FORM 1 621869

REGULATION 9

COMMONWEALTH OF AUSTRALIA

PATENTS ACT 1952

APPLICATION FOR A STANDARD PATENT

We, SOCIETE DES PRODUITS NESTLE S.A., a Swiss body corporate of Vevey, Switzerland, hereby apply for the grant of a Standard Patent for an invention entitled:-

"PROTECTION OF A FOOD AGAINST OXIDATION"

which is described in the accompanying Complete Specification.

Details of basic application:-

Number: 1715/88-8

Country: Switzerland

Date: 6th May, 1988

Our address for service is: SHELSTON WATERS
55 Clarence Street
SYDNEY, N.S.W. 2000.

DATED this 18th Day of April, 1989
SOCIETE DES PRODUITS NESTLE S.A.

by

Fellow Institute of Patent Attorneys of Australia
of SHELSTON WATERS

S006844 18/04/89

To: The Commissioner of Patents
WODEN A.C.T. 2606

File: D.B. S-86

Fee: $140.00

# CONVENTION APPLICATION BY A COMPANY

FORM 8 - REGULATION 12 (2)

NO 2438/02

AUSTRALIA
PATENTS ACT 1952

62 1069

## DECLARATION IN SUPPORT OF A CONVENTION APPLICATION FOR A PATENT

In support of the Convention Application made by

(a) Here Insert (in full) Name of Company.

(a) SOCIETE DES PRODUITS NESTLE S.A.

(hereinafter referred to as "Applicant") for a patent for an invention entitled:

(b) Here Insert Title of Invention.

(b) "Protection of a food against oxidation"

(c) and (d) Here Insert Full Name and Address of Company Official authorised to make declaration.

I, (c) Andrzej LEDZION

of (d) En la Priauraz, 1807 Blonay, Switzerland

do solemnly and sincerely declare as follows:

1. I am authorised by Applicant to make this declaration on its behalf.

2. The basic Application(s) as defined by section 141 of the Act was / were made

(e) Here insert Basic Country followed by date of Basic Application.

(f) Here insert Full Name(s) of Applicant(s) in Basic Country.

in (e) Switzerland on the 6th day of May 1988

by (f) Société des Produits Nestlé S.A.

in ..... on the ..... day of ..... 19

by .....

in ..... on the ..... day of ..... 19

by .....

in ..... on the ..... day of ..... 19

by .....

(g) Here Insert (in full) Name and Address of actual Inventor or Inventors.

3. (g) Josef BURRI, of ch. de la Pierraz 25, 1066 EPALINGES, Switzerland / Manfred Paul GRAF, of ch. de la Baye 12, 1807 BLONAY, Switzerland

Pierre LAMBELET, of Tour Ronde 7, 1806 SAINT LEGIER, Switzerland / Jurg LOELIGER, of ch. de Pierre à Fleur 6, 1802 CORSEAUX, Switzerland is/are

the actual Inventor(s) of the invention and the facts upon which Applicant is entitled to make the Application are as follows:

See reverse side of this form for guidance in completing this part.

the applicant is the assignee of the actual inventors

4. The basic Application(s) referred to in paragraph 2 of this Declaration was/were the first Application(s) made in a Convention country in respect of the invention, the subject of the Application.

DECLARED at Vevey, Switzerland

this 11 APR 1989 day of ..... 19

(h) Personal Signature of Declarant (c) (no seal, witness or legalisation).

(h)

(12) PATENT ABRIDGMENT (11) Document No. AU-B-33117/89
(19) AUSTRALIAN PATENT OFFICE (10) Acceptance No. 621869

(54) Title
**PROTECTION OF A FOOD AGAINST OXIDATION**

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(21) Application No. : **33117/89** (22) Application Date : **18.04.89**

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(71) Applicant(s)
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(74) Attorney or Agent
**SHELSTON WATERS , 55 Clarence Street, SYDNEY NSW 2000**

(56) Prior Art Documents
**US 3686001**

(57) Claim

1. The use of vanillin for the protection of a food against oxidation by addition of 5 to 5000 µg vanillin per g food dry matter.

4. The use in accordance with claim 1, substantially as herein described with reference to any one of the examples.

COMMONWEALTH OF AUSTRALIA 21803

FORM 10

PATENTS ACT 1952

COMPLETE SPECIFICATION

FOR OFFICE USE:

Class Int.Class

Application Number:
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Complete Specification Lodged:
Accepted:
Published:

Priority:

Related Art:

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Name of Applicant: SOCIETE DES PRODUITS NESTLE S.A.

Address of Applicant: Vevey, Switzerland

Actual Inventor: Josef Burri, Manfred Paul Graf, Pierre Lambelet and Jurg Loeliger

Address for Service: SHELSTON WATERS, 55 Clarence Street, Sydney

Complete Specification for the Invention entitled:

"PROTECTION OF A FOOD AGAINST OXIDATION"

The following statement is a full description of this invention, including the best method of performing it known to us:-

This invention relates to a process for the protection of a food against oxidation and to the use of vanillin for this purpose.

