescribed in Directive 80/778/EC		Rejection of specific batch De-metallisation step	
		Use of deioniser	Water's electrical conductivity	<20 ms/cm	Continuous recording of deioniser	Automatic discontinuation of deioniser, analysis of water samples	

Malting (CCP2)	C	Use of indirect heating systems, control low-NO_x burners	NDMA production during kilning	2.5 ppb	Continuous checking the area, specific analyses	Rejection or mixing with other batches	Quality control manager
	P	Control of time, temperature and RH	Colour and flavour development	Specified by particular plant	Continuous monitoring of processing conditions	Mixing with other malts, rejection of specific batch	Quality control manager
	M	Proper hadling operations after production	Mycotoxin production	0.004 mg/L	Visual inspection of fungi development, HPLC, ELISA, EPS analysis	Rejection of specific batch	Quality control manager
Mashing (CCP3)	C	Control of temperature, CIP	NDMA production, detergent residues	2.5 ppb None	Continuous recording of the processing	Adjust lautering program	Quality control manager
Lautering (CCP4)	C	Schedule Inspection, under plate cleaning	ATNC	<20 ppb	Microbiological and chemical analyses	Proper maintain, re-lautering of the batch,	Quality control manager
Boiling (CCP5)	C	Correct use of boiler treatment chemicals	Contamination with detergents	0	CIP system	Repair CIP, batch rejection	Quality control manager
Fermentation (CCP6)	M	Aeration of wort, use of yeast for max 6 generations	Poor yeast viability, “stuck” fermentation	Min 90% viable yeast cell	Yeast concentration, fermentability, O_2 concentration in the wort	Increase propagation frequency, wort aeration	Quality control manager

(*continued*)

Table 1. *Continued.*

Process Step	Hazards (P, M, C)[a]	Control-Preventive Measures	CCP Parameter	Critical Limit	Monitoring Procedures	Corrective Actions	Responsible Personnel
Fermentation (CCP6)	M	Inspection of CIP system and equipment	*Lactobacilli, acetic acid bacteria and wild yeasts*	Presence in 1 mL plate +1 mL actidione	Plate count method, or a rapid detection method	Proper disinfection of equipment, reprocessing of the batch	Quality control manager
Filtration (CCP7)	C	Use CO_2, prefilling of filter with water	O_2 uptake	>0.2 ppm dissolved O_2	Measurement of dissolved O_2	Survey of filtration for increased O_2 pick up	Quality control manager
Bottle/can inspector (CCP8)	C	GMP	Cleaning performance	No solids, no liquid remnants	Elaborate electronic recognition systems after CIP	Rewashing of bottles, CIP system inspection	Quality control manager
	P	Certified supplier, proper handling of bottles	Bottles proper for foods and drinks, bottles condition	Cracks/scratches absence	On-line visual control	Rejection of faulty bottles	Trained personnel
Bottle/can filler (CCP9)	C	Installation of controlling equipment on the CIP system	Contamination with detergents	Complete absence	Organoleptic examination of filled bottles	Batch rejection	Trained personnel
Bottle/can sealer (CCP10)	P	Correct installation of equipment	Blow-off effect	Occurrence reduced to an acceptable level	Control set sealing pressure	Automatic removal of destroyed bottles	Trained personnel
Bottle/can pasteurization (CCP11)	P	Running pasteuriser according to program	Oxidation caused of wrong temperature-time set	Max. 65°C for 20 min, quick cooling at the exit	Continuous on-line time-temperature checking	Adjust temperature, maintain equipment	Technical manager

Bottle/can inspection (CCP12)	P	Regular inspection of the machinery	Physical damage	Occurrence reduced to an acceptable level	On-line monitoring	Equipment standardisation	Technical manager
Labeling (CCP13)	P	Careful selection of the etiquettes	Misplaced etiquettes	Reduced to an acceptable level	Visual checks, control of the equipment	Relabeling the specific batch	Trained personnel
Bottle/can packaging (CCP14)	P	Correct installation of the equipment	Bottles condition during palletisation	Absence of rifts in the lute, crack or scratches	On-line visual control	Adjust the equipment parameters (speed, pressure)	Technical manager
Storage (CCP15)	P	Control storage conditions	Organoleptic condition of beer	Specified by the particular plant	Scheduled controls of finished product	Adjust the storehouse conditions	Trained personnel

[a]P, M, C stand for physical, microbiological and chemical hazards, respectively.

than malt are sometimes used as an additional source of extract to supplement malt. Unmalted cereal adjuncts usually contain no active enzymes and therefore rely on malt or exogenous enzymes to provide the necessary enzymes for starch conversion (19).

Yeast growth cannot be separated from the fermentation process and it is necessary to the production of both beer and fresh yeast for use in subsequent fermentations. The quality control of yeasts comprises a) the selection, maintenance and supply of a suitable strain and b) the routine assessment of purity and detection of microbial contamination (CCP) (20).

Malting (CCP2)

This process involves steeping the barley in a shallow bed of water at a temperature of 10–15°C, so that its moisture content amounts to 45 wt.-% of barley. Barley is then allowed to germinate under controlled temperature conditions at approximately 15°C and RH100% with constant turning to prevent matting the rootlets. The barleycorn undergoes germination through air passage via the germinating malt for 3–5 days. Gentle heating stops germination due to moisture removal and promotes formation of flavor compounds. The kiln temperature regime is crucial for the color of malt and the survival of enzymes to be used in the mashing process. Kilning duration usually varies between 24 and 48 h. Time, temperature and moisture content are varied to control color and flavor development. Chemical, microbiological and physical hazards may be encountered in this step. In particular, nitrosodimethylamine (NDMA) production during kilning (reaction of NO_x with organic materials) constitutes a chemical hazard with a critical limit (CL) at 2.5 ppb, because of its suspected carcinogenic effect. In addition, mycotoxin production more than 0.004 mg/L and color and flavor alteration represent chemical and physical hazards, respectively. The NDMA content in malt can be controlled by using indirect heating systems or by carefully maintained and controlled low-NO_x burners. Regular checks should nevertheless be carried out by the maltster, so that the residual risk caused by polluted air is kept as low as possible (17). The finished malt has its rootlets removed and is screened to produce the uniform quality. During the malting process two important changes occur: a) the barley develops its own enzyme systems; and b) the naturally produced enzymes start to break down the cell structure of the endosperm (19). Malt quality control tests include hot water extract, color, soluble nitrogen, total nitrogen, moisture, enzyme activities, viscosity, and lautering prediction tests. The microbiological status of malt used in the following steps (CCP) is very much dependent on its handling operations after production (16).

Milling

The main function of dry or wet milling is to reduce the malt particle size to form grist (ground or milled grain). The particle size reduction facilitates the

extraction of soluble components, mainly sugars, and nitrogenous compounds from the endosperm (21).

Mashing (CCP3)

Mashing, the first step in wort production, involves extracting soluble materials from the milled malt. This is accomplished by feeding the grist through Steel's masher, a hydrator consisting of a large-bore tube bent at right angles. During its passage through the vertical portion of tube the grist is spayed with hot water (typically 65°C) and then mixed with the help of a revolving screw (22). The floating endosperm particles hydrate and undergo further amylolytic scission by α- and β-amylases. Processors adjust the pH and temperature conditions to allow both enzymes with a range of susceptibility to pH and temperature to work effectively. NDMA production (CL = 2.5 ppb), as well as possible detergent residues, constitute potential chemical hazards for public health. Continuous monitoring at the processing and adjustment of the lautering program and Cleaning In Place (CIP) system when deviation occurs are proper preventive and corrective actions, respectively.

Lautering (CCP4)

The lauter tun is a vessel normally rinsed thoroughly with a sparging or hot water delivery system before receiving the mash, which precipitates at the flat floor of slotted stainless steel or brass plates. At tun center there is a lautering machine, on the shaft of which rotating rakes are attached to facilitate draining the wort into a collection vessel called grant. The wort is recirculated through the lauter tun until it reaches a certain degree of clarity, whereupon it is delivered to the kettle (21). In lautering, production of Apparent Total *N*-nitroso compounds (ATNC) above the CL of 20 ppb constitute a CCP that should be monitored with chemical and microbiological analyses. Scheduled inspection and under-plate cleaning can prevent insufficient separation of trub from wort (23).

