By some accounts, human beings have been aging distilled spirits in wooden containers for almost five hundred years. Despite billions of person-hours of experience, the myriad of chemical reactions responsible for the flavor of wood-aged spirits are not fully understood. Spirits derive their distinct characteristics over time while stored in wooden containers in part by the production and presence of esters. Esters are compounds made by chemically bonding acid molecules and alcohol molecules to form new compounds, often with pleasant aromas and tastes. This process is known as “esterification.” In addition to esterification, wood-aged spirits derive additional characteristics through other processes, including extraction of flavor compounds and sugar from the wood container (e.g., the ubiquitous oak barrel). These processes are not necessarily separate or distinct, and can interact with and affect each other.

Traditional, producing wood-aged distilled spirits has included carboxylic acid esterification, phenolic acid esterification, and the formation of complex esters, including both phenolic and carboxylic acids. Carboxylic acid esters are responsible for the fruity aromas and tastes in distilled spirits. Carboxylic precursor acids are derived from the yeast and bacteria during fermentation. If organic materials are used for the container in which these reactions occur, those materials also influence the process. For example, where a charred or toasted oak barrel is used, carboxylic precursor acids are also derived from thermal decomposition of the oak polymer, hemicellulose, found in the inner lining of the barrel. Carboxylic precursor acids are largely responsible for “off-flavors” in distilled spirits. Off-flavors are various flavorful or aromatic compounds present in spirits that are often described by connoisseurs using color terms (e.g., “sulfury”, “soyolent”, “meaty”, “acidic”, “metallic”, “vegetal”, etc.). Phenolic acid esters are responsible for spicy and sometimes fruity or smoky aromas in distilled spirits. Phenolic precursor acids are derived from yeast and smoke or burned ingredients in the fermentation, and are also derived from broken down oak polymers found within the inner lining of the barrel. Complex esters are responsible for complex honeyed aromas in distilled spirits. The complex esters are produced during fermentation or from the chemical reactions of both carboxylic and phenolic acids with alcohols during the time in the barrel or other container.

Fischer esterification of fatty acids and alcohol is a well-understood and commonly practiced chemical reaction. A typical laboratory process involves heating a solution of fatty acids and alcohols under reflux in the presence of an acid catalyst. In laboratory settings, strong acids (e.g., sulfuric acid) are typically employed as the catalyst, but this can be incompatible with spirit making where other functional groups are sensitive to stronger acids and where chemical additives are typically prohibited. It has long been known that Fischer esterification can also be completed using weak acid catalysts, but at the expense of relatively slow reaction rates. Where charred or toasted oak barrels are used during the maturation of distilled spirits, weak acids may be gradually extracted from organic material in the walls of the barrel. It typically takes years for esters to accumulate using weak acid catalysts, although it has been observed that in warmer environments (i.e., modestly heated within the range of normal atmospheric conditions less than 120° F) the process can be accelerated significantly (from decades to years).

Wood extraction is the process that gives distilled spirits their color and astringent "oaky" taste. Tannins (actually polyphenols), are one component typically extracted from organic material (typically present in the container) during the maturation process.

Tannin extraction from oak is well understood and has been practiced for hundreds of years in the leather tanning industry. It is also well understood in industries outside of distilled spirits that tannin extraction takes place faster at higher temperatures. For distilled spirits, tannin extraction is typically practiced over a period of many years as a component of the aging process. For tanning leather, however, the process is often conducted at elevated temperatures, and typically takes only 1-2 days. But, it should be noted however, that the tolerances required in tannin extraction for tanning leather are much less precise than those for distilled spirits. In addition, tannin extraction in leather tanning is typically performed with water, whereby it also serves as a delivery medium to the leather, wherein it was allowed to evaporate leaving the tannins behind. A direct application of this process is not possible in producing quality distilled spirits.

Attempts have been made to accelerate maturation of distilled spirits by cycling or varying pressures over relatively large ranges (e.g., between -2 and 10 ATM; see US Patent Publication No. 2013/0149423). These processes generally do not yield a product close enough to that produced by traditional means. Other environmental conditions are more important to achieve characteristics associated with a mature flavor.
[0010] Esterification and the extraction of wood compounds from the wood container are some of the primary reactions taking place in the maturation process of distilled spirits. Because these processes run concurrently, and often interact with or depend on each other as well as the material and other conditions of their environment over time, it is very difficult to deviate very far from traditional methods while still achieving similar results. For example, rapid tannin extraction may not provide enough time for interaction with a wood container to pick up some of the more subtle and complex flavors present in traditionally aged spirits. Much of the expense in spirit making stems from the long latency in creating the end product. Stock must be stored, often in climate-controlled environments, and tested repeatedly during maturation. It is difficult to predict markets many years out. Makers that produce too much product fail to maximize their investment, whereas those that produce too little fail to capture potentially significant portions of the upside.