Vanillin is known for its strong flavouring power. It is thus used in known manner as an agent for flavouring foods, such as creams, ice creams or chocolates for example.

It is also known that certain fat-soluble derivatives of vanillin, particularly certain vanillyl acylamides, may be used for the protection of foods against oxidation. However, it has never been proposed to use vanillin as such for the protection of foods against oxidation. This is because vanillin shows hardly any solubility in fats.

The object of the present invention is to provide a process for the effective protection of a food using a substance generally accepted as innocuous to the human organism.

The present invention relates to the use of vanillin for the protection of a food against oxidation in a quantity of 5 to 5000 µg vanillin per g food dry matter.

It has surprisingly been found that vanillin lends itself as such to use as an antioxidant in foods and that it provides as such for the effective protection of foods against oxidation in the concentrations indicated.

If less than 5 µg vanillin is added per g food dry

matter, the food is in danger of being inadequately protected against oxidation. If more than 5000 µg is added, the protection afforded may be no greater than that obtained by adding quantities within the indicated range.

In the context of the present invention, the term vanillin is understood to encompass each of the substances of identical or similar formulae which are known for their flavouring power characteristic of vanilla, more especially the active principle of an extract of vanilla planifolia Andr. (Orchidacea), namely 3-methoxy-4-hydroxybenzaldehyde, the compound synthetized from eugenol or lignin which has the same structure, or 3-ethoxy-4-hydroxybenzaldehyde.

Accordingly, to carry out the process according to the invention or for the use according to the invention, it is possible to use a natural extract containing vanillin, a purified natural vanillin or a synthetic vanillin. It is preferred to use purified vanillin, for example in the form of a fine powder or alcoholic solution.

The vanillin may be added to the food by thorough mixing with the food during or after its production. This presupposes that the food or its constituents are preferably in subdivided, dry, pasty or liquid form during or after production.

In view of the volatility of vanillin, its thorough mixing with the food is preferably carried out at a relatively moderate temperature with no excessive heat treatment thereafter.

Thus, the thorough mixing step is preferably carried out by dry mixing, after drying, in the production of dehydrated flakes, for example, by the cylinder drying of an aqueous suspension of food material.

The fact that vanillin effectively affords protection of a food against oxidation by simple final dry addition in powder form is one of the most surprising aspects of the present invention.

The following Examples, in which the percentages are by weight, are intended to illustrate the process and the use according to the invention.

Example 1

To prepare dehydrated precooked cereal flakes, grains of whole wheat are ground the day before in a hammer mill. Four batches of an aqueous dispersion of 5 kg wheat flour in 20 kg water are prepared.

Each batch is heat-treated for 60 s at 130 °C in a scraped-surface heat exchanger and is then immediately dried for 10 s on a two-cylinder dryer heated with steam at 4 bar to a residual moisture content of 3.4-3.8%. Four batches of cereal flakes are thus obtained.

One batch is kept without vanillin for comparison. 100 µg vanillin powder per g flakes are added dry with thorough stirring to the second batch, 200 µg vanillin powder per g flakes to the third batch and 500 µg vanillin powder per g flakes to the fourth batch.

The four batches are subjected to the following oxidation test:

Oxidation test

The cereal flakes are placed in 400 ml lacquered tin-plate cans in an amount of 40 g flakes per batch per can. The cans are then hermetically sealed and stored at 30°C.

The gases in the head space of the cans containing the sample flakes of the four batches are analyzed after 1, 2 and 3 months by determination of the pentane content, pentane being a by-product of the degradation of lineoleic acid by oxidation, and the residual oxygen content. The pentane content (in M pentane in 5 ml head space gas at 1013 mbar) is determined by gas-phase chromatography and the residual oxygen content (in % head space gas) by measurement of the paramagnetic suscept-

ibility.

The results of this test are shown in Table I below:

TABLE I

| Samples | Storage period: 1 month pentane ($10^{-9}$M) | 1 month $O_2$ (%) | 2 months pentane ($10^{-9}$ M) | 2 months $O_2$ (%) | 3 months pentane ($10^{-9}$M) | 3 months $O_2$ (%) |
|---|---|---|---|---|---|---|
| Without vanillin (comparison) | 4.3 | 18.8 | 9.1 | 17.6 | 13 | 16.4 |
| With 100 µg/g vanillin | 4.2 | 18.4 | 7.5 | 17.4 | 8 | 17.3 |
| With 200 µg/g vanillin | 3.2 | 18.5 | 4.8 | 17.6 | 6 | 17.3 |
| with 500 µg/g | 1.0 | 18.8 | 1.4 | 18.2 | 1.6 | 18 |

It can be seen that, after storage for only one month at 20°C, there is already a difference between the comparison sample and the samples containing vanillin, the difference being more pronounced, thelarger the quantity of vanillin added. After 2 and 3 months, the reduction in the production of pentane and in the consumption of oxygen as a result of the addition of vanillin, i.e. the reduction in oxidation, is very clear.

