Chapter 6

Advances in Distilled Beverages Authenticity and Quality Testing

Teodora Emilia Coldea, Elena Mudura and
Carmen Socaciu

Additional information is available at the end of the chapter

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Abstract

Given the advent of the consumers and producers demands, researches are focusing lately to develop innovative, cost-effective, progressively complex alcoholic beverages. As alcohol consumption has a heavy impact on social environment and health, fast and safe solutions for industrial application are needed. In this chapter, the recent advances in the field of alcoholic beverages authenticity and quality testing are summarised. Solutions for the online monitoring of the process of distilled beverages are offered and the recent methods for identification of raw material and process formed biomarkers of distilled beverages are presented.

Keywords: distilled beverages, authenticity, biomarkers

1. Introduction

Distilled beverages are important for consumers, producers and agricultural sector. Last decades presented us continuously changed requirements and descriptive practices for high level of consumer's protection with impact on the market transparency and fair competition. Both traditional methods and innovative technologies applied in distilled beverages production are focusing on their quality improvement.

The principal requirement set for an alcoholic beverage can be summarised as: are intended for human consumption, have specific sensory properties, with a minimum ethyl alcohol content of 15% v/v produced either by distillation with addition of flavourings, of naturally fermented products, or by addition of plant ethanol macerates, or by blending of flavourings, sugars, other sweetening products, or other agricultural origin products. Spirit drinks can also be produced by blending of different spirit drinks with ethyl alcohol of agricultural origin and other alcoholic or non-alcoholic beverages.

Generally, spirit drinks can be classified as with or without extract content. The presence of flavourings, sugars or other sweetening ingredients are forbidden in rum, whisky, vodka, grain spirit, wine spirit, grape or other fruit marc spirit, fruit, cider and perry spirits. No addition of other sources of ethanol of agricultural origin and no colour improvements by the addition of caramel are allowed in fruit spirits. In the category of distilled alcoholic beverages containing extract, we can specify plant macerates based spirit drinks, gin, aquavit, aniseed-flavoured spirit drinks, bitter, liqueur and mead nectar.

The European Commission prepared a list with the specific parameters and geographical indications of alcoholic beverages in countries across Europe. For example, only for fruit spirits are recognised 70 denominations, such as *Schwarzwälder Kirschwasser*, *sliwovitz*, *eau-de-vie*, *pálinka* and *țuica*.

The European countries have an old tradition in fruit growing and valorising in traditional distilled beverages. The traditional methods used to obtain distilled beverages involve the distillation of fermented plant material (fruits or cereals) in copper stills with open fire, maturing and conditioning in oak barrels, for at least 3 months. Usually distillation is repeated twice, such as in Romania or Hungary, frequently the ethyl alcohol content ranging over 50% v/v. No matter the production process applied, the flavour and taste of these distilled beverages should indicate the origin of the raw material used. Several stages, quality testing of the raw material, its preparation and fermentation are key factors determining the distilled beverages quality, with respect to their specific bouquet. Additionally, some of the major volatiles found especially in fruit distillates, such as methanol, furfural, isobutylic alcohol and acetaldehyde have toxic potential. Of main interest for consumer's health is the amount of methanol, which is the second compound found in fruit distillates after ethanol. It is usually ingested by consumers in low doses, but can create serious problems especially in countries with high unrecorded alcohol consumption [1].

Fruit spirits, very popular worldwide, are recognised in Eastern and Central Europe as a part of tradition heritage. Are considered as therapeutic agents since Middle Ages. The most famous fruit used is plum. In Europe, the most important countries producing fruit distillates are Poland, Slovakia, Hungary, Bulgaria, Romania and Czech Republic. Are used two categories of fruits—the one with stones (genus *Prunus*—plums, cherries, sour cherries, apricots, peaches etc.) and without stones (pears, apples and other berries). Each type of fruit give specific minor volatile compounds responsible for the aroma of the distillate—alcohols, aldehydes, esters, acids and volatile phenols [2]. The quality of a spirit is strongly related to the primary flavour given by the natural aroma of the fruit, which is influenced by the geographical origin of the fruit, method of cultivation, storage or harvest period. In this chapter, we describe recent advances in the field of alcoholic beverages authenticity and quality testing and indicate solutions for risk compounds decreasing during processing, that can be applied through beverages online monitoring.

