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(71) Applicant: **LUCCIA B Private Company**
54622 Thessaloniki (GR)

(72) Inventors:
• **CHRISTOPOULOS, Miltiadis**
14123 Lykovrysi (GR)

- **MARKOU, George**
14123 Lykovrysi (GR)
- **PLAKANTONAKI, Sofia**
12241 Athens (GR)
- **PRINIOTAKIS, Georgios**
12241 Athens (GR)
- **PETROU, Argyrios**
Thessaloniki (GR)

(74) Representative: **Kouzelis, Dimitrios**
Intel-Lex
19, Loukianou Str.
10675 Athens (GR)

(54) **METHOD FOR GENERATING CELLULOSE PULP**

(57) The invention concerns a method (100) for generating cellulose pulp. The method (100) according to the invention comprises mixing (101) an organic material that is obtained from peaches with an aqueous solution for creating a first suspension; filtering (102) the first suspension to extract first solids from the first suspension; mixing (103) the first solids with an organic solvent for creating a second suspension; filtering (104) the second suspension to extract second solids from the second suspension; applying (105) an aqueous bleaching solution to the second solids to for creating a third suspension; and filtering (106) the third suspension to extract the cellulose pulp from the third suspension.

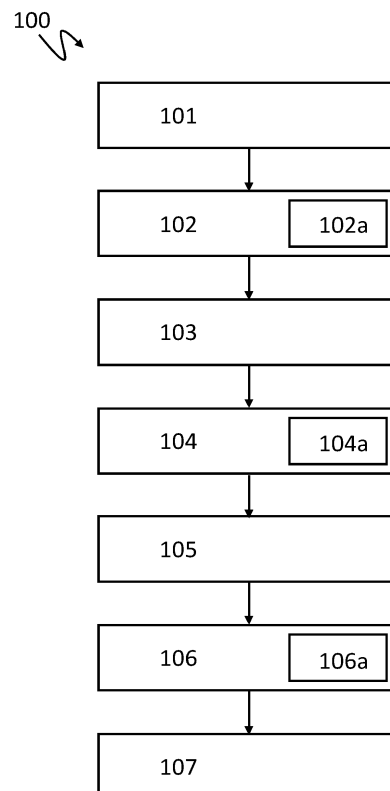


Fig.1

Description

Field of the invention

[0001] The invention relates to the field of generating cellulose pulp.

Background of the Invention

[0002] The needs of the modern textile industry in raw materials cannot be met with natural fibers, since the production cycle is given, the reserves are limited and it is necessary to commit significant cultivated areas to industrial crops such as cotton. Synthetic fibers cover the additional needs and take the largest market share but are petrochemical products, derived from non-renewable raw materials.

[0003] Man-made cellulosic fibers from regenerated cellulose are another option with increasing market share. Cellulosic fibers are currently produced mainly from wood while less than 1% is produced from recycled or other alternative raw materials. The use of agricultural crops instead of wood has the advantage of reducing deforestation, while the utilization of food industry waste makes it possible to recycle it creating products of high added value.

Summary of the Invention

[0004] A method for generating cellulose pulp according to the invention comprises mixing an organic material that is obtained from peaches with an aqueous solution for creating a first suspension, wherein the aqueous solution is an alkaline solution, filtering the first suspension to extract first solids from the first suspension, mixing the first solids with an organic solvent for creating a second suspension, filtering the second suspension to extract second solids from the second suspension, applying an aqueous bleaching solution to the second solids to for creating a third suspension, and filtering the third suspension to extract the cellulose pulp from the third suspension.

[0005] The method comprises mixing the organic material that is obtained from peaches with an aqueous solution for creating the first suspension, wherein the aqueous solution is an alkaline solution.

[0006] The organic material that is obtained from peaches is preferably peach residues from the food industry. In particular, the organic material is dried pitted peach waste. The organic material that is obtained from peaches preferably is peach residues without peach stones. Optionally, the method comprises a removal of cores from peaches and an extraction of juice from the peaches to create the organic material.

[0007] Adding the aqueous solution to the organic material supports the removal of hemicelluloses from the waste material. The mechanism can be divided in two successively stages. That is, a physical interaction

between cellulose and aqueous sodium hydroxide results in fibers of the organic material swelling. Further than that, this leads to a diffusion of hemicelluloses from the fibers interior to exterior via the swollen pores of the in the fiber wall of the fibers and their transfer to the aqueous solution.

