

DESCRIPTION

METHOD AND APPARATUS FOR PRODUCTION OF DIALKYLAMINOALKYL
(METH)ACRYLATE QUATERNARY SALT

5 TECHNICAL FIELD

The present invention relates to a method and apparatus for producing a dialkylaminoalkyl (meth)acrylate quaternary salt with high productivity under stable production conditions.

10 BACKGROUND ART

Dialkylaminoalkyl (meth)acrylate quaternary salts (hereinafter referred to as "DAM") are raw materials, etc., for cationic polymers, and are used for various purposes as raw materials for the production of polymer coagulants, paper
15 strength-reinforcing agents, antistatic agents, soil conditioners, and the like.

DAM is generally produced in the form of an aqueous solution of DAM. During production, using water as a solvent, raw materials are supplied in the presence of the solvent, and the
20 mixture is reacted while removing the heat of reaction. Conventionally, DAM is produced by a batch process (for example, Patent Documents 1 and 2).

However, such a method of producing DAM by a batch process has some drawbacks. Specifically, since the production is
25 performed in one reactor, it is necessary to newly feed raw materials on each DAM production process, and to remove products after the reaction is completed. In addition, a long period of time is required to stabilize production conditions. Thus, productivity is problematic. Moreover, the batch process makes it
30 difficult to stabilize production conditions such as the concentration of raw materials in a reaction mixture, the heat of the reaction, and the like. As a result, the quality of the obtained products tends to vary. Furthermore, in water or water-containing reaction solvent systems, acrylate is, when remained
35 in the reaction mixture, easily hydrolyzed to produce acrylic

acid. When polymers obtained by polymerization of DAM products, which contain acrylic acids as impurities, are used as flocculants, performance of some flocculants is degraded.

To solve these problems, the present inventors reported
5 a method of producing high-purity DAM with high productivity and few quality differences between products by continuously supplying a dialkylaminoalkyl (meth)acrylate, a quaternizing agent, and water to a reactor charged with a DAM aqueous solution as a reaction solvent, while continuously extracting the reaction
10 mixture from the reactor (Patent Document 3).

This method is excellent; however, when a low-boiling quaternizing agent is supplied as the quaternizing agent in the form of a gas, the supply pressure of the quaternizing agent should be above the pressure in the reactor. It is thus necessary
15 to increase pressure for the supply of a quaternizing agent. Further, the reaction mixture sometimes flowed backward into a supply pipe for the quaternizing agent, clogging the pipe. Moreover, when a liquid quaternizing agent was directly supplied to the reactor, since the agent was insufficiently dispersed in
20 the reaction mixture, reactivity was reduced.

Considering the above, the present inventors found that by supplying a quaternizing agent by an ejector, the following advantages are achieved: the reaction mixture does not flow backward into the quaternizing agent supply pipe; the
25 quaternizing agent is sufficiently dispersed in the reaction mixture; the solubility of the quaternizing agent in the reaction mixture is improved; and the reactivity becomes superior (Patent Document 4).

Patent Document 1: Japanese Unexamined Patent Publication No.
30 1992-217649

Patent Document 2: Japanese Unexamined Patent Publication No.
1996-268985

Patent Document 3: Japanese Unexamined Patent Publication No.
2003-342244

35 Patent Document 4: Japanese Unexamined Patent Publication No.

2004-155669

DISCLOSURE OF THE INVENTION

Problems to be Solved by the Invention

5 The method of continuously supplying a quaternizing agent by an ejector, as described in Patent Document 4, is an excellent method for producing DAM. However, due to the heat generation that accompanies a quaternization reaction, the method has a problem in that the temperature control in the reactor
10 becomes harder with increased reactivity. Generally, a reaction tank is cooled by a jacket-type heat exchanger of the reaction tank. It is difficult, however, to match the cooling capacity (the amount of heat removal) with the heat value; therefore, the temperature in the reactor rises. An excessive temperature rise
15 in the reactor causes problems such as the polymerization reaction of generated DAM and the hydrolysis of ester. Thus, stable production of high-grade DAM is difficult. For this reason, a method that secures temperature control in the reactor has been desired.

20 There is another problem in that when the amount of heat removal is small, a larger reactor or a plurality of reactors is necessary to increase the production volume.

 In light of above, an object of the present invention is to provide a method of producing high-purity DAM with high
25 throughput, even with the use of a compact reactor, by efficiently cooling the reaction mixture to control the temperature increase in the reactor. Another object of the present invention is to provide an apparatus suitable for the method.