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2. Distilled beverages: health-related aspects

The beneficial effects of moderate alcohol consumption on dysfunctions of the cardiovascular system, such as coronary heart disease, associated myocardial infarction [3, 4], are diminished by the effects of alcohol to human health causing pancreatitis, diabetes, liver cirrhosis or pancreatic cancer [5–10].

It is well known the excessive alcohol consumption impact on social environment and health, recent studies showing the consequences of alcohol even to younger consumers [11–13]. European countries are confronted with a high level of alcohol consumption. With a range of 7.4% alcohol exposure of young people at the age of 15–29 represents the third major risk factor for human health causing premature death in the EU countries [14]. Actually, recent statistics place Romania as the first country in Europe for illicit alcohol consumption. Still, an exact amount of alcohol consumed cannot be given because, especially East European countries have their own tradition for homemade producing and consumption of distilled beverages [15–20].

According to epidemiological findings, the long-term consumption of alcoholic beverages is related to the occurrence of malignant tumours of the oral cavity, pharynx, larynx, oesophagus, liver, colorectal and female breast. Recent literature proved that ethanol carcinogenic mechanism is strongly linked with its transformations to acetaldehyde, already known for the carcinogenic activity.

Additionally, numerous outbreaks of alcohol poisoning, sometimes leading to fatal risk, were encountered especially with methanol, from illicit sources of alcoholic beverages. Some of the common symptoms of acute methanol poisoning are those related to hangover—headache, vertigo and vomiting, but can also cause severe abdominal pain, blurred vision or back pain. Still, its metabolites, formaldehyde and formic acid, are more harmful [21].

Furfural, a volatile compound derived from fruit carbohydrates, presents also toxicity to human organism consisting of inducing pain, sore throat, diarrhoea, vomiting and headache [16, 21]. It is formed during the improper conducted distillation, where commonly in homemade production of distilled beverages is used direct heating [2], creating harmful effects, caramel colour, with irreversible burnt-bitter taste. Acetaldehyde is formed during fermentation, by the ethanol dehydrogenation, and presents toxic effects associated with hangover-like symptoms such as nausea, sweating, rapid pulse, and headache and vomiting. It is also known as a carcinogenic compound [22, 23]. Distilled beverages, especially the homemade ones, may contain some amounts of heavy metals—such as lead or copper. Lead occurrence in distilled beverages comes from the pesticides used in agriculture and remained in ground water. When the alcohol content of traditional beverages is adjusted, usually no water analyses are made, and, as a consequence, water can become a harmful source of chemical pollution. Copper provenience in distilled beverages, namely in traditional ones, is from distilling installation or, as in the case of lead, from the pesticides used in agriculture [24]. As a positive effect, copper is key determinant in the improvement of sensory characteristics of many alcoholic beverages [25].

Recently, European Food Safety Authority (EFSA) focused on ethyl carbamate content (a derivative from hydrogen cyanide (HCN) during fruit distillates processing). Even though European Regulation (110/2008) established limits for contaminants in alcoholic beverages, sometimes the imposed limits are still exceeded. The literature presents data on advanced techniques applied for determination of HCN, formed during fermentation process, from fresh fruit, fruit juice and kernel by [26].

3. Quality parameters of distilled beverages

The most important quality parameters, which are related to the safety of distilled beverage are ethanol content, esters, aldehydes, higher alcohols, methanol, furfural and HCN. These are also specified in the EU regulation for each type of product.

Depending on the product's type, different values for ethanol content are imposed by EU regulation (min. 96% v/v in ethanol of agricultural origin; min. 37.5% v/v in vodka, fruit spirits, rum and wine spirit; min. 40% v/v in whisky).