[0008] Mixing the organic material that is obtained from peaches with an aqueous solution is an alkali treatment. During the alkali treatment cellulose undergoes a peeling reaction that reduces its degree of polymerization and results in a low pulp strength. It is an endwise degradation process in which the reducing end group is cut off from the cellulose chain, producing soluble degradation products such as isosaccharinic acid. The use of moderate temperatures and alkali concentrations mitigates this phenomenon.

[0009] The method comprises the filtering of the first suspension to extract the first solids from the first suspension. With the filtering, the aqueous solution is removed from the process.

[0010] The method comprises the mixing of the first solids with an organic solvent for creating a second suspension. The first solid that has undergone the swelling procedure when the aqueous solution was added. With applying the organic solvent the process is more susceptible to further purification to remove unwanted substances. The use of the organic solvent demonstrated the ability to remove lignin and hemicelluloses under mild conditions, without significant cellulose degradation. The rate of delignification in acidic conditions is controlled by α -ether cleavage, while the likelihood of β -ether cleavage increases in more strongly acidic systems. Cellulose degradation is the result of acid hydrolysis, where H^+ ions permeate into the cellulose molecules, attach to β -1,4-glycosidic bonds, and thus shorten cellulose chains.

[0011] The method comprises filtering the second suspension to extract the second solids from the second suspension. With the filtering, the organic solvent is removed from the process.

[0012] The method comprises applying the aqueous bleaching solution to the second solids to for creating a third suspension. Treatment with the aqueous bleaching solution is performed to further promote the delignification of the pulp. Also, hemicelluloses that is produced because of the cellulose decomposition in the previous steps of the procedure is removed.

[0013] The method comprises filtering the third suspension to extract the cellulose pulp from the third suspension. Preferably, the cellulose pulp is extracted from the third suspension by filtering the third suspension.

[0014] The method according to the invention has the advantage that it allows using peach residues directly from the food industry and specifically canning and juice factories with the only pre-treatment being drying - a step that can be omitted since it is carried out to facilitate the transport of the raw material.

[0015] In this way, a circular economy is achieved by

utilizing waste mainly from the peach industry and turning it into raw material with high added value. It takes 10,000 liters of water to produce 1 kilo of cotton, meaning it takes about 2,700 liters to make 1 cotton t-shirt. In the present invention, the use of waste streams suggests that there is no water footprint for a cultivation stage.

[0016] The method allows to valorize peach waste and to turn the peach waste into valuable goods. This is achieved by an eco-friendly processes. In a climate changing environment, the shift to sustainable production is becoming a necessity. The increased activity in the modern agricultural sector produces plenty of wastes, the handling of which is putting more pressure to the public waste management systems. Agricultural wastes, such as peach wastes, are annually renewable and a low-cost source for natural cellulosic fibres. As there is a declining supply of natural raw materials in the textile industry, which makes the utilization of agricultural waste fibre an attractive option to make use of the tremendous wealth of natural plant fibre which is currently discarded. Cellulosic crops such as cotton take up space from food because of their higher monetary value. Therefore, fashion that is created using the method according to the invention may be considered to fuel food shortages.

[0017] The dependent claims define advantageous embodiments of the invention.

[0018] Preferably, the aqueous solution contains 2% to 3% w/v sodium hydroxide. The addition of sodium hydroxide supports the removal of hemicelluloses from the organic material.

[0019] Preferably, the mixing of the organic material with the aqueous solution and/or the mixing of the first solids with the organic solvent is performed at a temperature in a range between 75°C and 85°C. This improves a pulp strength of the cellulose pulp.

[0020] Preferably, the mixing of the organic material with the aqueous solution and/or the mixing of the first solids with the organic solvent is performed under continuous mechanical stirring. This leads to a more homogeneous structure of the cellulose pulp.

[0021] Preferably, the mixing of the organic material with the aqueous solution and/or the mixing of the first solids with the organic solvent is performed for at least 60 minutes, preferably for at least 90 minutes. This ensures that an optimal removal of hemicelluloses from the organic material is achieved.

[0022] Preferably, a first washing process is applied to the first solids that are extracted from the first suspension, wherein the first washing process preferably comprises rinsing the first solids with water. This ensures that the aqueous solution is removed from the first solids.

[0023] Preferably, the organic solvent is preferably a combination of formic acid 85% v/v and acetic acid 80% v/v added in equal proportions. Preferably, a ratio of the volumes between the first solids and the organic solvent is in a range between 1:6 to 1:12. This avoids that the fibers of the cellulose pulp are damaged while ensuring that all unwanted substances are removed from the first

solids.

