Abstract: A process for obtaining perfumes, colognes and purified alcohol based on the utilization of compositions containing natural hydro-colloid-forming polysaccharides obtained from animal, plant and microbial sources as well as polysaccharides that are modified chemically or physically which added to hydro-alcoholic solutions, in combinations or not, lead to the flocculation and improvement of sensorial characteristics of the end product, reducing the time taken for maturing and allowing the process to be performed at room temperature and improving the removal of substances that interfere on the perception of the scent notes of the fragrance. The composition may be applied in the manufacture of perfumes, hydro-alcoholic solutions, alcohol for special uses and other solutions and are particularly useful for accelerating the polishing and maturing of perfumes and colognes.
PROCESS FOR MANUFACTURING PERFUMES AND COLOGNES AND
FOR PURIFYING AND POLISHING ALCOHOL

Field of the invention

This invention involves a new process for producing perfumes and
colognes and for purifying and polishing alcohol with natural hydrocolloids
obtained from animal, plant or microbes to be used as clarifying agents and
for improving the sensorial aspects of hydro-alcoholic solutions. The product
have particularly wide application in the perfume industry and can be used for
the removal of undesirable substances though preserving the essential
characteristics of the final product.

Background of the invention

Until present the procedure for manufacturing perfumes generally
starts with the extraction of scents from flowers, fruits, barks, seeds,
exudates and other plant parts and products. The extract may be later
subjected to purification procedures. These natural extracts and synthetic
substances are combined at specific proportions by professional perfumers,
in order to reach the right sensorial effect, resulting in the creation of
exclusive formulas, having unique fragrances defined by the perception of
scent notes. The final product is the result of the dilution of this formula of
scented ingredients in a mixture of ethanol and water in concentrations that
can vary between 2 and 20 % of the concentrated formula. Other non-
scented components may also be added to the mixture in order to alter its
original color, improve its stability or add other properties to the product.

Many of the natural components of the perfume formulas contain a
series of impurities which need to be removed from the final product so that
the perfume reaches the level of purity, the scent quality and the level of
transparency that are required.

When the fragrance concentrate is added to the polar mixture of
ethanol and water the non-polar impurities precipitate as very fine particles
that tend to form a stable suspension. The composition of such particles
depends on the concentration of the concentrate and its physical and
chemical properties may vary significantly. Frequently the impurity particles
appear as paste matters or as liquid droplets. The usual procedure adopted for the removal of such particles is through filtration which may be done by different techniques and with the use of various equipments. When the impurities appears as paste or liquid materials its removal may require a cooling stage which may take a few hours or up to several days. This leads to the aggregation and solidifying or the particles allowing for an easier retention in the filters. The removal of non-soluble, finely spread particles in a suspension is also known as the polishing of the solution.

Besides the non-soluble impurities the solution containing the scented ingredients in water and ethanol also contain molecules having odors that interfere in the final perception of the fragrance. The removal of such molecules is achieved through traditional techniques involving the maintenance of the solution in still conditions for a certain period of time allowing for the progression of reactions of condensation and oxidation which leads to chemical changes of these molecules preventing the, otherwise, resulting interference on the perception of the different fragrance components which were originally planned by the perfumer. This procedure is known as fragrance maturing and may also involve the refrigeration of the mixture.

Therefore, traditional techniques used in perfume manufacturing involve stages of scent concentrate preparation, solubilization of the formula components in a hydro-alcoholic mixture, resting (maturing), refrigeration, filtration and bottling. Conditions adopted for each stage are determined by the manufacturer, taking into consideration the perfume composition and the quality requirements as defined by the manufacturer and the market. Stages of maturing and refrigeration are critical for the process and may be highly costly for the manufacturer, as they involve investing in the acquisition of expensive equipment, keeping the product that is being manufactured in storage for long periods of time, as well as significant expenses in energy for refrigeration.

Prior Art

Several clarification products have been utilized in the beverage industry which faces problems that are similar to those faced by the perfume
industry.

The Brazilian patent document PI 9700752-8 describes a composition obtained from seaweeds of the class RHODOPHYCEAE, which is rich in polyelectrolyte biopolymers, which, when added to hydro-alcoholic solutions that are contaminated with waxes, resins or phospholipids, triggers a flocculation process through polymeric bridges and, in the conditions found at hydro-alcoholic medium, forms a micro-net that is responsible for the sweep-flocculation leading to the clarification and polishing of the solution. Such mechanism, acting in combination with the results obtained experimentally, indicate a wide range of potential applications of these products in many different areas of knowledge.