Maximum level of methanol in ethanol of agricultural origin is 30 mg/100 mL p.a. High concentration of methanol in fruit distillates is directly related to the quantity of pectins present in fruits, which are methoxylated during fruit riping. Methanol forms when pectic substances hydrolyse under the influence of some pectolytic enzymes. As a result of pectases action, demethylation can occur releasing methanol together with pectic acid and pectol. One of the objectives of the second distillation is the concentration of methanol in overhead fraction, as to be removed and reduce its content in final distillate to a concentration in accordance with the maximum admissible levels (1200 mg/100 mL pure alc. in fruit distillates, 200 mg/100 mL p.a. in wine spirit, 1000 mg/100 mL p/a/ in cider spirit and 30 mg/100 mL p.a in ethyl alcohol of agricultural origin).

Furfural is a chemical compound that, in small amounts, contributes to the aroma and bouquet of fruit distillates, and is not allowed in ethanol of agricultural origin due to its possible toxic effect. Its health harmful effects are skin, eyes and respiratory tract irritation, headache, taste loss, skin allergies, respiratory difficulties, vomiting, thirst sensation and long exposure can affect the central nervous system, liver or blood.

The mean intake of ethyl carbamate from food is approximately 15 ng/kg bw per day, excluding the levels that come from alcoholic beverages. High levels of ethyl carbamate (ranging between 0.01 and 12 mg/L) can be found in distilled spirits, mainly in stone fruit spirits [20], depending on their origin [27]. As more than 80% of ethyl carbamate is formed, the next 4 h after distillation ends, it is important to avoid its accumulation by applying few measures: chemical elimination of cyanide in fermented juices and after distillation and the replacing of copper condensers with stainless steel ones or proper separation of heads, as HCN has a low boiling point—25.7°C [28].

Total volatile substances are a quality indicator for alcoholic beverages. The more amounts of volatile substances in fruit distillate (min. 200 mg/100 mL p.a.) or rum (min. 225 mg/100 mL p.a.) increases their quality.

Along with common analytical methods, it is imposed the necessity of some rapid, eco-friendly and cost-effective simplified alternative for alcoholic beverages assessment with applicability for both research and authorities.

4. Extraction methods applied in distilled beverages analysis

With the advent of the modern scientific revolution and the development of chemistry, alcoholic beverage sector becomes progressively complex, new ideas passing very fast from a research theme to a final market product. Many researches were conducted in this field and provide solutions with fast industrial applicability.

Given the large number of existed and daily worldwide innovated beverages, considerable progress in terms of extraction methods and analytical techniques have been made especially in the last decade. Different extraction methods such as liquid-liquid extraction (LLE), headspace solid-phase microextraction (HS-SPME), stir bar sorptive extraction (SBSE), gas-chromatography and ultra high performance liquid chromatography (UPLC) have been tested and discussed. **Table 1** summarises some of the techniques generally used for the compound extractions and the analytical methods applied for the characterisation of distilled beverages.

Type of distilled beverage	Investigated compound	Sample preparation	Analytical techniques	References
Non-aged fruit spirit	Volatile compounds	HS-SPME	GC-MS	[15, 29]
	Volatile compounds	–	GC-MS	[30, 31]
	Volatile compounds	LLE	GC-MS	[2]
	Volatile compounds	LLE	GC-O	[32]
	Volatile compounds	–	GC-FID	[16, 30, 31]
	Sensory parameters	–	Sensory analysis	[33]
	Volatile compounds	–	Ethanol, methanol (FTIR)	[2]
	Phenols	Solvent extraction	Total phenolics (UV-VIS) Antioxidant activity (DPPH, FRAP) Phenolics (HPLC)	[34]
	Metal content	–	Heavy metals (Atomic absorbtion spectrophometry)	[31, 35]
Vodka	Phenols	Solvent extraction	Total phenolics (UV-VIS) Antioxidant activity (DPPH, FRAP) Phenolics (HPLC)	[34]