[0024] Preferably, a second washing process is applied to the second solids that are extracted from the second suspension, wherein the second washing process preferably comprises rinsing the second solids with water. This ensures that the organic solvent is removed from the second solids.

[0025] Preferably, the aqueous bleaching solution is a hydrogen peroxide aqueous solution. In basic solutions, hydrogen peroxide is a strong reductant and oxygen gas is also produced. Sodium hydroxide acts as the solubilizing agent for extracting modified lignin fragments while hydrogen peroxide and oxygen -that is produced from the decomposition hydrogen peroxide- attack and fragment the residual lignin. Hemicelluloses produced because of the cellulose decomposition in the previous steps of the procedure can be removed through the same mechanism. Hydrogen peroxide bleaching is preferably performed together with an alkali source to produce the active perhydroxyl anion (HOO^-) and the perhydroxyl free radical that have been suggested as the intermediates in the brightening reactions for cellulose based product. Hydrogen peroxide decomposes to form water and oxygen and as a result no residues of the bleaching agent nor organochlorine by-products are found in the final product.

[0026] Preferably, twenty volumes of hydrogen peroxide aqueous solution are applied to one volume of the second solids when applying a hydrogen peroxide aqueous solution to the second solids, wherein the hydrogen peroxide aqueous solution has a concentration of 0.5-3.5% v/v. This avoids that the fibers of the cellulose pulp are damaged while ensuring that all unwanted substances are removed from the first solids.

[0027] Preferably, the hydrogen peroxide aqueous solution contains sodium hydroxide and has a pH level between ten and twelve.

[0028] Preferably, a third washing process is applied to the cellulose pulp that is extracted from the third suspension, wherein the third washing process preferably comprises rinsing the cellulose pulp with water. This ensures that the aqueous bleaching solution is removed from the second solids.

[0029] Preferably, the method comprises drying the cellulose pulp.

[0030] A method for producing a textile according to the invention comprises generating cellulose pulp using the method for generating cellulose pulp according to the invention and preferably comprises mixing the cellulose pulp with wood or cotton based cellulose pulp. The step of mixing the cellulose pulp with wood or cotton based cellulose pulp leads to a strengthening of the structure of the textile.

Brief description of the drawings

[0031]

Figure 1 shows a flowchart of a method according to the invention.

Detailed description

[0032] Figure 1 shows a flowchart of a method 100 for generating cellulose pulp according to an embodiment of the invention.

[0033] The target of the method 100 is to extract cellulose content present in unpitted peach waste from their native micro-environment in the form of pulp fiber. This is achieved by a treatment with hot aqueous solutions that results in the removal of soluble ingredients including sugars, phenolic components and soluble polysaccharides such as pectin.

[0034] In a first step 101, an organic material that is obtained from peaches is mixed with an aqueous solution for creating a first suspension, wherein the aqueous solution is an alkaline solution. The organic material is dried pitted peach waste. The aqueous solution contains 2% to 3% w/v sodium hydroxide.

[0035] The dried pitted peach waste is mixed with the aqueous solution containing 2-3% w/v NaOH, that is sodium hydroxide, within an appropriate stainless-steel container which is heated at a temperature of $80^{\circ}\text{C} \pm 5^{\circ}\text{C}$ under continuous mechanical stirring for 90 minutes. Thus, the mixing of the organic material with the aqueous solution in the first step 101 is performed at a temperature in a range between 75°C and 85°C and is performed under continuous mechanical stirring, wherein the mixing is performed for at least 60 minutes, here for 90 minutes.

[0036] In a first example, 200g of dried peach wastes from juice industry are used as organic material. The mixing is performed with 2.5%w/v NaOH with a solid (peach waste) to solvent ratio of 1:20 and the mixing is performed in a first bath at $80^{\circ}\text{C} \pm 5^{\circ}\text{C}$ for 90 minutes.

[0037] In a second step 102, which is performed after the first step 101, the first suspension is filtered to extract first solids from the first suspension. The second step 102 further comprises a first washing process 102a. The first washing process 102a is applied to the first solids that are extracted from the first suspension, wherein the first washing process comprises rinsing the first solids with water. Therefore, the first suspension is filtrated and the resulting first solids are washed with mains water to remove all remaining NaOH. The filtration is preferably done via the use of a series of sieves of progressively smaller mesh size with mesh openings from 1.6 mm up to $36\mu\text{m}$. Alternatively, other methods can be used such as bag filters, filter press and belt filter press. In this embodiment, the first solids are washed & filtered multiple times, preferably three times.

