

Description

This invention relates to a method for producing stabilized solutions of formaldehyde with methanol.

Such solutions are commonly used in reactions of formaldehyde with other products, in which it is of interest to have less water than if a conventional formaldehyde-in-water solution were used and where, furthermore, the presence of methanol is desired, for example, to act as solvent for the end product obtained.

US Patent 3.629.997 relates to a process for producing methanol-formaldehyde solution of low-water content. Part of the gaseous stream exiting from the catalyzer of a formaldehyde plant is diverted to concentrator-absorber and is scrubbed with the solution obtained in the main concentrator-absorber of the methanol formaldehyde plant. The maintenance of a particular temperature at the head of the concentrator-absorber allows a solution to be obtained at the foot of said concentrator-absorber containing 60-75% of formaldehyde, 15-25% of methanol and 5-15% of water. Nevertheless, this process always requires a distillation step and involves dilution of the formaldehyde solution obtained in the formaldehyde plant, unless the scrubbing water is reduced; but in this case adequate scrubbing of the gases is not obtained and methanol and formaldehyde are lost in them. On the other hand, the proportion of the aqueous-formaldehyde solution that is introduced into the concentrator-absorber to the concentrated formaldehyde recirculating solution entering the concentrator-absorber is in the range of 1:20 to 1:100, i.e. very high and, furthermore, a high temperature of over 85° has to be maintained. All of this leads to an increase in the energy cost.

Spanish patent 447,890 discloses a process for the preparation of urea formaldehyde glues and resins, based on the preparation of said glues and or resins from methanol, by preparing a concentrated aqueous formaldehyde solution which is reacted with urea.

The process described in the above patent is characterised by the combination of the following steps:

- a) methanol is catalytically oxidised in air;
- b) the gases from the catalytic oxidation of the methanol are condensed;
- c) the condensates are separated from the uncondensed gases;
- d) the uncondensed gases are cooled and washed in a scrubber with a cold polymerised formaldehyde solution which retains the methanol and formaldehyde contained in the gases;
- e) the solution obtained in step c) is distilled such that a concentrated aqueous formaldehyde solution and methanol are obtained separately;
- f) the solution obtained in step d) is distilled such

that the methanol is recovered and the formaldehyde is fixed;

g) a part of the formaldehyde solution obtained in step f) is pooled with the distilled condensate obtained in step e);

h) the concentrated aqueous formaldehyde solution from step e) is mixed with urea under certain pH and temperature conditions to obtain urea-formaldehyde glues or resins.

The above described steps are effected continuously and, in a preferred embodiment, atmospheric air is aspirated at a pressure of 101 KPa (760 mm Hg) with an appropriate compressor. The air is blown at a constant flow rate to a saturator and, therein, the air is bubbled through methanol, which is held at 50.5° C by means of a heating device. In turn, the methanol is supplied to the saturator from a reservoir at a constant rate thanks to an adequate regulation by way of a valve.

Thereafter, the mixture leaving the saturator, also at a constant methanol rate, is fed over a silver metal based catalyst held at a temperature of about 500° C such that it converts about 60% of the methanol into formaldehyde, in a conventional reaction, the corresponding hydrogen, part of which is oxidised in water by the air oxygen, being released. At the same time, a small amount of carbon dioxide is formed and practically all the air oxygen is converted during the reaction.

The gas mixture from the the catalyser is thereafter fed to the base of a scrubber receiving at the head thereof the condensates removed at the scrubber base, which are circulated by a pump and cooled by a cooler.

In the process of Spanish patent 447,890, the thus collected mixture is fed by a pump through the above mentioned remaining steps up to the preparation of the urea-formaldehyde glues and resins.

According to the present invention, the mixture cited in the foregoing paragraph forms the starting material for the preparation of said stabilised formaldehyde and methanol solutions. In the Examples given hereinafter, this mixture is referred to as: mixture from the scrubber base.

