(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) EP 1 551 463 B1

(12) **EUROPEAN PATENT SPECIFICATION**

(45) Date of publication and mention of the grant of the patent:
21.06.2006 Bulletin 2006/25

(21) Application number: **03784710.0**

(22) Date of filing: **08.08.2003**

(51) Int Cl.:
A61L 2/07 *(2006.01)*

(86) International application number:
PCT/PT2003/000010

(87) International publication number:
WO 2004/014436 (19.02.2004 Gazette 2004/08)

(54) **CORK PRODUCT TREATMENT PROCESS BY EXTRACTION OF COMPOUNDS DRAGGED IN WATER VAPOUR**

KORK-PRODUKT-BEHANDLUNGSVERFAHREN DURCH EXTRAKTION VON VERBINDUNGEN, DIE IN WASSERDAMPF MITGESCHLEPPT WERDEN

PROCEDE DE TRAITEMENT DE PRODUITS EN LIEGE PAR EXTRACTION DE COMPOSES ENTRAINES DANS LA VAPEUR D'EAU

(84) Designated Contracting States:
AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HU IE IT LI LU MC NL PT RO SE SI SK TR

(30) Priority: **13.08.2002 PT 10282902**

(43) Date of publication of application:
13.07.2005 Bulletin 2005/28

(73) Proprietor: **Amorim & Irmaos S.A.**
4535 Santa Maria de Lamas Codex (PT)

(72) Inventor: **CABRAL, Miguel**
4535 Santa Maria de Lamas Codex (PT)

(74) Representative: **Magalhaes Simoes, José Raul**
Simoes, Garcia, Corte-Real
& Associados - Consultores, Lda.
Av. Estados Unidos de América, No 131,7o-C
1700-173 Lisboa (PT)

(56) References cited:
EP-A- 0 931 553
EP-A- 1 224 946
WO-A-01/37887
US-A- 2 380 696

Note: Within nine months from the publication of the mention of the grant of the European patent, any person may give notice to the European Patent Office of opposition to the European patent granted. Notice of opposition shall be filed in a written reasoned statement. It shall not be deemed to have been filed until the opposition fee has been paid. (Art. 99(1) European Patent Convention).

EP 1 551 463 B1

Description

[0001] The present invention refers to a cork product treatment processes by extraction of compounds dragged in water vapour.

[0002] The process of the invention; hereinafter referred to as ROSA system, is based on the application of water vapour to cork granules, disks, and stoppers in order to achieve its deodorization, namely eliminating all the compounds dragged in water vapour, among which the 2,4,6-trichloroanizole.

[0003] Being a highly absorptive material, cork has an enormous ability to absorb compounds present in the cork environment, particularly those that present an unpleasant flavour and/or smell. These compounds can be transferred to the products contacting the cork, which, due to said contact, will often be damaged beyond repair.

[0004] Cork finds one of its most significant uses as a sealing element, namely as natural cork stoppers, composite stoppers, technical stoppers, Champagne stoppers, and stoppers with a capsule element. These products are used to seal wine products, which often have an aroma delicacy that is not compatible with any unpleasant flavour transmission from the packaging materials, namely from the sealing element.

[0005] However, there are many possible deleterious products that can be transmitted through cork stoppers to wine products.

[0006] Thus, compounds such as 2,4,6-trichloroanizole, 2,3,4,6-tetracholoroanizole, guaiacole, geosmine, 1-octen-3-one, 1-octen-3-ol, and methylisoborneol, are responsible for unpleasant aroma transmission to wine products, their presence in such products being often associated to the cork-sealing element. However, not all compounds have the same relevance in unpleasant aroma transmission to the wine products. 2, 4, 6-trichloroanizole, hereinafter referred to as TCA, is the most problematical because, among all the relevant compounds, is the one having the lowest sensorial detection limit, reaching values in the range of 2-6 ng/1. This compound transmits to the wine products aromas that are described as mouldy that render those wine products completely unfit. Presently, and mainly due to the presence of TCA in wines, alternative plastic sealing elements have been developed that, although without a technical performance similar to the cork sealing elements, endanger the cork industry since they do not transmit TCA to the wines.

[0007] The ROSA system is intended to greatly reduce compounds having unpleasant aromas, and particularly the TCA, in cork products, namely in cork granules, disks, and stoppers, so that the cork products will be no longer considered the cause of the wine product contamination by the above-mentioned compounds. Only this way the alternative sealing elements will no longer have a reason to exist and the cork products will regain the rightful market place that has been theirs for centuries.

State of the art

[0008] The strategies developed in order to solve the problem of TCA in cork products involve preventive and treatment aspects. The first of these aspects aims to avoid conditions appropriated to the TCA formation or to its migration to the wine products, and the second aspect deals with the treatment of cork products in order to remove the already existing TCA. Washing of cork products with hydrogen peroxide solution, the final treatment of these products with ozone or microwaves, the use of physical barriers, namely silicone ones, in order to prevent TCA from contacting with wine products, supercritical TCA extraction, with fluid carbon dioxide, from cork products, enzyme treatment, and cork granule treatment with water vapour are existing processes having as object the TCA removal in industrial plants.

[0009] Cork washing with hydrogen peroxide is a routine bleaching process used in the cork industry. The washing process is carried out at an industrial scale using a drum for natural, technical, and composite stoppers. Hydrogen peroxide, with its oxidative properties is known to act as disinfectant, therefore contributing to diminish the microbial load in cork stoppers. Microorganisms, and particularly fungi, are able to produce TCA due to their metabolism in the presence of primers. Thus, the peroxide washing acts as a preventive process in cork stopper contamination with TCA. Unfortunately it is not an effective process insofar as TCA contamination persists in cork products, despite the use of this washing process in the cork industry for the last 15 years.

[0010] Cork product ozonization is a process used in industry for microbial load reduction having in mind to prevent TCA production in the presence of primers. Ozonization can be simultaneous with the washing, by ozone injection into the washing water or by exposing the cork stoppers to a saturated ozone atmosphere. This process efficiency in terms of microbial load is well established, but TCA level reduction by ozone oxidative action is very doubtful. Industrial scale essays vie conducted showed no such reductions of TCA levels due to direct oxidative effect of ozone in saturated atmosphere.

[0011] The use of microwave in cork stopper TCA decontamination is a System that was presented to the industry as being completely effective. This system is presently used at an industrial level by a worldwide cork manufacturing company, under the designation Delfin. The system has significant effects in microbial load reduction but its effectiveness in TCA reduction is questioned in by Peter Godden, in Technical Review (Godden P. (2000) "Results of sensory and chemical evaluation of a batch of wine corks, to assess the ability of a new processing technique to reduce the incidence

of TCA-taint in wine, highlight the extreme care wineries should take in checking the quality of their closure supplies before use" (*Technical Review 43= 46*)).

[0012] The use of a physical barrier between the cork stopper and the wine product is a process currently used to prevent TCA migration from the cork stopper to said wine product, under the designation Cortex. This product is just a silicone barrier, with about 1 mm thick, adapted to the cork stopper base that contacts the wine in order to prevent TCA migration. Actually, this small silicone disk delays the TCA migration by physical effect and not because silicone is an effective TCA barrier. Thus, Cortex is not useful at all due to the fact that wine products can stay bottled longer than the time needed for TCA to migrate through the silicone disk to the wine products.

[0013] Recently, supercritical fluid CO_2 extraction was proposed as a good solution to solve the TCA problem in cork. This solution, according to its authors, has 97% efficiency and does not deform cork. As the results shown so far were obtained in a small amount of cork, they have to be confirmed at industrial scale. Additionally, the use of this process at industrial scale is expensive and has tight safety rules, since it uses pressures of about 100 bar. Therefore, although the process appears to work well at laboratory level, it is still not developed at an industrial level.