The patent document WO 98/000519 describes the use of a colloidal composition as a clarification agent and finishing of beverages produced by fermentation such as various kinds of beer, wines, low alcohol content beverages and their by products. The addition of Isinglass (collagen produced from fish) to beer is commonly used as a manner to promote its clarification and improving the efficacy of filtration. The composition described in that document includes polysaccharides such as pectin and may contain cooperating agents such as silicates. Those substances have nevertheless proven to be harmful as they may provoke allergic responses to the consumers and they also alter the original characteristics of the beverage.

The patent document WO 2006/032088 describes a special kind of pectin, obtained from plants, a non-allergenic polysaccharide which when added to beverages promotes their clarification at a level equivalent or even superior to that obtained through the use of Isinglass. The technique involves the use of this pectin combined with an \( \text{SO}_2 \) donor, such as sodium or potassium metabisulfite, at a proportion of 3.5 to 7.5 : 1, preferentially at 5:1.

Nevertheless, these clarification agents, in most cases interfere on the characteristics of the final product, modifying its aroma, taste or color.

The objective of the present invention is of introducing a new process for manufacturing perfumes and colognes as well as purifying the alcohol
used in their manufacturing. This process is capable of promoting their clarification within a short period of time, at room temperature, and is also capable of improving the removal of undesirable components that interfere in the fragrance without altering the sensorial features planned for the perfume or cologne.

Summary of the invention

This invention involves a process of obtaining perfumes and colognes and is based in the use of natural hydro-colloid-forming polysaccharides, which are obtained from animal, plant or microbial sources as well as from polysaccharides chemically or physically modified which are added to hydro-alcoholic solutions in combinations or not and act as flocculation agents and improving the sensorial features of the product, reducing the maturing time to a few hours even under ambient temperatures and helping with the removal of substances that interfere in the perception of the scent notes of the fragrances. In cases where the addition of different hydrocolloids in the solution, the first product may have the purpose of removing the undesirable components that are dissolved or suspended in the solution whereas the second product may serve as inducing agent leading to the formation of denser and more consistent flakes which are easier to remove from the solution.

The components that are the object of the present invention may be applied in the manufacture of perfumes, hydro-alcoholic solutions, alcohol for special uses, as well as other solutions, and are particularly useful at accelerating the polishing and maturing of perfumes and colognes.

Brief Description of the Figures

Figure 1 shows a scheme representing the removal of particles comparing the mechanisms involved in the traditional method (FIG. 1A) with the new process that utilizes hydrocolloids (FIG. 1B).

Figures 2A and 2B show Graphs 1 and 2 which compare the results obtained with the traditional process (Graph 1) with those obtained through the application of the newly invented process (Graph 2).

Figure 3 shows the chromatograms where the composition of a
perfume prepared traditionally (FIG. 3A) is compared with that of a perfume produced by utilizing the newly invented method involving hydrocolloids (FIG. 3B).

**Detailed Description of the Invention**

The process for obtaining the hydro-colloid compositions are not part of the invention but will be described herein in order to illustrate the properties and the general characteristics of the products involved.

In order to obtain the hydrocolloids from seaweeds, the algae are firstly washed and dried and subjected to a cleaning treatment with nitric acid or hydrochloric acid in concentrations which may vary from 0.5% to 5.0%, for a period of at least 10 minutes, under temperatures which may vary from 5°C to 40°C, depending on the degree of calcification of the algae. These are later washed in a neutralizing solution containing a selected hydroxide which may be calcium, sodium, potash or ammonium or their respective carbonates or bicarbonates at concentrations that may vary from 0.2% to 5.0%, for minimum periods of 10 minutes, under temperatures that may vary from 5°C to 40°C. The excess of neutralizing agent is removed in flowing water and the algae are then subjected to the extraction process by utilizing pure water as the solvent or water solutions containing mineral salts, alcohols or glycols in proportions that may vary according to the algae to be treated under temperatures which may vary from 50°C to 120°C, for periods varying from 20 minutes up to 2 hours. The volume of the extracting liquid is kept constant, if necessary through condensation and reflux. The resulting extract is then filtered or centrifuged for the removal of insoluble residues.

