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NEUTRALIZATION/WATER SEPARATION TANK FOR ESTERIFIED PRODUCT AND NEUTRALIZATION/WATER SEPARATION METHOD FOR ESTERIFIED PRODUCT

Abstract

A neutralization/water separation tank for an esterified product including: a neutralization part in which a crude product mixture containing alcohol and an ester compound, a neutralizing agent, and water are put to produce a neutralized mixture; a water separation part which divides the neutralized mixture into a floating layer and an aqueous layer; a first partition wall which extends upward from a bottom surface to provide an upper passage and by which the neutralization part and the water separation part are partitioned; and a second partition wall which extends downward from a ceiling to provide a lower passage, wherein the water separation part includes: a first water separation part into which the neutralized mixture is introduced from the neutralization part through the upper passage; and a second water separation part into which the neutralized mixture is introduced from the first water separation part through the lower passage, wherein the first water separation part and the second water separation part are partitioned by the second partition wall.

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Background/Summary

CROSS-REFERENCE TO RELATED APPLICATION [0001] The present application is a Divisional of U.S. patent application Ser. No. 17/605,184, filed on Oct. 20, 2021, which is a National Phase entry pursuant to 35 U.S.C. § 371 of International Application No. PCT/KR2020/008573, filed on Jul. 1, 2020, and claims the benefit of and priority to Korean Patent Application No. 10-2019-0115693, filed on Sep. 19, 2019, all of which are hereby incorporated by reference in their entirety for all purposes as if fully set forth herein.

TECHNICAL FIELD

[0002] The present invention relates to a neutralization/water separation tank for an esterified product and a neutralization/water separation method for an esterified product, and more particularly, to a neutralization/water separation tank for an esterified product and a neutralization/water separation method for an esterified product, in which, when preparing a plasticizer, both neutralization reaction and water separation occur well to improve efficiency.

BACKGROUND ART

[0003] A phthalate-based plasticizer accounts for 92% of the global plasticizer market until the 20th century (see Mustafizur Rahman and Christopher S. Brazel “The plasticizer market: an assessment of traditional plasticizers and research trends to meet new challenges” Progress in Polymer Science 2004, 29, 1223-1248). The phthalate-based plasticizer is an additive used for imparting flexibility, durability and cold resistance mainly to polyvinyl chloride (hereinafter, referred to as PVC) and lowering the viscosity during melting to improve processability. The phthalate-based plasticizer is added in various amounts to PVC and widely used in various applications from rigid products such as rigid pipes to soft products which may be used for such as food packaging materials, blood bags, flooring materials, etc. due to its soft and good flexibility. Therefore, it is more closely related to real life than any other material, and the direct contact with the human body may not avoidable.

[0004] However, in spite of the compatibility of the phthalate-based plasticizer with PVC and its excellent capability to impart flexibility, it has been argued recently about harmfulness of the PVC product containing the phthalate-based plasticizer that the phthalate-based plasticizer may leak out of the PVC product when used in real life, and act as a presumed endocrine disrupting (environmental hormone) substance and a carcinogen of the level of heavy metals (see N. R. Janjua et al. “Systemic Uptake of Diethyl Phthalate, Dibutyl Phthalate, and Butyl Paraben Following Whole-body Topical Application and Reproductive and Thyroid Levels Hormone in Humans” Environmental Science and Technology 2008, 42, 7522-7527). Especially, since the report about

the leakage of di-(2-ethyl hexyl) phthalate (DEHP), which has been the most used phthalate-based plasticizer in the US in the 1960s, out of the PVC product, the interest in environmental hormones have been added in the 1990s and global environmental regulations as well as various studies on hazards of the phthalate-based plasticizer to human have been started.

[0005] Therefore, in order to cope with environmental hormone problems and environmental regulations due to the leakage of the phthalate-based plasticizer, many researchers have been conducting research to develop a new, alternative, non-phthalate-based plasticizer which is free of phthalic anhydride used in the production of phthalate-based plasticizers or a leakage inhibition technology which may inhibit the leakage of the phthalate-based plasticizer to greatly reduce the hazards to human and be in accordance with environmental standards.

[0006] As a non-phthalate-based a plasticizer, terephthalate-based plasticizer has been getting the spot-light, because it is equivalent to the phthalate-based plasticizer in terms of physical properties, but is free of environmental issues. A variety of terephthalate-based plasticizers have been developed and research on the development of a terephthalate-based plasticizer having excellent physical properties, as well as researches on facilities for preparing such the terephthalate-based plasticizer have been actively conducted. In terms of process design, more efficient, economical and simple process design has been required.

DISCLOSURE OF THE INVENTION

Technical Problem

[0007] An object of the present invention is to provide a neutralization/water separation tank for an esterified product and a neutralization/water separation method for an esterified product, in which, when preparing a plasticizer, both neutralization reaction and water separation occur well to improve efficiency.