Boiling (CCP5)

Wort is boiled for up to 2 h at atmospheric pressure following the addition of hops (CCP). The shape of copper, boiling time, and temperature can affect the quality of produced beer. The major objectives of wort boiling are a) wort sterilization and enzyme inactivation, b) extraction of bitter and other substances from hops and formation of flavor compounds, and c) evaporation of excess water and wort concentration, evaporation of undesirable flavour volatiles. Wort contamination of the wort with *Enterobacteriaceae* from hops can result in various off-flavors, including "vegetable" and "phenolic" taints (24). Correct use of boiler treatment chemicals, steam condensate tasting for carrying over the taints, and operation of

phenol analyses are all essential to avoid chemical contamination and taints development (23).

Clarification

Wort clarification is conducted either through sedimentation or filtration. When whole hop cones are used, it is necessary to employ either a hop back or a hop separator–filter. The drop in hop usage and the widespread acceptance of preisomerized extracts led to utilization of a vertical cylinder known as whirlpool, which induces sustainable circulation of the trub collecting as a compact cone in the base. Whirlpools are more suited to larger worts and can also be used with ale. In modern breweries, centrifuges constitute a promising alternative to whirlpools (25).

Cooling

To prepare for fermentation, the clear hopped wort is cooled, usually in a plate heat exchanger. During cooling, it is advisable to aerate or even to oxygenate the wort, because next processing step involves yeast growth promoted in the presence of dissolved oxygen, despite the low dissolved oxygen concentration in wort (7–14 ppm) (22).

Fermentation (CCP6)

Fermentation aims at producing ethanol by fermenting yeasts. Yeasts vary in their behavior during fermentation; some strains tend to flocculate trap plug CO_2 and rising to the top, whereas others do not flocculate and precipitate. Several lagers are produced by bottom fermentation, while many types of ales and stouts are produced by top fermentation. *Saccharomyces cerevisiae* is usually the top fermenting yeast in the range of 18–22°C, whilst the bottom-fermenting are strains of *Saccharomyces uvarum* that function in the range of 7–15°C (26). Therefore, the temperature at which fermentation occurs is very crucial for the further stages of beer production. The modern use of cylindroconical vessels has reduced the fermentation period for ales and lagers from 7 to 2 or 3 days and from 10 to 7 days, respectively (27). Fermentation is monitored by taking samples for measuring the specific gravity and can be controlled by varying the cooling rate (20). "Stuck" fermentation where the required ethanol level is not attained and microbial contamination with *Lactic acid bacteria*, mainly *Lactobacilii* and *Pediococcus*, which cause taints during maturation or in bottle storage (28) represent microbiological hazards, which are the only hazard detected at this stage. Common causes for "stuck" fermentation include premature yeast flocculation and yeast failure to metabolize maltotriose due to repression by glucose (25). A minimum of 90% viable yeast cells (CL) can be applied to ensure the development of the process. During fermentation the pH

drops from 5.2 to 4.2 and by its completion the yeast is removed either as a top or bottom crop and retained to pitch the next fermentation. Apart from the conventional microbial detection methods with plate count, several rapid detection methods potentially applied in breweries, such as ATP bioluminescence, flow cytometry, and polymerase chain reaction, have been developed to reduce the incubation time from 3–4 days to 1–2 (29,30).

Maturation

Maturation includes all those changes occurring between the end of primary fermentation to beer filtration (31). Ale is matured at relatively warm temperatures, 12–20°C, while lagers are held under much cooler conditions. The warmer temperatures allow the rapid metabolism of any residual and priming sugars, as well as loss of green flavors, within 1–2 weeks, depending on beer type, yeast strain, wort composition, and primary fermentation conditions. In case of lager, the beer used to be held at refrigerated temperatures for up to several months after fermentation, allowing formation of protein/tannin complexes (18). Today, the enzyme addition has substantially shortened this process to several weeks, during which flavor matures. Enzymes, such as papain, may be added during transfer between fermentation and maturation tank. The dosage of the proteolytic enzyme varies depending on type of beer and process. Enzyme activity decreases progressively during maturation until its inactivation with pasteurization. Part of the enzyme absorbed in the yeast surface is removed during filtration (19).

Filtration (CCP7)

Beer produced during fermentation is turbid and should be clarified prior to its marketing. This turbidity is due to the presence of yeasts and proteinaceous materials associated with carbohydrates and polyphenols. The formation of these protein precipitates is attributed to cold temperature, low pH and poor solubility in alcoholic solutions (32). To prevent this from occurring in the final product, the beer may be subjected to various chill-proofing treatments during its storage. These treatments generally include the addition of clays to absorb the colloidal materials or proteolytic enzymes used to further solubilize the protein fraction (33). Since oxygen uptake during this process could severely affect the product organoleptic characteristics, a CCP of dissolved oxygen should be applied with a CL of 0.2 ppm (34).

Packaging and Sealing

The packing section comprises several CCPs including the containers to be used, their cleaning and disinfection (CCP8), the filler line (CCP9) and the sealer (CCP10). The bursting pressure of the bottles, as guaranteed by the manufacturer in his specifications for the new glass, may no longer be valid in case of reusable

bottles, due to the considerable physical stress during already exerted upon them during the filling process. Insufficient cleaning of reusable bottles due to low temperatures and concentrations of the employed cleaning solutions, as well as presence of extraneous entrapped materials within bottles and improper emptying, consist possible hazards. Moreover, cleaning solution remnants and shards introduced through the procedure pose problems under working conditions. The beer filler may be contaminated by cleaning and disinfection solutions. Contamination sources may be due to inadequate pressure or faulty CIP system resulting in cleaning and disinfecting solution remains in the pressure tank or the ring bowl of the filler (35,36). The crown corker should be correctly installed; the filling pressure of bottle caps on the mouths of the bottles should be adjusted to ensure a specified blow-off effect to avoid bottle bursting. After filling, there should be a full bottle inspector detecting glass particles in bottles or possible leakage (37).

Bottle Pasteurization (CCP11)

Pasteurization is carried out to ensure the beer shelf life over a period of months. This is accomplished by the development of tunnel pasteurization in which the beer bottle is subjected to 60°C for 20 min. Over-pasteurization, which causes oxidation and can adversely affect beer flavor (38) is a potential physical hazard. Furthermore, it is crucial to check the time-temperature procedure with adequate corrective actions for assuring the production of a satisfactory product.

Bottle Inspection (CCP12)

Bottle inspection after the pasteurization step is important to ensure that bottles have not been damaged during the process (39). Should such a situation occur, the equipment has to be standardized by the production engineer.

Labeling and Standardization (CCP13)

Labeling of the package should comply with the requirements of the Codex General for the labeling of prepackaged foods (40). This means that the name of the product shall be clearly declared, there must be a list of ingredients in descending order of proportion, no other fruit may be represented pictorially except those used, and "the date of minimum durability" will be declared by the month and year in uncoded numerical sequence.

Bottle/Can Packaging (CCP14)

Bottles (cans) are packaged into paperboard boxes of various sizes, according to the bottle or can dimensions. The encountered hazards can be of physical nature concerning the bottles (cans) condition during the procedure.

Storage (CCP15)

The finished beer undergoes chemical, microbiological and organoleptic analysis to ensure that its properties are within its specification range. A synoptical presentation of the occurring hazards, CCPs, CLs, and preventive corrective measures is given in Table 1.

SAKE

Introduction

Sake is a fermented liquor made from rice and coming in many varieties depending on the raw materials, manufacturing process and process after brewing (41). According to the earliest records, sake was originally brewed from rice that had been chewed to reach saccharification, followed by natural fermentation. Sake brewed this way was used as a sacred wine in the worship of the Shinto gods. This association with religion, Shintoism and Buddhism, has caused a deep intertwining of sake with the traditions and social customs of Japan. Thus, today sake is served at ceremonies and celebrations of all kinds (42). Sake has the highest alcohol percentage by volume of any fermented beverage. In its natural, undiluted state, it may contain a potent 20% ethanol compared to 3–5 % for beer or 9–12% for wine, which may reach higher values for fortified wines (43,44). The central brewers' union divides sake into four basic flavor types, on four axes of sweet, sour, bitter and umai. The latter is another translator's nightmare, which generally ends up translated as delicious. According to position established along these axes, sake is considered to be of "mature type," "fragrant type," "light and smooth type," or "full-bodied type" (Fig. 3). However, no set of criteria can adequately express the multiplicity of sensations that together create the flavor unique to any individual sake, but there is a perceived need for terms which quickly and simply give the general idea.