30 Means for Solving the Problem

 The inventors conducted extensive research to solve the above-described problems, and found that high-purity DAM can be produced with high productivity as follows. First, a part of the
35 reaction mixture is extracted from a reactor during the reaction,

and cooled through a heat exchanger. Then, the cooled reaction mixture is supplied to the reactor, together with a quaternizing agent, by an ejector. Consequently, the heat in the reactor can be effectively removed. As a result of further research based on
5 these findings, the present invention has been accomplished.

Specifically, the present invention provides a method and apparatus for producing dialkylaminoalkyl (meth)acrylate quaternary salts, as described below.

Item 1. A method of producing a dialkylaminoalkyl (meth)acrylate
10 quaternary salt by continuously supplying a dialkylaminoalkyl (meth)acrylate, a quaternizing agent, and water to a reactor, while continuously extracting a reaction mixture from the reactor,

wherein a part of the reaction mixture extracted from the reactor is cooled by passing through a heat exchanger, and
15 the cooled reaction mixture is supplied to the reactor together with a quaternizing agent through an ejector.

Item 2. A method according to Item 1, wherein the reactor is previously charged with a dialkylaminoalkyl (meth)acrylate quaternary salt aqueous solution before a dialkylaminoalkyl
20 (meth)acrylate, a quaternizing agent, and water are continuously supplied.

Item 3. A method according to Item 1 or 2, wherein the reaction mixture is maintained at a temperature of 30 to 80°C during reaction.

25 Item 4. A method according to any one of Items 1 to 3, wherein the reactor is a sealed reactor, the quaternizing agent is a quaternizing agent having a boiling point of 25°C or lower, and the pressure inside the reactor is maintained at 0.10 to 1 MPaG during reaction.

30 Item 5. A method according to any one of Items 1 to 4, wherein the dialkylaminoalkyl (meth)acrylate is dimethylaminoethyl (meth)acrylate, and the quaternizing agent is methyl chloride.

Item 6. A method according to any one of Items 1 to 5, further comprising supplying the reaction mixture continuously extracted
35 from the reactor to one or more sealed tanks, and gradually

adjusting the pressure of the reaction mixture in the tank to atmospheric pressure.

Item 7. An apparatus for producing a dialkylaminoalkyl (meth)acrylate quaternary salt, comprising:

- 5 a reactor (6) equipped with a dialkylaminoalkyl (meth)acrylate supply port (10), a water supply port (11), an ejector (1) for supplying a quaternizing agent, a reaction mixture extraction port (13), a circulating pump (7) for extracting a part of the reaction mixture, and a stirring blade
- 10 (9) as required;
- a flow path for supplying the reaction mixture extracted from the reactor (6) by the circulating pump (7) to the ejector (1); and
- a heat exchanger (8) disposed in the flow path, for
- 15 cooling the extracted reaction mixture.

Effect of the Invention

The DAM-producing method of the present invention allows efficient heat removal from a reactor. In particular, when

20 a quaternizing agent is supplied by using an ejector, reactivity is enhanced, increasing the heat value. Even in such a case, heat removal is readily carried out. Accordingly, temperature control in the reactor becomes extremely simple, and high-purity DAM can be produced with high productivity.

25

Brief Description of the Drawings

Fig. 1 schematically illustrates an ejector.

Fig. 2 schematically illustrates a reactor of the present invention comprising an ejector and an external heat

30 exchanger.

Description of Symbols

- 1: Ejector
- 2: Nozzle part
- 35 3: Suction part

- 4: Reaction mixture
- 5: Quaternizing agent
- 6: Reaction tank
- 7: Circulating pump
- 5 8: Heat exchanger
- 9: Agitator
- 10: Da supply pipe
- 11: Water supply pipe
- 12: Quaternizing agent supply pipe
- 10 13: Reaction-mixture extraction port
- 14: Jacket-type heat exchanger

BEST MODE FOR CARRYING OUT THE INVENTION

Embodiments of the present invention are described in
15 detail below. The present invention is not limited to these
embodiments, and can be suitably modified as long as the gist of
the invention is unchanged.

Note that the term (meth)acrylate in the specification
means acrylate or methacrylate.

20 1. Raw Materials Used

In the DAM-producing method of the present invention, a
dialkylaminoalkyl (meth)acrylate, a quaternizing agent, and water
are used as raw materials.

Specific examples of dialkylaminoalkyl (meth)acrylate
25 (hereinafter referred to as "Da") include dimethylaminoethyl
(meth)acrylate, diethylaminoethyl (meth)acrylate,
dipropylaminoethyl (meth)acrylate, dimethylaminopropyl
(meth)acrylate, etc.