[0008] The objects of the present invention are not limited to the aforementioned object, but other objects not described herein will be clearly understood by those skilled in the art from descriptions below.

Technical Solution

[0009] A neutralization/water separation tank for an esterified product according to an embodiment of the present invention for achieving the above object includes: a neutralization part in which a crude product mixture containing alcohol and an ester compound, a neutralizing agent, and water are put to produce a neutralized mixture; a water separation part which divides the neutralized mixture into a floating layer and an aqueous layer; a first partition wall which extends upward from a bottom surface to provide an upper passage and by which the neutralization part and the water separation part are partitioned; and a second partition wall which extends downward from a ceiling to provide a lower passage, wherein the water separation part includes: a first water separation part into which the neutralized mixture is introduced from the neutralization part through the upper passage; and a second water separation part into which the neutralized mixture is introduced from the first water separation part through the lower passage, wherein the first water separation part and the second water separation part are partitioned by the second partition wall.

[0010] In addition, the neutralization/water separation tank can further include a first discharge pump configured to discharge the resultant product contained in the floating layer from a sidewall of the second water separation part to the outside.

[0011] In addition, the neutralization/water separation tank can further include a second discharge pump configured to discharge the precipitated salt contained in the aqueous layer from the bottom surface of the water separation part to the outside.

[0012] In addition, the neutralization/water separation tank can further include a circulation pump configured to discharge the neutralized mixture from the neutralization part and put the neutralized mixture again into the neutralization part.

[0013] In addition, the neutralization/water separation tank can further include a propeller stirrer installed in the neutralization part to stir the neutralized mixture.

[0014] In addition, the bottom surface of the water separation part can be gradually inclined downward toward a center thereof.
[0015] In addition, the inclination can have a range of 5° to 45°.
[0016] In addition, the bottom surface of the neutralization part can be gradually inclined downward toward a center thereof.
[0017] In addition, the upper passage can have a height of 10% to 50% of a height of the first partition wall.
[0018] In addition, the lower passage can have a height of 10% to 50% of a height of the second partition wall.
[0019] A neutralization/water separation method for an esterified product according to an embodiment of the present invention for achieving the above object includes: a step of putting a crude product mixture containing alcohol and an ester compound, a neutralizing agent, and water into a neutralization part of a neutralization/water separation tank; a step of mixing the crude product mixture, the neutralizing agent, and the water to produce a neutralized mixture; a step of introducing the neutralization mixture into a first water separation part of the water separation part through an upper passage; a step of introducing the neutralization mixture into a second water separation part of the water separation part through a lower passage; a step of dividing the neutralization mixture into a floating layer and an aqueous layer; and a step of discharging the resultant product contained in the floating layer to the outside.
[0020] In addition, in the step of producing the neutralized mixture, a circulation pump can discharge the neutralized mixture from the neutralization part and put the neutralized mixture again into the neutralization part.
[0021] In addition, in the step of producing the neutralized mixture, the neutralized mixture can be stirred using a propeller stirrer.
[0022] In addition, a bottom surface of the water separation part can be gradually inclined downward toward a center thereof, and precipitated salt contained in the aqueous layer can be collected to the center of the bottom surface of the water separation part.
[0023] Particularities of other embodiments are included in the detailed description and drawings.
Advantageous Effects
[0024] The embodiments of the present invention can have at least the following effects.
[0025] Since the neutralization part, in which the neutralization reaction occurs, and the water separation part, in which the water separation occurs, are provided separately, both the neutralization reaction and the water separation can occur well to improve the efficiency.
[0026] In addition, since the neutralization part and the water separation part are combined into the one neutralization/water separation tank, the separate transfer pump may not be required, and thus, the total volume may not increase, the structure may be simplified, and the problem of the salt accumulation can be prevented from occurring.
[0027] The effects of the prevent invention are not limited by the aforementioned description, and thus, more varied effects are involved in this specification.

Description

BRIEF DESCRIPTION OF THE DRAWINGS
[0028] FIG. **1** is a schematic view of a neutralization/water separation tank, which is combined to allow both neutralization and water separation to occur together, according to a related art.
[0029] FIG. **2** is a schematic view of a neutralization/water separation device, in which a neutralization tank and a water separation tank are separated from each other, according to the related art.
[0030] FIG. **3** is a schematic view of a neutralization/water separation tank according to a first

embodiment of the present invention.
[0031] FIG. **4** is a schematic view of a neutralization/water separation tank according to a second embodiment of the present invention.
[0032] FIG. **5** is a schematic view of a neutralization/water separation tank according to a third embodiment of the present invention.
[0033] FIG. **6** is a schematic view of a neutralization/water separation tank according to a fourth embodiment of the present invention.