In other words, the present method is characterised in that the starting material is a liquid mixture comprising a formaldehyde concentration of from 48 to 55 wt%, a methanol concentration of from 20 to 30 wt% and an amount of water of from 32 to 15 wt%, said liquid mixture being tapped from a plant for the continuous preparation of urea-formaldehyde glues and resins, immediately after having subjected the methanol to catalytic oxidation in air, to condensation of the gases from said catalytic oxidation and to separation of the condensates from the uncondensed gases and in that said liquid mixture is stabilized by means of a base.

According to a further feature of the invention,

said liquid mixture is tapped continuously and the liquid mixture is stabilised by the continuous supply of said base and water, using the heat from the liquid mixture itself and from prior heating of the said water.

Preferably, according to the invention, the respective flow rates of said continuous supplies may be represented by magnitudes proportional to the following numbers: from 900 to 1100 for said liquid mixture; from 30 to 50 for said hot water and from 1 to 2 for said base.

According to a first development of the invention, said liquid mixture has a composition of 50.5 wt% of formaldehyde, and 26.0 wt% of methanol; said base is triethylamine; the flow rates of the liquid mixture, hot water and triethylamine are in an approximate ratio of 1000:40:1.4, a stabilised solution having a composition of 48.5 wt% of formaldehyde and 25.0% methanol being obtained.

According to another development of the invention, said liquid mixture is tapped batchwise and is stabilised by the addition of said base and water, the mixture being heated to a temperature of from 70° to 100° C for a time ranging from 5 to 30 minutes; more preferably, said tapped liquid mixture and said water and base additions represent magnitudes proportional to the following numbers: from 12,000 to 15,000 for said liquid mixture; from 500 to 625 for said water and from 15 to 20 for said base.

In an alternative development of the invention, said liquid mixture is tapped continuously and the liquid mixture is stabilised by the continuous supply of said base, followed by continuous low pressure distillation, giving, on the one hand, a concentrate to which methanol is added and, on the other hand, low concentration distillates which may be fed to a dealcoholisation column where the methanol and a formaldehyde-containing solution are separated and the respective flow rates of said continuous supplies preferably represent magnitudes proportional to the following numbers: from 900 to 1100 for said liquid mixture and from 1 to 2 for said base and wherein said low pressure ranges from 26 to 40 KPa (200 to 300 mm Hg).

In a subsequent development of the invention, said liquid mixture is tapped batchwise and is stabilised with the addition of said base, followed by low pressure distillation, giving, on the one hand, a concentrate to which methanol is added and, on the other hand, low concentration distillates which may be fed to a dealcoholisation column where the methanol and a formaldehyde-containing solution are separated, it being a preferred feature that said tapped liquid mixture and said addition of a base represent magnitudes proportional to the following numbers: from 12,000 to 16,000 for said liquid mixture and from 16 to 24 for said base and wherein said low pressure ranges from 21 to 32 KPa (160 to 240 mm Hg).

Examples of the invention are given hereinafter

without any limiting nature.

EXAMPLE 1

CONTINUOUS PREPARATION OF A 48/25 FORMALDEHYDE-METHANOL SOLUTION.

40 kg per hour of water and 1.4 kg per hour of triethylamine were added by a metering pump system to 1,000 kg per hour of the mixture coming from the base of the scrubber, having a composition of 50.5% of formaldehyde and 26.0% of methanol.

The temperature of the liquid from the base of the scrubber together with the water at 70° C was used to stabilise the formaldehyde-methanol solution.

The product obtained is stable for a period of over 6 months at 20° C and has a composition of 48.5% of formaldehyde and 25.0% of methanol.

EXAMPLE 2

BATCHWISE PREPARATION OF A 48/25 FORMALDEHYDE-METHANOL SOLUTION.

A reactor equipped with a stirrer and heating and cooling system was charged with a mixture having a composition of 50.5% of formaldehyde and 25% of methanol from the scrubber base. The flow rate was 1,000 kg per hour.

A 14 cu.m. capacity reactor was charged initially with 560 kg of water and 18 kg of 30% caustic soda, whereby 14 tonnes of a solution having a composition 48% of formaldehyde and 25% of methanol was obtained.

