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(54) **Process for the elimination of the haloanisoles and halophenols present in cork and installation to carry out said elimination**

(57) The present invention belongs to the field of processes for the elimination of the haloanisoles and halophenols present in cork as well as of the installations used to carry out said elimination, where the process for the elimination of halogenated compounds derived from

anisole and phenol present in cork is based on the thermal desorption in gaseous state of said compounds of the cork by the application of vacuum as well as the installation where said process is performed.

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Description**OBJECT OF THE INVENTION**

[0001] The present invention belongs to the field of the processes for the elimination of the haloanisoles and halophenols present in cork as well as of the installations used to carry out said elimination.

[0002] The main object of the present invention is a process for the elimination of halogenated compounds derived from anisole and phenol present in cork based on the thermal desorption in gaseous state of said compounds of the cork by the application of vacuum, as well as the installation where the previous process is carried out.

BACKGROUND OF THE INVENTION

[0003] Cork is a natural material, from the bark of a tree called "Quercus Suber". Its cellular structure is formed by units filled with air, disposed in a very regular manner.

[0004] Normally, its chemical composition varies in accordance with the type of cork, its origin and the age of the tree. The more typical components of cork are: suberin (in the order of 46%) , which is responsible for the great compressibility and elasticity of cork; lignin (25%) , cellulose and other polysaccharides (12%) , waxy substances (6%) , which are mainly responsible for the impermeability of the cork, tannins (6%) , and other compounds (5%) .

[0005] Its chemical composition, together with its characteristic structure give the cork physical properties that make it a material with multiple applications, among which we can highlight the manufacturing of stoppers for the closure of containers, for example bottles of wine, brandy, champagne, cava, whisky, etc. In this application, it is vital that the stopper respects the organoleptic characteristics of the content of the container.

[0006] In this regard, the cork industry has suffered what is called "corking", i.e. the appearance of an unpleasant taste and/or aroma in drinks where the bottles have been sealed with a cork stopper. This smell or taste, when present, deteriorates the quality of the bottle content, which involve significant economic losses for the industries.

[0007] Today, it is very well known that the substances responsible for this effect are halogenated compounds derived from anisole. Among them, we can highlight 2, 3, 4, 6- tetrachloroanisole (TeCA) , pentachloroanisole (PCA) , 2, 4, 6-tribromoanisole (TBA) , but especially 2, 4, 6- trichloroanisole (TCA) .

[0008] All these haloanisoles are highly contaminating, capable of ruining the organoleptic properties of any liquid, producing very unpleasant aromas and tastes, which could be called fungal or a mould. This is very possible due to their high volatility. Their olfactory and taste perception threshold is very low, so that a very small quantity is perceived either through taste or smell. For the case of TCA, 1.5-3 ng/l of said compound in an alcoholic solution such as wine, is sufficient to be detected.

[0009] Contamination by haloanisoles has its origin in other compounds, halophenols. These substances have been largely used as pesticides despite being highly contaminating and remaining in the atmosphere during long periods of time. Therefore, halophenols can be found as contaminants in soil, water and the atmosphere. Due to the high toxicity of these compounds, some microorganisms present in cork cannot degrade them when they come into contact with these compounds, for which reason they transform them into haloanisoles, through a biomethylation reaction. Therefore, it can be said that the true origin of cork contamination by haloanisoles is an environmental contamination problem.

[0010] The economic losses suffered by industries throughout the world due to haloanisole contamination are enormous and difficult to calculate. Therefore, there is growing interest in eliminating these contaminants from cork. Despite the great effort that has been made to resolve this problem, there is currently no treatment that is totally effective, having developed different strategies such as avoiding the formation of haloanisoles, avoiding the migration of haloanisoles to the liquid or eliminating the haloanisoles found in cork.

[0011] Document ES2076185 details a process based on washing the stopper with an aqueous solution of alkaline nature, in order to avoid the formation of haloanisoles by the elimination of the microorganisms that produce them.