MODE FOR CARRYING OUT THE INVENTION

[0034] Advantages and features of the present invention, and implementation methods thereof will be clarified through following embodiments described with reference to the accompanying drawings. The present invention can, however be embodied in different forms and should not be construed as limited to the embodiments set forth herein. Rather, these embodiments are provided so that this disclosure will be thorough and complete, and will fully convey the scope of the present invention to those skilled in the art. Further, the present invention is only defined by scopes of claims. Like reference numerals refer to like elements throughout.
[0035] Unless terms used in the present invention are defined differently, all terms (including technical and scientific terms) used herein have the same meaning as generally understood by those skilled in the art. Also, unless defined clearly and apparently in the description, the terms as defined in a commonly used dictionary are not ideally or excessively construed as having formal meaning.
[0036] In the following description, the technical terms are used only for explaining a specific exemplary embodiment while not limiting the present invention. In this specification, the terms of a singular form may include plural forms unless specifically mentioned. The meaning of “comprises” and/or “including” does not exclude other components besides a mentioned component.
[0037] Hereinafter, preferred embodiments will be described in detail with reference to the accompanying drawings.
[0038] FIG. **1** is a schematic view of a neutralization/water separation tank **3**, which is combined to allow both neutralization and water separation to occur together, according to a related art.
[0039] In order to prepare a plasticizer, a mixing step, a neutralizing step, a water-separating step, a purifying step, and a filtering step are largely performed. In the mixing step, carboxylic acid and alcohol are mixed to cause an esterification reaction, thereby producing a crude product mixture containing the alcohol and the ester compound. In the neutralizing step, a neutralizing agent that is a basic aqueous solution is mixed to the crude product mixture to neutralize the crude product mixture. In the water-separating step, the neutralized mixture is divided into a floating layer **22** containing organic matters and an aqueous layer **23** containing salt **24**. In the purifying step, when the resultant product is discharged from the floating layer **22**, the residual alcohol is removed. Then, in the filtering step, the resultant product is filtered using a filter to acquire a plasticizer that is a final product.
[0040] In the various steps, as illustrated in FIG. **1**, the neutralization and water separation method according to the related art are performed in a neutralization/water separation tank **3** having the form of a single combined container. In addition, the inside of the neutralization/water separation tank **3** is divided into a first space **31** and a second space **32** by a partition wall **33**. The partition wall **33** extends upward from a bottom surface **34** of the neutralization/water separation tank **3** to form an upper passage **331**.
[0041] First, the crude product mixture is put into the first space **31** of the neutralization/water separation tank **3** through a first inlet **351**, and the neutralizing agent, which is the basic aqueous solution, and water are also put into the first space **31** through a second inlet **352**. Then, a circulation pump **36** connected to the first space **31** operates to mix and neutralize the crude product mixture, the neutralizing agent, and the water, thereby producing a neutralized mixture **21**. Then, immediately, the mixture **21** is divided into the floating layer **22** containing the organic matters and the aqueous layer **23** containing the salt **24**. When a water level of the neutralized

mixture **21** is higher than that of the partition wall **33**, the floating layer **22** overflows to flow into the second space **32** through the upper passage **331** disposed above the partition wall **33**. Also, a first discharge pump **371** connected to the second space **32** operates to discharge the resultant product from the floating layer **22** to the outside. The salt **24** precipitated in the first space **31** does not flow into the second space **32** through the upper passage **331** and is precipitated in a lower portion of the first space **31**. The precipitated salt **24** is discharged from the aqueous layer **23** to the outside by a second discharge pump **372** connected to the first space **31**.

[0042] However, when external energy is applied to the neutralized mixture **21** within the first space **31** so that the neutralized mixture **21** quickly flows, the neutralization reaction may occur well. When the external energy is not applied to the neutralized mixture **21** to leave the neutralized mixture **21**, the water separation may occur well. That is, since the conditions in which the neutralization reaction and the water separation occur well are contradicted, both the neutralization reaction and the water separation may not occur well, and thus, there is a problem that the efficiency is deteriorated.

[0043] FIG. **2** is a schematic view of a neutralization/water separation device **3***a*, in which a neutralization tank **31***a* and a water separation tank **32***a* are separated from each other, according to the related art.

[0044] In order to solve the above problem, as illustrated in FIG. **2**, the neutralization tank **31***a* and the water separation tank **32***a* are separately separated from each other. That is, a crude product mixture is put into a neutralization tank **31***a* of the neutralization/water separation tank **3***a* through a first inlet **351***a*, and a neutralizing agent, which is a basic aqueous solution, and water are also put into the neutralization tank **31***a* through a second inlet **352***a*. Then, a circulation pump **36***a* connected to the neutralization tank **31***a* operates to mix and neutralize the crude product mixture, the neutralizing agent, and the water, thereby producing a neutralized mixture **21**. Also, the neutralized mixture **21** is transferred to the water separation tank **32***a* by using a transfer pump **38**. The inside of the water separation tank **32***a* is divided into a first space **321** and a second space **322** by a partition wall **33***a*. Also, the partition wall **33***a* extends upward from a bottom surface of the water separation tank **32***a* to form an upper passage **331***a*.