The product was stabilised when the reactor charge was complete, by heating to 90° C for 15 minutes. The product was cooled and drained off.

During the time used for stabilising the solution, cooling and draining the reactor, the plant described in Spanish patent 447,890 continued running to produce the urea-formaldehyde glues and resins.

EXAMPLE 3

CONTINUOUS PREPARATION OF A 55/35 FORMALDEHYDE-METHANOL SOLUTION.

1.5 kg per hour of 30% caustic soda were added by a metering pump system to 1,000 kg per hour of the mixture coming from the base of the scrubber, having a composition of 50.8% of formaldehyde and 20.0% of methanol and was fed to a continuous distillation or concentration system operating at an absolute pressure of 33 KPa (250 mm Hg). 505 kg per hour of a concentrate having a composition of 74% of formaldehyde and 12.0% of methanol and 495 kg per hour of distillates having a composition of 25.0% of formaldehyde and 28.2% of methanol were obtained, the re-

mainder being water.

These distillates were also continuously fed to a dealcoholisation column where the methanol was removed and is recovered for the formaldehyde production system. At the column base formaldehyde with 34.8% methanol was obtained.

179 kg per hour of methanol was supplied continuously to the concentrate resulting from the distillation to adjust the product concentration.

684 kg per hour of a solution containing 54.6% of formaldehyde and 35.0% of methanol were obtained.

EXAMPLE 4

BATCHWISE PREPARATION OF A 55/35 FORMALDEHYDE-METHANOL SOLUTION.

A 14 cu.m. capacity reactor equipped with a stirrer and heating, cooling and condenser system and vacuum distilling equipment was charged with a mixture having a composition of 54.8% of formaldehyde and 20.2% of methanol from the scrubber base. The flow rate was 1,000 kg per hour.

When the 14 tonne charge was completed, 20 kg of triethylamine were added and vacuum distillation was effected (absolute pressure 26 KPa (200 mmHg)).

The distillation was terminated when the formaldehyde concentration reached 74% and 7,770 kg having a 12% methanol content had been obtained.

2,758 kg of methanol were added and a charge of 10,528 kg having a 54.7% formaldehyde and 35.0% methanol composition was completed. The product obtained was stable for over 6 months at 20° C.

7,639 kg of distillate having a 25% formaldehyde and 25% methanol composition were obtained, the remainder being water. This material was suitably metered by a pump to a dealcoholisation column. 5,720 kg of a methanol-free 33.2% formaldehyde solution was obtained, the remainder being water.

During the time used for distilling and stabilising the solution, cooling and draining the reactor, the plant described in Spanish patent 447,890 continued running to produce the urea-formaldehyde glues and resins.

Claims

1. A method for producing stabilized solutions of formaldehyde with methanol, characterised in that the starting material is a liquid mixture comprising a formaldehyde concentration of from 48 to 55 wt%, a methanol concentration of from 20 to 30 wt% and an amount of water of from 32 to 15 wt%, said liquid mixture being tapped from a plant for the continuous preparation of urea-formaldehyde glues and resins, immediately af-

ter having subjected the methanol to catalytic oxidation in air, to condensation of the gases from said catalytic oxidation and to separation of the condensates from the uncondensed gases, and in that said liquid mixture is stabilized by means of a base.