Type of distilled beverage	Investigated compound	Sample preparation	Analytical techniques	References
Rum	Phenols	Solvent extraction	Total phenolics (UV-VIS) Antioxidant activity (DPPH, FRAP) Phenolics (HPLC)	[34]
Bitter	Phenols	Solvent extraction	Total phenolics (UV-VIS) Antioxidant activity (DPPH, FRAP) Phenolics (HPLC)	[34]
Bitter liqueur	Phenols	Solvent extraction	Total phenolics (UV-VIS) Antioxidant activity (DPPH, FRAP) Phenolics (HPLC)	[34]
Fruit liqueur	Volatile compounds	HS-SPME	GC-MS	[31]
	Phenols	Solvent extraction	Total phenolics (UV-VIS) Antioxidant activity (DPPH, FRAP) Phenolics (HPLC)	[34]
Aged distillate	Phenols	Solvent extraction	Total phenolics (UV-VIS)	[34, 36–39]
	Phenols	Solvent extraction	Phenolics (HPLC)	[33, 34, 36, 37, 40–44]
	Phenols	Solvent extraction	Phenolics (UPLC)	[45]
	Phenols	Solvent extraction	Antioxidant activity (DPPH, FRAP)	[33, 34]
			Antioxidant activity (ABTS)	[39]
	Colour parameters	–	Colour intensity and hue (UV-VIS)	[36]
	Sensory parameters	–	Sensory analysis	[36, 37, 46]
	Volatile compounds	LLE	GC-MS GC-FID	[46, 47]
		SPME	GC-MS	[44]
		–	GC-O	[48]
	Volatile compounds	Stir bar sorptive extraction (SBSE)	GC-MS	[49, 50]

Table 1. Some techniques used for the extraction of distilled beverages interest compounds and the analytical methods applied for their quality, safety and authenticity assessment.

5. Authenticity biomarkers

5.1. Raw material biomarkers

The quality of final beverage is strongly dependent of the quality of raw material used, variety, harvesting methods applied and storage conditions. In this context, several studies focused the chemical characteristics, volatile and microbiological aspects of vegetal matrices used in production of distilled beverages [20, 51–54]. The applied techniques in volatile compounds assessment are infrared spectroscopy [55], gas-chromatography coupled with mass-spectrometry [51, 52, 56, 57] and gas-chromatography with flame ionisation detector [53, 58].

Liquid chromatography was applied for identification and quantification of phenolic compounds in apple [53, 59, 60], nectarin, peaches and plums [61].

Free fatty acids from fruits are considered main responsible for the beverages aroma studies found [51, 62–64]. Fatty acid esters are contributors of fruit distillates flavour giving the fruity and flowery notes [2].

5.2. Biomarkers formed during fermentation process

Fermentation represents one of the most important factors for the quality and complexity of distilled beverages. Despite common laboratory methods used to test de-fermented marc quality, recent studies applied advanced methods. The quality of raw material subjected to fermentation, yeast specie and the inoculum amount, type and hygiene of the vessels [65], temperature and duration of the process are parameters intensively tested in the past years [20, 26, 28, 66, 67]. Yeast species used in fermentation have an important role in defining the final bouquet of the beverage, a gas-chromatography analysis proved [68]. Molecular techniques were used for distinguishing different types of microorganisms involved in the fermentation of cachaça [69] with impact on their volatile profile [70].

Ethanol, the first fermentation metabolite, along with high alcohols, aldehydes and fatty acids with their esters, are responsible for the flavour formation. In the case of traditional fruit, distillates are not involved in selected yeast cultures. The fermentation is carried out spontaneously, in the presence of *Kloeckera apiculata* yeast species present on fruit peels surface. Yeast microbiota species responsible for alcoholic fermentation of fruits depends especially by the region of raw material, the fermentation process applied, type of the final beverage, initial cellular concentration, temperature, pH, sulphur dioxide content and ethanol concentration. Fermentation microbiota is formed of *Saccharomyces cerevisiae* and other spontaneous species, which should not exceed 10^6–10^9 CFU/mL, in order not to inhibit the *S. cerevisiae* biological activity. During the fermentation, *S. cerevisiae* will dominate the must microbiota due to their resistance to formed ethanol. Along with *S. cerevisiae*, which represents the majority of must yeast when must ethanol concentration exceeds 5% v/v, fermentation mibrobiota is formed by *K. apiculata* (representing 98% of the viable microbiota before must exceeded 5% v/v ethanol concentration), *Rhodotorula mucilaginosa*, *R. graminis* and *Aureobasidium* sp. [66]. These spontaneous yeast species can demonstrate the authenticity of a specific fermented beverage. Contrarily, must fermentation in the presence of selected strains of *S. cerevisiae*, provide more complex volatile profile of the final distillate [71]. Even more, were found ethyl decanoate and ethyl-2-*trans*-4-*cis*-decadienoate as authenticity biomarkers for pear distillates [72]. The higher number of viable fermentation yeast contributes to more esters formation. During fermentation, are formed also eugenol (that comes from phenol or ferulic acid), acetic acid and other ethyl esters [73].