[0038] In a third step 103, which is performed after the second step 102, the first solids are mixed with an organic solvent for creating a second suspension. The organic solvent is a combination of formic acid 85% v/v and acetic acid 80% v/v added in equal proportions. That is, the same volume of acid 85% v/v and acetic acid 80% v/v is

combined to create the organic solvent.

[0039] The first solids are treated with the organic solvent that is a combination of formic acid 85% v/v and acetic acid 80% v/v added in equal proportions.

5 The second suspension is heated at a temperature of $80^{\circ}\text{C} \pm 5^{\circ}\text{C}$ under continuous mechanical stirring for 90min. The first solid (peach waste) to organic solvent ratio is preferably in the range from 1:6 to 1:12. Consequently, the organic solvent is a combination of formic acid 85% v/v and acetic acid 80% v/v added in equal proportions, wherein the ratio of the volumes or the weight between of first solids and of organic solvent is in a range between 1:6 to 1:12.

10 **[0040]** The first solids are mixed with the organic solvent within an appropriate stainless-steel container which is heated at the temperature of $80^{\circ}\text{C} \pm 5^{\circ}\text{C}$ under continuous mechanical stirring for 90 minutes. Thus, the mixing of the first solids with the organic solvent in the third step 103 is performed at a temperature in a range between 75°C and 85°C and is performed under continuous mechanical stirring, wherein the mixing is performed for at least 60 minutes, here for 90 minutes.

20 **[0041]** In the first example, the mixing is performed with a ratio of formic acid 85% v/v to acetic acid 80%v/v of 1:1, wherein a solid to solvent ratio is 1:6 and the mixing is performed in a second bath at $80^{\circ}\text{C} \pm 5^{\circ}\text{C}$ for 90 minutes.

25 **[0042]** In a fourth step 104, which is performed after the second step 103, the second suspension is filtered to extract second solids from the second suspension. The fourth step 104 further comprises a second washing process 104a. The second washing process 104a is applied to the second solids that are extracted from the suspension, wherein the second washing process 104a comprises rinsing the second solids with water. Therefore, the second suspension is filtrated and the resulting second solids are washed with mains water to remove all remaining organic solvent. The filtration is preferably done via the use of a series of sieves of progressively smaller mesh size with mesh openings from 1.6mm up to $36\mu\text{m}$. Alternatively, other methods can be used such as bag filters, filter press and belt filter press. In this embodiment, second first solids are washed & filtered multiple times, preferably three times. The second suspension is filtrated and the resulting second solids are washed with mains water until all acid is removed.

30 **[0043]** In a fifth step 105, which is performed after the fourth step 104, an aqueous bleaching solution is applied to the second solids to for creating a third suspension. The aqueous bleaching solution is a hydrogen peroxide aqueous solution. Twenty volumes of the hydrogen peroxide aqueous solution are applied to one volume of the second solids when applying the hydrogen peroxide aqueous solution to the second solids, wherein the hydrogen peroxide aqueous solution has a concentration of 0.5-3.5% v/v. The hydrogen peroxide aqueous solution contains sodium hydroxide and has a pH level between 10 and 12. With this, the second solids are further purified and bleached with 20 volumes of hydrogen peroxide

aqueous solution at concentrations between 0.5-3.5%v/v containing NaOH to adjust pH levels between 10 and 12.

[0044] It is noted that alternative agents can be used as aqueous bleaching solution. Exemplary agents are, sodium hypochlorite NaOCl, which can be used at room temperature, sodium chlorite (NaClO₂), which can be used at 80°C ± 5°C, and ozone. This list is not exhaustive. Enzymatic treatment with cellulase is also possible according to the literature. Hydrogen peroxide has the advantage of not leaving residual concentrations, but it is susceptible to the presence of transition metals, pH level and high temperature.

[0045] In the first example, 0.5% v/v H₂O₂ containing NaOH 4g/L is applied with a solid to solvent ratio of 1:20 in a third bath at 80°C ± 5°C for 90 minutes.