[0012] A similar process is disclosed in patent ES2255458, which considers the inhibition of the substances causing the bad smell, by the addition and enrichment of cork with carbonate and bicarbonate salts of alkaline and earth alkaline metals. In this case, the invention is based on causing a chemical change that means the microorganisms are modified, with the consequent inhibition in haloanisole formation.

[0013] Microwave radiation has been proposed in order to eliminate haloanisoles from the stopper in patent ES2310030 where it states that the action of this radiation sterilises the cork and consequently reduces its contamination. The process attempts to go beyond this, not just treating the surface, but applying a variable power that manages to reach the inside of the cork.

[0014] There are other techniques using electronic accelerators that, via β emissions, eliminate the microorganisms with the consequent total sterilization of the cork stopper.

[0015] The use of physical barriers to prevent haloanisoles from coming into contact with the liquid is another of the strategies used. This field includes document ES2148796 the process whereof is based on coating the cork, or cork

derivative, with a substance, generally silicone, which prevents the contaminating substances from the stopper coming into contact with the packaged liquid.

[0016] All these processes are aimed at preventing the formation of the contaminant or that it comes into contact with the packaged liquid, but none of them is capable of eliminating the haloanisoles and other already existing contaminants.

To resolve this problem, different alternatives have been proposed based on different physicochemical products.

[0017] An elimination process based on an absorption process has been proposed in document WO01/41989. This consists of eliminating the contaminant present in cork, by contact with an aqueous suspension of activated charcoal, from coconut shell. Suitable conditions will favour the migration of the contaminant to the active charcoal, being adsorbed in it.

[0018] Another apparently effective physicochemical process for the elimination of contaminants is extraction. In this context, numerous processes have been proposed.

[0019] Patent ES2216965 carries out the extraction with a dense fluid, proposing CO₂ as most appropriate. In accordance with this invention, pressurized CO₂ (100-300 bar), is placed in contact with the cork at a certain temperature (40-80 °C), thus managing to eliminate the undesired substances. This process is also used to extract numerous compounds in cork, for which reason the addition of a co-solvent is advised to improve the extraction selectivity. The main drawback of this process is its high cost.

[0020] A variant of this method can be considered in document EP2033751, wherein the inert gas (carbon dioxide, nitrogen or noble gases such as argon or mixtures) is used to treat the cork with the same purpose. In this case, the pressure conditions under which the process is carried out are moderated, making the process significantly cheaper.

[0021] On the other hand, the extraction of haloanisoles with steam has become one of the most typical processes, with numerous variants having been proposed.

[0022] A widely known process is that set down in document ES2268459, where the significant extraction of compounds present in cork, in particular TCA, is carried out by steam distillation. The steam and the water at a certain pressure (less than 0.8 bar) make it possible to expulse the contaminants present inside the cork cells. This process is similar to that included in patent ES2019562, with the exception that the first makes it possible to treat the cork in all its varieties, whether granulated cork, discs or stoppers, whilst the second is only considered valid for small-sized cork. Furthermore, the latter comprises some additional stages, such as grinding, preheating and cooling of the cork.

[0023] Another process that uses steam for the extraction of the contaminant is disclosed in patent ES2237133. In this case, the process is favoured by the use of pressurized steam (2-30 bar). The treatment has a short duration, under 1 minute and is followed by a fast and brutal expansion, until the receptacle reaches atmospheric pressure.

[0024] Another form of favouring extraction with steam is including other components in the stream. Thus, patent ES2247180 contemplates the use of gaseous mixtures formed by steam and an organic solvent, specifically ethanol, in the presence of air and semi-continuously.

[0025] Patent ES2259547 proposes the elimination of the contaminants by a process based on extraction with a liquid, generally water with surfactants, which is performed in three stages. In the first, the cork is placed in contact with the liquid, providing heat energy and creating a vacuum atmosphere (low pressure). That depression makes the bubbles of air trapped in the cork pores escape to the outside, facilitating the entry of the liquid intended for decontamination. In the second stage, the cork is left at rest and in the third it is dried applying different methodologies known in the sector.