[0045] When the neutralized mixture **21** is put into the first space **321** through the transfer pump **38**, the neutralized mixture **21** is immediately divided into a floating layer **22** containing organic matters and an aqueous layer **23** containing salt **24**. When a water level of the neutralized mixture **21** is higher than that of the partition wall **33***a*, the floating layer **22** overflows to flow into the second space **322** through the upper passage **331***a* disposed above the partition wall **33***a*. Also, a first discharge pump **371** connected to the second space **322** operates to discharge the resultant product from the floating layer **22** to the outside. The precipitated salt **24** is discharged from the aqueous layer **23** to the outside by a second discharge pump **372** connected to the first space **321**. Thus, in the neutralization tank **31***a*, external energy is applied to the neutralized mixture **21**, and thus the neutralization reaction occurs well. In the water separation tank **32***a*, the external energy is not applied to the neutralized mixture **21** to leave the neutralized mixture **21**, and thus the water separation occurs well to improve efficiency.

[0046] However, since the neutralization/water separation device **3***a* requires a separate transfer pump **38**, there is a problem that a total volume increases, and the structure is complicated. In addition, when an operation of the neutralization/water separation device **3***a* is stopped, the neutralized mixture **21** may remain in the transfer pump **38**, and the salt **24** may be precipitated. When the salt **24** is accumulated in the transfer pump, a pressure inside the transfer pump increases, and thus, the efficiency and lifespan of the pump decrease.

[0047] FIG. **3** is a schematic view of a neutralization/water separation tank **1** according to a first embodiment of the present invention.

[0048] According to a first embodiment of the present invention, since a neutralization part **11**, in which a neutralization reaction occurs, and a water separation part **12** in which water separation

occurs, are provided to be separated from each other, both the neutralization reaction and the water separation may occur well to improve efficiency. In addition, since the neutralization part **11** and the water separation part **12** are combined into one neutralization/water separation tank **1**, a separate transfer pump is not required, and thus, a total volume does not increase, and a structure is simplified, and salt **24** is prevented from being accumulated.

[0049] For this, the neutralization/water separation tank **1** for an esterified product according to an embodiment of the present invention includes: a neutralization part **11** in which a crude product mixture containing alcohol and an ester compound, a neutralizing agent, and water are put to produce a neutralized mixture **21**; a water separation part **12** which divides the neutralized mixture **21** into a floating layer **22** and an aqueous layer **23**; a first partition wall **131** which extends upward from a bottom surface **14** to provide an upper passage **1311** and by which the neutralization part **11** and the water separation part **12** are partitioned; and a second partition wall **132** which extends downward from a ceiling **14***a* to provide a lower passage **1321** in the water separation part **12**. The water separation part **12** includes: a first water separation part **121** into which the neutralized mixture **21** is introduced from the neutralization part **11** through the upper passage **1311**; and a second water separation part **122** into which the neutralized mixture **21** is introduced from the first water separation part **121** through the lower passage **1321**, and the first water separation part **121** and the second water separation part **122** are partitioned by the second partition wall **132**.

[0050] In order to prepare a plasticizer as described above, it is first subjected to a mixing step. In the mixing step, when carboxylic acid and alcohol are mixed to cause an esterification reaction, a crude product mixture containing the alcohol and the ester compound is produced.

[0051] Here, the carboxylic acid can be an alkyl carboxylic acid having 2 to 24 carbon atoms, a cycloalkyl carboxylic acid having 3 to 24 carbon atoms, an aromatic carboxylic acid having 6 to 24 carbon atoms, or a mixture thereof. For example, the carboxylic acid can be selected from caprylic acid, caproic acid, lauric acid, octanoic acid, decanoic acid, dodecanoic acid, ethanoic acid (acetic acid), propionic acid, butyric acid, pentanoic acid, hexanoic acid, ethylhexanoic acid, cyclohexane carboxylic acid, benzoic acid, cyclohexane 1,2-dicarboxylic acid, cyclohexane 1,3-dicarboxylic acid, cyclohexane 1,4-dicarboxylic acid, terephthalic acid, isophthalic acid, phthalic acid, and a combination thereof, preferably can be cyclohexane 1,2-dicarboxylic acid, cyclohexane 1,3-dicarboxylic acid, cyclohexane 1,4-dicarboxylic acid, terephthalic acid, isophthalic acid, phthalic acid, or combinations thereof, but is not limited thereto.

[0052] The alcohol can be aliphatic alcohol having a C1-C20 alkyl group, preferably aliphatic primary alcohol having a C3-C10 alkyl group. Here, the alcohols can have linear or branched alkyl and be alcohol mixed between structural isomers, and alcohols having different carbon numbers can be added as a mixture.