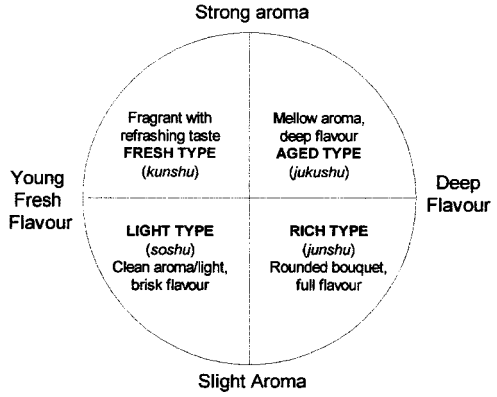

Figure 3. Main flavor types for sake characterization (43).

Sake Main Production Stages

The main stages for sake production are schematically presented in Figure 4.

Raw Materials (CCP1)

The main ingredients of Japanese sake are rice, sake rice, sake yeast and water. The rice most suitable for sake should consist of large grains and should be soft with a white part at its center, due to coarse cell structure. Rice should comply with the maximum residue limits for pesticides and insecticides established by the Codex Alimentarius Commission for this commodity (45) (CCP, chemical hazard). For Japanese sake, yellow *koji* mold (*Aspergillus oryzae*) is used. Sake yeast (*Saccharomyces cerevisiae*) is a microbe converting the occurring glucose and minerals in rice and water into alcohol. Employment of bubble-free type yeast eliminates

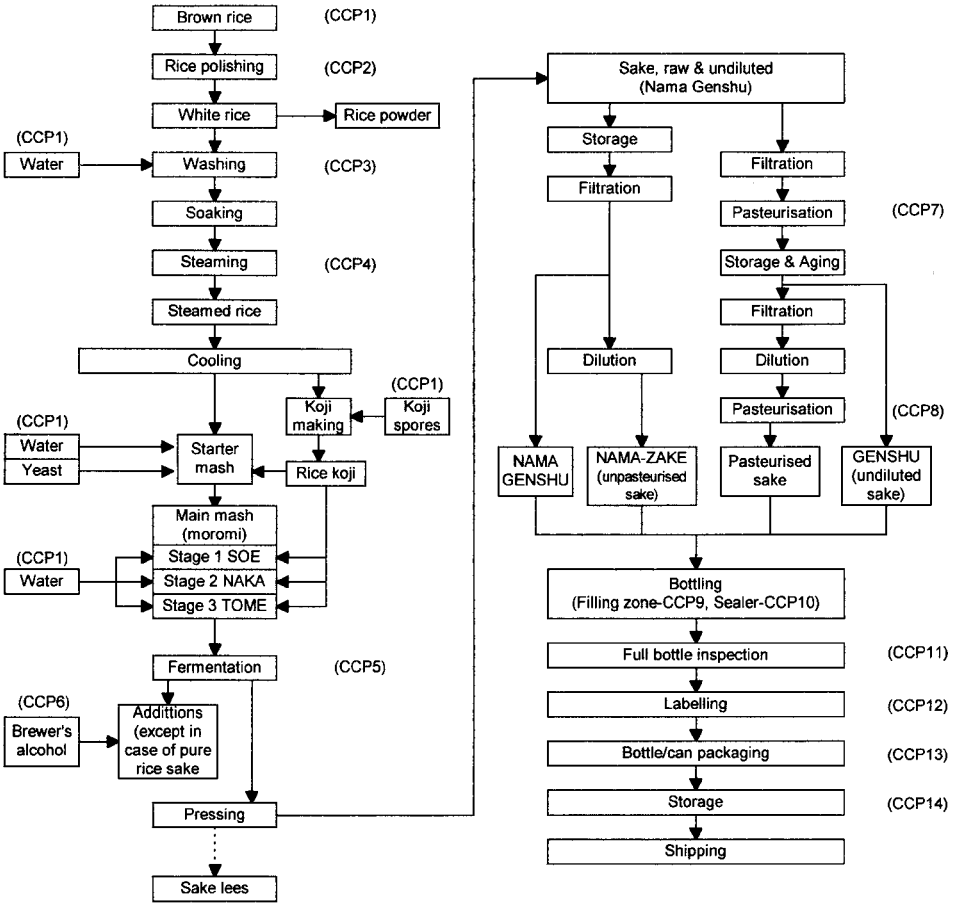

Figure 4. Process flow diagram of sake production (26,46,47).

the bubble removal step, thus shortening the brewing period and reducing the cost. Should the factory wish to employ a specific yeast, an adequate disinfection of the building interior is required, otherwise undesirable bacteria may be introduced which could prove hazardous to human health (CCP, microbiological hazard) (46).

Rice Polishing (CCP2)

The brown rice used for sake production must be first polished to remove the outer portion of the grain, which contains fats, proteins, minerals and amino acids that can cause unpleasant flavors, leaving the starch residues that are located in the center of the grain. Nowadays, machines are programmed to automatically remove whatever portion of the rice is required for the specific sake (47). The rice polishing ratio (73–35%) is expressed by the following formula (43):

$$\text{Rice polishing ratio} = (\text{weight of white rice/weight of brown rice}) \times 100 \quad (1)$$

The polishing process should be gently carried out, because friction results in heat generation, thereby greatly affecting water absorption and rice grain structure. Broken grains are unlikely to satisfactorily ferment (47). Maybe the most important stage in sake production consists of yeast starter mash production, which can take place either with the classical Kimoto or slightly revised Yamahai process, or with the new "high speed" methods (48).

Washing (CCP3)

After the rice has been polished, rice powder clinging to the grain surface is removed by washing. Washing can be carried out either mechanically or manually (laborious hand washing) and should result in removing most of the organic and inorganic impurities, reaching the CLs set by Codex Alimentarius of 1.5% and 0.1% m/m respectively.

Soaking (Steeping)

Soaking allows rice to absorb the desired amount of water that is crucial to establishing the rice consistency. For sake produced "en masse," simply dumping into a vat overnight for as long as 14 h is a usual case (47). However, high polished rice may be soaked within minutes. In such a case an error of a minute might prove to have dire consequences for the end product (43).

Steaming (CCP4)

Steaming aims at softening the rice grains and breaking down the starch molecules, thus encouraging the growth of *Aspergillus oryzae* and eliminating all

other microorganisms, leaving an initially sterile environment prone to sake mold propagation. Presence of lactic acid bacteria (LAB) and yeasts may occur at the end of this step, representing a microbiological hazard and resulting in considerable organoleptic losses. The time can vary from 20 to 60 min, depending on the brewer and apparatus employed (40–60 and 20 min for traditional and automated, respectively) (43,46).

Cooling

The ensuing division of steamed rice is mainly related to its further use. A part of it is directly cooled by air blower, whereas 20–30% is transferred to a heated culture room to be infected with bacteria spores (*Aspergillus oryzae*) for sake mold production.

Koji

Since rice grains contain no sugar, it is the action of *koji* mold that converts the starch in the grains to sugar. The steamed rice is first cooled to 15–36°C before being transferred to the *koji* culture room (30°C). Spores of the mold are sprinkled like fine dust on the rice when it has cooled down to 33°C. After the spores are kneaded into the steamed rice, the rice is heaped and wrapped in cloths to prevent heat and moisture loss, which are two crucial factors for satisfactory bacterial growth. To maintain uniform temperature and moisture, rice is spread and mixed twice, the first time after 20 hours (upon the appearance of white flecks) and then 7–8 h thereafter, accompanied by a distinctive aroma release (48).