Various compounds can be used as a quaternizing agent,
30 insofar as they can quaternize the amino group of Da. Either
liquid or gaseous quaternizing agents are available. However,
from the standpoint of easy removal of unreacted quaternizing
agent from the reaction mixture after the reaction, easy supply
to an ejector, and excellent dispersibility in the reaction
35 mixture, quaternizing agents in the form of a gas or with a low

boiling point (hereinafter referred to as "low-boiling-point quaternizing agents") are preferable. Specifically, a quaternizing agent having a boiling point of 25°C or lower, preferably 20°C or lower, and more preferably 10°C or lower, at
5 atmospheric pressure, can be used.

Specific examples of such a quaternizing agent include methyl chloride, ethyl chloride, and other alkyl halides, as well as benzyl chloride and other arylalkyl halides, etc. Among them, alkyl halides are preferable, alkyl halides having an alkyl group
10 containing 1 to 5 carbon atoms are more preferable, and methyl chloride is particularly preferable.

The quaternizing agent can be supplied to a reactor in various forms. For example, the quaternizing agent can be supplied in the form of a gas, liquid, or gas-liquid mixture.

15 As the above-mentioned water used in the present invention, the purest possible water free from metals, etc., is preferably used. Specific examples include distilled water, ion exchange water, etc.

2. Production of DAM

20 The present invention is a method of producing DAM, comprising continuously supplying Da, a quaternizing agent, and water to a reactor, while continuously extracting a reaction mixture from the reactor, wherein a part of the reaction mixture extracted from the reactor is cooled by passing through a heat
25 exchanger; and the cooled reaction mixture is supplied to the reactor together with the quaternizing agent using an ejector. Here, the term "reaction mixture" refers to a solution containing DAM generated by the reaction of raw materials in the reactor.

Fig. 2 shows an example of an apparatus for carrying
30 out the DAM-producing method of the present invention. The apparatus comprises a reactor (6) provided with a Da supply port (10), a water supply port (11), an ejector (1) for supplying a quaternizing agent, a reaction mixture extraction port (13), a circulating pump (7) for extracting a part of the reaction
35 mixture, and a stirring blade (9) as required; a flow path for

supplying the reaction mixture extracted from the reactor (6) by the circulating pump (7) to the ejector (1); and a heat exchanger (8) disposed in the flow path, for cooling the extracted reaction mixture. The apparatus may further comprise a finishing tank and an ageing tank.

The method and apparatus of the present invention are described in detail below with reference to Figs. 1 and 2.

Reactor

The reactor used in the present invention is not limited in size, material, and structure as long as raw materials can be reacted to produce DAM.

As the reactor, those whose inner surface is coated and/or lined with glass, Teflon[®], or the like are preferable, so that elution of metal components into the obtained DAM aqueous solution can be avoided and a high-purity DAM aqueous solution can be obtained. When a low-boiling-point quaternizing agent is used as a quaternizing agent, sealable or pressurizable apparatuses are preferably used as a reactor. Examples of such a reactor include autoclaves, etc. Moreover, a reactor equipped with a jacket-type heat exchanger is preferable for easy temperature control (Fig. 2).

In the present invention, the reactor is provided with an ejector for use in supplying the quaternizing agent. Fig. 1 shows a typical example of an ejector. In Fig. 1, (1) indicates an ejector, (2) indicates a nozzle part, and (3) indicates a suction part. The ejector is also referred to as an aspirator. As shown in Fig. 1, a liquid 4 is supplied to the nozzle part (2) at high speed, thereby reducing the pressure inside the ejector. By the resulting driving force, a liquid, gas, or gas-liquid mixture (5) can be supplied from the suction part (3) into the ejector. The reactor may have one or more ejectors, and the optimal number of ejectors can be determined depending on the size of the reactor and the size of the reaction.

In the present invention, a reaction mixture (4) is extracted from the reactor by the circulating pump (7) and then

supplied to the nozzle part (2) of the ejector. By the driving force generated here, a quaternizing agent is supplied from the suction part (3) into the reaction mixture. Generally, a reaction-mixture extraction part is mounted in the bottom of the reactor, and the ejector is mounted in the upper portion of the reactor. The suction part (3) of the ejector may be installed outside the reactor. Fig. 2 shows an example in which the tip (reaction-mixture discharge part) of an introducing pipe of the ejector is placed in the reaction mixture. The tip may also be placed above the surface of the reaction mixture. In a preferable embodiment, the tip of the introducing pipe of the ejector is placed in the reaction mixture. Particularly, the tip of the introducing pipe is preferably placed in an almost intermediate position between the bottom of the reactor and the surface of the reaction mixture.

In the present invention, mixing of the gas and liquid in the reactor is sufficiently carried out by using an ejector alone; however, four-retreating blades, a disk turbine, and like stirring blades may be used together with an ejector, as required.

