(γ2) feeding said water stream

into a distillation column configured to bring into contact said liquid stream with said aqueous stream;

(δ) discharging from the device water comprising aniline or aniline and N-methylaniline; and

(ε) discharging from the device aqueous an aniline-free or aniline-free and N-methylaniline-free leucoindigo solution.

14. Method of item 12 or 13, wherein said (γ1) liquid stream and said (γ2) water stream are fed into the column via inlets in the sidewalls of the column, and said (δ) water comprising aniline or aniline and N-methylaniline is discharged at the top of the column, and the (ε) aqueous aniline-free or aniline-free and N-methylaniline-free leucoindigo solution is discharged at the bottom of the column.

15. Method of any one of claims 13 or 14, wherein the distillation column is a column provided with trays or packing materials.

16. In yet another aspect, the present disclosure refers to stable aqueous leucoindigo solution comprising an aromatic amine in the form of aniline or aniline and N-methylaniline, wherein said leucoindigo is in the form of an alkali metal salt;

wherein the concentration of the aromatic amine is below 40 ppm determined according to ISO 14362-1:2017(E); and

wherein the concentration of the leucoindigo salt is in a concentration range of from 25 to 45 % by weight based on the total weight of the solution, and wherein the stability of the solution is measured at a temperature of 23 °C; or

CLAIMS

1. Method of removing an aromatic amine in the form of aniline or aniline and N-methylaniline from an aqueous leucoindigo solution comprising said aromatic amine in order to obtain a purified leucoindigo solution, wherein said leucoindigo is in the form of an alkali metal salt, the method comprising step (A1):

   (A1) subjecting the aqueous leucoindigo solution to distillation;

   wherein step (A1) further comprises one of the following steps (B1), (C1) or (D1):

   (B1) adding water to the aqueous leucoindigo solution prior to the distillation according to step (A1);

   (C1) adding water to the aqueous leucoindigo solution simultaneously to the distillation according to step (A1);

   (D1) adding water to the aqueous leucoindigo solution after the distillation according to step (A1) and redistilling the obtained solution;

   wherein water is distilled off in a weight which at least equals the weight of said aqueous leucoindigo solution used in step (A1).

2. Method of making an aqueous aniline-free or aniline-free and N-methylaniline-free leucoindigo solution from an aqueous leucoindigo solution comprising aniline or aniline and N-methylaniline, the concentration of aniline or aniline and N-methylaniline being determined according to ISO 14362-1:2017(E), wherein said leucoindigo is in the form of an alkali metal salt, the method comprising step (A1):

   (A1) subjecting the aqueous leucoindigo solution to distillation;

wherein step (A1) further comprises one of the following steps (B1), (C1) or (D1):

(B1) adding water to the aqueous leucoindigo solution prior to the distillation according to step (A1);

(C1) adding water to the aqueous leucoindigo solution simultaneously to the distillation according to step (A1);

(D1) adding water to the aqueous leucoindigo solution after the distillation according to step (A1) and redistilling the obtained solution;

wherein water is distilled off in a weight which at least equals the weight of said aqueous leucoindigo solution used in step (A1).

3. Method of claim 1 or 2, wherein step (A1) further comprises step (A2):

(A2) subjecting the aqueous leucoindigo solution obtained in step (A1) to distillation;

wherein step (A2) further comprises one of the following steps (B2), (C2) or (D2):

(B2) adding water to the aqueous leucoindigo solution prior to the distillation according to step (A2);

(C2) adding water to the aqueous leucoindigo solution simultaneously to the distillation according to step (A2);

(D2) adding water to the aqueous leucoindigo solution after the distillation according to step (A2) and redistilling the obtained solution.

4. Method of any one of claims 1 to 3, wherein water is added in a weight sufficient to allow distilling off water in a weight which at least is one and half times the weight of said aqueous leucoindigo solution used in step (A1).

5. Method of any one of the preceding claims, wherein water is added in a weight sufficient to allow distilling off water in a weight which at least is twice the weight of said aqueous leucoindigo solution used in step (A1).

