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(54) **PROCESS FOR APPLYING POLYMERIC COATING ON CORK STOPPERS**

VERFAHREN ZUM AUFBRINGEN EINER POLYMERBESCHICHTUNG AUF KORKEN

PROCÉDÉ D'APPLICATION D'UN REVÊTEMENT POLYMÈRE SUR DES BOUCHONS EN LIÈGE

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Description**FIELD OF THE INVENTION**

[0001] The present invention relates to a process for applying a polymeric coating on cork stoppers, which coating is based on cross-linked silicone (PDMS), in order to delay the migration of coloured compounds from stoppers into alcoholic beverages. In particular, the process of the present invention provides a polymeric coating that significantly delays the migration of phenolic/polyphenolic compounds from stoppers into white alcoholic beverages.

BACKGROUND OF THE INVENTION

[0002] One of the most important applications of cork is as a seal, namely as natural cork stoppers, agglomerated cork stoppers, technical stoppers, Champagne corks and cork stoppers with cap. These stoppers are widely used in sealing alcoholic beverages. However, cork stoppers did not yet succeeded entering the market of white beverages with high alcohol content due to the yellowish colour they confer to these beverages over time.

[0003] Phenolic compounds such as ellagic acid, gallic acid and vanillin and polyphenolic compounds such as ves-calagin and castalagin present in the cork migrate to white alcoholic beverage conferring it an undesirable yellowish coloration. The higher the alcoholic content of the beverage, the bigger the migration of this type of compounds.

[0004] There are several known processes aiming at the elimination of the undesirable compounds of the cork material. The approaches used to eliminate said compounds are also different.

[0005] Patent application US 5174956, published December 29, 1992, describes a method for deodorization of cork using steam to remove substances originating unpleasant flavour, namely 2,4,6-trichloroanisole (TCA). The method describes the use of steam in cork boards within a container with a pressure equal or higher than 1 atm and a temperature equal or higher than 100 °C.

[0006] International Patent Application WO 0141989, published June 14, 2001, describes a physical chemical method to remove undesirable odours from cork. This process makes use of an aqueous suspension of activated carbon obtained from coconut. Washing cork with this suspension eliminates the unpleasant flavours from the cork.

[0007] International Patent Application WO 2004014436, published February 19, 2004, discloses an extraction system whereby compounds are dragged with water vapour allowing a significant reduction of compounds with unpleasant flavours.

[0008] European Patent EP 1444075 B1, published August 11, 2004, discloses a process that makes use of hydroalcoholic mixtures in gaseous state, at temperatures up to 100 °C and pressures up to 2 bar, as means for extraction of phenolic compounds.

[0009] US Patent 7192490 B1, published March 20, 2007, discloses a process for extraction of organic compounds from cork by means of a dense fluid under pressure (10 bar to 600 bar), in particular a supercritical fluid, at a temperature in the range of 10 °C to 120 °C.

[0010] The above mentioned methods involve the use of organic and inorganic solvents which are generally expensive and have a limited efficacy in extracting coloured compounds from cork. Indeed, these methods allow only a partial extraction of phenolic and polyphenolic compounds present in cork and therefore only moderately delay their migration from the cork stopper to the beverage. Thus, these methods are not sufficiently effective for applying in cork stoppers to be used in said white alcoholic beverages.

[0011] Although they reside within the same technical field of the present invention, the preceding patent documents have different approaches in order to solve the described problem.

[0012] There are other prior art documents that disclose the application of coatings on cork stoppers, which purpose is to establish physical barriers to the migration into the beverages of impurities present in cork stoppers, in particular TCA.

[0013] International Patent Application WO 9628378, published September 19, 1996, discloses a coating and the process thereof for applying it to prevent tainting by TCA. The disclosed coatings are polyethylene dispersions, gels such as ethylene vinyl acetate (EVA), polyvinylidene chloride (PVDC) dispersions, among others. Polyvinyl chloride (PVC) and polyurethane are the preferred coatings. The application of the coating on cork stoppers is carried out by heating (microwave or hot air) or by pre-treatment of cork with UV radiation and other oxidation treatments, in order to improve the coating adhesion and its incorporation. This process does not delay the migration of phenolic or polyphenolic compounds into white alcoholic beverages for an acceptable time period.

[0014] US Patent 6348243, published February 19, 2002, discloses a silicone oil based coating which is applied by immersion. A pre-treatment is also disclosed in which the cork is washed with one or more solvents so as to reduce the concentration of undesired compounds by extraction. Silicone oil has a low efficacy barrier effect to permeation of coloured compounds of cork into beverages because it is oil and not a cross-linked film. Therefore, its characteristics of delaying migration of coloured substances are lost over time, not solving the above described problem.

[0015] European Patent EP 0773090 B1, published September 3, 2003, discloses a coating method for stoppers with

a silicone elastomer. This invention aims to improve the performance of treated cork in terms of sealing, which is useful for cork stoppers of lower quality, in view of the shortage of raw material and its increasing price. The application of the coating is made by immersion and under vacuum.

[0016] European Patent EP 2001759 B1, published September 27, 2007, relates to the application of a coating on cork which is made up of two films, the first being hydrophilic (such as for example cellulose) and the second hydrophobic (such as for example silicone). This document does not mention the use of the coating as in this invention, nor comments on the performance as a barrier to the migration of polyphenolic compounds and also the mechanical performance of said coatings.