Hydrocolloids from animal sources may be obtained from shrimp and similar crustacean, usually as residues from seafood processing industries. This raw material is treated in order to remove coarser residues and is subjected to a demineralization treatment with an acid solution at concentrations ranging from 2.5% to 10.0%. The calcium-rich acid solution is discarded and the demineralized residue is then extracted with a solution of sodium hydroxide at 5 % for the removal of the proteins. The alkaline protein solution is removed, the solid materials are washed with water until a
neutral pH is reached and is then treated with a sodium hypochlorite solution containing 0.3 to 6% of active chlorine for the removal of color and odor. The polysaccharide hence obtained is chitin which is then treated with a concentrated sodium hydroxide solution at 60°C for 2-4 hours until its deacetylation. The raw chitosan is dissolved into an aqueous acetic acid solution, the non-soluble impurities are removed by filtration and the clear solution is precipitated by adding a 8% sodium hydroxide solution to it. The pure chitosan that precipitates is washed in water until a neutral pH is reached. This is then dried and ground. The chitosan solution used in the newly described process can be prepared by adding water or acid solutions to the dried chitosan at concentrations that are adequate for obtaining the solution that is needed.

Pectin hydrocolloids can also be obtained from fruits such as apples - whole fruits, apple peels and residues obtained from the apple juice industry may serve as raw material. The dry materials are treated with a hot acid solution at temperatures that may vary from 60 to 100°C. The acid solution may be prepared from mineral or organic acids and should have a pH between 1.5 and 3.5. Depending on the conditions of the raw material and the characteristics needed for the pectin the extraction may take 30 minutes up to several hours. Subsequently the material is filtered through activated charcoal for color removal, it is vacuum concentrated and is then precipitated in ethanol. The precipitated pectin is removed by filtration, washed in ethanol and left to dry. The dry pectin is ground and, depending on the intended end use, mixed with calcium salts or organic acids. The pectin solution utilized in the newly invented process may be prepared by simply adding water to dry pectin readily available in the market at the appropriate concentrations.

In order to obtain hydrocolloids from gum Arabic or gum acacia the raw gum obtained from the exudate from trunks of acacia tree (Acacia Senegal) is dissolved in water and filtered for the removal of insoluble impurities, clarified with activated charcoal and filtered once again as to obtain a transparent solution. This solution can be used directly in the process of fragrance production or it can be spray-dried or concentrated and
precipitated with ethanol, filtered and dried. The gum arabic solution is used in the newly invented process by the simple addition of water to the gum at concentrations that are adequate for obtaining the hydro-colloid solution needed for the process.

Obtaining hydrocolloids from guar seeds \( \textit{(Cyamopsis tetragonolobus)} \) involves removing the hulls, grinding and sieving them in order to obtain a powder. The fine powder is then suspended in ethanol for removal of additional impurities, filtered and dried. The solution of gum guar used in the newly invented process may be prepared by simply adding water to the dried gum at concentrations that are adequate for obtaining a solution of hydrocolloids adequate for the process.

In order to obtain hydrocolloids from the bacterium \textit{Xanthomonas campestris}, a culture medium containing an aqueous solution of sucrose, peptone, urea, potassium hydrogen phosphate, iron chelated with EDTA and magnesium salts is seeded with a suspension of \textit{X. campestris} and left to ferment in aerated conditions under continuous agitation under a temperature of 28°C for 5 days. The gum produced by the bacterium is precipitated with ethanol 98°GL, and is then dried and ground. The solution of xanthan gum used in the newly invented process may be prepared by simply adding water to the dried gum at adequate concentrations in order to prepare the hydro-colloid solution to be used.

The particle removal mechanism is presented in Fig. 1, included in this report as one of its parts and illustrates how the traditional and the newly invented process compare.

When the filtering medium is placed transversally to the direction of the flow and the traditional method is used (FIG. 1A), smaller particles pass through the filter pores and "soft" particles clog the filter pores or deform and pass through the filter media.

When hydrocolloids are used the particles are captured by the polymeric net and are trapped in the flakes allowing them to be intercepted by the filters (FIG. 1B).

