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(54) Title: HYGIENE ARTICLE WITH ODOUR CONTROL SUBSTANCE AND METHOD FOR PRODUCING IT

(57) Abstract: A hygiene article such as a sanitary napkin:, panty liner, diaper, pant diaper, adult incontinence guard, underlay or wipe, wherein said hygiene article contain an odour control substance in the form of an oxidized lipid. The oxidized lipid has a peroxide value as measured by AOCS Official Method Cd 8-53 between 100 and 1000 meq/kg and contains an -antioxidant in an amount of at least 0,15 wt% as calculated on the weight of the oxidized lipid.

HYGIENE ARTICLE WITH ODOUR CONTROL SUBSTANCE AND METHOD FOR PRODUCING IT

TECHNICAL FIELD

The present invention refers to a hygiene article such as a sanitary napkin, panty liner, diaper, pant diaper, adult incontinence guard, underlay or wipe, said hygiene article containing an odour control substance in the form of an oxidized lipid. The invention also refers to a method for producing a hygiene article containing an odour control substance in the form of an oxidized lipid.

BACKGROUND OF THE INVENTION

Odour control has become an important factor in hygiene articles. Odours occur e.g. as a result of discharges from the wearer of an absorbent hygiene article or as a result of the storage of bodily fluids in the article. These odours can be embarrassing for the wearer of the article. It is important, therefore, to reduce or prevent odours from occurring in absorbent articles while they are being worn. It is also desirable to be able to clean the urogenital area from odour substances by a wipe and/or to prevent odour to occur.

Examples of odour substances that may occur in absorbent hygiene articles are sulphur compounds, aldehydes, indoles, amines etc.

Various methods are used to prevent or reduce odours in absorbent hygiene articles that have arisen in conjunction with the discharge of bodily fluids. The methods are based on 1) masking of the odours; 2) a chemical reaction, for example in the form of neutralization, with an acid/base system; 3) adsorption/absorption of odours involving the creation of surfaces which exhibit a special affinity to the odours or large specific surfaces/cavities which are able to bind the odours concerned and thus to prevent them from remaining in gaseous form, or 4) bacteria inhibitors which reduce/control the growth of bacteria and associated odours that have arisen because of high bacteria counts.

Perfumes or fragrances are used, for example, in order to mask odours. Maskers do not remove the odourss and must be added in an appropriate quantity to ensure that the odour is covered and that the perfume does not smell too strongly. Zeolites, silicone dioxide, clays, active carbon and/or cyclodextrin, for example, are used for the adsorption of odour substances. Some of these are susceptible to moisture, however, which restricts

their effectiveness. Sodium bicarbonate, citric acid and/or superabsorbent materials with a low pH are used for the neutralization of certain odours. Bacteria can generate substances with an unpleasant odour, and copper acetate, a superabsorbent material with silver ions and/or an acidic superabsorbent material can be used to reduce the growth of bacteria. The above-mentioned odour control substances are effective against different kinds of odours and act with different mechanisms.

A number of odours are hydrophobic, and such odours are absorbed and/or adsorbed by hydrophobic odour control substances. Hydrophobic odouriferous substances include, for example, certain organic acids, sulphur compounds, aldehydes, indole, amines, etc., which commonly occur in conjunction with the use of absorbent articles.

Described in US 6 147 028 is an odour control substance in the form of polysiloxane-coated starch granules that are used in absorbent products. The starch granules have a hydrophobic surface, and they absorb hydrophobic material from the air.

US 6 479 150 describes material layers of thermoplastic fibres with a hydrophobic odour control substance that is modified with a surface-active substance in order to make the layer wettable. The odour control substance is, for example, an aromatic odour control substance.

Previously disclosed odour control substances suffer from the disadvantage, among other things, that they are difficult to distribute uniformly throughout the whole of the absorbent product. This is attributable to the fact that previously disclosed odour control materials often consist of solid particles, which cannot be distributed continuously over the internal and external surfaces of the product and as such reduce the degree of coverage. The possibility of trapping undesirable odours in an effective manner is reduced in this way.

GB 1 282 889 discloses a deodourant composition comprising at least one calcium, aluminium, magnesium or zinc salt of an unsaturated aliphatic hydroxycarboxylic acid having at least 17 carbons. It is further told that these metal salts can be combined with saturated aliphatic hydroxycarboxylic acids and unsaturated aliphatic hydroxycarboxylic acids. The saturated hydroxycarboxylic acids may either be naturally saturated or derived from oxidation products of unsaturated fatty acids, such as oleic acid, ricinoleic acid, linoleic acid and linolenic acid. These unsaturated fatty acids upon mild oxidation lead to

corresponding pure hydroxycarboxylic acids. Pure hydroxycarboxylic acids have very low oxidizing ability on other substances and a peroxide value close to 0 meq/kg.

WO 2008/058565 discloses absorbent articles comprising a peroxy compound as an organic zinc salt, in particular zinc ricinoleate, which are told to have a synergistic effect in the suppression of malodours, such as ammonia.

WO 2009/082287 discloses an absorbent article comprising oxidized lipids as odour control substance. The lipids are oxidized under controlled conditions, e g with ozone, to have a peroxide value of at least 20 meq/kg.

WO 2010/039064 discloses a wipe to which has been added an oxidized lipid as odour control substance. The lipids are oxidized to have a peroxide value of at least 20 meq/kg.

The need remains to develop odour control substances for hygiene products, and one object of the present invention is to develop a hygiene article comprising an effectively functioning odour control material in the form of an oxidized lipid, wherein the formation of undesired volatile substances caused by decomposition of the oil is reduced or prevented also during long storage periods.