20 Supply of Raw Materials

In the present invention, each raw material is continuously supplied in a constant amount to the reactor. This can suppress the increase in the amount of reaction heat, compared to batch-type reactions.

25 The quaternizing agent is supplied to the reactor through the suction part of the ejector. Da and water can be supplied in accordance with a standard manner. The raw materials can be supplied in any order. For example, when a low-boiling-point quaternizing agent is used as the quaternizing agent in the present invention, the low-boiling-point quaternizing agent is 30 supplied to the reactor through an ejector. Then, while keeping the pressure in the reactor constant, Da and water are continuously supplied.

Further, when a low-boiling-point quaternizing agent is 35 used as the quaternizing agent of the present invention, using a

sealed reactor, the pressure inside the reactor during the reaction is maintained at 0.1 to 1 MPaG, preferably 0.1 to 0.8 MPaG, and more preferably 0.1 to 0.6 MPaG. As a result, a larger amount of quaternizing agent can be dissolved in the reaction mixture.

In the present invention, it is preferable that a DAM aqueous solution is previously charged in the reactor as a reaction solvent, and then Da, a quaternizing agent, and water are continuously supplied to the reactor. When raw materials are reacted without solvent, crystalline DAM is obtained. Due to the hygroscopic nature, DAM in a crystal form attaches to the inner wall of the reactor, and thus heat removal becomes difficult. Additionally, since DAM takes the form of crystals, an oxygen-free condition occurs, causing the polymerization of DAM. In order to solve this problem, some methods use water as a solvent. In this case, however, the Da of the raw materials is hydrolyzed, leading to decreased product purity. In contrast, the present invention preferably uses a DAM aqueous solution as a reaction solvent to efficiently produce high-purity DAM.

The type of DAM contained in the above DAM aqueous solution is not limited. Usually, the same type of DAM as the obtained DAM is used. The concentration of the DAM aqueous solution is also unlimited and can be selected from a wide range, as required. Usually, the concentration of the DAM aqueous solution is 50 to 84 wt.%, preferably 75 to 84 wt.%, and more preferably 78 to 83 wt.%. Moreover, the concentration of a DAM aqueous solution obtained by supplying raw materials for reaction is adjusted within the above range.

In the present invention, the above-mentioned DAM aqueous solution (reaction solvent) and each raw material can be supplied to the reactor in any order as long as the reaction of the raw materials proceeds. Specifically, in one method, the DAM aqueous solution is previously charged in the reactor before each raw material is continuously supplied; in another method, the DAM aqueous solution is supplied to the reactor simultaneously with

or during the supply of the raw materials.

The rate of supplying each raw material may be suitably determined depending on the properties of each raw material, the size of the reactor, the production volume per unit time, etc.

5 For example, Da is preferably supplied at a rate of 3.0 to 70 mass%/hour, and more preferably 5.0 to 35 mass%/hour, with respect to the mass of the reaction mixture in the reactor.

10 A quaternizing agent is preferably supplied at a rate of 1.0 to 30 mass%/hour, and more preferably 2.0 to 15 mass%/hour, with respect to the mass of the reaction mixture in the reactor.

Water is preferably supplied at a rate of 1.0 to 30 mass%/hour, and more preferably 2.0 to 15 mass%/hour, with respect to the mass of the reaction mixture in the reactor.

15 The rate of supplying each raw material within this range is preferable, because reactions progress with a suitable proportion of raw materials. This enables the production of high-purity DAM with high productivity. The proportion of quaternizing agent to Da is preferably almost equimolar in the reaction mixture.

20 Moreover, Da, a quaternizing agent, and water may be supplied either into the reaction mixture or to the upper space of the reaction mixture in the reactor. As described above, since the quaternizing agent is supplied to the reaction mixture through an ejector, it is preferably supplied into the reaction mixture. Furthermore, in order to avoid deposition of DAC
25 crystals during the supply of Da, which causes obstruction of the introducing pipe, Da is preferably supplied from the tip of a Da-introducing pipe located in the upper space above the reaction mixture.

30 External Heat Exchanger

When each raw material is continuously supplied to the reactor and a quaternizing agent is supplied by an ejector, the amount of reaction heat per unit time tends to increase as the reactivity of the quaternization reaction tends to rise. In the
35 present invention, however, the removal of reaction heat is

extremely immediately carried out, since a part of the reaction mixture is cooled by an external heat exchanger, and is supplied and circulated into the reactor through an ejector. Therefore, control of the reaction temperature is far easier. When using
5 only a reactor equipped with a jacket-type heat exchanger, temperature control is difficult. Particularly, larger production volumes require considerable amounts of heat removal, and thus a larger reactor or a plurality of reactors must be used.

