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(54) **Antimicrobial packaging based on the use of natural extracts and the process to obtain this packaging**

Antimikrobielle Verpackung auf Basis natürlicher Extrakte und Herstellungsverfahren für entsprechende Verpackung

Emballage antimicrobienne à base d'extraits naturels et procédé de fabrication de l'emballage

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Description**OBJECT OF THE INVENTION**

[0001] The object of the present invention corresponds to an antimicrobial packaging based on the use of natural extracts and also to the process followed to obtain this packaging. The packaging corresponding to the object of the invention is characterised in that of its antimicrobial, antifungal and also antioxidant properties.

[0002] To achieve the specified objectives, a vehicle is used, herein referred to as the active liquid, which is applied to a support made of plastic materials, paper, cardboard, aluminium etc., of a rigid or flexible format.

[0003] The present invention is characterised in that the active liquid can be applied to different packaging in the form of different types of coatings or as a spray.

[0004] The active liquid is comprised of resins, which are selected depending on the type of packaging, solvents that give the system fluidity, additives that confer it flexibility and, finally, of active components, which in this case are natural extracts with or without fixatives.

[0005] Therefore, the present invention can be included within the area of varnishes used to coat packaging used for the storage and conservation of products, mainly but not exclusively, to store food products, in order to improve the characteristics of the packaging and the packaged product.

BACKGROUND OF THE INVENTION

[0006] To date, active packaging is one of the most important technological innovations to preserve food products. This packaging is designed in order to provide the packed product with an additional protection, using the interaction between the food or product contained in the packaging to improve its preservation and acceptability.

[0007] The actions carried out to improve the quality of the products inside the packaging include:

- the addition of some compounds into the package, such as oxygen scavengers, moisture absorbers, ethylene absorbers and others.
- The addition of new components, dissolved or incorporated into the packaging material, such as antioxidants or others.

[0008] Examples are disclosed in FR 1 266 546, US 6 106 876 and WO 0 244 055.

[0009] These solutions, already known as protective measures and as means to improve the packaged products, present some drawbacks, such as involving the incorporation of foreign bodies inside the package, alteration of the organoleptic properties of the products by introducing foreign elements or irregular distribution of the protective properties.

[0010] This can even lead to a complete loss of the intended activity (loss of efficacy) when there is no direct contact between the product to be protected and the packaging, since protection is achieved by direct contact.

[0011] It is also a clear drawback to have to make a film in each case with the additional requirement of having to store films of different composition.

[0012] Until now, only one type of packaging which includes an antioxidant formulation has been described, also by the authors of this patent. The object of the invention can not under any circumstances be considered a later version of the same patent, since the objective of the first patent was only to prevent oxidation occurring in the packaging, without any antimicrobial or antifungal activity, and used different products to achieve this.

[0013] There are no known cases of packaging with antimicrobial and/or antifungal activity where the liquid used for these purposes is incorporated into the manufactured packaging, since in other solutions proposed for similar purposes the elements used to achieve these objectives require this to be incorporated in the formats during the polymerisation process and while obtaining the plastic material.

[0014] On the other hand, some processes achieve the same objectives but use chemical compounds obtained synthetically. This is a drawback from the consumer's perspective since these products must first be approved and present potential unknown risks for human health.

[0015] To achieve these properties, natural extracts or essential oils are used that form part of an active liquid which is applied as a coating to the packaging. In this way, active agents are immobilised on the packaging material and the system as a whole presents the antimicrobial or antifungal properties. To achieve this antimicrobial and antifungal action, direct contact with the food product to be protected is not required since the protection is given by controlled release of the antimicrobial agents in the active liquid used to manufacture the packaging.

[0016] The use of natural extracts has the advantage that all of these are classified as GRAS material (Generally Recognized as Safe) and are derived from plants used as condiments in food products.

[0017] Hence, another objective of the invention is to overcome previous drawbacks of:

- incorporating antimicrobial products during the manufacturing process of the packaging, with the incorporation of these to the final manufactured packaging format.
- the need for direct contact between the food products to be protected and the packaging with the protection formulation, since a vapour phase is produced responsible for protection of the food products.
- using chemical products of unique formulation obtained synthetically and with unknown health risks.