SUMMARY OF THE INVENTION

The above defined problem is solved in the present invention by a hygiene article such as a sanitary napkin, panty liner, diaper, pant diaper, adult incontinence guard, underlay or wipe, said hygiene article containing an odour control substance in the form of an oxidized lipid, wherein said oxidized lipid has a peroxide value as measured by AOCS Official Method Cd 8-53 between 100 and 1000 meq/kg and contains an antioxidant in an amount of at least 0.15 wt% as calculated on the weight of the oxidized lipid.

The oxidized lipids may have a peroxide value as measured by AOCS Official Method Cd 8-53 of at least 150, preferably at least 200 and not more than 350, preferably not more than 325 meq/kg.

The antioxidant may be an oil soluble antioxidant.

The oil soluble antioxidant may be chosen from vitamin E, gum guaiac, propyl gallate, butylated hydroxyanisol, butylated hydroxytoluene or 2,4,5-trihydroxy hydro-quinone, vitamin K, vitamin A, vitamin D or carotenoids.. Vitamin E exists in eight different forms including four tocotrienols (α, β, γ, δ) and four tocopherols (α, β, γ, δ). Examples of carotenoids are β-carotene, lutein, lycopene.

The oxidized lipid may contain at least 0.2 wt% and more preferably at least 0.5 wt% antioxidant

The oxidized lipid may contain up to 5 wt%, preferably up to 3 wt% and more preferably up to 2 wt% antioxidant.

The lipids may be fatty acids or derivatives thereof.

The fatty acid derivatives may be esters of fatty acids, especially triglycerides.

At least part of the fatty acids and/or fatty acid derivatives may be unsaturated.

The oxidized lipids may be oxidized by treatment with oxygen

The invention further refers to a method of producing a hygiene article such as a sanitary napkin, panty liner, diaper, pant diaper, adult incontinence guard, underlay or wipe, said hygiene article containing an odour control substance, said method comprising the steps of oxidizing a lipid so that it will have a peroxide value as measured by AOCS Official Method Cd 8-53 between 100 and 1000 meq/kg, subsequently adding an antioxidant to the oxidized lipid and adding said oxidized lipid containing the antioxidant to said hygiene article or to a component which is to be incorporated in said hygiene article.

The lipid may be oxidized with oxygen gas.

The lipid may be exerted to UV radiation during oxidation.

The antioxidant may be added in an amount at least 0.15 wt% as calculated on the weight of the oxidized lipid.

DEFINITIONS

The term "hygiene article" refers to an article used for personal hygiene especially for absorbing bodily exudates, like urine, feces and menstrual fluid, or for wiping the skin to remove such bodily exudates. Examples of such hygiene articles include feminine hygiene products such as sanitary napkins, panty liners and sanitary panties, diapers and pant diapers for infants and incontinent adults, incontinence pads, diaper inserts, underlays, washcloths, patches, towelettes, napkins, wet wipes, and the like. The invention mainly refers to disposable hygiene articles, which means articles that are not intended to be laundered or otherwise restored or reused after a single use.

The term "lipid" denotes all fat-soluble (lipophilic), naturally-occurring substances, such as fats, oils, waxes, cholesterol, steroids, monoglycerides, diglycerides, triglycerides, phospholipids, and others.

By "oxidized lipids" is meant that the lipids have undergone an oxidation process wherein oxygen has been introduced in the lipid molecular structure. The oxidation agent is any agent, which leads to oxidation of the lipid structure, e.g. oxygen gas, ozone or peroxides.

By "antioxidant" is meant a compound that inhibits the oxidation of other molecules. Oxidation reactions can produce free radicals and antioxidants have the ability to eliminate such intermediates.

DETAILED DESCRIPTION OF THE INVENTION

It has been shown that oxidized lipids, are very effective in reducing certain odouriferous substances which are commonly occurring in hygiene articles. Natural animal-derived or plant-derived lipids are very often mixtures of mono-, di- and triglycerides and free fatty acids. The lipids can be purified, hydrated, refined, modified and used individually or in different mixtures. Examples of suitable lipids which originate from animals can be found in bees waxes, emu oil, lactis lipida, lanolin, shark's liver oil, lard, whale oil, butter fat and tallow. Examples of suitable lipids which originate from plants can be found in apricot kernel oil, ground nut oil, avocado oil/wax, blackcurrant seed oil, borage seed oil, Brazil nut oil, castor oil, cocoa butter, coconut oil, maize oil, cotton seed oil, rose hip seed oil, evening primrose oil, grape seed oil, linseed oil, mango seed oil, rose oil, olive oil, orange

wax, palm oil, ground nut oil, rice wax, sesame seed oil, shea butter, soybean oil, sunflower seed wax, peanut oil, sesame oil, safflower oil, tobaccoseed oil, poppyseed oil, teased oil, kapok oil, rice bran oil, sorghum oil, crambe oil, linseed oil, perilla oil, hempseed oil, tung oil, oiticica oil, palm kern oil, sweet almond oil and wheat germ oil. Further examples of lipids are waxy oils, which are esters of mono-alcohols, for example Jojoba oil, phospholipids etc.