[0014] Another process proposed as being effective in TCA reduction is the use of enzymes, namely lacase-polifenol-oxidase. This enzyme can polymerise phenols therefore preventing their metabolic conversion into anizoles by fungi action. Although the use of this enzyme is a good solution to prevent TCA formation, it does not act on the TCA already present and absorbed in cork products, therefore preventing its polymerisation. Consequently, the already existing TCA remains in cork products and can, therefore migrate to the wine products. Essays made in several laboratories confirm that this product is not effective in eliminating TCA.

[0015] Finally, cork granules treatment with water vapour in autoclave or with vapour jet in an industrial scale drum has been the current practice, either in Portugal (autoclave) or in Japan (vapour jet in an industrial scale drum). The use of autoclave both with cork granules and with cork planks does not seem to significantly reduce TCA in these products. The use of water vapour in a industrial scale drum seems to achieve a significant reduction of TCA in cork granules, but this process has the drawback that it uses a drum that prevents the continuous cork granule treatment, which is essential to the industrial scale feeding of extruding and moulding machinery used in continuous production of champagne, technical and composite stoppers.

[0016] EP-A-1 224 946 discloses an apparatus and process for the decontamination of cork granules in order to minimise the presence of compounds that would generate unpleasant smell. The granules are fed to the apparatus and travel through it by a mixing device have a rotating shaft comprising outlet orifices for a gas in order to thoroughly mix the granules. The shaft is provided with mixing blades and the decontamination is carried out by means of microwave radiation.

Description of the ROSA System for extracting water vapour dragged compounds

[0017] The ROSA System is a system that allows the significant extraction of compounds absorbed in cork products, dragged by water vapour, in particular TCA. This system comprises two different types of apparatus. One is adapted for use with cork granules that continuously extracts said compounds, and the other one is adapted to disks and stoppers, using a drum to clean these products batch wise.

[0018] The present invention will now be described with reference to the annexed drawings, in which:

Figure 1 is a schematic view of the A type apparatus of the present invention;

Figure 2 is a schematic view of the B type apparatus of the present invention;

Figure 3 is a bar graph showing the reduction of 2,4,6-TCA in cork granules with 2/3 mm particle size after the use of the volatile compounds extraction system;

Figure 4 is a bar graph showing the reduction of 2,4,6-TCA in cork granules with 2/3 mm particle size after the use of the volatile compounds extraction system;

Figure 5 is a bar graph showing sensorial analysis of granules treated with the volatile compounds extraction system;

Figure 6 is a bar graph showing the mechanical changes in composite stoppers, manufactured out of cork granule, to which the volatile compounds extraction system was applied;

Figure 7 is a bar graph showing disk visual class reduction after the use of volatile compounds extraction system;

Figure 8 is a bar graph showing significant stopper visual class reduction after the use of volatile compounds

extraction system;

Figure 9 is a bar graph showing disk 2,4,6-TCA reductions after the use of volatile compounds extraction system;

Figure 10 is a bar graph showing ∅ 26 mm disk visual class reduction after the use of volatile compounds extraction system;

Figure 11 is a bar graph showing the natural cork stopper 2,4,6-TCA reduction after the use of volatile compounds extraction system;

Figure 12 is a bar graph showing natural cork stopper visual class change after the use of volatile compound extraction system;

A Type Apparatus - Used with cork granules

[0019] This apparatus (Fig. 1) comprises a stainless steel cylinder, with 2500 mm length and 250 mm diameter, having several openings, namely: four side openings (A) to the inlet of the water vapour coming from a generating source (I), which inlet that can be controlled by faucets (B); an opening (C) located on the underside of the apparatus for granule recovery after treatment; an opening (D) located on the upper side for the exhaustion of the water vapour after granule treatment.

[0020] Inside the cylinder is provided a screw propeller (K) turning about a central shaft, driven by an motor (F) that forces a rotation movement.

[0021] The clearance between the cylinder and the screw propeller is small so that a certain portion of the cork granules that are at a certain pitch of blades of the screw propeller can only proceed to the next pitch of blades by way of the rotation screw propeller motion. The cork granule is unloaded into a feeding hopper, provided with a lock (E) that controls the amount of granules entering the system. The water vapour is generated in an external boiler, the flow rate being controlled by a pressure control valve (J). The system is also equipped with a temperature controller (H) and a pressure gauge (G).

[0022] The A type apparatus of the ROSA system uses the following operating conditions:

Cylinder capacity: 8 kg of granules (with the system stopped)

Temperature: 100ºC to 125ºC

Gauge pressure: 0,2 to 0,8 bar

Contact time: 6 to 65 minutes

Rotation velocity: this operating condition depends On the contact time

System throughput: since this system is a continuous one its throughput depends on the contact time that is used.

B Type Apparatus - Used for cork disks and stoppers

[0023] This apparatus (Figure 2) comprises a stainless steel cylinder, located horizontally along its longitudinal axis, having in one top a lid that tightly seals by means of thread screws (A). Inside this cylinder is provided another smaller cylinder (B) in concentric arrangement regarding the first one, which surface is made of perforated plate. This inner cylinder has a longitudinal opening in order to allow the loading of the cork product to be treated (disks or stoppers). The inner cylinder rotates about a central shaft (C) provided with openings in its surface through which the water vapour is introduced into the apparatus. The water vapour is generated in an external boiler (D), the flow rate being controlled by a pressure control valve (E). The system is also equipped with a temperature controller (F) and a pressure gauge (G).

[0024] The outer cylinder has two further openings:

an opening located on its underside (H) for draining the residual water resulting from small-scale condensation, and

an opening located on the upper side (I) for the exhaustion of water vapour after the treatment.

[0025] After introducing the cork product to be treated in the cylinder, the lid (A) is closed and the system is driven

through the rotation movement imparted by the motor (J) and by the continuous introduction of water vapour inside the system.

[0026] The B type apparatus of the ROSA system uses the following operating conditions:

Capacity: about 10,000 ∅ 26,5 mm x 6,5 mm thick disks and about 2,000 stoppers of a 38 x 24 mm gauge

Temperature: 100°C to 125°C

Pressure: 0,2 to 0,8 bar

Contact time: 6 to 65 minutes

Rotation velocity: 1 to 10 rpm

Efficiency of the ROSA system

[0027] The ROSA system efficiency was assessed in several stages. First it was studied at laboratory scale, followed by pilot industrial scale tests.

[0028] On a laboratory scale it has been used cork granules, disks and stoppers, naturally and experimentally contaminated with TCA in order to verify the TCA reduction magnitude that could be achieved by the system in this compound. The TCA determination was made using the GC-MS coupled with SPME technique, after 24 hours maceration in 10% ethanol. Each granule sample consists of about 2 g of granules with particle dimensions of 1-2 rom. Each disk and stopper sample corresponds to a simultaneous maceration of 50 6x26 mm disks or of 50 28 x 24 mm stoppers.

[0029] The results are shown in Tables 1 and 2. In these tables it is evident the amount of TCA reduction achieved, in some Cases the presence of TCA having been determined in the water vapour condensate after distillation, where large amounts of TCA were found, proving the efficiency of the extraction system by water vapour TCA dragging.

Table 1: TCA before and after treatment by water vapour distillation in TCA naturally contaminated samples

		10 min.		20min.			30 min		60 min.	
	Dimensions									
		Before	After	Before	After	Cond.*	Before	After	Before	After
Granules	0,5-1**	24,6***	7.3	24,6	7,1	79,4	24,6	4		
	2-3			14,2	4	60				
				10,6	1,5	68				
Disks							32,75	1,9	32,75	1,5

*TCA analysis after vapour condensation; **mm; *** TCA ng/l

Table 2: TCA before and after treatment by water vapour distillation in TCA experimentally contaminated samples

		10 min.		20 min.			30 min		60 min.	
	Dimensions									
		Before	After	Before	After	Cond.*	Before	After	Before	After
Granules	3 - 4**	181	37,5				181	32,5	181	28,0
	4 - 6			268,3	50,2	4430				
				323.8	47.8	4458				
				330,7	55,1	4482				
				267,3	50,1	4517				
Disks		41,4	11,1	65,9	3,6	63	41,4	7	41,4	5
				47,4	2,8	63				
				41,3	1,8	63				
Natural Stoppers	33X24			47,4	9,6					

Table continued

Dimensions	10 min.		20 min.			30 min		60 min.	
	Before	After	Before	After	Cond.*	Before	After	Before	After
			92,2	15,9					

* TCA analysis after vapour condensation; **mm; *** TCA ng/l

[0030] Industrial scale pilot tests were performed, namely with the A type apparatus for granules and the B type apparatus for disks and stoppers.