[0026] Furthermore, the olfactory and taste perception threshold of haloanisoles is so low (for example, for the case of TCA 1.5- 3.5 ng/l in a hydroalcoholic solution (wine) is sufficient to be detected) which require haloanisole elimination levels so high that the majority of the aforementioned methods do not manage to achieve this.

[0027] On other occasions, the cost of the installations, processing and the energy used are so high that they make many of the processes achieved unfeasible.

[0028] On the other hand, cork is a delicate material, with a chemical structure and highly defined properties. Vigorous treatments, wherein high temperatures and/or pressures are used produce significant alterations and degradations in its structure and composition. Others, methods based on the extraction of contaminants with liquid solvents (water, ethanol, etc.) or gaseous (steam, CO₂, etc.) eliminate other valuable components of cork, such as waxes, suberin, tannins, etc. together with the contaminants, thus altering its properties.

[0029] On other occasions, wherein substances are used to eliminate the contaminants, they in turn contaminate the cork.

[0030] The applicant is not aware of the existence of treatments that combine the conditions necessary to eliminate the haloanisoles present in cork effectively, for all forms of cork, both in large pieces and granular, without having to carry out a previous dissolution in a liquid or in a supercritical fluid for the later extraction of the contaminants, and where said processes do not cause alterations in the chemical composition and structure of the cork.

DESCRIPTION OF THE INVENTION

[0031] The present invention relates to a process for the elimination of the haloanisoles and halophenols present in

cork comprising a stage of thermal desorption in gaseous state of the haloanisoles and halophenols present in cork by the application of a vacuum pressure.

[0032] To achieve and guarantee that the haloanisoles and halophenols are found in gaseous state, the desorption stage must take place at a temperature higher than the boiling temperature of said contaminant compounds, i.e. of haloanisoles and halophenols, temperatures that vary depending on the vacuum pressure applied to the cork.

[0033] The melting and boiling points of said contaminant compounds quickly decrease when the pressure is reduced, so that if it is very low the compounds will be in gaseous state at relatively low temperatures. Table 2 shows how the boiling point of 2, 4, 6- trichloroanisole (TCA) changes with pressure. In accordance with these data, TCA at a pressure of 0.1 mbar would be in gaseous state at 19.5 °C; at 1 mbar it would require 56 °C; at 10 mbar 98 °C and at 1000 mbar 239 °C. Thus, the thermal desorption of those contaminants can be performed at moderate temperatures, if very low pressures are used.

[0034] Table 1 shows said points for the haloanisoles and halophenols most frequently found in cork. In general they are compounds with high boiling points that increase as the number of substituted chlorines in the molecule increases. Haloanisoles have slightly lower melting and boiling points than halophenols, for which reason they are rather more volatile.

Table 1. Melting and boiling points of halophenols and haloanisoles at atmospheric pressure.

Compound	Melting temp (°C)	Boiling temp (°C)
2,4,6-trichlorophenol (TCF)	69.5	246
2,3,4,6-tetrachlorophenol (TeCF)	115	288
pentachlorophenol (PCF)	190	310
Tribromophenol (TBF)	95	286
2,4,6-trichloroanisole (TCA)	60-62	240
1,2,3,5-tetrachloroanisole (TeCA)		280
pentachloroanisole (PCA)		305
Tribromoanisole (TBA)	88	298

[0035] These melting and boiling points rapidly decrease when the pressure is reduced below atmospheric pressure, i.e. in vacuum conditions, so that if it is very low, the compounds will be in gaseous state at relatively low temperatures. Table 2 shows how the boiling point of TCA changes with pressure. In accordance with these data, TCA at a pressure of 0.1 mbar would be in gaseous state at 19.5 °C; at 1 mbar it would require 56 °C; at 10 mbar 98 °C and at 1000 mbar 239 °C. Thus, the thermal desorption of those contaminants is possible at ambient temperature, if vacuum pressures in the order of 0.01 mbar are used.

