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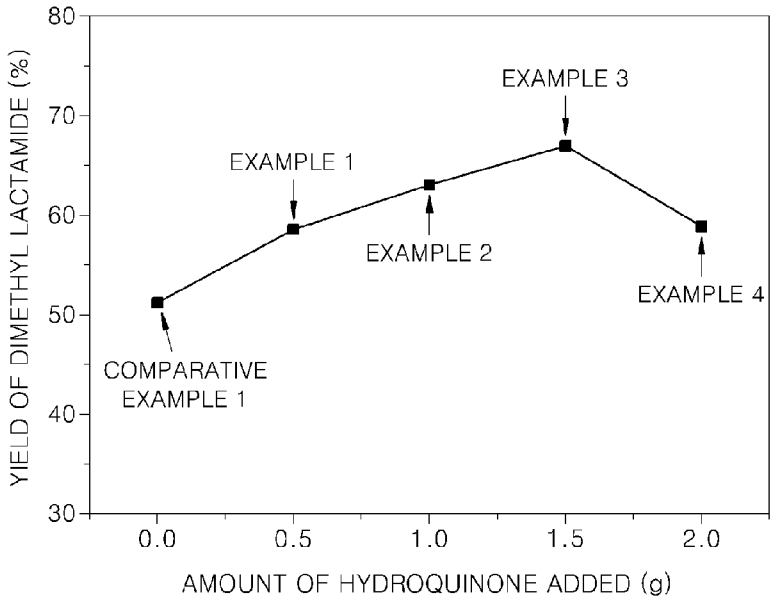
(54) Title: METHOD OF PREPARING DIALKYL LACTAMIDES

[Fig. 1]



(57) Abstract: A method of preparing dialyl lactamides. The method of preparing dialkyl lactamides includes preparing ammonium lactate by reacting at least one compound selected from the group consisting of lactic acid and esters thereof with at least one amine compound, preparing dialkyl lactamides by heat-treating the ammonium lactate, and adding at least one of a polymerization inhibitor and an anti-polymerization agent to a reaction mixture of at least one of the preparing of the ammonium lactate and the preparing of the dialkyl lactamides.

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Description

Title of Invention: METHOD OF PREPARING DIALKYL LACTAMIDES

Technical Field

[1] The present invention relates to a method of preparing dialkyl lactamides, and more particularly, to a method of preparing dialkyl lactamides, which includes adding at least one of a polymerization inhibitor and an anti-polymerization agent to reactants and/or a reaction mixture.

Background Art

[2] Dialkyl lactamides are substances that can replace dialkylacetamides. Due to their high solubility in resins, dialkylacetamides are widely used in the manufacture of synthetic fibers and films and in fiber coating. Recently, demand for dialkylacetamides has rapidly increased with a new market for LCD cleaners and with increased demand for clothes in accordance with the world economic recovery. However, due to their high volatility, dialkylacetamides cause environmental pollution and are toxic to human beings. Since dialkyl lactamides can replace dialkylacetamides without the foregoing problems, demand for dialkyl lactamides will greatly increase as the growth of semiconductor and LCD industries continues.

Disclosure of Invention

Technical Problem

[3] The present invention provides a method of preparing dialkyl lactamides, the method including adding at least one of a polymerization inhibitor and an anti-polymerization agent to reactants and/or a reaction mixture before and/or during a reaction.

Solution to Problem

[4] According to an aspect of the present invention, there is provided a method of preparing dialkyl lactamides, the method including:

[5] preparing ammonium lactate by reacting at least one compound selected from the group consisting of lactic acid and esters thereof with at least one amine compound represented by Formula 1 below;

[6] preparing dialkyl lactamides by heat-treating the ammonium lactate; and

[7] adding at least one of a polymerization inhibitor and an anti-polymerization agent to a reaction mixture of at least one of the preparing of the ammonium lactate and the preparing of the dialkyl lactamides:

[8] Formula 1

[9] HNR_1R_2

[10] where R_1 and R_2 are each independently a substituted or unsubstituted C_1-C_{100} alkyl

group.

[11] The esters of lactic acid may include at least one compound selected from the group consisting of lactide and monobasic esters of lactic acid.