Triglycerides are commonly occurring in many natural fats and oils, such as rapeseed oil, olive oil, maize oil, sunflower oil, palm oil, cocoanut oil and butter, palm oil, cacao butter, theobroma oil etc. Most of the naturally occurring triglycerides contain a mixture of saturated and unsaturated fatty acids, while the proportion of saturated and unsaturated fatty acids varies between the different oils. This proportion is usually given as the quotient: unsaturated/saturated. The unsaturated fatty acids may either be monounsaturated or polyunsaturated. The most commonly occurring fatty acids in triglycerides are palmitic acid, a saturated fatty acid, oleic acid, a monounsaturated fatty acid, linoleic and linolenic acids, which are polyunsaturated fatty acids.

The composition of some common natural oils are given in Table 1 below, which is taken from Bailey´s Industrial Oil and Fat products, vol.1, editor: Daniel Swern, John Wiley & Sons Inc., New York,1979.

Table 1

Vegetable oil	Saturated fatty acids (weight-%)	Unsaturated fatty acids (weight-%)
Olive oil	9.3-18.8	81.1-89.0
Sunflower oil	8.7-14.2	85-91
Rapeseed oil	6.2-9.5	90.5-93.8
Maize oil	12-18	82-88
Cocoa butter	59.8	40.2

Such oils and fats normally contain small amounts of antioxidants, either naturally occurring or added by a supplier, so that autoxidation caused by contact with air is substantially prevented or delayed.

The lipids used in the present invention are oxidized by an oxidizing agent. Examples of useful oxidizing agents are: oxygen gas, ozone, peroxides, peroxy acids and nitrogen

dioxide. The naturally occurring antioxidants will be destroyed during such oxidation (J. Am. Oil Chem. Soc. (2009) 86:895-90; Sabilov et al; Effects of Temperature and UV Light on Degradation of α-Tocopherol in Free and Dissolved Form.

The reactivity of different lipids is dependent on the number of double bonds, i.e. the degree of unsaturation. Saturated lipids oxidize very slowly while lipids with a high degree of unsaturation oxidize more rapidly.

The oxidation should be performed under controlled conditions until a desired peroxide value is reached. Preferably the oxidized lipids should have a peroxide value as measured by AOCS Official Method Cd 8-53 between 100 and 1000 meq/kg. The oxidized lipids may have a peroxide value of at least 150 or at least 200 meq/kg. They may further have a peroxide value of not more than 350 or not more than 325 meq/kg.

The lipids may be oxidized by any suitable method and by any suitable oxidation agent, for example by oxygen, ozone, mixtures of ozone/air or ozone/oxygen. The preferred oxidation agent according to the invention is oxygen gas. The lipids may further be exerted to UV-radiation during oxidation with oxygen gas.

At the oxidation process a series of peroxidic products may be formed, such as hydroperoxides, ozonides, diperoxides, peroxides and polyperoxides. Certain by-products may also be formed, for example ketones and aldehydes, which are less desired. These by-products may be removed by washing the lipids with a solvent after the oxidation process. Alternatively volatile undesired substances may be removed by evaporation, for example under vacuum.

Oxidation with oxygen gas instead of ozone reduces the formation of at least some of these undesired by-products. However the problem of autoxidation and/or decomposition of the oxidized lipids during storage remains. Such autoxidation may result in the formation of undesired by-products, such as aldehydes and ketones. These by-products have an unpleasant odour.

Addition of an antioxidant to the oxidized lipids after oxidation will prevent autoxidation and/or decomposition and the formation of such undesired by-products. The antioxidant is preferably oil-soluble. Examples of useful oil-soluble antioxidants are vitamin E, gum

guaiac, propyl gallate, butylated hydroxyanisol, butylated hydroxytoluene or 2,4,5-trihydroxy hydro-quinone, vitamin K, vitamin A, vitamin D or carotenoids.. Vitamin E exists in eight different forms including four tocotrienols (α, β, γ, δ) and four tocopherols (α, β, γ, δ). Examples of carotenoids are β-carotene, lutein, lycopene.

The antioxidant should be added to the oxidized lipid in an amount of at least 0.15 wt%, preferably at least 0.2 wt% or more preferably at least 0.5 wt% antioxidant as calculated on the weight of the oxidized lipid. The amount of antioxidant may be up to 5 wt%, preferably up to 3 wt% or more preferably up to 2 wt% antioxidant as calculated on the weight of the oxidized lipid.

The antioxidant that has been tested in the examples below is α-tocopherol.

One or more essential oils, such as chamomile, clove, lemon, lavender, rosemary and sandalwood may optionally be added to the oxidized lipid.

The oxidized lipids may thus be added to wood pulp fibres, for example cellulosic fluff pulp, which frequently is used in the absorbent core of a hygiene absorbent article, such as a sanitary napkin, panty liner, diaper, pant diaper, adult incontinence guard or underlay. Alternatively or in addition the oxidized lipids may be added to the topsheet of such an absorbent article or any additional functional layer contained in the absorbent article, such as liquid receiving layer, liquid distribution layer, liquid storage layer etc.

The pulp fibres treated with oxidized lipid may be mixed with untreated pulp and/or with superabsorbent material to form an absorbent core. An absorbent core can contain between 0.2 and 50% by weight, preferably between 0.5 and 40% by weight, more preferably between 1 and 35% by weight and most preferably between 3 and 30% by weight of added oxidized lipids calculated on the total weight of the hydrophilic fibres, for example pulp fibres, contained in the absorbent core.

In certain areas, for example along the edges of the absorbent core and/or other functional layer of the absorbent article, a higher proportion of the oxidized lipids may be used than in the central area of the absorbent core. The fibres in the edge areas may then be more or less saturated with the oxidized lipids and function as leak barriers.