Main Mash (Moromi) and Fermentation (CCP5)

In fermentation, the occurring chemical hazards are related to heavy metals presence (As < 0.2, Cd < 0.01, Pb < 0.3 mg/L), pesticide residues (as mentioned in Codex Alimentarius) and residues of detergents (absence) and ethylene glycole (absence). Their CLs can be determined and monitored with specific chemical analyses. The ingredients of main mash (water, *koji* rice and steamed rice) are added to the starter mash in three steps (moving from small to bigger recipient) over a period of 4 days at successively lower temperatures, thus preventing the growth of airborne bacteria (Table 2). A day after the addition of all the ingredients, formation of a moist surface showing clear cracks occurs. Furthermore, the mash begins to bubble (indication of fermentation progress) as gas is given off during the burgeoning fermentation. The fermentation can take place at various temperatures and its duration depends on it; that is, at lower temperatures it takes up to two weeks but the sake aroma is much more appealing compared to that formed at higher temperatures. The characteristic sake aroma results from combined flavor

Table 2. Quantities of Ingredients at Each Stage of Mixing the Main Mash (Moromi)

	Yeast Starter (Moto)	1st Addition	2nd Addition	3rd Addition	4th Addition[a]	Total
Total rice (kg)	210	470	850	1470		3000
Steamed rice (kg)	140		330	650	1230	2350
Koji rice (kg)	70	140	200	240		650
30% Brewer's alcohol (L)					1200	1200
Water (L)	230	420	1010	2030	360	405

[a]Traditional brewers mix the final mash in three stages. The fourth addition of alcohol and water is a controversial postwar development (Kondo, 1984).

components of a number of compounds produced during fermentation (49). The elevated alcohol content of the fermented sake is related to lipid metabolism of yeast in the presence of proteolipid provided by the *koji* molds (50,51).

Additions (CCP6)

The addition of alcohol at this stage is carried out unless it is clearly stated that sake does not contain any alcohol from extraneous sources. The added alcohol should not contain methanol, or if it does, the content of the latter should be less than 0.5 g/L because of its toxicity (CCP, chemical hazard).

Pressing

Automatic machine presses (consisting of a series of panels with balloon-like sacks attached) are most widely used nowadays instead of the traditional time-consuming method using long bags. The remained caked lees are employed for pickle production and cooking or sedimentation of rice particles may occur. Alternatively, sedimentation of rice particles at the bottom of the tank may take place.

Filtration

Coloring and aging (maturation) inhibition can be effected by using activated charcoal filters.

Pasteurization (CCP7 and CCP8)

Heating sake, preferably twice at 65°C, kills off the remaining yeast, stops enzyme action and deactivates the lactic acid bacteria that will eventually spoil sake. This process represents a microbiological hazard for which the specific plant may

set CLs. However, in recent years, refrigerated storage and transport have made unpasteurized sake, with characteristic aroma, available to the consumer (43).

Dilution

The produced sake in its raw state (Genchu) contains more than 20% alcohol by volume, but it is generally diluted to about 15–16 vol.-%.

Bottling/Storage/Distribution

The applied procedures are similar to those mentioned for the beer production.

A summary of the occurring hazards, CCPs, CLs, and preventive and corrective measures is given in Table 3.

WINE

Introduction

Wines are made from the fruit of *Vitis vinifera*, of which there are a great number of varieties growing in many parts of the world. The history of wine is inextricably interwoven with human history. It might be as true to say that it was with wine that civilization began, for the vine takes longer to mature than any other crop, and does not produce grapes for wine making until its fourth year. It is not exactly known when men first had wine, but it was accepted as a gift from the gods: the Egyptians attributed it to Osiris and the Greeks to Dionysos. Mesopotamia and the Caucasian slopes were no doubt early sources of wine from where it was spread to Egypt and Greece and then to the rest of the world (52).

Wine Main Production Stages

The main stages for wine production are schematically presented in Figure 5.

Harvesting (CCP1)

Grape harvesting is a CCP comprising both physical and chemical hazards. Physically, the grapes should be sound without rotten parts; otherwise oxidative and microbial contamination can rapidly develop. Therefore, harvesting should be conducted with the greatest possible care and an efficient disease management system should be applied (53,54). Pesticides play an important role in pest management, but they should be handled with care because they constitute chemical hazards (55). At the time of harvest, the grapes must have also reached the correct maturity when Brix and Total Acidity (TA) levels indicate maturity of wine. Since pesticide and fungicide residues on the surface of the berries constitute chemical

hazards, Oliva et al. (56) proposed a rapid and simple gas chromatographic method for their determination. The maximum residue limits for pesticides in grapes and wines are provided by Codex Alimentarius (45) and Organisation International du Vin (57). Finally, the bulk bins used for grapes transportation, should be effectively decontaminated to avoid any microbial infection.

Stemming

Stemming includes the removal of stem, leaves, and grape stalks before crushing. This procedure has several advantages because the total volume of processed product drops by 30%, thus resulting in smaller tanks, and eventually increasing the product's alcoholic content (58). However, the end of fermentation and the alcohol content of finished product depend mostly on the Brix level of initial grapes. Stemmers usually contain a perforated cylinder allowing berries to pass through but prevent the passage of stems, stalks and leaves.

Crushing

Crushing typically immediately follows stemming, since some crushing of the fruit occurs during stemming. The released juice is highly susceptible to oxidative browning and microbial contamination. The most common crushing processes involve pressing the fruit against a perforated wall or passing the fruit through a set of rollers. It is very important to avoid crushing the seeds to preclude contaminating the must with seed oils, the oxidation of which could produce rancid odors and constitute an undesirable source of bitter tannins. Equally important is the proper handling of product, because inappropriate timing might lead to a sudden start of alcoholic fermentation and consequently to higher fermentation temperatures, while a delay might cause microbial contamination and oxidative browning (59).

Maceration

Maceration is the breakdown of grape solids after crushing of grapes. While maceration is always involved in the initial stage of red wine fermentation, the long-standing trend has been to limit maceration in white wine production. Temperature and duration of maceration depend on grape and wine variety. Usually for white and rose wines the maceration time is less than 24 h, red destined for early consumption is macerated for 3–5 days and red for aging is macerated from 5 days to 3 weeks. Fermentation usually occurs during this or at the end of maceration. The amount of the antimicrobial to be used, usually added to white musts that are most sensitive to oxidation, depends on the crop health and maceration temperature. Sulfur dioxide has a distinct advantage over other antimicrobial agents, because of the relative insensitivity of the wine yeasts to its action. However, it is also toxic, or inhibitory, to most bacteria and yeasts (i.e., *Candida, Pichia, Hansenula*) at low concentrations (60) and has a rather low retention capability after the clarification step (61).

Table 3. Summary of Hazards, CCPs, CLs, Monitoring, Corrective Actions, and Personnel Responsible for Sake Production

Process Step[a]	Hazards (M, C, P)[b]	Control-Preventive Measures	CCP Parameter	Critical Limits (CLs)	Monitoring Procedures	Corrective Actions	Responsible Personnel
Incoming raw materials (CCP1)	C	Certified suppliers, efficient disease management system in use	Pesticide residues in water	MRLs as described by Codex Alimentarius	Specific chemical analysis	Rejection of specific batch Change supplier	Quality control manager
		Proper water decontamination Certified suppliers	Heavy metals presence in water	Within specifications prescribed in Directive 80/778/EC		Evaluation of the decontaminating methods	
	M	Certified suppliers, proper preparation	Microbial contamination of the culture	100% clean	Microbiological analysis	Rejection of specific batch	Quality control manager
		Proper water decontamination	Water microbiological quality	Absence of pathogens		Inspection of the equipment	
Rice polishing (CCP2)	C	Certified supplier, efficient disease management system in use	Pesticide residues in polished rice	MRLs as described by Codex Alimentarius	Specific chemical analysis	Rejection of specific batch Change supplier	Quality control manager
Washing (CCP3)	P	Certified suppliers, installation of automatic separator	Animal impurities Other organic and inorganic mater	0.1% m/m 1.5% m/m 0.1% m/m	Specific examination	Rewashing of specific batch, change supplier	Quality control manager
Steaming (for unpasteurised sake) (CCP4)	M	GMP, scheduled microbiological controls	Presence of yeasts and LAB	Set by the specific plant	Microbiological analysis	Specific batch reprocessing, CIP standar-disation	Quality control manager, Trainned personnel

Fermentation (CCP5)	C	Material control, GMP corrosion checks	Heavy metal presence Pesticide residues	As < 0.2, Cd < 0.01, Pb < 0.3 (mg/L)	Specific chemical analysis	Demetallisation Change supplier Rejection of specific batch	Quality control manager
		GMP, use of nontoxic glycole	Residues of ehtylene glycole & detergents	0	Specific chemical analysis	Dilution with large quantities, machinery modification	
Alcohol addition (CCP6)	C	Certified supplier	Methanol content	<0.5 g/L	GC examination	Rejection of specific batch	Quality control manager
Pasteurization (CCP7 & CCP8)	M	Running of pasteuriser according to program	Detection of yeasts, LAB % enzymatic activity	Set by the specific plant	Microbiological analysis	Temperature adjustment, batch reprocessing, proper machinery disinfection	Quality control manager Technical manager

[a]Regarding the procedures of bottling, storage and distribution, the CCPs are similar to those mentioned in Table 1 for beer production.
[b]M, C, P stand for microbiological, chemical and physical hazards, respectively.