The compositions obtained, according to the newly invented process,
act in hydro-alcoholic perfume solutions or in alcohol for removal of particles in suspension as double-effect flocculating agents, hydrocolloids such as those previously mentioned as examples as well as others that are also adequate but haven't been mentioned form a complex mesh with waxes, phytoestersols and other non-scented components that are present in the fragrance raw materials and flocculation occurs due to the intra-molecular polymeric bonds, combined with the formation of an immobilizing micro-net. As a result the particles are incorporated in the polymeric flakes and dragged to the bottom of the maturing tank and can then be removed by filtration.

The performance of the compositions produced according to the process of the present invention removing odorous molecules which interfere with the final perception of the fragrance from the scented water-alcohol solutions or from the alcohol used their formula can be explained by two possible mechanisms: the formation of charge complexes between the odorous molecules and the functional groups of the hydrocolloids or the formation of chemical derivatives between low-molecular weight odorous molecules such as acetaldehyde, present in ethyl alcohol, and functional groups present in the hydro-colloid molecules. Aldehydes such as acetaldehyde may form polymeric ketals or other derivatives with the hydroxyl groups of the hydro-colloid. Removal of the flakes formed by the insoluble hydrocolloids then leads to the removal of the odorous molecules which interfere on the perception of the notes from the perfume solution or from the alcohol. This invention then makes it unnecessary the long periods of maturing of the mixture that leads to the removal of such molecules, mainly by oxidation of aldehydes, which are traditionally utilized.

Fragrance formula compositions vary significantly between different perfumes and so does the quality of the alcohol provided by each manufacturer. Therefore the amount of the components included in the composition of the new invention required for the removal of impurities needs to be determined experimentally for each product being treated. Quantities of hydrocolloids between 0.5% and 3% in relation to the hydro-alcoholic solution have been shown to be sufficient in most cases. When the addition of two
different hydrocolloids is necessary amounts of the first component between 0.5 to 3% and of around 5% of the second component measured in relation to the first component have been shown to be sufficient in most cases.

In order to have the efficiency and benefits of the newly invented process properly evaluated some of the experiments that have been performed will be presented next as examples. These serve only to illustrate the invention and do not represent any form of limitation to the invention. Similarly, examples are presented of applications of the newly invented process.

Example 1: Increase in the efficiency of removal of suspended insoluble particles through the application of the newly invented process.

The efficiency of the process involving the utilization of the composition of the present invention was demonstrated through a series of experiments where it was possible to evaluate and compare the efficiency of the traditional method with the new process that utilizes the addition of hydrocolloids for the removal of insoluble particles that are responsible for the final turbidity in perfumes.

A perfume was produced by utilizing the traditional system by adding 10 grams of the aroma concentrate to 90 mL of a hydro-alcoholic solution with 85 % of alcohol and 15 % of water. The resulting mixture was homogenized and then filtered through a conventional filter lined with two 3μ filter paper sheets and then through two 1μ filter paper sheets. The resulting filtrate was then placed under refrigeration at 4°C in order to evaluate the possible occurrence of precipitation after the filtration.

For the perfume produced with the composition described in the new invention 10 grams of the aroma concentrate to 90 mL of a hydro-alcoholic solution with 85 % of alcohol and 15 % of water and then 2 grams of a solution of hydro-colloid obtained from red seaweeds were added to the mixture. The mixture was then homogenized and 0.1 grams of a saline solution of hydrocolloids obtained from brown seaweeds were added. The resulting mixture was homogenized and then filtered through a conventional
filter lined with two 3 µ filter paper sheets followed by filtering through two 1 µ filter paper sheets. The resulting filtrate was then placed under refrigeration at 4°C in order to evaluate the possible occurrence of precipitation after the filtration.

Turbidity of the two solutions was then analyzed through spectrophotometry by evaluating their absorbance under different times of maturing. Absorbance measurements were made with a spectrophotometer HACH mod. 4000U, at 265 nm.

Graphs shown in Figures 2A and 2B show the benefits that were reached when the process and composition of the present invention was utilized. Figure 2A (Graph 1) shows the results of the experiment obtained without the use of the composition of the invention and Figure 2B (Graph 2) shows the results of the experiment where the newly invented process was used.

Even after a month of rest under refrigeration, when the traditional method of maturing was used (Graph 1) the solution still had a turbidity of 70%. On the other hand, when the newly invented process was used (Graph 2) a significantly more efficient removal of the impurities occurred immediately after the treatment. It is also noteworthy that samples that were submitted to the conventional process still had a cloudy appearance after four months of having been manufactured.