In garment-like article, such as diapers and pant article the treated fibres can be positioned in a waistband region or in areas around the leg openings. They can then prevent odours from escaping from the article. Alternatively the oxidized lipids can be added to elastic means forming part of for example waist elastics or leg elastics in an absorbent article.

In the same manner as described above with respect to the absorbent core, the oxidized lipids can be uniformly or non-uniformly distributed in any other layer in an absorbent article, such as the topsheet, a liquid receiving layer, a liquid distribution layer or the like. The proportion by weight of the oxidized liquid in these layers can be the same as for the absorbent core.

Other methods for applying the oxidized lipids to a component in an absorbent article are by spraying, coating or impregnation.

Depending on the kind of absorbent article product, the proportion of oxidized lipids will vary. Panty liners, for example, do not require the same quantity of odour control material as an incontinence product.

Odours in absorbent articles differ in respect of their character. In order to achieve an even better odour-controlling effect, other types of odour control materials or odour control substances can also be used in the absorbent articles according to the present invention. These can be acidified cellulose fibres, for example, and/or superabsorbent materials with a low pH. Cellulose fibres can be acidified, for example, by the addition of a buffer/acid. The acidic odour control materials deal with odouriferous substances that are alkaline, for example, such as amines and ammonia. Acidic odour control materials are capable, if added in a sufficient quantity, of lowering the pH and, by so doing, of inhibiting the growth/activity of bacteria which in turn produce substances that are able to contribute to a badodour.

Other odour control substances can also be added to the article, for example chitosan, activated carbon, zeolites, clay, silicone dioxides, cyclodextrin, starch-based odour control substances and esters. The esters can be selected from among cyclical esters or esters selected from among isomentyl acetate, isomentyl propionate, isomentyl isobutyrate, isomentyl crotonate and isomentyl butyrate.

As mentioned above essential oils, such as lavender, sandalwood, lemon, chamomile, clove and rosemary oil may be added to the oxidized oil.

The oxidized lipids can be added to the pulp fibres, or other fibres, in conjunction with the production of the fibres or be added in the production apparatus in which the absorbent articles are produced.

For wipes the amount of oxidized lipids added may vary dependant on the intended use. For example in personal hygiene higher amounts of the oxidized lipids may be used in the urogenital area, where it is an advantage that the lipids remain on the skin, than in hand wiping, where it may be desired that only small amounts of the lipids remain.

The wipe can contain between 0.01 and 15 g/g, preferably between 0.1 and 8 g/g, more preferably between 0.2 and 4 g/g and most preferably between 0.3 and 3 g/g of added oxidized lipids calculated on the total weight of the wipe. The amounts may differ dependant on the intended use.

Upon use of the wipes the composition containing the oxidized lipids is preferably transferred and delivered to the skin thereby serving as a skin treatment agent, especially for odour control and/or bacteria control.

The oxidized lipids may be distributed evenly throughout the wipe. Alternatively, the oxidized lipids may be localized in specific areas of the wipe, especially on the surface thereof, so as to be easily released from the wipe and transferred to the skin

The composition with which the wipes of the present invention is impregnated may, in addition to the oxidized lipids, contain one or more of the following components: a viscosity regulating agent, a carrier for the oxidized lipid or an agent for improving the adhesion of the composition to the skin. Examples of viscosity regulating agents include polyethylene glycol (PEG) and glycerol. Quaternary tensides may be used as agents for improving the adhesion to skin. Other components which may be contained in the composition are cleaning agents, skin care agents, antibacterial agents, fragrances etc

The presence of oxidized lipids in wipes may also inhibit the growth/activity of bacteria which in turn produce substances that are able to contribute to a bad odour. The inhibition of growth/activity of unwanted bacteria is also important for hygienic reasons, both in the urogenital area, but also in handwiping. Frequent handwiping occurs for example in restaurants, kitchens, in medical care premises, in schools, in day care centers, industry, workshops etc. Besides the odour and bacteria control effects, the oxidized lipids may also have a skin care effect.

Other odour control substances can also be added to the wipe, for example chitosan, activated carbon, zeolites, clay, silicone dioxides, cyclodextrin, starch-based odour control substances and esters. The esters can be selected from among cyclical esters or esters selected from among isomentyl acetate, isomentyl propionate, isomentyl isobutyrate, isomentyl crotonate and isomentyl butyrate.

The carrier material used in the wipe should be chosen so that it can hold the oxidized lipids in its porous structure and release it to the skin when the wipe is used. It shall preferably also be capable to absorb substances that have been wiped off the skin. Examples of suitable carrier materials are fibrous materials such as tissue paper, airlaid tissue and different type of nonwoven materials. Examples of nonwoven materials are hydroentangled webs, spunbond, meltblown, thermobonded webs etc. Further examples of carrier materials are foams, nets, films etc. In the case of films the oxidised lipids may be applied between film layers and exposed when separating the film layers from each other and/or applied in formed recesses in the film.

The structure of the carrier material is important for its function to hold liquid substances. A material that is especially suitable in this respect is hydroentangled webs.

Fibres that are useful in fibrous carrier materials are pulp fibres, cotton fibres, bamboo fibres and other natural fibres, regenerated cellulose fibres such as viscose, lyocell, polyolefin fibres, like polyethylene and polypropylene, polyester and mixtures thereof.

For a so called wet wipe a suitable fibre composition may be a mixture of viscose fibres and polyester fibres, for example 70 wt% viscose fibres and 30 wt% polyester.

A common fibre composition in other type of wipes is a mixture of pulp fibres and polypropylene.

A suitable basis weight for a personal hygiene wipe is between 30 and 70 g/m^2, preferably between 40 and 50 g/m^2.