[0031] Regarding the cork granules, Figs. 3, 4 and 5 show the achieved results. Fig. 3 shows the results of 2-3 mm granules treatment in the A type apparatus, using different residence times, one "rapid" module with a treatment of about 6 minutes, and one "slow" module with a treatment of about 20 minutes. Each group consists of 3 dark bars and 3 light ones, referring the same type of granule that are sampled 3 times before treatment and 3 times after treatment. TCA reductions are significant, depending however on the treatment time, being higher after a longer treatment. The average values and the corresponding standard deviations are shown in figure. Fig. 4 refer to the treatment of about 40 kg of granules in the A type apparatus for 20 minutes. Sampling was made before treatment in 24 samples collected, and after treatment the same number of samples was collected. The results show an average reduction of about 83,4%. Fig. 5 shows the results of sensorial analysis to sample pairs before and after a 20 minutes treatment. A panel of 15 trained tasters has been used and they have been asked if they would identify unpleasant aromas in the samples, and when those unpleasant aromas were found within the same pair, which sample had the most intense aroma. The results are evident with almost all of the treated samples considered clean by most of the tasters.

[0032] Since the TCA extraction results Were very good, composite stoppers were produced out of the granules that had been treated in the A type apparatus and their mechanical performance was compared to that of composite stoppers produced with granules of the same batch, but that had not been treated with the ROSA system. The results (Fig. 6) show that there is no significant difference in terms of mechanical performance, showing that the ROSA system does not affect the mechanical properties of the cork granules.

[0033] The B type apparatus was construed for the treatment of natural disks and stoppers, since the A type apparatus produced important deformations in the cork pieces, therefore preventing its intended use as sealing elements or parts of sealing elements (Fig. 7 and 8).

[0034] The TCA reductions achieved with this apparatus in disks were highly significant (Fig. 9) without significant changes in the visual class (Fig. 10). The TCA mean reduction was 75%, while in the visual classes the reduction was 8,6% in class A and 2,3% in class B.

[0035] Tests were made in natural stoppers using the B type apparatus. Again, the results in terms of TCA reduction were very good (Fig. 11), having been achieved average reductions of about 70% and the visual class changes produced by this system having little significance (Fig. 12).

[0036] Considering the above results we can conclude that extraction system of water vapour dragged compounds, particularly TCA, is highly effective in reducing cork granules, disks, and stoppers contamination by these compounds, either using a continuous operated apparatus type for granules or using another type, batch operated, for disks and stoppers.

Claims

1. Cork product treatment process by extraction of compounds dragged in water vapour, to eliminate said compounds, particularly 2,4,6-trichloroanizole that transmit unpleasant taste and/or smell to wine products through stoppers, wherein the treatment is made continuously and without interruptions, in-line with the production circuit, by either an A type apparatus comprising a 2500 mm length and 250 mm diameter stainless steel cylinder, having several openings, namely: four side openings (A) to the inlet of the water vapour coming from a generating source (I), which inlet can be controlled by faucets (B); an opening located on the underside (C) of the apparatus for granule recovery after treatment; an opening located on the upper side (D) for the exhaustion of water vapour after granule treatment, having inside the cylinder a screw propeller (K) rotating about a central shaft, driven by a motor (F) producing a rotating movement, in the case of cork granules for the production of composite stoppers, and technical, and champagne stopper bodies, or **batchwise by** a B type apparatus comprising a stainless steel cylinder, located horizontally along its longitudinal axis, having in one top a lid that is tightly sealed by means of threaded screws (A), inside this outer cylinder being provided another smaller diameter cylinder (B) in concentric arrangement regarding the first

one, whose surface is made of a perforated plate, this inner cylinder having a longitudinal opening allowing the loading of the cork product to be treated consisting of disks or stoppers , the inner cylinder rotating about a central shaft (C) provided with openings along its surface for the introduction of water vapour inside the apparatus, the water vapour being generated in an external boiler (D), the flow rate being controlled by a pressure control valve (E), the apparatus being also equipped with a temperature controller (F) and a pressure gauge (G), the outer cylinder having two more openings: one opening located on its underside (H) for draining the residual water, resulting from small scale condensation; and one opening located on the upper side (I) for the exhaustion of water vapour after the treatment, in the case of natural cork disks for the production of technical, and champagne stoppers.

2. Process according to claim 1, **characterized in that** the A type apparatus is used with the following operating conditions: cylinder capacity: 8 kg of granule with the system stopped; a temperature of 100°C to 125°C; a gauge pressure of 0,2 to 0,8 bar; a contact time of 6 to 65 minutes; and a rotation speed depending on the contact time.

3. **Process according to claim 1, characterized in that the** B type apparatus **is used with** the following operating conditions: a capacity of about 10,000 ∅ 26,5 mm x 6,5 mm thick disks and about 2,000 stoppers with a 38 x 24 mm gauge; a temperature of 100°C to 125°C; a pressure of 0,2 to 0,8 bar; a contact time of 6 to 65 minutes and a rotation velocity of 1 to 10 rpm.

Patentansprüche

1. Behandlungsprozess für Korkprodukte durch Extraktion von Verbindungen, die in Wasserdampf mitgeschleppt werden, um diese Verbindungen zu beseitigen, insbesondere 2,4,6-Trichloranisol, das Weinprodukten einen unangenehmen Geschmack und/oder Geruch durch Verschlüsse verleiht, wobei die Behandlung kontinuierlich und ohne Unterbrechungen während des Produktionskreislaufs durch entweder eine Vorrichtung des A-Typs erfolgt, die einen Edelstahlzylinder mit einer Länge von 2500 mm und einem Durchmesser von 250 mm aufweist, der mehrere Öffnungen hat, nämlich: vier Seitenöffnungen (A) für den Einlass von Wasserdampf, der von einer Generatorquelle (I) kommt, wobei der Einlass durch Hähne (B) gesteuert werden kann; eine Öffnung (C) an der Unterseite der Vorrichtung zur Rückgewinnung von Granulat nach der Behandlung; eine Öffnung (D) an der Oberseite für den Auslass von Wasserdampf nach der Behandlung des Granulats, mit einem Schneckenpropeller (K) im Innern, der sich um eine mittlere Welle dreht, die von einem Motor (F) angetrieben wird, um eine Rotationsbewegung zu erzeugen, im Fall von Korkgranulat zur Herstellung von Verbundverschlüssen, technischen Verschlüssen und Champagner-Verschlusskörpern, oder chargenweise durch eine Vorrichtung des B-Typs, die einen Edelstahlzylinder aufweist, der waagrecht entlang seiner Längsachse angeordnet ist und an einer Oberseite einen Deckel hat, der mittels Gewindeschrauben (A) dicht verschlossen ist, wobei innerhalb dieses äußeren Zylinders ein weiterer Zylinder (B) mit kleinerem Durchmesser in konzentrischer Anordnung zum ersten vorgesehen ist, dessen Oberfläche aus einem perforierten Blech besteht, wobei dieser innere Zylinder eine längliche Öffnung hat, um das zu behandelnde Korkprodukt laden zu können, das aus Scheiben oder Verschlüssen besteht, der innere Zylinder sich um eine mittlere Welle (C) dreht, die mit Öffnungen entlang ihrer Oberfläche zum Einleiten von Wasserdampf in die Vorrichtung versehen ist, wobei der Wasserdampf in einem externen Kessel (D) erzeugt wird, die Durchflussrate von einem Druckregelventil (E) gesteuert wird, die Vorrichtung außerdem mit einem Temperaturregler (F) und einem Manometer (G) ausgerüstet ist und der äußere Zylinder zwei weitere Öffnungen hat: eine Öffnung (H) an seiner Unterseite zum Ablassen des Restwassers aufgrund geringfügiger Kondensation; und eine Öffnung (I) an der Oberseite für den Auslass von Wasserdampf nach der Behandlung im Fall von Naturkorkscheiben für die Herstellung von technischen Verschlüssen und Champagner-Verschlüssen.