The present invention comprises a flow path for
10 supplying the reaction mixture extracted from the reactor (6) by the circulating pump (7) to the ejector (1), and a heat exchanger (8) disposed in the flow path, for cooling the extracted reaction mixture. The flow path may be provided with one or two or more heat exchangers.

15 A part of the reaction mixture is extracted from the reactor by a circulating pump, and then supplied to a heat exchanger. Heat exchangers made of stainless steel, titanium, or glass lining are preferable. For example, a multi-tube heat exchanger, plate-type heat exchanger, spiral-type heat exchanger,
20 etc. are available.

The cooled reaction mixture passed through the heat exchanger is supplied to the nozzle part of the ejector, while high pressure is maintained. Then, high driving force is generated at the nozzle part for aspirating the quaternizing
25 agent (Fig. 2).

In this way, a large amount of reaction mixture is continuously cooled and circulated through an external heat exchanger in a short time in the present invention. This ensures extremely high cooling efficiency even when the amount of
30 reaction heat increases. Accordingly, it is possible in the present invention to keep the inside of the reactor at a constant temperature in a step to continuously supply each raw material to the reactor and in a reaction step. The temperature in this case is preferably 30 to 80°C, and more preferably 40 to 70°C.

35 Extraction of Reaction Mixture

In the present invention, each raw material is supplied while the reaction mixture is extracted from the reactor. The timing of extracting the reaction mixture is not limited. The reaction mixture may be extracted either simultaneously with the start of the raw material supply, or some time after the start of raw material supply. Moreover, the reaction mixture can be extracted continuously, or may be extracted intermittently, as required. A part of the extracted reaction mixture is supplied to the nozzle part (2) of the ejector (1) through the circulating pump.

The rate of extraction of the reaction mixture may be suitably determined according to the purposes. The reaction mixture is preferably extracted at a rate of 5.0 to 130 mass%/hour, and more preferably 9.0 to 65 mass%/hour, with respect to the mass of the reaction mixture in the reactor. Under such conditions, reactions progress with a suitable raw material proportion, ensuring the production of high-purity DAM with high productivity.

Finishing Tank

In the present invention, it is preferable that the reaction mixture continuously extracted from the reactor be supplied to one or more sealed finishing tanks, and that the pressure of the reaction mixture in the finishing tank be subsequently adjusted to atmospheric pressure. Here, the term finishing tank means a tank connected to the reactor and/or an ageing tank, described later. Such a finishing tank is used to remove unreacted components of the quaternizing agent, etc., in the reaction mixture after reaction, particularly, when using a low-boiling-point quaternizing agent as the quaternizing agent, the unreacted low-boiling-point quaternizing agent, by reducing the pressure of the reaction mixture to atmospheric pressure, and further aerating the reaction mixture as required.

For example, when methyl chloride is used as a low-boiling-point quaternizing agent, unreacted methyl chloride is dissolved in the reaction mixture in the reactor. Unreacted

methyl chloride in the reaction mixture is hydrolyzed to hydrogen chloride, which is unfavorable for the quality of final products. Therefore, it is necessary to remove unreacted methyl chloride. However, if the pressure of the reaction mixture is immediately
5 returned to atmospheric pressure, methyl chloride is vaporized at a stretch, and simultaneously oxygen dissolved in the reaction mixture is also vaporized. As a result, the reaction mixture enters a low-oxygen condition, thereby causing the polymerization of DAM in the reaction mixture.

10 This problem can be resolved by using a finishing tank to gradually reduce the pressure of the extracted reaction mixture. The reaction mixture may be supplied either continuously or intermittently to the finishing tank; however, continuous supply is preferred for the stable production of DAM.

15 The number of finishing tanks may be at least one, but a larger number of tanks are preferable in terms of removing unreacted components after reaction, particularly, when using a low-boiling-point quaternizing agent as the quaternizing agent, the unreacted quaternizing agent. The number of finishing tanks
20 is generally one or more, preferably two or more, and more preferably three or more. The size, material, and structure of the finishing tank are suitably selected according to raw materials used, the production volume of DAM, the purposes, etc. Specifically, those exemplified as the above-mentioned reactor
25 can be used.

 The pressure inside the finishing tank is not limited. Preferably, the pressure is not more than that in the reactor, more preferably atmospheric pressure to 0.7 MPaG, and further preferably atmospheric pressure. The polymerization of DAM in the
30 reaction mixture can be preferably prevented in this pressure range.