The composition comprising the oxidized lipids may be added to the carrier material by spraying, coating or impregnation.

EXAMPLES

Example 1. Oxidation of vegetable oils

200 mL of vegetable oil (sunflower oil, corn oil or olive oil) was placed in a beaker of 11 cm, heated and oxygen bubbling through the oil (gas flow=60 l/h), a magnetic stirrer was used in the beaker. Simultaneously, a Mineralight UV Lamp, Model S-68, 220Volts, 50 Hz, 0.65 Amps was used for sample irradiation. The distance between the UV lamp and the oil surface was 5 cm. Depending on the oil, reaction time and temperature for achieving the highest peroxide value change. The results are given in Table 2.

Table 2. Reaction conditions to achieve the highest peroxide value

Vegetable Oil	Time (hours)	Temperature (°C)
Sunflower Oil	22.1	80
Corn Oil	6.0	110
Olive Oil	24.0	110

The degree of oxidation was tested by determining the peroxide value according to the test method AOCS Official Method Cd 8-53: Peroxide Value Acetic Acid – Chloroform Method. The peroxide value for both the starting oils and the oxidized oils was determined. The results are given in Table 3 below.

Table 3. Peroxide values of native and oxidized oils

Oil	Peroxide Value (meq/Kg)
Sunflower Oil	7
Oxidized Sunflower Oil	396
Corn Oil	4
Oxidized Corn Oil	366

Olive Oil	8
Oxidized Olive Oil	347

Example 2. Analysis of odour reduction

Treatment of pulp with oils

Sheets of fluff-grade bleached sulphate pulp from International Paper, were impregnated with a solution of the tested oil in acetone. To a pulp sheet weighing 4 g was added 1.71 g oil in 1.71 g acetone. The solution was evenly distributed over the surface of the sheets. When the acetone had evaporated, the sheets contained 30 wt% oil and 70 wt% pulp fibers. The sheets were defibrated in an IKA A11 basic Analytical mill to produce fluffed pulp during approximately 1 minute. Following the same procedure pulps with 3 wt% and 10 wt% were prepared. In those cases 0.12 g and 0.44 g of oil were added respectively.

For the experiment four (4) malodour substances were used: dimethyl sulfide (DMS), dimethyl disulfide (DMDS), isovaleraldehyde and diacetyl. Due to the difference between the solubility of the compounds two different solutions were prepared (Solution A and Solution B)

- Solution A:
  - Solution A.1: In a vial wih septum were placed 22,14 g polyethyleneglycol with a molar weight 300 g/mole (PEG300), 115 µL DMS, 90 µL DMDS and 120 µL Isovaleraldehyde.
  - Solution A.2: From solution A.1 were taken 50 µL and placed in another vial with 22,44 g PEG300.
- Solution B:
  - Solution B.1: To a 250 ml volumetric flask were added 105 µL diacetyl using distilled water as solvent.
  - Solution B.2: From solution B.1 were taken 0,6 ml and placed in a vial with 9,4 ml of distilled water.

Odour reduction measurement:

1 g treated pulp was placed in a 60 ml vial, after which 3.9 ml of 0.01 phosphate buffer saline pH 7.4 from Sigma was added with 0.1 mL of the desired odour solution (A.2 or B.2). The final concentrations were 300 ng/ml for dimethyl sulfide (DMS), dimethyldisulfide (DMDS) and Isovaleraldehyde (IVAL), 600 ng/ml for diacetyl.

The vial was then placed in an oven and after 3 hours at a temperature of 30°C, after this time a SPME fibre (Supelco), 75 um Carboxen-PDMS, was injected into the headspace above the pulp and after additional 0.5 h the SPME fiber was analyzed with gas chromatography (GC), thermo Finnigan Trce, with a MS detector. The chromatographic

analysis was carried out on a Termo Finnigan Trace GC with a MS detector. The GC was equipped with a split/splitless injector, mode splitless and 180°C in the transfer line. The column used was a ZB-624 (Zebron),30 m, 0.25 mm i.d, 1.40 µm film thickness. The MS mode was full scan, when the compounds were analysed the following mass numbers were chosen:

62 for DMS, 58 for IVAL,

94 for DMDS, 86 for diacetyl.

The peak area of each odour substance was determined for samples with treated pulp and an untreated reference pulp, results are shown in Table 4. The odour reduction in comparison to untreated pulp was made by equation (1):

$$Odour\ reduction\ (\%) = \left[1 - \left(\frac{Actual\ peak\ area}{Peak\ area\ of\ sample\ with\ untreated\ pulp}\right)\right] x100\% \qquad (1)$$

Table 4. Results odour reduction (%)

	Peroxide Value (meq/kg)	DMS	IVAL	DMDS	Diacetyl
30% Sunflower Oil	7	0	0	31.6	4.7
3% oxidized sunflower oil	396	99.9	99.6	72.6	61.7
10% oxidized sunflower oil	396	99.8	98.3	81.8	66.9
30% oxidized sunflower oil	396	99.9	99.6	99.0	83.4
30% Corn Oil	4	87.1	66.0	88.4	2.8
3% oxidized corn oil	366	99.9	98.1	62.3	72.8
10% oxidized corn oil	366	100	98.2	81.5	79.6
30% oxidized corn oil	366	100	98.8	98.3	88.4
30% Olive oil	8	84.0	69.2	73.0	4.5
3% oxidized olive oil	347	78.7	88.9	47.8	39.3
10% oxidized olive oil	347	95.6	98.7	70.9	45.6
30% oxidized olive oil	347	99.4	99.8	89.4	65.3

Example 3. Preparation of oxidized sunflower oils with different peroxides values

200 mL of sunflower was placed in a beaker of 11 cm, heated and gas bubbling through the oil (gas flow=60 l/h), a magnetic stirrer was used in the beaker. Simultaneously, a Mineralight UV Lamp, Model S-68, 220Volts, 50 Hz, 0.65 Amps was used for sample irradiation. The distance between the UV lamp and the oil surface was 5 cm. Different reaction conditions were tested. Sunflower oil was oxidized at different temperatures: 55,

80 and 110 °C. The influence of the bubbled gas was also tested. In this case it was bubbled oxygen, air or no gas bubbling. Results are shown in Table 5.