DESCRIPTION OF THE INVENTION

[0018] The antimicrobial packaging which corresponds to the object of the invention, basically consists in packaging of any material, format and arrangement, to which an active liquid has been applied which, among other elements, contains essential oils with an antimicrobial, antifungal and presumably also an antioxidant action.

[0019] The active liquid can be applied to the final format of the manufactured packaging before it is filled with the desired product, therefore it is not necessary to have a large store or active packaging of different characteristics for the different products to be packaged.

[0020] The active liquid is comprised of:

- a formulation made up of:
 - o a resin base such as nitrocellulose, acrylic, vinylic or others, chosen depending on the type of packaging to which the liquid is applied, to achieve good adhesion.
 - o Solvents which give the active liquid fluidity.
 - o Additives to give flexibility or other characteristics to the coating.
 - o Natural extracts based on essential oils that contain the active agents together with appropriate fixatives, which produce blocking or immobilization effects of the agents required for the desired antimicrobial and antifungal characteristics and permit controlled release of the active agents.

[0021] The natural extracts used, among others, can correspond to cinnamon, clove, ginger, rosemary, oregano, dill, basil etc. or other similar compounds derived from plants, either alone, or combined in appropriate proportions depending on the desired effect. The proportion of extracts ranges from 0.1 % to 10% in weight of the active liquid.

[0022] The natural extracts added to the base formulation can be in the form of an essence alone or combined with a fixative which retains the volatile elements of the natural extracts and which play the most active role in inhibiting growth of bacteria, yeasts and fungi.

[0023] The desired objective is complete inhibition of growth of bacteria, yeasts and fungi, producing their total elimination and preventing subsequent growth.

[0024] Depending on the type and the concentration of the extract used and whether or not it is used as a fixative, complete inhibition or at least a reduction in colony forming units (CFU) is achieved for some types of bacteria, fungi or yeasts.

[0025] Since certain food types favour the growth of certain bacteria and/or fungi, different types or combinations of natural extracts are required in each case, in order to achieve complete inhibition of all the possible bacteria, fungi and yeasts that can arise.

[0026] In some cases, the combination of natural extracts has been found to have a synergic effect on its inhibition properties, significantly improving the active efficiency.

[0027] The following tables are included in an attempt to demonstrate the effect of natural extracts added to a basic formulation applied to packaging, where the following abbreviations are used:

- CFUs per ml = colony forming units per ml
- Control = the standard microorganism culture produced in the Petri dish used in laboratory experiments.
- P0 = the basic formulation without any extract.
- PCA1 = the basic formulation to which cinnamon essence is added at 1% in weight of the active liquid.
- PCA4 = the basic formulation to which cinnamon essence is added at 4% in weight of the active liquid.
- PPeCA1 = the basic formulation to which cinnamon essence is added at 1 % together with a fixative.
- PPeCA4 = the basic formulation to which cinnamon essence is added at 4% together with a fixative.

• BACTERIA DIFFUSION ASSAYS (TABLE 1)

CFUs/ml	Control	P0	PCA1	PCA4	PPeCA1	PPeCA4
<i>S.aureus</i>	3.2E + 4	6.1 E+4	3.2E+4	3.4E+4	1.5E+4	NO
<i>L. monocitogene</i>	1.0E+5	2.6E+4	1.0E+5	1.0E+5	2.5E+4	9.1E+ 3
<i>E. faecalis</i>	3.4E + 4	2.9E+4	3.0E+4	3.0E+4	3.1 E+4	1.0E + 3
<i>B.cereus</i>	1.4E + 4	2.4E+4	1.8E+4	8.6E+4	8.6E+4	NO
<i>E. coli</i>	6.1 E+ 3	2.0E +3	1.9E+3	2.6E+3	7.0E+2	2.0E+1
<i>Y. enterocolitica</i>	1.5E+4	2.1 E+4	1.9E+4	1.7E+4	2.1 E+4	NO
<i>S. choleraesuis</i>	100	80	80	50	30	NO
<i>P. aeruginosa</i>	2.4E+4	2.3E+4	3.0E+4	2.1 E+4	1.9E+4	2.1 E+4

• FUNGI DIFFUSION ASSAYS (TABLE 2)

CFUs/ml	Control	P0	PCA1	PCA4	PPeCA1	PPeCA4
<i>C. albicans</i>	7.8 E+3	6.8E+3	6.6E+3	7.2 E+3	NO	NO
<i>A.flavus</i>	320	380	410	150	NO	NO

[0028] As can be observed in table 1, the number of colony forming units (CFUs/ml) of bacteria is zero, i.e. there are NO colony forming units for most of the bacteria and for any of the fungi and yeasts for a 4% extract of cinnamon essence with fixative.