 In the present invention, a gas containing at least oxygen can be blown into the reaction mixture transferred to the finishing tank. This is preferable because oxygen is supplied to
35 the reaction mixture in the finishing tank to prevent the

polymerization of DAM, and unreacted components after reaction, particularly, when using a low-boiling-point quaternizing agent as the quaternizing agent, the unreacted low-boiling-point quaternizing agent, can be removed. The type of such a gas is not limited as long as at least oxygen is contained. Generally, air is used.

As described above, in order to suppress the hydrolysis of Da in the reactor, it is preferable to produce DAM at a high concentration. However, to reduce viscosity for easy handling and to prevent crystallization in winter, final products preferably contain 60 to 80 mass% of DAM. In this case, water is preferably added to the finishing tank to adjust the concentration suitable for final products.

Ageing Tank

In the present invention, it is preferable that the reaction mixture continuously extracted from the reactor is further supplied to one or more sealed ageing tanks. The term ageing tank means a tank connected to the reactor and the finishing tank. Such an ageing tank is used to increase the reaction rate for producing DAM (to age) more than that in the reaction mixture extracted from the reactor. The reactor, ageing tank, and finishing tank are usually connected in this order.

Specifically, a reaction can be aged by, for example, maintaining the pressure in the ageing tank below the pressure in the reactor, as well as reducing the temperature in the ageing tank below the temperature in the reactor.

The use of an ageing tank is preferable because even when reaction does not sufficiently progress in the reactor due to reaction conditions, etc., the reaction can sufficiently progress in the ageing tank. In addition, residual unreacted raw materials in products can be avoided. Although the reaction mixture may be supplied either continuously or intermittently to the ageing tank, continuous supply is preferred for the stable production of DAM.

The pressure and temperature in the ageing tank are not

limited. The pressure in the ageing tank is generally lower than the pressure in the reactor. When using an ageing tank, the pressure in the ageing tank can be adjusted to atmospheric pressure. The pressure is preferably atmospheric pressure to 0.8 MPaG, and more preferably atmospheric pressure to 0.6 MPaG. The temperature in the ageing tank may be generally below the temperature in the reactor, and is preferably 30 to 60°C. The pressure in the ageing tank is preferably maintained constant. Further, the temperature in the ageing tank is also preferably maintained constant.

In the present invention, it is preferable that the reaction mixture, after being supplied to the ageing tank for ageing, is further supplied to the finishing tank. In addition to the effect of the ageing tank, this provides the effect of the finishing tank. That is, the polymerization of DAM in the reaction mixture is prevented, and unreacted components after reaction, particularly, when using a low-boiling-point quaternizing agent as the quaternizing agent, the unreacted low-boiling-point quaternizing agent, can be removed.

In this case, it is preferable to gradually reduce the pressures in the reactor, ageing tank, and finishing tank. This can prevent the polymerization of DAM in the low-oxygen conditioned reaction mixture, which occurs because the quaternizing agent dissolved in the reaction mixture is vaporized at a stretch, and simultaneously oxygen dissolved in the reaction mixture is also vaporized. The pressure in each tank in this case may be adjusted to a suitable level for the reactor, ageing tank, and finishing tank. When the ageing tank is adjusted to atmospheric pressure, only aeration and the addition of water may be performed in the finishing tank.

The number of ageing tanks in the present invention may be at least one; a larger number of tanks is preferable, however, in terms of productivity improvement. The number of ageing tanks is generally one or more, preferably two or more, and more preferably three or more. When two or more ageing tanks are

connected, it is preferable to gradually reduce the pressure. Specifically, when two ageing tanks are connected, the pressure is preferably 0.05 to 0.8 MPaG in the first ageing tank, and atmospheric pressure to 0.5 MPaG in the second ageing tank.

5 Moreover, the size, material, and structure of the ageing tank may be suitably selected according to the raw materials used, the production volume of DAM, the purposes, etc. Specifically, those exemplified as the reactor can be used.

10 The residence time ($[\text{the mass of reaction mixture}] / [\text{the rate of extraction of reaction mixture (mass/hour)}]$) in the present invention can be determined to cover various ranges, depending on reaction conditions, the number of finishing tanks or ageing tanks, and other conditions. Usually, for a two-tank continuous process using a finishing tank, the above residence
15 time is generally 4 hours or more, preferably 4.5 hours or more, and more preferably 5 hours or more. For a three-tank continuous process using an ageing tank and a finishing tank, the residence time is generally 1 hour or more, preferably 1.5 hours or more, more preferably 2 hours or more, and further more preferably 2 to
20 15 hours. For the residence time in this range, the amount of residual acrylic acid derived from the above-mentioned raw materials is preferably decreased. The amount of residual acrylic acid derived from the raw materials in DAM obtained in the present invention is usually 1,500 ppm or less, preferably 1,200
25 ppm or less, and more preferably 1,000 ppm or less.