[0011] Consumers of distilled spirits are often educated and discerning. Many will refuse to consume or pay a premium for non-authentic tasting products. What is needed is a means by which the quality and complexities associated with traditionally aged spirits can be achieved in a significantly reduced timeframe.

SUMMARY

[0012] In one embodiment, the disclosure provides a process for producing a spirit having characteristics associated with a matured distilled spirit. The process comprises contacting an unmatured distilled spirit with wood to form a distilled spirit mixture and maintaining the temperature of the distilled spirit mixture between about 140° F and about 170° F for a period of time ranging from about 12 hours to about 336 hours in a sealed vessel or under reflux such that evaporation of volatile compounds is prevented.

[0013] In another embodiment, the disclosure provides a process for producing a matured distilled spirit comprising: (a) obtaining a marker concentration of a chemical compound in a target matured distilled spirit thereby giving a target marker concentration, (b) contacting an unmatured distilled spirit with wood to form a distilled spirit mixture; (c) measuring a marker concentration of the distilled spirit mixture; and (d) heating the distilled spirit mixture to a temperature between about 140° F and about 170° F such that evaporation of volatile compounds is prevented until the marker concentration of the distilled spirit mixture reaches the target marker concentration.

[0014] In still another embodiment, the disclosure provides a process for producing a distilled spirit having characteristics associated with a mature spirit comprising: contacting an unmatured distilled spirit having a concentration of ethyl decanoate with wood to give a distilled spirit mixture and maintaining the temperature of the distilled spirit mixture between about 140° F and about 170° F in a sealed vessel or under reflux such that evaporation of volatile compounds is prevented. The concentration of ethyl decanoate increases by a factor of at least about 1.7X throughout the process.

[0015] Other features and aspects of the disclosure will be provided in more detail herein.

BRIEF DESCRIPTION OF THE DRAWINGS

[0016] FIG. 1 depicts an embodiment of the invention pertaining to a wooden barrel with a cutaway to show the barrel's internals.

[0017] FIG. 2 depicts an embodiment of the invention pertaining to a sealed vessel or container with a cutaway to show the vessel's internals.

DETAILED DESCRIPTION

[0018] The invention pertains to a process for producing a distilled spirit having characteristics associated with a matured distilled spirit. The distilled spirit produced in accordance with the process has many of the characteristics associated with a matured distilled spirit produced in accordance with industry standards, but is advantageously produced in a much shortened timeframe. By contacting an unmatured distilled spirit with wood at increased temperatures, the maturation process can be shortened without reducing the quality of the spirit.

[0019] As used herein, a spirit refers to any distilled spirit. In particular embodiments, the spirit is a sugar cane-based, grain-based, fruit-based, or agave-based spirit such as rum, tequila, mescal, whiskey, brandy, gin, or combinations thereof.

[0020] The process involves contacting an unmatured distilled spirit with wood under particular conditions. A distilled spirit having the characteristics associated with a matured distilled spirit, as used herein, describes a spirit, which by one or more chemical markers, has attained characteristics associated with spirits aged in accordance with industry standards. Such standards, for example, include aging in wood over a period of time greater than 1, 5, 10, 15, 20, 25 or 30 years. The characteristics associated with a matured distilled spirit may include taste, aroma and body profiles, such as smoothness. In one embodiment, a chemical marker associated with a matured distilled spirit may include the spirit's ethyl decanoate concentration. The ester concentration of an unmature spirit varies significantly from one style of distilled spirit to another. But, an increasing concentration of ethyl decanoate indicates progression of maturation. In an unmatured spirit, ethyl decanoate concentration generally ranges from about 0.5mg/liter to about 20 mg/liter. Its concentration increases by a factor of at least about 1.5X throughout the process. In other embodiments, the ethyl decanoate concentration increases by a factor of at least about 1.7X throughout the process.

[0021] Commonly, ethyl acetate concentration is used as a marker for the progress of aging where higher amounts indicate greater maturation. But, ethyl acetate is not itself desirable. Interestingly, the process described herein results in a decrease in ethyl acetate concentration from the concentration in the unmatured distilled spirit. The concentration of ethyl acetate decreases by at least about 40% to about 80% or more as a result of the process described herein. In one embodiment, the concentration of ethyl acetate decreases by at least about 50%

[0022] The unmatured distilled spirit is a spirit that has not attained the markers or characteristics associated with a matured distilled spirit. The "unmatured distilled spirit," as described herein, refers to white or raw spirits, as well as partially matured spirits, provided that the unmatured distilled spirit is lacking in certain characteristics of a matured spirit.