[0017] International Patent Application WO 2007115612 A1, published on October 18 2007, relates to a method for coating cork stoppers with liquid reactive polyurethane coating agents, said coating being used to retain any aroma-active substances in the cork. Drying and crosslinking is carried out at Temperatures between 25°C and 130°C during a period between 30s and 12h.

[0018] Usually the last step of the production of cork stoppers consists of a stopper surface treatment with silicone and/or paraffin. However, the surface treatments commonly used are not intended for reduction of the amount of coloured substances that migrate from cork into the beverage, but only the lubrication of stoppers in order to get a better insertion/removal of the stopper into/from the bottle.

[0019] Thus, the processes of prior art employed to reduce the migration of coloured compounds from cork into beverages have essentially the purpose of extracting TCA from cork stoppers or limiting its migration into beverages. In fact cork stoppers did not yet succeed in entering the market of white alcoholic beverage which demonstrates the inefficacy of these processes to solve the problem of phenolic/polyphenolic compounds migration into these beverages.

[0020] That is to say, cork stoppers treated with the above mentioned processes, when inserted in bottles with white alcoholic beverages, produce an undesirable yellowish colour over time due to migration of said phenolic/polyphenolic compounds.

[0021] The present invention solves the problem of migration of coloured substances from the cork stopper into beverages, by applying a coating on cork stoppers using a process that ensures that all surface of the stopper is coated, as well as the existing pores in the cork stopper. By preventing or significantly limiting the formation of pores in the coating during its application, the process of the present invention provides a high efficacy coating.

SUMMARY OF THE INVENTION

[0022] The coating process of the present invention provides a coating on cork stoppers that achieve a significant reduction of migration of visually undesirable compounds from cork stoppers into beverages, enabling the successful marketing of these stoppers for white alcoholic beverages.

[0023] A coating with pores has a lower barrier effect than a free-pore coating. The coating process for cork stoppers of the invention provides an impermeability of stoppers to those undesirable compounds which was never achieved with the related prior art processes and coatings.

[0024] The present invention discloses a process for applying a polymeric coating on cork stoppers comprising the steps of:

1. depositing a polymeric film on cork stoppers providing a non-cured polymeric coating on said stoppers;
2. gradual curing of the polymeric coating by applying a temperature variation rate in the range of about 3 °C/min to about 6 °C/min, in order to gradually raise the temperature from about 40 °C to about 110 °C;
3. final curing of the polymeric coating at constant temperature in the range of about 70 °C to 120 °C for at least 5 minutes.

[0025] In one aspect of the invention, step 1 is made by bringing stoppers into contact with a polymeric suspension at a temperature in the range of 15 °C to 25 °C with a contact time between the stoppers and the polymeric suspension higher than 10 minutes.

[0026] In another aspect, the cork stoppers in step 1 have a moisture content in the range of 8% to 25%.

[0027] In yet another aspect, the polymer of step 1 is PDMS.

[0028] In another aspect of the invention, step 1 is carried out in a non-perforated drum.

[0029] Yet another aspect of the invention, steps 2 and 3 are carried out in a perforated drum.

[0030] In another aspect of the process of the invention, step 3 further includes water vaporization to ensure the control of the final moisture content of cork stoppers and the absence of pores in the coating.

[0031] Furthermore, the process comprises the steps of:

1. depositing PDMS on cork stoppers at a temperature of about 20 °C, by bringing the stoppers into contact with a polymeric suspension of PDMS for about 15 minutes;

2. applying a temperature variation rate of about 4 °C/min so as to gradually raise a temperature of about 40 °C to about 90 °C;
3. applying a temperature of about 90 °C for about 8 minutes in the presence of water vapour,

wherein step 1 is carried out in a non-perforated drum with stoppers having a moisture content in the range of 10% to 15% and steps 2 and 3 are carried out in a perforated drum.

[0032] The present disclosure also relates to a cork stopper, coated according to the process above defined, having a moisture content in the range of 5% to 8% and having a coating with a thickness in the range of 5 μm to 100 μm .

[0033] In one aspect of the disclosure, said cork stopper has a coating with a thickness in the range of 10 μm to 50 μm .

[0034] Also disclosed is the use of said cork stopper on bottles having white alcoholic beverages.

BRIEF DESCRIPTION OF THE DRAWINGS

[0035] The present invention is described by the following detailed description which makes reference to the accompanying drawings, in which:

Fig. 1 is a graph of absorbance at 420 nm of hydroalcoholic solutions at a temperature of 35 °C, in total maceration and contact with stoppers treated (PDMS) according to the process of the present invention and with stoppers not treated (Ref).

Figs. 2 to 5 are graphs of absorbance at 420 nm over time of tequila, vodka, gin and an hydroalcoholic solution, respectively, in bottles uprightly stored and undergone daily manual agitation, in contact with stoppers treated (PDMS) according to the process of the present invention and with stoppers not treated. The limit of visual colour detection corresponds to the indicated absorbance of 0.012.

DETAILED DESCRIPTION OF THE INVENTION

[0036] The present invention discloses a coating process with polymeric film that efficiently delays the migration of coloured compounds from cork stoppers into hydroalcoholic beverages. In particular, this invention solves the problem of migration of phenolic and polyphenolic compounds from cork into alcoholic beverages in an economically and viable way, advantageously allowing the use of cork stoppers in, for example, white beverages.

[0037] In the context of the present description, the terms "white alcoholic beverage" or "white beverage" refer to a transparent beverage having an high alcoholic content (above 20% vol alcohol).