**Example 2: Preservation of the components of the perfume after the application of the newly invented process**

The purpose of this assay was that of verifying the state of preservation of the perfume components after processing with the hydrocolloids as described in the invention.

The comparative analysis of the perfume composition prepared with the traditional process with that obtained by applying the hydro-colloid composition and the process described in the new invention was made with High Resolution Gas Chromatography. A chromatograph HP 5890 series II, equipped with a column DB-1 of 30 m, film thickness of 1 µm and internal
diameter of 0.25 mm, with Helium as carrier gas at a 50 cm/min flow rate was utilized. Analysis were performed with temperature starting at 100°C and the rate of temperature increase was of 10°C/min until reaching a maximum of 250°C, with maintenance at this temperature.

Resulting chromatograms can be seen in FIGS 3A and 3B included in this report.

As can be observed when the chromatogram resulting from the analysis of the composition of the perfume manufactured traditionally (FIG. 3A) is compared with that obtained for the perfume prepared with the hydrocolloid composition, as described in the new invention (FIG. 3B) it is clearly demonstrated that the use of the newly invented process removes the insoluble components from the hydro-alcoholic mixture but does not alter the composition of the volatile components which are essential for the perfume.

**Example 3: Efficiency of the application of the newly invented process on the perfume stability**

The purpose of this essay was checking the efficiency of the hydrocolloid composition, as described in the new invention, regarding the parameters of stability of the end products. Table I (see below) shows the results of the comparative analysis of the stability of a perfume prepared by following the traditional process and those of a perfume prepared by utilizing the hydro-colloid composition as described in the newly invented process.

Perfume stability tests were performed under different conditions and any changes in perfume properties were recorded for the time lapse since manufacturing when it occurred. Results have clearly shown that the preparation of a perfume involving the use of the newly invented process with hydrocolloids generates a transparent and stable perfume under a range storage conditions as early as 24 hours after manufacturing whereas the perfume prepared following the traditional procedure remained cloudy even after 90 days of it having been manufactured.
Example 4: Sensorial performance of the perfume after the application of the newly invented process

The objective of this essay was that of verifying the sensorial performance of a perfume prepared by using the newly invented process.
involving hydrocolloids that provoke flocculation and hence accelerate perfume maturing.

For this particular study three traditional scent families were included. These are known by perfumists as "Fresh Fougere", "Fresh Floral Fresco with Green and Woody notes" and "Floral Floral", and are regarded as representing various fine perfumes. Perfumes were prepared by using identical preparations of each of the perfume concentrates belonging to each of the three scent families. These perfumes were prepared either by following the traditional technique or by following the newly invented process. Scent attributes were evaluated by a panel including trained experts and perfumists. The results are presented below in Table II.

**TABLE (II)**

<table>
<thead>
<tr>
<th>Perfume</th>
<th>Olfactory family</th>
<th>Conventional process</th>
<th>New process</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample 1</td>
<td>Fresh Fougère</td>
<td>&quot;open&quot;</td>
<td>More harmony between the notes</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Enhanced citric note &quot;Fresh&quot;</td>
<td>No sharp notes</td>
</tr>
<tr>
<td>Sample 2</td>
<td>Fresh flower with wood &amp; green notes</td>
<td>Sharp notes</td>
<td>Matured</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Lack of &quot;harmony&quot; between the notes</td>
<td>More balanced</td>
</tr>
<tr>
<td>Sample 3</td>
<td>Flower</td>
<td>Bottom notes are ready perceived</td>
<td>More harmony between the notes</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Green notes appear</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Preserves &quot;fresh&quot; notes</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Highlights &quot;noble&quot; notes</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Galbanum, hyacinth and ylang</td>
</tr>
</tbody>
</table>

The results that were obtained demonstrate that the preparation of a perfume that follows the newly invented process yields products that have a better olfactory performance and preserves and highlights the most valuable olfactory notes giving the perfume a higher degree of harmony.

**Example 5: Sensorial performance of perfumes prepared with alcohol of different origins and degrees of purify after being subjected to the newly invented polishing process**

One of the critical aspects in the production of high quality perfumes is
the choice of the alcohol to be used in the perfume formulation. It is known that the level of purity in the alcohol that is chosen affects directly the olfactory performance of the perfume. This limitation causes many problems for the perfume manufacturers which are often restricted to a rather limited range of choices for this fundamental raw material.