[0053] The esterification reaction of the alcohol component and the carboxylic acid can be performed in the presence of a catalyst. A material commonly used in the esterification reaction of alcohol can be used as the catalyst. For example, the catalyst can be one or more selected from acid catalysts such as sulfuric acid, hydrochloric acid, phosphoric acid, nitric acid, paratoluenesulfonic acid, methanesulfonic acid, ethanesulfonic acid, propanesulfonic acid, butanesulfonic acid, and alkyl sulfuric acid, metal salts such as aluminum lactate, lithium fluoride, potassium chloride, cesium chloride, calcium chloride, iron chloride, and aluminum phosphate, metal oxides such as heteropolyacids, and organic metals such as natural/synthetic zeolites, cation and anion exchange resins, tetraalkyl titanate, and polymers thereof, but is not limited thereto.

[0054] Next, a neutralizing step of neutralizing the crude product mixture with the basic neutralizing agent and a water-separating step of dividing the neutralized mixture into a floating layer **22** and an aqueous layer **23** are performed. As illustrated in FIG. **3**, the neutralization/water separation method for the esterified product according to the first embodiment of the present invention is performed in the neutralization/water separation tank **1** having the form of a single combined container. In addition, the inside of the neutralization/water separation tank **1** is divided

into the neutralization part **11** and the water separation part **12** by the first partition wall **131**.
[0055] The neutralization part **11** provides a space in which the crude product mixture containing the alcohol and the ester compound, the neutralizing agent, and the water are put to produce the neutralized mixture **21**. As illustrated in FIG. **3**, a circulation pump **16** configured to discharge the neutralized mixture **21** from the neutralization part **11** and put the neutralized mixture **21** again into the neutralization part **11** can be connected to the neutralization part **11**. The circulation pump **16** can operate so that the neutralized mixture **21** is quickly and uniformly stirred and neutralized.
[0056] The water separation part **12** provides a space in which the neutralized mixture **21** is accommodated to be divided into the floating layer **22** and the aqueous layer **23**. Here, the floating layer **22** is a portion that contains organic matters to serve as a plasticizer later, and the aqueous layer **23** is a portion that contains water and salt **24**, which are produced by the neutralization reaction.
[0057] The first partition wall **131** partitions the neutralization part **11** from the water separation part **12**. Thus, external energy may be applied to only the neutralization part **11**, and thus, the neutralization reaction and the water separation may occur more efficiently. The first partition wall **131** extends upward from a bottom surface **14** of the neutralization/water separation tank **1** to form the upper passage **1311** thereabove. That is, the neutralization part **11** and the water separation part **12** are not completely isolated by the first partition wall **131** and are connected to each other through the upper passage **1311**. Therefore, when a water level of the neutralized mixture **21** neutralized in the neutralization part **11** is higher than the first partition wall **131**, the neutralized mixture **21** overflows to flow to the water separation part **12** through the upper passage **1311** disposed above the first partition wall **131**.
[0058] It is preferable that the upper passage **1311** has a height of 10% to 50% of the height of the first partition wall **131**. If the upper passage **1311** has a height lower than 10% of the height of the first partition wall **131**, an excessively large amount crude of product mixture, neutralizing agent, and water may be put to cause an overflow of the neutralized mixture **21**. Also, if the upper passage **1311** has a height higher than 50% of the height of the first partition wall **131**, the neutralized mixture **21** may be immediately introduced into the water separation part **12** before the neutralization reaction proceeds sufficiently.
[0059] The inside of the water separation part **12** is divided into a first water separation part **121** and a second water separation part **122** by the second partition wall **132**. The first water separation part **121** provides a space by which the neutralization part **11** and the second water separation part **122** are spaced apart from each other. In addition, the second water separation part **122** provides a space in which the neutralized mixture **21** is accommodated to be divided into the floating layer **22** and the aqueous layer **23**. The neutralized mixture **21** has large kinetic energy to flow from the neutralization part **11** into the first water separation part **121** through the upper passage **1311**. Also, the neutralized mixture **21** flows from the first water separation part **121** into the second water separation part **122** through the lower passage **1321**. Here, since the neutralization part **11** and the second water separation part **122** are spaced apart from each other by the first water separation part **121**, the kinetic energy of the neutralized mixture **21** is reduced in the second water separation part **122**. Thus, the water separation of the neutralized mixture **21** may occur well so that the neutralized mixture **21** is more quickly and clearly divided into the floating layer **22** and the aqueous layer **23**.
[0060] The second partition wall **132** partitions the first water separation part **121** from the second water separation part **122**. As a result, when the neutralized mixture **21** is put into the second water separation part **122**, the kinetic energy may be significantly reduced, and thus the water separation may occur more efficiently. The second partition wall **132** extends downward from a ceiling **14***a* of the neutralization/water separation tank **1** to form a lower passage **1321** therebelow. That is, the first water separation part **121** and the second water separation part **122** are not completely isolated by the second partition wall **132** and are connected to each other through the lower passage **1321**. Thus, the neutralized mixture **21** put into the first water separation part **121** naturally flows into the

second water separation part **122** through the lower passage **1321** disposed below the second partition wall **132**.