[0029] Also, in table 2 we can observe that colonies of fungi are not formed with 1% extracts of cinnamon combined with fixative.

[0030] Logically, these experiments have been carried out with natural extracts such as cloves, basil, rosemary etc. producing in some cases a decline in the number of colony forming units (CFUs) and a radius of inhibition inside which no colony develops, while in other cases there is total inhibition, i.e. there is no growth of a specific bacteria, yeast or fungus.

[0031] The process to obtain the antimicrobial packaging has the following steps:

- Preparation of a basic formulation comprised of a resin substrate, some solvents and some additives to confer the final characteristics to the coating.
- Dispersion and dissolution of natural extracts in the basic formulation that contain antimicrobial and fungicidal active agents. The solution or dispersion is made using Cowles type rotor stator assemblies; wet mills or other similar devices.
- Incorporation of the active liquid to the chosen package or support.
- Drying of the packaging, either in the open air or with forced air.

[0032] Incorporation of the active liquid to the package can be done:

- by impregnating the support in the active liquid
- by coating the different forms of the support
- by spraying the active liquid on the support

[0033] The packaging to which the liquid is applied can be made of plastic, paper, cardboard, tinfoil, steel, aluminium or glass, either of one material or in several layers. Materials used can be virgin or recycled.

[0034] The supports to which the active liquid can be applied are:

- Polypropylene and all its forms
- Polyethylene and all its forms
- Polyester (PET, PETG)
- PVDC
- Polystyrene and all its forms
- PVC

- Polyamide
- Polycarbonate
- Cellulotic films (cellophane) and all its forms
- KRAFT
- 5 - Paper and all its forms
- Aluminium
- Tinfoil
- Steel
- Glass

10 **[0035]** And all the complexes that can be formed from these materials (by adhesion, lamination or extrusion).

DESCRIPTION OF THE DRAWINGS

15 **[0036]** As a complement to the present descriptive report and to make it easier to understand, this is accompanied by a set of plans. The most significant aspects of the invention are represented in the figures of these plans, these must be considered as illustrative and in no way limit the scope of the invention.

[0037] Figure 1. This shows a schematic representation of the steps of the manufacturing process of the antimicrobial packaging based on the essence of the object of the invention.

20 **[0038]** Figure 2. This shows a schematic representation of the materials used to manufacture the active liquid applied to the final packaging.

PREFERRED EMBODIMENT OF THE INVENTION

25 **[0039]** In view of the figures, a preferable application of the proposed invention is described.

[0040] Figure 1 represents the steps required for the process to manufacture the antimicrobial packaging which constitutes the object of the invention and consists of the following steps:

- Preparation (1) of a basic formulation, which is comprised of a resin substrate, some solvents and some additives which confer the coating with its final characteristics.
- 30 - Dispersion and dissolution (2) of natural extracts that contain the active antimicrobial and antifungal agents in the basic formulation. The dissolution or dispersion is carried out using stirrer systems such as "Cowles" type rotor stator assembly, wet mills or by other similar methods.
- Incorporation (3) of the active liquid to the chosen packaging or support.
- 35 - Drying (4) of the packaging, either in the open air or by forced air.

[0041] The active liquid can be incorporated to the packaging by:

- Impregnation (3.1) of the packaging by the active liquid in spray.
- 40 - Coating (3.2) of the different packaging formats.
- By spraying (3.3) the packaging with the active liquid.