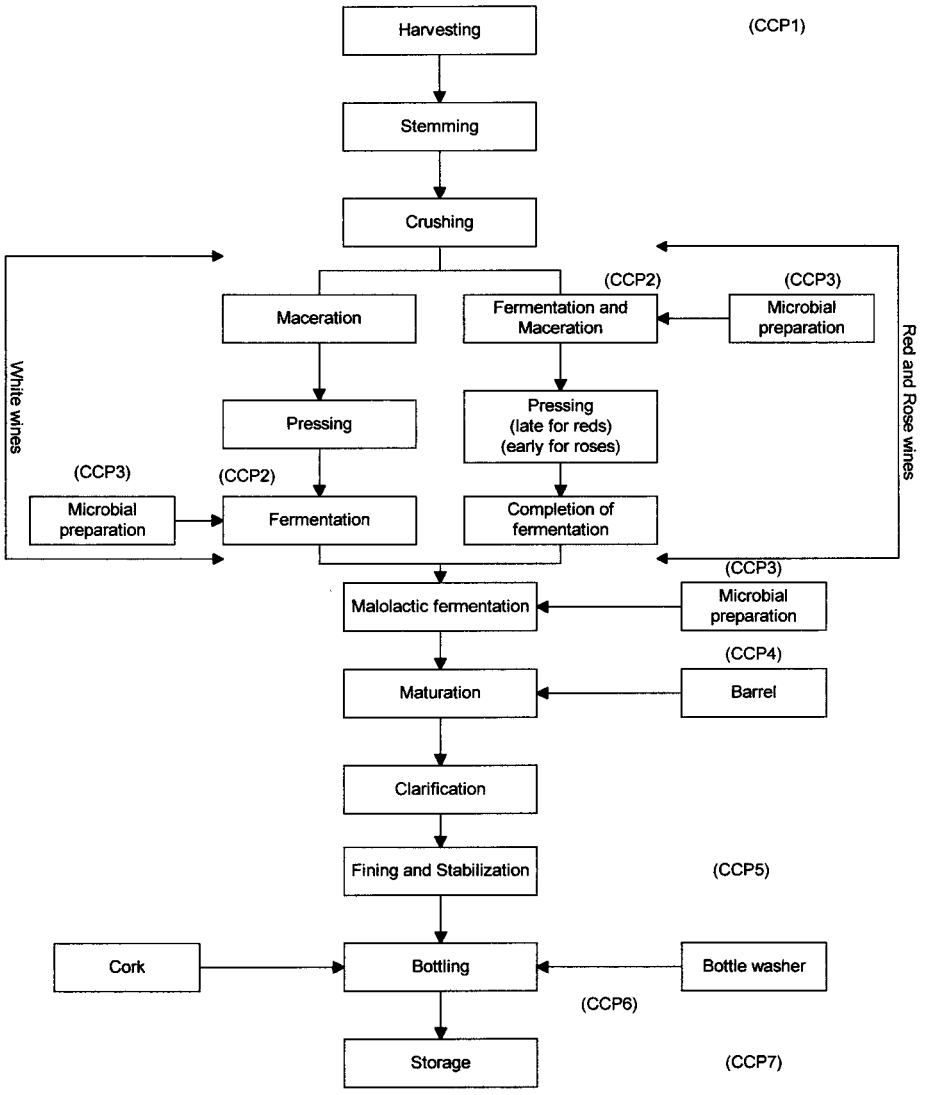

Figure 5. Process flow diagram of wine production (35,52,58).

Pressing

The must is allowed to remain in the press for several minutes, during which juice runs out under its own weight. Depending on the press type (horizontal, pneumatic, continuous screw presses), the produced juice and wine fractions vary in terms of their physicochemical properties. Combining different wine fractions, the winemaker can influence the character of the wine. However, a potential hazard might be the occurrence of oxidation reactions if there is a delay in the process (52).

Alcoholic Fermentation (CCP2)

Alcoholic fermentation is usually carried out by strains of *Saccharomyces cerevisiae* because this species is remarkably tolerant to high sugar, ethanol, and sulfur dioxide concentrations and also grows at low pH values typical for grape must (pH 3.2–4). The culture of *Saccharomyces cerevisiae* is either part of the indigenous microflora or may be partially added to achieve a population of about 10^5 to 10^6 cells/ml in the must (CCP3, microbiological hazard) (62). Possible contamination of must with killer yeasts (a property mainly present in wild strains of *Saccharomyces* but also in other yeast genera such as *Candida, Debaryomyces, Hansenula, Kluyveromyces, Pichia, Torulopsis and Cryptococcus*) may result in stuck fermentation (63). Attention should be paid to the added amount of sulfur dioxide (total SO_2; 175 and 225 mg/L for red and white wine, respectively) in order to inhibit, if not to kill, most of the indigenous yeast population of grapes (64), as well as acidity adjustment, and to sugar and tannin concentration of the juice.

In fermentation the encountered chemical hazards consist of heavy metals presence ($As < 0.2, Cd < 0.01, Cu < 1, Pb < 0.3$ mg/L), methanol content (300 and 150 mg/L for red and white wine, respectively), ethyl carbamate content, pesticide residues (as mentioned in the Codex Alimentarius) and residues of detergents (absence) and ethylene glycol (absence). CLs may be established and monitored with specific chemical analyses. Special attention should be paid regarding the ethyl carbamate content, because there is no legislative action against it in Europe, contrary to the United States (<15 ppb and <60 ppb for table and desert wines, respectively) and Canada (30 ppb and 100 ppb for table and desert wines, respectively). The latter is formed from reaction of alcohols with substances rich in nitrogenous compounds, mainly urea and aminoacids like arginine and citruline. Its control is carried out with gas chromatography and its prevention can be accomplished by avoiding intensive organic fertilization of vines, high temperatures at the end or after the alcoholic fermentation, using yeast cultures tested for low urea and ethyl carbamate production, employing urease, and determining urea when long storage is intended and carried out. The fermentation temperature is one of the most crucial factors affecting yeast metabolism both directly and indirectly. For white and red wines the desirable temperature varies within the range of 8–15°C and 25–28°C, respectively. Any presence of residual sugars (i.e., sucrose, glucose, fructose) by the end of fermentation is a hazard that might cause microbial destabilization of wine. The fermentation process requires no oxygen. Nevertheless, traces of oxygen at the beginning of the exponential phase of yeast growth speed up the fermentation because the yeast population increases and the average cell viability prolonged. The pH might affect the process only at extreme values (<3.0) where the growth of fermentative yeasts is inhibited (59).

Finally, the fungicide residues in the must might play an inhibitory role in the yeast's growth and undermine the sensory qualities of the wine by affecting biosynthetic pathways (65–67).

Malolactic Fermentation

Early onset and completion of malolactic fermentation allows the prompt addition of sulfur dioxide, storage at cool temperatures, and clarification. It is conducted by lactic acid bacteria (*Oennococcus oenos*), which directly decarboxylate L-malic acid (dicarboxylic acid) to L-lactic acid (monocarboxylic acid). This metabolism results in acidity reduction and pH increase, which are in turn related to an increased smoothness and drinkability of red wines but might also generate a flat taste (68,69). The initial pH, the sulfite concentration (70), the phenolics and the anthocyanin content (71) of juice/wine strongly affect whether, when, and how (with what species) malolactic fermentation will occur. Bacterial viruses (phages) can severely disrupt malolactic fermentation by attacking the *Oennococcus oenos* cells, thus causing microbial destabilization of wine (72). Therefore, to assure the development of malolactic fermentation, winemakers inoculate the wine with one or more strains of *Oennococcus oenos* (CCP3) (73,74). After fermentation, the wine's desirable total acidity is generally considered to vary within the range of 0.55–0.85% (white and red wines toward the upper and lower end, respectively). Whenever, the total acidity surpasses those limits, acidification and deacidification techniques should be in place (35).