[0038] The term "about" in the context of the present description means a variation of $\pm 5\%$ of the stated value. For example, the term "about 2.0" means " 2.0 ± 0.1 ".

[0039] Unless otherwise noted, the ranges of values which are given in the present description are intended to provide a simplified and technically acceptable way to denote each individual value within the specified range. Each individual value of said range should be construed as it has been individually mentioned. By way of example, the term "1 to 2" means any value in this range, for example 1.0, 1.1, 1.2, 1.3, 1.4, 1.5, 1.6, 1.7, 1.8, 1.9, 2.0. All values mentioned in present description should be construed as approximate values, for example, the reference to "2.0" means "about 2.0".

[0040] The term "phenolic compounds" in the context of the present refers to compounds that give colour to white beverages, such as for example ellagic acid and gallic acid.

[0041] The term "polyphenolic compounds" in the context of the present refers to more complex compounds which also give colour to white beverages, namely vescalagin and castalagin.

[0042] The term "coloured compounds" in the context of the present refers to phenolic compounds and/or polyphenolic compounds.

[0043] The term "polymeric film" refers to any food-grade, cross-linkable polymer based material, which is depositable in the form of a non-cured coating layer on the outer surface of cork stoppers. The curing process provides a polymeric coating film.

[0044] The preferred coating of the present invention is made of cross-linkable silicone, named polydimethylsiloxane and herein referred to as PDMS.

[0045] In the process of the invention the use of silicone resin PDMS is preferred, as this resin provides a low permeability to coloured polyphenolic compounds and also has a high adhesion to cork stoppers. Moreover, PDMS can be produced in compatible way to human food and forms a transparent coating having good flexibility that follows the natural elasticity of cork stoppers. PDMS also acts as cork stopper auto-lubricant, thus facilitating the insertion/removal of the stopper into/from the bottle.

[0046] It was surprisingly found that the deposition of a polymeric film, such as a PDMS silicone resin film, by the process of the present invention, allows the production of a coating with very low permeability to coloured compounds,

efficiently operating as a physical barrier.

[0047] The process for applying a polymeric coating on cork stoppers comprises the steps of:

1. depositing a polymeric film on cork stoppers providing a non-cured polymeric coating on said stoppers;
2. gradual curing of the polymeric coating by applying a temperature variation rate in the range of about 3 °C/min to about 6 °C/min, in order to gradually raise the temperature from about 40 °C to about 110 °C;
3. final curing of the polymeric coating at constant temperature in the range of about 70 °C to 120 °C for at least 5 minutes.

[0048] Step 1 of depositing a polymeric film preferably comprises the application of a polymeric suspension on cork stoppers. This coating step must be carried out in a non-perforated drum so as to avoid any losses of polymeric material. The drum should be a rotary drum so as to promote a better contact between stoppers and silicone resin.

[0049] The contact between stoppers and silicone resin should preferably be done at room temperature (between 15 and 25 °C) so that the surface coating of stoppers is as much uniform as possible and also the resin curing process does not take place extemporaneously.

[0050] The contact time between stoppers and silicone resin should be higher than 10 minutes, preferably between 10 and 30 minutes, more preferably between 10 and 20 minutes, to ensure a uniform distribution of the resin on the surface of stoppers.

[0051] As known by a skilled person, the presence of suitable final moisture content in finished cork stoppers (namely 5 to 8%) is a key factor for keeping the mechanical and elastic properties of stoppers. To reach such conditions in finished cork stoppers, step 1 of the process of present invention must be carried out preferably on stoppers with moisture content in the range of 8% to 25%, more preferably in the range of 10% to 15%.

[0052] As known by a skilled person, in the course of the conventional process of production of cork stoppers, there is a washing step of cork stoppers. In terms of global productive process of cork stoppers, in which the process of the invention is included, said washing step is upstream of step 1 of the present invention. In order to ensure a suitable final moisture content (at the end of the process of the invention), the stoppers should start step 1 of the invention as soon as they finish said washing, so that the polymeric suspension is preferably applied on stoppers with a moisture content in the range of 10% to 15%, as above mentioned. This restriction helps to easily obtain said final moisture content of 5 to 8% which contributes to the good quality of the final stopper.

[0053] In conventional production processes, the stoppers are dried after the washing, and only after that drying step they start the coating procedure. These processes of the prior art yield quite dry stoppers (having moisture content below 5%) and therefore with a lower quality than those obtained by the process of the present invention.

[0054] Also note that the thickness of the polymeric coating obtained in the process of the present invention is important.

[0055] In one hand, the thickness of the coating should not be visually perceived in the cork stopper but, on the other hand, should be sufficiently thick to ensure that the permeation characteristics to coloured compounds are not at risk, such as its delaying capacity to phenolic and polyphenolic compounds, as well as its mechanical strength. Accordingly, the thickness of the coating should be in the range of 5 μm to 100 μm , preferably in the range of 10 μm to 50 μm .

[0056] In order to obtain a thickness within said range of values, a suitable amount of polymeric suspension should be used per total surface area of the stoppers to be coated, when carrying out step 1. Industrially, this is a fast method for obtaining the desired thicknesses.

[0057] Thus, it was found that by using 2.5 kg of polymeric suspension in step 1 of the present invention, it is possible to obtain thicknesses in the range of 10 μm to 50 μm in batches of stoppers containing 15000 stoppers with 26 x 19.5 mm or with 30 x 19.5 mm (length x diameter).

[0058] In the processes of the prior art, after a coating step, for example with silicone, the cork stoppers undergo a curing procedure usually between 90 °C and 100 °C.