[12] The monobasic esters of lactic acid may be represented by Formula 2 below:

[13] Formula 2

[14] R_3-OC(=O)CH(OH)CH_3

[15] where R_3 is a substituted or unsubstituted C_1-C_{10} alkyl group, a substituted or unsubstituted C_1-C_{10} alkoxy group, a substituted or unsubstituted C_6-C_{24} aryl group, a substituted or unsubstituted C_6-C_{24} cycloalkyl group, a substituted or unsubstituted C_6-C_{24} heterocycloalkyl group, or a substituted or unsubstituted acyl group.

[16] The polymerization inhibitor and the anti-polymerization agent may include at least one compound selected from the group consisting of hydroquinone monomethyl ether, phenothiazine, p-benzoquinone, toluhydroquinone, 4-tertiary butyl catechol, hydroquinone, mono-tert-butylhydroquinone, chloranil, p-nitrosophenyl, 3-mercaptopropyltrimethoxysilane, 3-(methacryloxypropyl)trimethoxysilane, and 4-hydroxy-2,2,6,6-tetramethyl-1-piperidinyloxy.

[17] The amount of the amine compound may be in a range of 100 to 1500 parts by mole based on 1 part by mole of a total of lactic acid and esters thereof.

[18] The total amount of the polymerization inhibitor and the anti-polymerization agent may be in a range of 0.1 to 10 parts by weight based on 100 parts by weight of the total of lactic acid and esters thereof.

[19] The preparing of the ammonium lactate may be performed at a temperature in a range of 50 to 70℃.

[20] The preparing of the dialkyl lactamides may be performed at a temperature in a range of 100 to 200℃.

Advantageous Effects of Invention

[21] According to an embodiment of the present invention, there is provided a method of preparing dialkyl lactamides with high yield by inhibiting a side-reaction in which lactic acid, esters of lactic acid, lactate anions, an amine compound, or any combination thereof form an oligomer by adding at least one of a polymerization inhibitor and an anti-polymerization agent to reactants and/or a reaction mixture before and/or during the reaction.

Brief Description of Drawings

[22] The above and other features and advantages of the present invention will become more apparent by describing in detail exemplary embodiments thereof with reference to the attached drawing in which:

[23] FIG. 1 is a graph illustrating yields of dimethyl lactamides according to the amount

of hydroquinone used as a polymerization inhibitor according to Examples 1 to 4 and Comparative Example 1.

Mode for the Invention

[24] Hereinafter, a method of preparing dialkyl lactamides according to an embodiment of the present invention will be described more fully with reference to the accompanying drawing, in which exemplary embodiments of the invention are shown. The "dialkyl lactamides" used herein refers to dialkyl lactamide and/or derivatives thereof. Here, the "dialkyl lactamides" may also include N,N-dialkyl lactamides.

[25] A method of preparing dialkyl lactamides according to an embodiment of the present invention includes preparing ammonium lactate by a reaction between at least one compound selected from the group consisting of lactic acid and esters thereof and at least one amine compound represented by Formula 1 below, and preparing dialkyl lactamides by heat-treating the ammonium lactate.

[26] Formula 1

[27] HNR_1R_2

[28] In Formula 1, R_1 and R_2 are each independently a substituted or unsubstituted C_1-C_{100} alkyl group.

[29] The term "substituted" used herein indicates that a hydrogen atom of a compound is substituted with a halogen atom, a hydroxy group, an alkyl group, an alkoxy group, an amine group, or a combination thereof.

[30] The method of preparing dialkyl lactamides further includes adding at least one of a polymerization inhibitor and an anti-polymerization agent to the reaction mixture of at least one of the preparing of ammonium lactate and the preparing of the dialkyl lactamides. Here, the "reaction mixture of the preparing of ammonium lactate" includes at least lactic acid and/or esters thereof, and the "reaction mixture of the preparing of dialkyl lactamides" includes at least ammonium lactate.

[31] The preparation of dialkyl lactamides according to the method may be carried out in the presence of an acetylation catalyst such as $M(ClO_4)_2 \cdot 6H_2O$ (M: Mn, Co, Ni, Cu, Zn) or in the absence of a catalyst.

[32] The preparing ammonium lactate may be performed at a temperature in the range of 50 to 70℃. If the temperature is within the range described above, ammonium lactate may be obtained with high yield without an excessive evaporation loss of the amine compound.