Table 5. Influence of reaction conditions on the peroxide value

Reaction time (h)	110 °C, oxygen	80 °C, oxygen	55 °C, Oxygen	80°C, no gas bubbling	80°C, air
1,5	85,4				
3,5	306,7	12,4	7,9	8,8	3,0
5	335,1				
5,5	277,5				
6	287,5	23,0	11,2	12,9	6,0
7,5	272,7	41,6	25,1	14,4	23,8
16		71,7			
20		101,0			
21		142,6			
22		341,6			
22,1		395,9			
24		379,2	34,3	89,7	101,6
28		-	37,4	235,2	197,2
32		336,5	40,9	323,7	353,1
48		326,3	65,3	362,1	327,8
56		294,6	72,4	374,4	298,3
72		170,6	78,6	357,6	275,0

White spaces mean that the measurement was not made

Example 4.Tests with different amounts of added oils having different peroxides values

This experiment was made using 3% and 30% addition of the sunflower oil. The different peroxides values were tested, for sunflower oil the peroxides values were: 14, 34, 65, 101, 235, 324, 363 and 396 meq/Kg. The measurement of peroxide value and the odour characterization was made as described in example 1 and 2. Tables 6 and 7 show the results.

Table 6. % odour reduction after 3wt% addition oxidized sunflower oil

	% odour reduction after 3wt% addition oxidized oil				
Peroxide Value (meq/Kg)	DMS	IVAL	DMDS	Diacetyl	
14	63,4	39,5	23,0	2,5	
34	74,3	78,2	28,8	26,8	
65	74,5	89,6	33,0	36,6	
101	85,6	97,9	33,5	43,5	
235	99,9	97,6	51,2	52,9	
323	99,9	96,7	51,9	55,3	
363	99,9	98,3	68,5	56,5	
396	99,9	99,4	72,6	61,7	

Table 7. % odour reduction after 30wt% addition of oxidized sunflower oil

	% odour reduction after 30wt% addition of oxidized oil				
Peroxide Value (meq/Kg)	DMS	IVAL	DMDS	Diacetyl	
14	73,3	72,4	53,5	9,7	
34	87,8	81,7	71,0	23,1	
65	92,5	88,2	71,1	45,9	
101	99,9	94,4	77,9	66,9	
235	100,0	99,2	99,9	77,2	
323	100,0	99,6	98,7	78,3	
363	100,0	100,0	98,6	80,2	
396	99,9	99,6	99,0	83,4	

Example 5. Preparation of oxidized vegetable oils containing α-tocopherol

After completed oxidation an antioxidant in the form of α-tocopherol was added to some of the oxidized oils from Example 1. The following amounts of α-tocopherol were added: 0.15 wt%, 0.5 wt%, 1 wt%. The amounts of α-tocopherol were calculated on the weight of the oxidized oil.

Example 6. Measurements of volatile substances from pulp samples containing oxidized vegetable oils with and without α-tocopherol after different storage times

SPME-GC–MS was used to determine the relative changes in volatiles formed in sunflower oil during the storage. 1 g of oxidized oil was placed into a vial. The SPME needle was pierced through the septum of the sample vial, exposing the fiber to the

headspace of the sample for 30 min. This allowed the analytes to reach their near-equilibrium concentration with the active phase of the fiber. Subsequently, the needle was pierced through the inlet septum on the GC injector and the fiber was exposed for 5 min. The MS spectra library was used for identification of the measured compounds.

The analyses were conducted after 0, 2, 4, 8 and 12 weeks of storage in order to study the stability of oxidized sunflower oil. Since the oxidized oils have a variety of volatile compounds, only five of them were chosen. In this way, it is easier to see how the chosen volatile compounds change over time; results are shown in Table 8 and 9.

Table 8. Pulp + 30% oxidized sunflower oil

	Peak Area (1 x10^5)				
Compound	0 weeks	2 weeks	4 weeks	8 weeks	12 weeks
n-pentane	5,5	139,0	185,5	421,9	252,0
heptane	0,2	15,0	24,0	33,4	74,0
pentanal	9,6	90,3	84,4	123,1	529,2
octane	0,9	87,1	144,9	277,4	362,6
hexanal	34,3	183,9	190,1	239,4	50,8

Table 9. Pulp + 30% oxidized sunflower oil containing 1wt% α-tocopherol

	Peak Area (1 x10^5)				
Compound	0 weeks	2 weeks	4 weeks	8 weeks	12 weeks
n-pentane	1,9	1,8	1,2	1,2	1,1
heptane	2,1	1,2	0,3	0,1	0,0
pentanal	28,4	28,7	30,7	34,2	49,2
octane	1,0	0,1	0,0	0,3	0,3
hexanal	2,2	10,2	13,5	18,3	18,8

Example 7. Analysis of odour reduction after 12 weeks of storage

It was important to test if oxidized oils containing α-tocopherol were able of reducing odours. In this experiment, the efficiency of oxidized sunflower oil after 12 weeks was also tested. Added levels of α-tocopherolwere 1%, 0.2% and 0.01%.