[0059] It was found that by heating cork stoppers between 90 °C and 100 °C, an expansion of the air contained within the cork and its release through the silicone film take place. In this way, throughout the polymer curing process pores are formed in the coating, providing lower barrier performance than in the case of a coating without pores.

[0060] Surprisingly, it was found that such pores can be eliminated or significantly reduced in number from the polymeric coating, by means of an intermediate step of gradual curing of the coating under certain conditions, after step 1. In this way, the air present within the cork may expand and subsequently leak out to the atmosphere before the cure is complete, thus providing a more consistent polymeric film.

[0061] The elimination or significant reduction of the pores is carried out in step 2 of the present invention, wherein the coated stoppers of step 1 are submitted to a temperature variation rate in the range of 3 °C/min to 6 °C/min, preferably at about 4 °C/min, in order to gradually raise the temperature from about 40 °C to about 110 °C, preferably from about 60 °C to about 90 °C.

[0062] After step 2 of the present invention and taking into consideration the complete cure of the coating, step 3 of the invention is carried out by submitting the cork stoppers to a constant temperature in the range of about 70 °C to

about 120 °C, for at least 5 minutes, preferably for about 5 to 10 minutes, more preferably more for about 10 minutes.

[0063] Both step 2 and step 3 of the present invention can be carried out in a drum, preferably a perforated drum.

[0064] Step 3 of thermal curing completion can be further carried out in the presence of water vapour by introduction of said water vapour inside the curing drum, in order to ensure the control of the final moisture content of the cork stoppers resulting from the process of the invention.

[0065] In one preferred embodiment of the present invention, the process comprises:

1. depositing PDMS on cork stoppers at a temperature of about 20 °C, by bringing the stoppers into contact with a polymeric suspension of PDMS for about 15 minutes;
2. gradual curing of the polymeric coating by applying a temperature variation rate of about 4 °C/min, in order to gradually raise a temperature from about 40 °C to about 90 °C;
3. final curing of the polymeric coating at about 90 °C for about 8 minutes in the presence of water vapour,

wherein step 1 is carried out in a non-perforated drum with stoppers having a moisture content in the range of 10% to 15% and steps 2 and 3 are carried out in a perforated drum.

[0066] As already mentioned, the coated cork stoppers obtained by the process of the invention should have a moisture content in the range of 5% to 8% and the coating should have a thickness in the range of 5 µm to 100 µm. The subsequent application of said stoppers in bottles having white alcoholic beverages ensures a barrier effect to the migration of coloured compounds into the beverage, for time periods not previously observed with coated stoppers of the prior art.

EXAMPLES

Example 1 - Preparation of the Polymeric Formulation

[0067] Before step 1 of the invention, the polymeric formulation to apply on the cork stoppers is prepared. The preferred formulation is prepared with polydimethylsiloxane (PDMS) with vinyl groups, a cross-linking agent and a catalyst. The amount of catalyst to add to the formulation has to be appropriate (about 1%) to make the final PDMS formulation compatible with human food (food-grade).

[0068] In the following Tables 1 and 2, the main characteristics of the polymeric formulation and the amounts of the components in percentage of total weight of the formulation are respectively described.

Table 1 - Polymeric formulation

Components	Viscosity (mPa.s)	density (25 °C) (g.cm ⁻³)	cure T (°C)
PDMS	400-600	0.97	90 - 100
Cross-linking agent	22	1.0	
Catalyst	850	0.98	

Table 2 - Weight percentage of the formulation components

Component	% w/w
PDMS	96.6
Cross-linking agent	2.4
Catalyst	1.0

Example 2 - Application of PDMS coating on Cork Stoppers According to the Process of the Invention. Assessment of Extraction of Coloured Compounds after Maceration of Treated Stoppers (Accelerated Regimen)

[0069] This example refers to the application on cork stoppers of a PDMS coating of the type defined in Example 1, by means of the process of the present invention. The PDMS polymeric suspension was prepared by adding cross-linking agent and catalyst to PDMS according to Example 1.

[0070] Film deposition was done on 100 cork stoppers (by the process of the present invention) by immersion in a mixing rotary drum at room temperature of about 20 °C for about 15 minutes. The stoppers were previously washed and

showed an approximate moisture content of 15% before were mixed in the drum.

[0071] After this first step, the cork stoppers were placed in a perforated rotary drum for curing the film. The rotary drum was at 60 °C and a temperature variation rate of 3 °C/min was set until reaching the temperature of 90 °C. Next, the stoppers were kept at 90 °C for 10 minutes in the presence of water vapour in the cure drum.

[0072] After coating, the stoppers were inserted in 60 mL flasks containing a 50% (v/v) hydroalcoholic solution. In 10 flasks, 5 random coated stoppers of the treated batch and 5 other non-coated stoppers were inserted. Next, the flasks were placed on a horizontal shaker at a temperature of 35 °C. These conditions established the accelerated regimen of colouration of the solution, in order to provide an accelerated assessment of the results.

[0073] The extracted coloured compounds in contact with the hydroalcoholic solution show a colour with a maximum absorbance peak of about 420 nm.

[0074] In Fig. 1, the PDMS coated stoppers are indicated with "PDMS" and the stoppers without coating are indicated with "Ref". This second group of stoppers is a control for the colour analysis.

[0075] Fig. 1 shows the increasing absorbance of the hydroalcoholic solutions in contact with treated and not treated cork stoppers. After 20 days, the relative difference of absorbance at 420 nm between the two types of stoppers is higher than 80%, showing very significant difference.