This study deals with the evaluation of the influence of the treatment of alcohol with the newly invented process on the sensorial performance of perfumes prepared with alcohol of different origins and degrees of purify.

For this study two traditional scent families were chosen. These are known by the perfumers as "Fresh Fougere" and "Floral Floral", and are regarded as representative of numerous fine perfumes.

Perfumes were prepared by using identical preparations of each of the perfume concentrates belonging to each of the two scent families. These perfumes were prepared with alcohol of three different sources, named manufacturers 1, 2 and 3. Experts ranked the alcohol produced by the three manufactures in a decreasing order of sensorial quality for the production of fine perfumes, in terms of presence of interfering odorous substances. Therefore alcohol from manufacturer 1 was regarded as "premium" alcohol, alcohol from manufacturer 2 was considered as slightly inferior or "second choice" whereas the alcohol from manufacturer 3 was considered as inferior and inadequate for the production of fine perfumes. These were prepared either by following the traditional technique or by following the newly invented process. Scent attributes were evaluated by a panel including experienced evaluators and perfumers. The results are presented below in Boxes 1 and 2 below.

The results obtained for this essay clearly shows that the preparation of a perfume involving the use of the newly invented process improves the olfactory performance of the perfume removing the sharp notes that usually remains from the impurities contained in poor quality alcohol preserving the valuable olfactory notes and upgrading the perfume harmony even when such low quality alcohol are used in their preparation.
Box 1

<table>
<thead>
<tr>
<th>Eau de cologne with 10% masculine fragrance (Fougere Fresh family)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Alcohol</strong></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td>Manuf. 1</td>
</tr>
<tr>
<td>Premium</td>
</tr>
<tr>
<td>Manuf. 2</td>
</tr>
<tr>
<td>Second choice</td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td>Manuf. 3</td>
</tr>
<tr>
<td>Poor quality</td>
</tr>
</tbody>
</table>

Box 2

<table>
<thead>
<tr>
<th>Eau de cologne with 10% feminine fragrance (Floral Floral family)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Alcohol</strong></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td>Manuf. 1</td>
</tr>
<tr>
<td>Premium</td>
</tr>
<tr>
<td>Manuf. 2</td>
</tr>
<tr>
<td>Second choice</td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td>Manuf. 3</td>
</tr>
<tr>
<td>Poor quality</td>
</tr>
<tr>
<td></td>
</tr>
</tbody>
</table>

The following examples have the purpose of illustrating several possibilities of applications of the newly invented process.

**Example 6: Perfume manufacturing process based on the utilization of a flocculation system promoted by seaweed hydrocoHoïds**

The adoption of the newly invented system is rather simple and allow increasing perfume production even in smaller scale, in simpler and less expensive manufacturing plants, it reduces the need of further investing in
equipment and reduces the energy expenses for refrigeration of the solution
during production. Besides that, the process allows the manufacturer to
preserve the original composition of the perfume, to maintain and highlight
the valuable scent notes and also improves the perfume harmony. The new
process also helps the experts involved in formulation by reducing the
restrictions related to the type of alcohol that may be used in the perfume
manufacture.

The fragrance concentrate is placed in a tank where it is mixed with
water and alcohol. 20 ml of a solution of hydrocolloids obtained from red
seaweeds are added under continuous agitation for each liter of the perfume
solution as well as 1 ml of a solution of brown seaweed hydrocolloids. The
solution is left to rest at room temperature. Depending on the fragrance
the time needed for complete formation of the flakes can vary from 2 to 12 hours.
After the flake-forming process ends the raw product is filtered through a
common filter lined with 14 micra porosity filter paper. The transparent
perfume is then bottled. As an alternative, and depending on the fragrance
that is being used filter papers with porosity varying between 0.5 and 30
micra may be used. Depending on the production scale of the perfume or
colony different filtration industrial equipments may be used such as bag
filters, press filters, cartridge filters, basket filters and the like, as well as
associations of these different equipments.