Acetaldehyde, along with ethyl hexanoate, octanoate, decanoate and dodecanoate esters, which after distillation and ageing increase, are also compounds formed during fermentation [74].

Acetaldehyde, ethyl acetate and amyl alcohols, are the main responsible of distilled beverages aroma, formed during fermentation, their amounts influencing the quality of distillates [75].

5.3. Biomarkers formed during distillation process

The influence of distillation on the distillate bouquet is decisive. Distillation influences both the products quality and safety. Unwanted fractions, considered as toxic for human organism, are separated in this step. Separation depends on the distillation procedure (simple, double with or without rectification), temperature, duration, compounds separation grade and the fermented marc quality. The right moment for distillation is very important, because influences also the distillation yield, and should be performed immediately after the fermentation ends. The way distillation is conducted strongly influences the distillate quality. Marc heating should be made gradually and uniform, in order to avoid the burning taste and smell.

By distillation, components of a homogenous liquid are separated based on their constituents boiling temperatures and vapour pressures. The most volatile components have higher vapour pressures, so it will concentrate in the vapour phase, and will form the condensate. The low volatile compounds will remain in the residual liquid. The volatile compounds formed in fermentation are extracted by distillation. Usually, are used stainless steel installation, but the traditional method includes copper alembic. Copper has an important role as reaction catalyser and formation of aromatic substances. Firstly, marc is introduced in boiling kettle, from where will be obtained the first distillate, which is subjected to a new distillation. Redistillation aims the separation of heads and tails fractions. After the second distillation, ethanol content will be range 50% v/v.

The principal compounds resulted from distillation are water, ethanol and hundreds of volatile compounds, contributors to distillate flavour. During distillation three main fractions are collected: heads, middle fraction and tails (fusel oils).

Acetaldehyde, propionaldehyde, methanol and some esters (ethyl acetate, methyl acetate and acetal) form heads fraction. They are highly toxic compounds, present unpleasant smell and taste and are separated after the first distillation. Middle fraction (ethanol content interval between 20 and 72% v/v) represents 30–45% of the entire distillate, and it contains ethanol, higher alcohols, acetals, which gives distillate specific aroma. Tails represent a high amount of volatile compounds, with high boiling point and water solubility. This fraction (10–15% of the entire amount of distillate) includes furfural, acetic acid, ethyl lactate, fatty acids with high molecular mass, fatty acids esters, volatile acids (propionic, butyric, isovalerianic and capronic) and higher alcohols (amylic, isoamylic and hexylic), and is also a strongly toxic fraction, with unpleasant sensorial properties, which is the reason for need of their separation. Heads are subjected to a new distillation, while tails are added to the marc, in order to perform the second distillation.