Example 3 - Application of PDMS coating on Cork Stoppers According to the Process of the Invention. Assessment of Extraction of Coloured Compounds from Treated Usual Stoppers (Normal Regimen)

[0076] The conditions for applying the process of the present invention were identical to those of the preceding assay.

[0077] After application of the process as in Example 2, the resulting coated stoppers were placed so as to seal bottles containing different white alcoholic beverages.

[0078] Bottles having Tequila, Vodka, Gin and a hydroalcoholic solution similar to Example 2 were used. The assessment was carried out at normal regimen, *i. e.*, treated and not treated cork stoppers in bottles were assessed. In this way, non-accelerated conditions were used for the extraction of undesirable compounds for those beverages.

[0079] In Figs. 2 to 5 several plots of absorbance change at 420 nm over time of several beverages in contact with treated stoppers (PDMS) and not treated (Ref) are shown. In graphs of Figs. 2 to 5 a visual level of perception of colour is also indicated (horizontal line at absorbance of 0.012). The bottles were kept upwardly in an incubator at 35 °C and were subjected to daily manual agitation.

[0080] In the assay with treated stoppers it was not possible to detect any visible colour with naked eye (absorbance higher than 0.012) in all different beverages, even after 175 days of assay. In contrast, the beverages sealed with non-treated stoppers (Ref), such as for example tequila and vodka, showed colour after only 23 days. These results were obtained from an average of 10 absorbance values.

[0081] Based on these results it is possible to conclude that the polymeric coating process of the present invention is very efficient in comparison to non-coated stoppers. The results obtained with cork stoppers coated by the process of the invention foresees its use in bottles containing white alcoholic beverages, for much longer time periods than prior art stoppers (treated or not treated).

Claims

1. A process for applying a polymeric coating on cork stoppers comprising the steps of:

1. depositing a polymeric film on cork stoppers providing a non-cured polymeric coating on said stoppers;
2. gradual curing of the polymeric coating by applying a temperature variation rate in the range of about 3 °C/min to about 6 °C/min, in order to gradually raise the temperature from about 40 °C to about 110 °C;
3. final curing of the polymeric coating at constant temperature in the range of about 70 °C to 120 °C for at least 5 minutes.

2. The process according to claim 1, wherein step 1 is made by bringing stoppers into contact with a polymeric suspension at a temperature in the range of 15 °C to 25 °C with a contact time between the stoppers and the polymeric suspension higher than 10 minutes.

3. The process according to claim 1 or 2, wherein in step 1 the cork stoppers have a moisture content in the range of 8 % to 25%.

4. The process according to any of claims 1 to 3, wherein the polymer of step 1 is PDMS.

5. The process according to any of preceding claims, wherein step 1 is carried out in a non-perforated drum.
6. The process according to any of preceding claims, wherein steps 2 and 3 are carried out in a perforated drum.
7. The process according to any of preceding claims, wherein step 3 further includes water vaporization to ensure the control of the final moisture content of cork stoppers and the absence of pores in the coating.

8. A process for applying a polymeric coating on cork stoppers comprising the steps of:

1. depositing PDMS on cork stoppers at a temperature of about 20 °C, by bringing the stoppers into contact with a polymeric suspension of PDMS for about 15 minutes;
2. applying a temperature variation rate of about 4 °C/min so as to gradually raise a temperature of about 40 °C to about 90 °C;
3. applying a temperature of about 90 °C for about 8 minutes in the presence of water vapour,

wherein step 1 is carried out in a non-perforated drum with stoppers having a moisture content in the range of 10% to 15% and steps 2 and 3 are carried out in a perforated drum.

Patentansprüche

1. Verfahren zum Aufbringen einer Polymerbeschichtung auf Korkstopfen, folgende Schritte umfassend:

1. Abscheiden eines Polymerfilms auf Korkstopfen, wodurch eine ungehärtete Polymerbeschichtung auf den Stopfen bereitgestellt wird,
2. schrittweises Härten der Polymerbeschichtung durch Anwenden einer Temperaturveränderungsrate im Bereich von etwa 3 °C/min bis etwa 6 °C/min, um die Temperatur schrittweise von etwa 40 °C auf etwa 110°C zu erhöhen,
3. abschließendes Härten der Polymerbeschichtung bei konstanter Temperatur im Bereich von etwa 70 °C bis 120°C für mindestens 5 Minuten.

2. Verfahren nach Anspruch 1, wobei Schritt 1 erfolgt, indem Stopfen in Kontakt mit einer Polymersuspension mit einer Temperatur im Bereich von 15 °C bis 25 °C gebracht werden, mit einer Kontaktzeit zwischen den Stopfen und der Polymersuspension von mehr als 10 Minuten.

3. Verfahren nach Anspruch 1 oder 2, wobei in Schritt 1 die Korkstopfen einen Feuchtigkeitsgehalt im Bereich von 8 % bis 25 % aufweisen.

4. Verfahren nach einem der Ansprüche 1 bis 3, wobei das Polymer von Schritt 1 PDMS ist.

5. Verfahren nach einem der vorhergehenden Ansprüche, wobei Schritt 1 in einer nicht-perforierten Trommel ausgeführt wird.

6. Verfahren nach einem der vorhergehenden Ansprüche, wobei die Schritte 2 und 3 in einer perforierten Trommel ausgeführt werden.