**Example 7: Perfume manufacturing process based on the utilization of
a flocculation system promoted by apple pectin**

The fragrance concentrate is placed in a tank where it is mixed with
water and alcohol. To the perfume solution 30 mL of an aqueous solution of
apple pectin at 1% for each liter of the perfume solution are added under
continuous agitation. The solution is left to rest at room temperature.
Depending on the fragrance the time needed for complete formation of the
flakes can vary from 2 to 12 hours. After the flake-forming process ends the
raw product is filtered through a common filter lined with 14 micra porosity
filter paper. The transparent perfume is then bottled. As an alternative, and
depending on the fragrance that is being used filter papers with porosity varying between 0.5 and 30 micra may be used. Depending on the production scale of the perfume or colony different filtration industrial equipments may be used such as bag filters, press filters, cartridge filters, basket filters and the like, as well as associations of these different equipments.

**Example 8: Perfume manufacturing process based on the utilization of a flocculation system promoted by *Xanthomonas campestris* (xanthan gum) hydrocolloids**

The fragrance concentrate is placed in a tank where it is mixed with water and alcohol. To the perfume solution 10 mL of an aqueous solution of Xanthan Gum at 1% for each liter of the perfume solution are added under continuous agitation. The solution is left to rest at room temperature. Depending on the fragrance the time needed for complete formation of the flakes can vary from 2 to 12 hours. After the flake-forming process ends the raw product is filtered through a basket filter equipped with a filter media with a porosity of 30 microns, followed by a cartridge filter with a porosity of 1 micron, for the complete removal of the flakes that are formed. The transparent perfume is then bottled.

**Example 9: Manufacturing process of alcohol - perfume manufacturing degree - through the application of a system involving flocculation with seaweed hydrocolloids**

The use of this process on low quality alcohols allows the alcohol manufacturer or the perfume manufacturer to purify alcohol, removing impurities that occur at low quantities in alcohol such as aldehydes, but that interfere in the perception of fragrances in a perfume. Alcohol obtained through this treatment has an appropriate sensorial performance and this leads to its upgrade to perfume application degree.

Alcohol is taken to a tank where hydro-colloid solutions are added for treatment. 30 mL of a red seaweed hydro-colloid solution is added for each liter of alcohol under continuous agitation followed by 1.5 mL of brown
seaweed hydrocolloids. This mixture is left to rest at room temperature for 4 hours. After the flake-forming process ends the alcohol is filtered through a basket filter with a porosity of 25 microns for complete removal of flakes. Alcohol obtained through this process has a smoother odor and may be used in perfume manufacturing without restrictions.
CLAIMS

1. PROCESS FOR MANUFACTURING PERFUMES AND COLOGNES utilizing natural hydrocolloids characterized by such hydrocolloids being incorporated to hydro-alcoholic solutions as a clarification agent and for improvement of the sensorial features of such hydro-alcoholic solutions.

2. PROCESS FOR MANUFACTURING PERFUMES AND COLOGNES as in claim 1, characterized by such hydrocolloids being obtained from animal, plant or microbial sources.

3. PROCESS FOR MANUFACTURING PERFUMES AND COLOGNES as in claim 1, characterized by such hydrocolloids being obtained from seaweeds.

4. PROCESS FOR MANUFACTURING PERFUMES AND COLOGNES as in claim 3, characterized by such seaweeds being selected from red and brown algae.

5. PROCESS FOR MANUFACTURING PERFUMES AND COLOGNES as in claim 2, characterized by such hydrocolloids being obtained from Xanthomonas campestris.

6. PROCESS FOR MANUFACTURING PERFUMES AND COLOGNES as in claim 2, characterized by such hydrocolloids being obtained from fruits, fruit parts and seeds.

7. PROCESS FOR MANUFACTURING PERFUMES AND COLOGNES as in claim 1, characterized by pectin from fruits, particularly from apple being utilized as flocculating agents.

8. PROCESS FOR MANUFACTURING PERFUMES AND COLOGNES as in claim 1, characterized by chitosan obtained from animals, chitosan from crustacean sources in particular, being used as a flocculating agent.

9. PROCESS FOR MANUFACTURING PERFUMES AND COLOGNES as in claim 1, characterized by guar gum being used as flocculating agent.

10. PROCESS FOR MANUFACTURING PERFUMES AND COLOGNES as in claim 1, characterized by xanthan gum being used as flocculating agent.

II. PROCESS FOR MANUFACTURING PERFUMES AND COLOGNES
as in claim 1, characterized by such hydrocolloids added to hydro-alcoholic mixtures leading to the removal of insoluble components from the hydro-alcoholic mixture without altering its composition in terms of volatile components of importance for the perfume.