2. Prozess nach Anspruch 1, **dadurch gekennzeichnet, dass** die Vorrichtung des Typs A unter folgenden Betriebsbedingungen arbeitet: Fassungsvermögen des Zylinders: 8 kg Granulat bei angehaltenem System; Temperatur 100°C bis 125°C; Überdruck 0,2 bis 0,8 bar; Kontaktzeit 6 bis 65 Minuten; und Drehzahl in Abhängigkeit von der Kontaktzeit.

3. Prozess nach Anspruch 1, **dadurch gekennzeichnet, dass** die Vorrichtung des Typs B unter folgenden Betriebsbedingungen arbeitet: Fassungsvermögen von ca. 10.000 Scheiben mit einem Durchmesser von 26,5 mm und einer Dicke von 6,5 mm und ca. 2000 Verschlüssen der Abmessung 38 x 24 mm; Kontaktzeit 6 bis 65 Minuten und Drehzahl 1 bis 10 U/min.

Revendications

1. Procédé de traitement de produits en liège par extraction de composés entraînés dans de la vapeur d'eau, afin d'éliminer lesdits composés, en particulier, le 2,4,6-trichloroanizole, qui transmettent un goût et/ou une odeur désagréables à des produits vinicoles par l'intermédiaire des bouchons, dans lequel le traitement est réalisé en continu et sans interruptions, à la chaîne dans le circuit de production, soit par un dispositif de type A comportant un cylindre en acier inoxydable d'une longueur de 2 500 mm et d'un diamètre de 250 mm, comportant plusieurs ouvertures, à savoir : quatre ouvertures latérales (A) pour l'admission de la vapeur d'eau provenant d'une source génératrice (I), laquelle admission peut être commandée par des robinets (B), une ouverture située sur le côté inférieur (C) du dispositif pour une récupération des granulés après traitement, une ouverture située sur le côté supérieur (D) pour l'aspiration de la vapeur d'eau après le traitement des granulés, comportant à l'intérieur du cylindre un propulseur à vis (K) tournant autour d'un arbre central, entraîné par un moteur (F) produisant un mouvement rotatif, dans le cas de granulés de liège pour la production de bouchons composites, et de corps de bouchons techniques ainsi que de champagne, soit par lots grâce à un dispositif de type B comprenant un cylindre en acier inoxydable, positionné horizontalement le long de son axe longitudinal, comportant au sommet un couvercle qui est scellé hermétiquement au moyen de vis filetées (A), à l'intérieur de ce cylindre extérieur étant prévu un autre cylindre de diamètre plus petit (B) en agencement concentrique par rapport au premier cylindre, dont la surface est constituée d'une plaque perforée, ce cylindre intérieur comportant une ouverture longitudinale permettant au chargement du produit en liège d'être traité pour constituer des disques ou des bouchons, le cylindre intérieur tournant autour d'un arbre central (C) doté d'ouvertures le long de sa surface pour l'introduction de vapeur d'eau à l'intérieur du dispositif, la vapeur d'eau étant générée dans une chaudière externe (D), le débit étant régulé par une vanne de régulation de pression (E), le dispositif étant également doté d'un régulateur de température (F) et d'un manomètre (G), le cylindre extérieur comportant deux ouvertures supplémentaires : une ouverture située sur son côté inférieur (H) pour une évacuation de l'eau résiduelle, résultant d'une condensation à petite échelle, et une ouverture située sur le côté supérieur (I) pour l'évacuation de la vapeur d'eau après le traitement, dans le cas de disques en liège naturel pour la production de bouchons techniques et de champagne.

2. Procédé selon la revendication 1, **caractérisé en ce que** le dispositif de type A est utilisé dans les conditions de fonctionnement suivantes: capacité de cylindre: 8 kg de granulés avec le système arrêté, une température de 100°C à 125°C, une pression de manomètre de 0,2 à 0,8 bar, un temps de contact de 6 à 65 minutes et une vitesse de rotation dépendant du temps de contact.

3. Procédé selon la revendication 1, **caractérisé en ce que** le dispositif de type B est utilisé dans les conditions de fonctionnement suivantes : une capacité d'environ 10 000 disques de 26,5 mm de diamètre et 6,5 mm d'épaisseur et environ 2000 bouchons d'un calibre de 38 x 24 mm, une température de 100°C à 125°C, une pression de 0,2 à 0,8 bar, un temps de contact de 6 à 65 minutes et une vitesse de rotation de 1 à 10 tr/min.