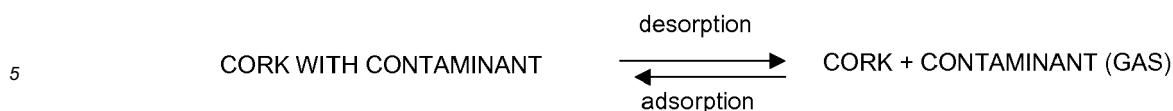
Table 2. Evolution of the boiling temperature of 2,4,6-trichloroanisole (TCA) with the pressure.

Pressure (mbar)	Boiling temperature (°C)
0.05	10
0.10	19.5
0.50	44.5
1	56
10	98
100	157
500	215
1000	239

[0036] These vacuum conditions in the desorption process are not only aimed at lowering the boiling point of the haloanisoles and halophenols present in cork, but also displacing the adsorption-desorption balance established, in favour of desorption.

[0037] The contaminants remain in the structure of the cork adsorbed in its surface in accordance with the following

dynamic balance:



[0038] This balance is displaced in favour of desorption as the vacuum conditions remove the molecules of the haloanisole and halophenol contaminants that are desorbed from the cork structure. That displacement of the balance in favour of desorption is also favoured by a temperature rise, since with heat the bonds are broken that keep the contaminants bound to the cork surface. These bonds can be strong, for which reason it does not only need to reach a certain temperature so that the contaminant is in gaseous state, but it also needs a somewhat higher temperature to break those bonds.

[0039] The invention also relates to the installation to carry out the elimination process of the haloanisoles and halo-phenols present in cork described above.

[0040] The installation comprises a tank where the cork to be treated is placed, a tank which is capable of withstanding vacuum pressure conditions of at least 0.01 mbar. The tank also comprises a heating device of the cork.

[0041] The installation further comprises a vacuum application device which is connected to the tank for the application of the vacuum pressure.

20 **[0042]** The cork on which said process for the elimination of the haloanisoles and halophenols is applied and which is contained in the installation tank also described above can be in any form and size: pieces of bark, sheets, discs, stoppers, granules, etc.

[0043] The main advantages provided by the process and installation of the present invention with respect to the known processes and installations are:

- 25
- It is a simple process that is easy to carry out since it is performed in a single stage.
 - No additional substance is used during the process, so that there is no possibility of contamination of the cork.
 - The costs of the installation are very low if compared with that of other processes, such as that of extraction with supercritical CO₂, steam, etc.
- 30
- The handling and energy costs are very small, since the temperatures at which the cork is heated are low and in some cases they may not exist. On the other hand, the energy costs to make the corresponding vacuum are very reduced.
 - It allows treating large volumes of cork in a single treatment without excessively increasing the prices of the installation, making the process very economical. Tanks of 10, 20 m³ and greater can be easily used.
- 35
- The control over the temperature and the vacuum also allow the cleaning process of the cork to be easily controlled, avoiding its degradation and the elimination of valuable substances such as waxes, tannins, etc.
 - It can be applied to any format and size of cork such as: pieces of bark, large sheets, discs, stoppers, granular cork, etc.
 - It can be used with cork recently taken from the tree and before or after mechanical and/or chemical treatments that are generally known and typical in cork processing, such as boiling or treatment with hot water.
- 40
- It can be used after other cork purification treatments, which due to their limitations cannot achieve the desired degree of cleaning.

DESCRIPTION OF THE DRAWINGS

45 **[0044]** To complement the description being made and in order to aid towards a better understanding of the characteristics of the invention, in accordance with a preferred example of practical embodiment thereof, a set of drawings is attached as an integral part of said description wherein, with illustrative and non-limiting character, the following has been represented:

50 Figure 1.- Shows a diagrammatic view of the installation for the elimination of the haloanisoles and halophenols present in cork of the present invention.

PREFERRED EMBODIMENT OF THE INVENTION

55 **[0045]** Below, a preferred and non- limiting embodiment is described of the process for the elimination of the haloanisoles and halophenols present in cork of the present invention, as well as of the installation to carry out said elimination process.