2. The method of claim 1, wherein said liquid mixture is tapped continuously and the liquid mixture is stabilised by the continuous supply of said base and water, using the heat from the liquid mixture itself and from prior heating of said water.
3. The method of claim 2, wherein the respective flow rates of said continuous supplies represent magnitudes proportional to the following numbers: from 900 to 1100 for said liquid mixture; from 30 to 50 for said hot water and from 1 to 2 for said base.
4. The method of claim 3, wherein said liquid mixture has a composition of 50.5 wt% of formaldehyde, and 26.0 wt% of methanol; said base is triethylamine; the flow rates of the liquid mixture, hot water and triethylamine are in an approximate ratio of 1000:40:1.4, there being obtained a stabilised solution having a composition of 48.5 wt% of formaldehyde and 25.0% methanol.
5. The method of claim 1, wherein said liquid mixture is tapped batchwise and is stabilised by the addition of said base and water, the mixture being heated to a temperature of from 700 to 100° C for a time ranging from 5 to 30 minutes.
6. The method of claim 5, wherein said tapped liquid mixture and said water and base additions represent magnitudes proportional to the following numbers: from 12,000 to 15,000 for said mixture; from 500 to 625 for said water and from 15 to 20 for said base.
7. The method of claim 6, wherein said liquid mixture has a composition of 50.5 wt% of formaldehyde and 25.0 wt% of methanol; said base is 30% caustic soda; and the liquid mixture, water and caustic soda supplies are in an approximate ratio of 13,422:560:18, there being obtained a stabilised solution having a composition of 48 wt% of formaldehyde and 25 wt% of methanol.
8. The method of claim 1, wherein said liquid mixture is tapped continuously and the liquid mixture is stabilised by the continuous supply of said base, followed by continuous low pressure distillation, giving, on the one hand, a concentrate to which methanol is added and, on the other hand, low concentration distillates which may be fed to

mainder being water.

These distillates were also continuously fed to a dealcoholisation column where the methanol was removed and is recovered for the formaldehyde production system. At the column base formaldehyde with 34.8% methanol was obtained.

179 kg per hour of methanol was supplied continuously to the concentrate resulting from the distillation to adjust the product concentration.

684 kg per hour of a solution containing 54.6% of formaldehyde and 35.0% of methanol were obtained.

EXAMPLE 4

BATCHWISE PREPARATION OF A 55/35 FORMALDEHYDE-METHANOL SOLUTION.

A 14 cu.m. capacity reactor equipped with a stirrer and heating, cooling and condenser system and vacuum distilling equipment was charged with a mixture having a composition of 54.8% of formaldehyde and 20.2% of methanol from the scrubber base. The flow rate was 1,000 kg per hour.

When the 14 tonne charge was completed, 20 kg of triethylamine were added and vacuum distillation was effected (absolute pressure 26 KPa (200 mmHg)).

The distillation was terminated when the formaldehyde concentration reached 74% and 7,770 kg having a 12% methanol content had been obtained.

2,758 kg of methanol were added and a charge of 10,528 kg having a 54.7% formaldehyde and 35.0% methanol composition was completed. The product obtained was stable for over 6 months at 20° C.

7,639 kg of distillate having a 25% formaldehyde and 25% methanol composition were obtained, the remainder being water. This material was suitably metered by a pump to a dealcoholisation column. 5,720 kg of a methanol-free 33.2% formaldehyde solution was obtained, the remainder being water.

During the time used for distilling and stabilising the solution, cooling and draining the reactor, the plant described in Spanish patent 447,890 continued running to produce the urea-formaldehyde glues and resins.

Claims

1. A method for producing stabilized solutions of formaldehyde with methanol, characterised in that the starting material is a liquid mixture comprising a formaldehyde concentration of from 48 to 55 wt%, a methanol concentration of from 20 to 30 wt% and an amount of water of from 32 to 15 wt%, said liquid mixture being tapped from a plant for the continuous preparation of urea-formaldehyde glues and resins, immediately af-

ter having subjected the methanol to catalytic oxidation in air, to condensation of the gases from said catalytic oxidation and to separation of the condensates from the uncondensed gases, and in that said liquid mixture is stabilized by means of a base.