7. Verfahren nach einem der vorhergehenden Ansprüche, wobei Schritt 3 ferner eine Wasserverdampfung beinhaltet, um die Steuerung des abschließenden Feuchtigkeitsgehalts der Korkstopfen und das Nicht-Vorhandensein von Poren in der Beschichtung zu gewährleisten.

8. Verfahren nach einem der vorhergehenden Ansprüche, folgende Schritte umfassend:

1. Abscheiden von PDMS auf Korkstopfen bei einer Temperatur von etwa 20 °C, indem die Stopfen für etwa 15 Minuten mit einer Polymersuspension von PDMS in Kontakt gebracht werden,
2. Anwenden einer Temperaturveränderungsrate von etwa 4 °C/min, um eine Temperatur schrittweise von etwa 40 °C auf etwa 90 °C zu erhöhen,
3. Anwenden einer Temperatur von etwa 90 °C für etwa 8 Minuten in Gegenwart von Wasserdampf,

wobei Schritt 1 in einer nicht-perforierten Trommel ausgeführt wird, wobei die Stopfen einen Feuchtigkeitsgehalt im Bereich von 10 % bis 15 % aufweisen, und die Schritte 2 und 3 in einer perforierten Trommel ausgeführt werden.

5 Revendications

1. Procédé d'application d'un revêtement polymère sur des bouchons en liège comprenant les étapes de :

1. dépôt d'une pellicule polymère sur des bouchons en liège fournissant un revêtement polymère non durci sur lesdits bouchons ;
2. durcissement graduel du revêtement polymère en appliquant une vitesse de variation de la température dans la plage d'environ 3° C/min à environ 6° C/min, afin d'augmenter graduellement la température d'environ 40° C à environ 110° C ;
3. durcissement final du revêtement polymère à une température constante dans la plage d'environ 70° C à 120° C pendant au moins 5 minutes.

2. Procédé selon la revendication 1, dans lequel l'étape 1 est effectuée en mettant en contact les bouchons avec une suspension polymère à une température dans la plage de 15° C à 25° C avec un temps de contact entre les bouchons et la suspension polymère supérieur à 10 minutes.

3. Procédé selon la revendication 1 ou 2, dans lequel dans l'étape 1 les bouchons en liège ont une teneur en humidité dans la plage de 8 % à 25 %.

4. Procédé selon l'une quelconque des revendications 1 à 3, dans lequel le polymère de l'étape 1 est du PDMS.

5. Procédé selon l'une quelconque des revendications précédentes, dans lequel l'étape 1 est réalisée dans un tambour non perforé.

6. Procédé selon l'une quelconque des revendications précédentes, dans lequel les étapes 2 et 3 sont réalisées dans un tambour perforé.

7. Procédé selon l'une quelconque des revendications précédentes, dans lequel l'étape 3 inclut en outre une vaporisation d'eau pour garantir le contrôle de la teneur en humidité finale des bouchons en liège et l'absence de pores dans le revêtement.

8. Procédé selon l'une quelconque des revendications précédentes, comprenant les étapes de :

1. dépôt de PDMS sur les bouchons en liège à une température d'environ 20° C, en mettant les bouchons en contact avec une suspension polymère de PDMS pendant environ 15 minutes ;
2. application d'une vitesse de variation de la température d'environ 4° C/min de sorte à augmenter graduellement une température d'environ 40° C à environ 90° C ;
3. application d'une température d'environ 90° C pendant environ 8 minutes en la présence de vapeur d'eau,

dans lequel l'étape 1 est réalisée dans un tambour non perforé avec des bouchons ayant une teneur en humidité dans la plage de 10 % à 15 % et les étapes 2 et 3 sont réalisées dans un tambour perforé.