[0023] The term "distilled spirit mixture," as used herein, refers to any distillate on the spectrum from matured to unmatured. The term "distillate" as used herein refers to the liquid composition in the vessel and may include an unmatured distilled spirit, a matured distilled spirit, or a distilled spirit mixture.

[0024] The unmatured distilled spirit is contacted with wood. In one embodiment, the wood is provided as the vessel for holding the distillate. In such embodiments, the vessel is, for example, an oak barrel. In other embodiments, wood may be provided to the inside of the reaction vessel, which is optionally made of wood. For example, wood chips may be added such that they are submerged or floating on top of the distillate. Wood may also be provided as various structural configurations within the vessel including as baffles or packing. In still other embodiments, wood may include wood extracts and raw acids designed to mimic the characteristics of wood. In a preferred embodiment, the wood is oak.

[0025] The unmatured distilled spirit is contacted with the wood at a temperature ranging from about 140° F to about 170° F. In alternate embodiments, the temperature ranges from about 140° F to about 150 °F, from about 145° F to about 150° F, from about from about 150° F to about 160° F, or from about 160° F to about 170 °F.

[0026] The time period needed to convert the unmatured distilled spirit to a distilled spirit having characteristics associated with a matured spirit...
starting composition of the unmatured distilled spirit and the temperature at which the process is conducted. In some embodiments, a temperature between about 140°F and about 170°F is provided for a period of time ranging from about 12 hours to about 336 hours. In alternate embodiments, a temperature between about 140°F and about 170°F is provided for a period of time ranging from about 12 hours to about 24 hours, from about 24 hours to about 48 hours, from about 48 hours to about 96 hours, from about 96 hours to about 168 hours, from about 168 hours to about 226 hours, or from about 226 hours to about 336 hours.

[0027] Generally, heat is applied continuously through the given time period. But heat may also be provided for the given time period in a manner that is not continuous, for example, at intervals so long as heat is provided totaling the stated time period. At the end of this time period, no further heating or aging is needed to achieve the distilled spirit having characteristics associated with a matured distilled spirit. For example, no further heating at a temperature of 140°F to about 170°F is provided to the distilled spirit having characteristics associated with a mature distilled spirit.

[0028] The heating is conducted in a sealed vessel or under reflux such that evaporation of volatile compounds is prevented. The pressure inside the vessel can and will vary in different embodiments of the invention. In general, the internal pressure of the vessel housing the reaction components climbs to a maximum of about 6 pounds per square inch (psi). The typical range of pressures in the vessel is between 2 psi and 6 psi, but can be altered to relieve pressure and maintain an operating pressure of less than 1 psi while still achieving the desired chemical reactions.

[0029] In one embodiment, the temperature is maintained between about 140°F and about 150°F for a period of time ranging from about 168 hours to about 226 hours.

[0030] In another embodiment, the temperature is maintained between about 150°F and about 160°F for a period of time ranging from about 12 hours to about 48 hours.

[0031] Of the various advantages of the present invention, one is that no additional ingredients or acids are provided to facilitate the process. In this regard, the process is free of additives. Without being bound to any particular theory, it is thought that the inventive process rapidly extracts acids from the wood. These acids facilitate the esterification process without the need for additives beyond the wood and the unmatured distilled spirit.

[0032] The disclosure also provides a process for producing a distilled spirit having characteristics of a matured distilled spirit comprising (a) obtaining a marker concentration of a chemical compound in a target matured distilled spirit thereby giving a target marker concentration, (b) contacting an unmatured distilled spirit with wood to form a distilled spirit mixture; (c) measuring the marker concentration of the distilled spirit mixture; and (d) heating the distilled spirit mixture to a temperature between about 140°F and about 170°F in a sealed vessel or under reflux such that evaporation of volatile compounds is prevented until the target concentration of the distilled spirit mixture reaches the target marker concentration.

[0033] The step of obtaining the marker concentration of a chemical compound in a target matured distilled spirit may involve selection of a target matured distilled spirit and measurement of its marker concentration. In other embodiments, this step involves using a reference or standard for the target marker concentration. The target marker concentration is then used to determine length of time needed for the reaction between the unmatured distilled spirit and the wood. One or more measurements of the distilled spirit mixture formed by the contacting of the unmatured distilled spirit and the wood is taken after the initial contacting of the unmatured distilled spirit and the wood. Such measurements can be taken by techniques known in the art such as mass spectroscopy, high performance liquid chromatography, gas chromatography, or colorimetric methods.