[0046] The elimination process comprises a stage of thermal desorption in gaseous state of the haloanisoles and

halophenols of the cork, by application of a vacuum pressure.

[0047] Preferably, the vacuum pressure is in the range between 1000 mbar and 0.01 mbar, and the temperature between 310 °C and 0 °C, so that the process would eliminate the following compounds to a level below the olfactory and taste perception threshold in the event that the cork is used as the stopper of an alcoholic solution such as wine:

2,4,6-trichlorophenol (TCF)
2,3,4,6-tetrachlorophenol (TeCF)
pentachlorophenol (PCF)
Tribromophenol (TBF)
2,4,6-trichloroanisole (TCA)
1,2,3,5-tetrachloroanisole (TeCA)
pentachloroanisole (PCA)
Tribromoanisole (TBA)

[0048] where the perception threshold for the previous compounds is 1.5-3 ng/l of compound in an alcoholic solution such as wine.

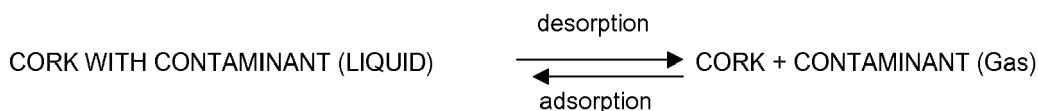
[0049] More preferably, the vacuum pressure is in the range between 1000 mbar and 0.01 mbar, and the temperature for the thermal desorption in cork of between 246 °C and 0 °C, so that the process manages to eliminate the following compounds to a level below the olfactory and taste perception threshold in the case that the cork is used as the stopper of a bottle of an alcoholic solution such as wine:

2,4,6-trichlorophenol (TCF)
2,4,6-trichloroanisole (TCA)

where the perception threshold for the previous compounds is situated at 2.5 ng/l of compound in an alcoholic solution such as wine.

[0050] More preferably, the selective elimination of 2, 4, 6- trichloroanisole (TCA) is carried out by applying a vacuum pressure of 0.1 mbar with temperature conditions of thermal desorption of at least 19.5 °C; or applying a vacuum pressure of 1 mbar with temperature conditions of thermal desorption of at least 56 °C; or by applying a vacuum pressure of 10 mbar with temperature conditions of at least 98 °C or by applying a vacuum pressure of 1000 mbar with temperature conditions of thermal desorption of at least 239 °C, in addition to any other pressure- temperature pair included in the previous extreme values and proportional thereto and that a person skilled in the art would be capable of formulating in light of the previous values.

[0051] Temperatures and vacuums wherein the haloanisoles do not reach boiling point, but they do reach melting point melting, could also be used. In this case, the haloanisole would be in liquid state, although there would exist a liquid-gas balance.



[0052] In this way, as there is a continued vacuum that removes the contaminant (gas), the cork would end up free from contaminant.

[0053] In these vacuum and temperature conditions, wherein the contaminant contained in cork would be in liquid state, the desorption process is much slower and less efficient than when vacuums and temperatures are used that guarantee that the contaminant is in gaseous state; for which reason the use of those vacuums and temperatures are less recommendable.

[0054] The time of the desorption stage is another variable that can be controlled in said process. The longer the time the cork is subjected to the thermal desorption stage, the greater the elimination of the haloanisoles and halophenols will be.

[0055] In short, it can be said that the thermal desorption process can be controlled in accordance with the temperature,

the pressure and the treatment time.

[0056] These three variables make it possible to operate in highly varied conditions:

- With high temperatures and large vacuums, the treatment time is shortened since the process is fast. However, in these conditions other less volatile substances than those present in cork can also be desorbed, such as waxes, tannins etc., and if the temperature is very high the cork may suffer degradation.
- With low temperatures and small vacuums, the time is lengthened and the least volatile contaminants may not be totally eliminated; however, the chemical composition and structure of the cork will remain very stable.
- Intermediate temperatures and large vacuums may be the most recommendable conditions.