2. The method of claim 1, wherein said liquid mixture is tapped continuously and the liquid mixture is stabilised by the continuous supply of said base and water, using the heat from the liquid mixture itself and from prior heating of said water.
3. The method of claim 2, wherein the respective flow rates of said continuous supplies represent magnitudes proportional to the following numbers: from 900 to 1100 for said liquid mixture; from 30 to 50 for said hot water and from 1 to 2 for said base.
4. The method of claim 3, wherein said liquid mixture has a composition of 50.5 wt% of formaldehyde, and 26.0 wt% of methanol; said base is triethylamine; the flow rates of the liquid mixture, hot water and triethylamine are in an approximate ratio of 1000:40:1.4, there being obtained a stabilised solution having a composition of 48.5 wt% of formaldehyde and 25.0% methanol.
5. The method of claim 1, wherein said liquid mixture is tapped batchwise and is stabilised by the addition of said base and water, the mixture being heated to a temperature of from 700 to 100° C for a time ranging from 5 to 30 minutes.
6. The method of claim 5, wherein said tapped liquid mixture and said water and base additions represent magnitudes proportional to the following numbers: from 12,000 to 15,000 for said mixture; from 500 to 625 for said water and from 15 to 20 for said base.
7. The method of claim 6, wherein said liquid mixture has a composition of 50.5 wt% of formaldehyde and 25.0 wt% of methanol; said base is 30% caustic soda; and the liquid mixture, water and caustic soda supplies are in an approximate ratio of 13,422:560:18, there being obtained a stabilised solution having a composition of 48 wt% of formaldehyde and 25 wt% of methanol.
8. The method of claim 1, wherein said liquid mixture is tapped continuously and the liquid mixture is stabilised by the continuous supply of said base, followed by continuous low pressure distillation, giving, on the one hand, a concentrate to which methanol is added and, on the other hand, low concentration distillates which may be fed to



a dealcoholisation column where the methanol and a formaldehyde-containing solution are separated.

9. The method of claim 8, wherein the respective flow rates of said continuous supplies represent magnitudes proportional to the following numbers: from 900 to 1100 for said liquid mixture and from 1 to 2 for said base and wherein said low pressure ranges from 26 to 40 KPa (200 to 300 mm Hg).
10. The method of claim 8 or claim 9, wherein said liquid mixture has a composition of 50.8 wt% of formaldehyde and 20.0 wt% of methanol; said base is 30% caustic soda; and the liquid mixture and caustic soda supply rates are in an approximate ratio of 1000:1.5; the concentrate has a composition of 74 wt% of formaldehyde and 12 wt% of methanol; said methanol being added at a rate, relative to said concentrate, of approximately 505:180, there being obtained a stabilised solution having 55 wt% of formaldehyde and 35 wt% of methanol.
11. The method of claim 1, wherein said liquid mixture is tapped batchwise and is stabilised with the addition of said base, followed by low pressure distillation, giving, on the one hand, a concentrate to which methanol is added and, on the other hand, low concentration distillates which may be fed to a dealcoholisation column where the methanol and a formaldehyde-containing solution are separated.
12. The method of claim 11, wherein liquid mixture tapping and said addition of a base represent magnitudes proportional to the following numbers: from 12,000 to 16,000 for said liquid mixture and from 16 to 24 for said base and wherein said low pressure ranges from 31 to 32 KPa (160 to 240 mm Hg).
13. The method of claim 11 or claim 12, wherein said liquid mixture has a composition of 54.8 wt% of formaldehyde and 20.2 wt% of methanol; said base is triethylamine; the liquid mixture and triethylamine supply rates are in an approximate ratio of 14,000:20; the concentrate has a composition of 74 wt% of formaldehyde and 12 wt% of methanol; said methanol being added at a rate, relative to said concentrate, of approximately 1,110:400, there being obtained a stabilised solution having 54.7 wt% of formaldehyde and 35 wt% of methanol.