[0061] It is preferable that the lower passage **1321** has a height of 10% to 50% of the height of the second partition wall **132**. If the lower passage **1321** has a height lower than 10% of the height of the second partition wall **132**, a flow amount of neutralized mixture **21** may be significantly reduced, and an entire process time may be excessively consumed. Also, if the lower passage **1321** has a height higher than 50% of the height of the second partition wall **132**, since the neutralization part **11** and the second water separation part **122** are not sufficiently spaced apart from each other by the first water separation part **121**, the kinetic energy of the neutralized mixture **21** flowing into the second water separation part **122** may not be significantly reduced, and thus the efficiency of the water separation may be deteriorated.

[0062] A first discharge pump **171** for discharging the resultant product contained in the floating layer **22** to the outside can be connected to a sidewall of the second water separation part **122**. The first discharge pump **171** is connected to the sidewall of the second water separation part **122** at a height corresponding to the position at which the floating layer **22** is floated. Since the amount of crude product mixture, neutralizing agent, and water to be put and the amount of resultant product to be discharged by the first discharge pump **171** can be adjusted through a first inlet **151** and a second inlet **152**, the position at which the floating layer **22** is floated can be easily adjusted. That is, when the amount to be put increases, or the amount to be discharged decreases, the position at which the floating layer **22** is floated may increase in height, and when the amount to be put decreases, or the amount to be discharged increases, the position at which the floating layer **22** is floated may decrease in height. However, since it is preferable that the floating layer **22** is floated to a position higher than that of at least the lower passage **1321**, the first discharge pump **171** can be connected to a position higher than the lower passage **1321**.

[0063] A second discharge pump **172** for discharging the precipitated salt **24** contained in the aqueous layer **23** to the outside can be connected to the bottom surface **14** of the water separation part **12**. While the neutralized mixture **21** flows through the first water separation part **121** and the second water separation part **122**, the neutralized mixture **21** can be divided into the floating layer **22** and the aqueous layer **23**, and the salt **24** contained in the aqueous layer **23** can be precipitated in a lower portion of the water separation part **12**. The salt **24** is a compound in which an acidic anion and a basic cation are generated while the crude product mixture is neutralized by the neutralizing agent. Thus, the second discharge pump **172** is connected to the bottom surface **14** of the second water separation part **122** to discharge the precipitated salt **24** from the aqueous layer **23** to the outside.

[0064] A neutralization/water separation method for esterified product according to an embodiment of the present invention includes: a step of putting a crude product mixture containing alcohol and an ester compound, a neutralizing agent, and water into a neutralization part **11** of a neutralization/water separation tank **1**; a step of mixing the crude product mixture, the neutralizing agent, and the water to produce a neutralized mixture **21**; a step of introducing the neutralization mixture **21** into a first water separation part **121** of the water separation part **12** through an upper passage **1311**; a step of introducing the neutralization mixture **21** into a second water separation part **122** of the water separation part **12** through a lower passage **1321**; a step of dividing the neutralization mixture **21** into a floating layer **22** and an aqueous layer **23**; and a step of discharging the resultant product contained in the floating layer **22** to the outside.

[0065] Particularly, first, the crude product mixture containing the alcohol and the ester compound is put into the neutralization part **11** of the neutralization/water separation tank **1** through a first inlet **151**, and the neutralizing agent, which is the basic aqueous solution, and water are also put into the neutralization part **11** through a second inlet **152**. Also, the crude product mixture, the neutralizing agent, and the water are mixed with each other to be neutralized, thereby producing the neutralized mixture **21**. An aqueous solution of a basic substance such as alkali metal hydroxide,

alkaline earth metal hydroxide, or a mixture thereof can be used as the neutralizing agent. For example, an aqueous NaOH solution or an aqueous KOH solution can be used as the neutralizing agent.

[0066] Also, a circulation pump **16** connected to the neutralization part **11** operates. The circulation pump **16** discharges the neutralized mixture **21** from the neutralization part **11** and then puts the neutralized mixture **21** again into the neutralization part **11**. Thus, the neutralized mixture **21** can be quickly and uniformly neutralized.

[0067] The crude product mixture can be separately put into the neutralization tank through the first inlet **151**, and the neutralizing agent and the water can be separately put into the neutralization tank through the second inlet **152**. However, as illustrated in FIG. **3**, the first inlet **151** and the second inlet **152** can be connected to each other while the crude product mixture, the neutralizing agent, and the water are put. As a result, the crude product mixture, the neutralizing agent, and the water can be previously mixed before being put into the neutralization tank and then be put into the neutralization tank through a third inlet **153**. Thus, the neutralized mixture **21** can be more quickly and uniformly neutralized when compared to a case in which the circulation pump **16** operates solely to neutralize the neutralized mixture **21**, as described later.