Many studies were performed for the identification of the different types of distilled beverages composition. A study performed in Thailand on rice distillate found volatiles identified by GC-MS have the strong odorant capacity: ethyl acetate, ethyl butyrate, ethyl decanoate, acetaldehyde, ethyl laureate, ethyl caprilate, 2-phenethyl acetate, 1-hexanol, isoamylic alcohol and 2-furaldehyde [76]. The volatile phenols identified were 4-ethylguaiacol, eugenol and 4-ethylphenol, responsible of strong floral and spicy notes, and they should be only in moderate amounts when compared to the other compounds. Grape marc distillate contains higher alcohols (1-hexanol, *trans*-3-hexen-1-ol, *cis*-3-hexen-1-ol and *trans*-2-hexen-1-ol), fusel alcohols (1-propanol, 2-butanol, isobutanol, 1-butanol, isoamyl alcohol, 1-pentanol, 1-octen-3-ol, 1-heptanol, 1-octen-4-ol, 1-octanol, 1-nonanol, 1-decanol, benzyl alcohol and 2-phenyl-ethanol), fatty acids (isobutyric, butyric, pentanoic, hexanoic, heptanoic, octanoic, decanoic, dodecanoic, tetradecanoic and hexadecanoic acids), fatty acids esters (ethyl propanoate, ethyl isobutyrate, ethyl butyrate, ethyl 2-methyl butyrate, ethyl 3-methyl butyrate, ethyl pentanoate, ethyl hexanoate, ethyl heptanoate, ethyl octanoate, ethyl nonanoate, ethyl decanoate, ethyl dodecanoate, ethyl tridecanoate, ethyl tetradecanoate, ethyl hexadecanoate, ethyl octadecanoate, ethyl linoleate and ethyl linolenate), acetic esters (ethyl acetate, propyl acetate, isobutyl acetate, isoamyl acetate and hexyl acetate), hydroxi and dicarboxy acids esters (ethyl lactate and isoamylic lactate) and other esters [77].

Moderate presence of ethyl acetate is considerate as a positive aspect due to its aroma flavour. Though, acetals in large amounts indicate a possible microbial contamination. Acetaldehyde brings hazel, cherry and overripe apple flavours.

Furfural is formed during distillation by acid hydrolysis, by fermented pentoses heating or by Maillard reactions, especially by direct heating in copper alembic. The distillation method applied influences especially the amount of esters in the final distillate, mainly considering the increasing of ethyl decanoate and ethyl palmitate [78]. When distillation columns are used, a significant increase of esters is observed, 20% more higher alcohols, less aldehyde (a decrease with 40%) and 10% less methanol, when compared to alembic distillation. Higher concentrations of methyl and ethyl acetates indicate the incorrect fractions separation (heads) during the distillation process [65]. Lactate ethyl (found in tails fraction), with unpleasant aroma, is considered a marker of the long fermented marc storage before distillation, when unwanted malolactic fermentation takes place. Fatty acids esters (caproic, caprilic, capric and lauric), with a fruity floral flavour, are formed during fermentation. Ethyl caproate presents a banana flavour, ethyl caprylate is less perfumed, and giving a grape oil flavour, ethyl caprate is less intense, while ethyl laurate gives a candle wax smell. Another source for fatty acids esters formation can be explained by the yeasts thermal degradation and autolysis during distillation.

Methanol is separated in heads and tails. Still, a small amount of methanol is separated also in middle fraction [79], depending on the fruits processing method (crushing or pressing), storage duration, fruits initial composition, pH, fermentation and distillation temperatures [65].

Higher alcohols, isoamyl alcohol and 1-propanol in the greatest amount, are known as fusel oils, having a higher boiling point than ethanol. They present a strong flavour and are retrieved in large amounts in distillate beverages, depending on the raw material and yeasts species used during fermentation, as these compounds are derived from the sugars and amino acids metabolised by yeasts [79].

Aldehydes, the most volatile compounds in distilled beverages, are formed during fermentation and are considered the main compounds resulted from the biochemical reaction when yeasts use amino acids and sugars. Acetaldehyde is the most abundant (90% of total aldehydes) and it is accumulated in heads fraction [79]. Acrolein is another important aldehyde present in heads fraction of the distillate, has a strong spicy aroma and is produced by bacteria from glycerol.

5.4. Aged distillates biomarkers

All distilled beverages need a maturation period before consumption. During maturation, a physical structuring of the ethanol and water molecules is produced and as a result, distillate becomes smoother and less pungent [80].

Biochemical process during distilled beverages ageing depends on the temperature, oxygen and the chemical composition of the ageing or maturation materials. The storage materials are very important for the distillate safety. When plastic material is used, unwanted high extraction yield of phthalate compounds was observed especially in illegally produced strong alcoholic beverages [81].

Distillates ageing can be performed in different wooden barrels or by the addition of different wood powders and other wood fragments.

Ever since OIV approved the use of wood alternatives for barrels, different methods are applied to shorten the ageing period of alcoholic beverages and enhance their phenolic and flavour profile [82]. Recently, more attention is given to the use of wooden fragments and powders for the rapid inducing ageing character of brandies [37, 46, 83–85], with reducing the lasting period between days to some weeks. The cost and difficulty of wooden barrels handling, guided actors in beverage industry to these less expensive alternatives.