Treatment of pulp with oils

Sheets of fluff-grade bleached sulphate pulp from International Paper, were impregnated with a solution of the tested oil in acetone. To a pulp sheet weighing 4 g was added 1.71 g oil in 1.71 g acetone. The solution was evenly distributed over the surface of the sheets. When the acetone had evaporated, the sheets contained 30 wt% oil and 70 wt% pulp fibers. The sheets were defibrated in an IKA A11 basic Analytical mill to produce fluffed pulp during approximately 1 minute.

For the experiment three (3) malodour substances were used: dimethyl sulfide (DMS), dimethyl disulfide (DMDS) and isovaleraldehyde (IVAL)

- Solution A:
  - Solution A.1: In a vial wih septum were placed 22,14 g polyethyleneglycol with a molar weight 300 g/mole (PEG300), 115 µL DMS, 90 µL DMDS and 120 µL Isovaleraldehyde.
  - Solution A.2: From solution A.1 were taken 50 µL and placed in other vial with 22,44 g PEG300.

Odour reduction measurement:

1 g treated pulp was placed in a 60 ml vial, after which 3.9 ml of 0.01 phosphate buffer saline pH 7.4 from Sigma was added with 0.1 mL of the odour solution. The final concentrations were 300 ng/ml for dimethyl sulfide (DMS), dimethyldisulfide (DMDS) and Isovaleraldehyde (IVAL). The vial was then placed in an oven and after 3 hours at a temperature of 30°C, after this time a SPME fibre (Supelco), 75 um Carboxen-PDMS, was injected into the headspace above the pulp and after additional 0.5 h the SPME fiber was analyzed with gas chromatography (GC), thermo Finnigan Trce, with a MS detector. The chromatographic analysis was carried out on a Termo Finnigan Trace GC with a MS detector. The GC was equipped with a split/splitless injector, mode splitless and 180°C in the transfer line. The column used was a ZB-624 (Zebron),30 m, 0.25 mm i.d, 1.40 µm film thickness. The MS mode was full scan, when the compounds were analysed the following mass numbers were chosen:

62 for DMS, 58 for IVAL,

94 for DMDS, 86 for diacetyl.

The peak area of each odour substance was determined for samples with treated pulp and an untreated reference pulp, results are shown in Table 10. The odour reduction in comparison to untreated pulp was made by equation (1):

$$Odour\ reduction\ (\%) = \left[1 - \left(\frac{Actual\ peak\ area}{Peak\ area\ of\ sample\ with\ untreated\ pulp}\right)\right] x100\% \qquad (1)$$

Table 10. Odour reduction after addition of α-tocopherol and comparison between samples after 0 and 12 weeks

	DMS	IVAL	DMDS
30% Oxidized Sunflower oil - 0 weeks	99,9	99,6	99
30% Oxidized Sunflower oil - 12 weeks	100	95,0	80,6
30% Oxidized Sunflower oil + 1% α-tocoph.- 0 weeks	80,7	95,5	79,5
30% Oxidized Sunflower oil + 1% α-tocoph.- 12 weeks	84,3	96,5	74,7
30% Oxidized Sunflower oil + 0.2% α-tocoph.	91,1	98,5	71,0
30% Oxidized Sunflower oil + 0.01% α-tocoph.	98,4	99,0	71,2

CLAIMS

1. A hygiene article such as a sanitary napkin, panty liner, diaper, pant diaper, adult incontinence guard, underlay or wipe, said hygiene article containing an odour control substance in the form of an oxidized lipid ***characterized in*** that said oxidized lipid has a peroxide value as measured by AOCS Official Method Cd 8-53 between 100 and 1000 meq/kg and contains an antioxidant in an amount of at least 0.15 wt% as calculated on the weight of the oxidized lipid.

2. A hygiene article as claimed in claim 1, ***characterized in*** that the oxidized lipids have a peroxide value as measured by AOCS Official Method Cd 8-53 of at least 150, preferably at least 200 and not more than 350, preferably not more than 325 meq/kg.

3. A hygiene article as claimed in claim 1 or 2, ***characterized in*** that the antioxidant is an oil soluble antioxidant.

4. A hygiene article as claimed in claim 3, ***characterized in*** that the oil soluble antioxidant is chosen from vitamin E, gum guaiac, propyl gallate, butylated hydroxyanisol, butylated hydroxytoluene or 2,4,5-trihydroxy hydro-quinone, vitamin K, vitamin A, vitamin D or carotenoids.

5. A hygiene article as claimed in claim 4, ***characterized in*** that the oil soluble vitamin is α-tocopherol.

6. A hygiene article as claimed in any of the preceding claims, ***characterized in*** that the oxidized lipid contains at least 0.2 wt% and more preferably at least 0.5 wt% antioxidant.

7. A hygiene article as claimed in claim 6, ***characterized in*** that the oxidized lipid contains up to 5 wt%, preferably up to 3 wt% and more preferably up to 2 wt% antioxidant.

8. A hygiene article as claimed in any of the preceding claims, ***characterized in*** that the lipids are fatty acids or derivatives thereof.

9. A hygiene article as claimed in claim 8, ***characterized in*** that the fatty acid derivatives are esters of fatty acids, especially triglycerides.