Patentansprüche

1. Verfahren zur Herstellung von stabilisierten Lösungen aus Formaldehyd mit Methanol, dadurch gekennzeichnet, daß das Ausgangsmaterial ein flüssiges Gemisch ist, umfassend Formaldehyd in einer Konzentration von 48 bis 55 Gew.-%, Methanol in einer Konzentration von 20 bis 30 Gew.-% und Wasser in einer Menge von 32 bis 15 Gew.-%, wobei das flüssige Gemisch aus einer Anlage zur kontinuierlichen Herstellung von Harnstoff/Formaldehyd-Leimen und -Harzen abgezogen wird, unmittelbar nachdem das Methanol einer katalytischen Oxidation an Luft unterworfen worden ist, um die Gase von dieser katalytischen Oxidation zu kondensieren und die Kondensate von den nicht kondensierten Gasen abzutrennen und daß das flüssige Gemisch mit Hilfe einer Base stabilisiert wird.
2. Verfahren nach Anspruch 1, wobei das flüssige Gemisch kontinuierlich abgezogen wird, und das flüssige Gemisch stabilisiert wird durch kontinuierliche Zufuhr der Base und von Wasser unter Anwendung von Wärme aus dem flüssigen Gemisch selbst und durch Vorerhitzen des Wassers.
3. Verfahren nach Anspruch 2, wobei die jeweiligen Fließgeschwindigkeiten der kontinuierlichen Zufuhr in Größenordnungen liegen, die proportional sind den folgenden Zahlen: 900 bis 1100 für das flüssige Gemisch, 30 bis 50 für das heiße Wasser und 1 bis 2 für die Base.
4. Verfahren nach Anspruch 3, wobei das flüssige Gemisch eine Zusammensetzung von 50,5 Gew.-% Formaldehyd und 26,0 Gew.-% Methanol aufweist, die Base Triethylamin ist, die Fließgeschwindigkeiten des flüssigen Gemisches, heißen Wassers und Triethylamins etwa im Verhältnis von 1000:40:1,4 liegen, wobei eine stabilisierte Lösung erhalten wird mit einer Zusammensetzung von 48,5 Gew.-% Formaldehyd und 25,0 % Methanol.
5. Verfahren nach Anspruch 1, wobei das flüssige Gemisch ansatzweise abgezogen wird und stabilisiert wird durch Zusatz der Base und von Wasser, und wobei das Gemisch auf eine Temperatur von 70° bis 100°C während eines Zeitraums im Bereich von 5 bis 30 min erhitzt wird.
6. Verfahren nach Anspruch 5, wobei die Zugabemengen des abgezogenen flüssigen Gemisches, des Wassers und von Base Werten entsprechen, proportional den folgenden Zahlen: 12 000 bis 15 000 für das Gemisch; 500 bis 625 für das Wasser



und 15 bis 20 für die Base.

7. Verfahren nach Anspruch 6, wobei das flüssige Gemisch eine Zusammensetzung von 50,5 Gew.-% Formaldehyd und 25,0 Gew.-% Methanol aufweist; die Base 30%ige Natronlauge ist und die Zufuhrmengen des flüssigen Gemisches, von Wasser und Natronlauge etwa dem Verhältnis 13 422:560:18 entsprechen, wobei eine stabilisierte Lösung erhalten wird mit einer Zusammensetzung von 48 Gew.-% Formaldehyd und 25 Gew.-% Methanol. 5
8. Verfahren nach Anspruch 1, wobei das flüssige Gemisch kontinuierlich abgezogen wird und das flüssige Gemisch stabilisiert wird durch kontinuierliche Zufuhr der Base, gefolgt von einer kontinuierlichen Niederdruckdestillation, wobei man einerseits ein Konzentrat erhält, zu dem Methanol zugegeben wird, und andererseits Destillate niedriger Konzentration, die in eine Entalkoholisierungssäule geführt werden können, wo das Methanol und eine Formaldehyd-haltige Lösung getrennt werden. 10 15 20
9. Verfahren nach Anspruch 8, wobei die jeweiligen Fließgeschwindigkeiten der kontinuierlichen Zufuhr Werte darstellen proportional zu den folgenden Zahlen: 900 bis 1100 für das flüssige Gemisch und 1 bis 2 für die Base und wobei der Niederdruck im Bereich von 26 bis 40 kPa (200 bis 300 mm Hg) liegt. 25 30
10. Verfahren nach Anspruch 8 oder 9, wobei das flüssige Gemisch eine Zusammensetzung von 50,8 Gew.-% Formaldehyd und 20,0 Gew.-% Methanol aufweist, die Base 30%ige Natronlauge ist und die Zufuhrraten für das flüssige Gemisch und die Natronlauge etwa im Verhältnis 1000:1,5 liegen, das Konzentrat eine Zusammensetzung von 74 Gew.-% Formaldehyd und 12 Gew.-% Methanol aufweist, das Methanol mit einer Geschwindigkeit, bezogen auf das Konzentrat, von etwa 505:180 zugegeben wird, wobei eine stabilisierte Lösung erhalten wird, mit 55 Gew.-% Formaldehyd und 35 Gew.-% Methanol. 35 40 45
11. Verfahren nach Anspruch 1, wobei das flüssige Gemisch ansatzweise abgezogen wird und stabilisiert wird durch Zugabe der Base und anschließende Niederdruckdestillation, wobei man einerseits ein Konzentrat erhält, zu dem Methanol zugesetzt wird, und andererseits Destillate geringer Konzentration, die in eine Entalkoholisierungssäule geleitet werden können, wo das Methanol und eine Formaldehyd-haltige Lösung getrennt werden. 50 55