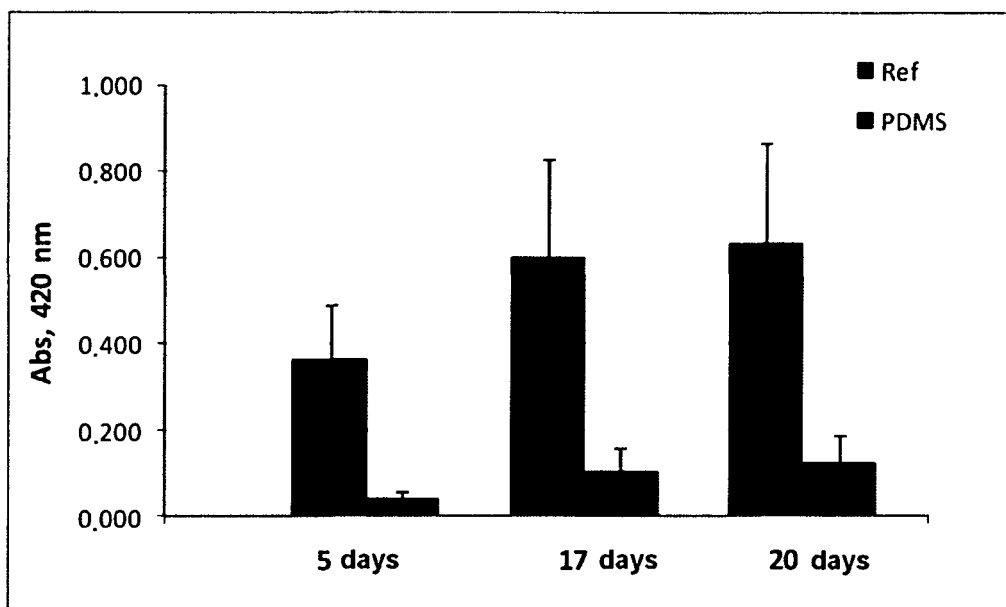


Fig. 1

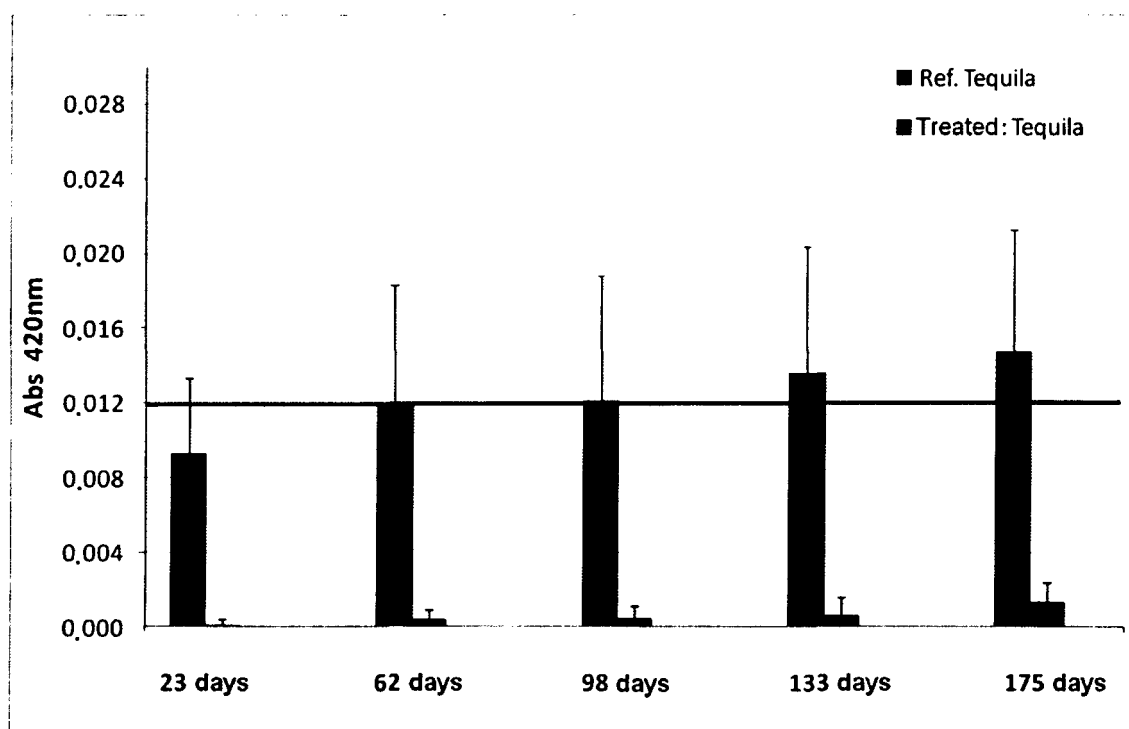


Fig. 2

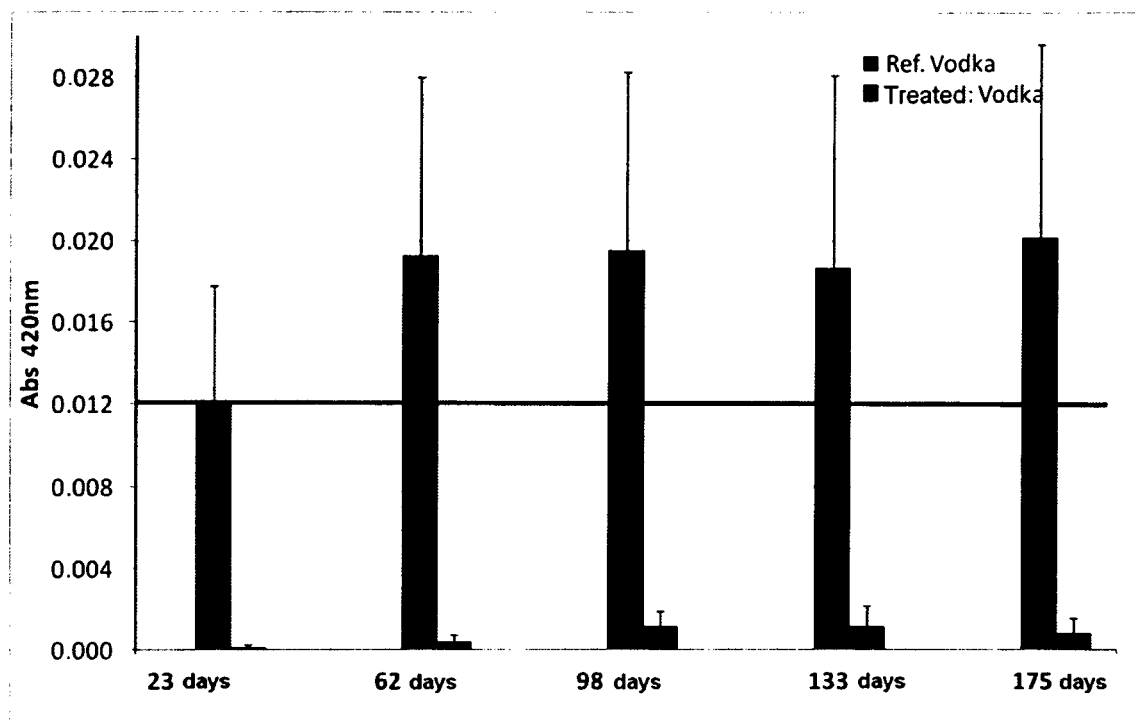


Fig. 3