[0068] When a water level of the neutralized mixture **21** neutralized in the neutralization part **11** is higher than the first partition wall **131**, the neutralized mixture **21** overflows to flow to the water separation part **12**, particularly the first water separation part **121** through the upper passage **1311** disposed above the first partition wall **131**. Also, the neutralized mixture **21** put into the first water separation part **121** naturally flows into the second water separation part **122** through the lower passage **1321** disposed below the second partition wall **132**.

[0069] As described above, the first water separation part **121** provides a space by which the neutralization part **11** and the second water separation part **122** are spaced apart from each other. Thus, since kinetic energy of the neutralized mixture **21** introduced into the second water separation part **122** decreases, the neutralized mixture **21** is more quickly and clearly divided into the floating layer **22** and the aqueous layer **23**. Also, a first discharge pump **171** connected to a sidewall of the second water separation part **122** operates to discharge the resultant product from the floating layer **22** to the outside. The discharged resultant product can be subjected to a purifying step of removing alcohol, which is the following process, and a filtering step to produce a final product, and the final product can be applied as a plasticizer. Also, a second discharge pump **172** connected to a bottom surface **14** of the water separation part **12** discharges the salt **24** contained in the aqueous layer **23** to the outside.

[0070] In the above-described method, both the neutralization reaction and the water separation occur well to improve efficiency. In addition, since the neutralization part **11** and the water separation part **12** are combined into one neutralization/water separation tank **1**, a separate transfer pump is not required, and thus, a total volume does not increase, and a structure is simplified, and salt **24** is prevented from being accumulated.

[0071] FIG. **4** is a schematic view of a neutralization/water separation tank **1***a* according to a second embodiment of the present invention.

[0072] According to a first embodiment of the present invention, a circulation pump **16** is connected to a neutralization part **11**. The circulation pump **16** discharges a neutralized mixture **21** from the neutralization part **11** and then puts the neutralized mixture **21** again into the neutralization part **11**. As a result, the neutralized mixture **21** can be quickly and uniformly stirred and neutralized.

[0073] However, as illustrated in FIG. **4**, in the neutralization/water separation tank la according to the second embodiment of the present invention, a propeller stirrer **18** is connected to the neutralization part **11**. The propeller stirrer **18** is a stirrer in which at least one propeller is disposed on a long rod so as to be immersed in a liquid, and the rod rotates to stir the liquid. As illustrated in FIG. **4**, the propeller stirrer **18** can be provided in two, but is not limited thereto. For example,

various numbers of propellers can be provided. Also, the propeller stirrer **18** can be installed substantially perpendicular to the neutralization part **11**, but is not limited thereto. For example, the propeller stirrer **18** can be installed in various manner such as being installed to be inclined or horizontally installed.

[0074] Since the propeller stirrer **18** is installed, a total volume of the neutralization/water separation tank **1** can be reduced when compared to a total volume of the circulation pump **16**, the installation may be easy, and the neutralized mixture **21** can be stirred more quickly and uniformly.

[0075] According to the first embodiment of the present invention, a first inlet **151** and a second inlet **152** are connected to each other during the process so that the crude product mixture, the neutralizing agent, and the water are mixed previously and then put into the neutralization tank through the third inlet **153**. However, according to the second embodiment of the present invention, since the propeller stirrer **18** is used instead of the circulation pump **16**, the neutralized mixture **21** can be stirred more quickly and uniformly. Thus, the first inlet **151***a* and the second inlet **152***a* do not need to be connected to each other during the process so that the crude product mixture is separately put into the neutralization tank through the first inlet **151***a*, and the neutralizing agent and the water are separately put into the neutralization tank through the second inlet **152***a*.

[0076] FIG. **5** is a schematic view of a neutralization/water separation tank **1***b* according to a third embodiment of the present invention.

[0077] According to the first and second embodiments of the present invention, the bottom surface **14** of each of the neutralization/water separation tanks **1** and la is flat. However, if the bottom surface **14** of the water separation part **12** is flat, the precipitated salt **24** is dispersed on the bottom surface **14** of the water separation part **12**. Thus, it may not be easy to allow the second discharge pump **172** to discharge the salt **24** to the outside.

[0078] In the e neutralization/water separation tank **1***b* according to the third embodiment of the present invention, a bottom surface **141** of the water separation part **12***a* can be gradually inclined toward a center thereof as illustrated in FIG. **5**. That is, the bottom surface **141** of the water separation part **12***a* can have a conical shape. Also, a second discharge pump **172** can be connected to the center of the bottom surface **141** of the water separation part **12***a*. Thus, when the salt **24** is precipitated, the salt **24** is collected to the center of the bottom surface **141** of the water separation part **12***a*, and the second discharge pump **172** can easily discharge the precipitated salt **24** to the outside.