[0057] Pressures below atmospheric pressure, up to 0.05 mbar and even lower, may be used. However, very large vacuums are difficult to achieve and require more expensive equipment.

[0058] On the other hand, cork is quite stable up to temperatures of 135 °C, so that higher temperatures can only be used during short periods of time. Temperatures between 100 and 135 °C are the most recommendable. Lower temperatures, even up to 0 °C, can be used, although the treatment time is lengthened, but they have the advantage that the energy cost to heat the cork is reduced or disappears. In this way, the range of usable temperatures can extend preferably from 0° C to approximately 240 °C.

[0059] Another important aspect to bear in mind is the quantity of water retained by the cork. Natural cork can retain water from environmental humidity or from previous treatments to which they have been subjected, such as boiling, steam extraction, etc, reaching values of 3-15%.

[0060] As water is a compound with a boiling point somewhat lower than that of haloanisoles and it is in larger amounts than them, during the thermal desorption process under vacuum, it is going to be preferably desorbed, consuming a great amount of energy during the process. Therefore, it is advisable to apply the thermal desorption treatment under vacuum with a cork which does not exceed a degree of humidity of 20%.

[0061] Previous studies performed in the laboratory have revealed that the efficiency and rapidity of thermal desorption under vacuum is greater if the contaminants present in cork (haloanisoles and halophenols) have been subjected to a recrystallization process, by means of boiling the cork in water and later drying, immediately before thermal desorption, so that the haloanisoles and halophenols are in recrystallized state.

EXAMPLE 1.

[0062] The following results were obtained in several tests performed in the laboratory:

Granular cork was used with a TCA contamination of 10.43 ng/l.

Applying the process of the invention during 10 hours at 100 °C and at a pressure of 0.1 mbar reduced its TCA level to 2.5 ng/l.

Applying the process of the invention during 10 hours at 115 °C and at a pressure of 0.1 mbar reduced its TCA level to 2.3 ng/l.

Applying the process of the invention during 15 hours at 100 °C and at a pressure of 0.1 mbar reduced its TCA level to 0.3 ng/l,

all under the TCA perception threshold in an alcoholic solution such as wine.

EXAMPLE 2.

[0063] As has been previously commented, pressures below atmospheric, up to 0.05 mbar and even lower, may be used.

[0064] In this regard, again using the granular cork with a TCA contamination of 10.43 ng/l and with vacuum pressures lower than that used in Example 1, it is observed that as the vacuum pressure decreases, the necessary time to reduce TCA levels to low and even zero levels, is increasingly lower, so that at vacuum pressures of 10^{-4} - 10^{-5} mbar the TCA levels are practically zero.

[0065] It has been observed that this reduction in the treatment time is very important since the process is more efficient and economical.

[0066] The following table shows the effect of the vacuum pressure in the cleaning treatment of samples of granular cork with an initial TCA concentration detected by migration of 10.43 ng/L.

Vacuum pressure (mbar)	Temperature (°C)	Treatment time (hours)	TCA level detected by migration (ng/l)	Total TCA (ng/g)
1.5 10 ⁻²	100	6	0.4	0,87
1 10 ⁻³	115	3	0.2	0.43
2 10 ⁻⁴	115	2	0.0	0.25
5 10 ⁻⁵	115	2	0.0	0.03
1 10 ⁻⁵	115	2	0.0	0.01

[0067] In the alcoholic drink cork stopper industry, contamination caused by TCA is determined by measuring (using UNE 56930 standard) the quantity of TCA capable of migrating from the cork to a hydroalcoholic solution which simulates wine. Logically, it is measured in ng of TCA/L of solution. The fact that values are obtained of 0.0 ng/L does not mean that the cork is totally free from TCA, but that it is not capable of migrating to the solution in the conditions established in the standard. It is for this reason that the Total TCA Content in cork is also shown in the previous table (in ng of TCA/g of cork).