[0042] Figure 2 shows that the active liquid (10) is comprised of:

- 45 - a basic formulation (5) comprised of
 - o A resin substrate (6) made of nitrocellulose, acrylics, vinylic or other compounds the selection of which is based on the type of support to which they are to be applied, in order to achieve good adhesion.
 - o Solvents (7) which confer fluidity to the active liquid.
 - 50 o Additives (8) to give flexibility or other characteristics to the coating.
- Natural extracts (9) based on essential oils that contain active agents which produce a blocking effect or immobilisation of the active agents that confer the desired antibacterial and fungicidal characteristics.

55 **[0043]** This description is sufficient for any expert in the area to understand the scope of the invention and the benefits that can be derived from it.

[0044] The materials and size, shape and arrangement of the components may vary provided that the essence of the invention remains the same.

[0045] The terms used in this descriptive report should always be interpreted in their broadest sense and not in a restrictive way.

5 Claims

1. Antimicrobial packaging based on the use of natural extracts that consists of packaging to which a coating or an active liquid is applied composed of a basic formulation that consists in:

- 10 - a resin substrate made of nitrocellulose, acrylics, vinyls etc.
- Solvents to confer the liquid fluidity.
- Additives to confer flexibility or other characteristics to the coating

15 **characterised in that** in the basic formulation, natural extracts based on essential oils of plants that contain anti-bacterial, antifungal and antioxidant active agents are dissolved or dispersed in a final proportion of natural extracts varies between 0.1% and 10% in weight of the final active liquid and wherein the natural extracts are based on volatile elements.

20 2. Antimicrobial packaging, according to claim 1, **characterised in that** the natural extracts can be selected among others from: cinnamon, clove, ginger, rosemary, oregano, dill, basil among others, either alone or as mixtures in appropriate combinations according to the type of products to be preserved, since the type of product stored determines the type of bacteria, yeasts or fungi that can develop.

25 3. Antimicrobial packaging, according to any of the previous claims **characterised in that** the packaging material is made from plastics, metals, paper, cardboard, tinfoil, steel, aluminium, steel, alone or in multiple layers, either virgin or recycled.

30 4. Antimicrobial packaging, according to claim 2, **characterised in that** more bactericidal agents are applied to the natural extracts in the form of essence or with a fixative that retains the volatile compounds.

35 5. Process to obtain an antimicrobial packaging based on the use of essential oils, according to previous claims, **characterised in that** the process consists of the following steps: - Preparation of a basic formulation, comprised of a resin substrate, some solvents and some additives to confer the final characteristics to the coating. - Dispersion and dissolution in the basic formulation of natural extracts that contain antimicrobial and fungicidal active agents. Incorporation of the active liquid to the chosen packaging or support. - Drying of the packaging.

6. Process to obtain antimicrobial packaging according to claim 6, **characterised in that** the dissolution or dispersion is done using stirrers of the Cowles rotor stator assembly type, wet mills or other similar methods.

40 7. Process to obtain an antimicrobial package according to claim 6 **characterised in that** the active liquid can be incorporated to the packaging: - by impregnating the package with active liquid - by coating the different forms of packaging. - by spraying the active liquid on the packaging.

45 8. Process to obtain antimicrobial packaging according to claim 6 **characterised in that** the packaging is dried in open air or by forced air.

Patentansprüche

50 1. Antimikrobielle Verpackung auf der Grundlage der Verwendung natürlicher Extrakte, bestehend aus einer Verpackung, auf die eine Beschichtung oder eine aktive Flüssigkeit aufgebracht wird, die aus einer Grundrezeptur besteht, enthaltend:

- 55 - ein aus Nitrozellulose, Acryl, Vinyl usw. hergestelltes Harzsubstrat,
- Lösungsmittel, um der Flüssigkeit Fluidität zu verleihen,
- Zusätze, um der Beschichtung Flexibilität oder weitere Kennwerte zu verleihen,

dadurch gekennzeichnet, dass in der Grundrezeptur natürliche Extrakte auf der Grundlage ätherischer Öle von

Pflanzen, die aktive antibakterielle, fungizide und oxidierungshemmende Wirkstoffe enthalten, mit einem Endgehalt an natürlichen Extrakten, der zwischen 0,1 und 10 Gew.-% der fertigen aktiven Flüssigkeit variiert, gelöst oder dispergiert sind und die natürlichen Extrakte auf flüchtigen Bestandteilen basieren.