[33] The preparing of dialkyl lactamides may be performed at a temperature in the range of 100 to 200℃. If the temperature is within the range described above, dialkyl lactamides may be obtained with high yield without an excessive energy loss by over-heating since water, which is generated as a by-product, is efficiently evaporated. In

the preparation of the dialkyl lactamides, water is generated as a by-product and it is immediately evaporated due to the high reaction temperature, to be discharged from a reactor.

[34] The esters of lactic acid may include at least one compound selected from the group consisting of lactide and monobasic esters of lactic acid.

[35] The monobasic esters of lactic acid may be represented by Formula 2 below.

[36] Formula 2

[37] R_3-OC(=O)CH(OH)CH_3

[38] In Formula 2, R_3 is a substituted or unsubstituted C_1-C_{10} alkyl group, a substituted or unsubstituted C_1-C_{10} alkoxy group, a substituted or unsubstituted C_6-C_{24} aryl group, a substituted or unsubstituted C_6-C_{24} cycloalkyl group, a substituted or unsubstituted C_6-C_{24} heterocycloalkyl group, or a substituted or unsubstituted acyl group.

[39] For example, the monobasic ester of lactic acid may be ethyl lactate.

[40] The amine compound may be dimethylamine. In this case, N,N-dimethyl lactamide may be prepared.

[41] The amount of the amine compound may be in the range of 100 to 1500 parts by mole based on 1 part by mole of a total of lactic acid and esters thereof. If the amount of the amine compound is within the range described above, ammonium lactate may be obtained with high yield without an excessive consumption of the amine compound. For example, the amine compound may be injected in a gaseous phase into a reactor filled with lactic acid and/or esters thereof, and optionally, with a polymerization inhibitor and/or an anti-polymerization agent. In this regard, the polymerization inhibitor and/or the anti-polymerization agent which are dissolved in lactic acid and/or esters thereof may be injected into the reactor. The reactor may be a batch reactor. The gaseous amine compound is continuously injected into the reactor to be in contact with the reaction mixture for a predetermined period of time and continuously discharged out of the reactor.

[42] The polymerization inhibitor or the anti-polymerization agent inhibits or prevents lactic acid, esters of lactic acid, lactate anion, and/or the amine compound alone or at least two thereof from being involved in a reaction to form an oligomer. Accordingly, the yield of the dialkyl lactamides as shown in Equation 1 below increases.

[43] Equation 1

[44] Yield of dialkyl lactamide (%) = (number of moles of generated dialkyl lactamide)/(number of moles of injected lactic acid and esters thereof) 100

[45]

[46] The polymerization inhibitor and the anti-polymerization agent may include at least one compound selected from the group consisting of hydroquinone monomethyl ether, phenothiazine, p-benzoquinone, toluhydroquinone, 4-tertiary butyl catechol, hy-

droquinone, mono-tert-butylhydroquinone, chloranil, p-nitrosophenyl, 3-mercaptopropyltrimethoxysilane, 3-(methacryloxypropyl)trimethoxysilane, and 4-hydroxy-2,2,6,6-tetramethyl-1-piperidinyloxy.

[47] The total amount of the polymerization inhibitor and the anti-polymerization agent may be in the range of 0.1 to 10 parts by weight based on 100 parts by weight of the total of lactic acid and esters thereof. If the total amount of the polymerization inhibitor and the anti-polymerization agent is within the range described above, dialkyl lactamides may be obtained with high yield without an excessive consumption of the polymerization inhibitor and the anti-polymerization agent.

[48] Hereinafter, one or more embodiments will be described in detail with reference to the following examples. However, these examples are not intended to limit the purpose and scope of the invention.

[49] Examples 1 to 4

[50] Preparation of dimethyl lactamide using lactic acid and dimethylamine

[51] First, a predetermined amount of hydroquinone (as shown in Table 1 below) was completely dissolved in 83.3 g of lactic acid (85% by weight). Then, the solution was added to a 150 ml batch reactor equipped with a gas inlet, a gas outlet, and a sampling port, and a reaction was carried out while continuously flowing a gaseous dimethylamine into the solution via the gas inlet at a rate of 250 ml/min. In this regard, the temperature of the reactor was maintained at 60℃ for 90 minutes, and the reactor was heated to 170℃ and maintained at the same temperature for 170 minutes. Then, unreacted dimethylamine was discharged out of the reactor via the gas outlet.