Examples

The present invention is described in detail below with reference to examples. Note that the term "%" means "mass%".

30 Example 1

Using a reactor as shown in Fig. 2, a methyl chloride adduct (hereinafter referred to as "DAC") of dimethylaminoethyl acrylate was produced.

As a reaction tank (6), a 10 m³ tank reactor comprising
35 an agitator (9) inside, a jacket-type heat exchanger (14), and a

glass-lined inner wall was used. The reaction tank (6) further comprises an ejector (1), a multi-tube heat exchanger (8) as the external heat exchanger (overall heat transfer coefficient $(U) \times$ heat transfer area $(A) = 5,972 \text{ kcal/hr}^\circ\text{C}$), and a circulating pump (7). In the multi-tube heat exchanger, water at 20°C cooled with brine was circulated by a pump (not shown).

After 8,000 kg of 79% DAC aqueous solution was charged in the reaction tank (6), the tank was sealed and warmed to 50°C .

Subsequently, while stirring the DAC aqueous solution with the agitator (9) of the reaction tank (6), the circulating pump (7) was activated to start circulation by supplying the DAC aqueous solution to a nozzle part of the ejector (1) through the multi-tube heat exchanger (8). Methyl chloride (hereinafter abbreviated as "MC") was supplied from a supply pipe (12) to the sealed reaction tank (6) via a suction part (3) of the ejector (1) so that the pressure inside the reaction tank (6) is 0.28 MPaG. MC was continuously supplied at a rate of 274 kg/hour, and in order to maintain the pressure inside the reaction tank (6) at 0.28 MPaG, only the amount of MC consumed by reaction was supplied.

Then, dimethylaminoethyl acrylate (hereinafter referred to as "DA") was continuously supplied at a rate of 730 kg/hour to the reaction tank (6) via a supply pipe (10). At the same time, water was continuously supplied at a rate of 263 kg/hour to the reaction tank (6) via a supply pipe (11). Using the jacket-type heat exchanger (14) and multi-tube heat exchanger (8), cooling water was circulated so that the reaction temperature was 50°C . The obtained DAC aqueous solution, of which the DAC concentration was 80%, was continuously extracted from a pipe (13).

The continuously-extracted DAC aqueous solution was supplied to an ageing tank (not shown), and aged at a reduced pressure of 0.1 MPaG. After ageing, the reaction mixture was further supplied to a finishing tank (not shown), and the pressure was adjusted to atmospheric pressure. Then, water was added to adjust the DAC concentration to 79%.

As a result, a 79% DAC aqueous solution could be stably and continuously produced at a reaction temperature of 50°C and at a rate of 1,250 kg/hour.

Example 2

5 DAC was produced in the same manner as in Example 1 except that DA, MC, and water were supplied at a rate of 876 kg/hour, 329 kg/hour, and 316 kg/hour, respectively.

 The result showed that although the capacity of the cooling water-circulating pump was full, a 79% DAC aqueous
10 solution could be stably and continuously produced at a reaction temperature of 51°C and at a rate of 1,500 kg/hour.

Comparative Example 1

 DAC was produced in the same manner as in Example 1 except that a reactor having no multi-tube heat exchanger was
15 used; DA, MC, and water were supplied at a rate of 526 kg/hour, 197 kg/hour, and 190 kg/hour, respectively; and only the jacket-type heat exchanger (14) was used to circulate cooling water so that the reaction temperature was 50°C.

 The continuous production of a 79% DAC aqueous solution
20 was started at a rate of 900 kg/hour. In the middle of the production, however, the reaction temperature exceeded the target temperature, and reached 54°C. As the reaction temperature continued to climb, cooling was insufficient, and thus the temperature was uncontrollable. Therefore, the operation was
25 halted.

Comparative Example 2

 Since a temperature increase occurred in the reaction mixture in Comparative Example 1, the continuous production of DAC was carried out with lower productivity.

30 DAC was produced in the same manner as in Comparative Example 1 except that DA, MC, and water were supplied at a rate of 394 kg/hour, 148 kg/hour, and 142 kg/hour, respectively.

 The result was that although the capacity of the cooling water-circulating pump was full, a 79% DAC aqueous
35 solution could be stably and continuously produced at a reaction

temperature of 51°C and at a rate of 675 kg/hour. However, this stable and continuous production was only possible with about half the production capacity of the Examples.