Maturation (CCP4)

The maturation step often lasts 6–24 months and takes place in oak barrels. During maturation a range of physical and chemical interactions occurs among the barrel, the surrounding atmosphere, and the maturing wine, leading to transformation of flavor and composition of wine (75). Here there is a CCP concerning the oak barrel, which should be fault-free and should have undergone a decontamination treatment. The wood also must be free of pronounced or undesirable odors, which could taint the wine (76). During the maturation period, several components of the wood (most of them phenolics) are extracted to the wine tannin (77,78). Since oak tannins can significantly add to the bitter taste of wine, white wines are usually matured in oak for shorter periods than red wines, and in conditioned barrels to release less extractable (79,80). Another CCP is related to the inhibition of the oxygen penetration through wood or during racking and sampling of wine. Although a slight oxidation is desirable, a more extensive one can cause various sensory changes, such as oxidized odor, browning, loss of color in red wines, activation of spoilage bacteria and yeasts, development of ferric casse, and precipitation of tannins (81). Limits on free and total SO_2 levels in finished wine are variable from country to country.

Clarification

Clarification involves only physical means of removing the suspended particulate matter. Juice clarification by racking, centrifugation or filtration often

improves the flavor development in white wine, and helps the prevention of microbial spoilage. If sufficient time is provided, racking and fining can produce stable, crystal clear wines, but now that early bottling in a few weeks or months after fermentation is employed, centrifugation and filtration are used to obtain the required clarity level (82). Microbial contamination of wine during the above mentioned procedures constitutes a potential problem for its stability (83). Racking is also effective on pesticide residue reduction of wine (84).

Stabilization (CCP5)

The reason for stabilization is production of a permanently clear and flavor fault-free wine. The most important procedures include a) tartrate stabilization by chilling the wine to near its freezing point and then filtering or centrifuging to remove the crystals, b) protein stabilization with absorption, denaturation, or neutralization by fining agents (bentonite) (85), c) polysaccharide removal with pectinases that hydrolyze the polymer, disturbing its protective colloidal action and filter plugging properties (82), and d) metal casse (Fe, Cu) stabilization. Ferric casse is controlled by the addition of agents (bentonites, proteins) controlling the flocculation of insoluble ferric complexes, whereas wines with copper content greater than 0.5 mg/L are particularly susceptible to copper casse formation (86). Legal residual copper levels in finished wines are variable and not all methods for copper removal are approved in all countries. In particular, all wine industry federal regulations for the US industry can be accessed via the Bureau of Alcohol, Tobacco and Firearms (BATF) (available at http://www.atf.treas.gov).

Bottling (CCP6)

Wine is bottled in glass bottles sealed with cork. The bottles must pass a decontaminating step and an inspection control to assure the absence of any defects and the stability of the product until its consumption (87). The cork should be correctly sized, 6–7 mm bigger than the inner neck diameter, to avoid any possible leaks. In bottling all three hazards may be encountered. In particular, cork microflora, residues of heavy metals, SO_2, pesticides and detergents, and absence of cracks, scratches and rifts in the lute represent microbiological, chemical and physical hazards. Although cork is noted for its chemical inertness in contact with wine, it might cause off-flavors when contaminated (88,89) or when the producers are not applying effective quality control (90). The CL for cork is absence of LAB and yeast, which can be assured with microbiological analysis. When long storage of wine is anticipated, longer and denser corks are preferred, because prolonged exposure slowly affects the cork integrity. Since on compression a plunger forces the cork down into the neck of the bottle, precaution must be taken against the buildup of microbes within the equipment (91,83), the lead transfer to wine through

the wine-cork-capsule system (92), and the oxidation during filling by flushing the bottles with carbon dioxide. Cork insertion may also occur under vacuum. The headspace oxygen might affect the product quality by causing the disease of the "bottle." The CL for SO_2 is 175 and 225 mg/L for red and white wine, respectively, for As < 0.2 mg/L, Cd < 0.01 mg/L, Cu < 1 mg/L, Pb < 0.3 mg/L, the residues of pesticides and insecticides in the final product are provided by Office International de la Vigne et du Vin (57).

Storage (CCP7)

Shipping and storage of wines at elevated temperatures can initiate rapid changes in color and flavor of wine. Direct exposure to sunlight corresponds to the effect of warm storage temperatures. Temperature affects reaction rates involved in the maturation, such as the acceleration of hydrolysis of aromatic esters and the loss of terpene fragrances (93). Temperature can also affect the wine volume and eventually loosen the cork seal, leading to leakage, oxidation, and possibly microbial formation resulting in spoilage of bottled wine.

The occurring hazards, CCPs, CLs, preventive and corrective measures are given synoptically in Table 4.

DISTILLED SPIRITS

Introduction

Distillation is one of the earliest examples of implementation of chemical technology. The process was known in China many hundred years before the birth of Christ and the first distilled beverage is believed to have been made from rice about 800 B.C. The first few years A.D., the Arabs learned the technology and from them, distillation was introduced to Western Europe (25). The spirit distillation industry comprises a heterogeneous assortment of manufacturing processes linked by yeasts as a common function. Distillery spirits are available in many forms, varying from pure alcohol to complex potable spirits. Nevertheless, they are all based on the same biochemical and physical principles and similar manufacturing stages (18). Gin and vodka typify non-cogeneric spirits. In the case of gin, the spirit is flavored with juniper and other "botanicals," while with vodka, the flavor is modified by filtration through charcoal. Both distillates can be produced from the several grains or potatoes, fermentation depending essentially on consistency and reliability of supply and quality and on economics and on the plant available (13). Ouzo, the most popular distilled spirit consumed in Greece, is traditionally manufactured from wine distillation. Its characteristic aroma and flavor are attributed to anethol, the main constituent of anise seed (94). Brandy is a spirit distilled from wine and is produced in all viticultural regions. In terms of quality the best-known brandies are Cognac and Armagnac. Both of these brandies are produced by distillation of white wine from geographically defined regions of France.

Table 4. Summary of Hazards, CCPs, CLs, Monitoring, Corrective Actions, and Personnel Responsible for Wine Production

Process Step	Hazards (C, M, P)[a]	Control-Preventive Measures	CCP Parameter	Critical Limits (CLs)	Monitoring Procedures	Corrective Actions	Responsible Personnel
Harvesting (CCP1)	P	Careful handling of grapes	Sound fruit without rotten parts	Reduced to acceptable level	Inspection during harvesting	Instruct personnel	Trained personnel
	C	Specify the last day of applying pesticides	Pesticide residues	Per pesticide according to Codex Alim.	Specific chemical analyses	Delay of harvesting date	Quality control manager
Fermentation (CCP2)	C	Material without heavy metals, corrosion checks	Heavy metals presence	As < 0.2, Cd < 0.01, Cu < 1, Pb < 0.3 (mg/L)	Specific chemical analyses	Rejection of specific batch, demetallisation	Quality control manager
		Certified suppliers, control of the product	Pesticide residues	Per pesticide according to Codex Alim		Rejection of specific batch	
		Careful maintain the equipment, use of non-toxic glucole GMP	Residues of ethylene glycole & detergents Methanol content	Absence 300 mg/L (red), 150 mg/L (white & rose)		Rejection of specific batch, dilution with large quantities, machinery modification	
		Avoid intensive fertilization Avoid high temperatures Use proper yeast cultures Employ urease	Ethyl carbamate formation	<15 (30) and <60 (100) ppb for table and desert wines in USA (Canada), respectively	Gas chromatography	Rejection of specific batch, dilution with large quantities	
Bacterial preparations (CCP3)	M	Certified suppliers, strictly following instructions	Microbiological contamination	100% clean	Microbiological analyses	Change supplier or method of preparation	Quality control manager

(*continued*)

Table 4. *Continued.*

Process Step	Hazards (C, M, P)[a]	Control-Preventive Measures	CCP Parameter	Critical Limits (CLs)	Monitoring Procedures	Corrective Actions	Responsible Personnel
Maturation (CCP4)	M	Certified suppliers, proper barrel decontamination	Microbiological contamination	Absence of yeasts, molds and lactic acid bacteria	Microbiological analyses	Rewash the barrel	Quality control manager
Stabilization (CCP5)	C	GMP, materials without heavy metals, calculation of ferrocyonide needed according to Fe present	Heavy metals presence	As < 0.2, Cd $<$ 0.01, Cu < 1, Pb < 0.3 (mg/L)	Specific chemical analyses	Rejection of specific batch, demetallisation	Quality control manager
			Residual ferrocyonide	Fe: 5 mg/L		Filtration or dilution with larger quantities	Quality control manager
Bottling (CCP6)	C	GMP, materials without heavy metals	Heavy metals presence	As < 0.2, Cd $<$ 0.01, Cu < 1, Pb < 0.3 (mg/L)	Specific chemical analyses	Rejection of specific batch, demetallisation	Quality control manager
		Certified suppliers, control of the product	Pesticide residues	By pesticide according to Codex Alim		Rejection of specific batch	
		GMP, avoidance of high doses	Detergent and SO_2 residues	None 175 mg/L (red), 225 mg/L (white, rose)		Modification of the CIP, rejection of batch	
	B	Inspection and screening of the bottling area	Insect presence in the full bottles	None	Visual inspection	Disinfect the area, rejection of specific batch	Trained personnel