[0068] Generally, stopper manufacturers and bottlers are more interested in the TCA migration level from the stopper to the wine, requiring values lower than 0.4 ng/L. They are currently starting to be more demanding and also require smaller total TCA levels, lower than 0.3- 0.4 ng/g.

[0069] Figure 1 shows the diagram of an installation to carry out the thermal desorption in vacuum conditions of the haloanisoles and halophenols present in cork of the aforementioned elimination process.

[0070] The installation comprises a tank (1) where the cork (3) to be treated is placed, a tank (1) which is capable of withstanding vacuum pressure conditions of up to at least 0.01 mbar. The tank (1) also comprises a device (2) for heating the cork (2), which is preferably a heat exchanger. The tank (1) is thermally insulated to minimize heat losses and thus reduce heating costs.

[0071] The installation also comprises a vacuum application device (5), which is preferably a vacuum pump, connected to the tank (1).

[0072] The installation also comprises a collection device (4) of the desorbed contaminants, i.e. of the haloanisoles and halophenols, which is disposed between the tank (1) and the vacuum application device (5) the mission of which is to collect the contaminants and any other desorbed substance. This avoids the contaminants from being emitted to the outside and becoming a new source of contamination of the cork. It also serves as a protection filter of the vacuum application device (5) which could be damaged by the desorbed substances. This collection device (4) of the desorbed contaminants may be a condenser, a cold trap, an adsorbent bed, or similar.

Claims

1. Process for the elimination of the haloanisoles and halophenols present in cork, **characterized in that** it comprises a stage of thermal desorption in gaseous state of the haloanisoles and halophenols present in cork by the application of a vacuum pressure.
2. Process for the elimination of the haloanisoles and halophenols present in cork according to claim 1, wherein the vacuum pressure is in the range between 1000 mbar and 0.01 mbar with a temperature of thermal desorption in cork of between 310 °C and 0 °C.
3. Process for the elimination of the haloanisoles and halophenols present in cork according to claim 2, wherein the vacuum pressure is in the range between 1000 mbar and 0.01 mbar with a temperature of thermal desorption in cork of between 246 °C and 0 °C.
4. Process for the elimination of the haloanisoles and halophenols present in cork according to claim 3, wherein the vacuum pressure is of 0.1 mbar with a temperature of thermal desorption of at least 19.5 °C.
5. Process for the elimination of the haloanisoles and halophenols present in cork according to any of claims 1 to 3, wherein the stage of thermal desorption is applied during 6 hours at 100 °C and at a pressure of 0.015 mbar to reduce the 2,4,6-trichloroanisole (TCA) content from 10.43 ng/l to 0.4 ng/l.
6. Process for the elimination of the haloanisoles and halophenols present in cork according to claim 1, wherein the

stage of thermal desorption is applied during 3 hours at 115 °C and at a pressure of $1 \cdot 10^{-3}$ mbar to reduce the 2,4,6-trichloroanisole (TCA) content from 10.43 ng/l to 0.2 ng/l.

7. Process for the elimination of the haloanisoles and halophenols present in cork according to claim 1, wherein the stage of thermal desorption is applied during 2 hours at 115 °C and at a pressure of $2 \cdot 10^{-4}$ mbar to reduce the 2,4,6-trichloroanisole (TCA) content from 10.43 ng/l to 0.0 ng/l.
8. Process for the elimination of the haloanisoles and halophenols present in cork according to claim 1, wherein the stage of thermal desorption is applied during 2 hours at 115 °C and at a pressure of $5 \cdot 10^{-5}$ mbar to reduce the 2,4,6-trichloroanisole (TCA) content from 10.43 ng/l to 0.0 ng/l.
9. Process for the elimination of the haloanisoles and halophenols present in cork according to claim 1, wherein the stage of thermal desorption is applied during 2 hours at 115 °C and at a pressure of $1 \cdot 10^{-5}$ mbar to reduce the 2,4,6-trichloroanisole (TCA) content from 10.43 ng/l to 0.0 ng/l.
10. Process for the elimination of the haloanisoles and halophenols present in cork according to any of claims 1 to 4, wherein the cork is presented in the form of piece of bark, sheet, disc, stopper or granules.
11. Process for the elimination of the haloanisoles and halophenols present in cork according to claim 10, wherein the cork has a degree of humidity of up to 20%.
12. Process for the elimination of the haloanisoles and halophenols present in cork according to claim 1, wherein it comprises an additional stage of recrystallization of the haloanisoles and halophenols, before thermal desorption.
13. Process for the elimination of the haloanisoles and halophenols present in cork according to claim 12, wherein the recrystallization comprises boiling in water and later drying of the cork.
14. Installation to carry out the process of elimination of the haloanisoles and halophenols present in cork according to any of the preceding claims, **characterized in that** it comprises a tank (1) where the cork (3) to be treated is placed and a vacuum application device (5) which is connected to the tank (1) for the application of the vacuum pressure.
15. Installation to carry out the process of elimination of the haloanisoles and halophenols present in cork according to claim 14, wherein the tank (1) comprises in turn a heating device (2) of the cork.