[0034] Once the chemical marker concentration of the distilled spirit mixture is obtained it may be compared to the target marker concentration in order to determine if heating at a temperature ranging from about 140°F and about 170°F can be discontinued. When the target concentration is not met, heating at a temperature ranging between about 140°F and about 170°F is continued. Where the target concentration is met, further heating at a temperature of 140°F to about 170°F is discontinued. Where the target concentration is met, further heating at a temperature between about 140°F and about 170°F is discontinued. Thus, the method provides a tunable process for producing a distilled spirit having characteristics associated with a matured distilled spirit in accordance with the invention. In one embodiment, the marker associated with a matured distilled spirit may be ethyl decanoate. The process disclosed above, however, is not limited to ethyl decanoate, but can be any other chemical compound that has different concentrations in a matured distilled spirit as compared to an unmatured distilled spirit. In one embodiment, the marker is a compound which has a higher concentration in the final product of the process than in the unmatured distilled spirit. In another embodiment, the marker is a compound which has a lower concentration in the final product of the process than in the unmatured distilled spirit.

[0035] Markers for the process include, but are not limited to ethyl acetate, ethyl butyrate, ethyl heptanoate, ethyl formate, ethyl octanoate, ethyl decanoate, and ethyl lactate. In some embodiments, semi-volatiles can be used as chemical markers. Individual semi-volatile compounds may be used as markers, or a fingerprint of more than one semi-volatile may be used as a marker.

[0036] The disclosure further provides a system for producing a distilled spirit having characteristics associated with a matured distilled spirit as depicted in Fig. 1, which shows a cutaway view of one possible internal configuration. The vessel 1 may be a wooden barrel.

[0037] In the embodiment depicted in Fig. 2, a cutaway is shown using dashed lines to reveal another possible internal configuration of the vessel 1, where vessel 1 may be a stainless steel container. In this embodiment, the wood 12 is provided as chips submerged in or floating on top of the distillate 2.

[0038] For both Fig. 1 and Fig. 2, heat may be applied to the distillate 2 inside the vessel 1 via a heat source 4. In one embodiment, the vessel is sealed under pressure. In another embodiment, the vessel is boiled under reflux to prevent evaporation of volatile compounds. Any method for introducing sufficient heat to the distillate including an internal electrical heating element 9 may be used. In such an embodiment, temperature may be controlled by manipulating electrical current in the internal electrical heating element 9 circuit. In alternate embodiments the heat source 4 is external and may be used with a conduit 16, conductor, or the like. In one non-limiting example, steam 18 may be created using an external boiler as the heat source in one embodiment, then passed through a conduit running through the vessel 1, wherein a conductive portion 16 of the conduit is submerged in the distillate 2. In such an embodiment, the temperature of the distillate 2 may be controlled by varying the amount of steam 18 produced by the boiler. Such control may be provided by valves or similar means which are not pictured.

[0039] In one embodiment, the temperature of the distillate 2 in the vessel 1 is brought up to a temperature of about 160°F for a period of up to 48 hours. This process simultaneously extracts tannins and catalyzing acids. In one embodiment, the temperature of the distillate is measured via a temperature sensor 5. Any known temperature sensor may be used. Temperature of the distillate 2 may be controlled by manually manipulating the heat source 4. In an alternate embodiment, an electronic temperature sensor is coupled to a controller 7. The controller 7 may be configured to present an alert (e.g., flash a light, produce a sound, produce an electrical signal, transmit a text message, e-mail or other electronic communication, etc.) when the temperature of the distillate 2 is outside of a provided range. Optionally, the controller 7 may be coupled to the heat source 4, in which case, the controller is configured to manipulate the heat source to keep the temperature of the distillate 2 within the provided range.
[0040] Heating in accordance with the invention triggers the esterification of free volatile acids and alcohols in the distillate 2, while the headspace 3 provides for reflux within the vessel 1 allowing any weak acids to be rapidly extracted from the wood. These weak acids, in combination with elevated temperatures, help catalyze the esterification of free acids in the distillate per the Fischer process, which would otherwise take many years in traditional containers.

[0041] The pressure inside the container may be measured by a pressure sensor 6. The pressure sensor 6 may be coupled to the controller 7. The pressure sensor may be configured to provide alerts if the pressure becomes too high. The pressure sensor may optionally be configured to communicate to open a release valve if the pressure is too high.