- 5 2. Antimikrobielle Verpackung nach Anspruch 1, **dadurch gekennzeichnet, dass** die natürlichen Extrakte unter anderen ausgewählt sein können aus: Zimt, Gewürznelke, Ingwer, Rosmarin, Origanum, Dill, Basilikum, entweder einzeln oder als Mischungen in geeigneten Kombinationen entsprechend der Art der zu konservierenden Produkte, da die Art des gelagerten Produkts die Art von Bakterien, Hefen und Pilzen bestimmt, die sich entwickeln können.
- 10 3. Antimikrobielle Verpackung nach einem der vorhergehenden Ansprüche, **dadurch gekennzeichnet, dass** das Verpackungsmaterial aus Kunststoffen, Metallen, Papier, Pappe, metallisiertem Papier, Stahl, Aluminium, Stahl, ein- oder mehrschichtig, neu oder recycelt hergestellt ist.
- 15 4. Antimikrobielle Verpackung nach Anspruch 2, **dadurch gekennzeichnet, dass** weitere bakterizide Wirkstoffe zusätzlich zu den natürlichen Extrakten in Form von Essenzen oder zusammen mit einem Fixierungsmittel angewendet werden, das die flüchtigen Komponenten zurückhält.
- 20 5. Verfahren zum Erhalt einer antimikrobiellen Verpackung auf der Grundlage der Verwendung ätherischer Öle gemäß den vorhergehenden Ansprüchen, **dadurch gekennzeichnet, dass** das Verfahren folgende Schritte umfasst:
 - Zubereiten einer Grundrezeptur enthaltend ein Harzsubstrat, Lösungsmittel und Zusatzstoffe, um der Beschichtung die End Eigenschaften zu verleihen,
 - Dispergieren und Auflösen der natürlichen Extrakte, die antimikrobielle und fungizide Wirkstoffe enthalten, in der Grundrezeptur,
 - 25 - Einbringen der aktiven Flüssigkeit in die gewählte Verpackung oder Unterlage,
 - Trocknen der Verpackung.
- 30 6. Verfahren zum Erhalt einer antimikrobiellen Verpackung nach Anspruch 6, **dadurch gekennzeichnet, dass** das Auflösen oder Dispergieren unter Verwendung von Rührwerken des Typs Cowles mit Rotor-Statoranordnungen, Nassmühlen oder nach ähnlichen Verfahren erfolgt.
- 35 7. Verfahren zum Erhalt einer antimikrobiellen Verpackung nach Anspruch 6, **dadurch gekennzeichnet, dass** die aktive Flüssigkeit in die Verpackung eingebracht werden kann durch:
 - Imprägnieren der Verpackung mit der aktiven Flüssigkeit,
 - Beschichtung der unterschiedlichen Formen der Verpackung,
 - Aufsprühen der aktiven Flüssigkeit auf die Verpackung.
- 40 8. Verfahren zum Erhalt einer antimikrobiellen Verpackung nach Anspruch 6, **dadurch gekennzeichnet, dass** die Verpackung an freier Luft oder durch Zwangsbelüftung getrocknet wird.

Revendications

- 45 1. Conditionnement antimicrobien basé sur l'utilisation d'extraits naturels consistant en un conditionnement auquel on applique un revêtement ou un liquide actif composé d'une formulation de base consistant en :
 - un substrat de résine fait à partir de nitrocellulose, acryliques, vinyliques, etc.
 - des solvants pour conférer la fluidité du liquide
 - 50 - des additifs pour conférer de la flexibilité ou d'autres caractéristiques au revêtement,

caractérisé en ce que dans la formulation de base, les extraits naturels basés sur des huiles essentielles de plantes contenant des agents antibactériens, antifongiques et antioxydants sont dissous ou dispersés dans une proportion finale d'extraits naturels variant entre 0,1% et 10% en poids du liquide actif final et dans lequel les extraits naturels

- 55 2. Conditionnement antimicrobien selon la revendication 1, **caractérisé en ce que** les extraits naturels peuvent être choisis parmi : cinnamome, clou de girofle, gingembre, romarin, origan, aneth, basilic, entre autres, soit seuls ou

mélangés dans des combinaisons appropriées selon le type de produits à conserver, car le type de produit stocké détermine le type de bactéries, levures ou champignons pouvant se développer.