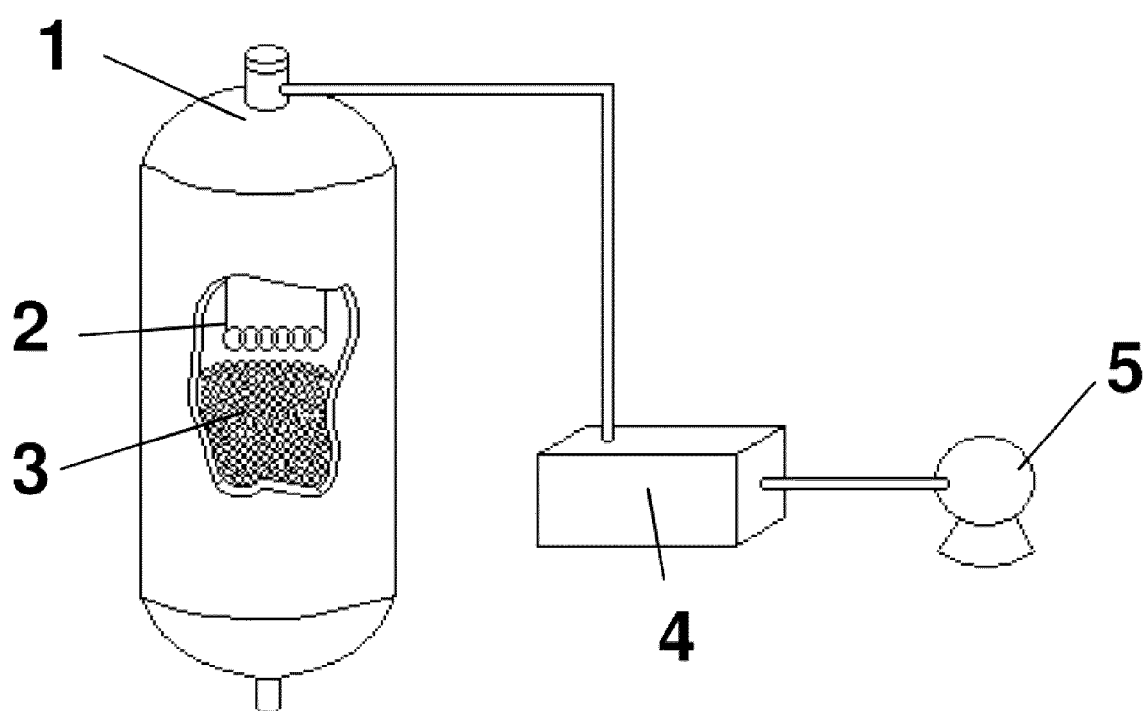


FIG. 1



EUROPEAN SEARCH REPORT

Application Number
EP 13 15 8850

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The present search report has been drawn up for all claims			
Place of search Munich		Date of completion of the search 4 June 2013	Examiner Bjola, Bogdan
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