6. Method of any one of the preceding claims, wherein distillation is performed under a flow of inert gas.

7. Method of any one of the preceding claims, wherein the concentration of the leucoindigo salt in the solution used in step (A1) is in the range of from 5 to 65 % by weight, based on the total weight of the solution.

8. Method of any one of the preceding claims, wherein the concentration of said aromatic amine in the solution used in step (A1) is in the range of from 2,000 ppm to 10,000 ppm, based on the total weight of the solution used in step (A).

9. Method of any one of the preceding claims, wherein the concentration of aniline in the solution used in step (A1) is in the range of from 1,000 ppm to 3,000 ppm and the concentration of N-methylaniline is in the range of from 500 to 2,000 ppm.

10. Method of any one of the preceding claims, wherein the weight of the added water and the water distilled off is selected such that the concentration of the leucoindigo salt in the purified solution is in the range of from 45 to 65 % by weight, based on the total weight of the solution.

11. Method of claim 10, wherein the concentration is adjusted to a range of from 10 to 45 % by weight, if necessary by addition of water.

12. Method of any one of the preceding claims, wherein any one of steps (B1), (C1) and (D1), or (B1), (C1) and (D1) and (B2), (C2), and (D2) comprises:

(α) providing a liquid stream comprising said aqueous leucoindigo solution comprising said amine(s);

(β) providing a water stream;

(γ) bringing into contact said liquid stream with said water stream.

13. Method of claim 12, wherein step (γ) comprises steps (γ1) and (γ2), and step (A1) or steps (A1) and(A2) comprises/comprise steps (δ) and (ε):

(γ1) feeding said liquid stream and

(γ2) feeding said water stream

into a distillation column configured to bring into contact said liquid stream with said aqueous stream;

(δ) discharging from the device water comprising aniline or aniline and N-methylaniline; and

(ε) discharging from the device aqueous an aniline-free or aniline-free and N-methylaniline-free leucoindigo solution.

14. Method of claim 12 or 13, wherein said (γ1) liquid stream and said (γ2) water stream are fed into the column via inlets in the sidewalls of the column, and said (δ) water comprising aniline or aniline and N-methylaniline is discharged at the top of the column, and the (ε) aqueous aniline-free or aniline-free and N-methylaniline-free leucoindigo solution is discharged at the bottom of the column.

15. Method of any one of claims 13 or 14, wherein the distillation column is a column provided with trays or packing materials.

16. Stable aqueous leucoindigo solution comprising an aromatic amine in the form of aniline or aniline and N-methylaniline, wherein said leucoindigo is in the form of an alkali metal salt;

wherein the concentration of the aromatic amine is below 40 ppm determined according to ISO 14362-1:2017(E); and

wherein the concentration of the leucoindigo salt is in a concentration range of from ~~10~~ 25 to 45 % by weight based on the total weight of the solution, and wherein the stability of the solution is measured at a temperature of 23 °C; or

wherein the concentration of the leucoindigo salt is in a concentration range of from 45 to 65 % by weight based on the total weight of the solution, and wherein the stability of the solution is measured at a temperature of 60 °C;

wherein the salt is in the form of a mixed sodium and potassium salt, wherein sodium and potassium are present in a molar ratio of from 2.33 : 1 to 1 : 2.33 or from above 3 : 1 to 10 : 1.

17. Stable aqueous leucoindigo solution of claim 16, wherein the concentration of the aromatic amine is below 30 ppm or 20 ppm or 5 ppm.

18. Method of making indigo, comprising step (D):

(D) oxidizing the aqueous leucoindigo solution as defined in any one of claims 16 to 17.

19. Method of claim 18, comprising step (C) prior to step (D):

(C) treating a textile with the leucoindigo solution as defined in any one of claims 16 to 17.

20. Method of making indigo, comprising steps (I) and (III):

(I) performing a method as defined in any one of claims 1 to 15, preferably claims 10 or 11 or any one of claims 12 to 15 as far as depending on claim 10 or 11;

(III) oxidizing the leucoindigo solution obtained in step (I).

21. Method of claim 20, comprising step (II) prior to step (III):

(II) treating a textile with the leucoindigo solution obtained in step (I).