12. PROCESS FOR MANUFACTURING PERFUMES AND COLOGNES as in claim 1, characterized by the addition of such hydrocolloids yielding products which are clearer and more stable under storage.

13. PROCESS FOR MANUFACTURING PERFUMES AND COLOGNES as in claim 1, characterized by the addition of such hydrocolloids resulting in products that have a better olfactory performance, preserving and highlighting the valuable scent notes and providing a higher level of harmony to the perfumes.

14. PROCESS FOR PURIFYING ALCOHOL characterized by the use of natural hydrocolloids obtained from animal, plant or microbial sources that act as flocculating agents and lead to an improvement of sensorial characteristics and by such hydrocolloids when added to poor quality alcohol allowing for an upgrade of its properties to a standard that is adequate for use in perfume manufacturing.
INTERNATIONAL SEARCH REPORT

International application Nº
PCT/BR2009/000437

A. CLASSIFICATION OF SUBJECT MATTER

IPC (2010.01) A61K8/73 A61K8/97 A61K8/98 A61K8/99 A61Q13/00 B01D21/01

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

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC (2010.01) A61K A61Q B01D

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

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

EPODOC, MEDLINE, SCIELO, BIOSYS, CHEMICAL ABSTRACTS, EMBASE, ELSEVIER BIOBASE, SCISEARCH

C. DOCUMENTS CONSIDERED TO BE RELEVANT

<table>
<thead>
<tr>
<th>Category</th>
<th>Citation of document, with indication, where appropriate, of the relevant passages</th>
<th>Relevant to claims Nº</th>
</tr>
</thead>
<tbody>
<tr>
<td>X</td>
<td>BR 9700752 A (BARRETO DAMIEL WEINGART [BR])</td>
<td>1-3, 12, 14</td>
</tr>
<tr>
<td></td>
<td>08 December 1998 (1998-12-08)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>See whole document.</td>
<td>4-11, 33</td>
</tr>
<tr>
<td></td>
<td>See whole document.</td>
<td></td>
</tr>
<tr>
<td>Y</td>
<td>FR 2829025 A1 (OREAL[FR])</td>
<td>Claims 1-7, 10-14</td>
</tr>
<tr>
<td></td>
<td>07 March 2003 (2003-03-07)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>See p.5, 1:0-p.6, 1.21.</td>
<td></td>
</tr>
<tr>
<td>Y</td>
<td>ES 2164566 A1 (LIPOTEC SA [ES])</td>
<td>Claims 1-14</td>
</tr>
<tr>
<td></td>
<td>16 February 2002 (2002-02-16)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>See col.2, 1.42-52.</td>
<td></td>
</tr>
</tbody>
</table>

☐ Further documents are listed in the continuation of Box C. □ Sec. Patent family annex.

☐ Special categories of cited documents:
- "A" document defining 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 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
26 March 2010

Date of mailing of the international search report
3 / March 2010

Name and mailing address of the ISA/BR

INSTITUTO NACIONAL DA PROPRIEDADE INDUSTRIAL
Rua Mayrink Veiga n°5, 18º andar
cep: 20090-050, Centro - Bis de Jabaquara/RJ
+55 21 2139-3663

Facsimile Nº:

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

Authorized officer

Mauricio da Silva Martins Almeida
Telephone Nº: +55 21 2139-3663/3742
<table>
<thead>
<tr>
<th>Category</th>
<th>Citation of document, with indication, where appropriate, of the relevant passages</th>
<th>Relevant to claims No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Patent documents cited in search report</td>
<td>Publication date</td>
<td>Patent family members</td>
</tr>
<tr>
<td>----------------------------------------</td>
<td>-----------------</td>
<td>-----------------------</td>
</tr>
<tr>
<td>BR 9700752 A</td>
<td>1998-12-08</td>
<td>None</td>
</tr>
<tr>
<td>FR 2829025 A1</td>
<td>2003-03-07</td>
<td>None</td>
</tr>
<tr>
<td>ES 2164566 A1</td>
<td>2002-02-16</td>
<td>ES 2164566 B1</td>
</tr>
<tr>
<td></td>
<td></td>
<td>JP 1500443 T</td>
</tr>
<tr>
<td></td>
<td></td>
<td>US 4851393 A</td>
</tr>
<tr>
<td></td>
<td></td>
<td>WO 8800952 A1</td>
</tr>
</tbody>
</table>

Form PCT/ISA/210 (patent family annex) (July 2009)