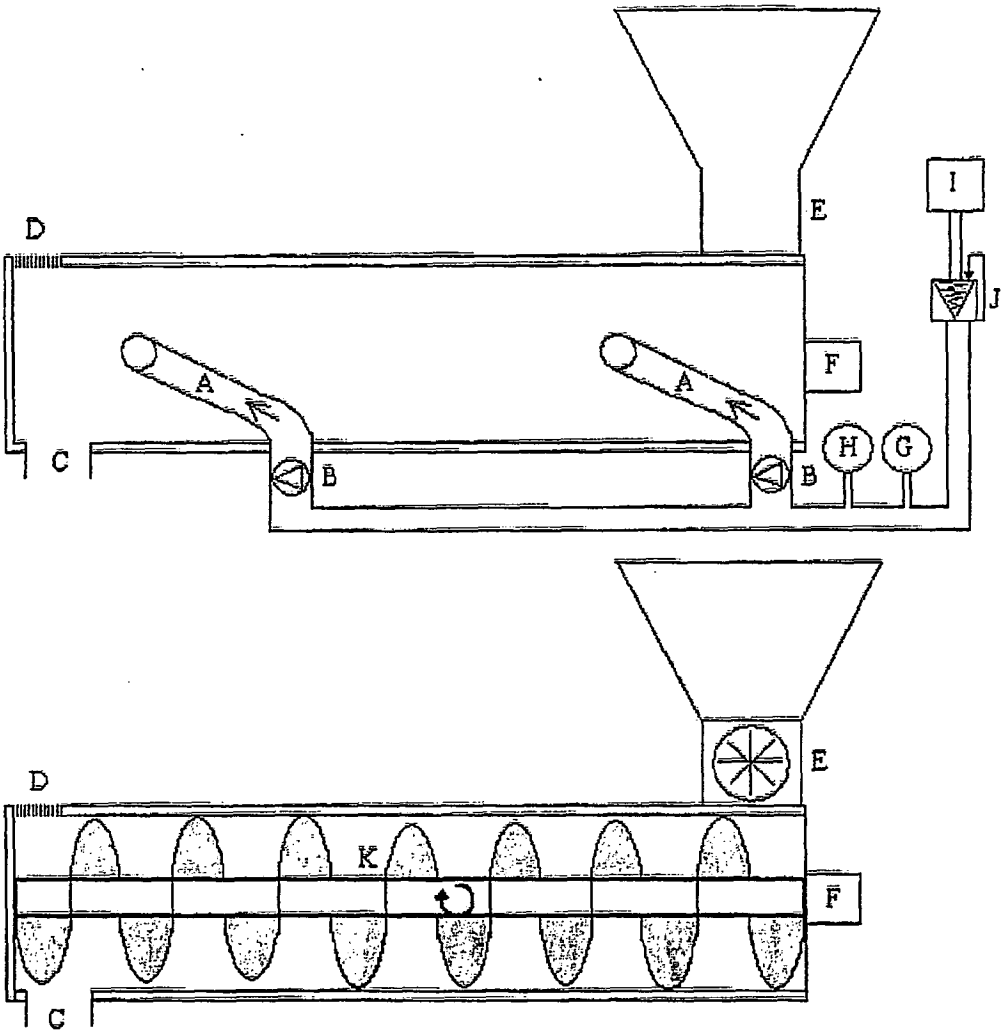


FIG. 1

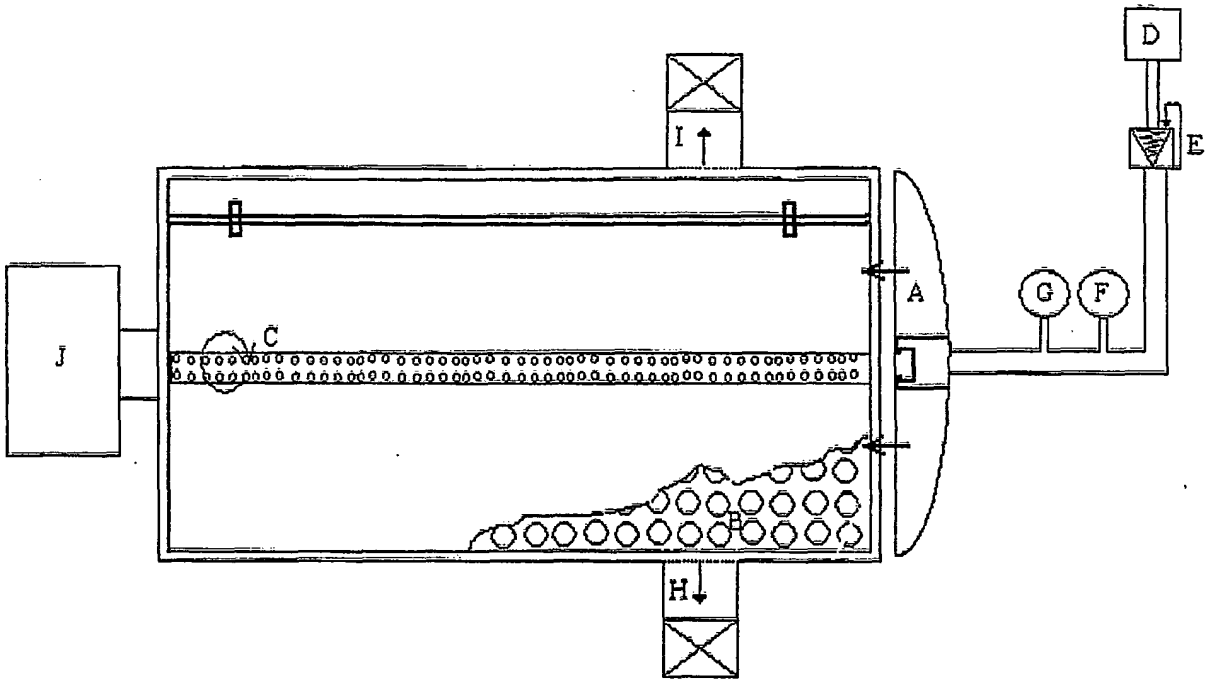


FIG. 2

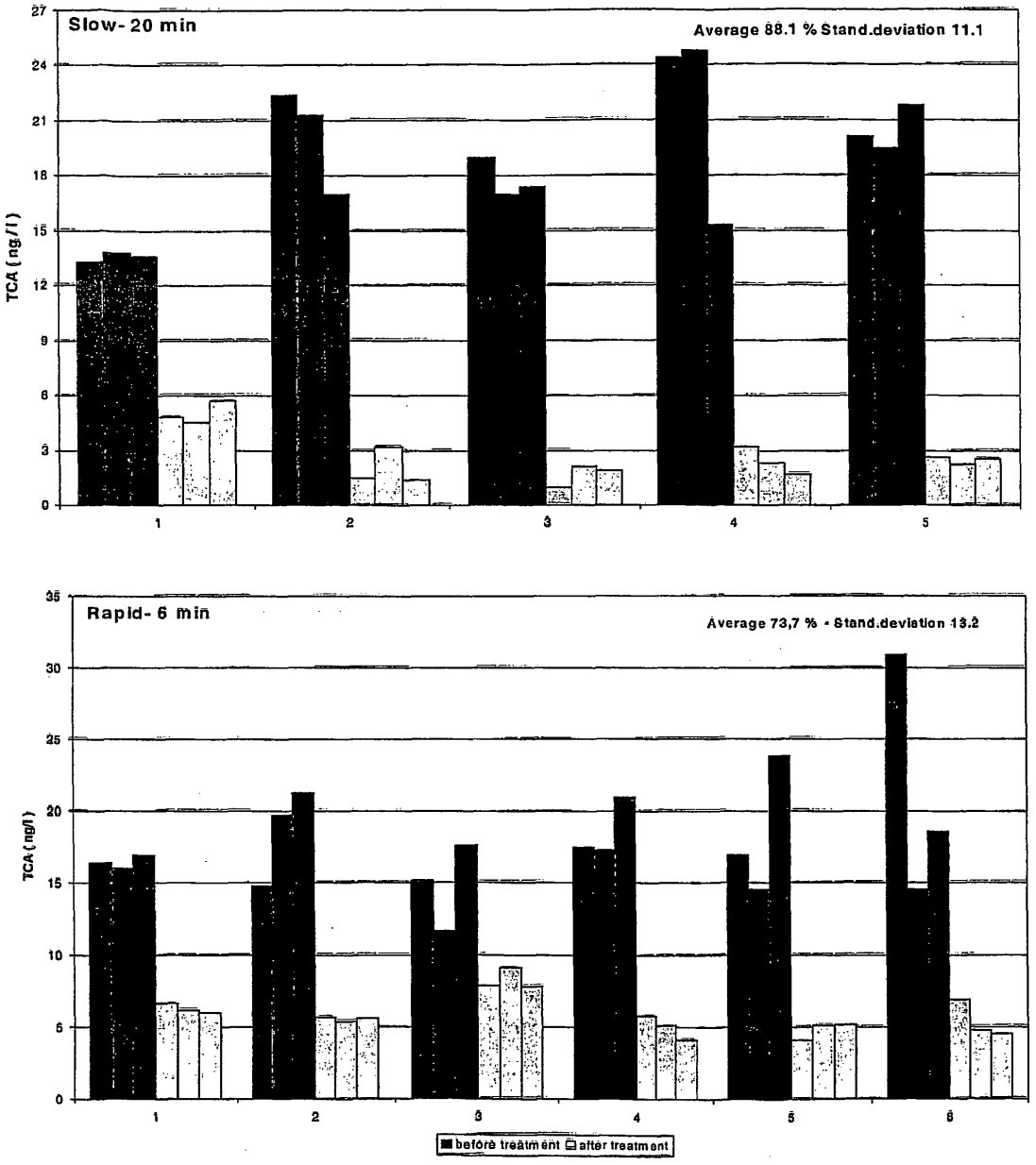

Slow- 20 min
Average 88.1 % Stand.deviation 11.1
TCA (ng/l)
0
3
6
9
12
15
18
21
24
27
1
2
3
4
5
Rapid- 6 min
Average 73,7 % - Stand.deviation 13.2
TCA (ng/l)
0
5
10
15
20
25
30
35
1
2
3
4
5
6
before treatment
after treatment