[0046] In a sixth step 106, which is performed after the fifth step 105, the third suspension is filtered to extract the cellulose pulp from the third suspension. The sixth step 106 further comprises a third washing process 106a. The third washing process 106a is applied to the cellulose pulp that is extracted from the third suspension, wherein the third washing process 106a comprises rinsing the cellulose pulp with water. Therefore, the third suspension is filtrated and the resulting cellulose pulp is washed with mains water to remove all remaining aqueous bleaching solution. The filtration is preferably done via the use of a series of sieves of progressively smaller mesh size with mesh openings from 1.6mm up to 36µm. Alternatively, other methods can be used such as bag filters, filter press and belt filter press. In this embodiment, the cellulose pulp is washed & filtered multiple times, preferably three times. The cellulose pulp is filtrated and the resulting cellulose pulp is washed with mains water until all aqueous bleaching solution is removed.

[0047] In a seventh step 107, which is performed after the sixth step 103, the washed cellulose pulp is dried. Preferably, the cellulose pulp is oven dried at 60°C until it reaches a stable weight, preferably over a time of 48hours.

[0048] The method 100 results in a cellulosic pulp with α-cellulose content greater than 92% and Degree of polymerization over 510. The overall yield of the process starting with the pitted peach waste is over 11%. With each step in which a filtering is performed, large foreign matters and precipitates, such as peach cores that were not extracted during the industrial peach processing process, are removed.

[0049] In the first example, the cellulose pulp is oven dried at 60°C for 48h, until it reaches a stable weight. The method leads to a yield of 11.6%, DP 567 and an α-cellulose content of the cellulose pulp of 94.9%.

[0050] The cellulose pulp can be used for the production of textiles, preferably mixed with dissolving pulp from conventional plant-based sources such as wood and cotton linter. This can be done by using a method for producing a textile that comprises generating cellulose pulp using the method 100 for generating the cellulose pulp and mixing the cellulose pulp with wood or cotton

based cellulose pulp.

Claims

1. Method (100) for generating cellulose pulp, the method comprising:

mixing (101) an organic material that is obtained from peaches with an aqueous solution for creating a first suspension, wherein the aqueous solution is an alkaline solution;
filtering (102) the first suspension to extract first solids from the first suspension;
mixing (103) the first solids with an organic solvent for creating a second suspension;
filtering (104) the second suspension to extract second solids from the second suspension;
applying (105) an aqueous bleaching solution to the second solids to for creating a third suspension; and
filtering (106) the third suspension to extract the cellulose pulp from the third suspension.

2. Method (100) according to claim 1, wherein the aqueous solution contains 2% to 3% w/v sodium hydroxide.

3. Method (100) according to any one of the previous claims, wherein the mixing (101) of the organic material with the aqueous solution and/or the mixing (103) of the first solids with the organic solvent is performed at a temperature in a range between 75°C and 85°C.

4. Method (100) according to any one of the previous claims, wherein the mixing (101) of the organic material with the aqueous solution and/or the mixing (103) of the first solids with the organic solvent is performed under continuous mechanical stirring.

5. Method (100) according to any one of the previous claims, wherein the mixing (101) of the organic material with the aqueous solution and/or the mixing (103) of the first solids with the organic solvent is performed for at least 60 minutes, preferably for at least 90 minutes.

6. Method (100) according to any one of the previous claims, wherein a first washing process (102a) is applied to the first solids that are extracted from the first suspension, wherein the first washing process preferably comprises rinsing the first solids with water.

7. Method (100) according to any one of the previous claims, wherein the organic solvent is preferably a combination of formic acid 85% v/v and acetic acid

80% v/v added in equal proportions.

8. Method (100) according to any one of the previous claims, wherein a ratio of the volumes between the first solids and the organic solvent is in a range between 1:6 to 1:12. 5

9. Method (100) according to any one of the previous claims, wherein a second washing (104a) process is applied to the second solids that are extracted from the second suspension, wherein the second washing process preferably comprises rinsing the second solids with water. 10

10. Method (100) according to any one of the previous claims, wherein the aqueous bleaching solution is a hydrogen peroxide aqueous solution. 15

11. Method (100) according to claim 10, wherein twenty volumes of hydrogen peroxide aqueous solution are applied to one volume of the second solids when applying the hydrogen peroxide aqueous solution to the second solids, wherein the hydrogen peroxide aqueous solution has a concentration of 0.5-3.5% v/v. 20
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12. Method (100) according to any one of claim 10 and 11, wherein the hydrogen peroxide aqueous solution contains sodium hydroxide and has a pH level between 10 and 12. 30