12. Verfahren nach Anspruch 11, wobei das Abziehen des flüssigen Gemisches und die Zugabe einer Base Werte darstellen, die proportional sind den folgenden Zahlen: 12 000 bis 16 000 für das flüssige Gemisch und 16 bis 24 für die Base und wobei der Niederdruck im Bereich von 31 bis 32 kPa (160 bis 240 mm Hg) liegt.

13. Verfahren nach Anspruch 11 oder 12, wobei das flüssige Gemisch eine Zusammensetzung von 54,8 Gew.-% Formaldehyd und 20,2 Gew.-% Methanol aufweist, die Base Triethylamin ist, die Zufuhrraten des flüssigen Gemisches und von Triethylamin in einem ungefähren Verhältnis von 14 000:20 liegen, das Konzentrat eine Zusammensetzung von 74 Gew.-% Formaldehyd und 12 Gew.-% Methanol aufweist und das Methanol in einer Geschwindigkeit zugegeben wird, bezogen auf das Konzentrat von etwa 1110:400 und wobei man eine stabilisierte Lösung erhält mit 54,7 Gew.-% Formaldehyd und 35 Gew.-% Methanol.

Revendications

1. Procédé de production de solutions stabilisées de formaldéhyde avec méthanol, caractérisé en ce que la matière de départ est un mélange liquide comprenant une concentration de formaldéhyde de 48 à 55 % en poids, une concentration de méthanol de 20 à 30 % en poids et un volume d'eau de 32 à 15 % en poids, ledit mélange liquide étant prélevé d'une installation pour la préparation en continu de colles et de résines d'urée formaldéhyde, immédiatement après avoir soumis le méthanol à une oxydation catalytique à l'air, pour condenser les gaz de ladite oxydation catalytique et pour séparer les condensés des gaz non-condensés, et en ce que ledit mélange liquide est stabilisé au moyen d'une base. 25 30
2. Procédé selon la revendication 1, dans lequel ledit mélange liquide est prélevé en continu et le mélange liquide est stabilisé par l'apport continu de ladite base et d'eau, en utilisant la chaleur du mélange liquide lui-même et en chauffant préalablement ladite eau. 35 40 45
3. Procédé selon la revendication 2, dans lequel les débits respectifs desdits apports continus représentent des grandeurs proportionnelles aux nombres suivants : de 900 à 1100 pour ledit mélange liquide ; de 30 à 50 pour ladite eau chaude et de 1 à 2 pour ladite base. 50 55
4. Procédé selon la revendication 3, dans lequel ledit mélange liquide a une composition de 50,5 % en poids de formaldéhyde, et 26,0 % en poids de 6



méthanol ; ladite base est de la triéthylamine ; les débits du mélange liquide, de l'eau chaude et de la triéthylamine sont dans un rapport approximatif de 1000:40:1,4, il a été obtenu une solution stabilisée ayant une composition de 48,5 % en poids de formaldéhyde et de 25,0 % de méthanol.