Free phenolic compounds from oak wood, in contact with oxygen produce quinones. Surface wood phenols, partially transformed by quinones, dissolved in distillate and, along with quinones oxidise distillate compounds with the formation of other phenolic compounds. Furfural can be formed also during ageing process of distillate. It results by pentose oxidation by wood extraction and by wood sugar residues.

Distillate storage in wooden barrels is producing a high ethanolic extraction of wood components in distillate, especially referring to gallic acid and quercetin. The action of tanase on gallic acid produces an oxidation reaction, resulting a gold-yellow colour of the distillate. Simultaneous are produced reactions of oxidation of ethanol, higher alcohols and aldehydes with formation of acids, which react with alcohols forming esters with specific flavour.

During ageing, as interaction between wood compounds (hemicelluloses, lignin, phenolic compounds, cellulose) and distillate, are produced physical (volume decreasing, alcohol content loss, colour intensity increasing, specific mass and extract increasing) and chemical transformations (oxido-reduction processes, pH modifications, new ester, acetals and aldehydes formations). Some of the substances absorbed from wood in distillate are colour, pectic and mineral compounds, amino acids and sugars. The most important are phenolic compounds with impact on both sensory properties of the distillate and on its antioxidant activities. Depending on wood species used in cooperage, different wood-related phenols are extracted in distilled beverages (**Table 2**). Despite oak (*Quercus robur* L.), the most frequently researched woods in cooperage are chestnut (*Castanea sativa* Miller), cherry tree (*Prunus avium* L.), walnut (*Juglans regia* L.), acacia

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(*Robinia pseudacacia* L.), mulberry (*Morus alba* and *Morus nigra*), ash (*Fraxinus excelsior* L. and *Fraxinus Americana* L.), beech (*Fagus sylvatica* L.), alder (*Alnus glutinosa* L.) and lime (*Tilia cordata* Miller) [33, 86–89], although only oak and chestnut are approved by OIV for (wine) ageing [89].

5.5. Rapid authenticity testing of distilled beverages

Ethyl alcohol used for the production of spirit drinks should be of exclusively agricultural origin (EUR Lex 110/2008). A key quality parameter is the maturation and ageing periods, respectively.

Phenolic compound	Oak	Chestnut	Cherry	Mulberry	Walnut	References
Gallic acid	*	*	*		*	[82, 86, 88, 90, 91]
Vanillic acid	*	*	*			[82, 86, 88, 91, 92]
Syringic acid	*	*	*			[82, 86, 88, 91]
Vanillin	*	*	*	*		[38, 82, 83, 86, 88, 90, 92]
Ellagic acid	*	*	*		*	[82, 86, 88, 91]
Trans caffeic acid	*					[91]
Trans cafftaric acid	*					[91]
5-OH-methyl-furfural	*					[82, 83]
Furfural	*	*				[38, 82, 92]
Syringaldehyde	*	*	*			[38, 82, 86, 88, 92]
Coniferaldehyde	*	*	*			[38, 82, 86, 88]
Sinapaldehyde	*	*	*			[82, 86, 88]
Eugenol	*	*	*	*		[38, 83, 90, 92]
Methoxyeugenol	*					[90]
Bnezene derivatives				*		[90]
Trimethoxyphenol			*			[90]
Benzaldehyde derivatives			*			[90]
Protocatechuic acid	*	*	*			[86, 88, 91]
p-Hydroxybenzoic acid	*		*			[88, 91]
Ferulic acid	*	*	*			[86, 88]
Protocatechuic aldehyde	*	*	*	*		[86, 88]
Vanillic aldehyde		*				[86]
Coumarin		*				[86]
Scopoletin	*	*				[86, 88]
Vescalagin	*	*	*			[86, 88]
Castalagin	*	*	*			[86, 88]