13. Method (100) according to any one of the previous claims, wherein a third washing process (106a) is applied to the cellulose pulp that is extracted from the third suspension, wherein the third washing process preferably comprises rinsing the cellulose pulp with water. 35

14. Method (100) according to any one of the previous claims, wherein the method comprises drying the cellulose pulp. 40

15. Method for producing a textile, the method comprising: 45
 generating cellulose pulp using the method (100) according to any one of the previous claims; and
 mixing the cellulose pulp with wood or cotton based cellulose pulp. 50
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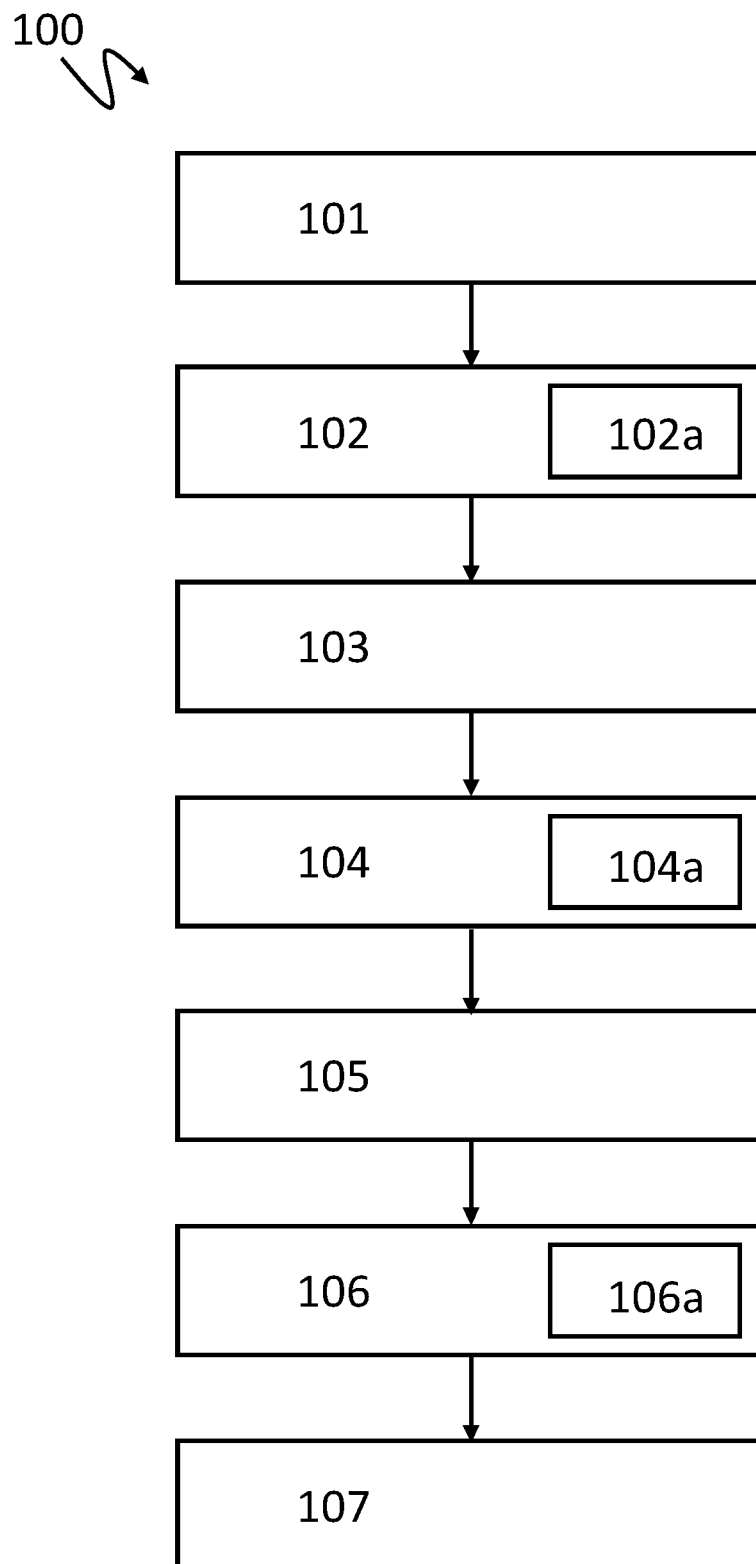


Fig.1



EUROPEAN SEARCH REPORT

Application Number

EP 23 20 3057

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The present search report has been drawn up for all claims			
Place of search		Date of completion of the search	Examiner
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