3. Conditionnement antimicrobien selon l'une quelconque des revendications précédentes, **caractérisé en ce que** la matière de conditionnement est réalisée en plastique, métal, papier, carton, feuille d'étain, acier, aluminium, acier, seuls ou en de multiples couches, soit vierges soit recyclés.
4. Conditionnement antimicrobien selon la revendication 2, **caractérisé en ce que** l'on applique plusieurs agents bactéricides aux extraits naturels sous forme d'essence ou avec un fixateur qui retient les composés volatils.
5. Procédé d'obtention d'un conditionnement antimicrobien basé sur l'utilisation d'huiles essentielles, selon les revendications précédentes, **caractérisé en ce que** le procédé consiste aux étapes suivantes : - Préparation d'une formulation de base, comprenant un substrat de résine, des solvants et des additifs pour conférer les caractéristiques finales au revêtement. - Dispersion et dissolution dans la formulation de base des extraits naturels contenant des agents antimicrobiens et fongicides actifs. - Incorporation du liquide actif au conditionnement ou support choisi. - Séchage du conditionnement.
6. Procédé d'obtention de conditionnement antimicrobien selon la revendication 6, **caractérisé en ce que** la dissolution ou dispersion est faite en utilisant des dispositifs d'agitation du type rotor-stator de Cowles, moulins humides ou d'autres méthodes similaires.
7. Procédé d'obtention d'un conditionnement antimicrobien selon la revendication 6 **caractérisé en ce que** le liquide actif peut être incorporé au conditionnement : - en imprégnant le conditionnement avec le liquide actif, - en revêtant les différentes formes de conditionnement, - en pulvérisant le liquide actif sur le conditionnement.
8. Procédé d'obtention d'un conditionnement antimicrobien selon la revendication 6, **caractérisé en ce que** le conditionnement est séché au plein air ou par ventilation forcée.

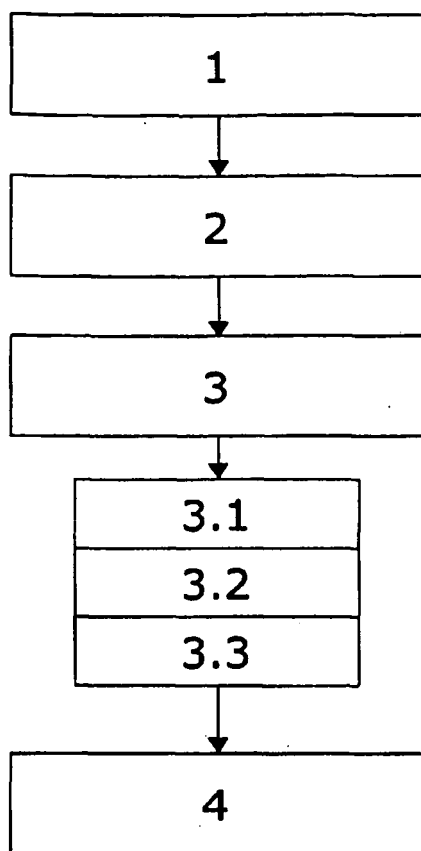


FIG. 1

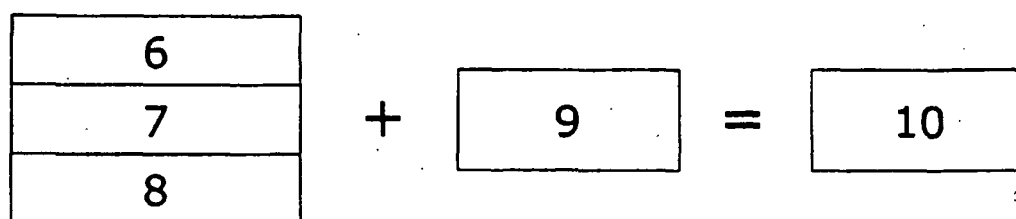


FIG. 2

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REFERENCES CITED IN THE DESCRIPTION

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