Phenolic compound	Oak	Chestnut	Cherry	Mulberry	Walnut	References
Acutissimin		*				[86]
Glucopyranose derivatives		*				[86]
Ellagic acid deoxyhexose		*				[86]
Ellagic acid dimer dehydrated		*				[86]
Valoneic acid dilactone		*				[86]
p-Coumaric acid	*	*	*			[86, 88]
cis p-Coumaric	*					[91]
trans p-Coumaric	*					[91]
cis Coutaric	*					[91]
trans Coutaric	*					[91]
cis p-Coumaric derivative	*					[91]
trans p-Coumaric derivative	*					[91]
Methyl vanillate			*			[86]
Methyl syringate			*			[86]
Benzoic acid			*			[86]
Flavan-3-ols			*			[86]
(+)-Catechin	*		*			[86]
(−)-Epicatechin	*					[91]
B-type procyanidin dimer			*			[86]
B-type procyanidin trimer			*			[86]
Naringenin			*			[86]
Isosakuranetin			*			[86]
Eriodictyol			*			[86]
Aromadendrin			*			[86]
Taxifolin			*	*		[86]
Guaiacol	*					[83]
trans-Oaklactone	*					[83]
cis-Oaklactone	*					[83]
o-Cresol	*					[83]
Ethyl guaiacol	*			*		[38, 83]
p-Cresol	*					[83]
2,6-Dimethoxy-phenol	*					[83]
Myricetin 3-O-glucoside	*					[91]
Quercetin 3-O-glucuronide	*					[91]
Quercetin 3-O-galactoside	*					[91]

Phenolic compound	Oak	Chestnut	Cherry	Mulberry	Walnut	References
Myricetin	*					[91]
Quercetin	*					[91]
Resveratrol trans 3-O-glucoside	*					[91]
Resveratrol cis 3-O-glucoside	*					[91]
Resveratrol trans	*					[91]
Resveratrol cis	*					[91]
Resveratrol				*		[91]
Oxyresveratrol				*		[91]
Dihydroxyresveratrol				*		[91]
Viniferin	*					[91]
2,3-Hydroxy-1-guayacyl-Propan-1-one	*					[91]
Methyl gallate	*					[91]
Tyrosol	*					[91]
Ethyl gallate	*					[91]
Tryptophol	*					[91]
4-Methylguaiacol				*		[38]
Sinapic acid	*	*	*			[88]
Caffeic acid	*	*	*			[88]
Roburin A	*	*				[88]
Roburin B	*	*				[88]
Roburin C	*	*				[88]
Roburin D	*	*				[88]
Roburin E	*	*				[88]
Grandinin	*	*				[88]

Table 2. Phenolic compounds identified in wood extracts as mentioned in different references.

The addition of flavourings, sugars or other sweetening products is forbidden for distilled beverages such as rum, whisky, fruit distillates or wine brandy. The addition of caramel in fruit distillates is not allowed, and in whiskey is allowed only the plain caramel (for colouring) [93], being considered as counterfeit.

Advanced techniques for laboratory analysis of the alcoholic beverages are the chromatographic ones. The expensive reference methods tend to be replaced by simpler ones, non-destructive and easy to handle. The Fourier Transform Infrared Spectroscopy (FTIR) technique, in combination with chemometrics is a fast and reproducible way to identify the authenticity and adulteration of beverages [19, 94]. As for wine, vinegar or olive oil, distilled beverages have also geographical indication denominations. Recent studies focused the discrimination of distilled beverages based on their raw material and geographical region [2, 95–97]. Due to the degradation of methoxylated pectins found in pulp, fruit distillates will content higher amounts of methanol in comparison with other matrices (cereal ethyl alcohol). Indirectly, methanol can be considered an authenticity marker of a natural fruit brandy, indicating the origin of raw material used [16].

Author details

Teodora Emilia Coldea[1]*, Elena Mudura[1] and Carmen Socaciu[2]

*Address all correspondence to: teodora.coldea@usamvcluj.ro

1 Department of Food Technology, Faculty of Food Science and Technology, University of Agricultural Sciences and Veterinary Medicine, Cluj-Napoca, Romania

2 Department of Food Science, Faculty of Food Science and Technology, University of Agricultural Sciences and Veterinary Medicine, Cluj-Napoca, Romania

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