FIG. 3

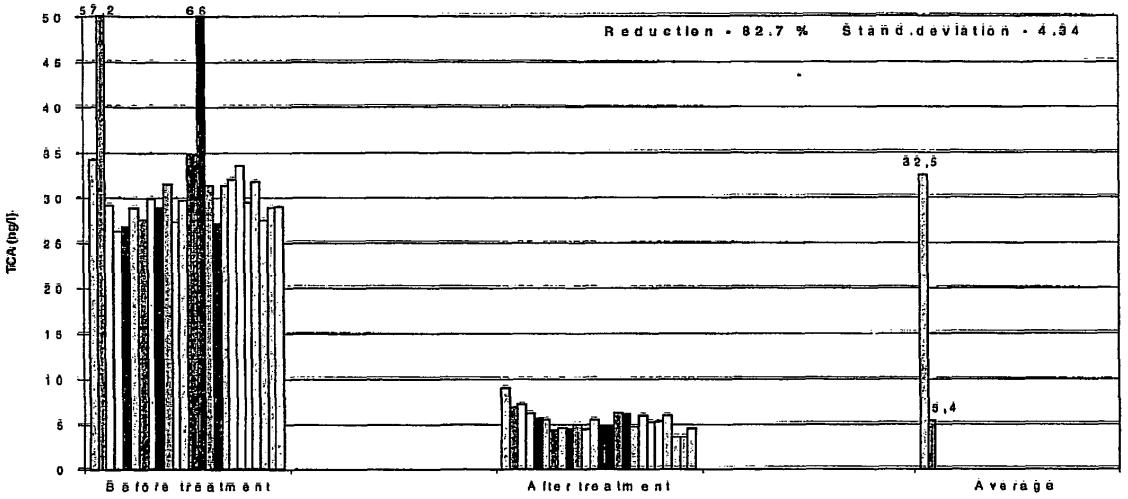

Reduction - 82.7 %
Stand.deviation - 4.34
TCA (ng/l)
50
45
40
35
30
25
20
15
10
5
0
57,2
66
32,5
5,4
Before treatment
After treatment
Average

FIG. 4

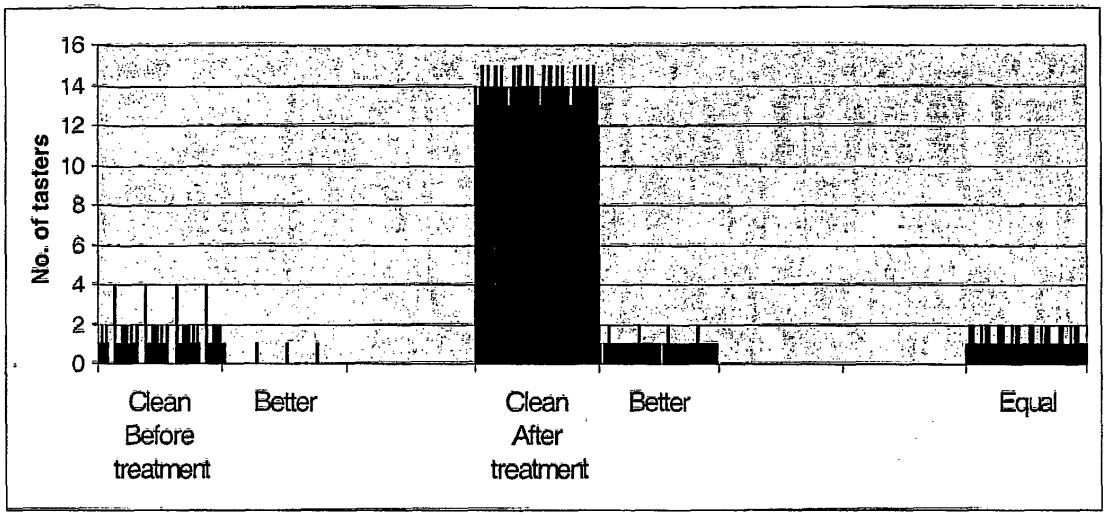

16
14
12
10
8
6
4
2
0
No. of tasters
Clean Before treatment
Better
Clean After treatment
Better
Equal

FIG. 5

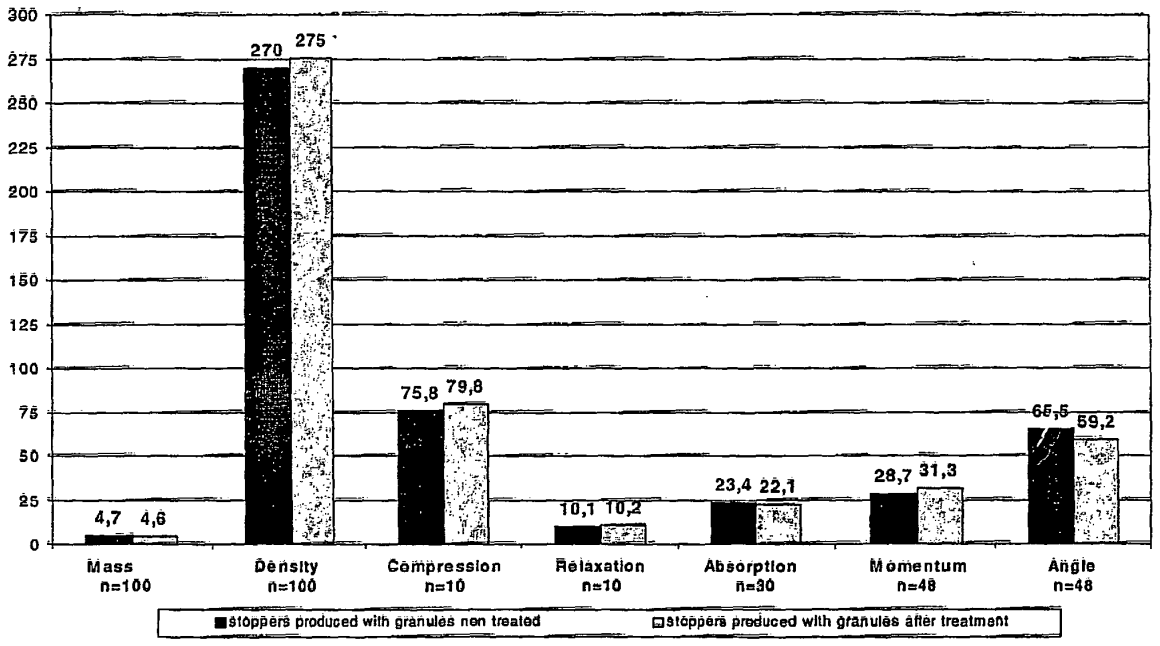
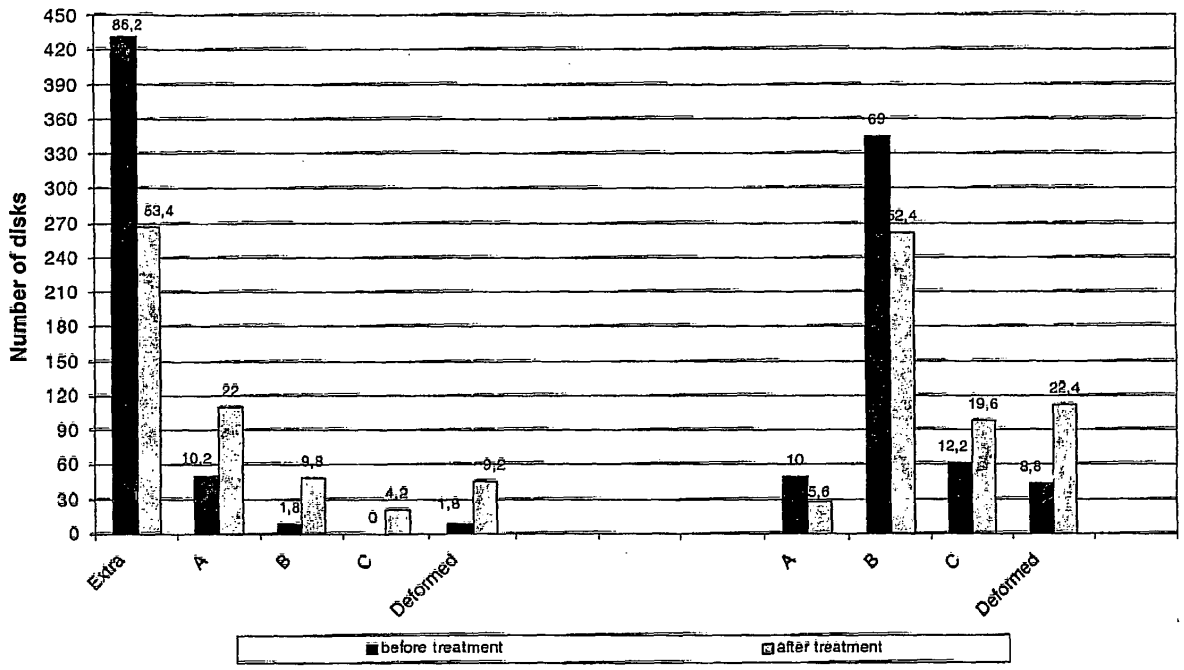