	P	Certified supplier, continuous inspection	Bottle condition	Absence of rifts in the lute, cracks, scratches,	On-line visual inspection	Rejection of faulty bottles	Trained personnel
		Certified supplier	Cork sizing	Proportional to the bottle	Sample measurements		
	M	Certified supplier, establishment of decontamination processes	Cork microflora	Yeast, LAB absence	Microbiological analyses	Rejection of faulty corks, decontamination process	Quality control manager
Storage (CCP7)	P	Control storage conditions and retail stores	Wine quality	Set by each plant	Organoleptic controls	Rejection of faulty batches	Trained personnel

[a]C, M, P symbols stands for chemical, microbiological and physical hazards, respectively.

Distilled Spirits Main Production Stages

The main stages for the production of the above mentioned distilled spirits are shown schematically in Figure 6.

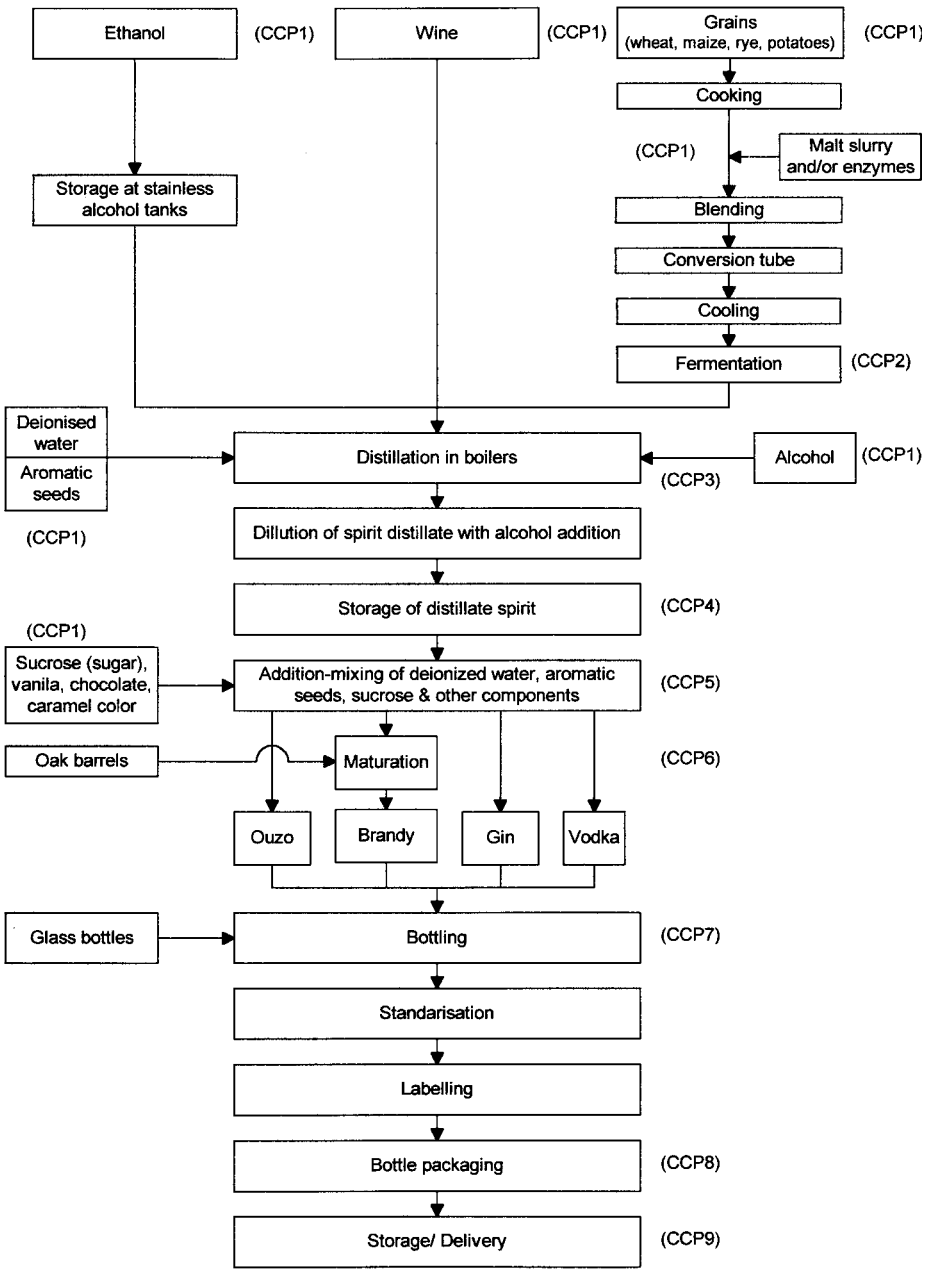

Figure 6. Process flow diagram of distilled spirits production (25,97).

Incoming Raw Materials (CCP1)

Incoming raw materials, such as alcohol, aromatic seeds (anise), sucrose, and glass bottles, reach the corresponding department of the factory in large containers. All materials are purchased against specifications agreed with the certified suppliers who are inspected, reviewed and assessed annually on basis of quality and availability of their raw materials. The wine used for ouzo and brandy production should comply with parameters of the finished products mentioned in Table 4. Alcohol is usually delivered in batches by large tankers consisting of one, two or three separate tanks. Alcohol must be of at least 96 vol.-% alcohol, free of volatile compounds that may affect the aroma of anise (*Pimpinella anisum*), having a methanol concentration lower than 0.5 g/L. Qualitative and quantitative measurements of each alcohol sample are taken by gas chromatography (GC). The grains should comply with pesticide and heavy metal residues set by Codex Alimentarius and national legislation, and they should also be mycotoxin-free, as earlier mentioned in the brewing section. Flavourful seeds are sampled and undergo microbiological and chemical analysis for *E. coli, B. cereus, Cl. perfrigens* and toxic metals as As, Cd, Hg. Microbiological control is based on prescribed instructions, including visual examination for undesirable mold or any other bacterial development and count after incubation of *Escherichia coli* (CCL $= 10^3$ cfu/g), *Bacillus cereus* (CCL $= 10^4$ cfu/g), and *Clostridium perfrigens* (CCL $= 10^3$ cfu/g). Chemical control includes toxicological analyses for high concentration levels of toxic or heavy metals, such as As (CCL = 10 mg/kg), Cd (CCL = 1 mg/kg), and Hg (CCL = 1 mg/kg) as well as the congealing and melting point of the essential oil anise (95). Other quality control tests could comprise specific gravity tests, refractive index, optical rotation and solubility in alcohol (96). Anethol, the main component of anise, should also undergo chemical analysis by GC, to ensure that its concentration in *cis*-anethol (toxic isomer) lies below 1%.

Cooking

This stage concerns solely the gin and vodka production from grains or potatoes. Cooking is required for maize and other cereals, as well as for potatoes. Batch or continuous cookers can be used and premalting is common practice. Malt is traditionally used for the conversion of starch to sugars, but has no role in flavor. Continuous cooking processes can be extended to include conversion. This involves cooling the cooked grain, adding malt slurry and blending, before passage to a conversion tube. A residence time of 10 min is sufficient for amylolysis to reach equilibrium. The mass is then cooled and transferred to the fermentation vessel. The most widely used enzymes are heat stable α-amylase and amyloglycosidase. The most efficient use is addition of α-amylase at 80°C, followed by amyloglycosidase at 55–60°C (25). The cooking stage requires careful control of temperature and pressure. The efficiency of conversion depends on concentration of grist, pH, and water composition.