Example 2

Four batches of cereal flakes are prepared from whole rice in the same way as described in Example 1. The flakes thus produced have a residual moisture content of 3.9%.

One batch is kept without vanillin for comparison. Respective quantities of 50 µg, 500 µg and 5000 µg vanillin per g flakes of the second, third and fourth batches are added dry with thorough stirring. The four batches are oxidation-tested at 37°C in the same way as described in

Example 1 and are then tasted by specialists in a test based on the odour emitted by the samples. The scale of marks awarded extends from 1 to 9, 1 representing a very heavily oxidized product, 9 an excellent, fresh product and 5 the limit of acceptability.

The results of these tests are shown in Table II below:

TABLE II

Storage period

| Samples | 3 months | | | 4 months | | | 6 months | | |
|---|---|---|---|---|---|---|---|---|---|
| | pentane ($10^{-9}$M) | $O_2$ (%) | taste | pentane ($10^{-9}$M) | $O_2$ (%) | taste | pentane ($10^{-9}$M) | $O_2$ (%) | taste |
| Without vanillin (comparison) | 2.9 | 20.0 | 6.3 | 6.3 | 19.8 | 6 | | | 5.3 |
| With 50 µg/g vanillin | 1.7 | 20.5 | 7.7 | 1.7 | 20.2 | 7.5 | 3.1 | 19.5 | 7.3 |
| With 500 µg/g vanillin | 0.0 | 20.3 | 6.7 | 0.7 | 19.9 | 5 | 1.0 | 19.7 | 6.3 |
| With 5000 µg/g vanillin | 0.5 | 20.3 | 6.0 | 4.3 | 20.1 | 5.3 | 0.9 | 19.8 | 6.0 |

It can be seen that all the results of the oxidation tests are conclusive while the tasting tests give corresponding results in the majority of cases.

Example 3

Four batches of cereal flakes are prepared from whole oats in the same way as described in Example 1. The flakes thus prepared have a residual moisture content of 1.7%.

One batch is kept without vanillin for comparison. Respective quantities of 50 µg, 500 µg and 5000 µg of vanillin per g flakes of the second, third and fourth batches are added dry with thorough stirring.

The four batches are oxidation-tested at 37°C in the same way as described in Example 1 and then tested for taste in the same way as described in Example 2.

The results of these tests are shown in Table III below.

TABLE III

| Samples | Storage period: 3 months pentane ($10^{-9}$M) | 3 months $O_2$ (%) | 3 months taste | 4 months pentane ($10^{-9}$M) | 4 months $O_2$ (%) | 4 months taste | 6 months pentane ($10^{-9}$M) | 6 months $O_2$ (%) | 6 months taste |
|---|---|---|---|---|---|---|---|---|---|
| Without vanillin (comparison) | 3.4 | 19.8 | 6 | 77.2 | 9.4 | 2 | | | 1 |
| With 50 µg/g vanillin | 6.2 | 19.4 | 5.3 | 48.8 | 11.3 | 1 | | | 1 |
| With 500 µg/g vanillin | 1.1 | 20.3 | 6 | 2.1 | 19.7 | 5.7 | 4.2 | 18.7 | 5.3 |
| With 5000 µg/g vanillin | 0.5 | 20.3 | 5.0 | 0.5 | 19.9 | 5 | 2.3 | 19.3 | 5.7 |

It can be seen that the flakes without vanillin are heavily oxidized on account of the relatively high fat content of oats. The antioxidant effect of adding vanillin is the more clearly apparent insofar as it is demonstrated both by the oxidation test and by the tasting test.

Example 4

To prepare dehydrated precooked cereal flakes, an aqueous dispersion of 500 kg oatmeal is prepared using such a quantity of water that the dispersion has a dry matter content of 40%.

The dispersion is heat-treated for 60 s at 130°C by injection of steam and is dried on a cylinder to a residual moisture content of 3.4%.

After taking some samples of flakes to be kept as such for comparison, 200 µg vanillin are added per g cereal flakes thus obtained.

The flakes are subjected to keeping tests in lacquered tinplate cans. Tastings by specialists after 2, 4 and 8 months reveal a considerable slowing down of the oxidative degradation of the flakes to which vanillin was added compared with the samples to which it was not added.

THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

1. The use of vanillin for the protection of a food against oxidation by addition of 5 to 5000 µg vanillin per g food dry matter.

2. The use claimed in claim 1, characterized in that the vanillin is in power form.

3. The use claimed in claim 1, characterized in that the vanillin is in the form of an alcoholic solution.

4. The use in accordance with claim 1, substantially as herein described with reference to any one of the examples.

DATED this 16th day of January 1992

SOCIETE DES PRODUITS NESTLE S.A.

Attorney: IAN T. ERNST

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