300
275
250
225
200
175
150
125
100
75
50
25
0
4,7
4,6
270
275
75,8
79,8
10,1
10,2
23,4
22,1
28,7
31,3
65,5
59,2
Mass n=100
Density n=100
Compression n=10
Relaxation n=10
Absorption n=30
Momentum n=48
Angle n=48
stoppers produced with granules non treated
stoppers produced with granules after treatment

FIG. 6



% Value on the top of column

Fig. 7

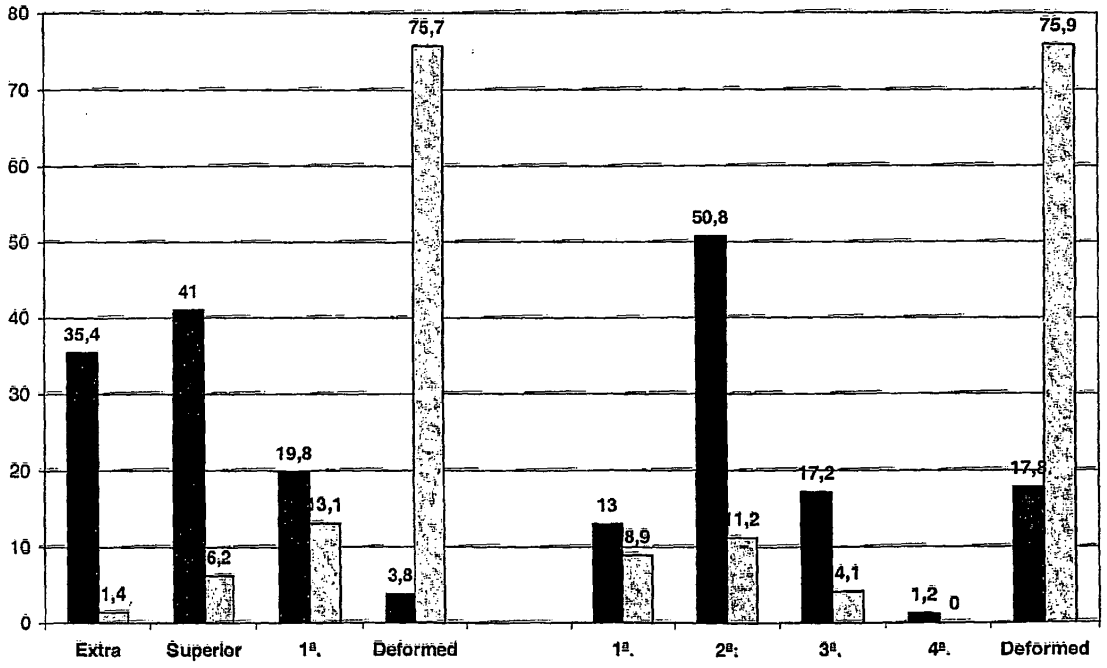

80
70
60
50
40
30
20
10
0
35,4
1,4
41
6,2
19,8
13,1
3,8
75,7
13
8,9
50,8
11,2
17,2
4,1
1,2
0
17,8
75,9
Extra
Superior
1ª.
Deformed
1ª.
2ª.
3ª.
4ª.
Deformed

FIG. 8

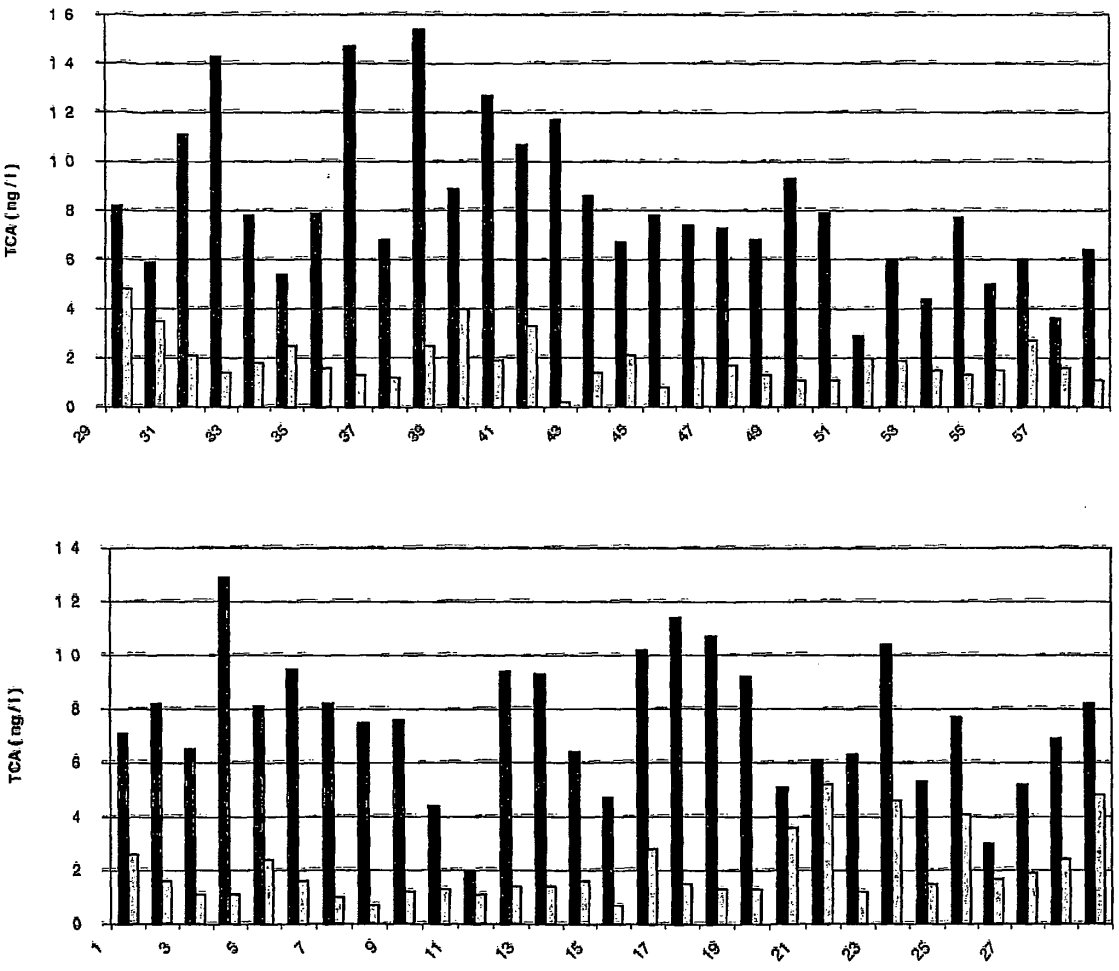

TCA (ng/l)
16
14
12
10
8
6
4
2
0
29
31
33
35
37
39
41
43
45
47
49
51
53
55
57
TCA (ng/l)
14
12
10
8
6
4
2
0
1
3
5
7
9
11
13
15
17
19
21
23
25
27