[0079] When an operation of the neutralization/water separation tank **1** is stopped, the salt **24** can also be precipitated in the neutralization part **11**. However, according to the first and second embodiments of the present invention, since the bottom surface **14** of the neutralization part **11** is flat, the precipitated salt **24** is dispersed on the bottom surface **14** of the neutralization part **11**. Particularly, when the neutralization/water separation tank **1** is stopped for a long time, the salt **24** precipitated at a corner of the bottom surface **14** of the neutralization part **11** can be accumulated and solidified. As a result, even if the neutralization/water separation tank **1** operates again, the salt **24** may not be mixed with the neutralized mixture **21** to remain at the corner of the bottom surface **14** of the neutralization tank.

[0080] In the neutralization/water separation tank **1***b* according to the third embodiment of the present invention, a bottom surface **142** of the neutralization part **11***a* can be gradually inclined toward a center thereof as illustrated in FIG. **5**. That is, the bottom surface **142** of the neutralization part **11***a* can also have a conical shape. Also, the circulation pump **16** can be connected to the center of the bottom surface **142** of the neutralization part **11***a*. Thus, even if the neutralization/water separation tank **1** is stopped for a long time, the precipitated salt **24** is collected to the center of the bottom surface **142** of the neutralization part **11***a*. Also, when the neutralization/water separation tank **1** operates again, the circulation pump **16** discharges the neutralized mixture **21** together with the precipitated salt **24** from the neutralization part **11***a* and again puts the neutralized mixture **21** again into the neutralization part **11***a*. Thus, the salt **24** precipitated on the bottom surface **142** of

the neutralization part **11***a* can be prevented from being accumulated and solidified.
[0081] FIG. **6** is a schematic view of a neutralization/water separation tank **1***c* according to a fourth embodiment of the present invention.
[0082] As illustrated in FIG. **6**, in the neutralization/water separation tank **1***c* according to the fourth embodiment of the present invention, a propeller stirrer **18** is connected to a neutralization part **11***a*. Thus, a total volume of the neutralization/water separation tank **1** may be reduced when compared to a total volume of a circulation pump **16**, the installation may be easy, and a neutralized mixture **21** may be stirred more quickly and uniformly.
[0083] Also, a bottom surface **141** of a water separation part **12***a* can be gradually inclined downward toward a center thereof. Also, a second discharge pump **172** can be connected to the center of the bottom surface **141** of a water separation part **12***a*. Thus, when salt **24** is precipitated, the salt **24** is collected to the center of the bottom surface **141** of the water separation part **12***a*, and a second discharge pump **172** can easily discharge the precipitated salt **24** to the outside.
[0084] Also, a bottom surface **142** of a neutralization part **11***a* can be gradually inclined downward toward a center thereof. Also, the circulation pump **16** can be connected to the center of the bottom surface **142** of the neutralization part **11***a*. Therefore, the salt **24** precipitated on the bottom surface **142** of the neutralization part **11***a* can be prevented from being accumulated and solidified.
[0085] Those with ordinary skill in the technical field of the present invention pertains will be understood that the present invention can be carried out in other specific forms without changing the technical idea or essential features. Therefore, the above-disclosed embodiments are to be considered illustrative and not restrictive. Accordingly, the scope of the present invention is defined by the appended claims rather than the foregoing description and the exemplary embodiments described therein. Various modifications made within the meaning of an equivalent of the claims of the invention and within the claims are to be regarded to be in the scope of the present invention.

Claims

1. A neutralization/water separation method for an esterified product, comprising: a step of putting a crude product mixture containing alcohol and an ester compound, a neutralizing agent, and water into a neutralization part of a neutralization/water separation tank; a step of mixing the crude product mixture, the neutralizing agent, and the water to produce a neutralized mixture; a step of introducing the neutralization mixture into a first water separation part of the water separation part through an upper passage; a step of introducing the neutralization mixture into a second water separation part of the water separation part from the first water separation part through a lower passage; a step of dividing the neutralization mixture into a floating layer and an aqueous layer; and a step of discharging s resultant product contained in the floating layer to the outside of the neutralization/water separation tank.
2. The neutralization/water separation method for an esterified product of claim 1, wherein, in the step of producing the neutralized mixture, a circulation pump discharges the neutralized mixture from the neutralization part and puts the neutralized mixture again into the neutralization part.
3. The neutralization/water separation method for an esterified product of claim 1, wherein, in the step of producing the neutralized mixture, the neutralized mixture is stirred using a propeller stirrer.
4. The neutralization/water separation method for an esterified product of claim 1, wherein a bottom surface of the water separation part is gradually inclined downward toward a center thereof, and wherein a precipitated salt contained in the aqueous layer is collected to the center of the bottom surface of the water separation part.