[52] Table 1

[Table 1]

	Example 1	Example 2	Example 3	Example 4
Amount of hydroquinone (g)	0.5	1.0	1.5	2.0

[53]

[54] Comparative Example 1

[55] Dimethyl lactamide was prepared in the same manner as in Examples 1 to 4, except that hydroquinone was not used.

[56] Evaluation Example

[57] In Examples 1 to 4 and Comparative Example 1, samples were collected from the reactor every 10 minutes after the reactor reached 170℃, and constituents of the samples and compositions of the constituents were analyzed using gas chromatography. Using the steady state data among the analyzed data, the yields of dimethyl lactamide were calculated by Equation 1 and the results are shown in Table 2

and FIG. 1. The yields of dimethyl lactamide reached the steady state after about 130 minutes since the reaction was initiated at 170℃ in Examples 1 to 4 and Comparative Example 1.

[58] Table 2

[Table 2]

	Example 1	Example 2	Example 3	Example 4	Comparative Example 1
Yield of dimethyl lactamide (%)	58.6	62.9	66.9	58.8	51.7

[59]

[60] Referring to Table 2, the yield of dimethyl lactamide according to Examples 1 to 4 was greater than that according to Comparative Example 1.

[61] In addition, referring to FIG. 1, the yield of dimethyl lactamide did not always increase as the amount of hydroquinone increased. The yield of dimethyl lactamide was maximized when the amount of hydroquinone was optimized.

[62] While the present invention has been particularly shown and described with reference to exemplary embodiments thereof, it will be understood by those of ordinary skill in the art that various changes in form and details may be made therein without departing from scope of the present invention as defined by the following claims.

Claims

[Claim 1] A method of preparing dialkyl lactamides, the method comprising:
preparing ammonium lactate by reacting at least one compound selected from the group consisting of lactic acid and esters thereof with at least one amine compound represented by Formula 1 below;
preparing dialkyl lactamides by heat-treating the ammonium lactate; and
adding at least one of a polymerization inhibitor and an anti-polymerization agent to a reaction mixture of at least one of the preparing of the ammonium lactate and the preparing of the dialkyl lactamides:
Formula 1
HNR_1R_2
where R_1 and R_2 are each independently a substituted or unsubstituted C_1-C_{100} alkyl group.

[Claim 2] The method of claim 1, wherein the esters of lactic acid comprise at least one compound selected from the group consisting of lactide and monobasic esters of lactic acid.

[Claim 3] The method of claim 2, wherein the monobasic esters of lactic acid are represented by Formula 2 below:
Formula 2
R_3-OC(=O)CH(OH)CH_3
where R_3 is a substituted or unsubstituted C_1-C_{10} alkyl group, a substituted or unsubstituted C_1-C_{10} alkoxy group, a substituted or unsubstituted C_6-C_{24} aryl group, a substituted or unsubstituted C_6-C_{24} cycloalkyl group, a substituted or unsubstituted C_6-C_{24} heterocycloalkyl group, or a substituted or unsubstituted acyl group.

[Claim 4] The method of claim 1, wherein the polymerization inhibitor and the anti-polymerization agent comprise at least one compound selected from the group consisting of hydroquinone monomethyl ether, phenothiazine, p-benzoquinone, toluhydroquinone, 4-tertiary butyl catechol, hydroquinone, mono-tert-butylhydroquinone, chloranil, p-nitrosophenyl, 3-mercaptopropyltrimethoxysilane, 3-(methacryloxypropyl)trimethoxysilane, and 4-hydroxy-2,2,6,6-tetramethyl-1-piperidinyloxy.

[Claim 5] The method of claim 1, wherein the amount of the amine compound is in a range of 100 to 1500 parts by mole based on 1 part by mole of a

total of lactic acid and esters thereof.

[Claim 6] The method of claim 1, wherein the total amount of the polymerization inhibitor and the anti-polymerization agent is in a range of 0.1 to 10 parts by weight based on 100 parts by weight of the total of lactic acid and esters thereof.

[Claim 7] The method of claim 1, wherein the preparing of the ammonium lactate is performed at a temperature in a range of 50 to 70℃.

[Claim 8] The method of claim 1, wherein the preparing of the dialkyl lactamides is performed at a temperature in a range of 100 to 200℃.

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[Fig. 1]