FIG. 9

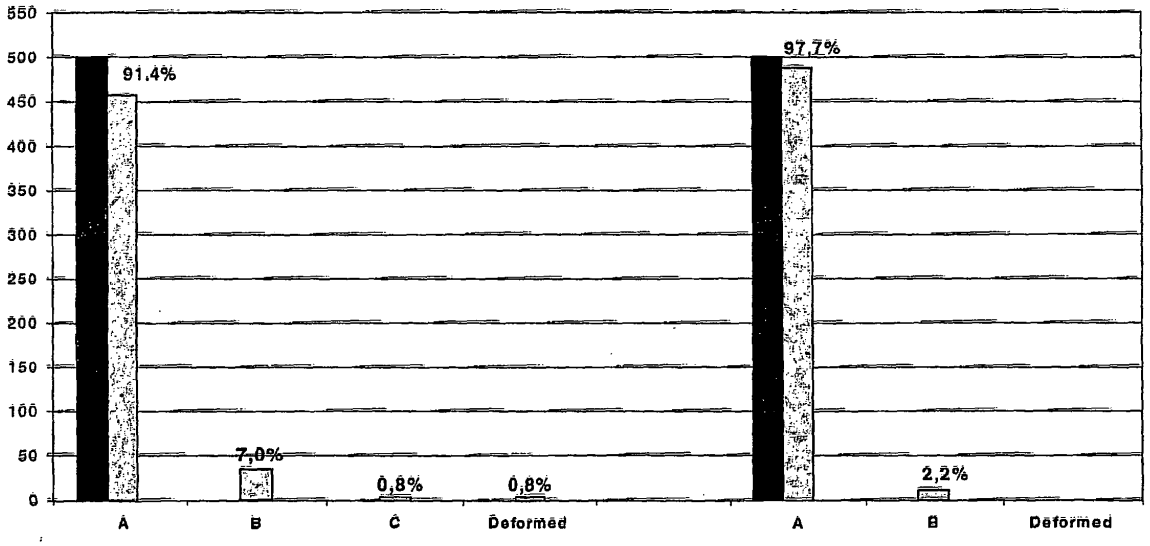

550
500
450
400
350
300
250
200
150
100
50
0
91,4%
7,0%
0,8%
0,8%
97,7%
2,2%
A
B
C
Deformed
A
B
Deformed

FIG. 10

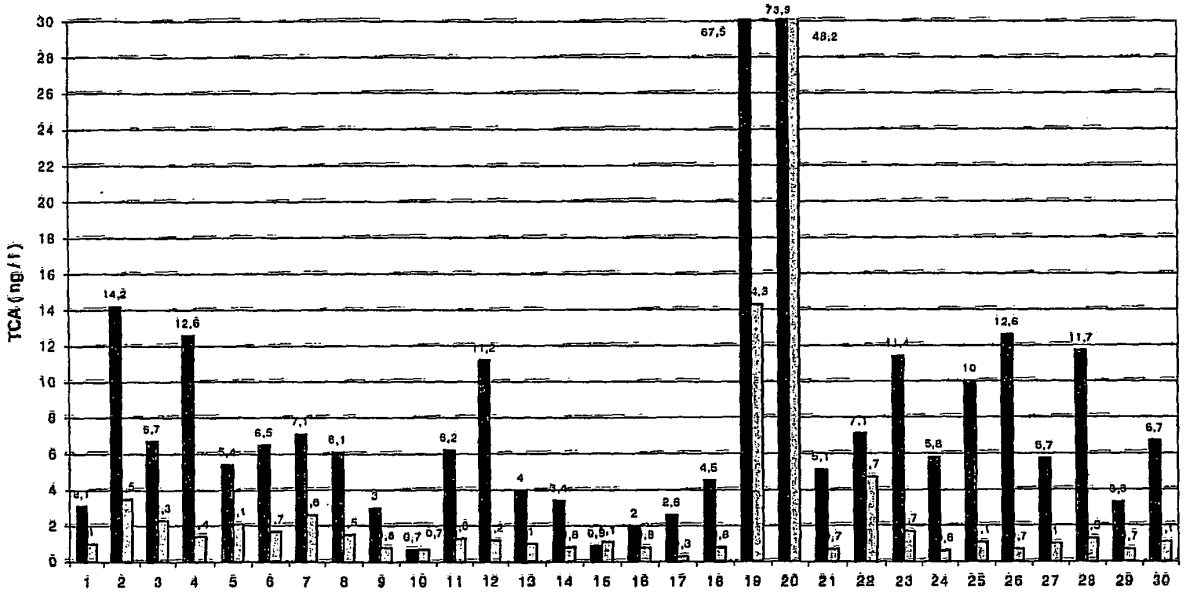

TCA (ng/l)
30
28
26
24
22
20
18
16
14
12
10
8
6
4
2
0
14,2
12,6
67,5
73,9
48,2
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30

FIG. 11

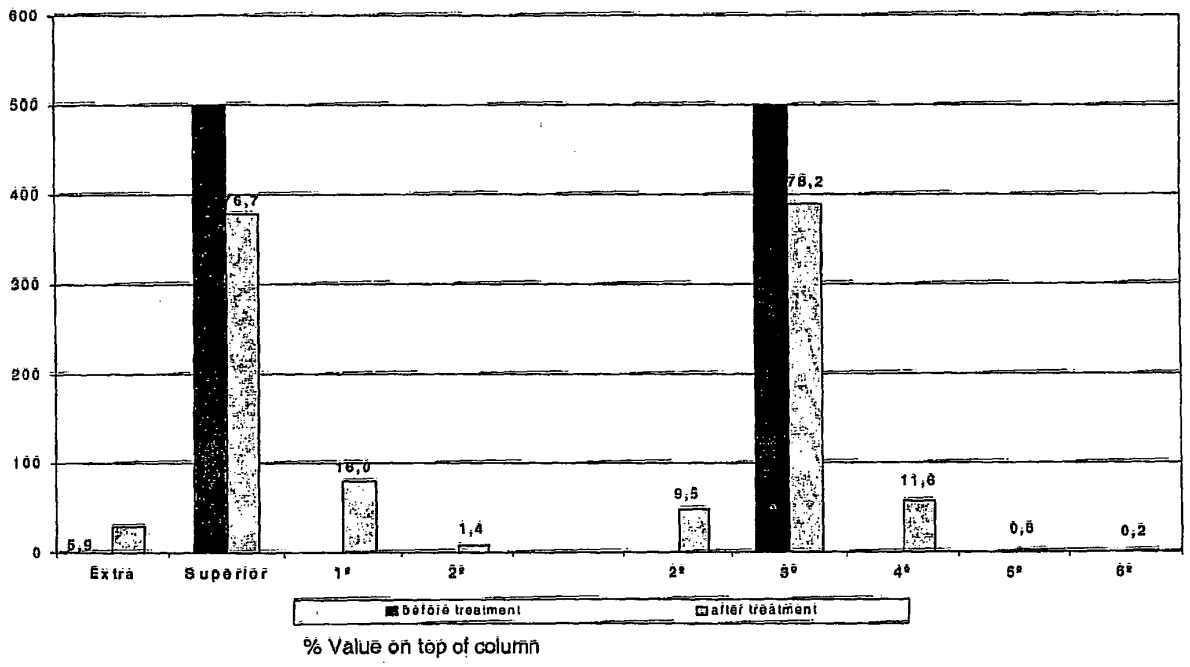

600
500
400
300
200
100
0
5,9
6,7
16,0
1,4
9,5
78,2
11,6
0,5
0,2
Extra
Superior
1#
2#
2#
3#
4#
5#
6#
before treatment
after treatment
% Value on top of column

FIG. 12