Table 1 shows the results of the Examples and

5 Comparative Examples.

Table 1

		Example 1	Example 2	Comparative Example 1	Comparative Example 2
Operational conditions	Setup load	1,250	1,500	900	675
	DAC 79% product (kg/hr)				
	DA supply (kg/hr)	730	876	526	394
	MC supply (kg/hr)	274	329	197	148
Heat-removal state	Setting of inside temperature (°C)	50	50	50	50
	Temperature of cooling water (°C)	26	28	24	23
	Actual inside temperature (°C)	50	51	54	51
	Cooling condition	Jacket-type and external heat exchangers	Jacket-type and external heat exchangers	Jacket-type heat exchanger only	Jacket-type heat exchanger only
	Temperature control	No problem	Cooling water-circulating pump full bore	Inside temperature exceeding 54°C, test stopped	Cooling water-circulating pump full bore
Amount of heat removal	Heat value (kcal/hr)	181,000	217,200	130,320	97,740
	Jacket-type heat exchanger (kcal/hr)	82,000	79,350	103,500	96,600
	External heat exchanger (kcal/hr)	143,328	137,356	-	-
	Total (kcal/hr)	226,128	216,706	103,500	96,600

Capacity of jacket-type heat exchanger: overall heat transfer coefficient (U) x heat transfer area (A) = 3,450

kcal/hr.°C

Capacity of external heat exchanger: overall heat transfer coefficient (U) x heat transfer area (A) = 5,972

kcal/hr.°C

The method of the present invention using an external heat exchanger resulted in sufficient reaction heat removal and high productivity.

On the other hand, the results of Comparative Examples 1 and 2, using only a jacket-type heat exchanger, are as follows. First, in Comparative Example 1, utilizing a smaller production volume of DAC aqueous solution (900 kg/hr) than the Examples, methyl chloride was continuously supplied to the same reactor as the Examples. However, the removal of reaction heat was insufficient, making temperature control difficult. Even if the production capacity is increased by force without using an external heat exchanger, temperature control is impossible, resulting in a risk. In Comparative Example 2, starting with a production volume of 675 kg/hr, temperature control was possible. Since the production volume was about half that of the Examples, a larger reactor, or two reactors, is necessary to secure the same production volume as the Examples.

Next, focusing on the amount of heat removal, 226,128 and 216,706 kcal/hr of heat removal were possible in Examples 1 and 2, whereas the amount of heat removal possible in Comparative Examples 1 and 2 was only 103,500 and 96,600 kcal/hr, which was about half that of the Examples.

Industrial Applicability

According to the DAM-producing method of the present invention, the temperature rise in the reactor can be controlled by efficiently cooling the reaction mixture, and hence high-purity DAM can be produced with high productivity.

Claims

1. A method of producing a dialkylaminoalkyl (meth)acrylate quaternary salt, comprising continuously supplying a dialkylaminoalkyl (meth)acrylate, a quaternizing agent, and
5 water to a reactor, while continuously extracting a reaction mixture from the reactor,

wherein a part of the reaction mixture extracted from the reactor is cooled by passing through an external heat exchanger provided outside the reactor, the cooled reaction
10 mixture is supplied to a nozzle part of an ejector while high pressure is maintained, and the cooled reaction mixture is supplied to the reactor together with the quaternizing agent through the ejector.

15 2. A method according to claim 1, wherein the reactor is previously charged with a dialkylaminoalkyl (meth)acrylate quaternary salt aqueous solution before continuously supplying a dialkylaminoalkyl (meth)acrylate, a quaternizing agent, and water.

20 3. A method according to claim 1 or 2, wherein the reaction mixture is maintained at a temperature of 30 to 80°C during reaction.

25 4. A method according to any one of claims 1 to 3, wherein the reactor is a sealed reactor, the quaternizing agent is a quaternizing agent having a boiling point of 25°C or lower, and the pressure inside the reactor is maintained at 0.10 to 1 MPaG during reaction.

30 5. A method according to any one of claims 1 to 4, wherein the dialkylaminoalkyl (meth)acrylate is dimethylaminoethyl (meth)acrylate, and the quaternizing agent is methyl chloride.

35 6. A method according to any one of claims 1 to 5, further comprising supplying the reaction mixture continuously extracted from the reactor to one or more sealed tanks, and

gradually adjusting the pressure of the reaction mixture in the tank to atmospheric pressure.

7. An apparatus for producing a dialkylaminoalkyl
5 (meth)acrylate quaternary salt, comprising:

a reactor (6) equipped with a dialkylaminoalkyl
(meth)acrylate supply port (10), a water supply port (11), an
ejector (1) for supplying a quaternizing agent, a reaction
mixture extraction port (13), a circulating pump (7) for
10 extracting a part of the reaction mixture, and a stirring blade
(9) as required;

a flow path for supplying the reaction mixture
extracted from the reactor (6) by the circulating pump (7) to the
ejector (1); and

15 a heat exchanger (8) disposed in the flow path, for
cooling the extracted reaction mixture.