10. A hygiene article as claimed in claim 8 or 9, ***characterized in*** that at least part of the fatty acids and/or fatty acid derivatives are unsaturated.

11. A hygiene article as claimed in any of the preceding claims, ***characterized in*** that said oxidized lipids are oxidized by treatment with oxygen.

12. A method of producing a hygiene article such as a sanitary napkin, panty liner, diaper, pant diaper, adult incontinence guard, underlay or wipe, said hygiene article containing an odour control substance as claimed in any of claims 1-11, ***characterized in*** oxidizing a lipid so that it will have a peroxide value as measured by AOCS Official Method Cd 8-53 between 100 and 1000 meq/kg, subsequently adding an antioxidant to the oxidized lipid and adding said oxidized lipid containing the antioxidant to said hygiene article or to a component which is to be incorporated in said hygiene article.

13. A method as claimed in claim 12, ***characterized in*** oxidizing the lipid with oxygen gas.

14. A method as claimed in claim 12 or 13, ***characterized in*** exerting the lipid to UV radiation during oxidation.

15. A method as claimed in any of claims 12-14, ***characterized in*** adding the antioxidant in an amount of at least 0.15 wt% as calculated on the weight of the oxidized lipid.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/SE2012/050926

A. CLASSIFICATION OF SUBJECT MATTER

IPC: see extra sheet

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC: A61F, A61L, C11B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE, DK, FI, NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-Internal, PAJ, WPI data, BIOSIS, COMPENDEX, MEDLINE, PUBCHEM

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 2010039064 A1 (SCA HYGIENE PROD AB ET AL), 8 April 2010 (2010-04-08); page 5, line 21 - page 6, line 6; pages 14-15; claims 1-2	1-15
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A	US 20020058917 A1 (HISANAKA TAKAYUKI ET AL), 16 May 2002 (2002-05-16); paragraphs [0035]-[0049], [0086]-[0123]; claims 1-4, 7	1-15
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A	US 20040122387 A1 (LONG ANDREW MARK ET AL), 24 June 2004 (2004-06-24); paragraph [0022]; claims 1-3	1-15
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☒ Further documents are listed in the continuation of Box C. ☒ See patent family annex.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier application or patent but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
13-05-2013	13-05-2013
Name and mailing address of the ISA/SE Patent- och registreringsverket Box 5055 S-102 42 STOCKHOLM Facsimile No. + 46 8 666 02 86	Authorized officer Ingemar Wistrand Telephone No. + 46 8 782 25 00

Form PCT/ISA/210 (second sheet) (July 2009)

INTERNATIONAL SEARCH REPORT

International application No.
PCT/SE2012/050926

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	JP 2003153945 A (FUKUMOTO KATSUMORI ET AL), 27 May 2003 (2003-05-27); (abstract) Retrieved from: WPI database; Original document: claims 1-3; [0021] --	1-15
A	US 5783247 A (VAN DER HEIJDEN ARNOLDUS ET AL), 21 July 1998 (1998-07-21); column 5, line 19 - column 6, line 13; claims 1, 10, 20-21 --	1-15
A	GB 1282889 A (GRILLO WERKE AG), 26 July 1972 (1972-07-26); pages 5-9; claim 1 --	1-15
A	US 20120027705 A1 (GAMMELSAETER RUNHILD ET AL), 2 February 2012 (2012-02-02); paragraph [0035]; claims 1, 8-9 -- --------	1-15

International application No.
PCT/SE2012/050926

Continuation of: second sheet

International Patent Classification (IPC)

A61L 15/46 (2006.01)
A61F 13/15 (2006.01)
C11B 5/00 (2006.01)

INTERNATIONAL SEARCH REPORT

International application No.
PCT/SE2012/050926

Box No. II Observations where certain claims were found unsearchable (Continuation of item 2 of first sheet)

This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☒ Claims Nos.: 1-2 and 12 in part
because they relate to subject matter not required to be searched by this Authority, namely:

See extra sheet

.../...

2. ☐ Claims Nos.:
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:

3. ☐ Claims Nos.:
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box No. III Observations where unity of invention is lacking (Continuation of item 3 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims.

2. ☐ As all searchable claims could be searched without effort justifying additional fees, this Authority did not invite payment of additional fees.

3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:

4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

☐ The additional search fees were accompanied by the applicant's protest and, where applicable, the payment of a protest fee.

☐ The additional search fees were accompanied by the applicant's protest but the applicable protest fee was not paid within the time limit specified in the invitation.

☐ No protest accompanied the payment of additional search fees.

Continuation of: Box No. II

Present claims 1-2 and 12 relate to a product and a method defined by reference to the following parameter:

"... has a peroxide value as measured by AOCS Official Method Cd 8-53 between 100 and 1000 meq/kg ..."

The use of these parameters in the present context is considered to lead to a lack of clarity within the meaning of Article 6 PCT. It is impossible to compare the parameters the applicant has chosen to employ with what is set out in the prior art. The lack of clarity is such as to render a meaningful complete search impossible.
Furthermore, an attempt is made to define the product and method by reference to a result to be achieved. Again, this lack of clarity in the present case is such as to render a meaningful search over the whole of the claimed scope impossible.

Consequently, the search has been restricted to the following:

A hygiene article (or a method of producing said article) containing an odour control substance in the form of oxidized lipid and that it further contains an antioxidant in an amount of at least 0.15 % as calculated on the weight of the oxidized lipid.

Thence it follows that a reasoned statement under Rule 43bis.1(a)(i) with regard to novelty, inventive step or industrial applicability only is established for those parts mentioned above.

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