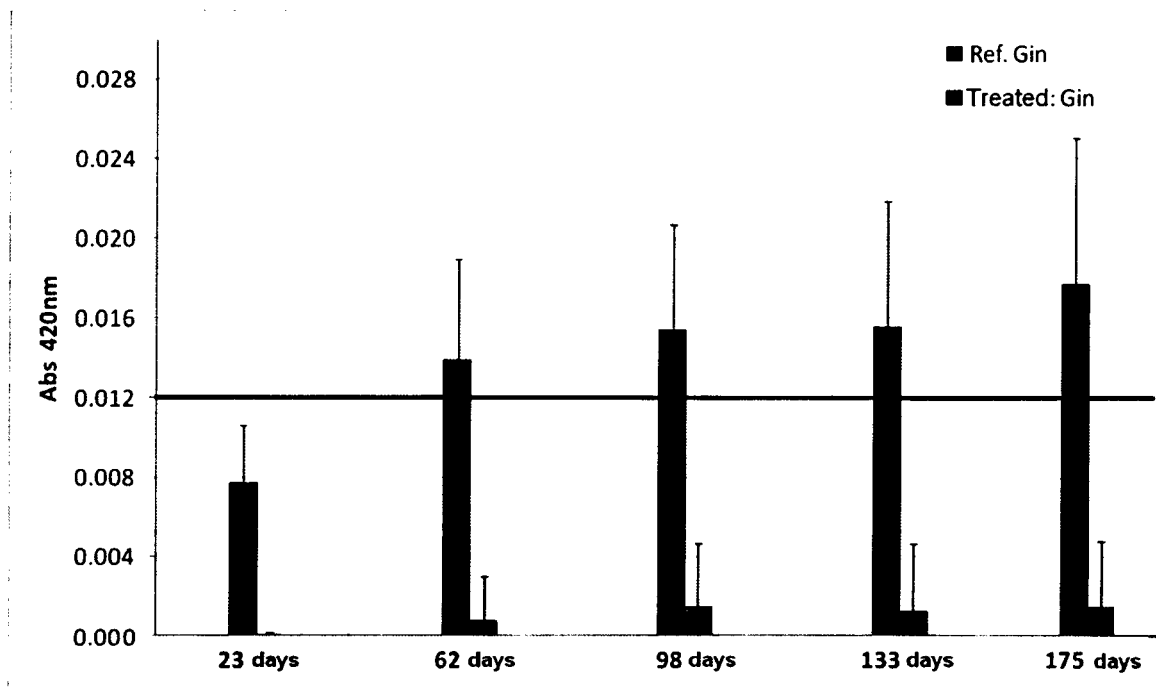


Fig. 4

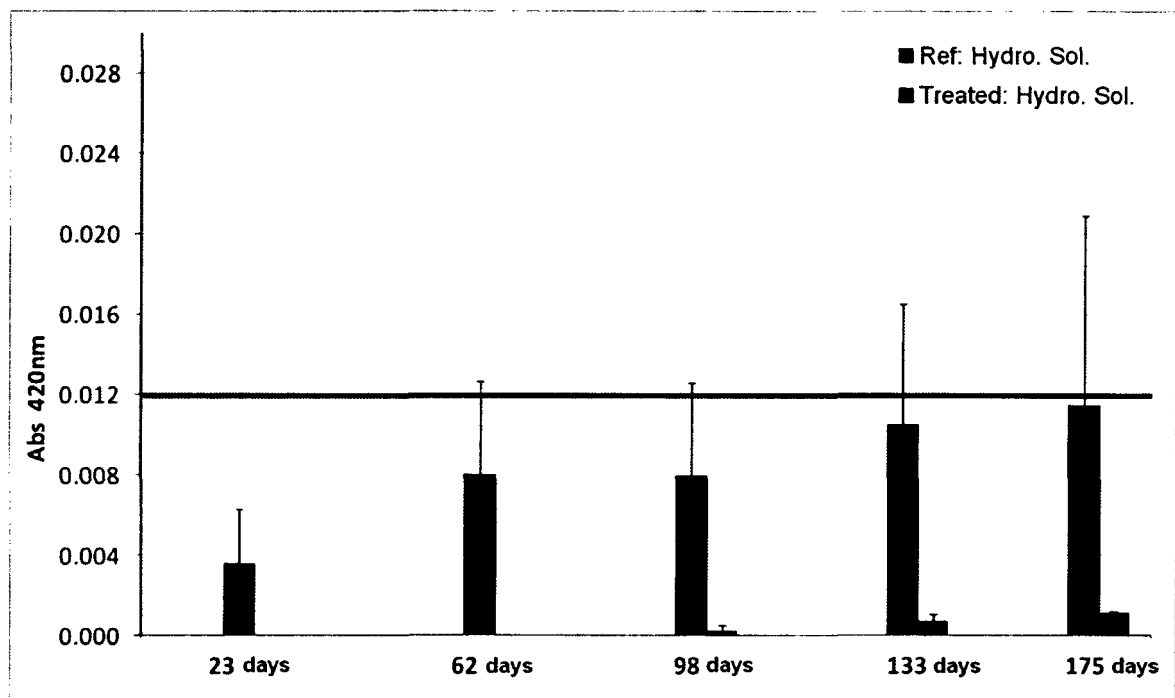


Fig. 5

REFERENCES CITED IN THE DESCRIPTION

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