Fermentation (CCP2)

Yeasts are selected in terms of their satisfactory performance in the particular type of mash used. The main criteria are fast fermentation rate, high ethanol yield, high ethanol tolerance and ability to ferment carbohydrates at relatively high temperatures. Overheating can be a serious problem and temperatures in the fermentation vessels must be carefully controlled. An infection-free yeast is also required for this stage (CCP). For this particular stage the CCPs are similar to those mentioned for wine production in Table 4.

Distillation (CCP3)

Alcohol of 96 vol.-%, deionized water, and flavorful seeds (anise, gum, etc.), wine or fermented grains are fed into the boilers at concentrations prescribed by the formulation for large-scale ouzo production, traditional production of ouzo and brandy, gin and vodka, respectively. Distillation is carried out within the range 63–80°C for 10 to 12 h. The percent alcohol volume of the final distillate amounts to about 5% v/v At this step a potential chemical hazard is the formation of ethyl carbamate, as mentioned in wine production. The CL for ethyl carbamate is different per product (i.e., 150 ppb for wine distillates, 400 ppb for fruit brandies, 60 ppm for rum, 70 ppm for sherry). Since inadequate thermal process might result in a possible microbiological hazard, on-line inspection of the thermal processing conditions and microbiological examination of the distillate are indispensable. Moreover, the distillate must satisfy the prescribed standards for the incoming alcohol (97). Were considerable deviations to be observed, the responsible person would need to order the redistillation or the rejection of the batch. Chocolate used for brandy production undergoes both physical control (microscopy, naked eye observation) for the inspection of presence of foreign materials and microbiological examination for *E. coli* (less than 10^3cfu/g) and *B. cereus* (CCL = 10^4 cfu/g) (98,99).

Dilution of Distillate with Alcohol Addition

The produced distillate has a high concentration of flavorful compounds and is diluted by adding alcohol of 96 vol.-%, thus resulting in a minimum concentration of distilled alcohol of 40% in the final product, in agreement with current legislation for ouzo production (95).

Storage of Spirit Distillate (CCP4)

The diluted distillate is transferred into stainless steel tanks, where it is stored for about 10–15 days stirred continuously so that all components are adequately dissolved. The concentration of *cis*-anethol should be accurately controlled by

Table 5. Summary of Hazards, CCPs, CLs, Monitoring, Corrective Actions, and Personnel Responsible for Distilled Spirits Production

Process Step	Hazards (M, C, P)[a]	Control-Preventive Measures	CCP Parameter	Critical Limits (CLs)	Monitoring Procedures	Corrective Actions	Responsible Personnel
Incoming raw materials (CCP1)	M	Control of storage conditions, Certified suppliers	*E. coli, B. cereus, Cl. perfrigens*	10^3, 10^4, 10^3 cfu/g respectively	Visual control for mold presence and microbiological control	Rejection of batch Change storage conditions	Quality control manager
	C	Certified suppliers	Toxic metals presence (Greek Food codex)	As < 1, Pd < 10, Cd < 1, Hg < 1 (mg/Kg)	Toxicological control with AAS	Change supplier	
			Methanol content in wine, alcohol, fermented grains	<0.5 g/L	Chemical analysis	Change supplier, Dilution with large quantities	
Distillation (CCP3)	M	GMP, control of distillation procedure, frequent cleaning	*E. coli, B. cereus, Cl. perfrigens*	10, 10^4, 10^3 cfu/g respectively	Microbiological control	Rejection/ redistillation of specific batch	Production manager
			Temperature and distillation time	63–80°C for 10–12 h	Time-temperature on-line monitoring		
	C	Urea determination Use proper yeast cultures	Ethyl carbamate formation	150 ppb,wine distillate 400 ppb,fruit brandies 60 ppm, rum 70 ppm, sherry	Gas chromatography	Rejection of specific batch, dilution with large quantities	
Storage of distillate (CCP4)	C		Content of total anethol in cis-anetol	<1%	HPLC analysis	Recall of specific distillate batch	Quality control manager

Addition of deionized water (CCP5)	C	Frequent control on the system in use GMP	1.Water quality	Within specifications prescribed in Directive 80/778/EC	Chemical and toxicological analysis with AAS	1.Pause of water flow and analysis of one or more samples	Quality control manager
		Use of deionizer	2.Electrical conductivity	<20 ms/cm	Continuous recording of deionizer	2.Automatic discontinuation of the deionizer	
Bottling (CCP7)	P	Supplier certificate	Bottles proper for foods and drinks, bottles condition	Absence of undesirable foreign materials & particles, rifts in the lute, cracks or scratches	On-line visual control empty and full bottle	Rejection of faulty bottles	Trained personnel
Bottle packaging (CCP8)	P	GMP, Testing of the machinery	Appearance of bottles	Absence of defects & correct labeling	On-line visual control	Rejection of faulty bottles and standardization of the equipment	Trained personnel
	C		Detergent remains	Complete absence	Chemical analysis	Inspection of CIP system	Quality control manager
Storage (CCP9)	C	Proper storage conditions	Alteration of organoleptic properties	Set by each plant	Organoleptic analysis	Rejection of faulty batch, Moderate storage conditions	Trained personnel

[a]M, C, P stands for microbiological, chemical, and physical hazards respectively.

HPLC. The CCL for *cis*-anethol is 1% of total anethol. In case of deviation, the specific batch distillate should be recalled.

Addition of Deionized Water (CCP5)

The stirred product is transferred into tanks, where the final product is prepared. Deionized water, aromatic substances (anethol or juniper) and sucrose are added in ratios, according to formulation, and the mixture is continuously stirred. The deionized water must comply with the standards as defined by Directive 80/778, where the CCL for electrical conductivity is 20 ms/cm and water conductivity values are monitored on-line.

Maturation (CCP6)

Unlike the other spirits mentioned, several brandies are aged for certain period in wood barrels. Aging involves several processes: complex phenolic substances as tannins are extracted from wood, structural molecules are depolymerised and extracted to the distillate, and reactions may occur between components of wood and distillate (100). These chemical reactions are very important for the organoleptic quality of the final products, which depends on composition of wood, different treatments in the manufacture of oak barrels and history of the oak barrel (76,101). Especially for brandy, the presence of scopoletin (determined with HPLC) is considered as a proof of maturation in oak barrels (101). The CL for this step is the same as mentioned for wine in Table 4.

Bottling (CCP7)

The end product is filtered and then pumped into filler machines. The bottles to be used must be supplied by certified suppliers and undergo a washing step (sterilization) and on-line visual control for the detection of undesirable foreign materials, particles, rifts in the lute, cracks or scratches. If any physical defects are detected, the bottles are rejected (CCP). Once the bottles are filled they are transferred to the sealing machine, which functions by exerting air pressure onto the heading of the bottle. The sealed bottles move to the standardization machine where a code number is printed, containing information about production time and the serial number of the tank where the final product was prepared. The code number is very important and useful for traceability reasons, such as possible recall of a certain batch of bottles, external audits and company internal control.

Labeling

Bottle labeling is carried out with a machine that heats and spreads the adhesive upon each label. Another automatic machine presses labels on the surface of bottles.

The label of the beverage should be in accordance with the principles of the Codex Stan 1–1985 (Rev. 1–1991) of the Codex Alimentarius (102).

Bottle Packaging (CCP8)

Bottles are packaged into paperboard boxes of various sizes, according to the dimensions of the bottles. The encountered hazards can be of physical, chemical, and microbiological origin (CCP). Visual control before packaging can assure that no defective bottles leave the plant. Chemical and microbiological control must be carried out to assure the efficiency of cleaning in place system (CIP) and to check the possibility of cross-contamination due to the remains of washing solutions.

Storage Distribution (CCP9)

During their storage and distribution the bottles of ouzo/brandy should be kept away from sunlight, that might affect their organoleptic properties (103). The occurring hazards, CCPs, CLs, control (preventive) and corrective measures and responsible personnel are summarized in Table 5.

CONCLUSIONS

The implementation of HACCP system to the drinks industry has been of a tremendous help in terms of providing the required assurance for worldwide trade expansion. Although the alcoholic beverages are comparatively safer than other foods and drinks because of their high alcohol content, identification of potential hazards and resumption of preventive and corrective actions (whenever required) is of primary importance. Establishment of critical control limits in conjunction with appropriate and effective monitoring procedures carried out by responsible personnel have managed to minimize the outbreaks of incidents that are hazardous and pernicious for human health.

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