5. Procédé selon la revendication 1, dans lequel ledit mélange liquide est prélevé par lots et est stabilisé par l'addition de ladite base et d'eau, le mélange étant chauffé à une température de 70° à 100° C pendant une période de temps de 5 à 30 minutes. 5
6. Procédé selon la revendication 5, dans lequel ledit mélange liquide prélevé et lesdites additions d'eau et de base représentent des grandeurs proportionnelles aux nombres suivants : de 12000 à 15000 pour ledit mélange ; de 500 à 625 pour ladite eau et de 15 à 20 pour ladite base. 10
7. Procédé selon la revendication 6, dans lequel ledit mélange liquide a une composition de 50,5 % en poids de formaldéhyde et de 25,0 % en poids de méthanol ; ladite base est de la soude caustique à 30 % ; et le mélange liquide, l'apport d'eau et de soude caustique sont approximativement dans un rapport de 13 422:560:18, il a été obtenu une solution stabilisée ayant une composition de 48 % en poids de formaldéhyde et de 25 % en poids de méthanol. 15
8. Procédé selon la revendication 1, dans lequel ledit mélange liquide est prélevé en continu et le mélange liquide est stabilisé par l'apport continu de ladite base, suivi par une distillation continue à basse pression, donnant, d'une part, un concentré auquel on ajoute du méthanol et, d'autre part, des distillats de faible concentration qui peuvent être fournis à une colonne de déalcoolisation où le méthanol et une solution contenant du formaldéhyde sont séparés. 20
9. Procédé selon la revendication 8, dans lequel les débits respectifs desdits apports continus représentent des grandeurs proportionnelles aux nombres suivants : de 900 à 1100 pour ledit mélange liquide et de 1 à 2 pour ladite base, et dans lequel ladite basse pression est comprise dans la gamme de 26 à 40 KPa (200 à 300 mm Hg). 25
10. Procédé selon les revendications 8 ou 9, dans lequel ledit mélange liquide a une composition de 50,8 % en poids de formaldéhyde et de 20,0 % en poids de méthanol ; ladite base est une soude caustique à 30 % ; et les taux des apports de mélange liquide et de soude caustique sont dans un rapport approximatif de 1000:1,5 ; le concentré 30

a une composition de 74 % en poids de formaldéhyde et de 12 % en poids de méthanol ; ledit méthanol étant ajouté dans un rapport, par rapport audit concentré, approximativement de 505:180, il a été obtenu une solution stabilisée ayant 55% en poids de formaldéhyde et de 35 % en poids de méthanol.

11. Procédé selon la revendication 1, dans lequel ledit mélange liquide est prélevé par lots et est stabilisé par l'addition de ladite base, suivi par une distillation à basse pression, donnant, d'une part, un concentré auquel on ajoute le méthanol et, d'autre part, des distillats de faible concentration qui peuvent être fournis à une colonne de déalcoolisation où le méthanol et une solution contenant du formaldéhyde sont séparés. 35
12. Procédé selon la revendication 11, dans lequel le mélange liquide prélevé et ladite addition d'une base représentent des grandeurs proportionnelles aux nombres suivants : de 12000 à 16000 pour ledit mélange liquide et de 16 à 24 pour ladite base et dans lequel ladite basse pression est comprise dans la gamme de 31 à 32 KPa (160 à 240 mm Hg). 40
13. Procédé selon les revendications 11 ou 12, dans lequel ledit mélange a une composition de 54,8 % en poids de formaldéhyde et de 20,2 % en poids de méthanol ; ladite base est de la triéthylamine ; les rapports des apports du mélange liquide et de la triéthylamine sont approximativement dans un rapport de 14000:20 ; le concentré a une composition de 74 % en poids de formaldéhyde et de 12 % en poids de méthanol ; ledit méthanol étant ajouté dans un rapport, par rapport audit concentré, approximativement de 1110:40, il a été obtenu une solution stabilisée ayant 54,7 % en poids de formaldéhyde et de 35 % en poids de méthanol. 45

